

Supporting Information for

Extending Pummerer Reaction Chemistry. Synthesis Studies in the Phakellin Alkaloid Area.

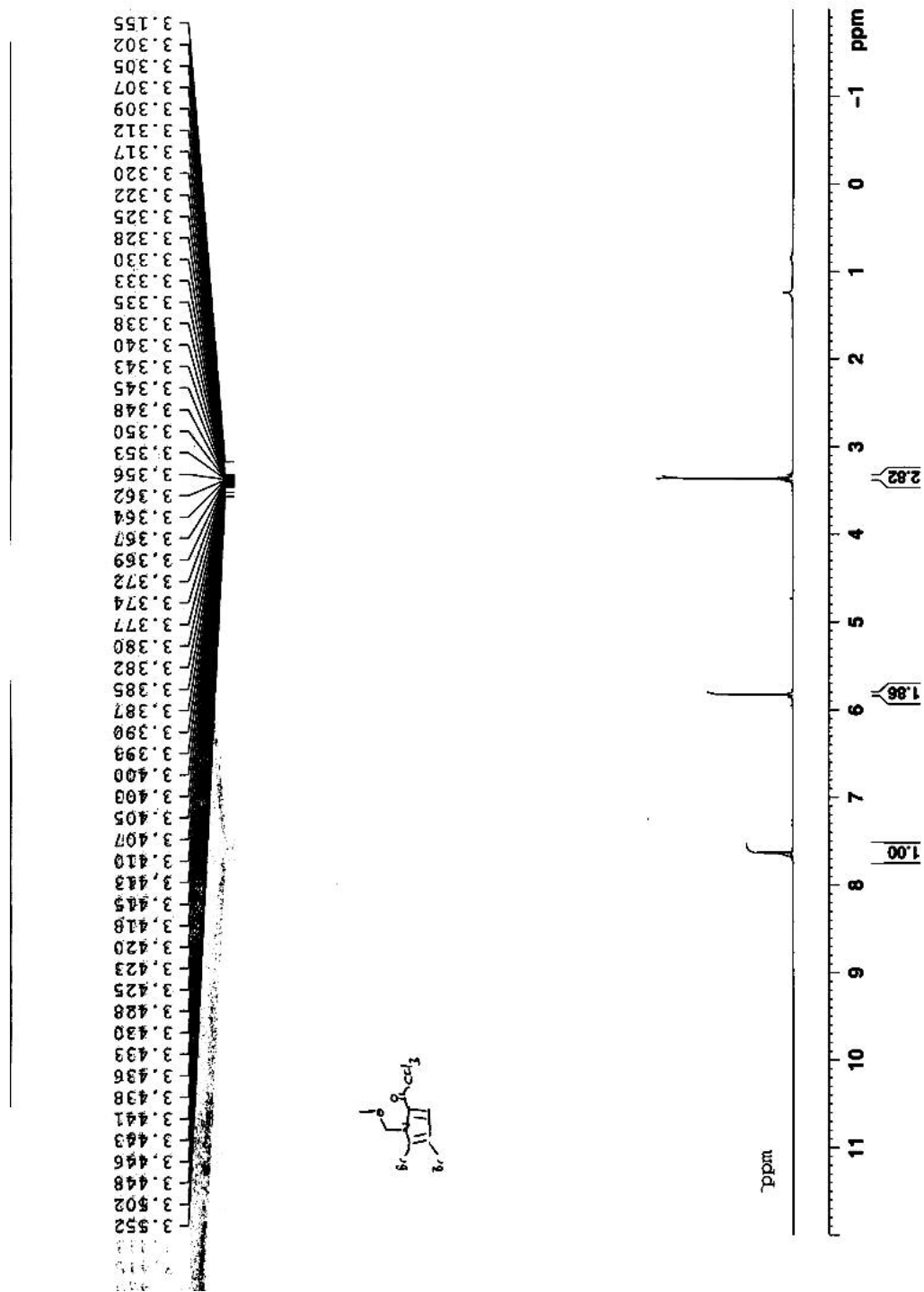
Ken S. Feldman,* Amanda P. Skoumbourdis and Matthew D. Fodor

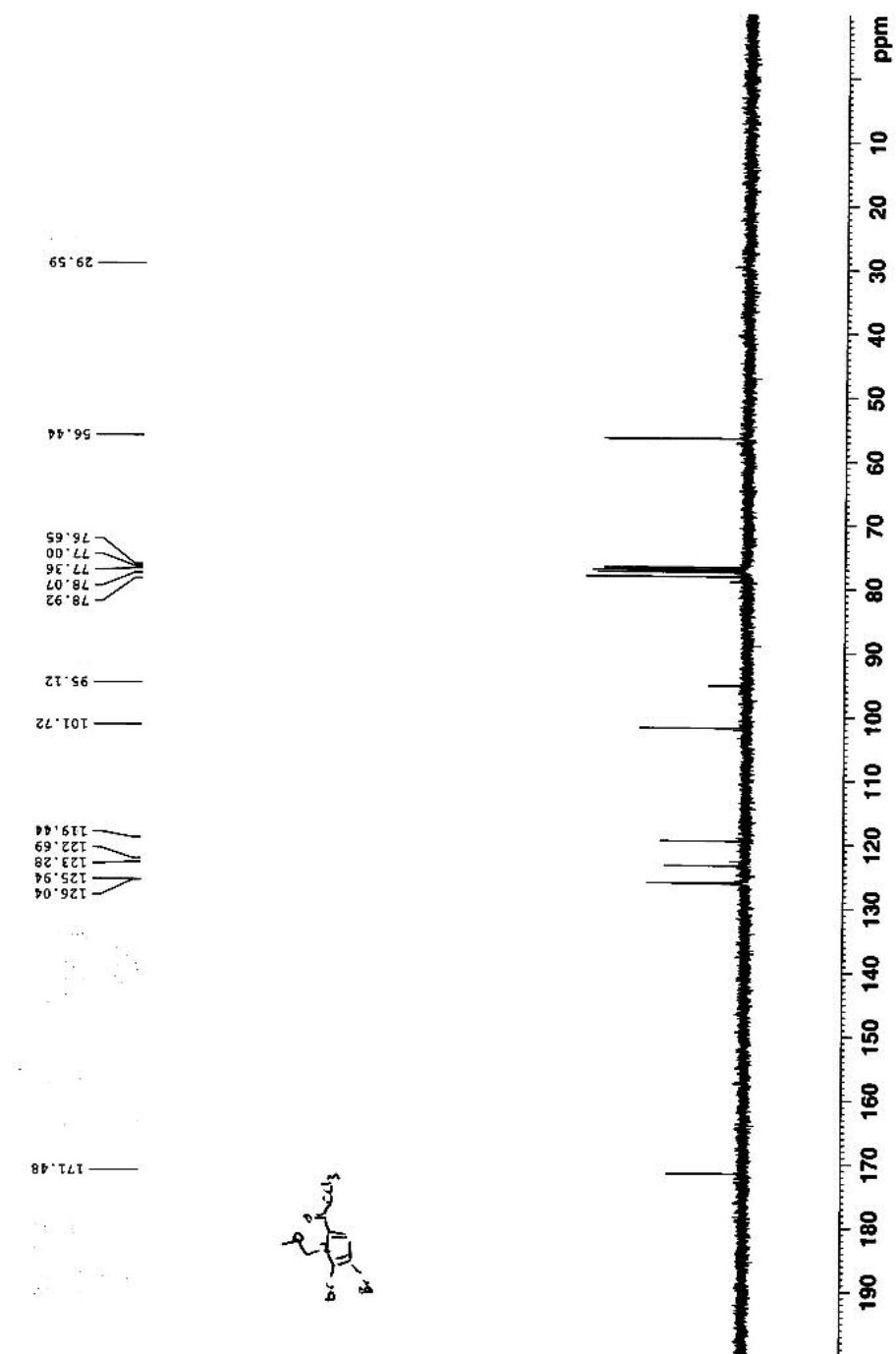
Department of Chemistry, The Pennsylvania State University, University Park, PA 16802

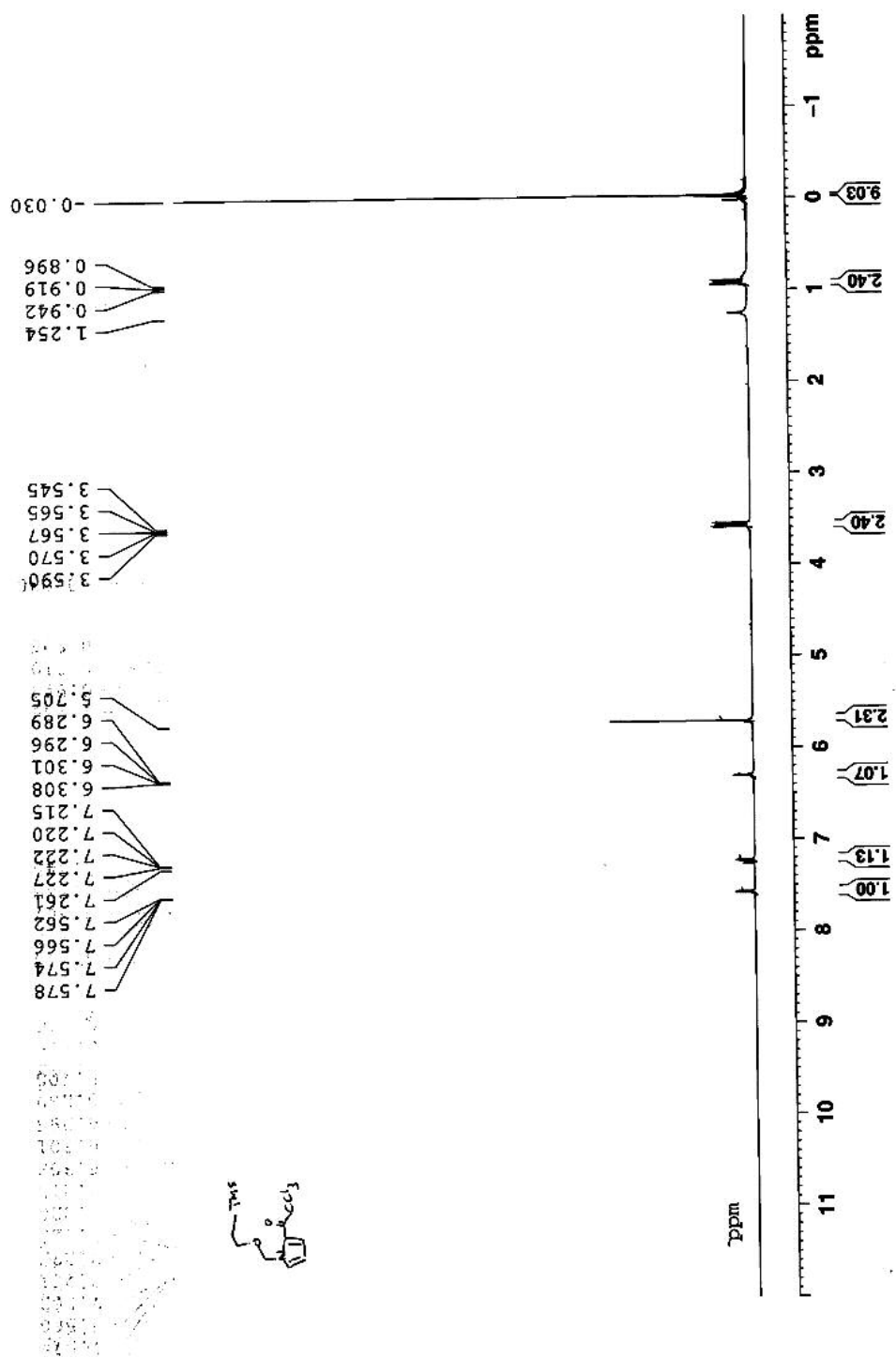
¹H NMR, ¹³C NMR, IR, MS and HRMS for compounds **2a**, **22b**, **24b**, **27**, **33**, **34**, **35c**, **35g**, **35h**, **36a-i**, **37a-i**, **5-(3-Amino-propyl)-2-benzenesulfinyl-imidazole-1-sulfonic Acid Dimethylamide**, **4,5-Dibromo-1H-pyrrole-2-carboxylic Acid [3-(3-dimethylsulfamoyl-2-phenylsulfanyl-3H-imidazol-4-yl)-propyl]-amide**, **4,5-Dibromo-1H-pyrrole-2-carboxylic Acid [3-(2-benzenesulfinyl-3-dimethylsulfamoyl-3H-imidazol-4-yl)-propyl]-amide**

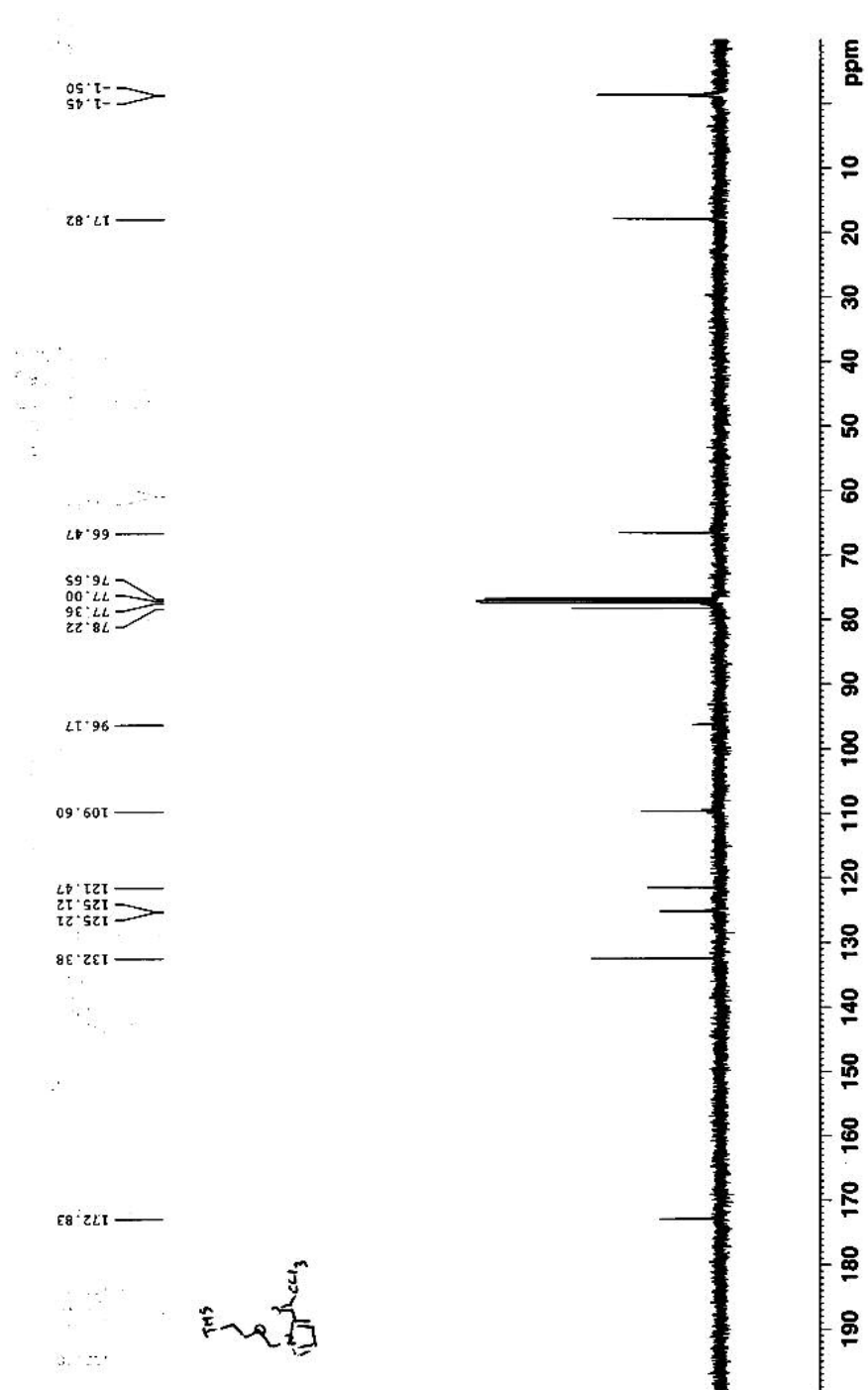
¹ H NMR 2a	S61
¹³ C NMR 2a	S62
¹ H NMR 2a (decomposition in DMSO- <i>d</i> ₆)	S63
¹³ C NMR 2a (decomposition in DMSO- <i>d</i> ₆)	S64
¹ H NMR 22b	S9
¹³ C NMR 22b	S10
¹ H NMR 24b	S55
¹³ C NMR 24b	S56
¹ H NMR 27	S57
¹³ C NMR 27	S58
¹ H NMR 33	S59
¹³ C NMR 33	S60
¹ H NMR 34	S11
¹³ C NMR 34	S12
¹ H NMR 35c	S5
¹³ C NMR 35c	S6
¹ H NMR 35g	S3
¹³ C NMR 35g	S4
¹ H NMR 35h	S7
¹³ C NMR 35h	S8
¹ H NMR 36a	S15
¹³ C NMR 36a	S16
¹ H NMR 36b	S29
¹³ C NMR 36b	S30
¹ H NMR 36c	S33

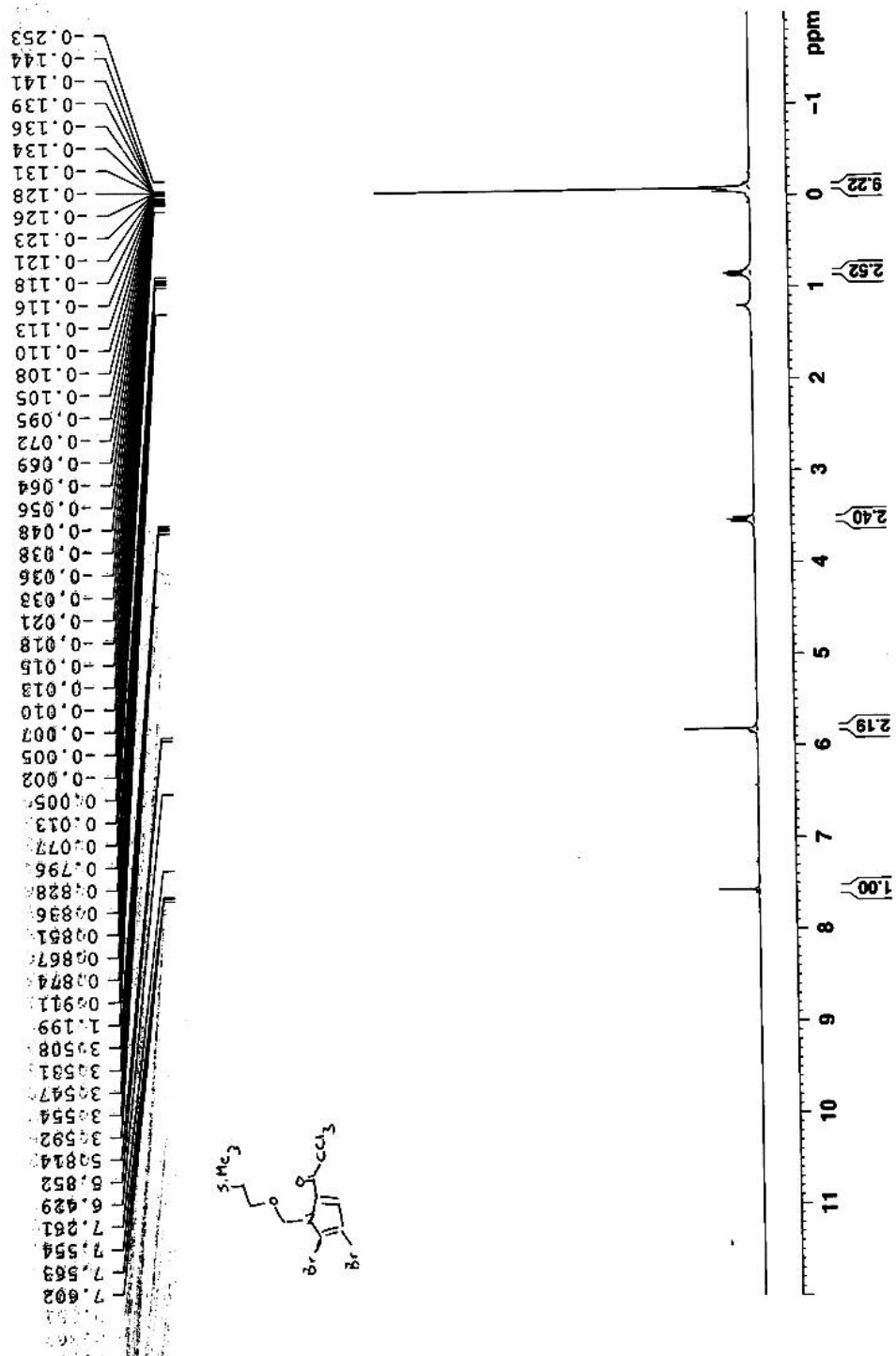
¹³ C NMR 36c	S34
¹ H NMR 36d	S23
¹³ C NMR 36d	S24
¹ H NMR 36e	S17
¹³ C NMR 36e	S18
¹ H NMR 36f	S25
¹³ C NMR 36f	S26
¹ H NMR 36g	S31
¹³ C NMR 36g	S32
¹ H NMR 36h	S35
¹³ C NMR 36h	S36
¹ H NMR 36i	S27
¹³ C NMR 36i	S28
¹ H NMR 37a	S37
¹³ C NMR 37a	S38
¹ H NMR 37b	S47
¹³ C NMR 37b	S48
¹ H NMR 37c	S51
¹³ C NMR 37c	S52
¹ H NMR 37d	S41
¹³ C NMR 37d	S42
¹ H NMR 37e	S39
¹³ C NMR 37e	S40
¹ H NMR 37f	S43
¹³ C NMR 37f	S44
¹ H NMR 37g	S49
¹³ C NMR 37g	S50
¹ H NMR 37h	S53
¹³ C NMR 37h	S54
¹ H NMR 37i	S45
¹³ C NMR 37i	S46
¹ H NMR 5-(3-Amino-propyl)-2-benzenesulfinyl-imidazole-1-sulfonic Acid Dimethylamide	S13
¹³ C NMR 5-(3-Amino-propyl)-2-benzenesulfinyl-imidazole-1-sulfonic Acid Dimethylamide	S14
¹ H NMR 4,5-Dibromo-1H-pyrrole-2-carboxylic Acid [3-(3-dimethylsulfamoyl-2-phenylsulfanyl-3H-imidazol-4-yl)-propyl]-amide	S19
¹³ C NMR 4,5-Dibromo-1H-pyrrole-2-carboxylic Acid [3-(3-dimethylsulfamoyl-2-phenylsulfanyl-3H-imidazol-4-yl)-propyl]-amide	S20
¹ H NMR 4,5-Dibromo-1H-pyrrole-2-carboxylic Acid [3-(2-benzenesulfinyl-3-dimethylsulfamoyl-3H-imidazol-4-yl)-propyl]-amide	S21
¹³ C NMR 4,5-Dibromo-1H-pyrrole-2-carboxylic Acid [3-(2-benzenesulfinyl-3-dimethylsulfamoyl-3H-imidazol-4-yl)-propyl]-amide	S22
General Experimental Procedures	S65

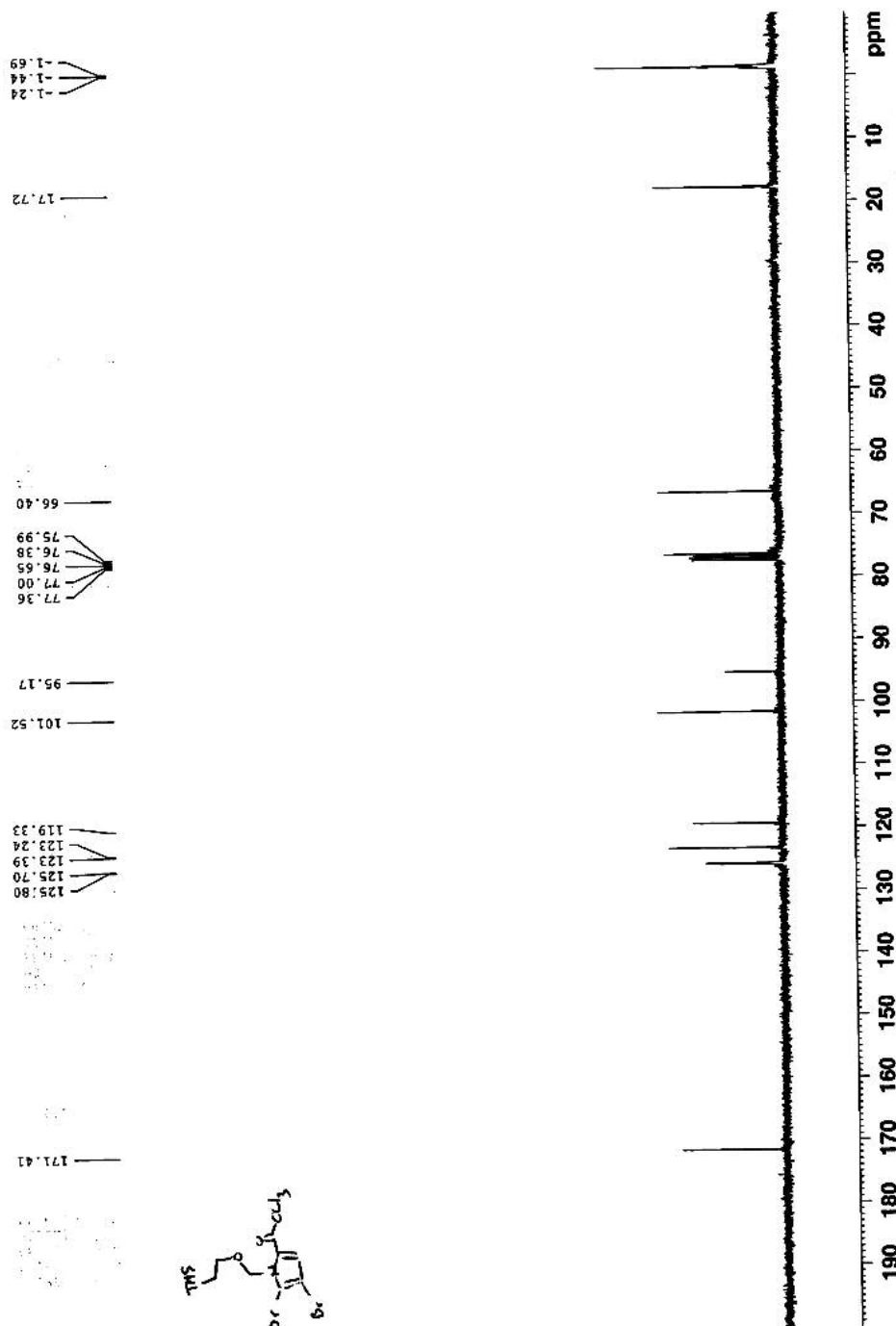




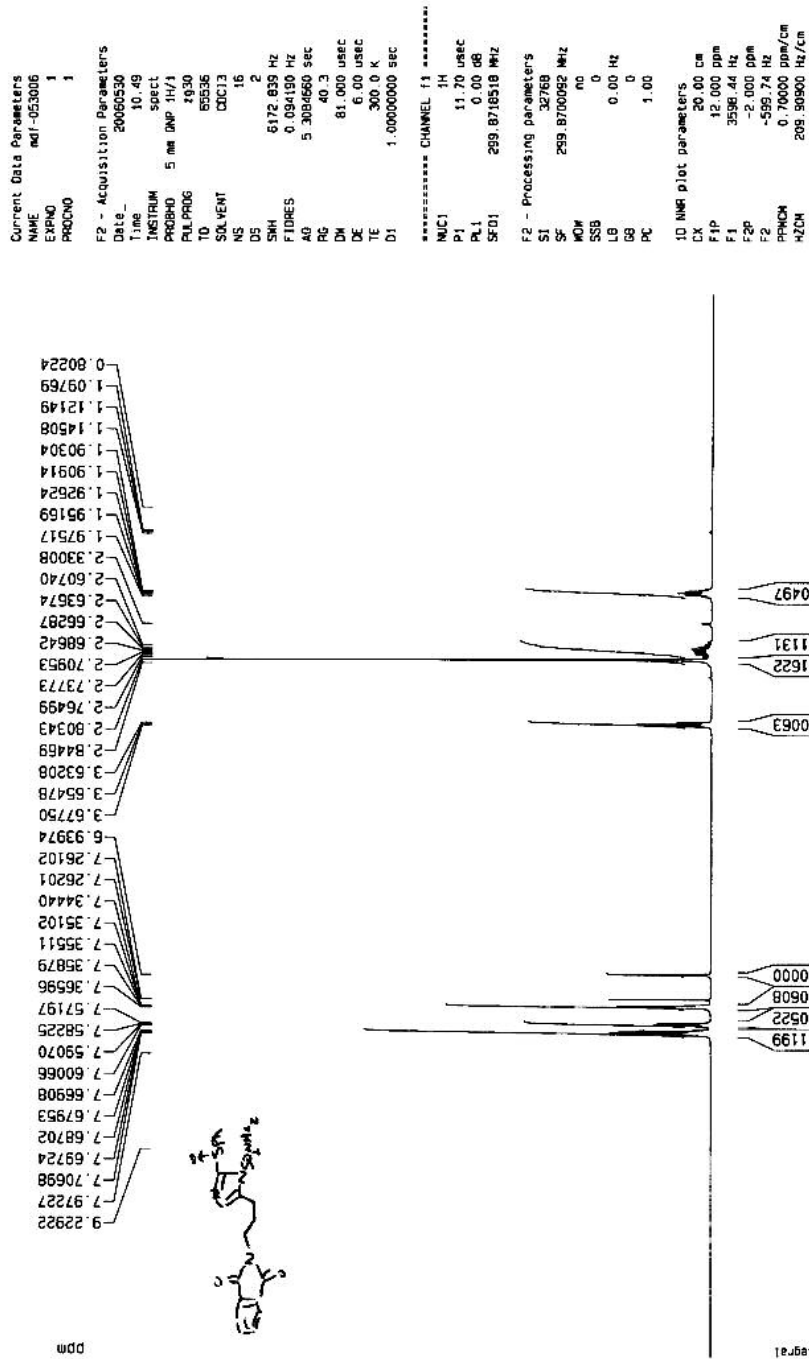




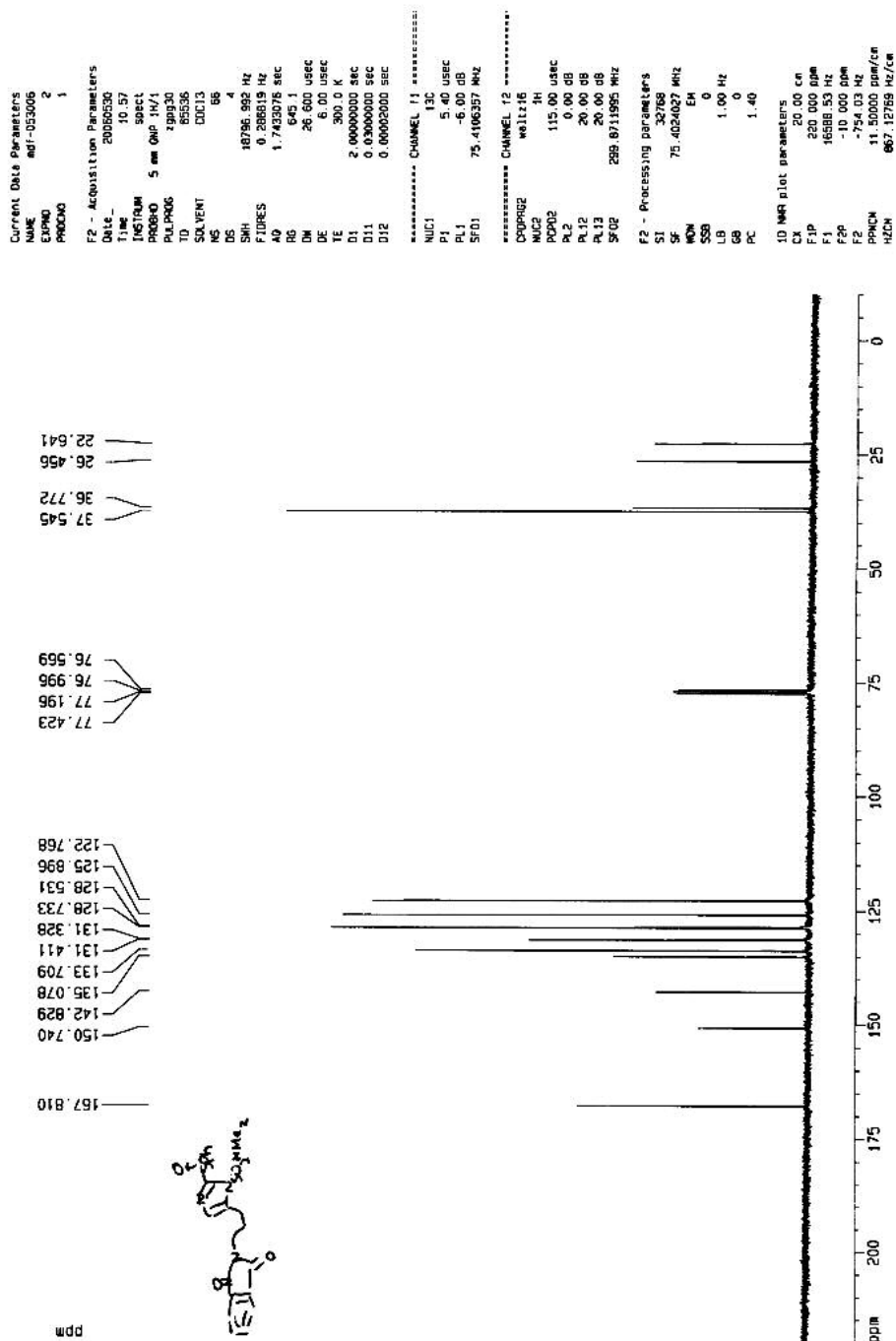




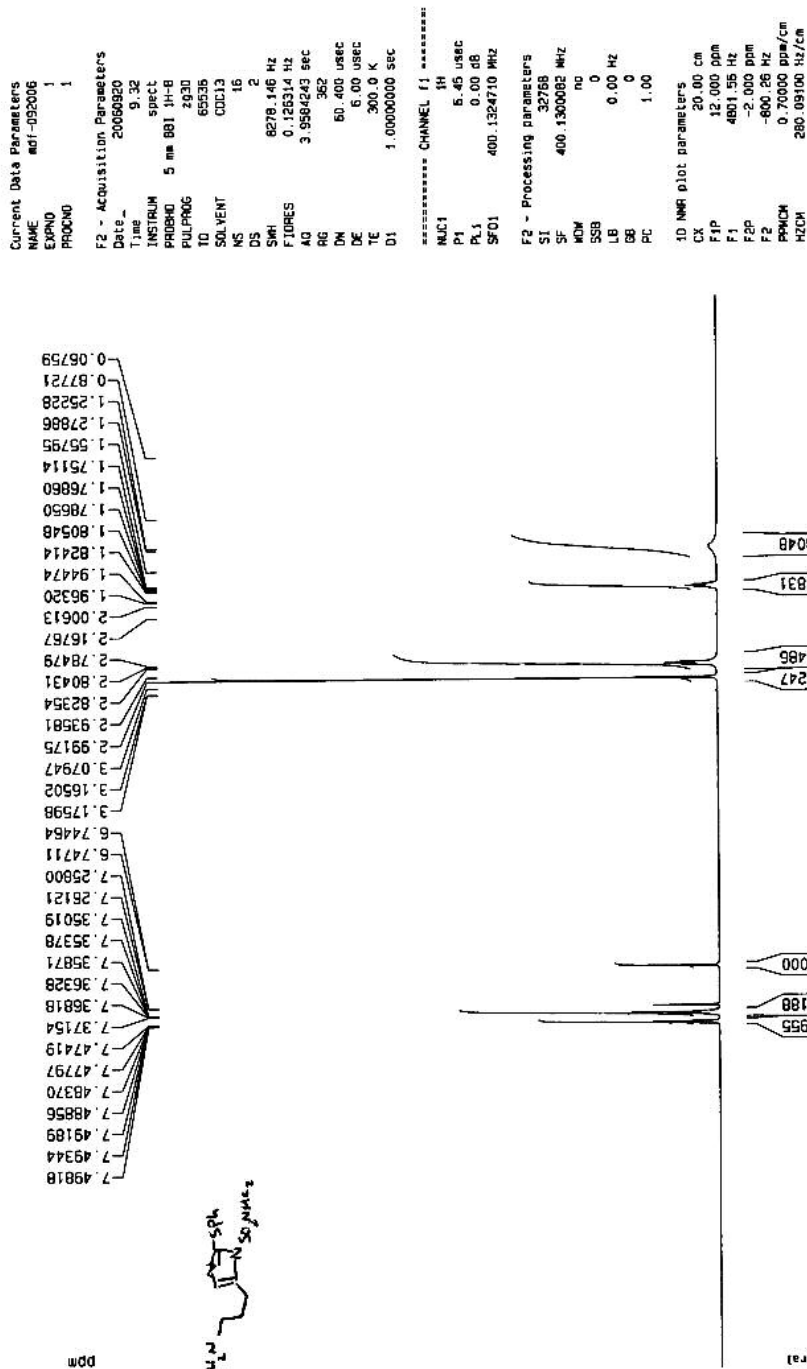
mdf-02-130 phthalimide - sulfoxide - 300MHz

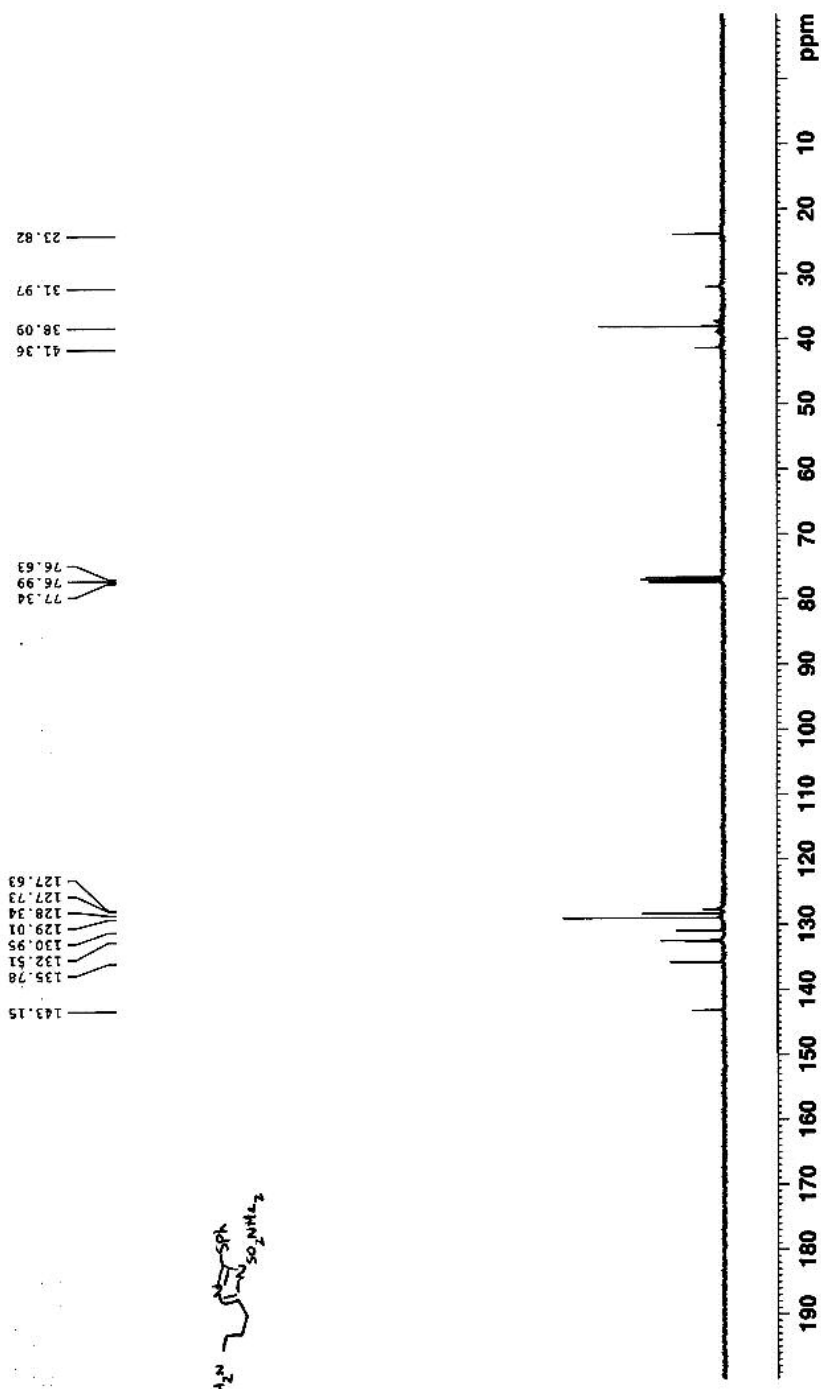


mdf-02-130 phthalimide - sulfoxide - 75 MHz

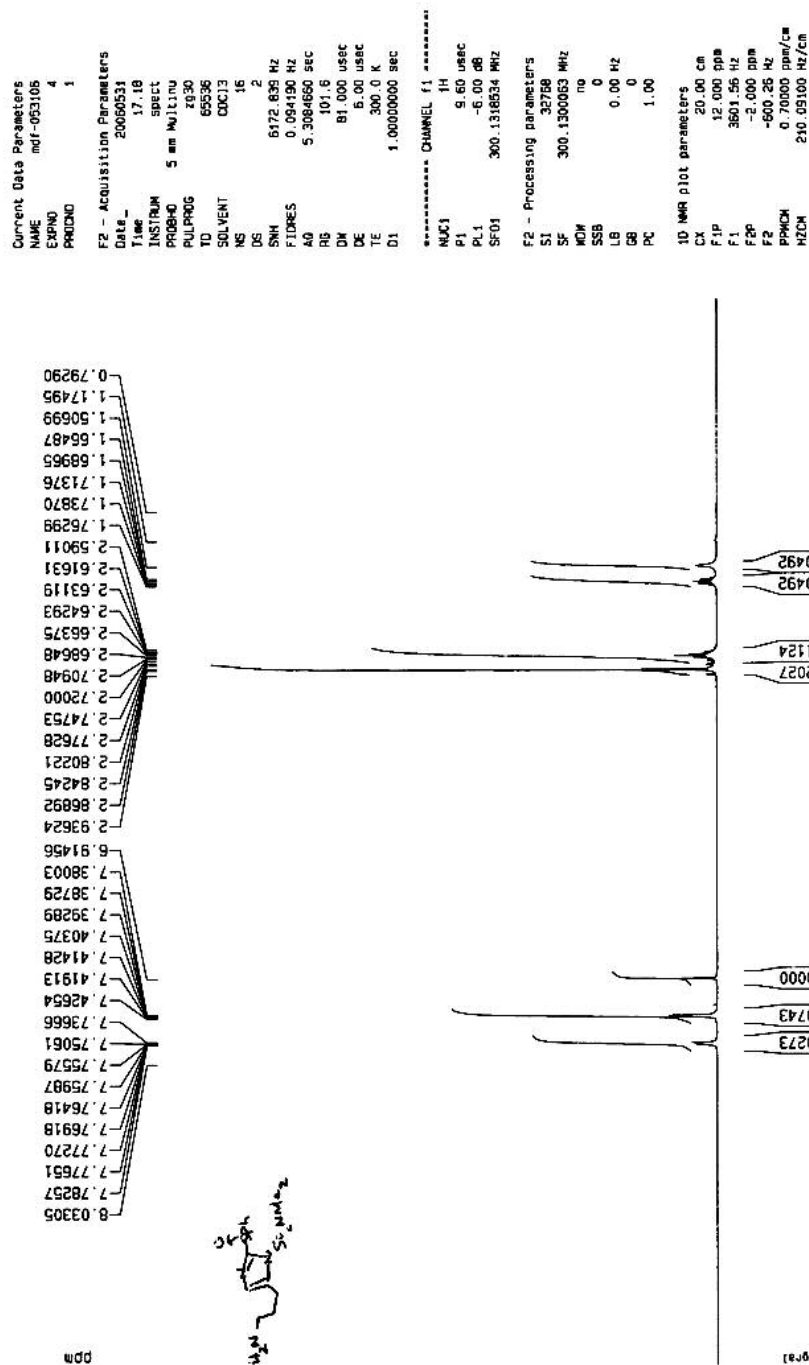


mdf-02-237 protected aminosulfide - 400MHz

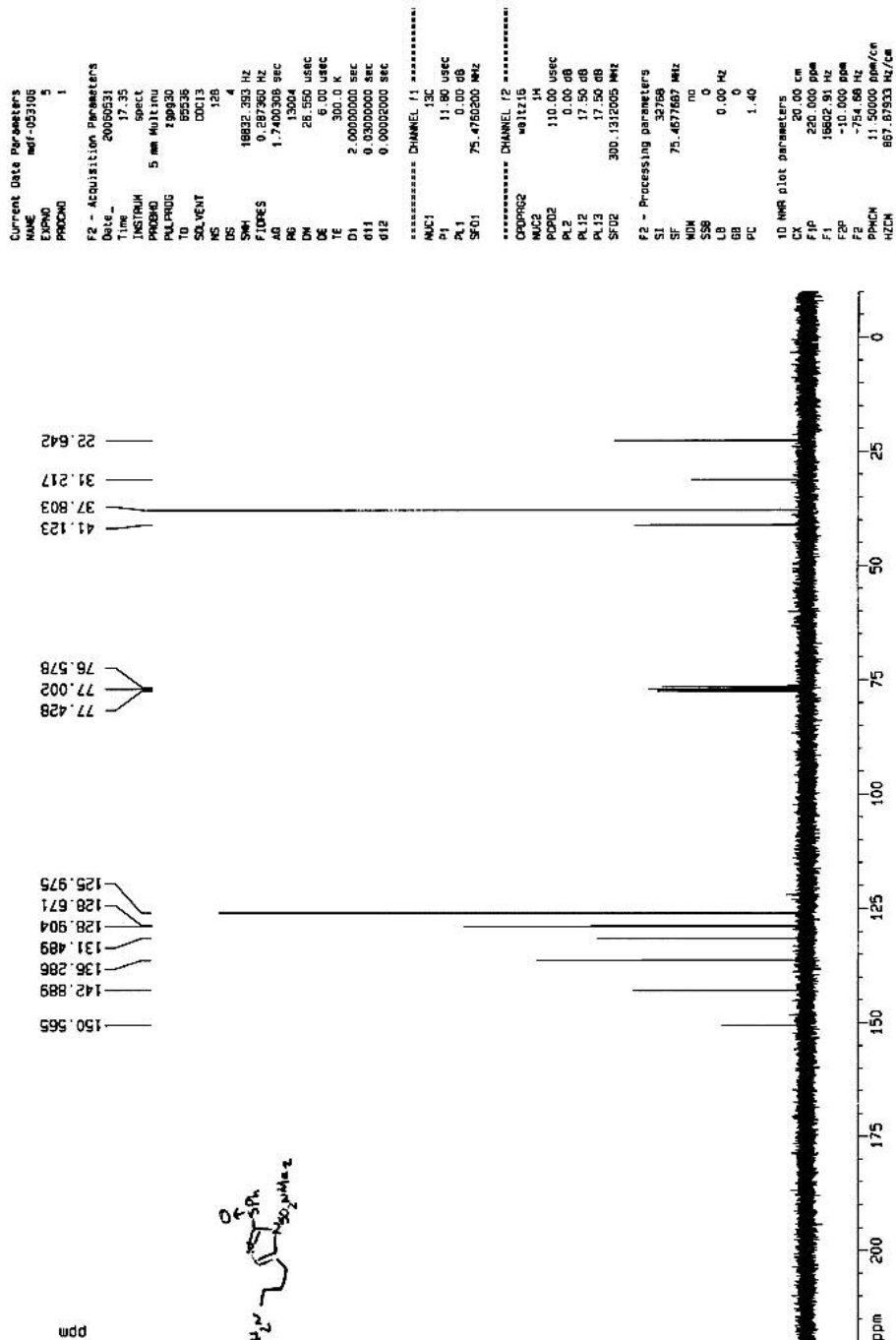




mdf-02-13: amine - sulfoxide - 300MHz



mdf-02-131 amine - sulfoxide - 75MHz



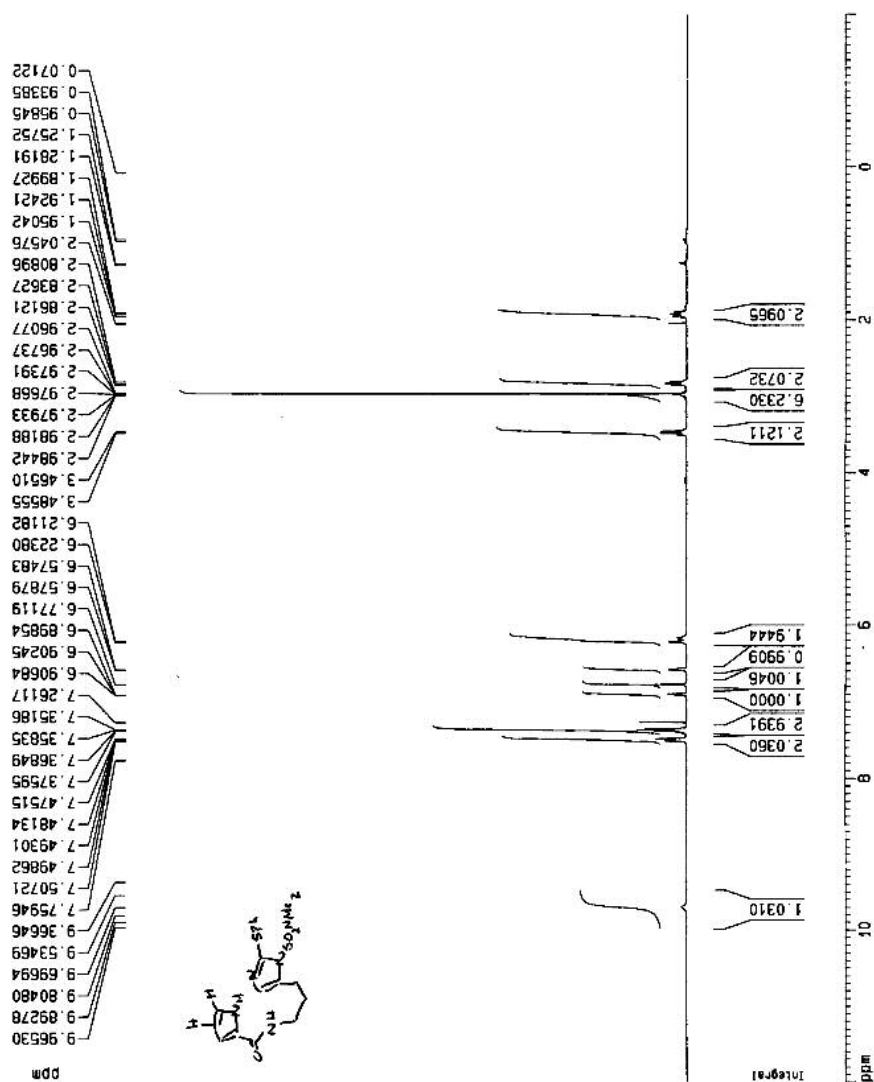
Current Data Parameters
NAME md1-051707
EXPNO 5
PROCNO 1

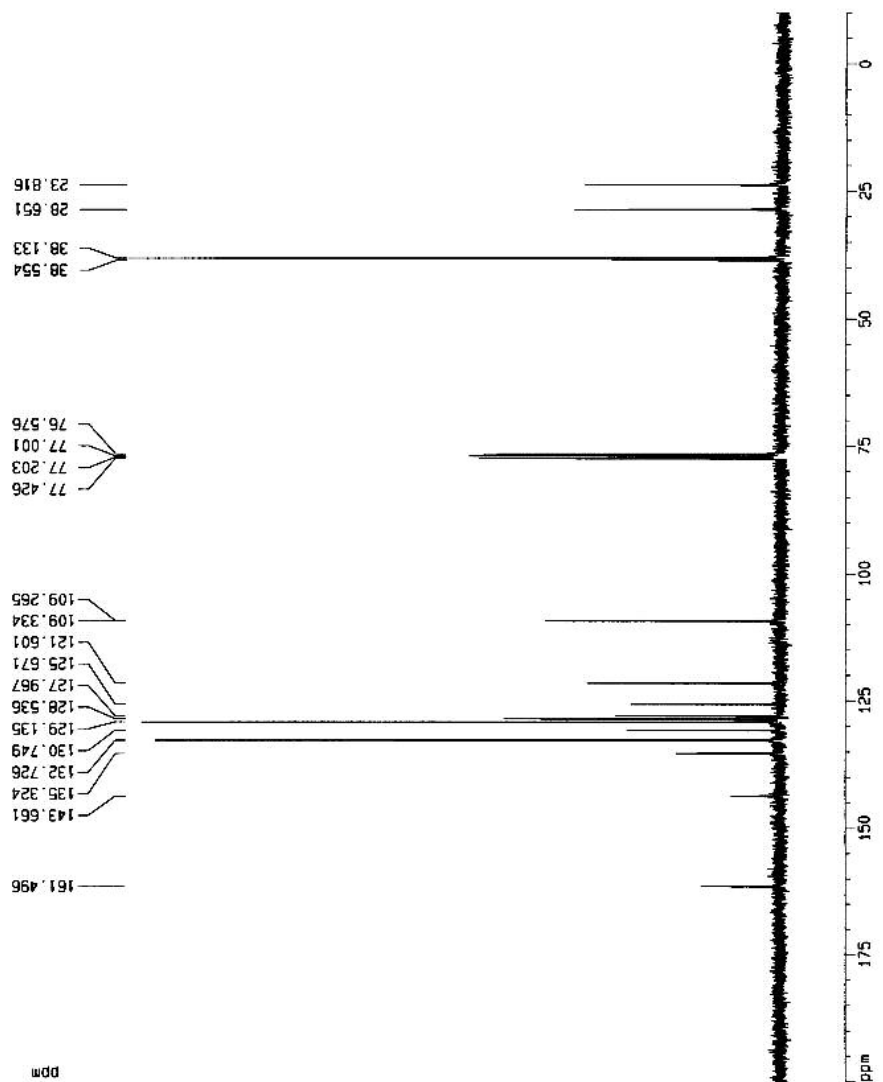
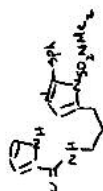
F2 - Acquisition Parameters
Date_ 20070517
Time 12.41
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT DMS
NS 20
DS 0
SWH 6172.839 Hz
FIDRES 0.376760 Hz
AQ 1.3271540 sec
RG 362
DM 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.0000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SFO1 299.8718518 MHz

F2 - Processing parameters
SI 32768
SF 299.870096 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
FIP 12.000 ppm
F1 3998.44 Hz
F2 -2.000 ppm
F2 -599.74 Hz
PPMCH 0.70000 ppm/cm
HZCM 209.90900 Hz/cm





Current Data Parameters
 NAME md1-051707
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20070517
 Time 17:21
 INSTRUM spect
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 135
 DS 0
 SWH 18796.582 Hz
 FIDRES 0.286013 Hz
 AQ 1.7433078 sec
 RG 655
 DM 26.600 usec
 DE 5.00 usec
 TE 300.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 D12 0.0002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 5.40 usec
 PL1 -6.00 dB
 SF01 75 4106357 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P2 115.00 usec
 PL2 0.00 dB
 PL12 20.00 dB
 PL13 20.00 dB
 SF02 299.671595 MHz

F2 - Processing Parameters
 SI 32768
 SF 75 4023501 MHz
 MVM EN
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 200.000 ppm
 F1 15080.48 Hz
 F2P -10.000 ppm
 F2 -10.000 Hz
 PRNCH 10 5000000/cu
 HZCN 791.72510 MHz

mdf-02-239 NH/monobromo/protected sulfido amide - 400MHz

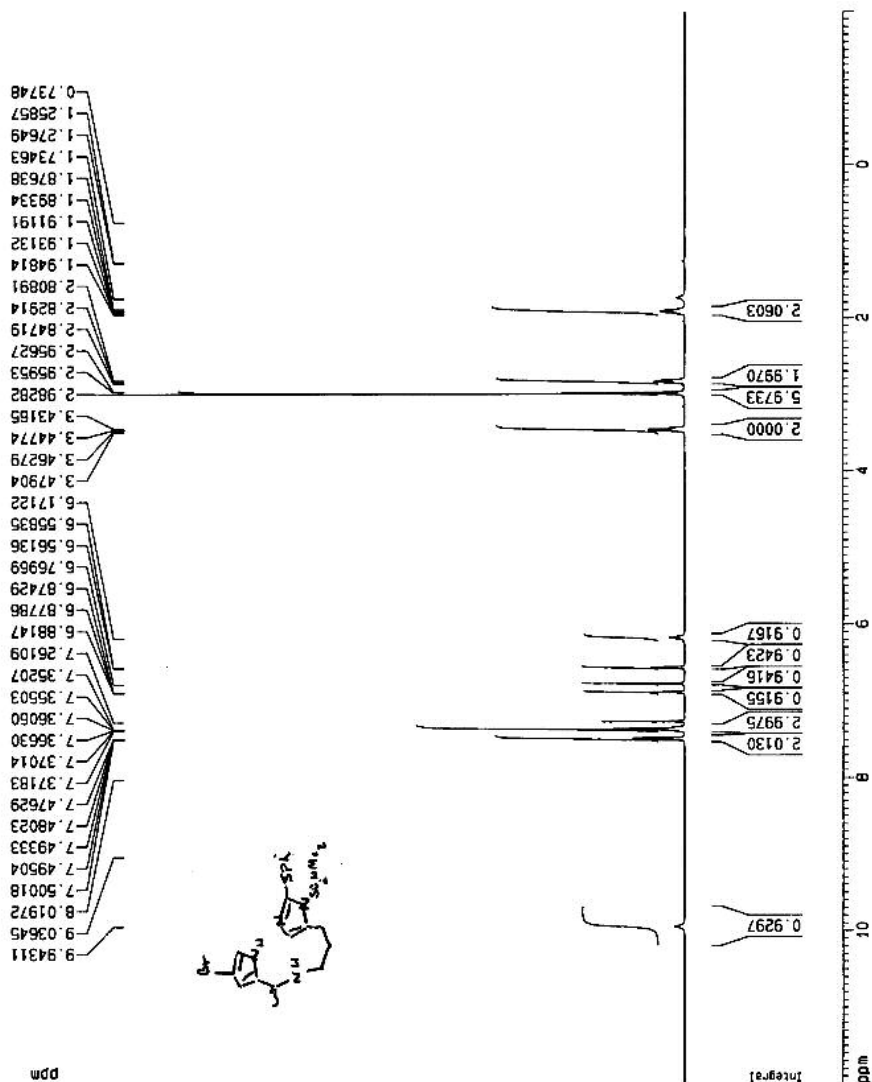
Current Data Parameters
 NAME mdf-022006
 EXPNO 6
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060920
 Time 9:55
 INSTRUM spect
 PROBHD 5 mm BBI 1H-B
 PULPROG zg30
 TO 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.125314 Hz
 AQ 3.9584243 sec
 RG 382
 DM 50.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

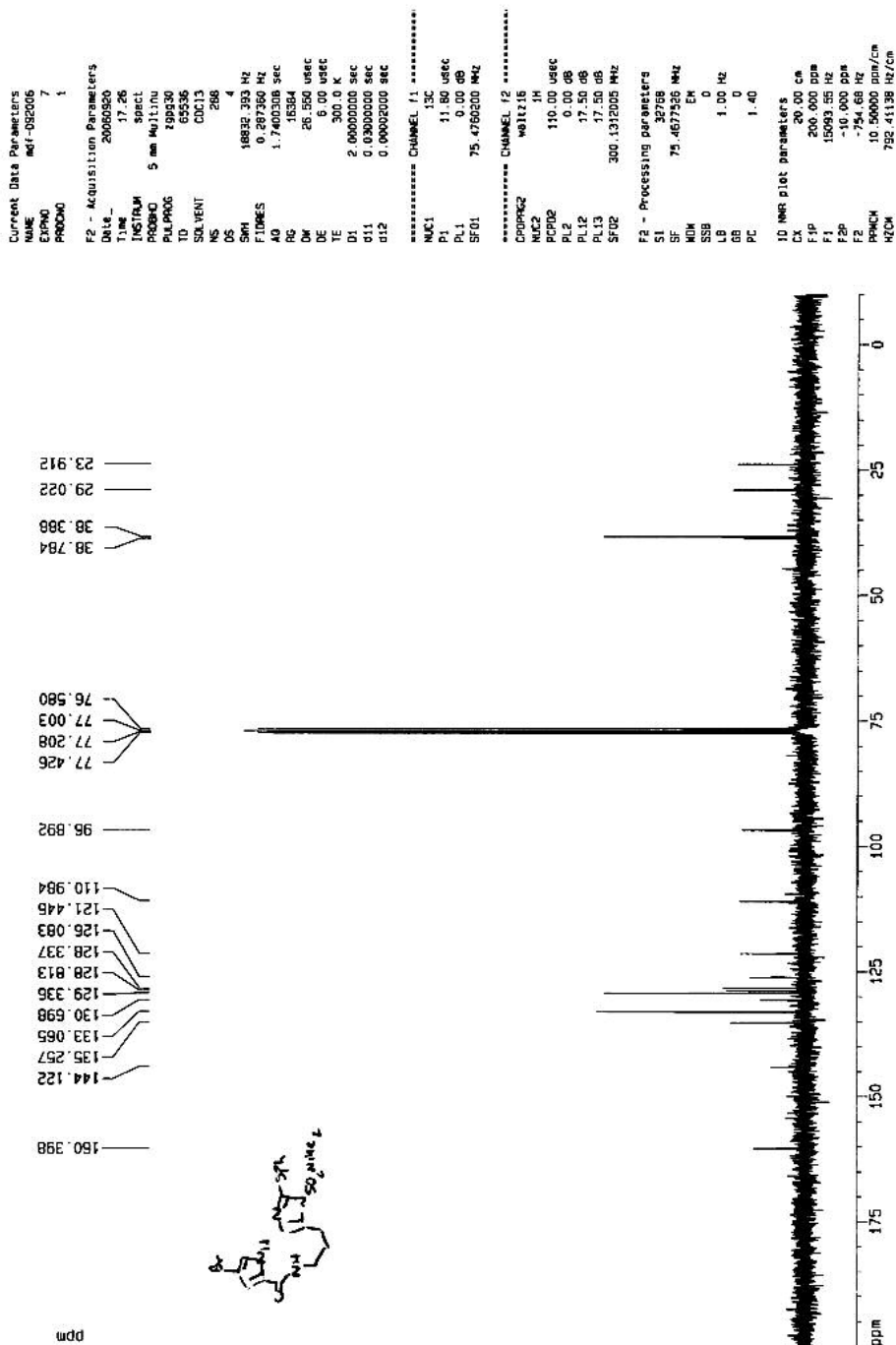
===== CHANNEL f1 =====
 NUC1 1H
 P1 6.45 usec
 PL1 0.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.130077 MHz
 MDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 FIP 12 000 gpm
 F1 4801.56 Hz
 F2 -2 000 gpm
 F2 -800.26 Hz
 FWHM 0.70000 ppm/cm
 WZCN 200.09100 Hz/cm



mdf-02-239 NH/monobromom/protected sulfido amide - 75MHz



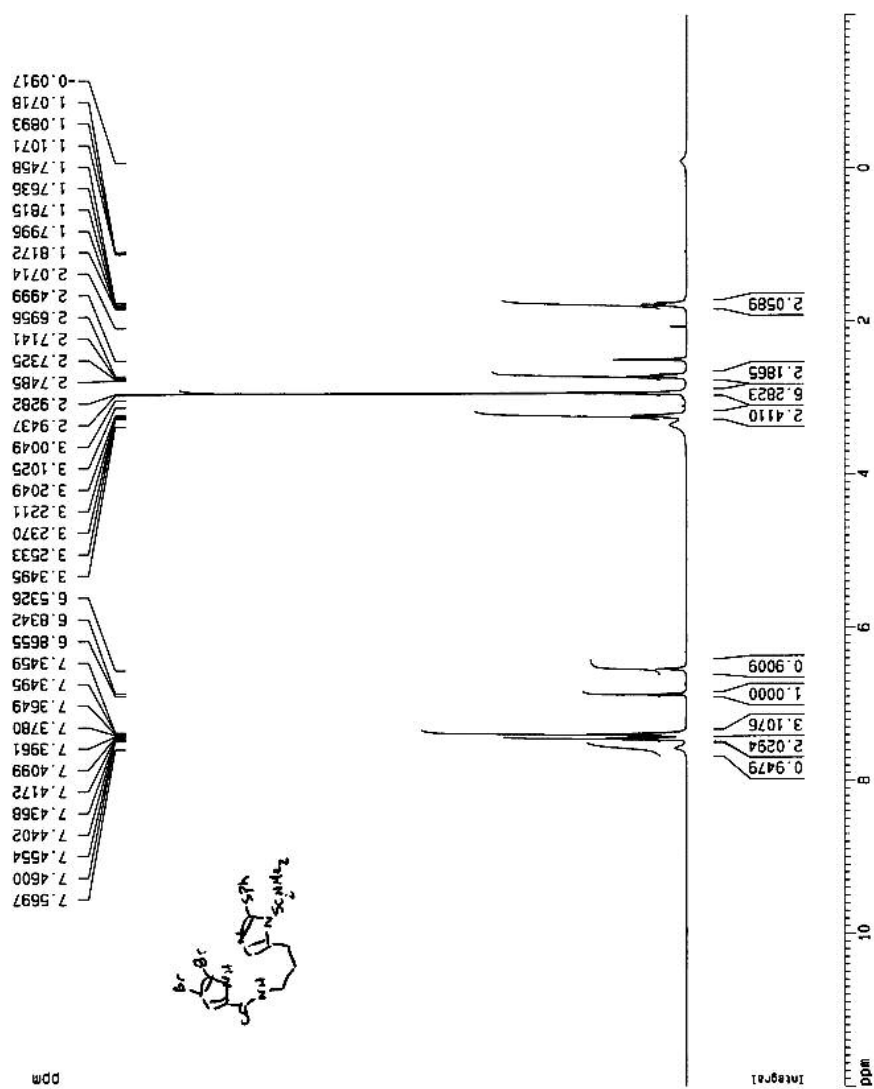
Current Data Parameters
 NAME mdf-051107
 EXPNO 1
 PROCNO 1

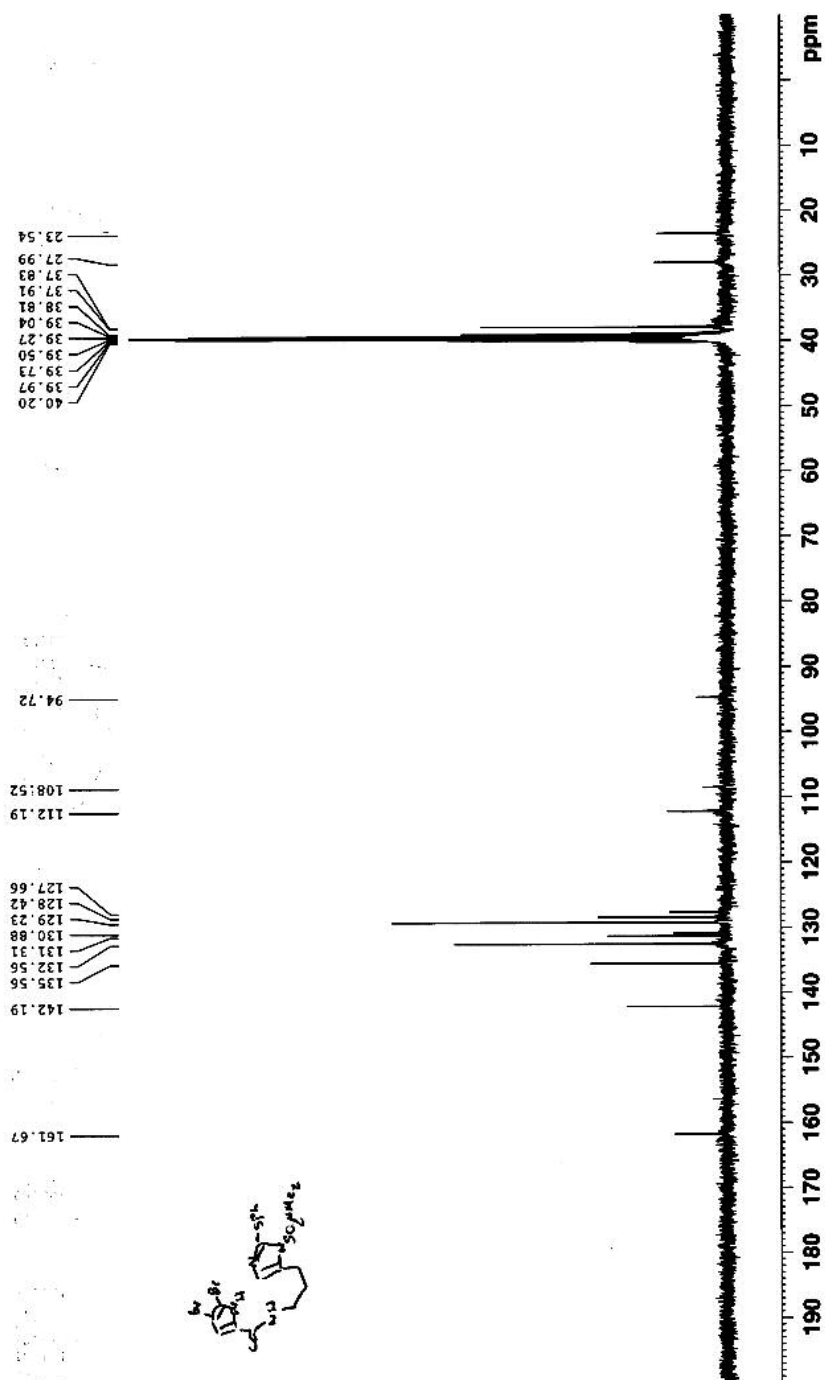
F2 - Acquisition Parameters
 Date_ 20070511
 Time 10:40
 INSTRUM spect
 PROBHD 5 mm BBI 1H-8
 PULPROG zg30
 TD 16384
 SOLVENT DMSO
 NS 18
 DS 0
 SFO1 8278.145 Hz
 FIDRES 0.505258 Hz
 AQ 0.986436 sec
 RG 181
 DW 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.00000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 6.45 usec
 PL1 0.00 dB
 SF01 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300031 MHz
 WDW em
 SSB 0
 LB 0.00 Hz
 DB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 12.000 ppm
 F1 4801.55 Hz
 F2P -2.000 ppm
 F2 -800.26 Hz
 PPM0K 0.70000 ppm/cm
 HZ0K 280.05100 Hz/cm





Current Data Parameters
NAME md1-061407
EXPNO 21
PROCNO 1

F2 - Acquisition Parameters

Date_ 20070614
Time 15.34
INSTRUM spect
PROBHD 5 mm BBI 1H-8
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 23
DS 0
SWH 8278.146 Hz
FIDRES 0.505258 Hz
AQ 0.9896436 sec
RG 40.3
DM 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.0000000 sec

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

NUC1 1H
P1 6.45 usec
PL 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters

SI 32768
SF 400.1300021 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1 12.000 ppm
F2 4801.55 Hz
F3 -2.000 ppm
F4 -800.25 Hz
P1 0.70000 ppm/cm
HZCM 280.05100 Hz/cm

3.30120
3.28648
2.92794
2.90719
2.75979
2.74139
2.72318
2.50010
1.85991
1.84227
1.82471

8.11150
7.74400
7.73598
7.72658
7.72208
7.55883
7.55001
7.54414
7.13412
6.82704



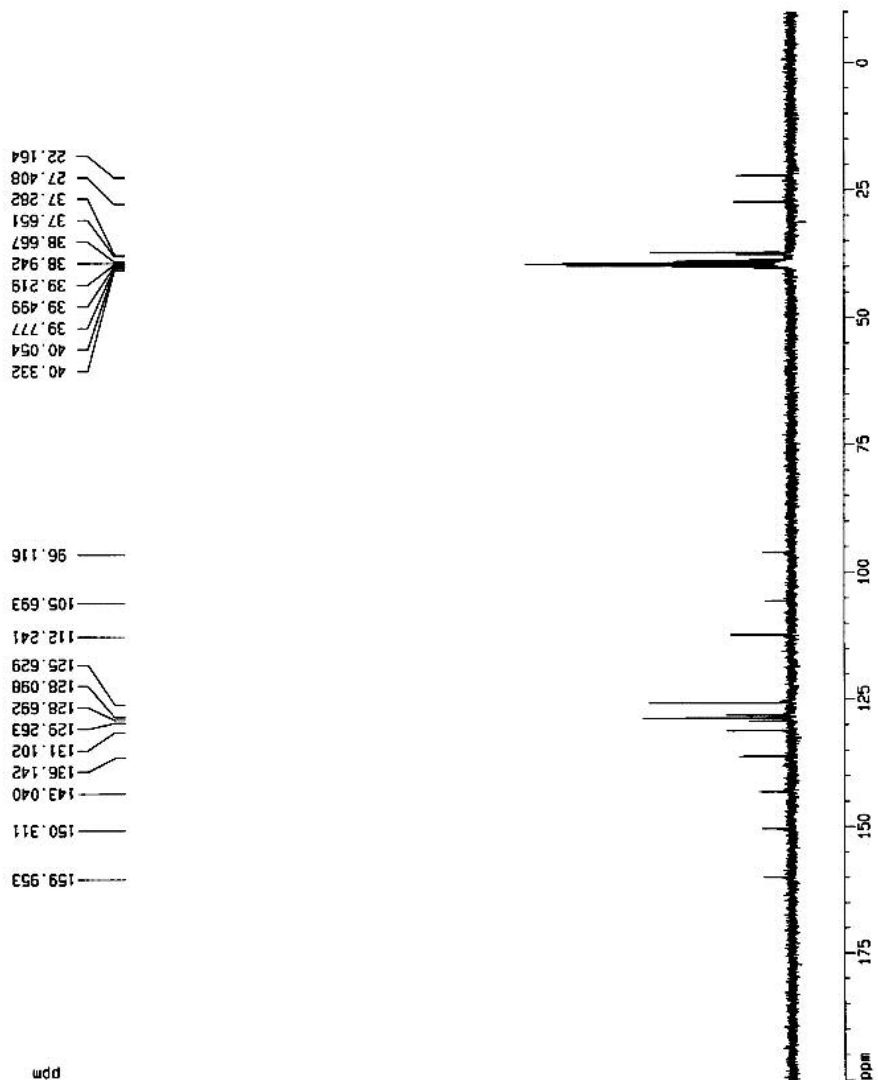
ppm

2.1185
6.1300
2.2605
2.1978

1.0000
2.0140
3.0258
0.9915
1.0279

Integral





Current Data Parameters
NAME nml-060107
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070601
Time 14.47
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 169
DS 4
SWH 19832.353 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 8192
ON 26.350 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00000000 sec

----- CHANNEL f1 -----
NUC1 ¹³C
P1 11.80 usec
PL 0.00 dB
SF01 75.4763200 MHz

----- CHANNEL f2 -----
CPDPRG2 mlti216
NUC2 ¹H
PCPD2 110.00 usec
PL2 0.00 dB
PL12 17.50 dB
PL13 17.50 dB
SF02 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4678108 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

10 NMR plot parameters
CX 20.00 cm
F1P 200.000 ppm
F1 15093.56 Hz
F2P -10.000 ppm
F2 -754.68 Hz
NUC1 ¹³C
NUC2 ¹H
AQ 1.7400308 sec

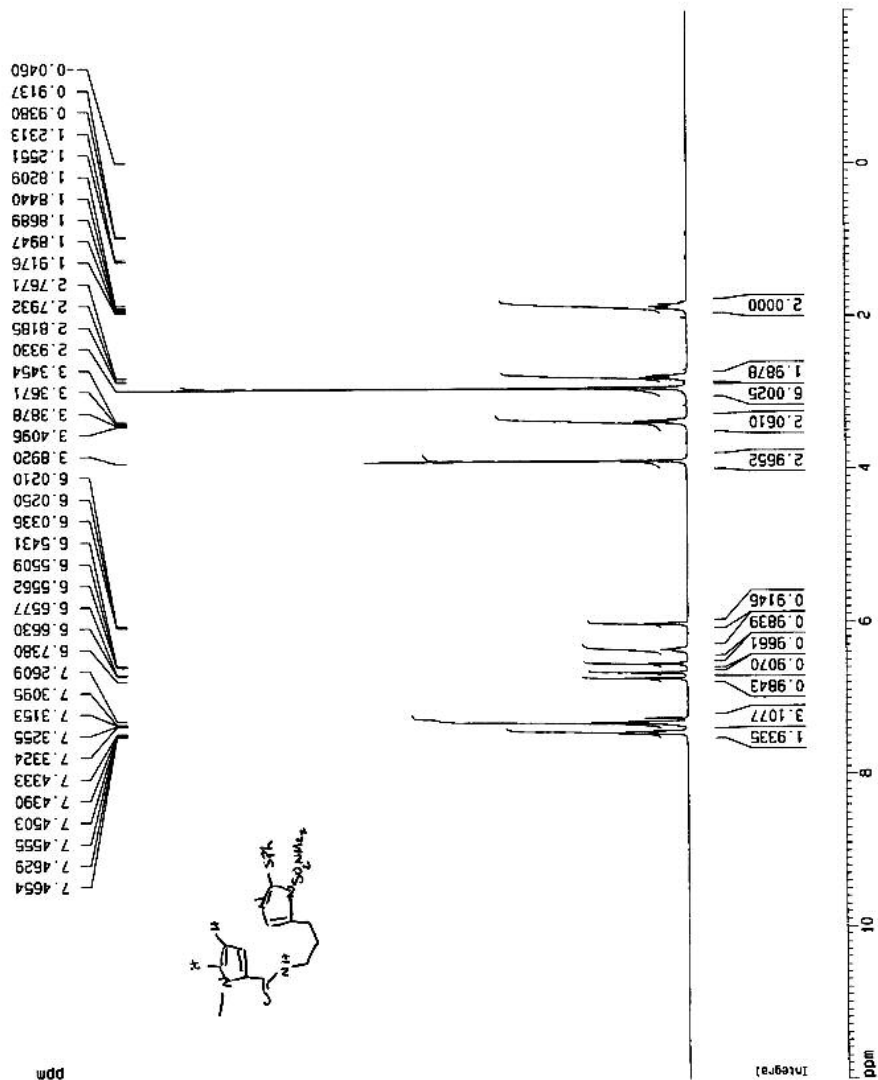
Current Data Parameters
NAME mdf-051507
EXPNO 2
PROCNO 1

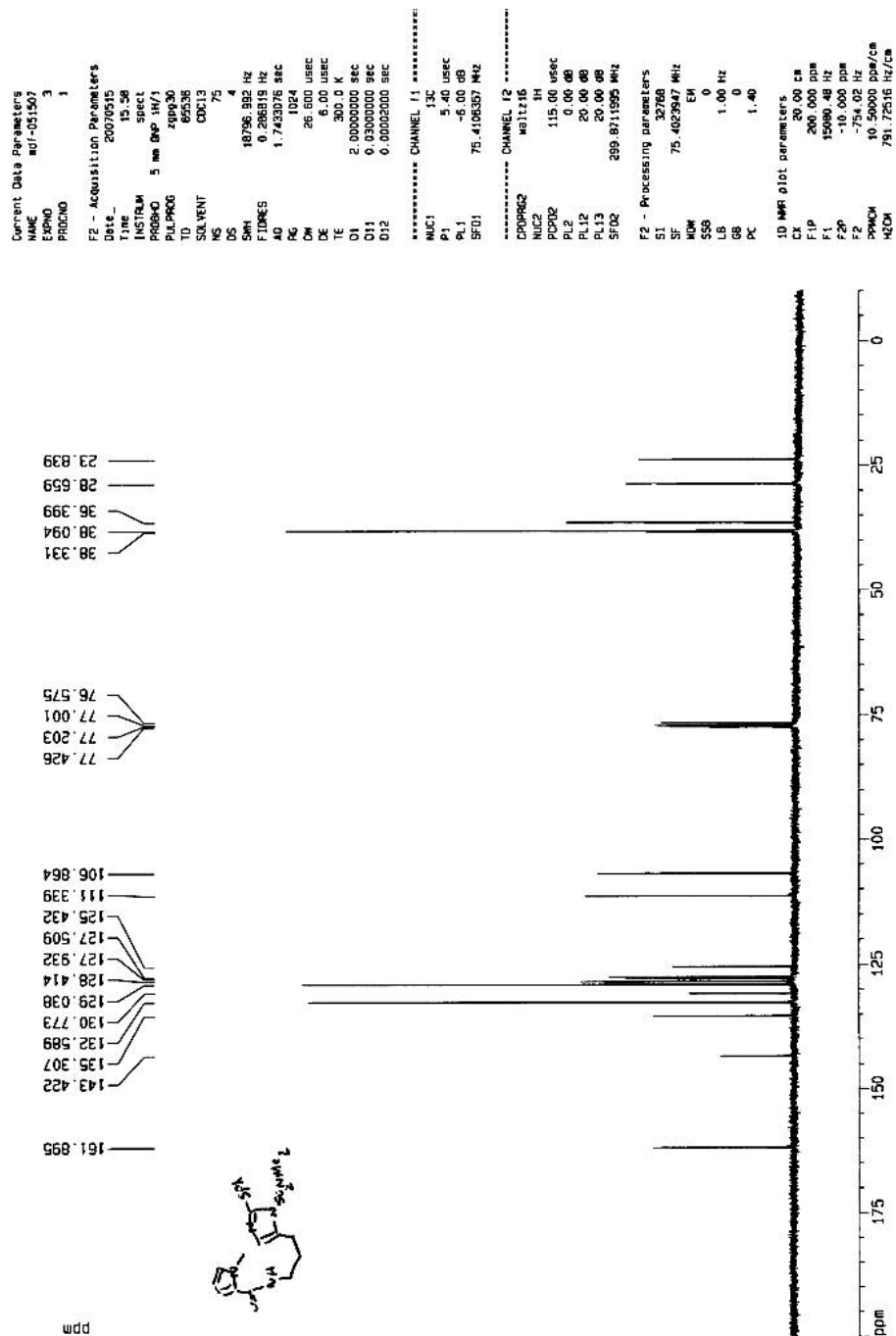
F2 - Acquisition Parameters
Date_ 20070515
Time 15.53
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 23
DS 2
SWH 6172.839 Hz
FIDRES 0.376760 Hz
AQ 1.3271540 sec
RG 50.8
DM 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SFO1 299.8718518 MHz

F2 - Processing parameters
SI 32768
SF 299.8700095 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
FIP 12.000 ppm
F1 3598.44 Hz
F2 -2.000 ppm
FZ -595.74 Hz
PRICK 0.70000 pps/cm
HZCM 209.90900 Hz/cm





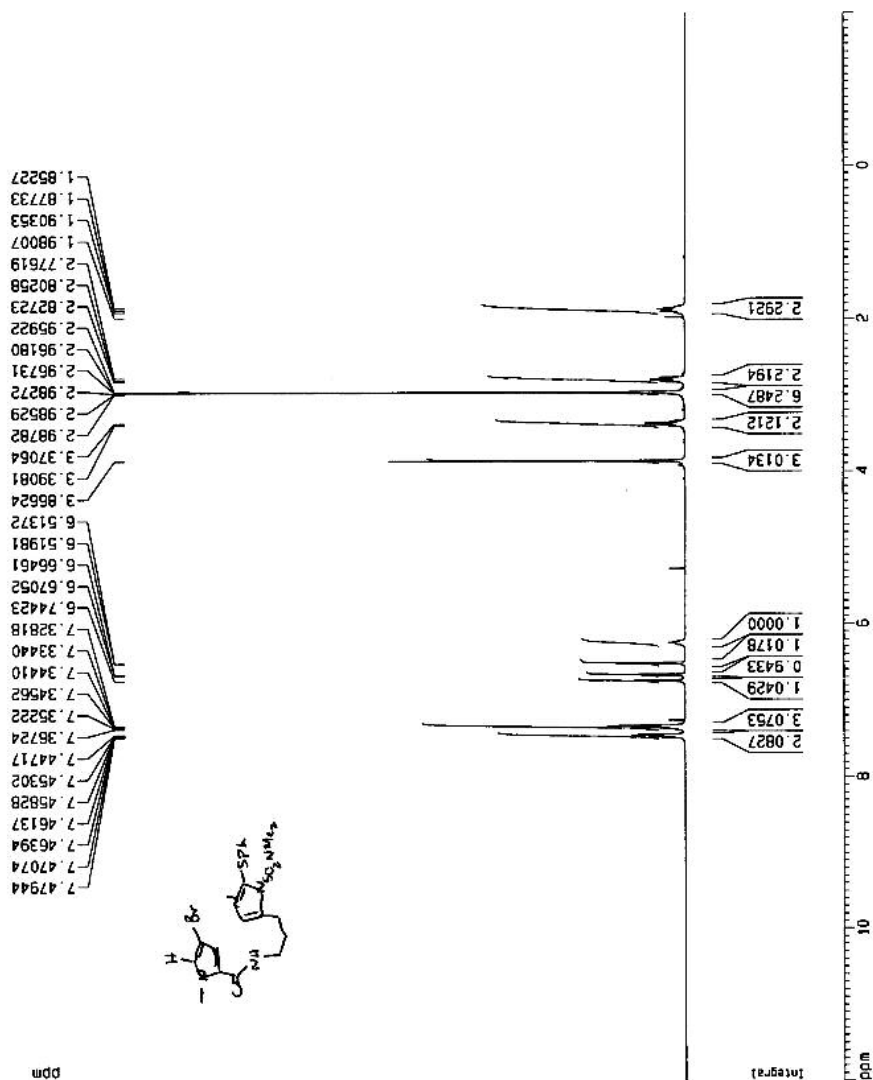
Current Data Parameters
NAME ndf-051507
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070515
Time 15.03
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 32
DS 2
SWH 6172.839 Hz
FIDRES 0.376760 Hz
AQ 1.3271540 sec
RG 71.8
DM 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 11.70 usec
PL1 0.00 dB
SF01 299.8718618 MHz

F2 - Processing parameters
SI 32768
SF 299.870084 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
FIP 12.000 ppm
F1 3598.44 Hz
F2P -2.000 ppm
F2 -595.74 Hz
PRMCM 0.70000 ppm/cm
HZCM 209.90900 Hz/cm



Current Data Parameters
NAME: m01-051507
EXPNO: 5
PROCNO: 1

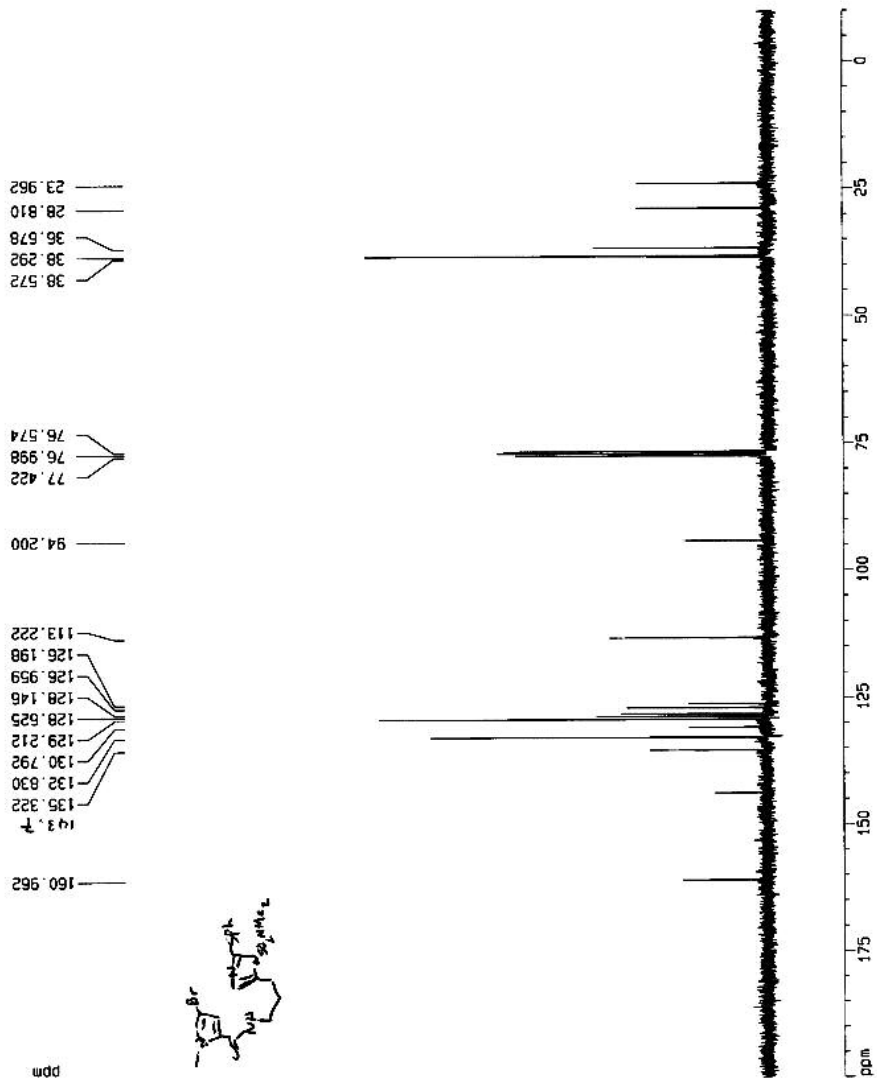
F2 - Acquisition Parameters
Date_: 20070515
Time: 16.11
INSTRUM: spect
PROBHD: 5 mm QNP 1H/1
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 76
DS: 4
SWH: 18796.982 Hz
FIDRES: 0.266919 Hz
AQ: 1.7433076 sec
RG: 1024
DM: 26.600 usec
DE: 6.00 usec
TE: 300.0 K
D1: 2.0000000 sec
D11: 0.0300000 sec
D12: 0.0000000 sec

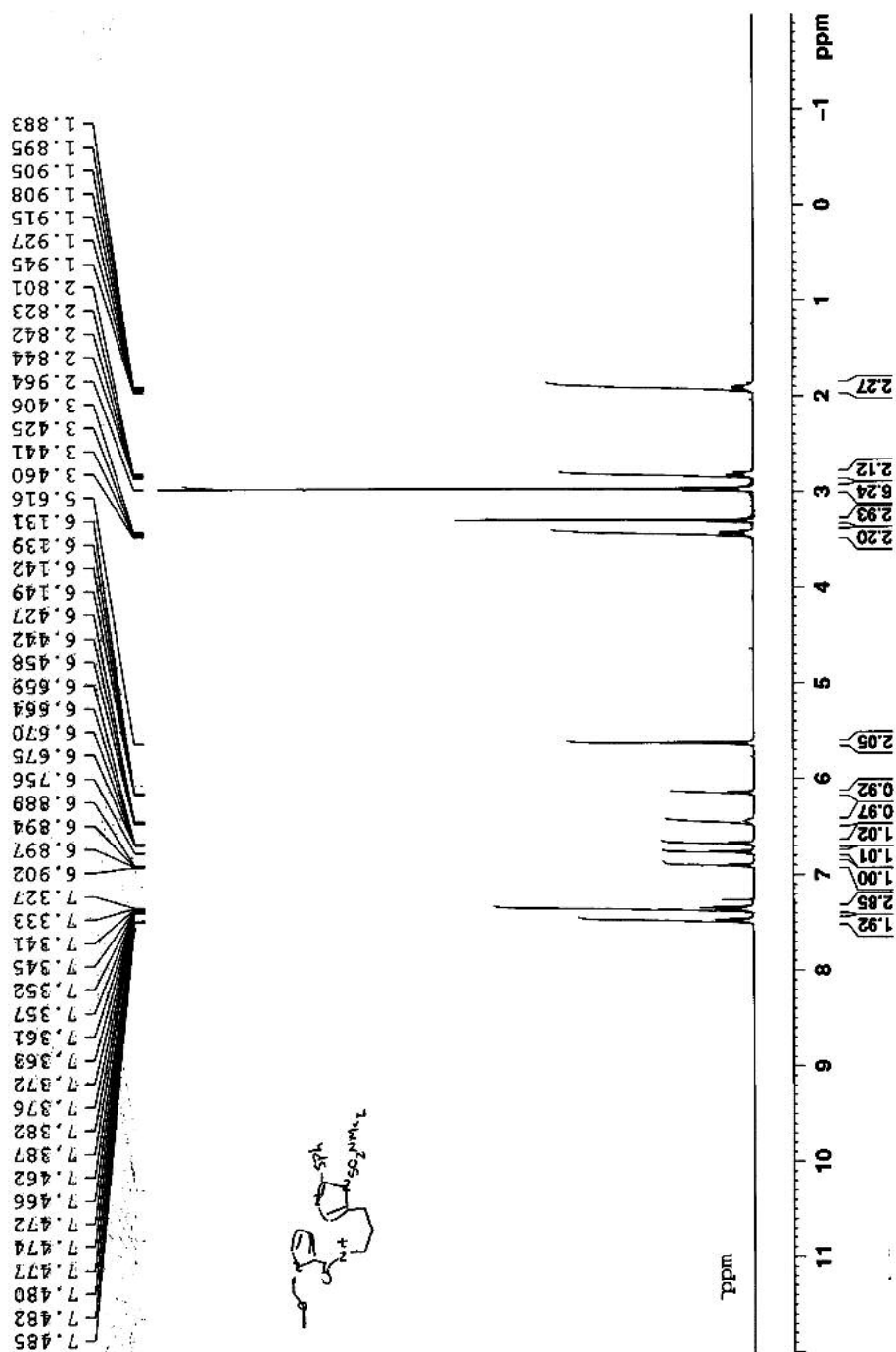
***** CHANNEL f1 *****
NUC1: 13C
P1: 5.40 usec
PL1: -6.00 dB
SF01: 75.4105357 MHz

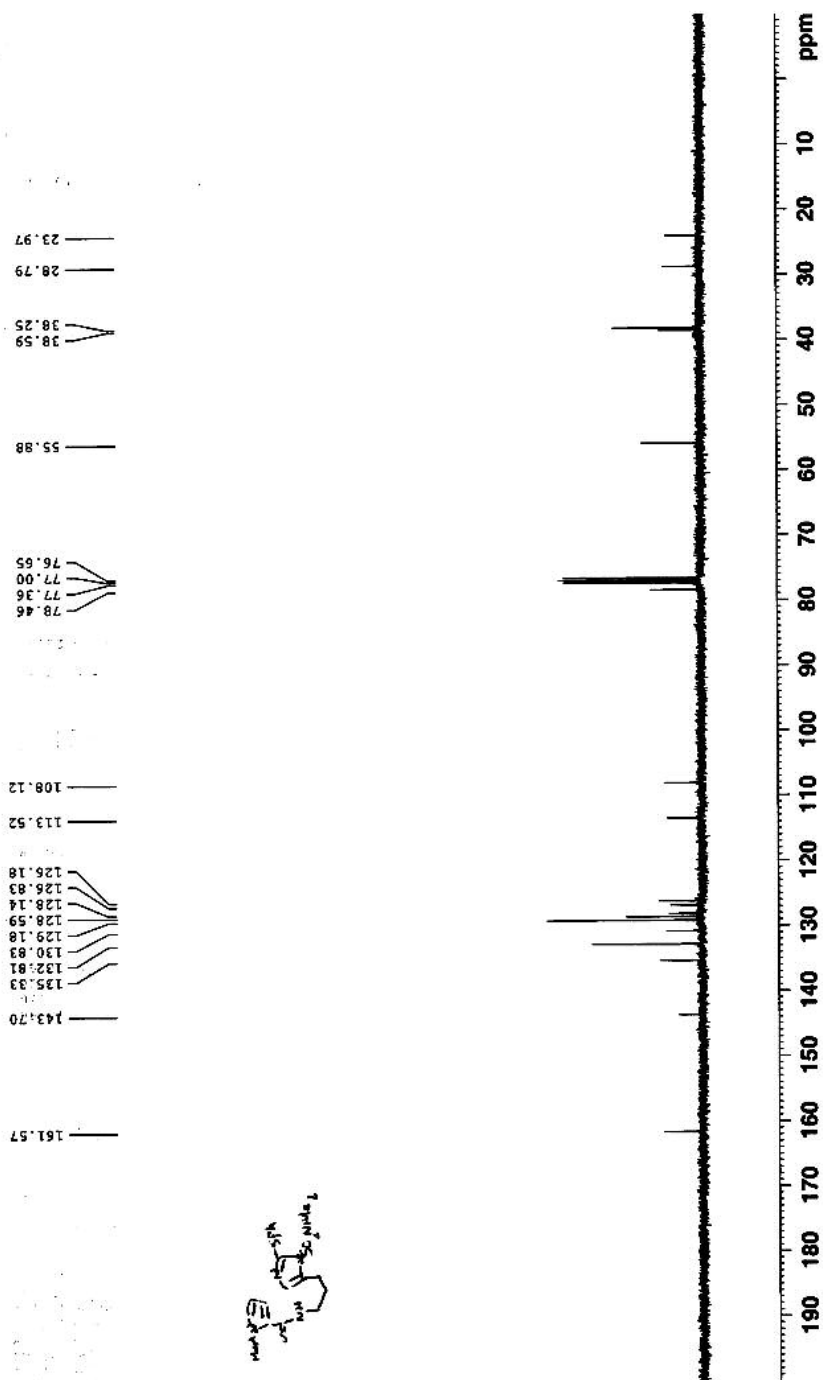
***** CHANNEL f2 *****
CPDPRG2: waltz16
NUC2: 1H
PCPD2: 115.00 usec
PL2: 0.00 dB
PL12: 20.00 dB
PL13: 20.00 dB
SF02: 299.8711953 MHz

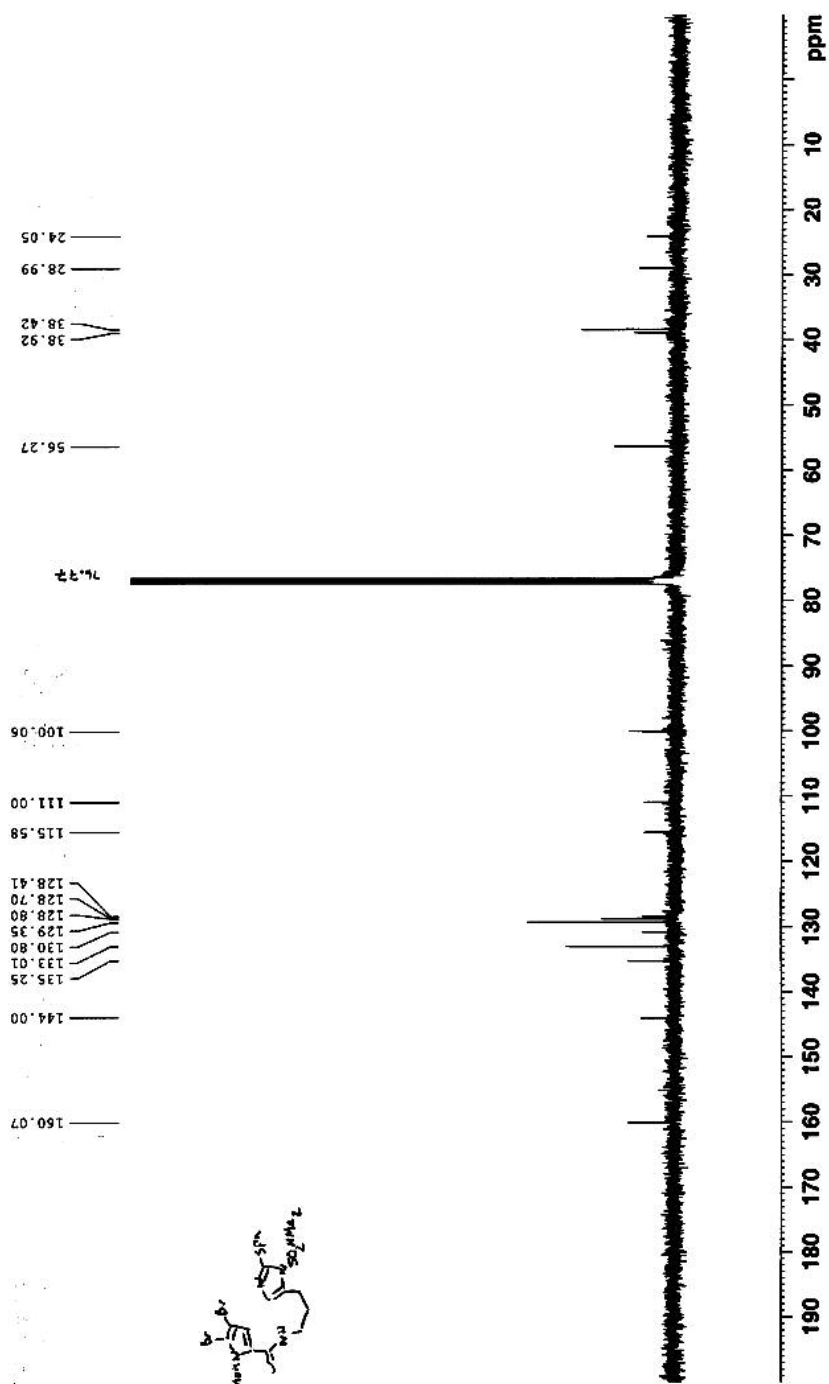
F2 - Processing parameters
SI: 32768
SF: 75.4023532 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40

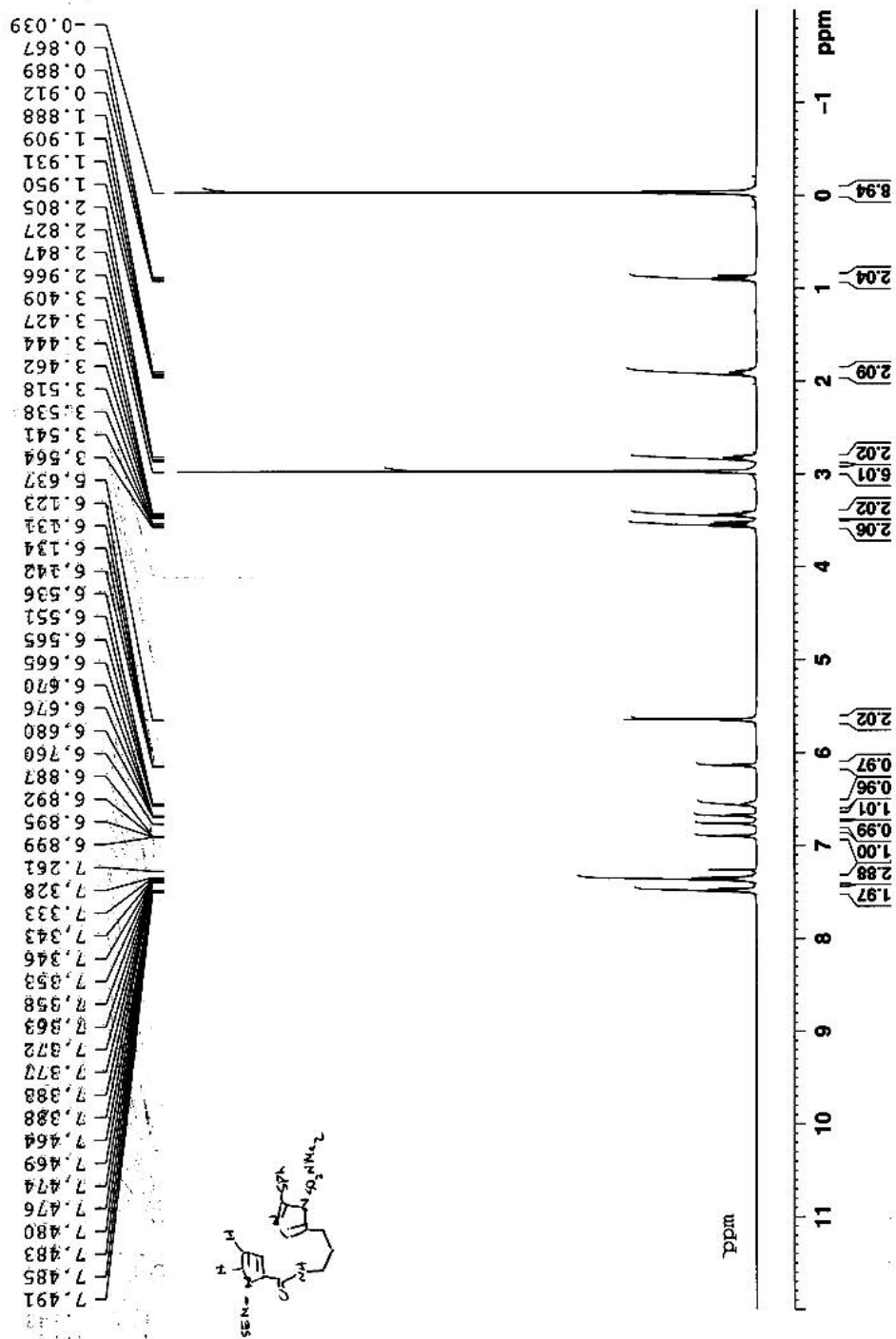
1D NMR plot parameters
CX: 20.00 cm
FIP: 200.000 ppm
F1: 15000.48 Hz
F2P: -10.000 ppm
F2: -754.02 Hz
PPMCON: 10.50000 ppm/cm
MZN: 791.72504 Hz/cm

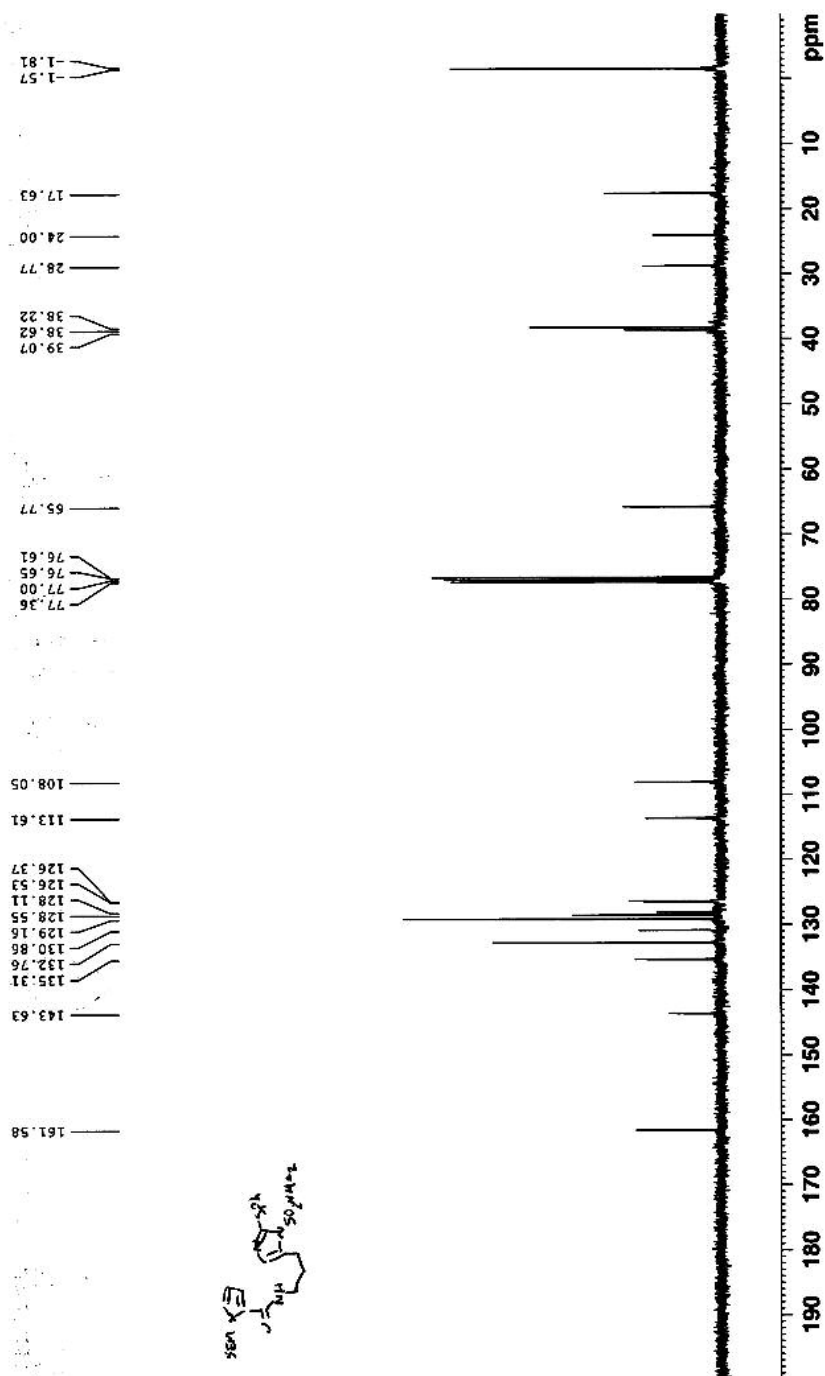














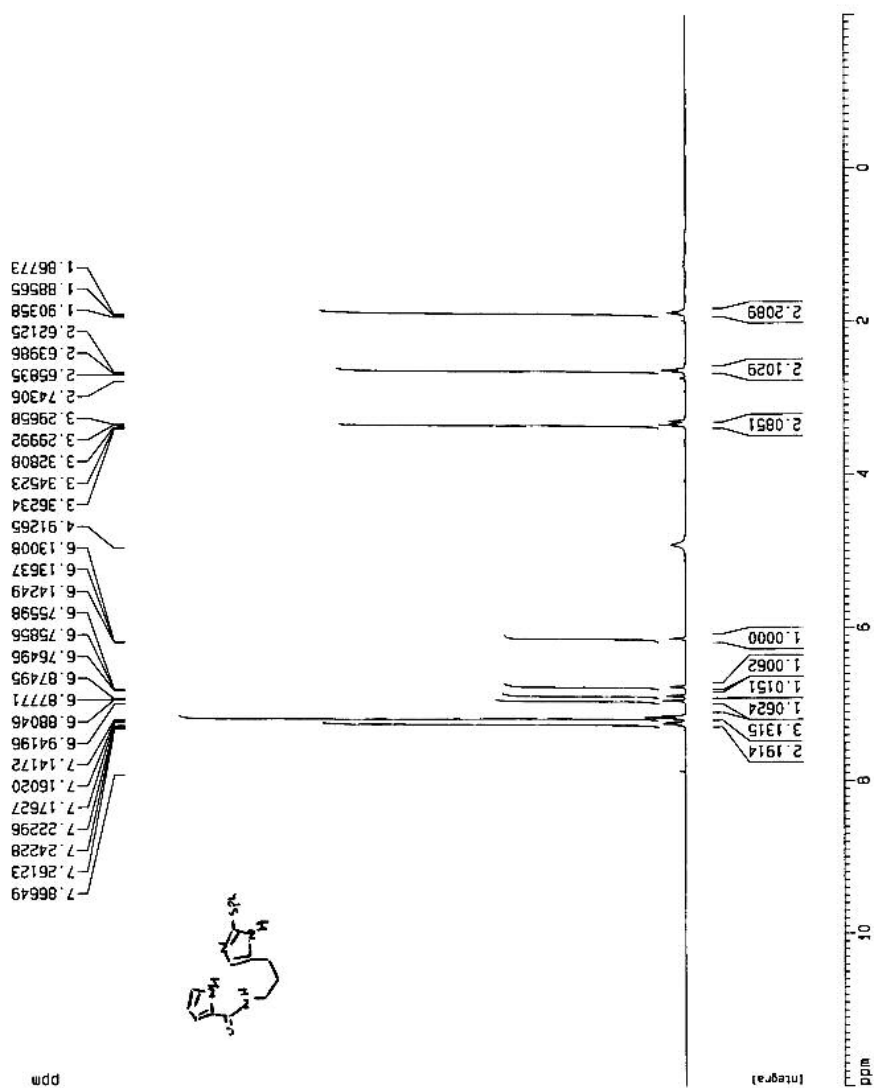
Current Data Parameters
NAME mdf-051507
EXPNO 5
PROCNO 1

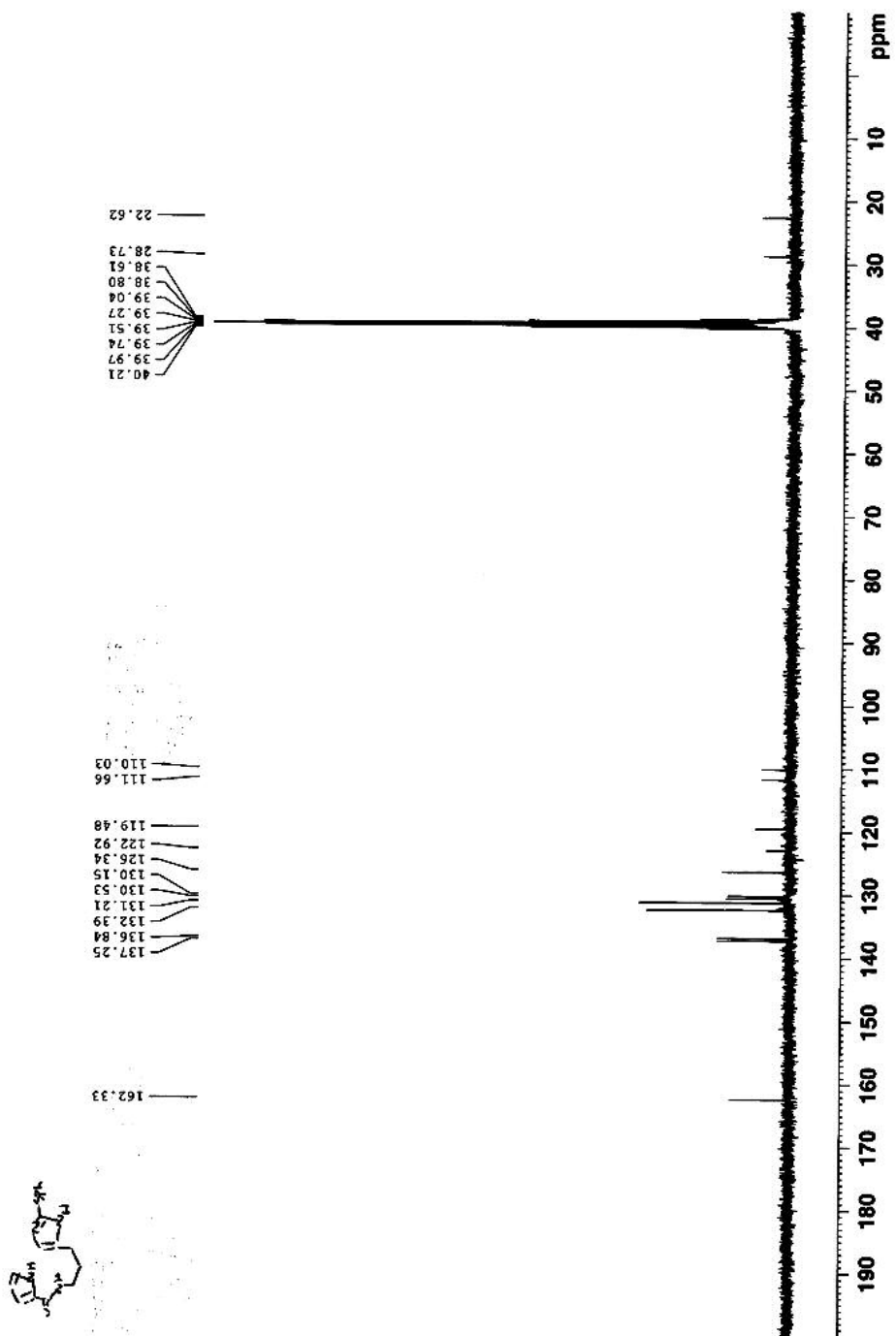
F2 - Acquisition Parameters
Date_ 20070519
Time 20.33
INSTRUM spect
PROBHD 5 mm BBI 1H-5
PULPROG zg30
TD 16384
SOLVENT MECH
NS 16
DS 0
SWH 8278.146 Hz
FIDRES 0.595258 Hz
AQ 0.5956436 sec
RG 228.1
DM 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300117 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

10 NMR plot parameters
CX 20.00 cm
F1P 12.000 ppm
F1 4801.56 Hz
F2P -2.000 ppm
F2 -800.25 Hz
PPM0 0.70000 ppm/cm
HZCM 280.09100 Hz/cm





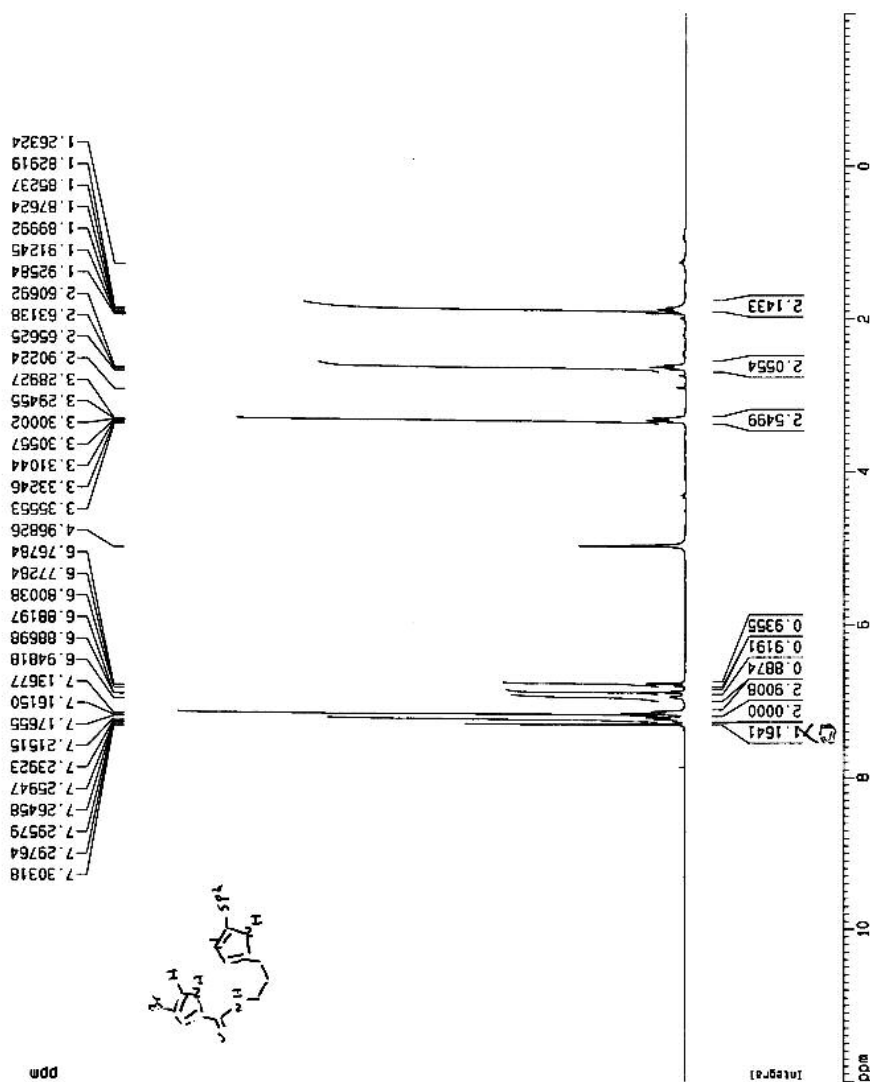
Current Data Parameters
NAME md-051607
EXPNO 7
PROCNO 1

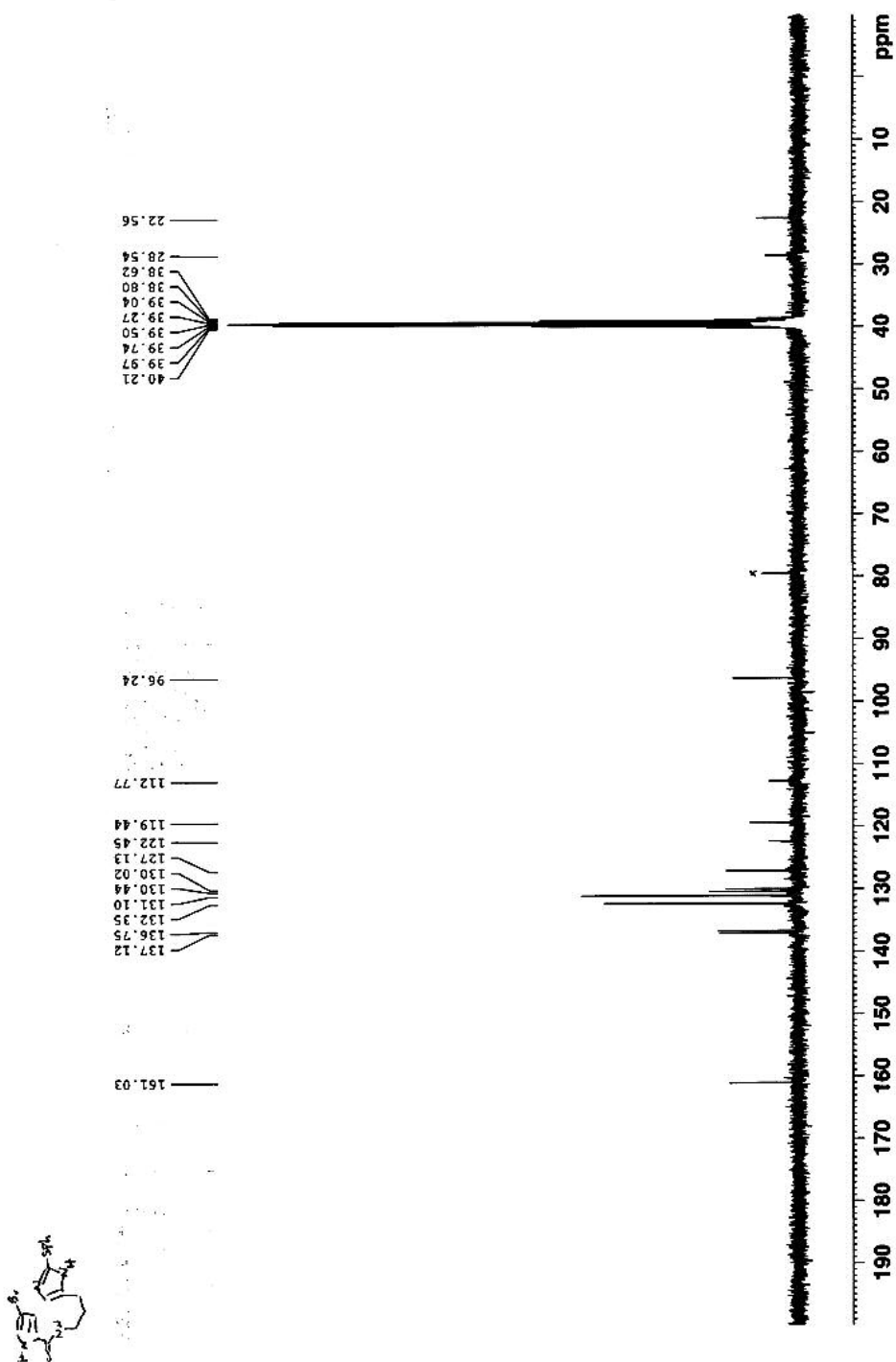
F2 - Acquisition Parameters
Date_ 20070516
Time 15:37
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg30
TO 16384
SOLVENT MeOH
NS 18
DS 0
SWH 6172.639 Hz
FIDRES 0.376750 Hz
AQ 1.3271540 sec
RG 203.2
DM 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

***** CHANNEL f1 *****
NUC1 ¹H
P1 9.50 usec
PL1 -6.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.130079 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

10 NMR plot parameters
CX 20.00 cm
F1P 12.000 ppm
F1 3601.56 Hz
F2P -2.000 ppm
F2 -600.26 Hz
PPMCM 0.70000 ppm/cm
HZCM 210.09100 Hz/cm





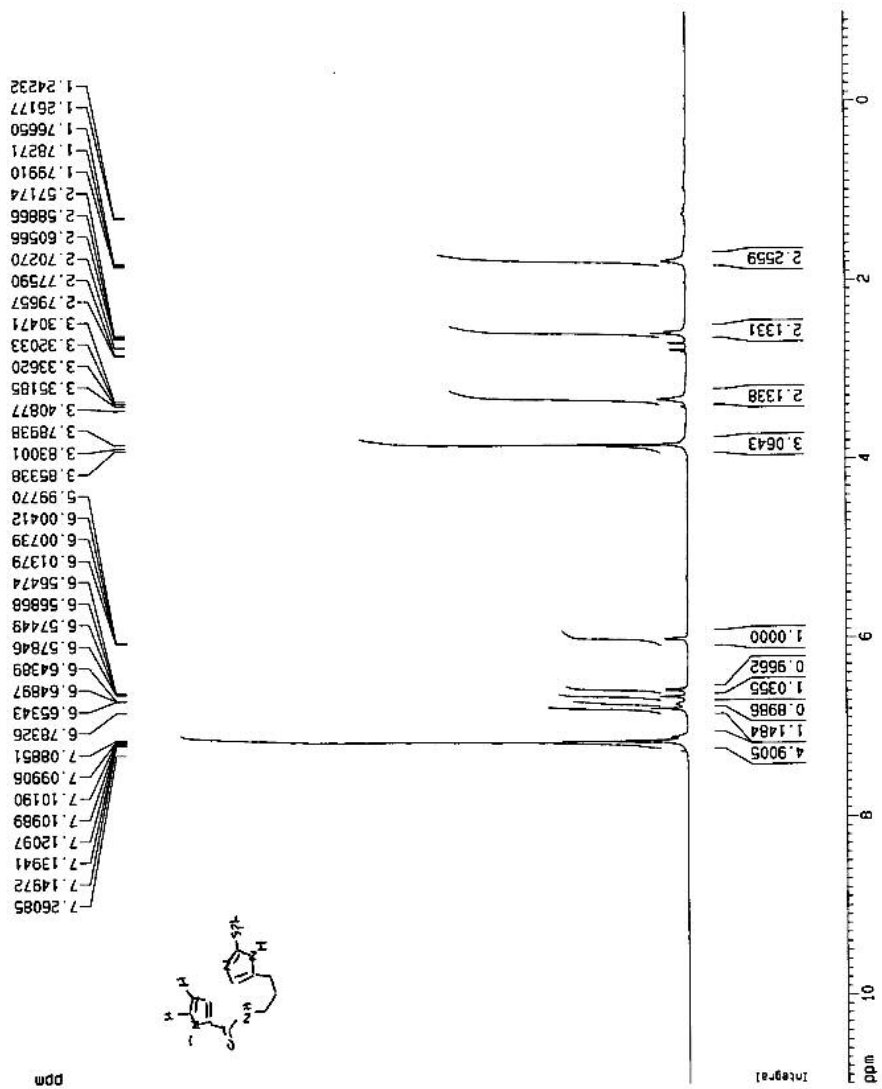
Current Data Parameters
NAME mcf-051707
EXPNO 20
PROCNO 1

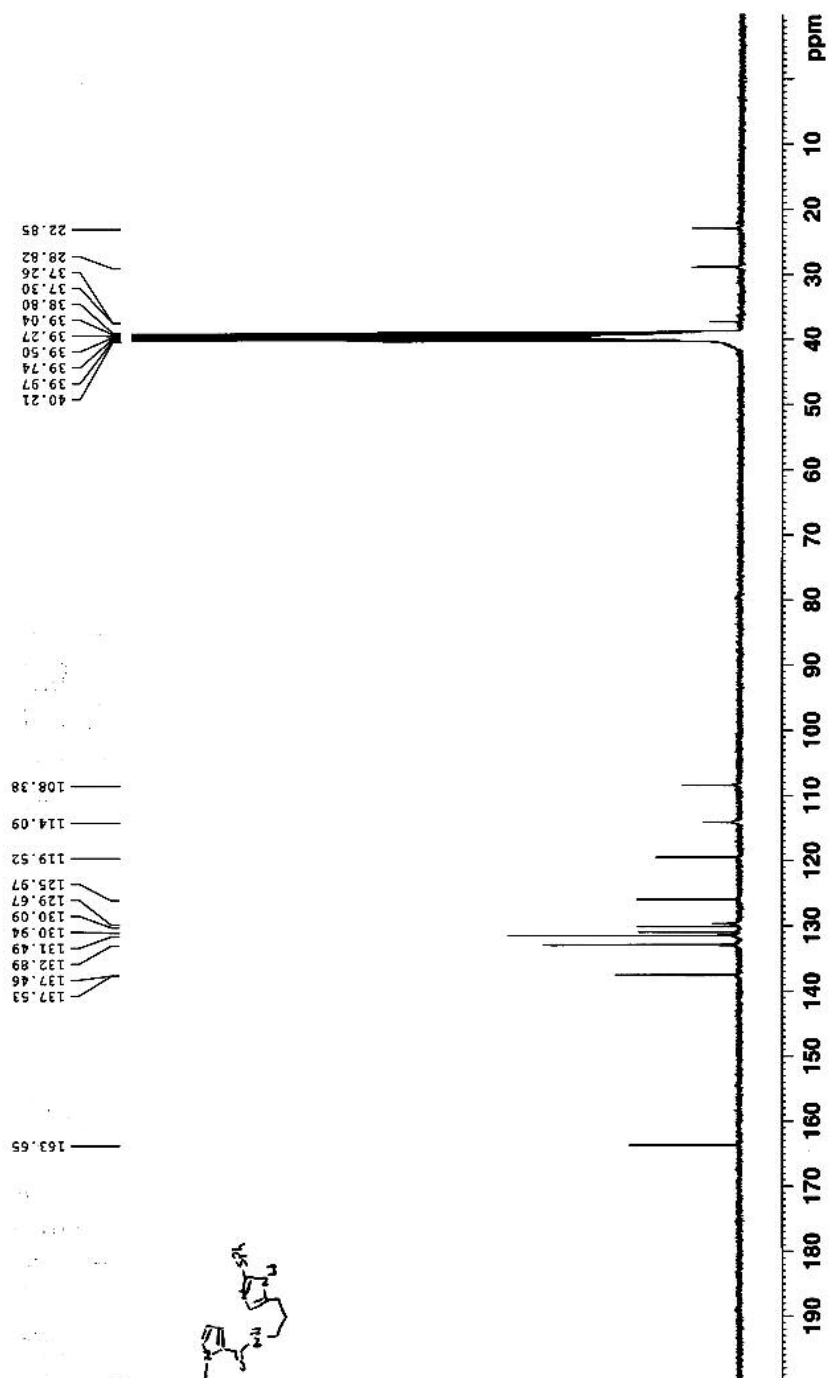
F2 - Acquisition Parameters
Date_ 20070517
Time 19.09
INSTRUM spect
PROBHD 5 mm BBI 1H-5
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.505258 Hz
AQ 0.9896435 sec
RG 40.3
DM 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

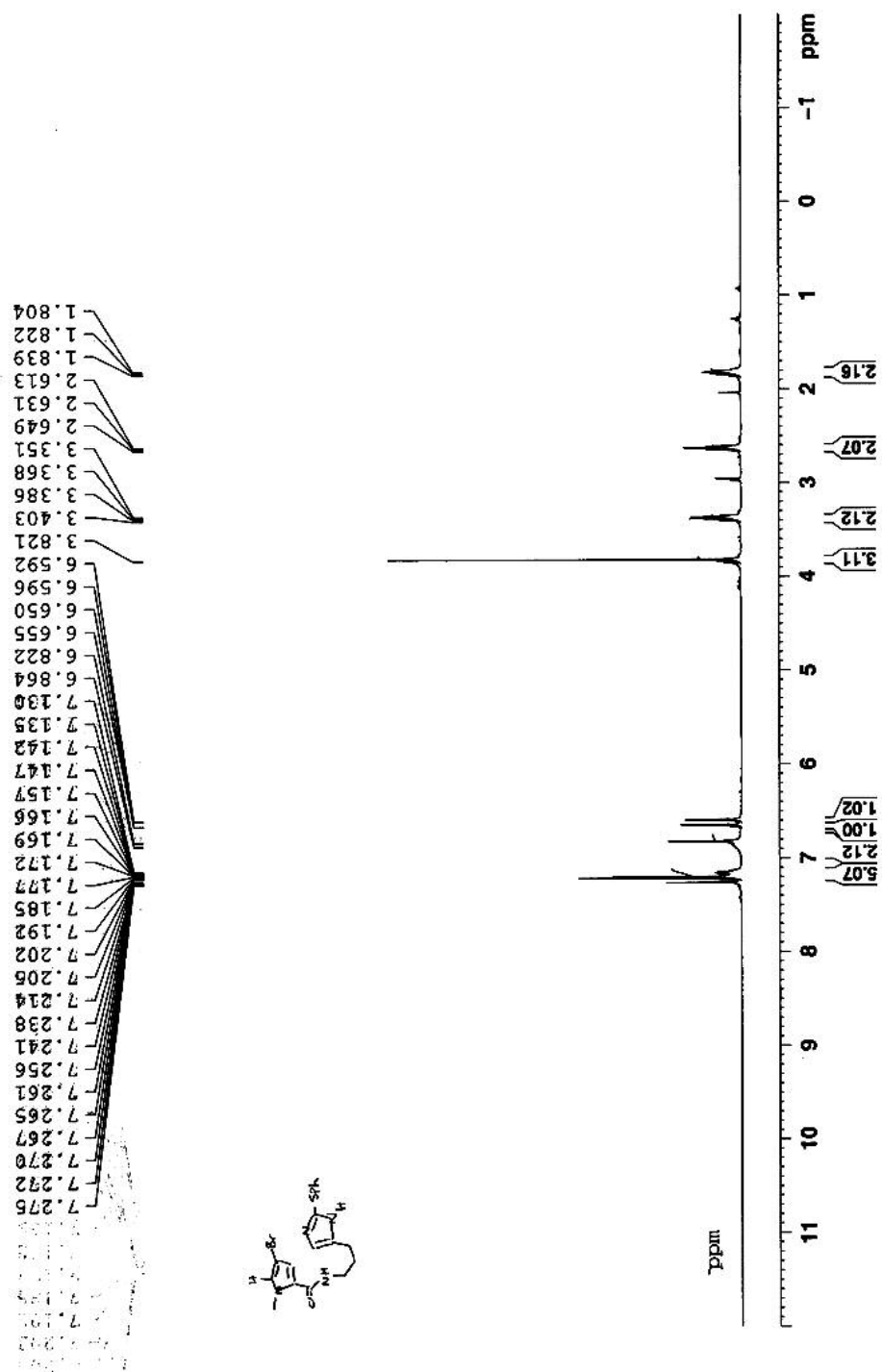
===== CHANNEL f1 =====
NUC1 1H
P1 6.45 usec
PL 0.00 dB
SFO1 400.1324710 MHz

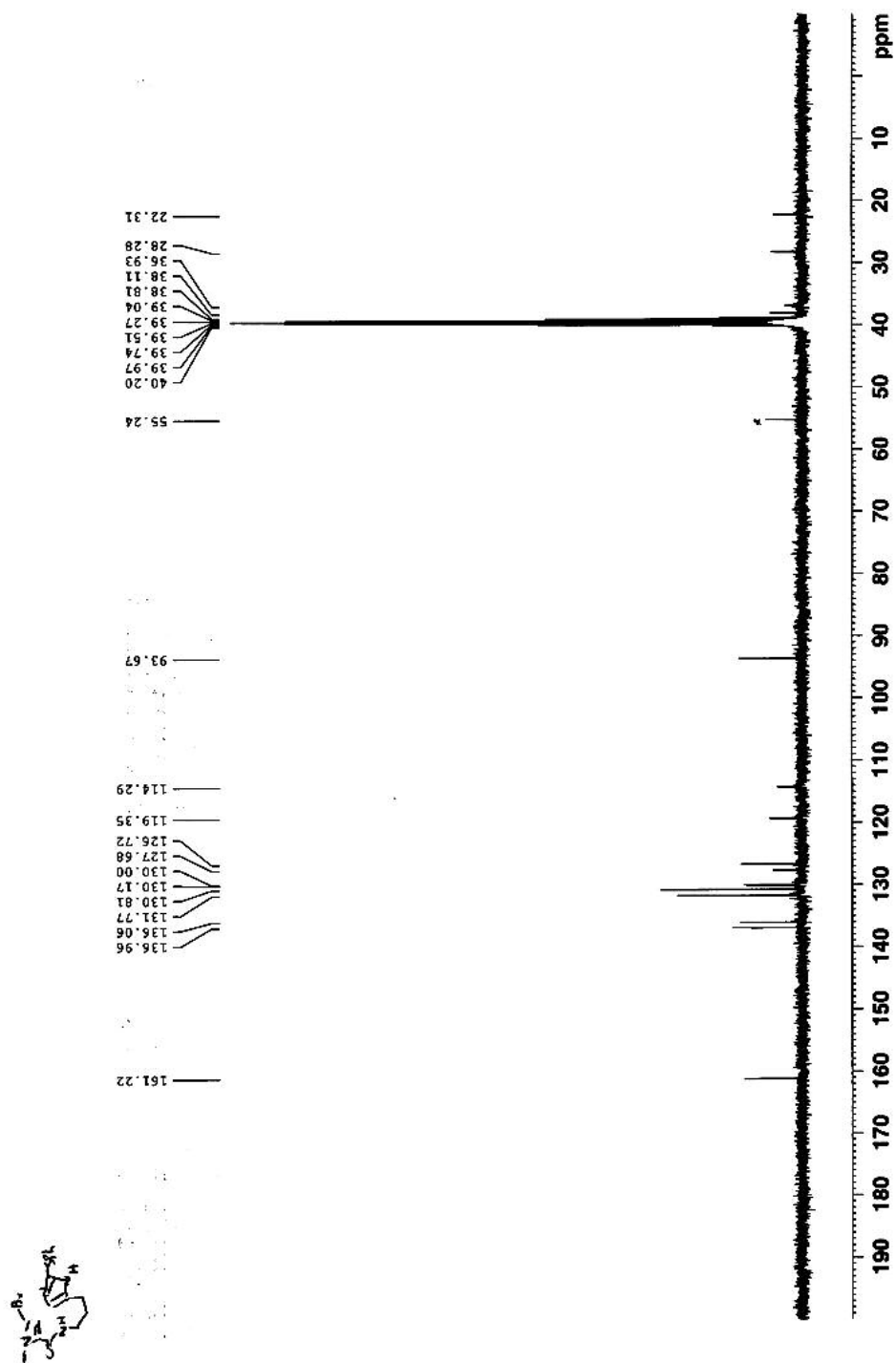
F2 - Processing parameters
SI 32768
SF 400.130085 MHz
MDM EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
FIP 11.000 ppm
F1 4401.43 Hz
F2P -1.000 ppm
F2 -400.13 Hz
PPMCH 0.60000 ppm/cm
HZCM 240.07800 Hz/cm









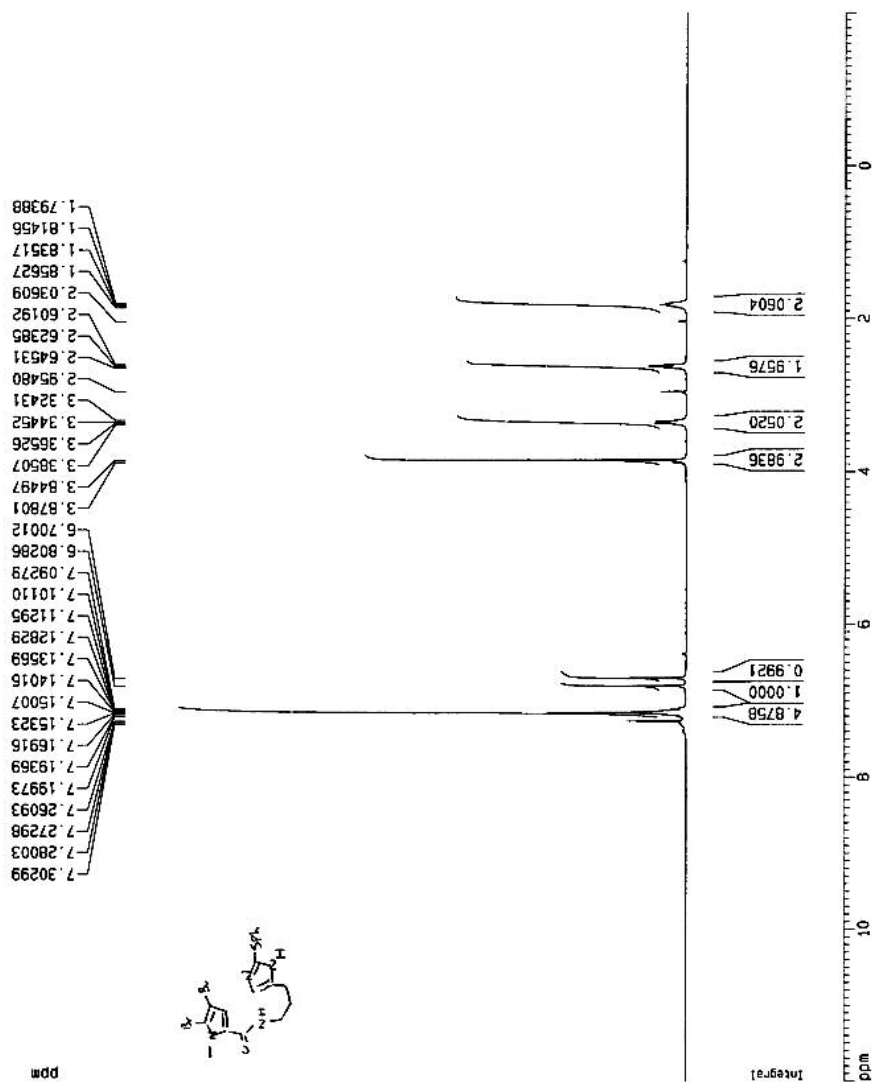
Current Data Parameters
NAME md1-051707
EXPNO 15
PROCNO 1

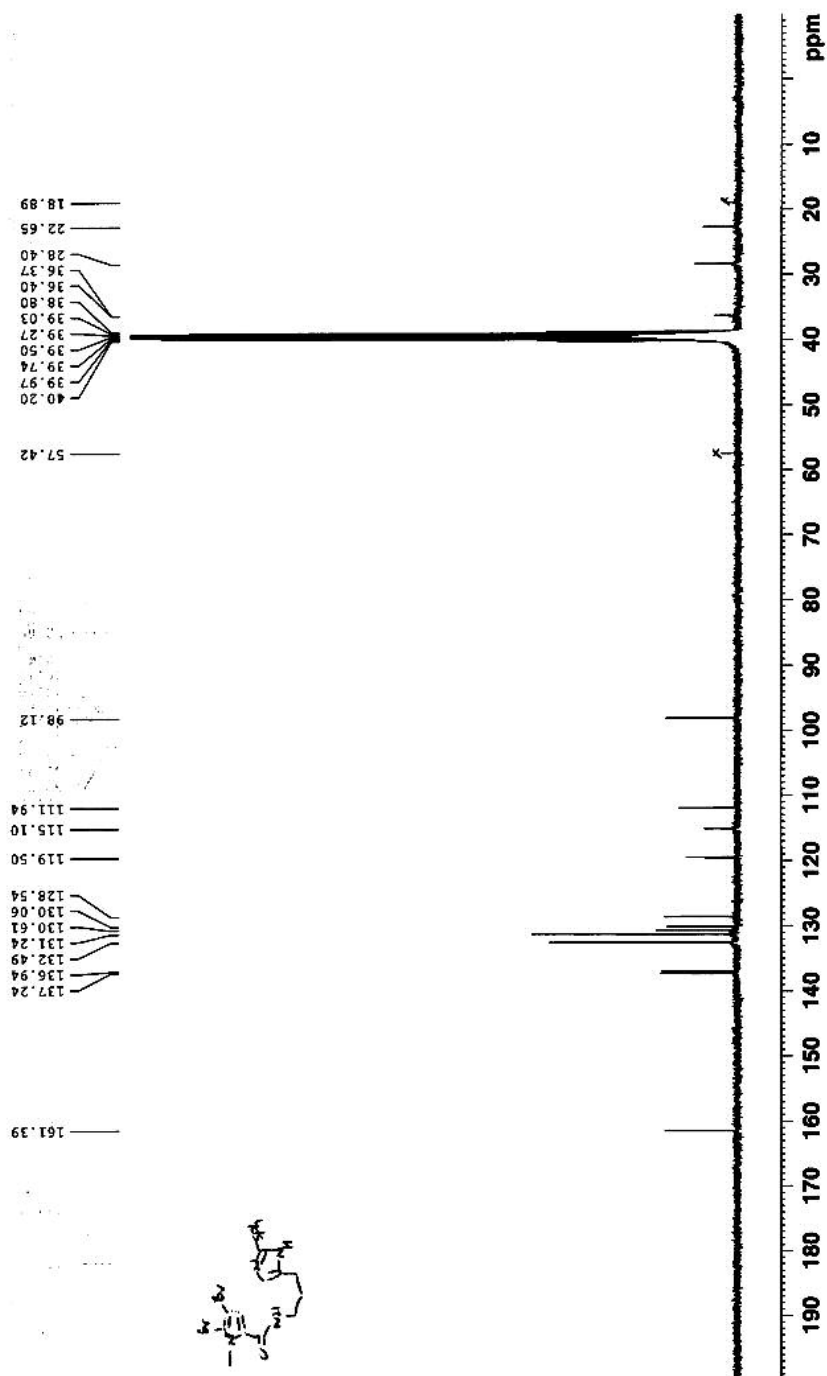
F2 - Acquisition Parameters
Date_ 20070517
Time 14:56
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg30
TD 65536
SOLVENT DMS
NS 16
DS 0
SWH 6172.839 Hz
FIDRES 0.376760 Hz
AQ 1.3271540 sec
RG 256
DM 81.000 usec
DE 5.00 usec
TE 300.0 K
D1 1.0000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 9.60 usec
PL1 -6.00 dB
SF01 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300051 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 12.000 ppm
F1 3601.56 Hz
F2P -2.000 ppm
F2 -600.26 Hz
PPMCM 0.70000 ppm/cm
HZCM 210.09100 Hz/cm





Current Data Parameters
 NAME mdf-051907
 EXPNO 6
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20070519
 Time 20:37
 INSTRUM spect
 PROBHD 5 mm BBI 1H-6
 PULPROG zgpg30
 TO 16384
 SOLVENT CDCl3
 NS 32
 DS 0
 SWH 8278.146 Hz
 FIDRES 0.505208 Hz
 AQ 0.9896436 sec
 RG 143.7
 DM 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec

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

NUC1 1H
 P1 6.45 usec
 PL1 0.00 dB
 SF01 400.1324710 MHz

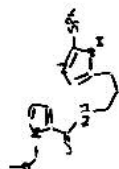
F2 - Processing parameters

SI 32768
 SF 400.1300072 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

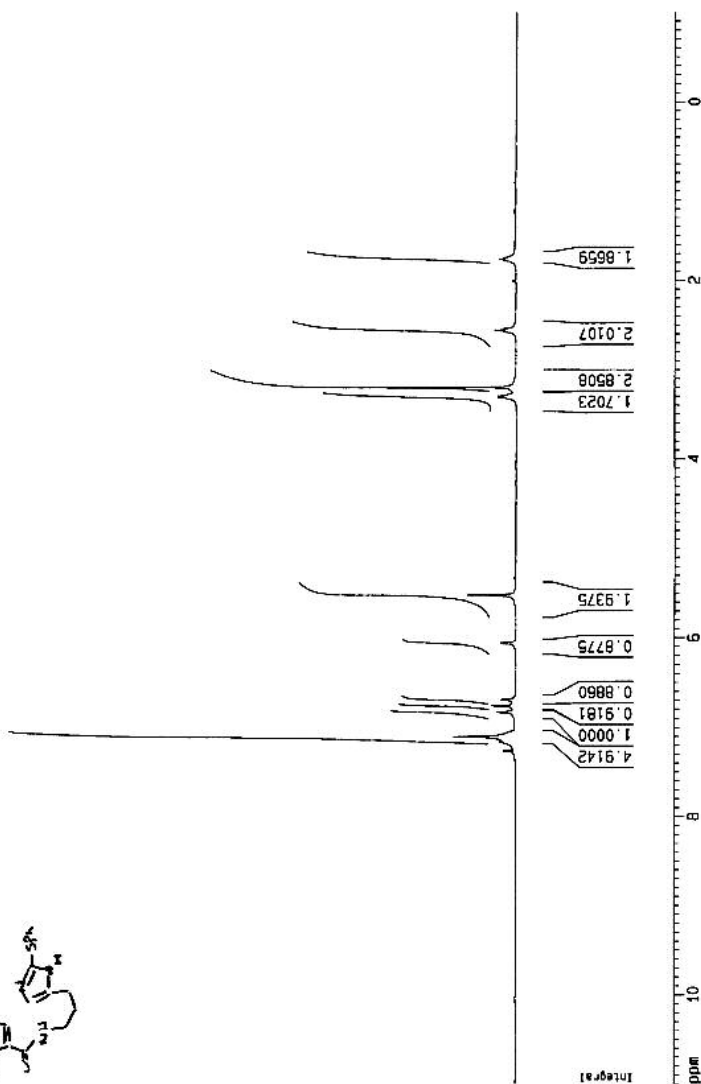
1D NMR plot parameters

CX 20.00 cm
 F1P 11.000 ppm
 F1 4401.43 Hz
 F2P -1.000 ppm
 F2 -480.13 Hz
 PPM0H 0.80000 ppm/cm
 MZCM 240.07800 Hz/cm

1.74433
 1.75078
 1.77711
 2.53966
 2.55652
 2.57346
 3.19666
 3.21782
 3.22761
 3.23015
 3.23559
 3.27975
 3.29501
 3.31010
 3.32499
 5.51717
 6.05435
 6.06115
 6.68293
 6.68851
 6.69213
 6.75603
 6.82560
 6.82918
 7.05416
 7.06152
 7.07499
 7.07930
 7.08301
 7.10042
 7.11653
 7.13560
 7.13860
 7.15826
 7.26095



ppm



Current Data Parameters
 NAME adf-052107
 EXPNO 31
 PROCNO 1

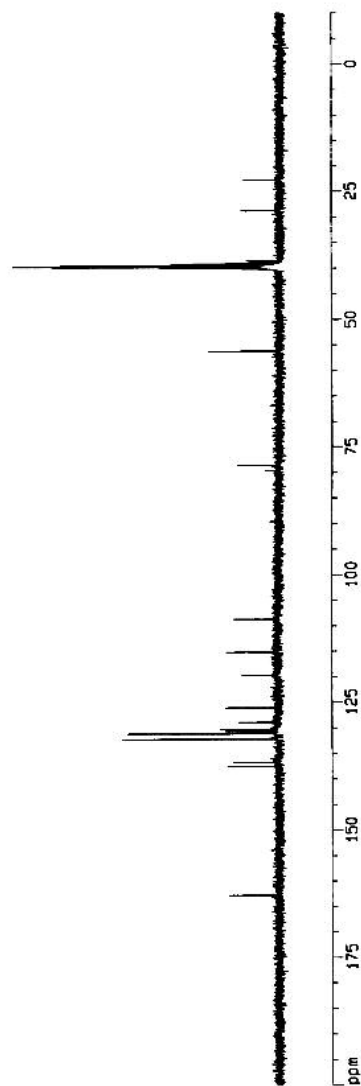
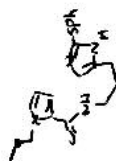
F2 - Acquisition Parameters
 Date_ 20070521
 Time 19.14
 INSTRUM spect
 PROBHD 5 mm BBI 1H-8
 PULPROG zgpg30
 TO 65536
 SOLVENT DMSO
 NS 129
 DS 4
 SWH 25125.629 Hz
 FIDRES 0.353387 Hz
 AQ 1.3542184 sec
 RG 65536
 DB 15.800 usec
 DE 6.00 usec
 TE 300.0 K
 T1 2.0000000 sec
 d11 0.0300000 sec
 d12 0.0005000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 15.35 usec
 PL1 -5.00 dB
 SF01 100.6237659 MHz

===== CHANNEL f2 =====
 CPOPRG2 waltz16
 NUC2 1H
 PPO2 114.00 usec
 PL2 0.00 dB
 PL12 24.00 dB
 PL13 24.00 dB
 SF02 400.1315005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127205 MHz
 NDM 64
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 200.000 ppm
 F1 20122.54 Hz
 F2P -10.000 ppm
 F2 -1006.13 Hz
 FWHM 10.300000 ppm/cg
 NZCM 1056.43559 Hz/cm



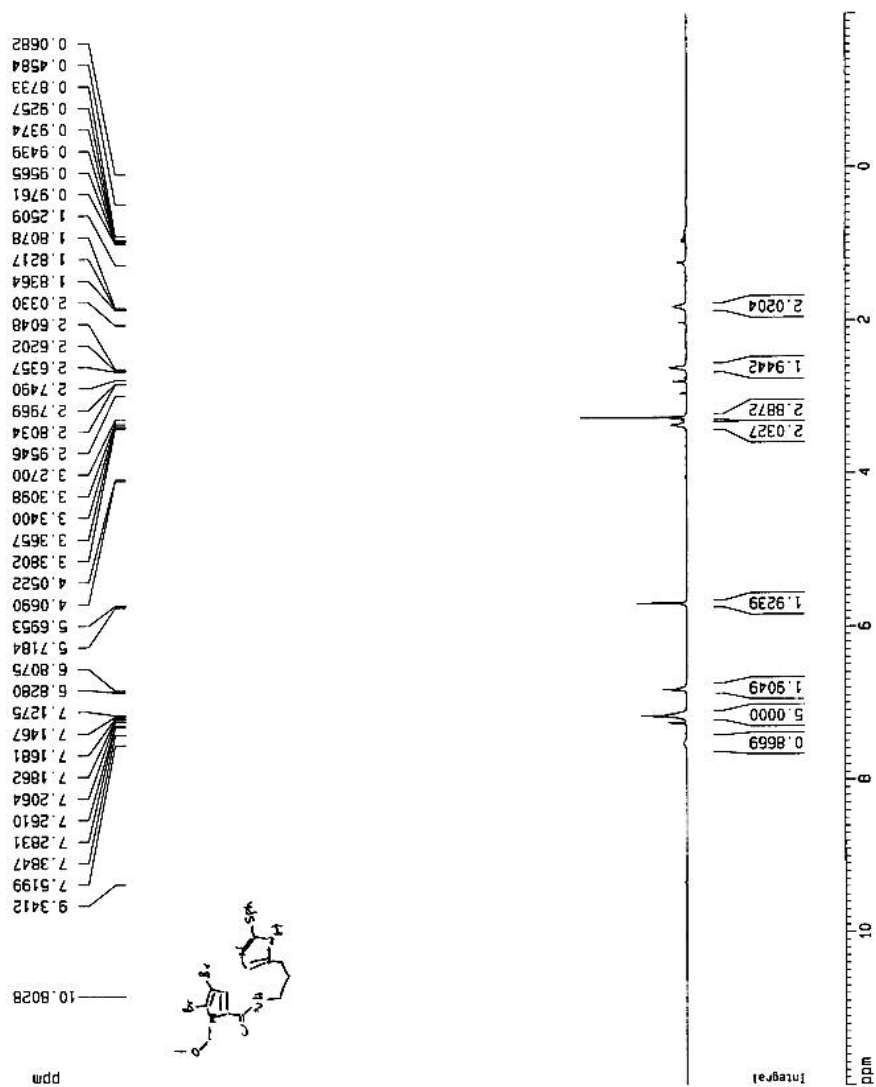
Current Data Parameters
 NAME mdf-051907
 EXPNO 1
 PROCNO 1

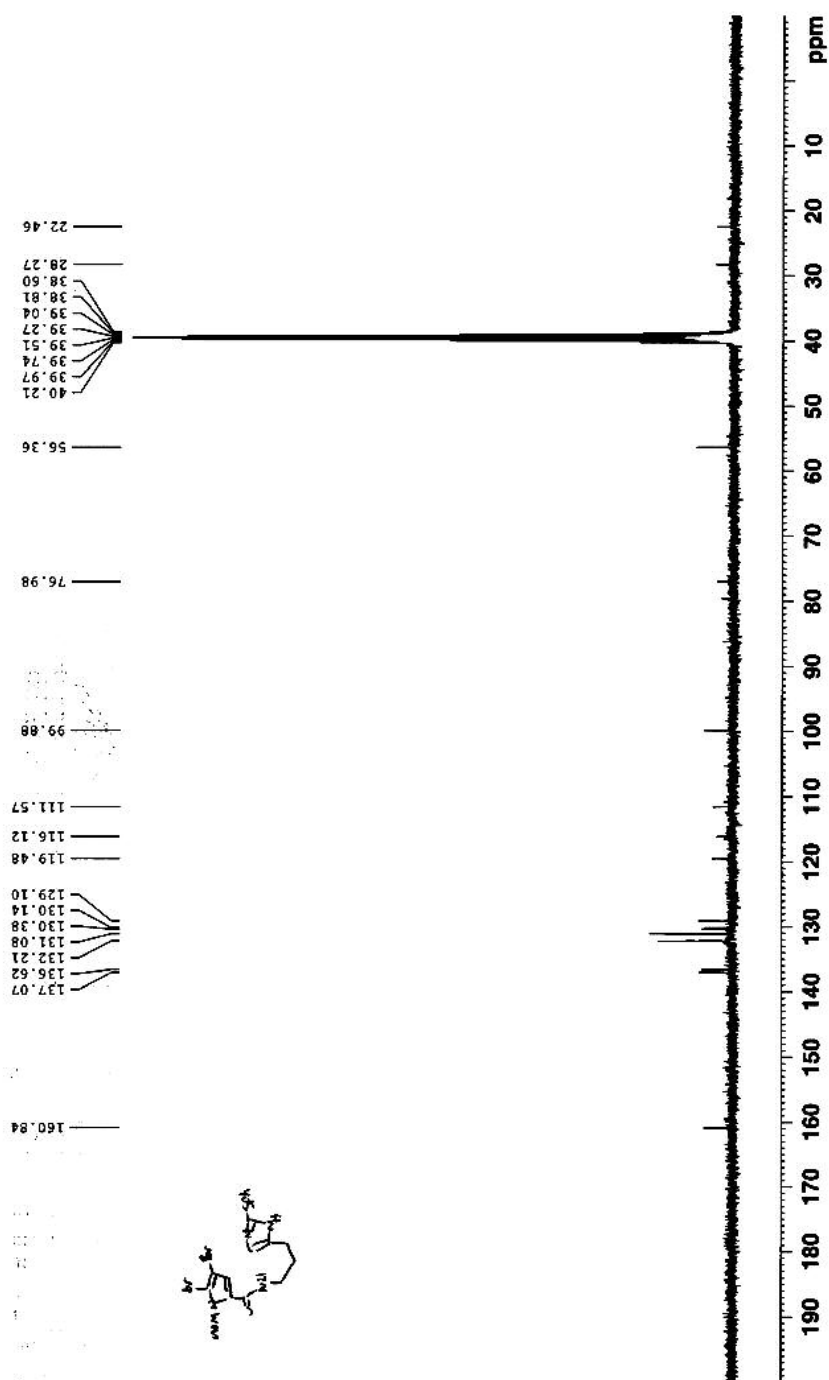
F2 - Acquisition Parameters
 Date_ 20070519
 Time 11:52
 INSTRUM spect
 PROBHD 5 mm BBI 1H-B
 PULPROG zg30
 TO 16384
 SOLVENT CDCl3
 NS 15
 DS 0
 SWH 8328.145 Hz
 FIDRES 0.565258 Hz
 AQ 0.9896436 sec
 RG 512
 DM 60.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 5.45 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.130067 MHz
 MDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 12.000 ppm
 F1 4801.56 Hz
 F2P -2.000 ppm
 F2 -800.25 Hz
 NUCLH 0.70000 ppm/cm
 HZCM 280.05100 Hz/cm





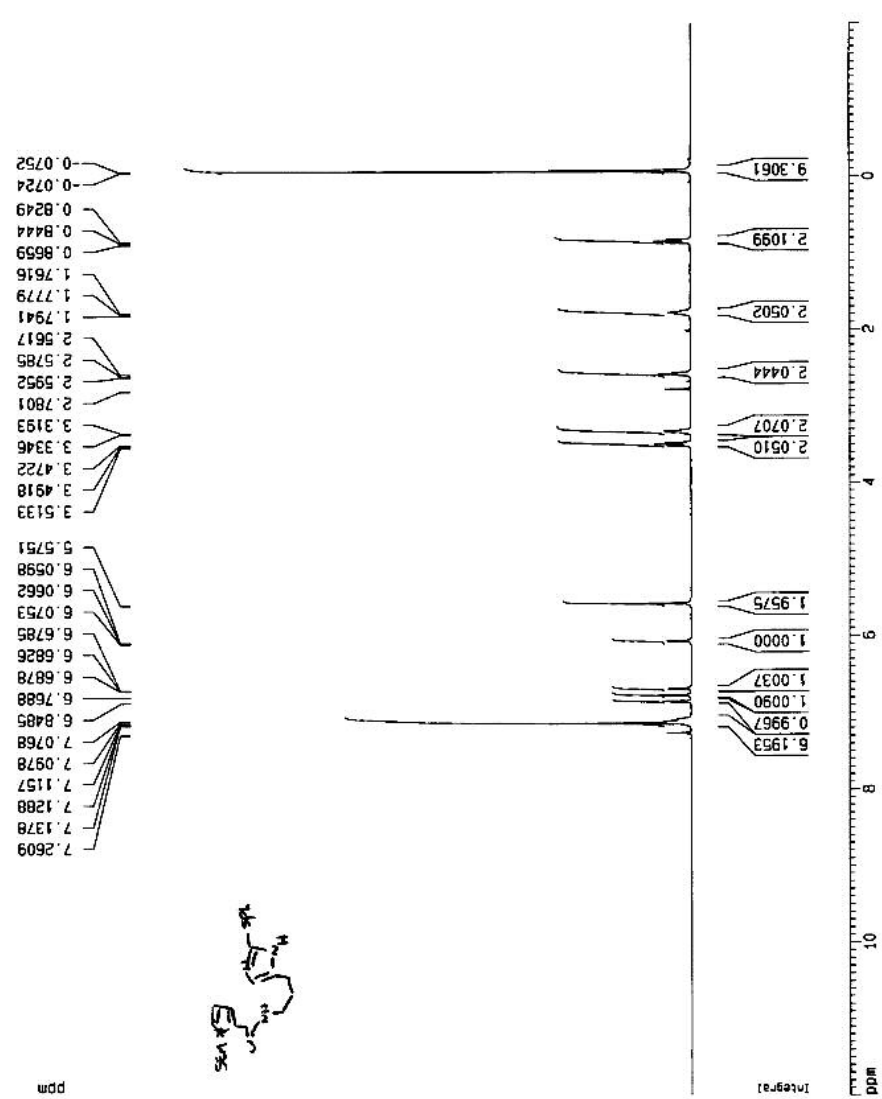
Current Data Parameters
NAME mdf-051907
EXPNO 7
PROCNO 1

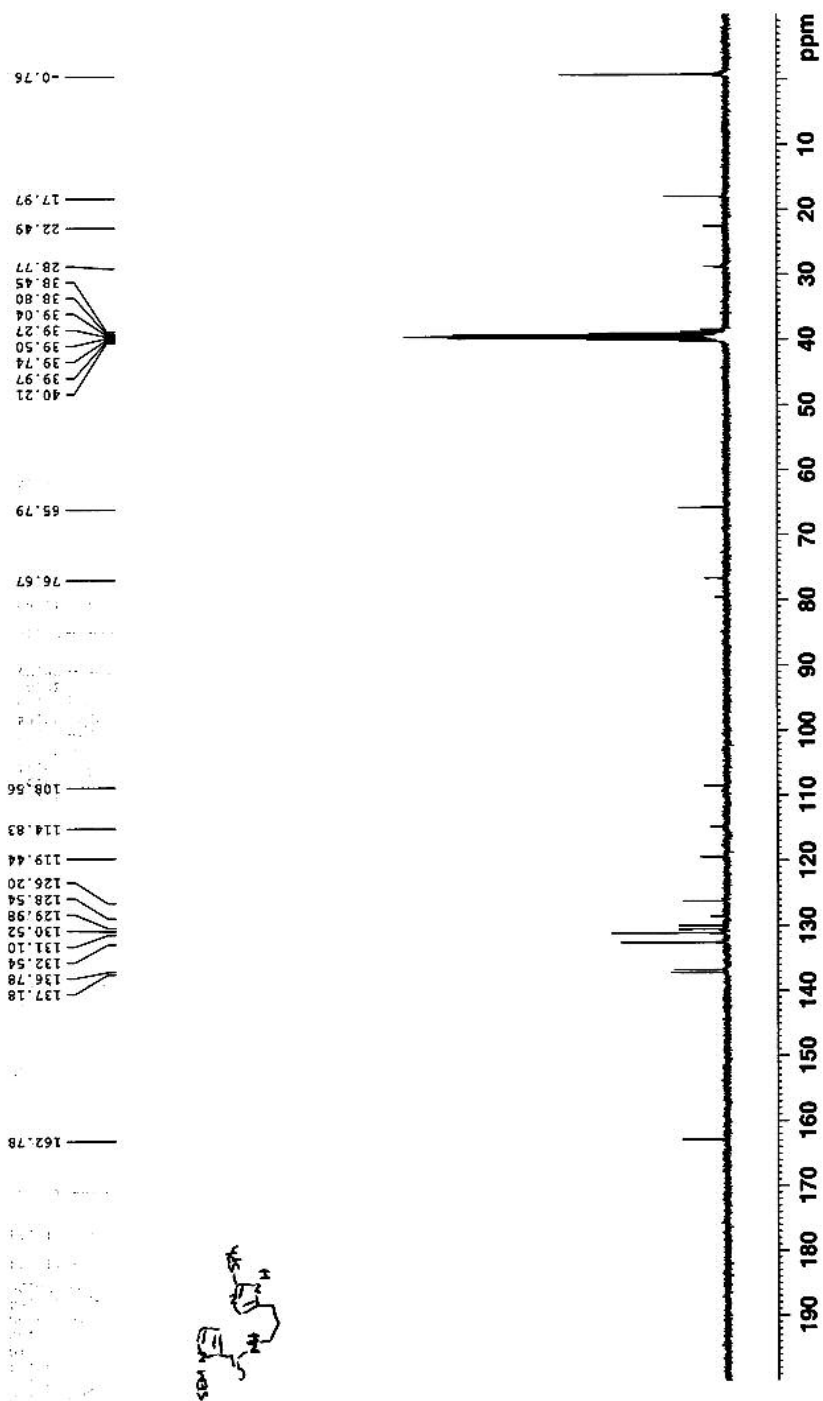
F2 - Acquisition Parameters
Date_ 20070519
Time 20.46
INSTRUM spect
PROBHD 5 mm BBI 1H-5
PULPROG zg30
TD 16384
SOLVENT DMS
NS 32
DS 2
SWH 8278.146 Hz
FIDRES 0.505236 Hz
AQ 0.9895436 sec
RG 161.3
DM 60.400 usec
DE 6.00 usec
TE 300.0 K
D1 1.0000000 sec

----- CHANNEL f1 -----
NUC1 1H
P1 6.45 usec
PL1 0.00 dB
SFO1 400.1324710 MHz

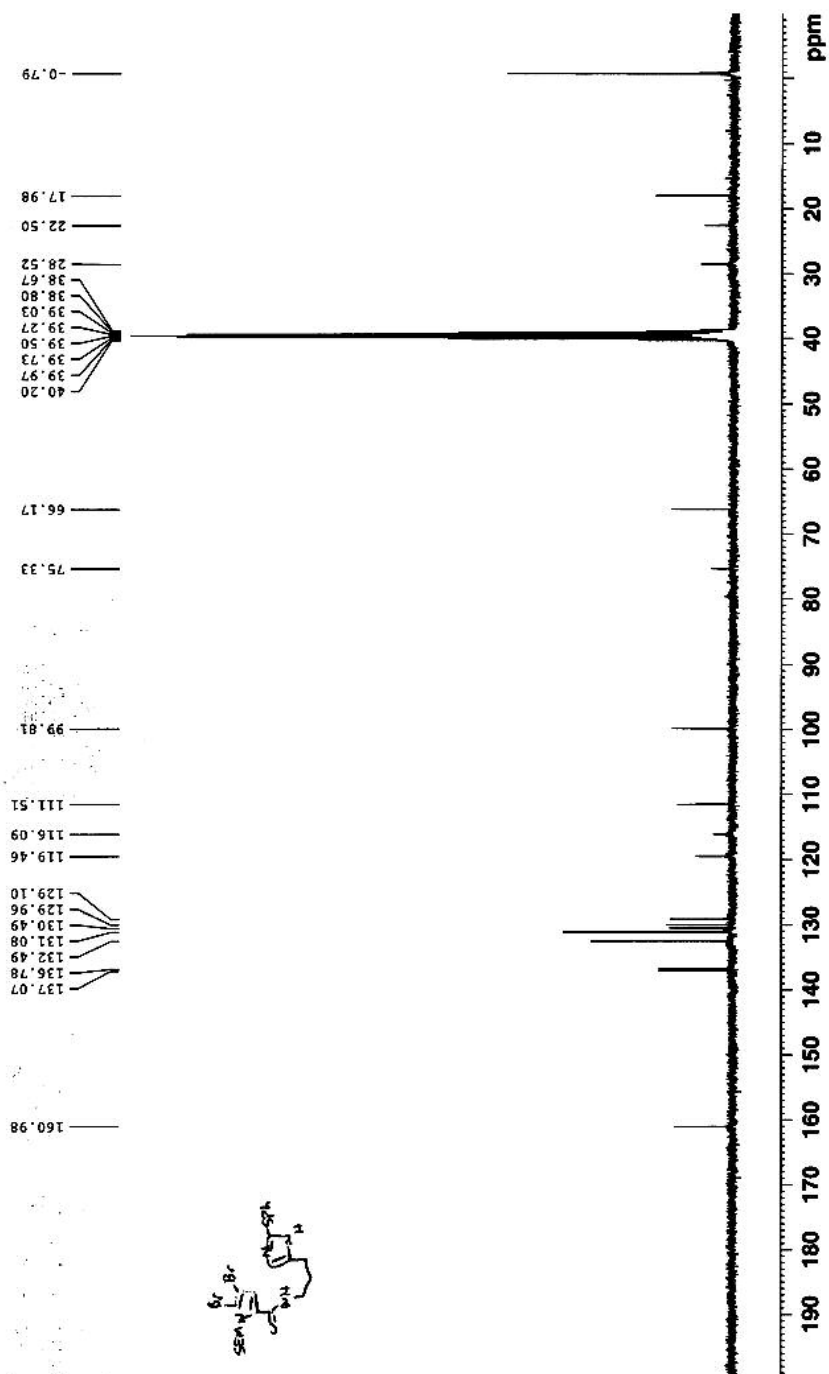
F2 - Processing parameters
SI 32768
SF 400.1300390 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CK 20.00 cm
F1P 12.000 ppm
F1 4801.55 Hz
F2P -2.000 ppm
F2 -800.26 Hz
PRGCM 0.70030 ppm/cm
HZCM 280.09100 Hz/cm









Current Data Parameters
 NAME mdf-061607
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20070615
 Time 15:19
 INSTRUM spect
 PROCNO 5 mm Multinu
 PULPROG zgpg30
 TO 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SNH 6172.839 Hz
 FIDRES 0.094150 Hz
 AQ 5.3084660 sec
 RG 101.5
 DM 81.000 usec
 DE 6.00 usec
 TE 300.0 K
 D1 1.0000000 sec

***** CHANNEL f1 *****

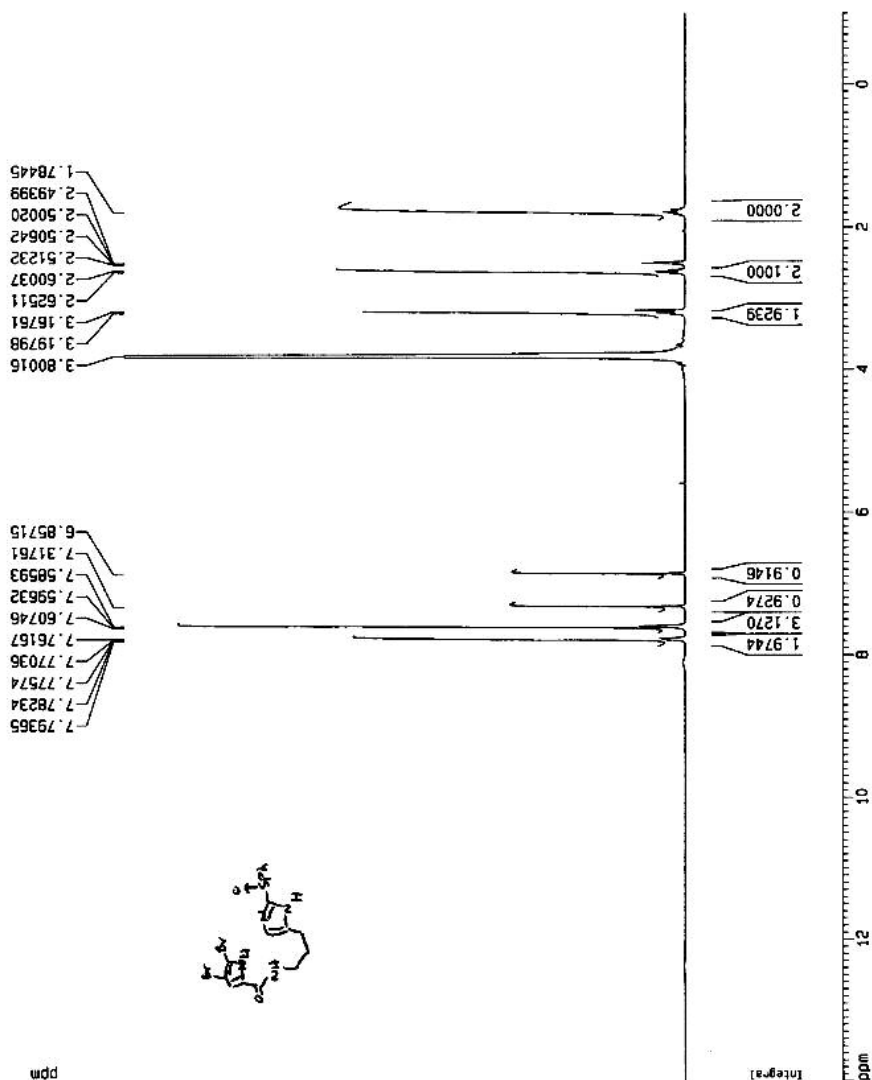
NUC1 1H
 P1 9.60 usec
 PL 1 -5.00 dB
 SF01 300.1310534 MHz

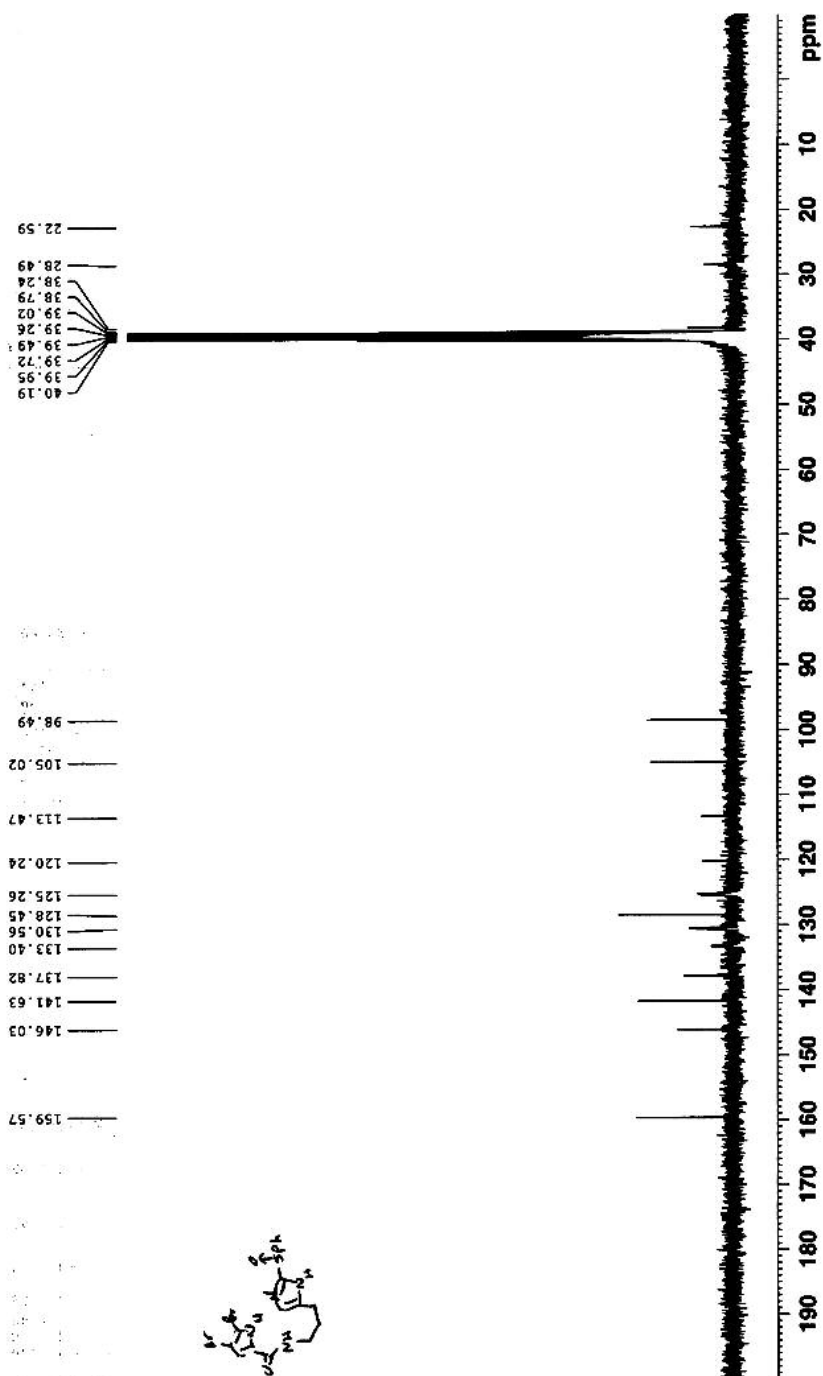
F2 - Processing parameters

SF 32768
 SF 300.1300016 MHz
 MDW no
 SSB 0
 LB 0.00 Hz
 DB 0
 PC 1.00

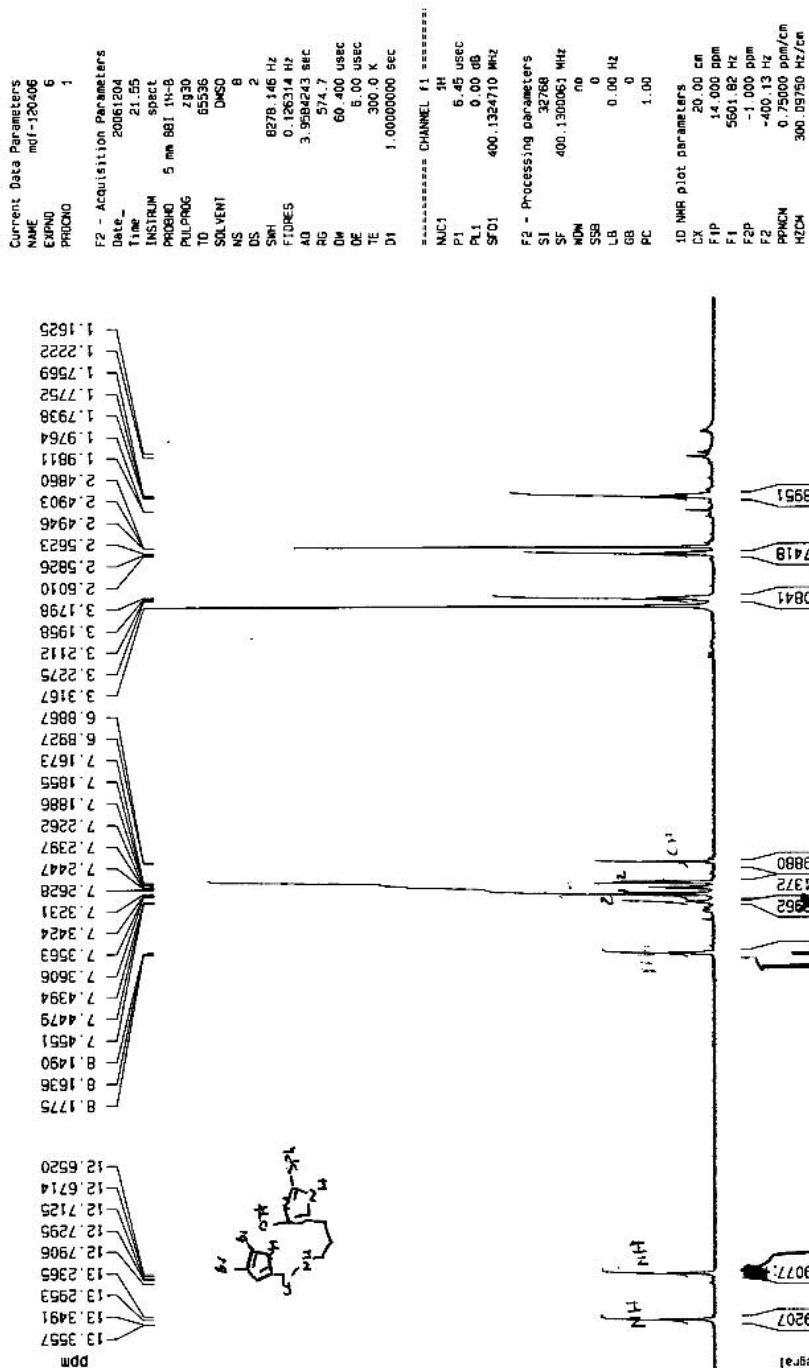
1D NMR plot parameters

CX 20.00 cm
 FIP 14.000 ppm
 F1 4201.82 Hz
 F2 -1.000 ppm
 PPMCH 0.75000 ppm/cm
 HZCM 225.09750 Hz/cm

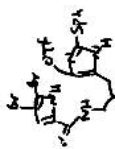
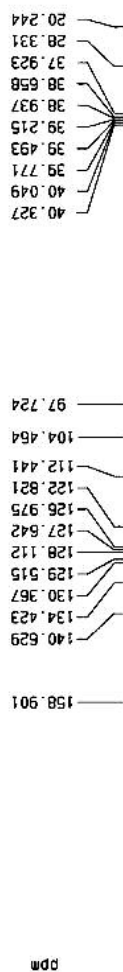




mlf-03-04s unknown product in DMSO - 400MHz



mdf-03-049 unknown product - 75MHz



Current Data Parameters
Name: mdf-03-049
EXPNO: 6
PROCNO: 1

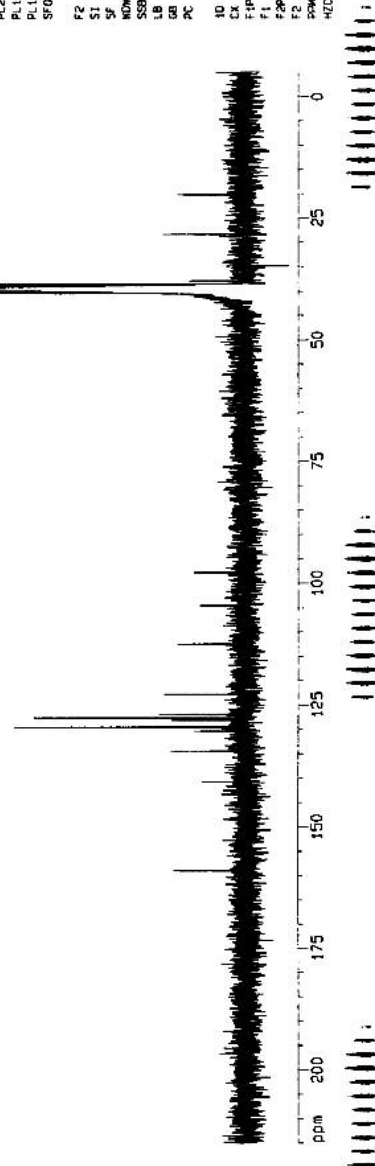
F2 - Acquisition Parameters
Date_ : 20081204
Time : 17.38
INSTRUM : spect
PROBHD : 5 mm WALT
PULPROG : zgpg30
TD : 65536
SOLVENT : DMSO
NS : 4096
DS : 4
SWH : 18832.393 Hz
FIDRES : 0.287360 Hz
AQ : 1.7400306 sec
RG : 8192
DM : 28.950 usec
DE : 6.00 usec
TE : 300.0 K
D1 : 2.00000000 sec
d11 : 0.03000000 sec
d12 : 0.00020000 sec

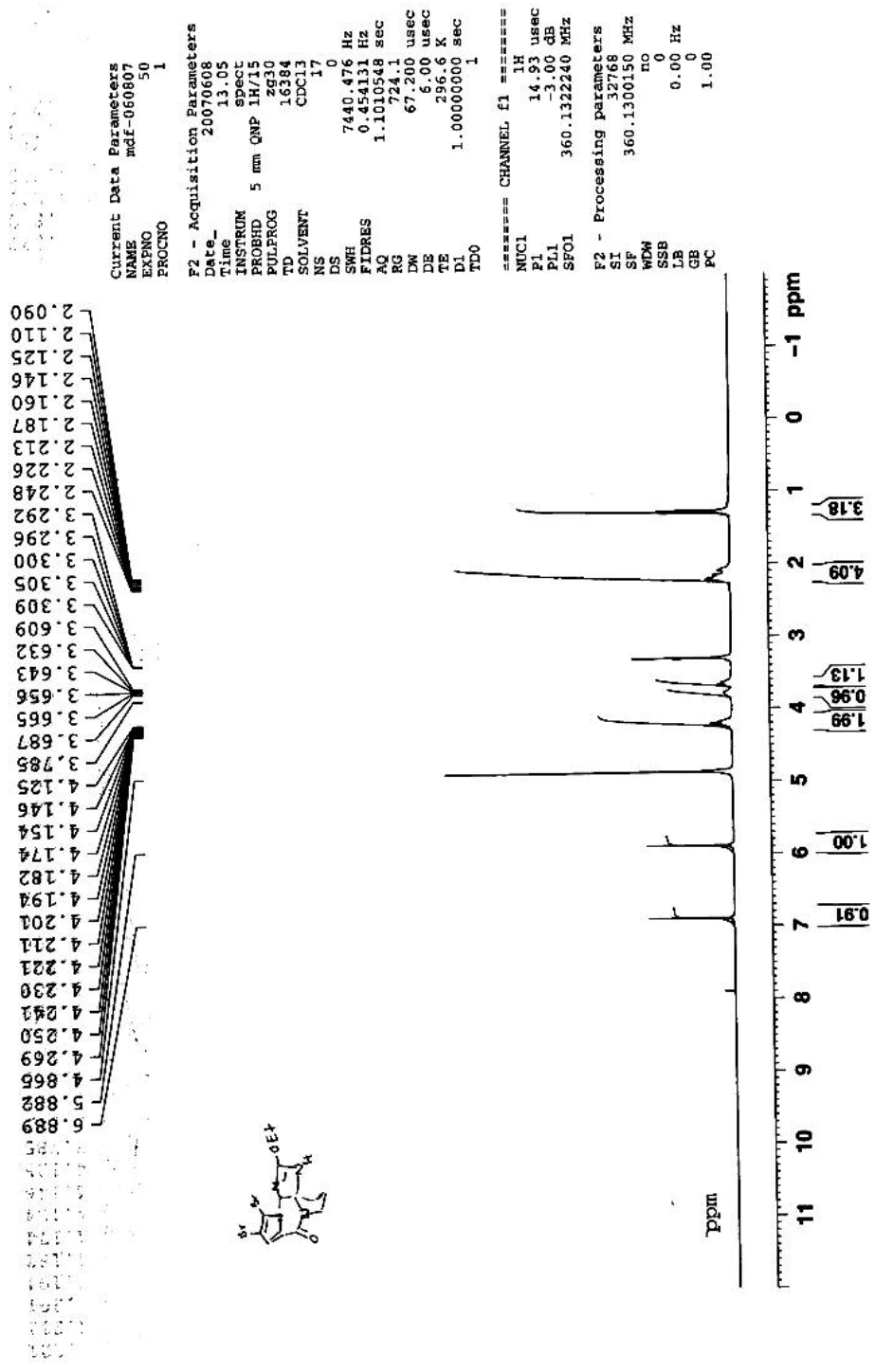
===== CHANNEL f1 =====
NUC1 : 13C
P1 : 11.80 usec
PL1 : 0.00 dB
SFO1 : 75.476000 MHz

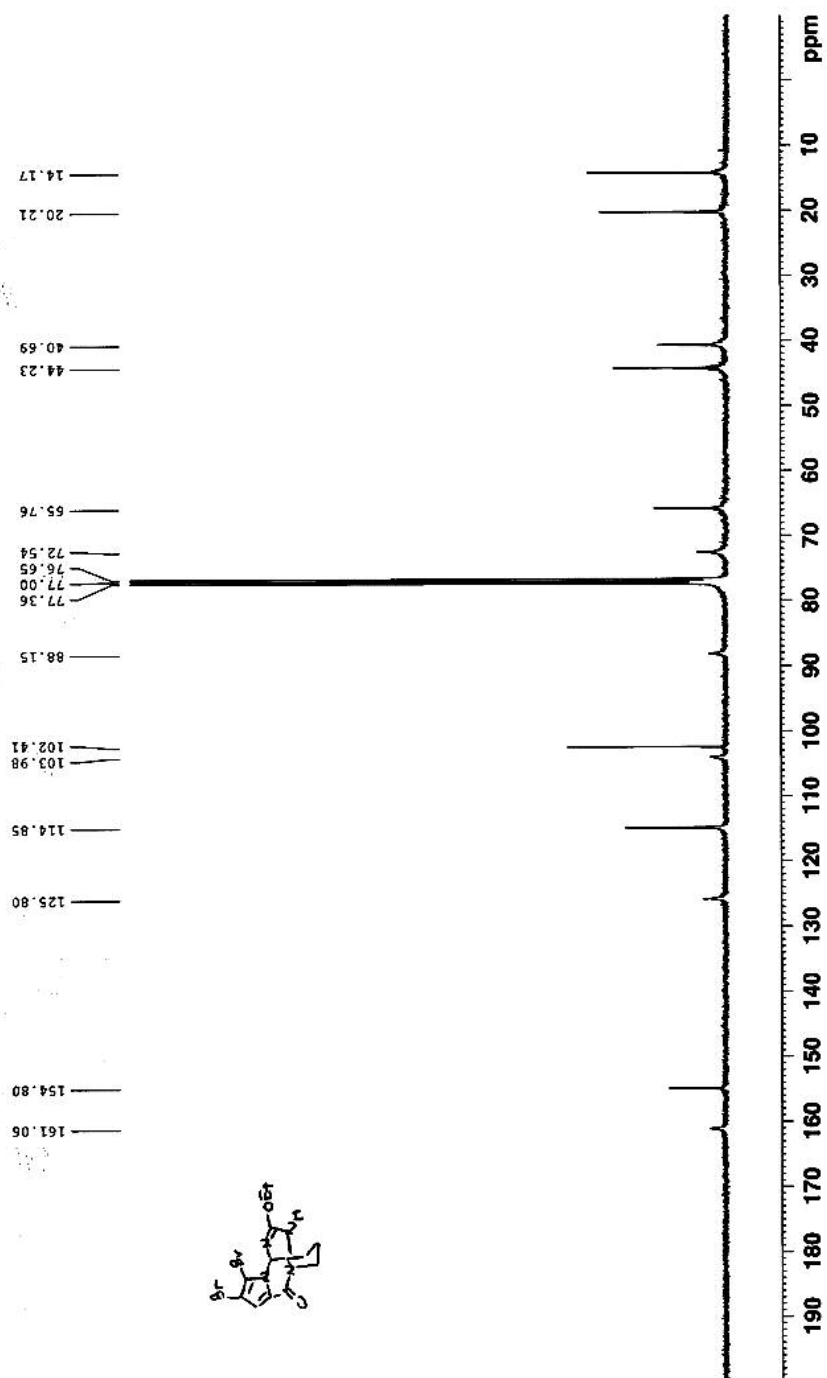
===== CHANNEL f2 =====
CPDPRG2 : waltz16
NUC2 : 1H
PCPD2 : 110.00 usec
PL2 : 0.00 dB
PL12 : 17.50 dB
PL13 : 17.50 dB
SFO2 : 300.131000 MHz

F2 - Processing parameters
SI : 32768
SF : 75.467784 MHz
WDW : EM
SSB : 0
LB : 1.00 Hz
GB : 0
PC : 1.40

10 MHz plot parameters
CX : 20.00 cm
FIP : 215.000 ppm
F1 : 16225.58 Hz
F2 : -5.000 ppm
F2 : -377.34 Hz
PRGM : 11.00000 ppm/cm
HZCM : 630.16569 Hz/cm







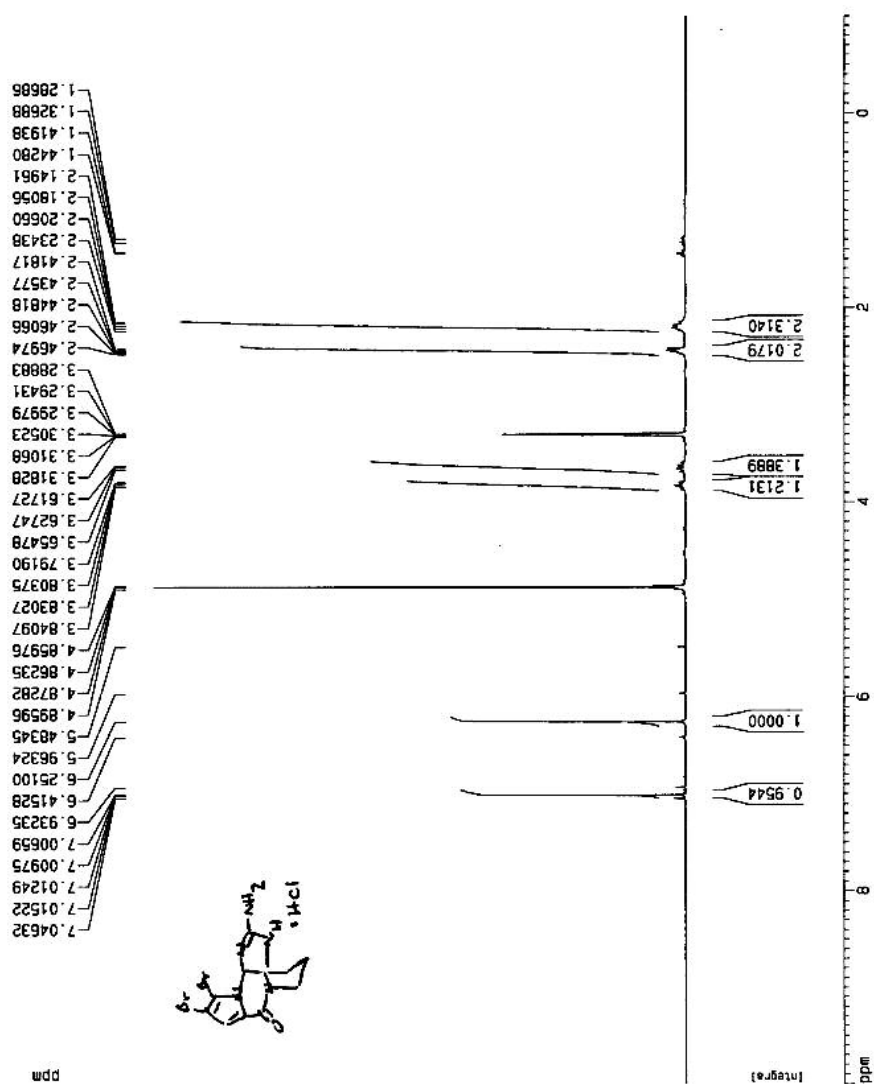
Current Data Parameters
NAME md-062107
EXPNO 11
PROCNO 1

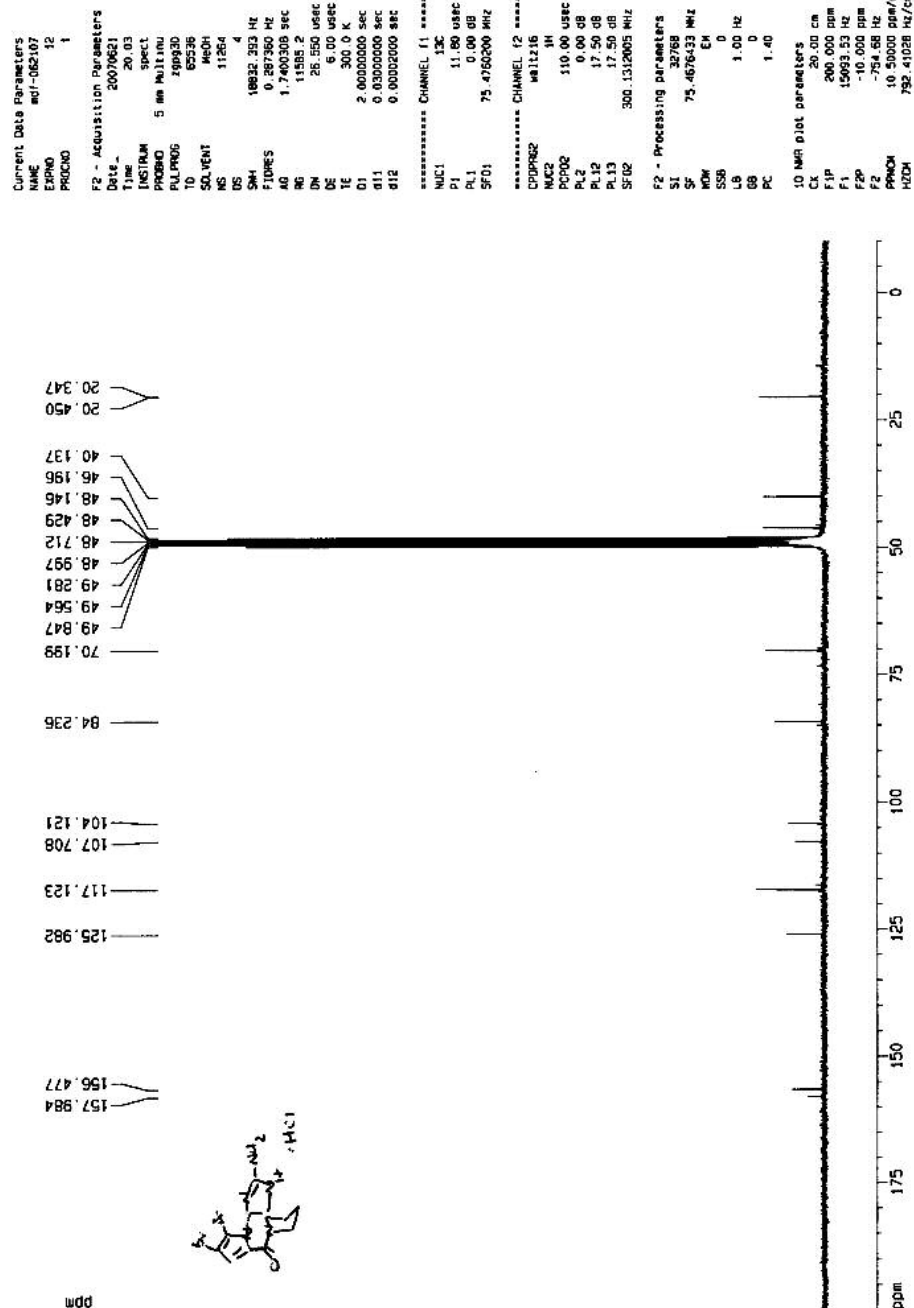
F2 - Acquisition Parameters
Date_ 20070621
Time 20.01
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zg30
TD 16384
SOLVENT MeOH
NS 16
DS 0
SWH 5172.839 Hz
FIDRES 0.376760 Hz
AQ 1.3271540 sec
RG 645.1
DM 81.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.0000000 sec

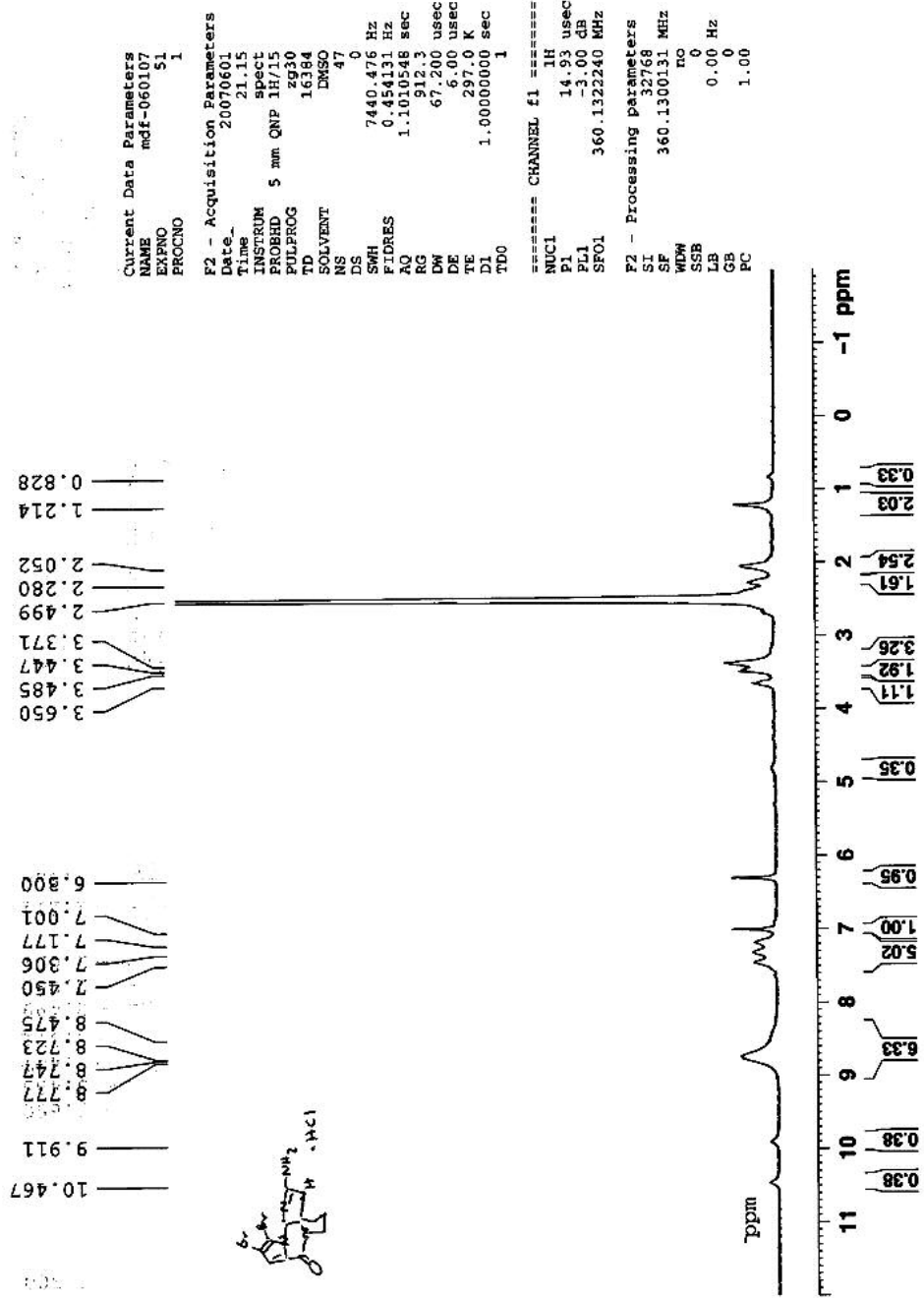
***** CHANNEL f1 *****
NUC1 1H
P1 9.60 usec
PL1 -6.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.130078 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 10.000 ppm
F1 3001.30 Hz
F2P -1.000 ppm
F2 -300.13 Hz
PPhOH 0.55000 ppm/cm
W2CN 165.07150 Hz/cm









Moisture- and oxygen-sensitive reactions were carried out in flame-dried glassware under an inert nitrogen atmosphere. Dry acetonitrile, dichloromethane, methanol and triethylamine were obtained by passing these solvents through activated alumina columns. Reagents were purchased at the highest commercial quality and used without further purification, unless otherwise stated. Diisopropylethylamine and 2,6-lutidine were distilled immediately prior to use. Reactions were monitored by thin layer chromatography carried out on 0.25 mm E. Merck silica gel plates (60F-254) with UV visualization. Purification of products via flash chromatography¹⁴ was performed with 40-63 μm silica gel and the solvent system indicated. Melting points are uncorrected. Copies of ^1H and ^{13}C NMR spectra are supplied in the Supporting Information as proof of purity.

General Procedure A: Phthalimide Hydrazinolysis. A stirring solution of phthalimidoimidazole in ethanol (0.1 M) was heated to reflux and treated with anhydrous hydrazine (20 equiv). A white precipitate began to form after approximately 5 min and the reaction was held at this temperature until the volume of this precipitate appeared to remain constant (ca. 30 min). The reaction solution was cooled to room temperature and partitioned between CH_2Cl_2 and H_2O (equal volumes). The aqueous layer was extracted once with CH_2Cl_2 and the combined organic fractions were washed with H_2O and brine, dried with Na_2SO_4 , and concentrated to give a yellow oil. Purification of this oil by flash column chromatography using 10-25% $\text{MeOH}(\text{NH}_3)/\text{CH}_2\text{Cl}_2$ as the eluent provided the pure aminoimidazole.

General Procedure B: Coupling of Trichloroacetyl Pyrrole Derivatives with Aminoimidazole Substrates. A stirring solution of aminoimidazole in acetonitrile (0.1 M) was treated with Na_2CO_3 (1 equiv) followed by addition of the corresponding acyl pyrrole (1 equiv). The resulting yellow solution was held at room temperature for 16 h. In cases where a solid precipitated, it was collected and rinsed with water (20 mL) and then with ether (50 mL) to yield an analytically pure product. In all other cases where no precipitate formed, the reaction mixture was concentrated in vacuo to yield a dark yellow oil. Purification of this oil by flash column chromatography using 10-100% EtOAc/hexanes as the eluent provided the pure carboxamide.

General Procedure C: Imidazole Deprotection. A stirring solution of carboxamide in THF (0.1 M) was heated to reflux. After 1 minute, 1.5 M HCl (4 equiv) was added and the reaction solution was held at reflux for 2 h after which time it was cooled to room temperature and poured into an ammonium hydroxide solution (an amount equal in volume to the HCl solution previously added). The resulting solution was partitioned between EtOAc (20 mL) and H_2O (10 mL) and the aqueous layer was extracted twice with EtOAc (2 x 20 mL). The organic fractions were combined, washed with brine (1 x 10 mL), dried with Na_2SO_4 and concentrated to give a yellow oil. Purification of this oil by flash column chromatography using 10-100% EtOAc/hexanes as the eluent provided the pure deprotection product.

General Procedure D: Pyrrole Protection. A stirring solution of acyl pyrrole in DMF (0.3 M) was treated with NaH (1.1 equiv) and the resulting yellow solution was

held at room temperature until the evolution of gas had ceased (ca. 10 min) after which the corresponding protecting agent (1.2 equiv) was added. After 1 hour, the resulting solution was partitioned between Et₂O (30 mL) and H₂O (10 mL) and the aqueous layer was extracted with Et₂O (3 x 10 mL). The combined organic fractions were washed with H₃PO₄ (3 x 10 mL), NaHCO₃ (3 x 10 mL), dried with Na₂SO₄, and concentrated to give a dark yellow oil. Purification of this oil by filtration through a short pad of silica using hexanes as the eluent provided the protected pyrrole.