

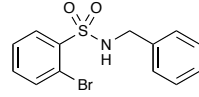
SUPPORTING INFORMATION

Title: Domino Heck–Aza-Michael Reactions: A One-Pot, Sequential Three-Component Approach to 1,1-Dioxido-1,2-benzisothiazoline-3-acetic Acid

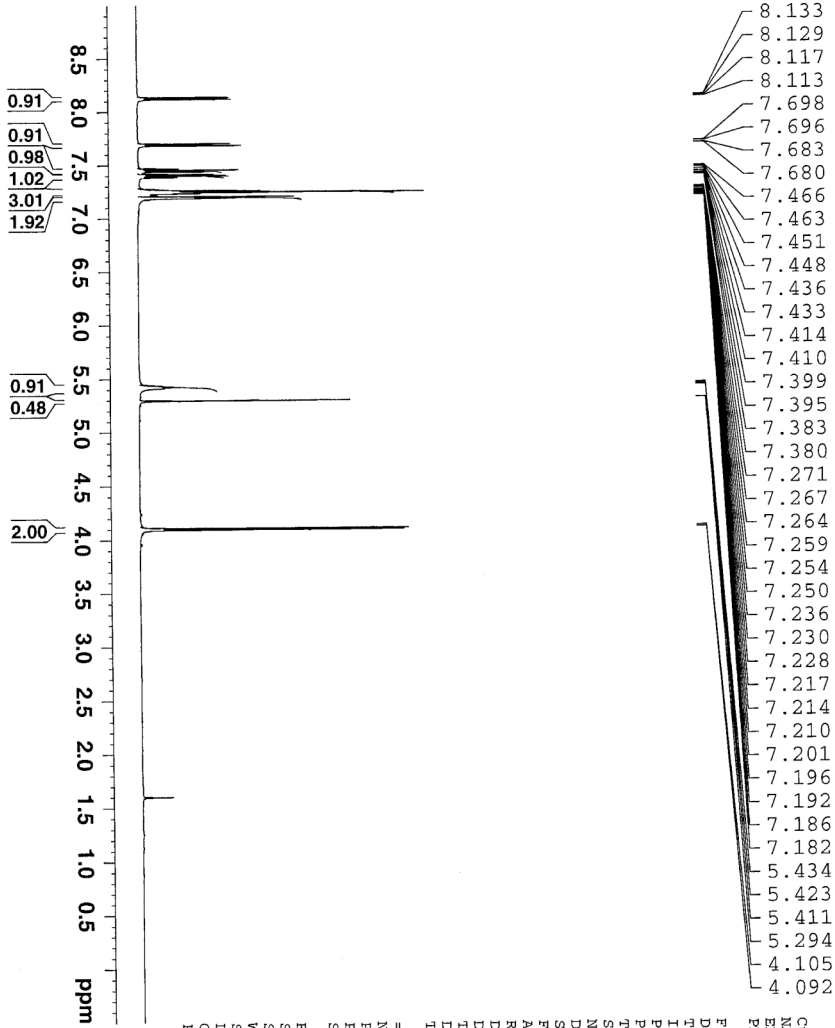
Author(s): Alan Rolfe, Kyle Young, Paul R. Hanson*

Ref. No.: O200800651

N-benzyl-2-bromobenzenesulfonamide (1)



PROTON CDCl3 opt/Topspin aro1fe 2



- 8.133
- 8.129
- 8.117
- 8.113
- 7.698
- 7.696
- 7.683
- 7.680
- 7.466
- 7.463
- 7.451
- 7.448
- 7.436
- 7.433
- 7.414
- 7.410
- 7.399
- 7.395
- 7.383
- 7.380
- 7.271
- 7.267
- 7.264
- 7.259
- 7.254
- 7.250
- 7.236
- 7.230
- 7.228
- 7.217
- 7.214
- 7.210
- 7.201
- 7.196
- 7.192
- 7.186
- 7.182
- 5.434
- 5.423
- 5.411
- 5.294
- 5.294
- 4.105
- 4.092



Current Data Parameters
 NAME AR_1273
 EXPNO 1
 PROCNO 1

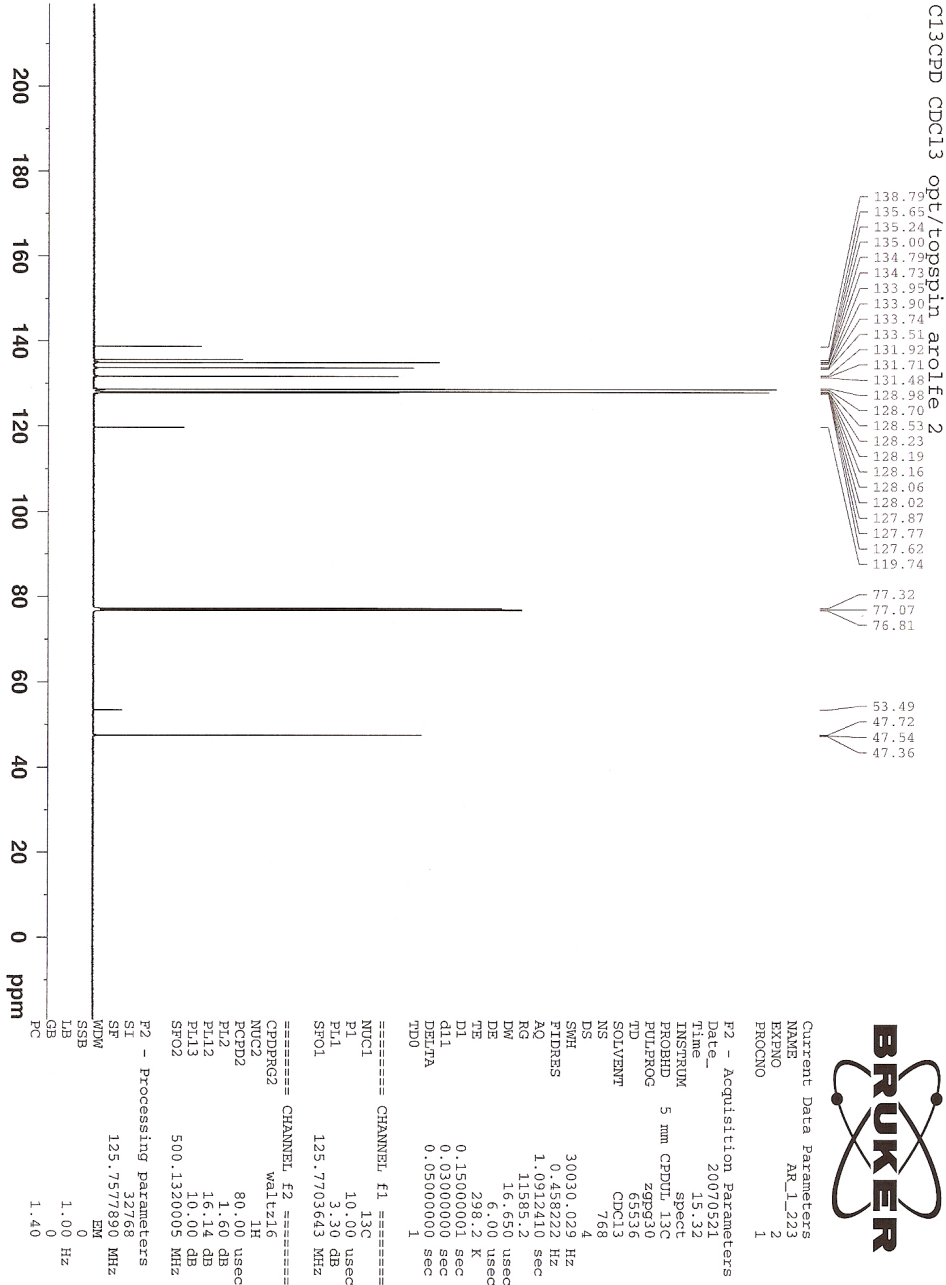
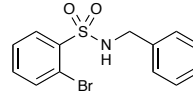
F2 - Acquisition Parameters
 Date_ 20070521
 Time 15.28

INSTRUM spect
 PROBHD 5 mm CPDUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRRS 0.157632 Hz
 AQ 3.1720407 sec
 RG 101.6
 DW 48.400 usec
 DE 6.00 usec
 TE 298.2 K
 D1 0.15000001 sec
 TD0 1

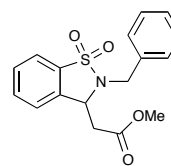
==== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 1.60 dB
 SFO1 500.1330885 MHz

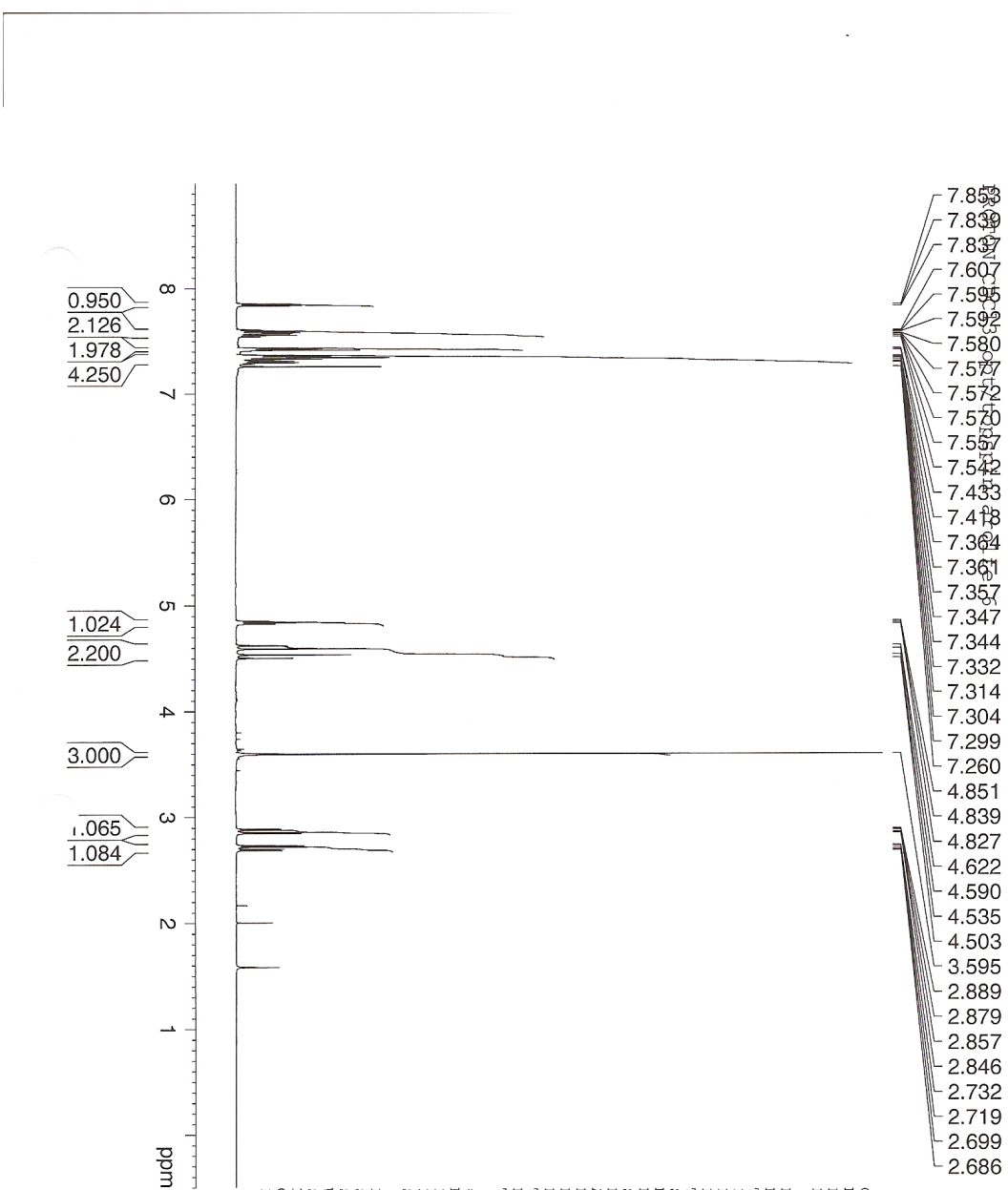
F2 - Processing Parameters
 SI 32768
 SF 500.1300143 MHz
 KDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

N-benzyl-2-bromobenzenesulfonamide (1)



Methyl 2-(2-benzyl-3-isothiazoline-1-yl)ethanoate 1,1' dioxide (2)





Current Data Parameters
 NAME AM_1224
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20070521
 Time 19.29
 INSTRUM spect
 PROBHD 5 mm CPUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1720407 sec
 RG 181
 DW 48.400 usec
 DE 6.00 usec
 TE 298.2 K
 D1 0.15000001 sec
 TD0 1

==== CHANNEL f1 =====
 NUCL1 1H
 P1 15.00 usec
 PL1 1.80 dB
 SFO1 500.1330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300139 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

- 7.859
- 7.839
- 7.837
- 7.607
- 7.595
- 7.592
- 7.580
- 7.577
- 7.572
- 7.570
- 7.557
- 7.542
- 7.433
- 7.418
- 7.364
- 7.361
- 7.357
- 7.347
- 7.344
- 7.332
- 7.314
- 7.304
- 7.299
- 7.260
- 4.851
- 4.839
- 4.827
- 4.622
- 4.590
- 4.535
- 4.503
- 3.595
- 2.889
- 2.879
- 2.857
- 2.846
- 2.732
- 2.719
- 2.699
- 2.686

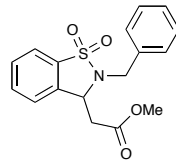
0.950
 2.126
 1.978
 4.250

1.024
 2.200

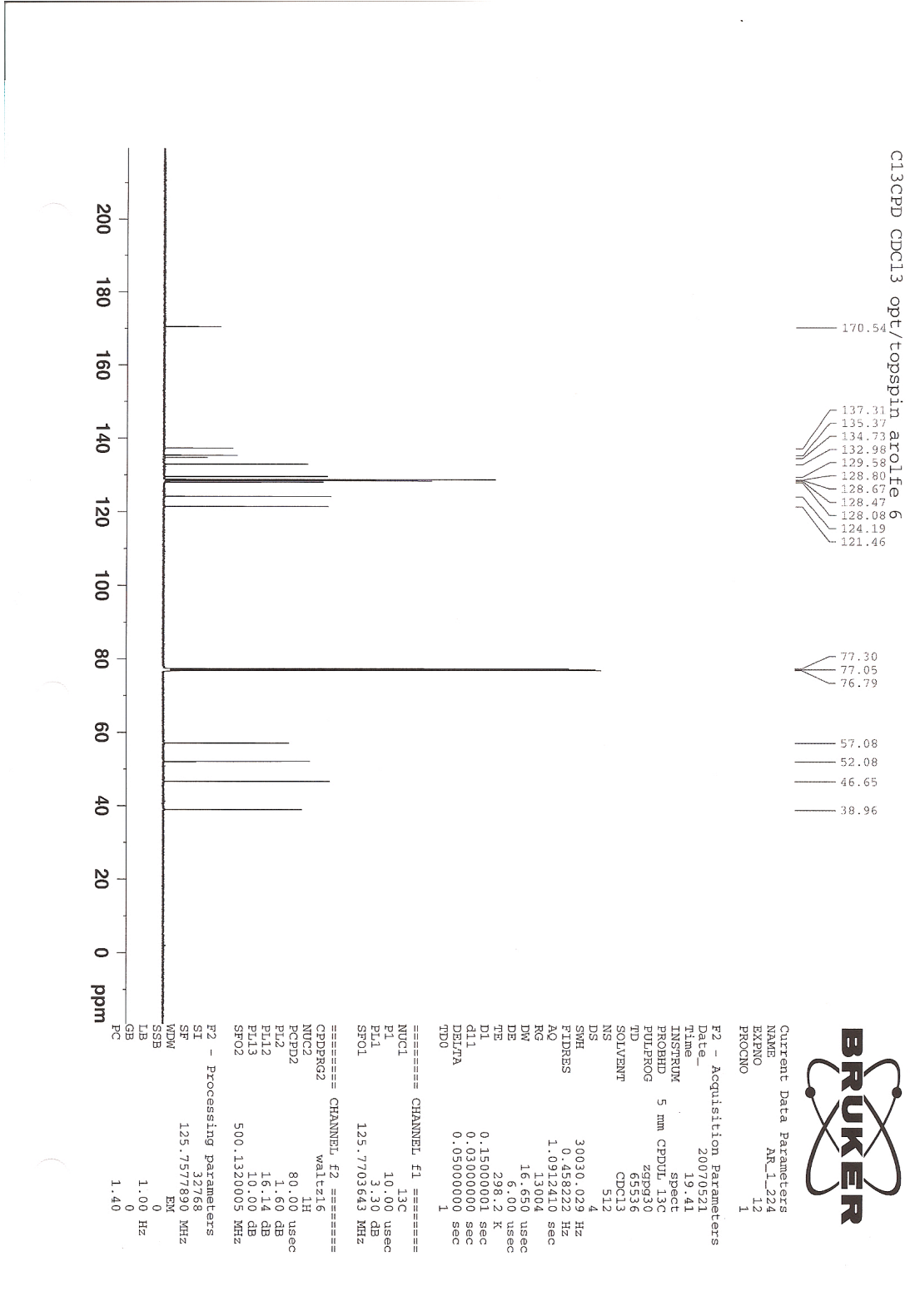
3.000

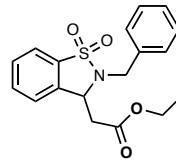
1.065
 1.084

ppm

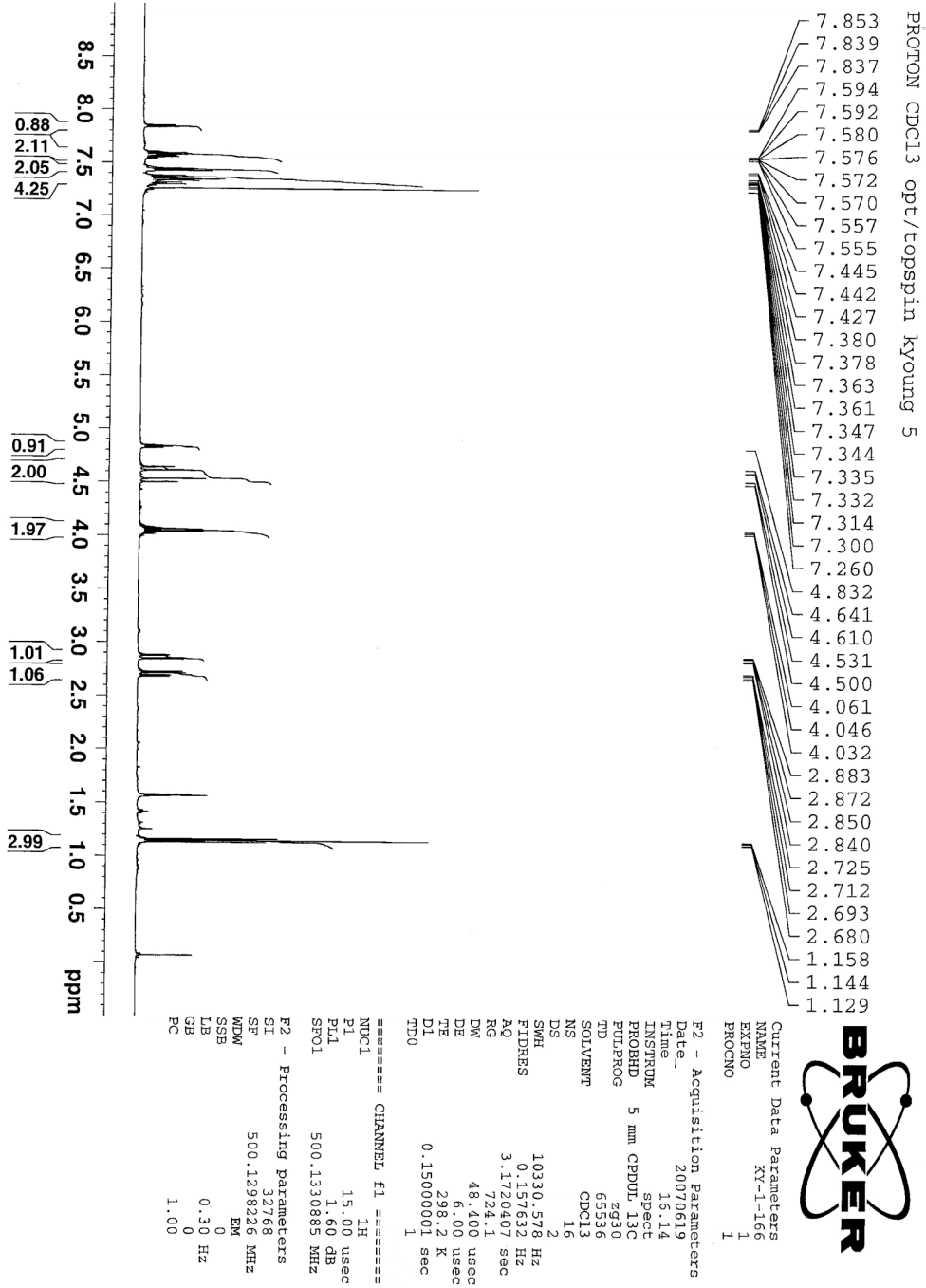


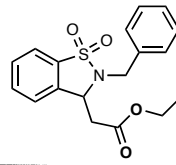
Methyl 2-(2-benzyl-3-isothiazoline-1-yl)ethanoate 1,1' dioxide (2)





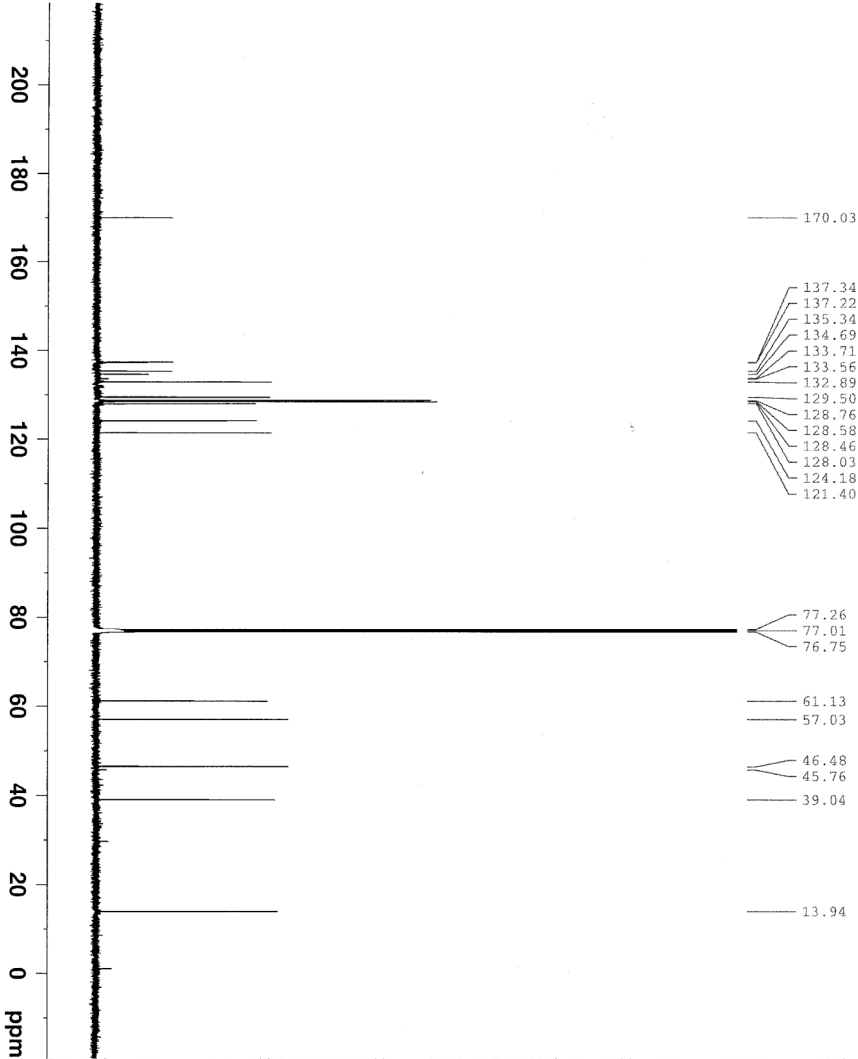
Ethyl 2-(2-benzyl-3-isothiazoline-1-yl)ethanoate 1,1' dioxide (3)





Ethyl 2-(2-benzyl-3-isothiazoline-1-yl)ethanoate 1,1' dioxide (3)

C13CPD CDCl3 opf/topsp1n kyoung 5



170.03
137.34
137.22
135.34
134.69
133.71
133.56
132.89
129.50
128.76
128.58
128.46
128.03
124.18
121.40

77.26
77.01
76.75
61.13
57.03
46.48
45.76
39.04

13.94

```

Current Data Parameters
NAME      K1-1-166
EXPNO    2
PROCNO   1

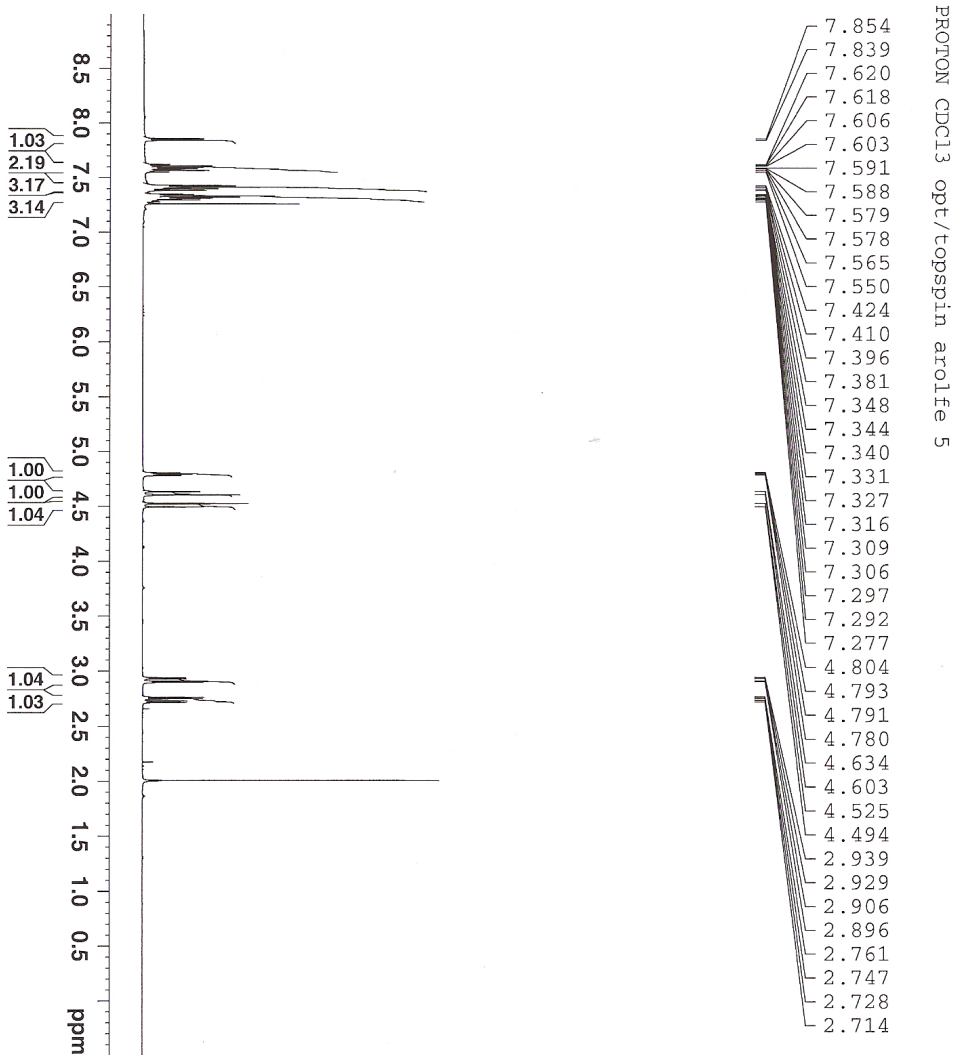
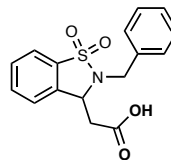
F2 - Acquisition Parameters
Date_    20070619
Time     16.26
INSTRUM  spect
PROBHD   5 mm CPDUL 13C
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       512
DS       4
SWH      30030.029 Hz
FIDRES   0.458222 Hz
AQ       1.0912410 sec
RG       13004
DW       16.650 usec
DE       6.00 usec
TE       298.2 K
D1       0.15000001 sec
d11      0.03000000 sec
DELTA    0.05000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1     13C
P1       11.00 usec
PL1      2.20 dB
SFO1     125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPDZ    80.00 usec
PL2      1.60 dB
PL12     16.14 dB
PL13     10.00 dB
SFO2     500.1320005 MHz

F2 - Processing parameters
SI       32768
SF       125.7577453 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
  
```

2-(2-Benzyl-3-isothiazoline-1-yl)ethanoic acid 1,1' dioxide (4)

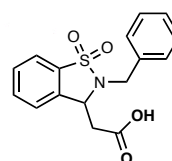


Current Data Parameters
 NAME AR_1_233
 EXPNO 11
 PROCNO 1

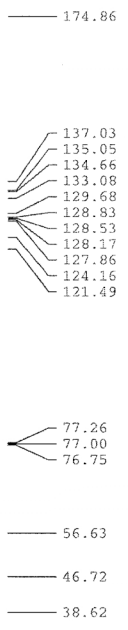
F2 - Acquisition Parameters
 Date_ 20070521
 Time 18.32
 INSTRUM spect
 PROBHD 5 mm CPDUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2

SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1720407 sec
 RG 203.2
 DW 48.400 usec
 DE 6.00 usec
 TE 298.2 K
 D1 0.15000001 sec
 TD0 1

==== CHANNEL f1 =====
 NUCL1 1H
 P1 15.00 usec
 PL1 1.60 dB
 SFO1 500.1330885 MHz
 F2 - Processing parameters
 SI 32768
 SF 500.1300150 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



2-(2-Benzyl-3-isothiazoline-1-yl)ethanoic acid 1,1' dioxide (4)



Current Data Parameters
 NAME AR_1_233
 EXPNO 12
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20070921
 Time 18.43
 INSTRUM spect
 PROBHD 5 mm CPDPL 13C
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 512
 DS 4
 SWH 30030.029 Hz
 FIDRES 0.458222 Hz
 AQ 1.0912410 sec
 RG 14596.5
 DW 16.650 usec
 DE 6.00 usec
 TE 298.2 K
 D1 0.15000001 sec
 d11 0.03000000 sec
 DELTA 0.05000000 sec
 TDO 1

==== CHANNEL f1 =====

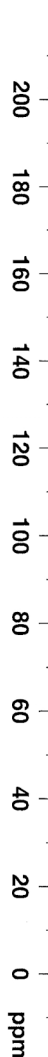
NUC1 13C
 P1 10.00 usec
 PL1 2.30 dB
 SFO1 125.7703643 MHz

==== CHANNEL f2 =====

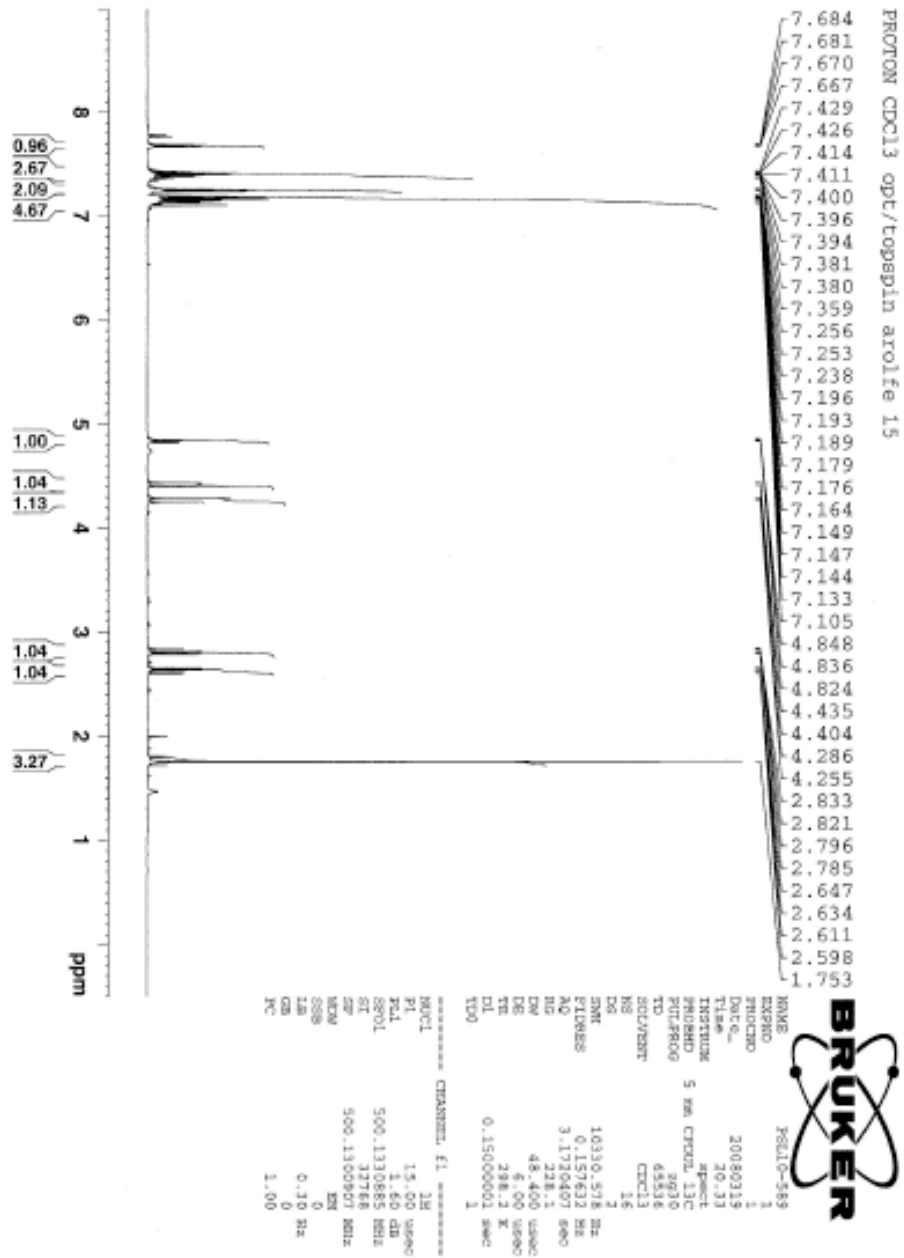
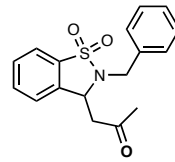
CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 1.60 dB
 PL12 16.14 dB
 PL13 10.00 dB
 SFO2 500.1320005 MHz

F2 - Processing parameters

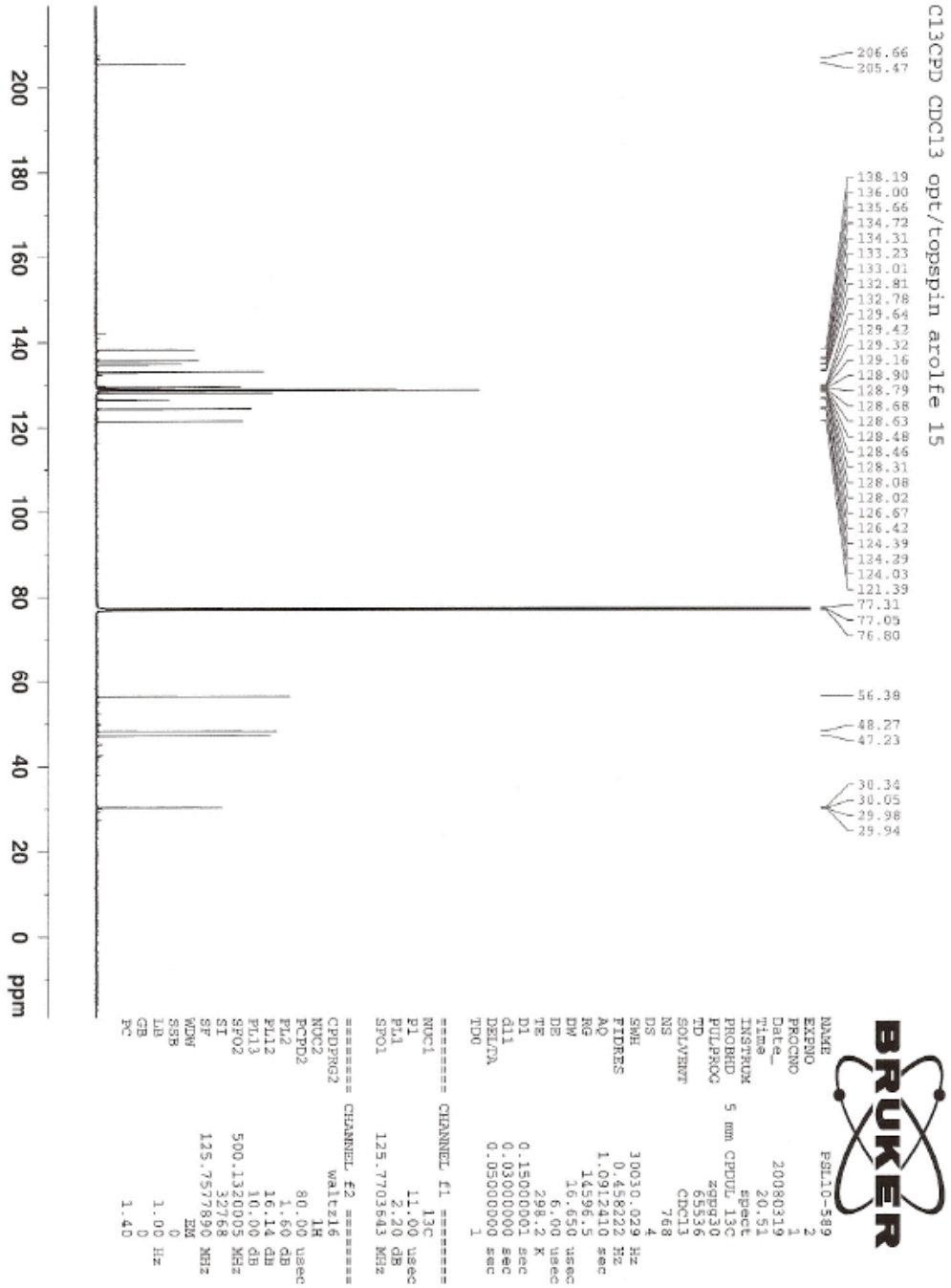
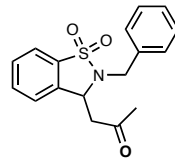
SI 32768
 SF 125.7577943 MHz
 MDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

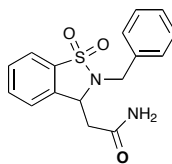


2-Benzyl-3-(2-propyl)isothiazoline-1-one 1,1' dioxide (5)

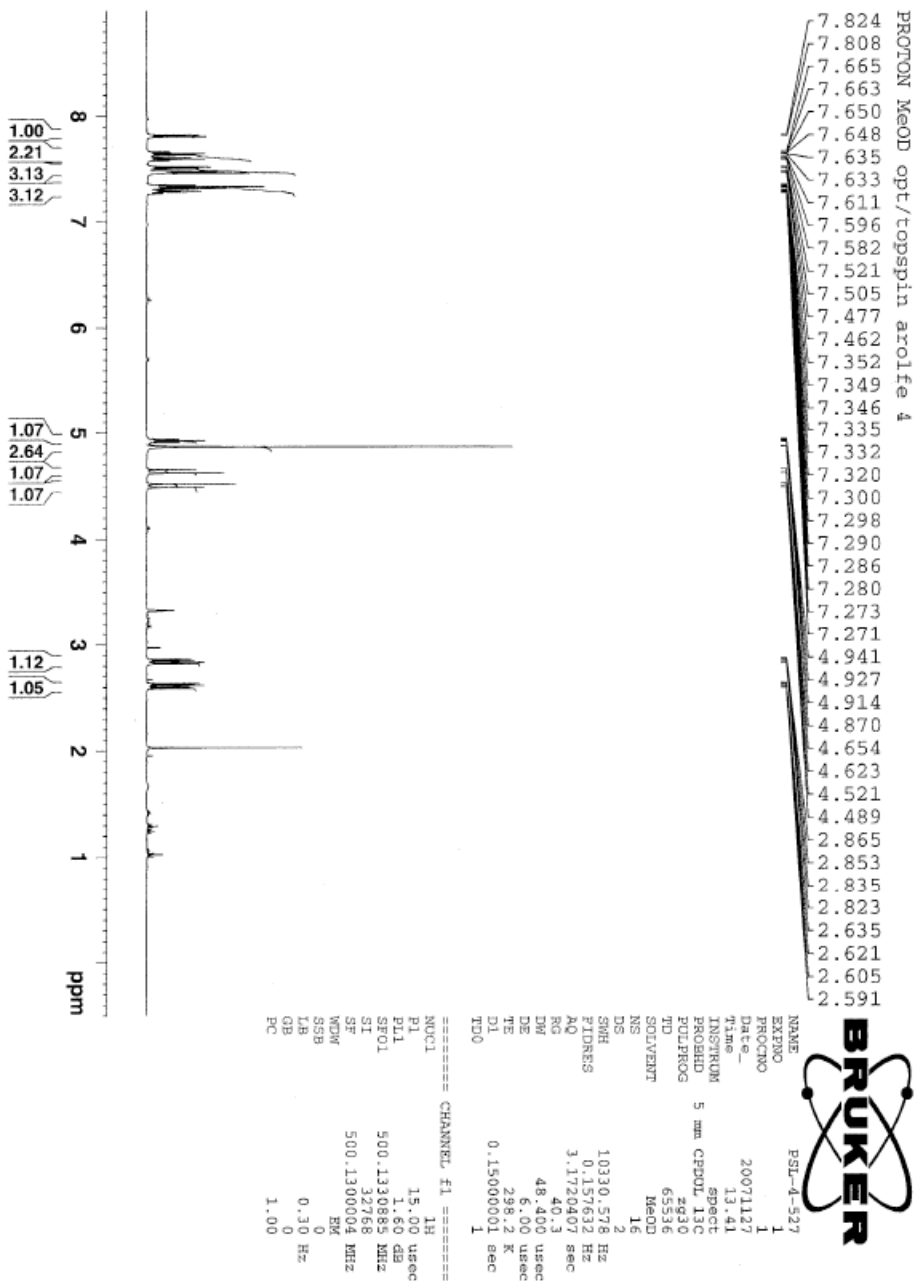


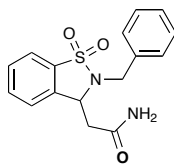
2-Benzyl-3-(2-propyl)isothiazoline-1-one 1,1' dioxide (5)



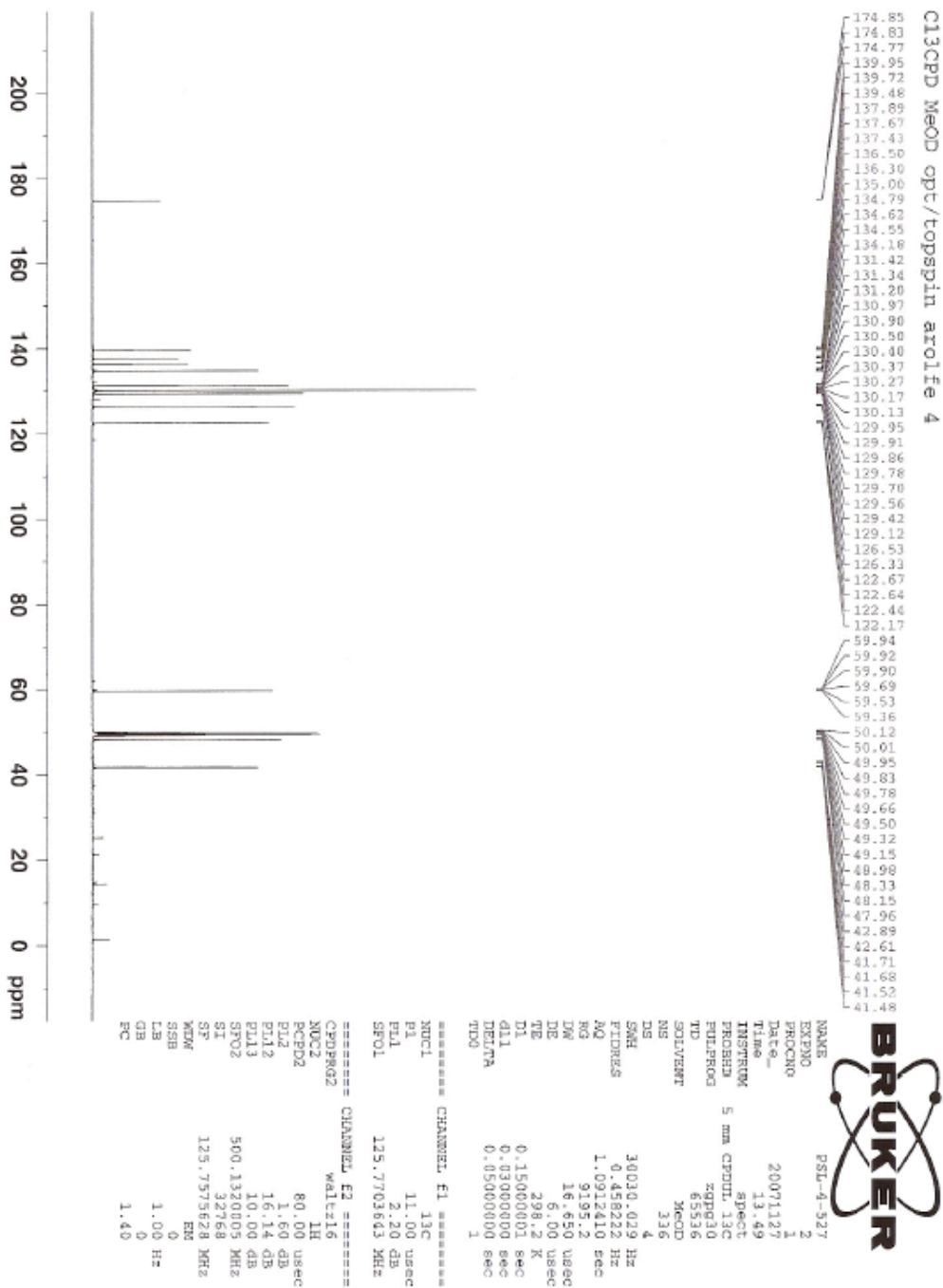


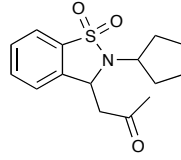
2-Benzyl-3-(2-propyl)isothiazoline-1-one 1,1' dioxide (6)



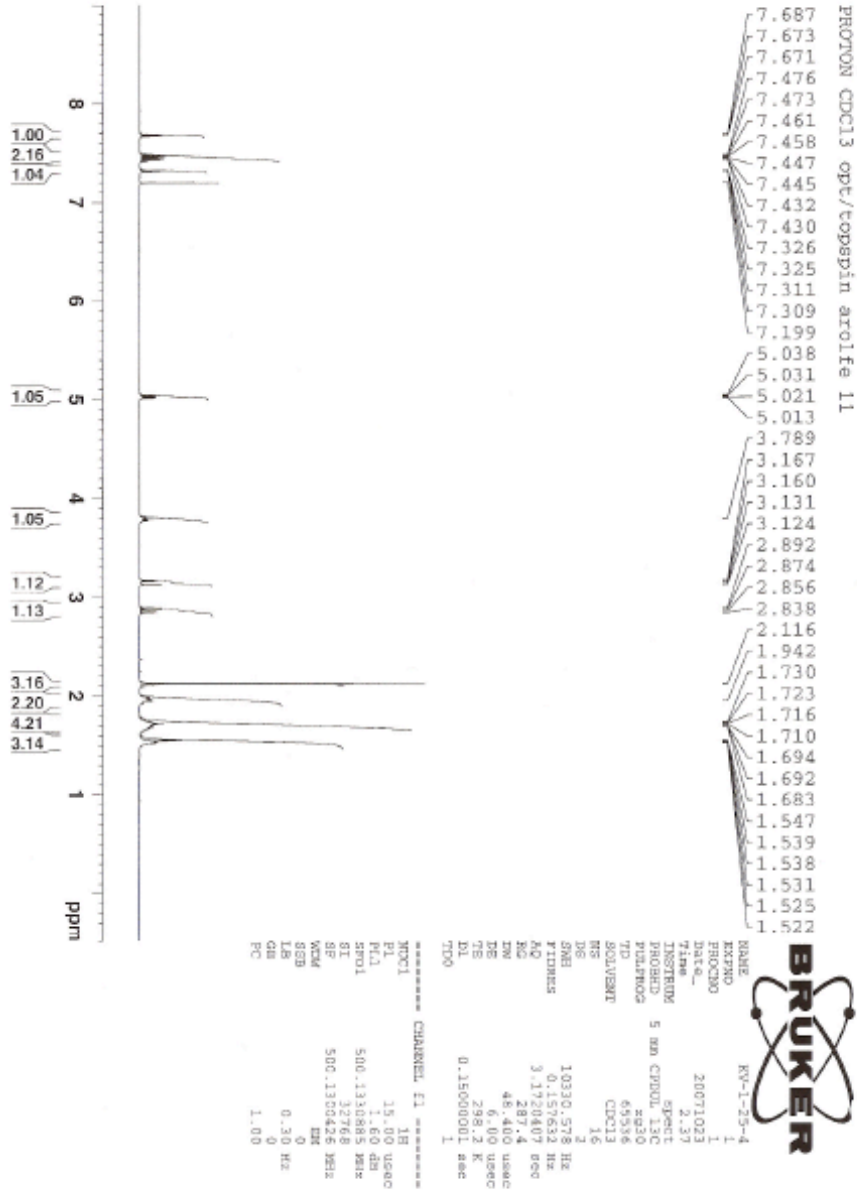


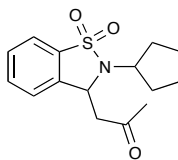
2-Benzyl-3-(2-propyl)isothiazoline-1-one 1,1' dioxide (6)



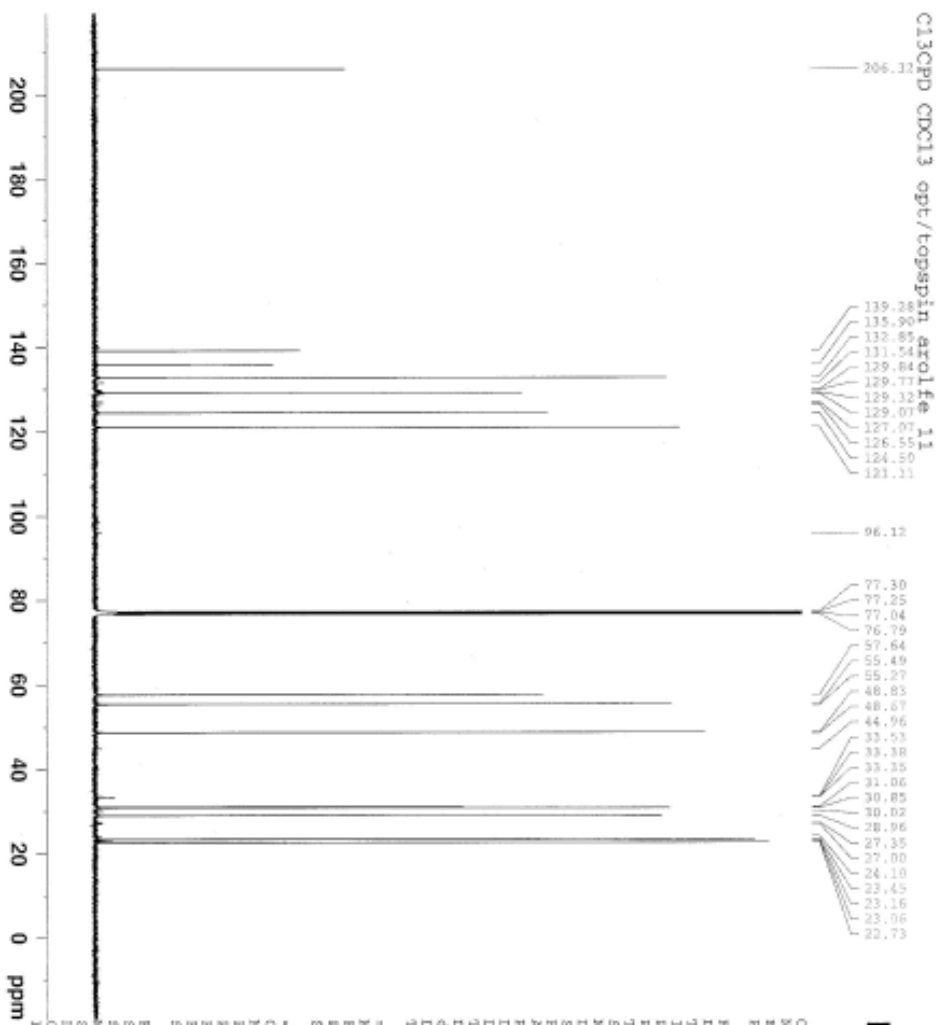


2-Benzyl-3-(2-cyclopentyl)isothiazoline-1-one 1,1' dioxides (7)





2-Benzyl-3-(2-cyclopentyl)isothiazoline-1-one 1,1' dioxide (7)



Current Data Parameters
 NAME RV-1-25-4
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20071023
 Time 2.55
 INSTRUM spect
 PROBRD 5 mm CPDML 13C
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 800
 DS 4
 SFO 36030.029 Hz
 FIDRES 0.45822 Hz
 AQ 1.0912410 sec
 RG 14596.5
 TM 16.650 urec
 DE 6.00 urec
 TE 298.2 K
 D1 0.1500001 sec
 d11 0.0300000 sec
 DELTA 0.0500000 sec
 TD0 1

***** CHANNEL F1 *****

NUC1 13C
 P1 11.00 urec
 PL 1.20 dB
 SFO1 125.7703643 MHz

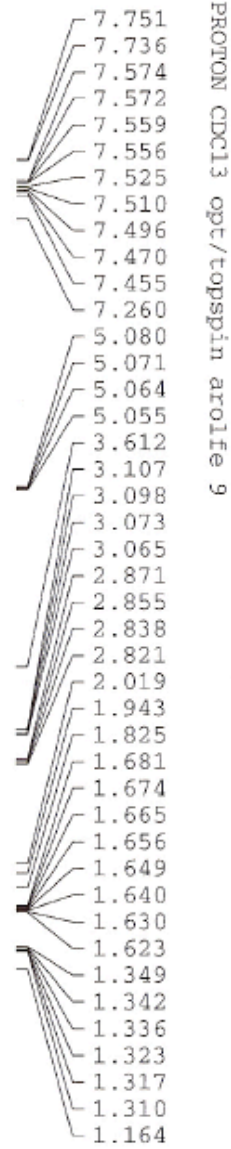
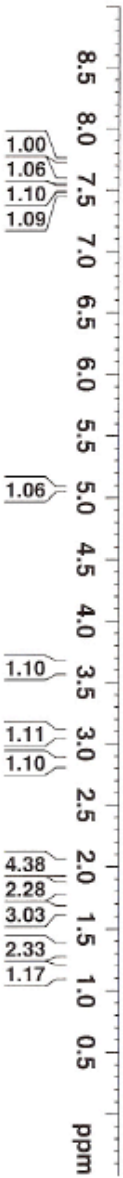
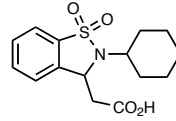
***** CHANNEL F2 *****

CPDPRG2 waltz16
 NUC2 1H
 P2 80.00 urec
 PL2 1.60 dB
 PL12 1.10 dB
 PL13 10.00 dB
 SFO2 500.1320005 MHz

F2 - Processing Parameters

SI 32768
 SF 125.7577890 MHz
 NSB 0
 GB 0
 PC 1.40

2-(2-Cyclohexyl-3-isothiazoline-1-yl)ethanoic acid 1,1' dioxide (**8**)



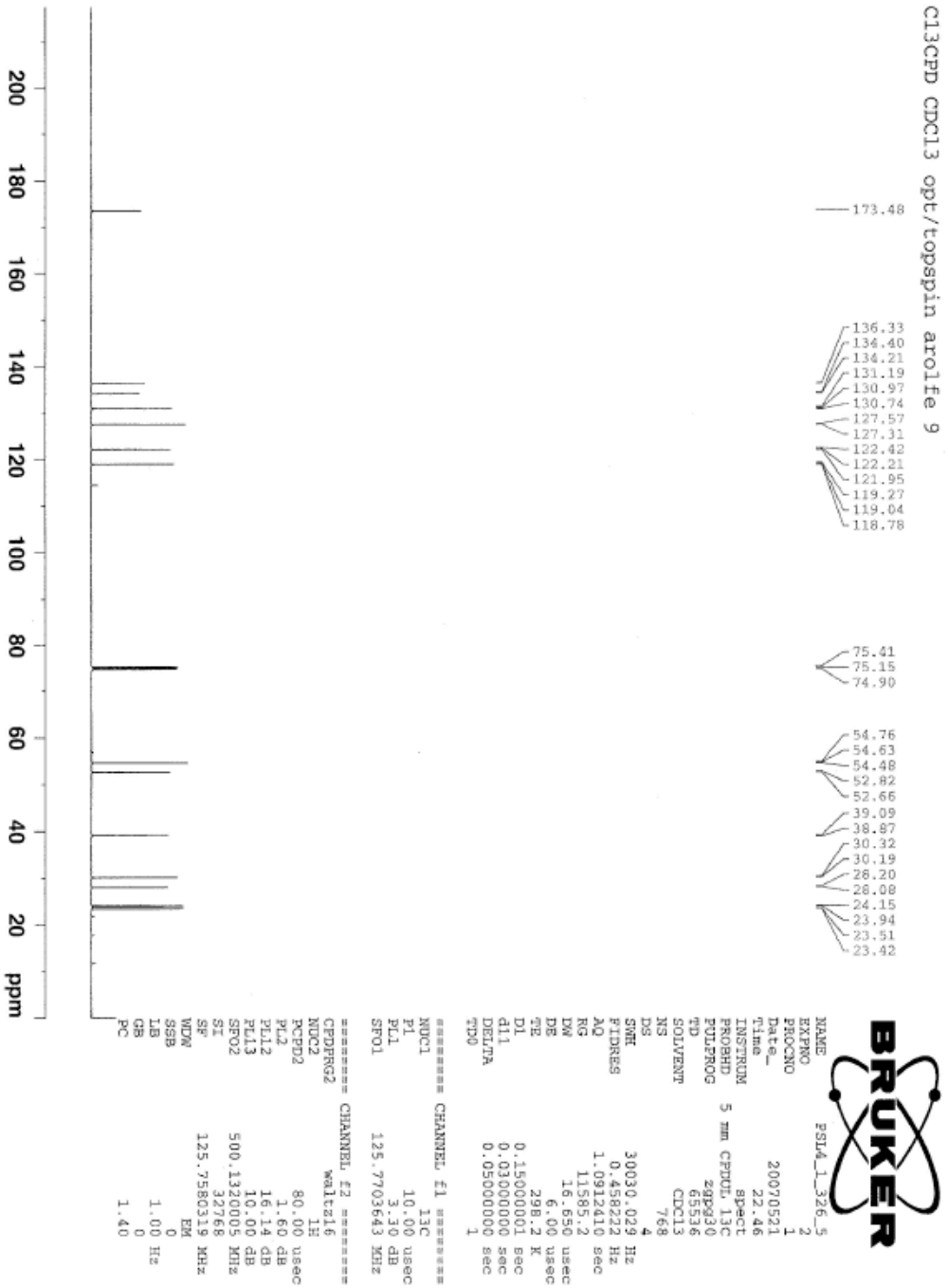
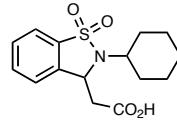
Current Data Parameters
 NAME PSL4_1_326_5
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20070521
 Time 22.28
 INSTRUM spect
 PROBHD 5 mm CPDUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SFO 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1720407 sec
 RG 45.3
 DW 48.400 usec
 DE 6.00 usec
 TE 298.2 K
 D1 0.15000001 sec
 TDO 1

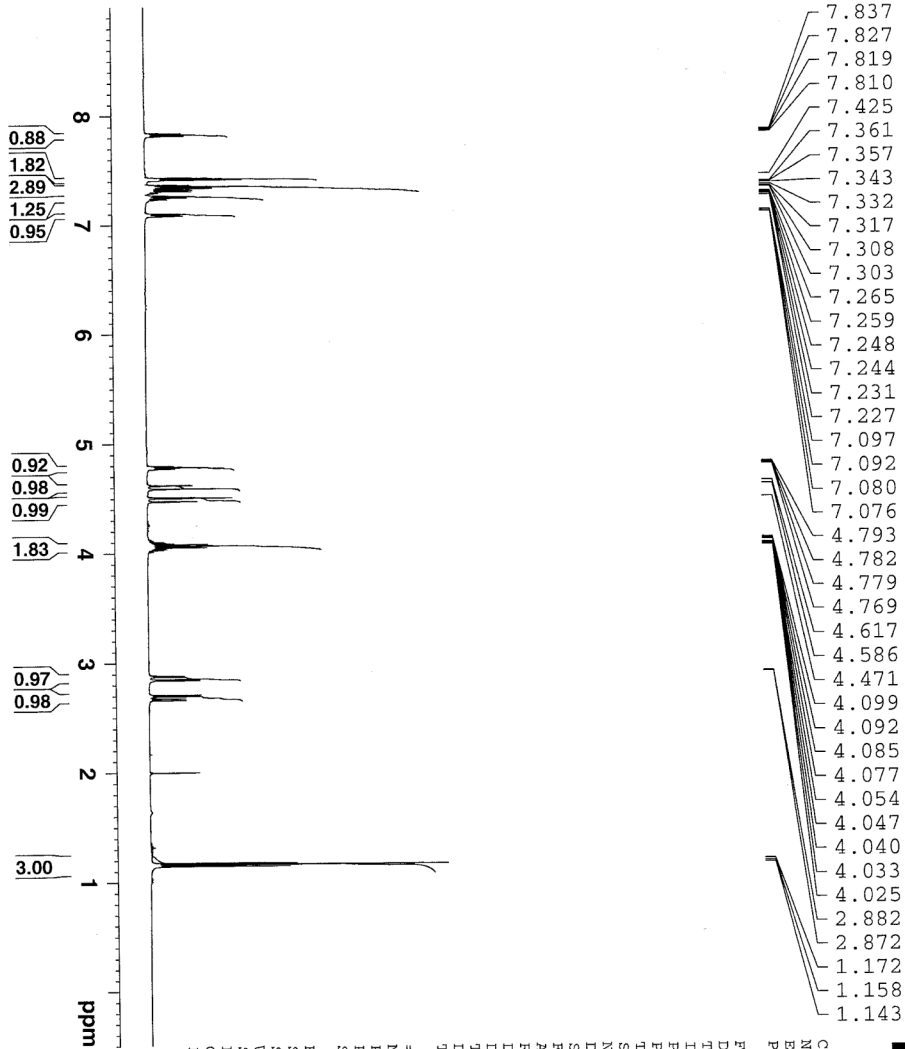
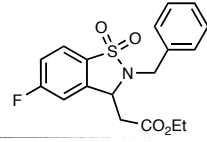
===== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL1 1.60 dB
 SFO1 500.1330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300134 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

2-(2-Cyclohexyl-3-isothiazoline-1-yl)ethanoic acid 1,1' dioxide (8)



Ethyl 2-(2-benzyl-6-fluoro-3-isothiazoline-1-yl)ethanoate 1,1' dioxide (9)



PROTON CDCl3 opt/topspin aro11fe 7



Current Data Parameters
 NAME KX_1_149_3
 EXPNO 1
 PROCNO 1

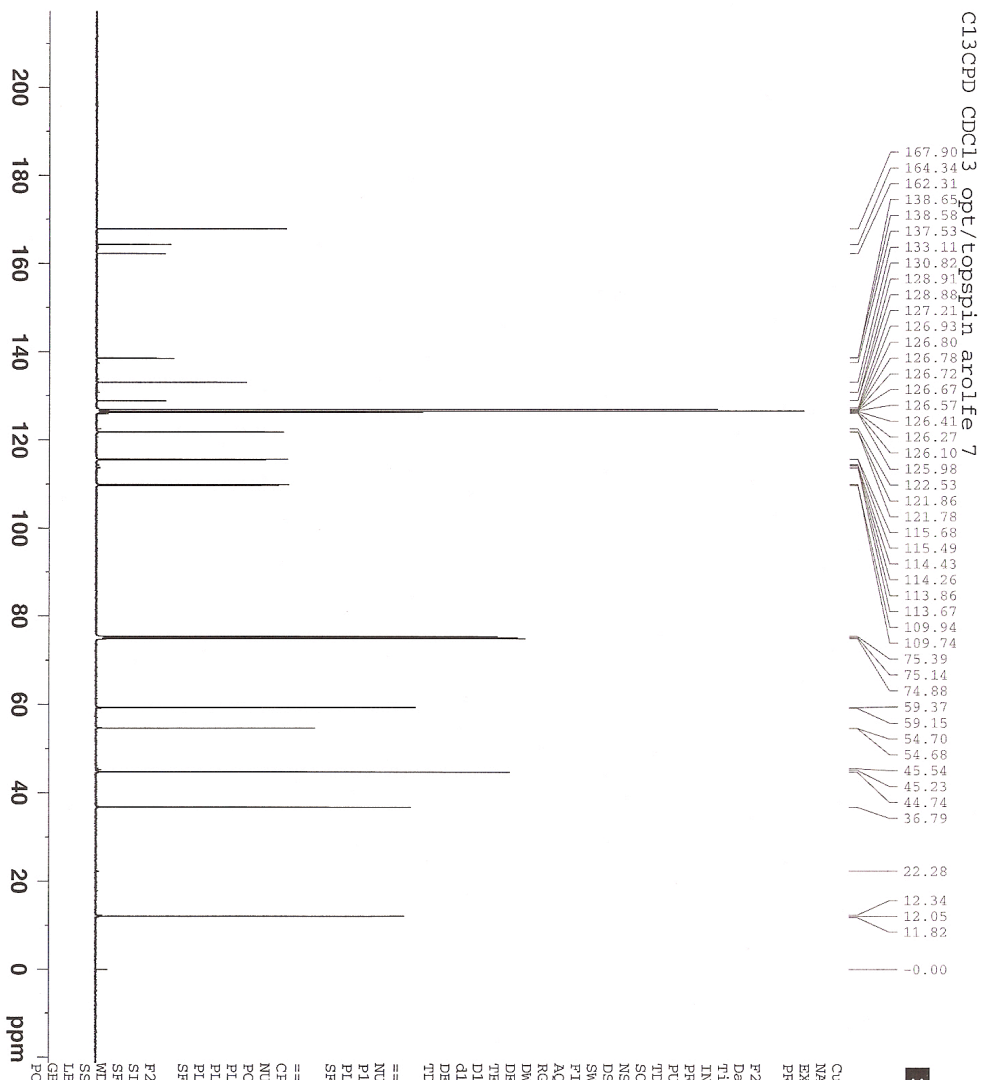
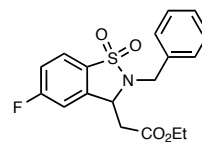
F2 - Acquisition Parameters

Date_ 20070521
 Time 20.27
 INSTRUM spect
 PROBHD 5 mm CPDPL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1720407 sec
 RG 64
 DW 48.400 usec
 DE 6.00 usec
 TE 298.2 K
 D1 0.15000001 sec
 TDO 1

==== CHANNEL f1 =====

NUC1 1H
 P1 15.00 usec
 PL1 1.60 dB
 SF01 500.1330885 MHz
 F2 - Processing parameters
 SI 32768
 SF 500.1300143 MHz
 MDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

Ethyl 2-(2-benzyl-6-fluoro-3-isothiazoline-1-yl)ethanoate 1,1' dioxide (9)



Current Data Parameters
 NAME KI_1_149_2
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

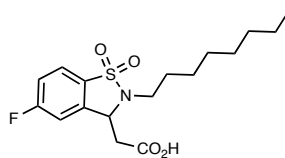
Date_ 20070521
 Time 20.39
 INSTRUM spect
 PROBHD 5 mm CPDUL 13C
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 4
 SWH 30030.029 Hz
 FIDRES 0.458222 Hz
 AQ 1.0912410 sec
 RG 11585.2
 DW 16.650 usec
 DE 6.00 usec
 TE 298.2 K
 D1 0.15000001 sec
 d11 0.03000000 sec
 DELTA 0.05000000 sec
 TDO 1

==== CHANNEL f1 =====
 NUC1 13C
 P1 10.00 usec
 PL 3.30 dB
 SFO1 125.7703643 MHz

==== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 ROPD2 80.00 usec
 PL2 1.50 dB
 PL12 16.14 dB
 PL13 10.00 dB
 SFO2 500.1320005 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7580317 MHz
 MDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

2-(6-Fluoro-2-octyl-3-isothiazoline-1-yl)ethanoic acid 1,1' dioxide (10)



PROTON CDCl3 opt/topspin aro1fe 8

- 7.808
- 7.798
- 7.790
- 7.781
- 7.269
- 7.265
- 7.259
- 7.252
- 7.248
- 7.235
- 7.231
- 7.179
- 7.175
- 7.163
- 7.159
- 4.878
- 4.868
- 4.864
- 4.854
- 3.334
- 3.304
- 3.087
- 3.077
- 3.043
- 2.859
- 2.845
- 2.826
- 2.811
- 1.742
- 1.734
- 1.721
- 1.712
- 1.705
- 1.386
- 1.372
- 1.356
- 1.326
- 1.319
- 1.304
- 1.300
- 1.231
- 0.879
- 0.852



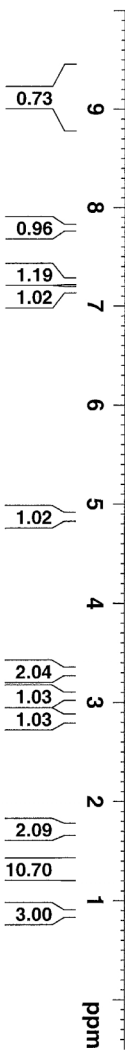
Current Data Parameters
 NAME PSL4_1_339_10
 EXPNO 1
 PROCNO 1

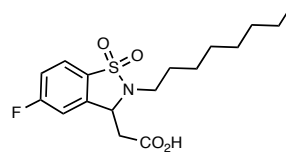
F2 - Acquisition Parameters

Date_ 20070521
 Time 21.25
 INSTRUM spect
 PROBHD 5 mm CPDUL 13C
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157633 Hz
 AQ 3.1720407 sec
 RG 50.8
 DW 48.400 usec
 DE 6.00 usec
 TE 298.2 K
 D1 0.15000001 sec
 TD0 1

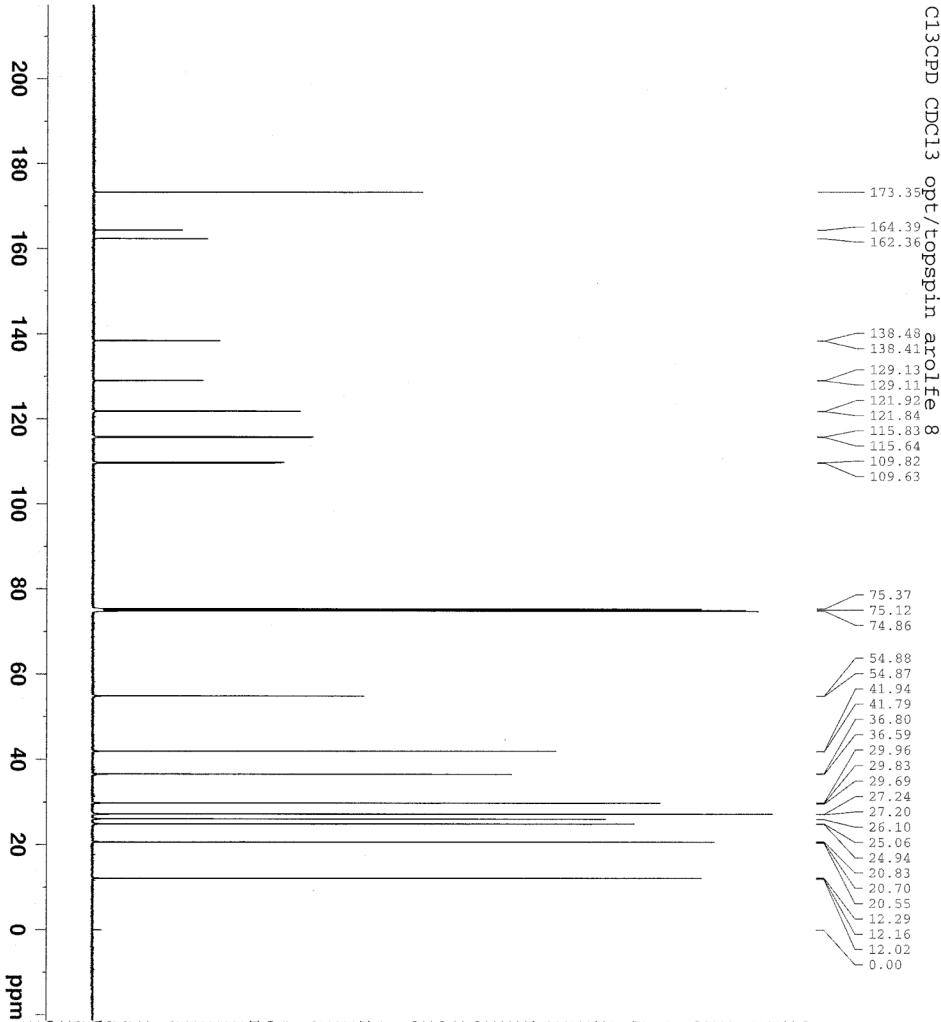
==== CHANNEL f1 =====
 NUC1 1H
 P1 15.00 usec
 PL 1.60 dB
 SF01 500.1330885 MHz

F2 - Processing parameters
 SI 32768
 SF 500.1300140 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





2-(6-Fluoro-2-octyl-3-isothiazoline1-yl)ethanoic acid 1,1' dioxido (10)



Current Data Parameters
 NAME PS14_1_339_10
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20070521
 Time 21.42
 INSTRUM spect
 PROBHD 5 mm CPDUL 13C
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 768
 DS 4
 SWH 30030.029 Hz
 FIDRES 0.458222 Hz
 AQ 1.0912410 sec
 RG 11585.2
 DW 16.650 usec
 DE 6.00 usec
 TE 298.2 K
 D1 0.15000001 sec
 d11 0.03000000 sec
 DELTA 0.05000000 sec
 TDO 1

==== CHANNEL f1 =====

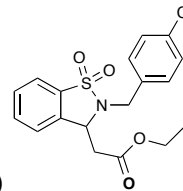
NUC1 13C
 P1 10.00 usec
 PL1 3.30 dB
 SFO1 125.7703643 MHz

==== CHANNEL f2 =====

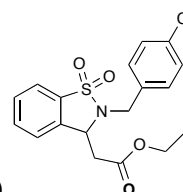
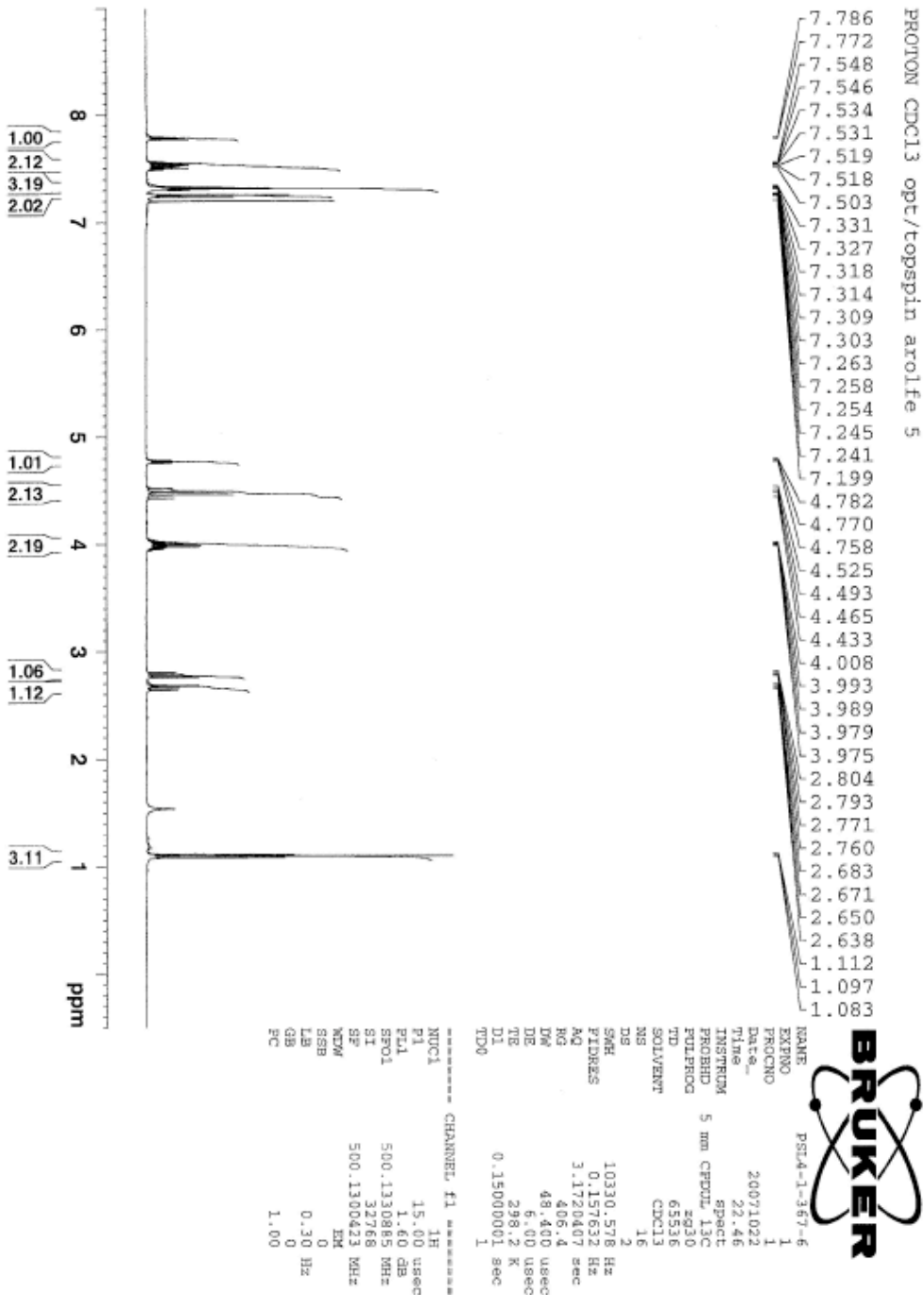
CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 1.60 dB
 PL12 16.14 dB
 PL13 10.00 dB
 SFO2 500.1320005 MHz

F2 - Processing parameters

SI 32768
 SF 125.7580326 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

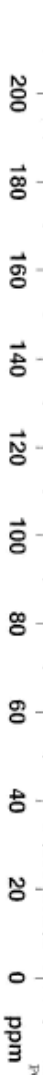


Ethyl 2-(2-(4-chlorobenzyl-3-isothiazoline-1-yl)ethanoate 1,1' dioxide (**11**)



Ethyl 2-(2-(4-chlorobenzyl-3-isothiazoline-1-yl)ethanoate 1,1' dioxide (**11**)

C13CPD CDC13 opt/topspin aro1fe 5



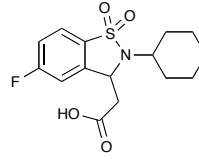
Current Data Parameters
 NAME FSI4-1-367-6
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071022
 Time 23.04
 INSTRUM spect
 PROBHD 5 mm CPDUI_13C
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 800
 DS 4
 SFR 30030.029 Hz
 FIDRES 0.448222 Hz
 AQ 1.0912410 sec
 RG 13004
 DM 16.630 usec
 DE 6.00 usec
 TE 298.2 K
 D1 0.15000001 sec
 d11 0.03000000 sec
 DELTA 0.05000000 sec
 TD0 1

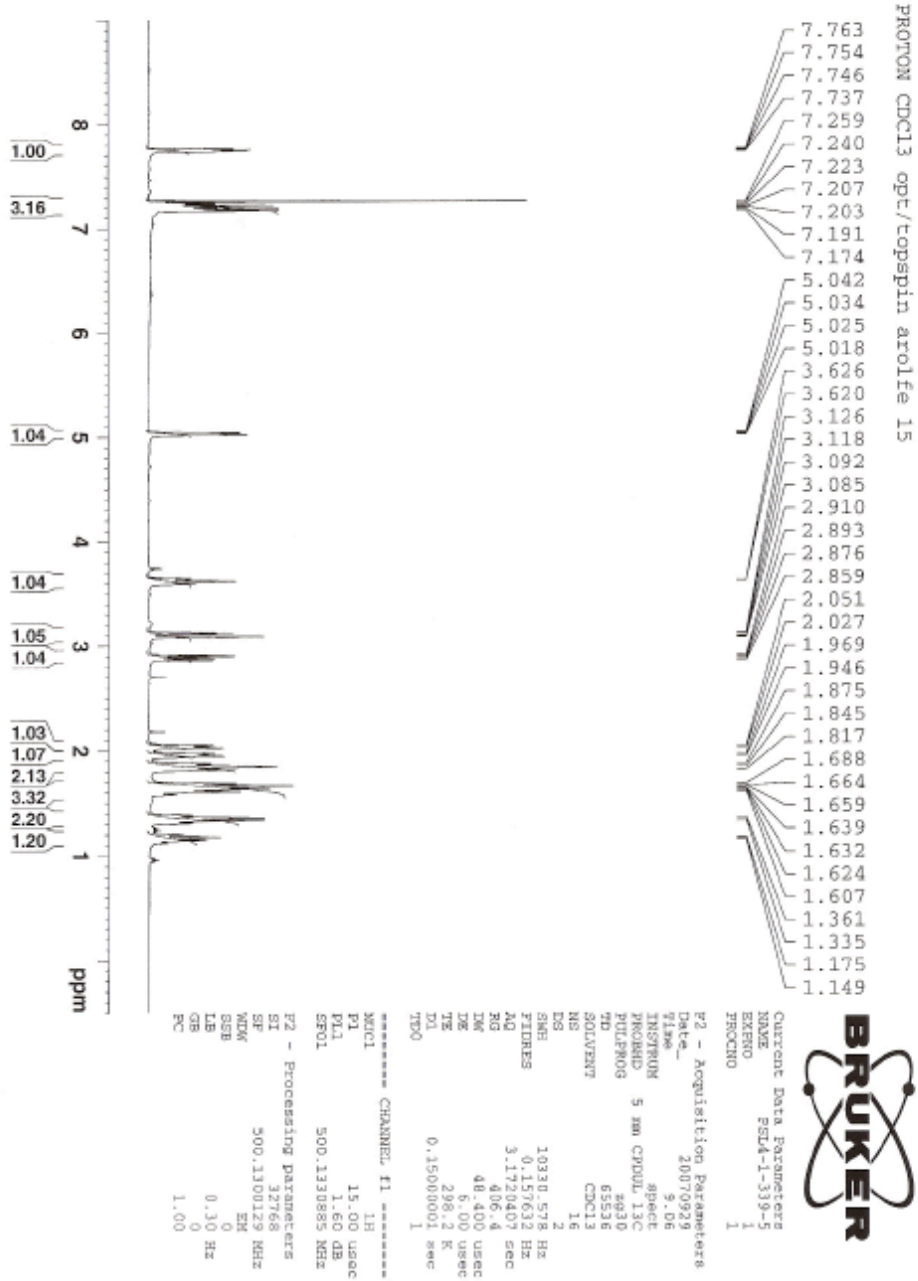
CHANNEL F1
 NUCL1 13C
 P1 11.00 usec
 PL1 2.20 dB
 SFO1 125.7703643 MHz

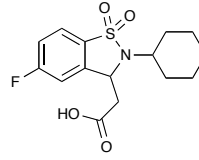
CHANNEL F2
 CPDPRG2 waite16
 NUCL2 1H
 PCPD2 80.00 usec
 PL2 1.60 dB
 PL12 16.14 dB
 PL13 10.00 dB
 SFO2 500.1320005 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7577890 MHz
 EN 64
 SR 1.00 Hz
 DB 0
 PC 1.40

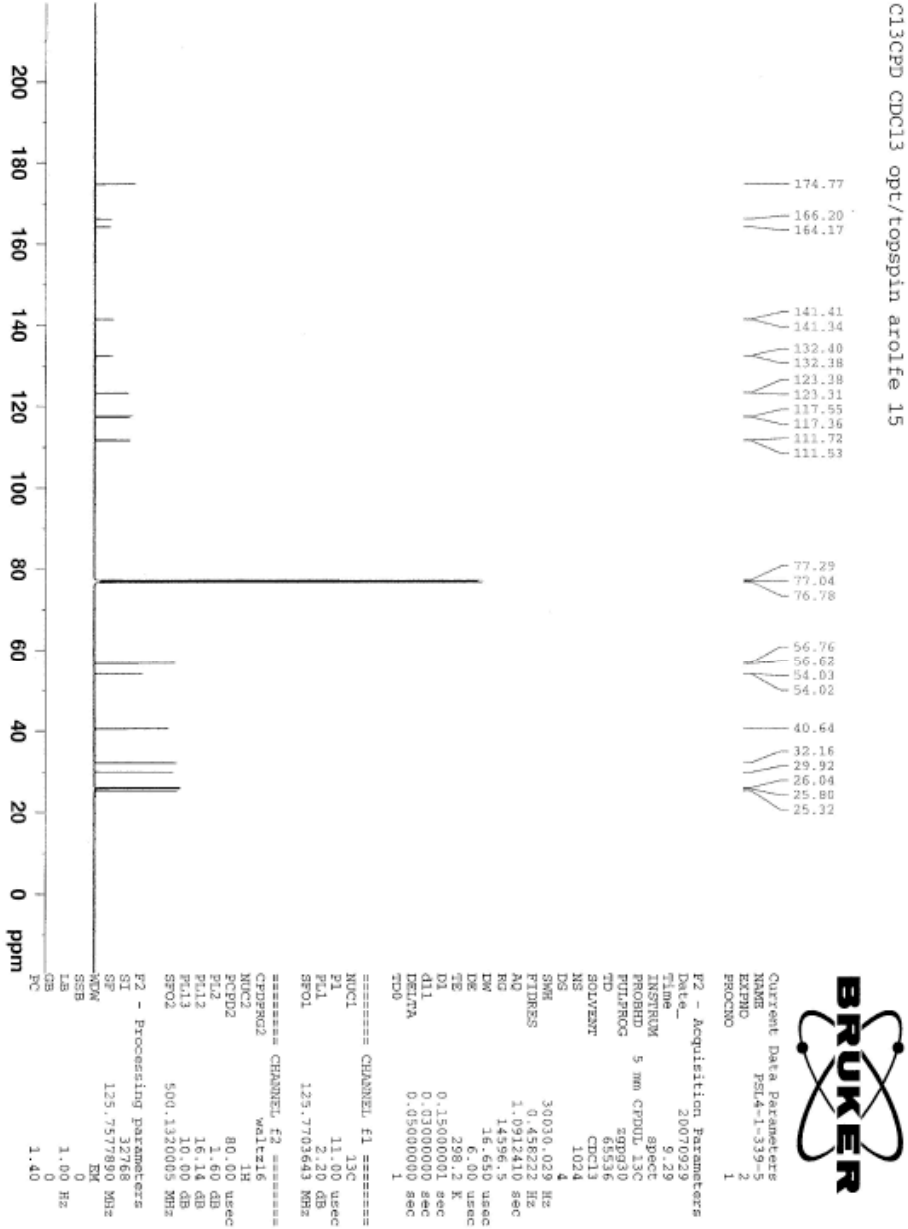


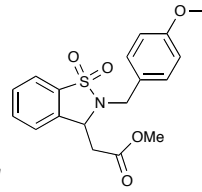
2-(2-Cyclohexyl-6-fluoro-3-isothiazoline-1-yl)ethanoic acid 1,1' dioxide (**12**)



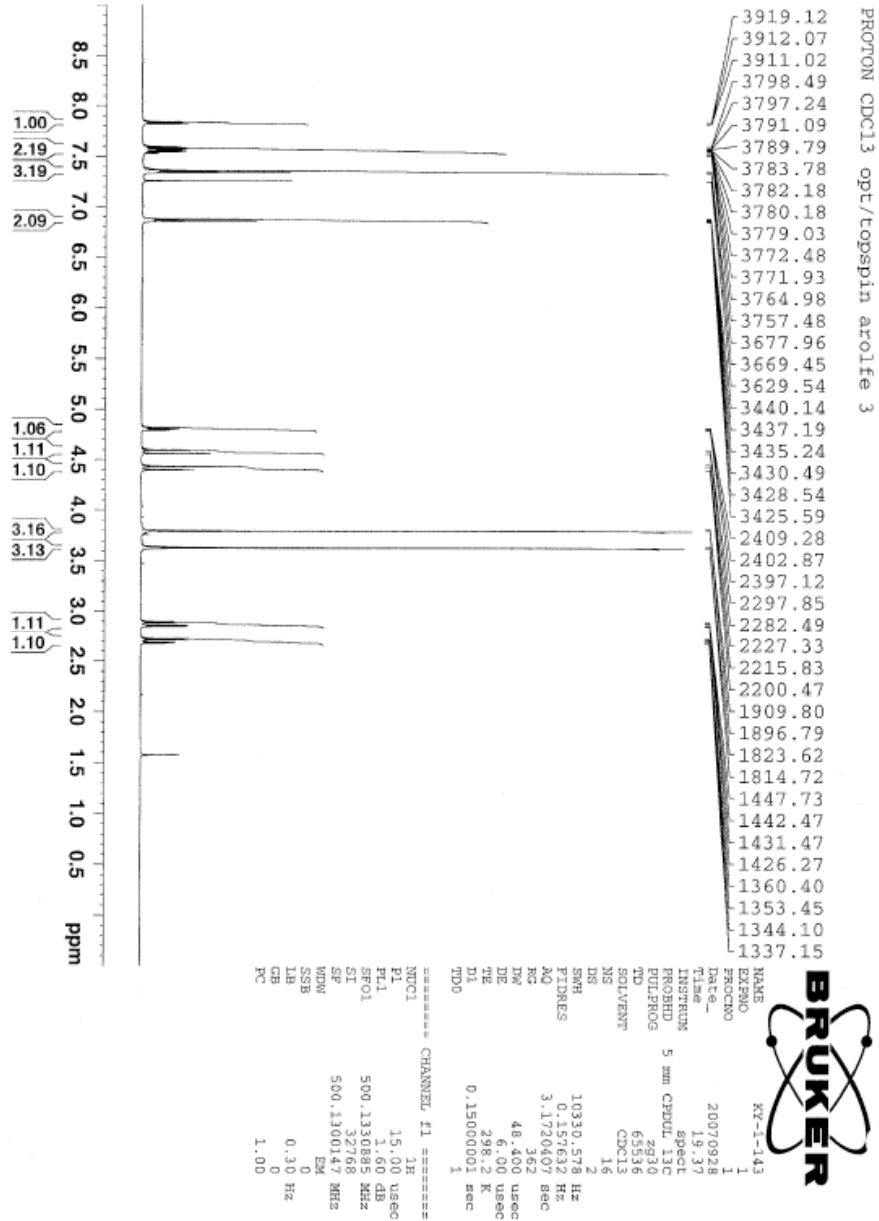


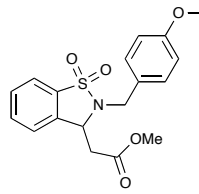
2-(2-Cyclohexyl-6-fluoro-3-isothiazoline-1-yl)ethanoic acid 1,1' dioxide (**12**)



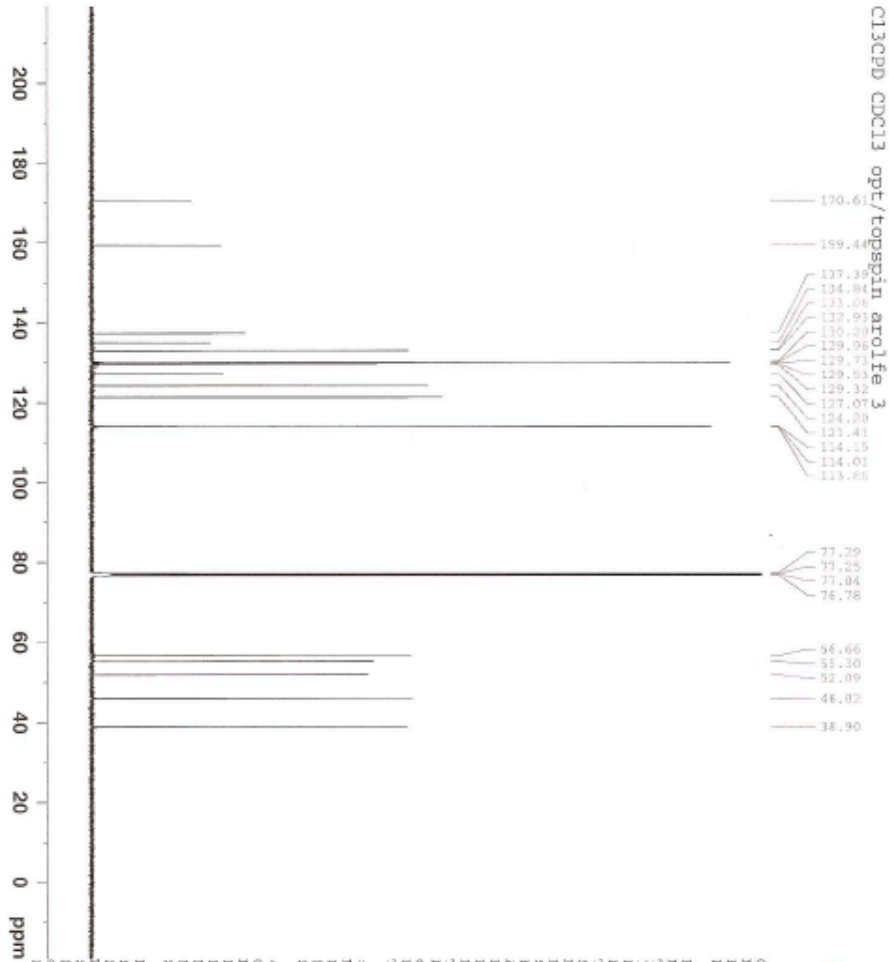


Methyl 2-(4-methoxybenzyl-3-isothiazoline-1-yl)methanoate 1,1'-dioxide (13)





Ethyl 2-(2-benzyl-5-(trifluoromethyl)isothiazoline-1-yl)ethanoate 1,1'-dioxide (13)



Current Data Parameters
 XANG XY-1-143
 EXTNO 2
 PROCNO 1

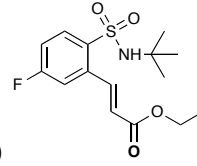
P2 - Acquisition Parameters

Date_ 20070220
 Time_ 20.00
 INSTRUM spect
 PROCNO 5
 PULPROG zgpg30
 TD 65536
 SFO1 125.7703643 MHz
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 30030.029 Hz
 FIDRES 0.458222 Hz
 AQ 1.0912410 sec
 RG 14596.5
 WC 18.650 usec
 DE 298.2
 TE 300.2 K
 D1 0.15000000 sec
 D11 0.03000000 sec
 DELTA 0.05000000 sec
 TDR 1

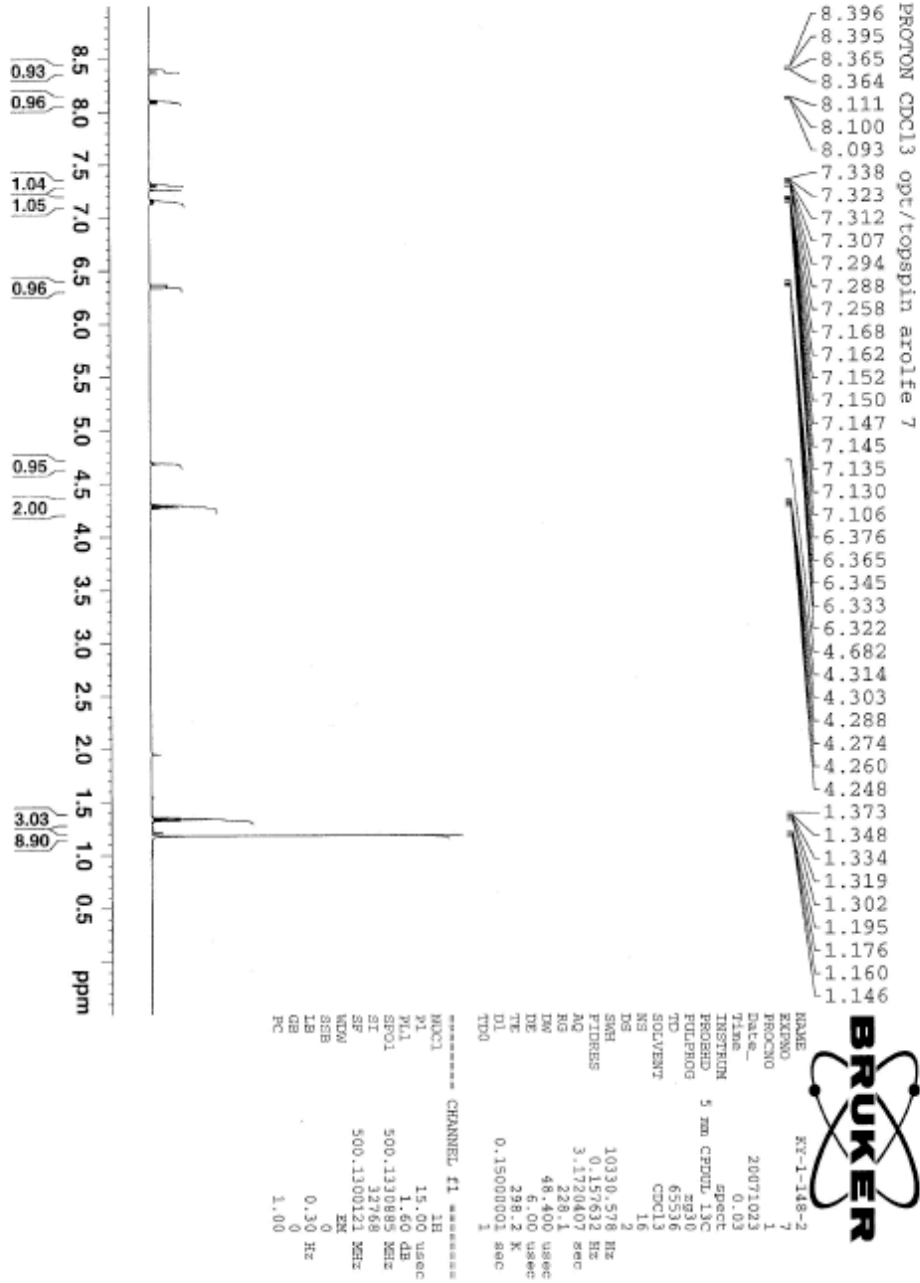
===== CHANNEL #1 =====
 NUCL1 13C
 P1 11.00 usec
 PL1 2.20 dB
 SFO1 125.7703643 MHz

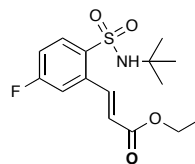
===== CHANNEL #2 =====
 CHPROG2 waltz16
 NUCL2 1H
 PCPD2 80.00 usec
 PL2 1.60 dB
 PL12 16.14 dB
 PL13 10.00 dB
 SFO2 500.1320005 MHz

P2 - Processing parameters
 SI 32768
 SF 125.7703643 MHz
 EX
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

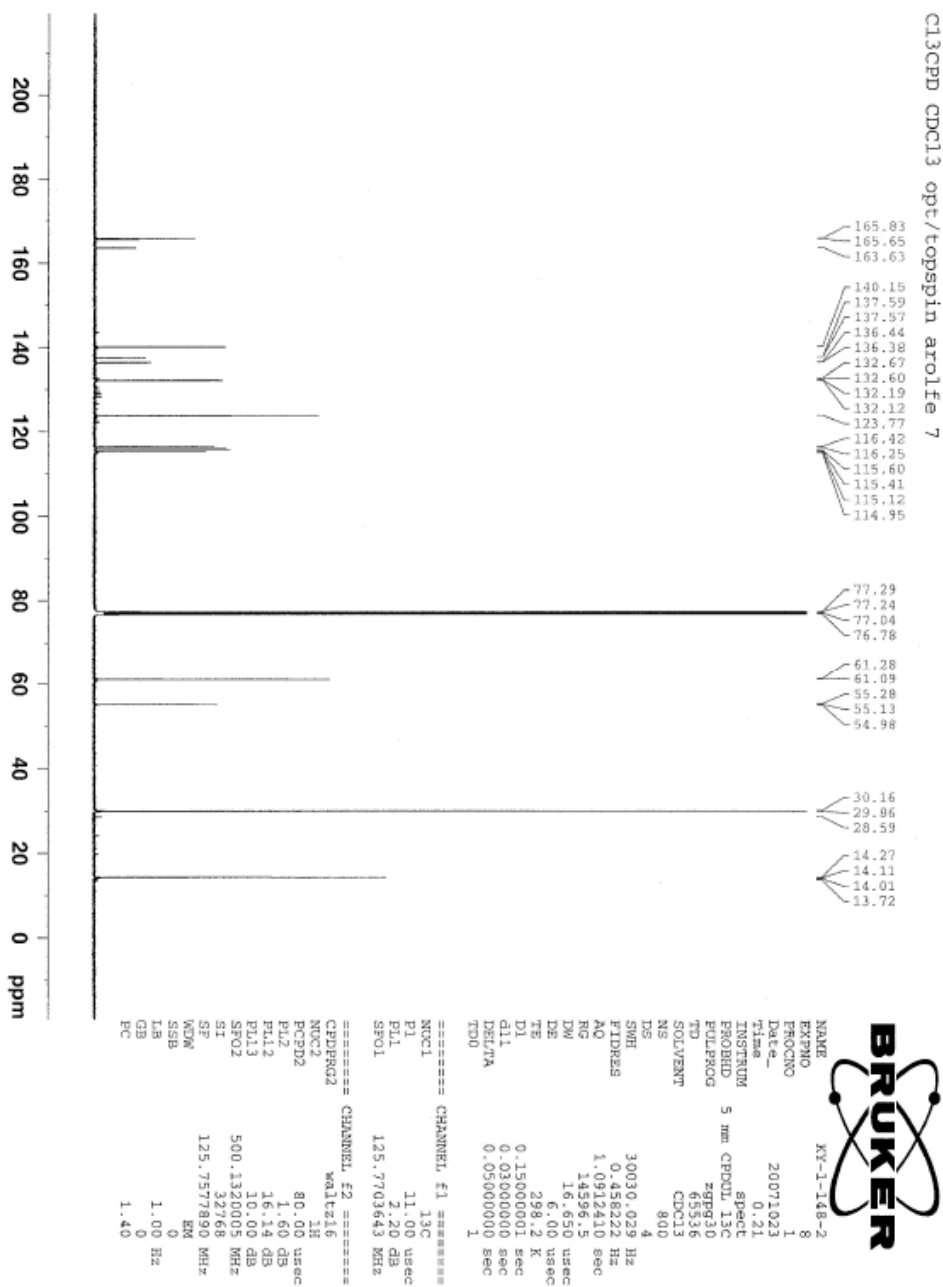


Ethyl 3-(2-(*N-tert*-butylsulfamoyl)-5-fluorophenyl)propanoate (**14**)

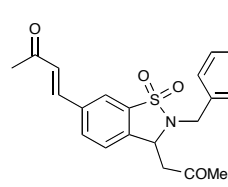




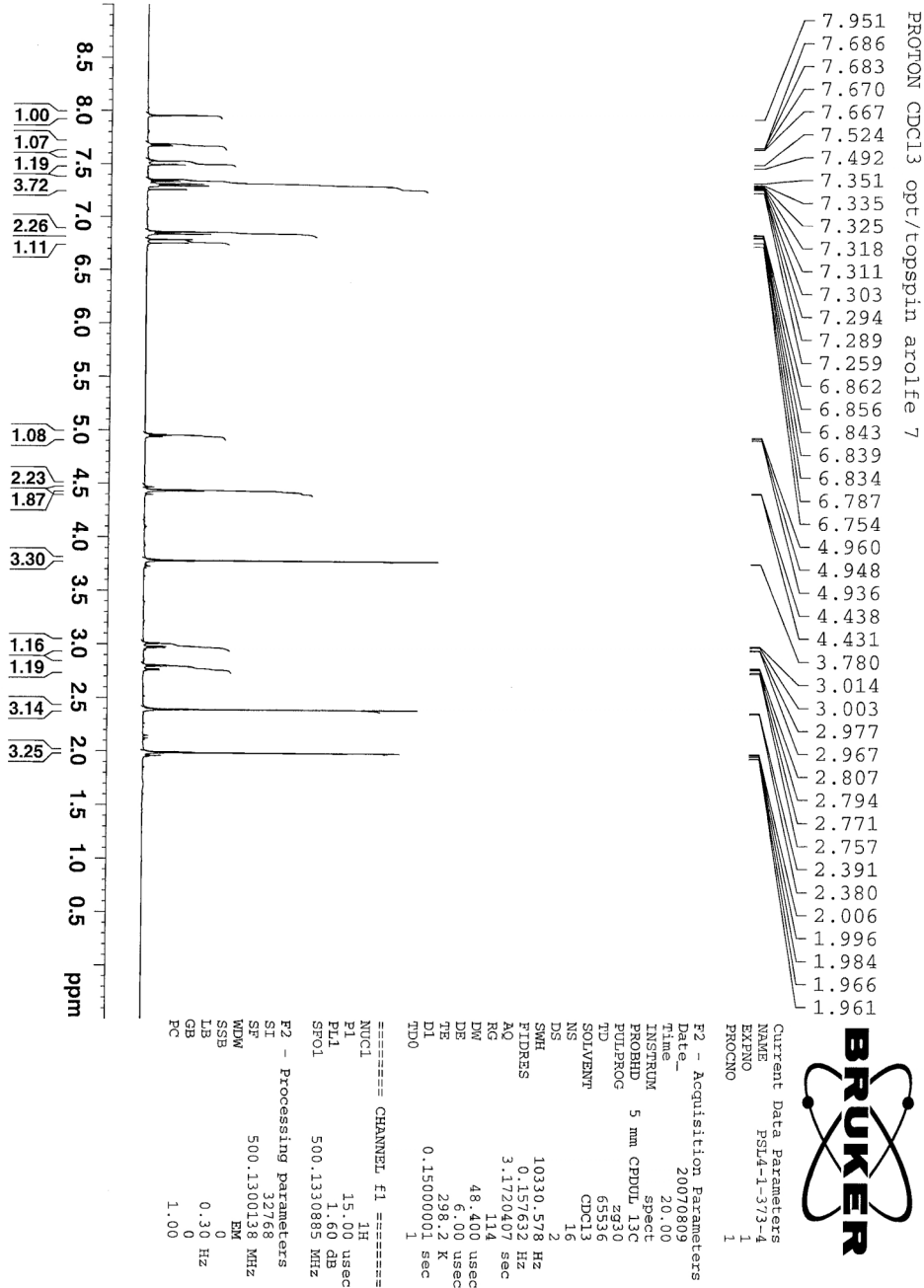
Ethyl 3-(2-(*N-tert*-butylsulfamoyl)-5-fluorophenyl)propanoate (**14**)



(E)-2-(4-methoxybenzyl)-6-(3-oxobut-1-enyl)-3-(2-oxopropyl)
one (16)

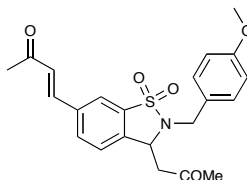


isothiazoline-1-

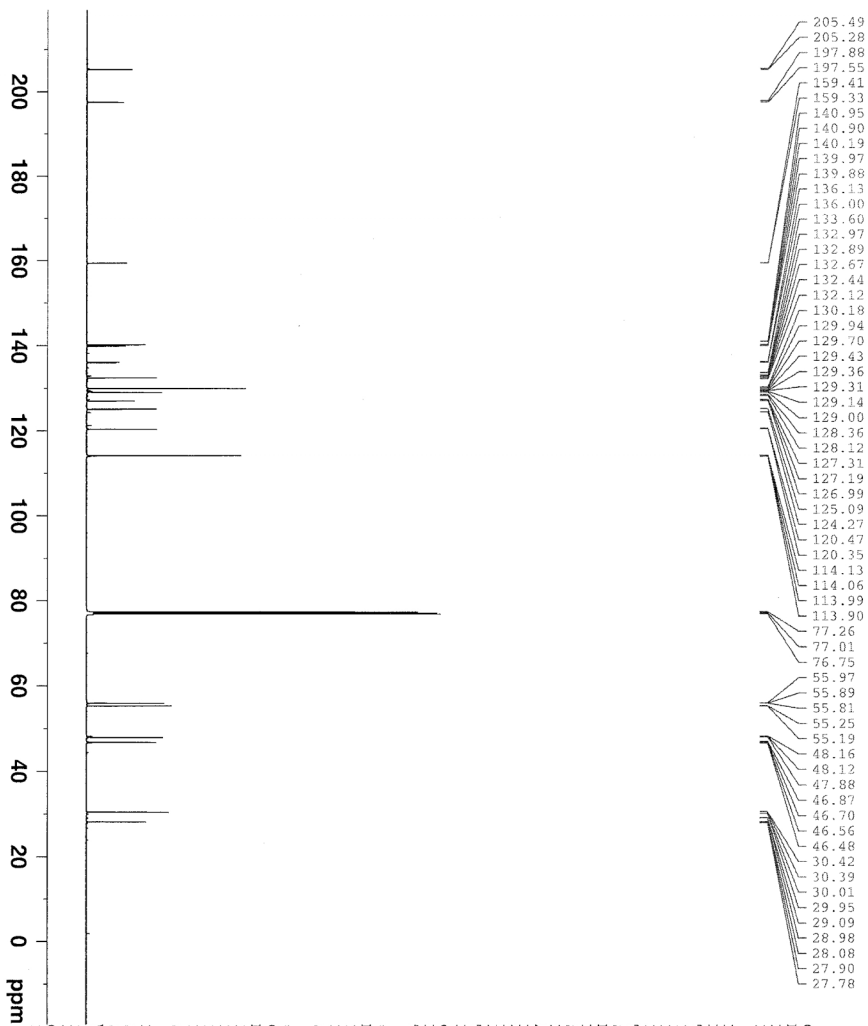


(E)-2-(4-methoxybenzyl)-6-(3-oxobut-1-enyl)-3-(2-oxopropyl)

(16)



isothiazoline-1-one



C13CPD CDCl3 opt/topspin arolfe 7



Current Data Parameters
 NAME ESI4-1-373-4
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20070809
 Time 20.18
 INSPRIM spect
 PROBD 5 mm CPDUT-13C
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 768
 DS 4
 SWH 30030.029 Hz
 FIDRES 0.456222 Hz
 AQ 1.0912410 sec
 RG 1625.5
 DW 16.650 usec
 DE 6.00 usec
 TE 298.2 K
 DL 0.15000001 sec
 dL1 0.03000000 sec
 DELTA 0.05000000 sec
 TDO 1

==== CHANNEL f1 =====

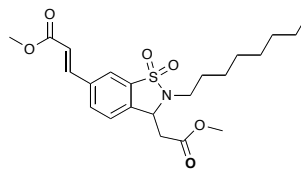
NUC1 13C
 P1 11.00 usec
 PL1 2.20 dB
 SFO1 125.7703643 MHz

==== CHANNEL f2 =====

CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 1.60 dB
 PL12 16.14 dB
 PL13 10.00 dB
 SFO2 500.1320005 MHz

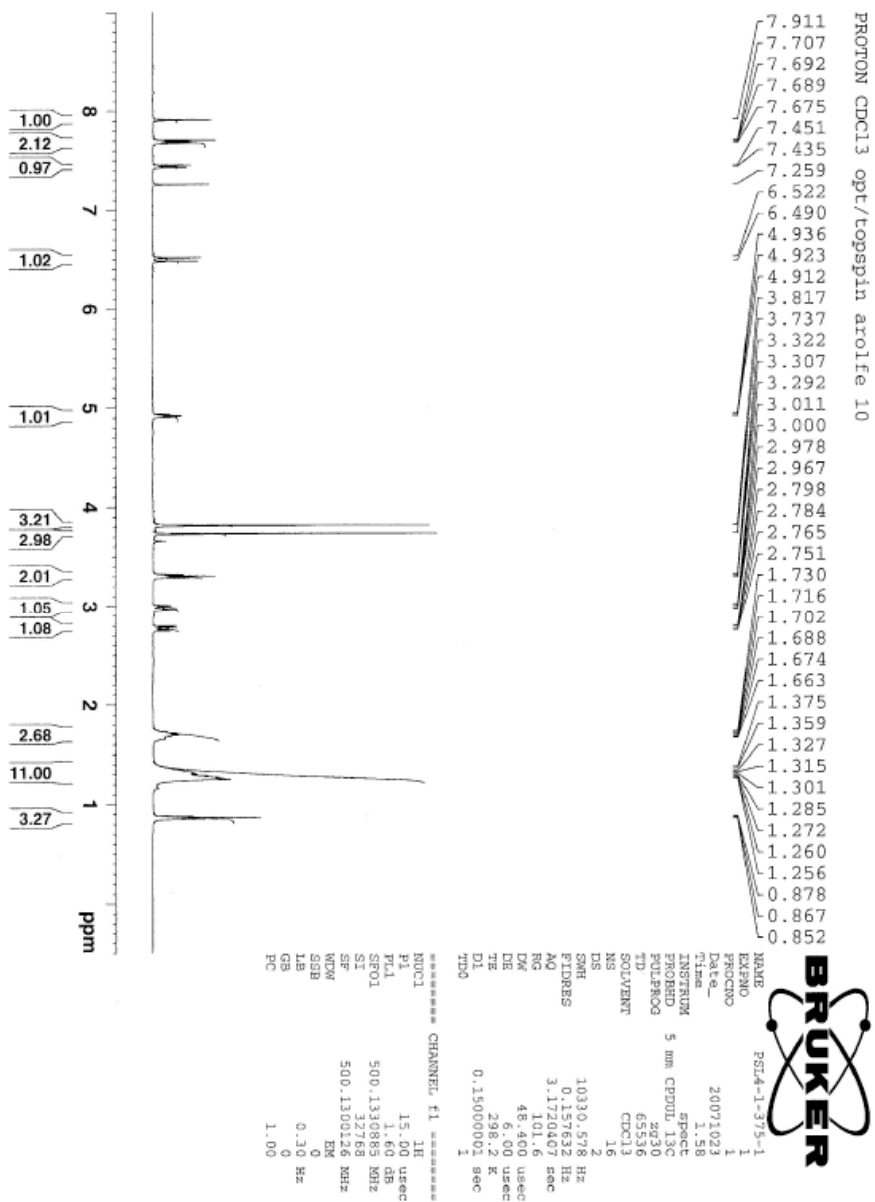
F2 - Processing parameters

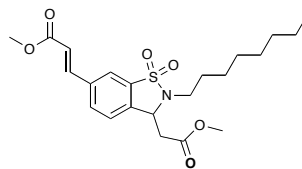
SI 32768
 SF 125.7577968 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



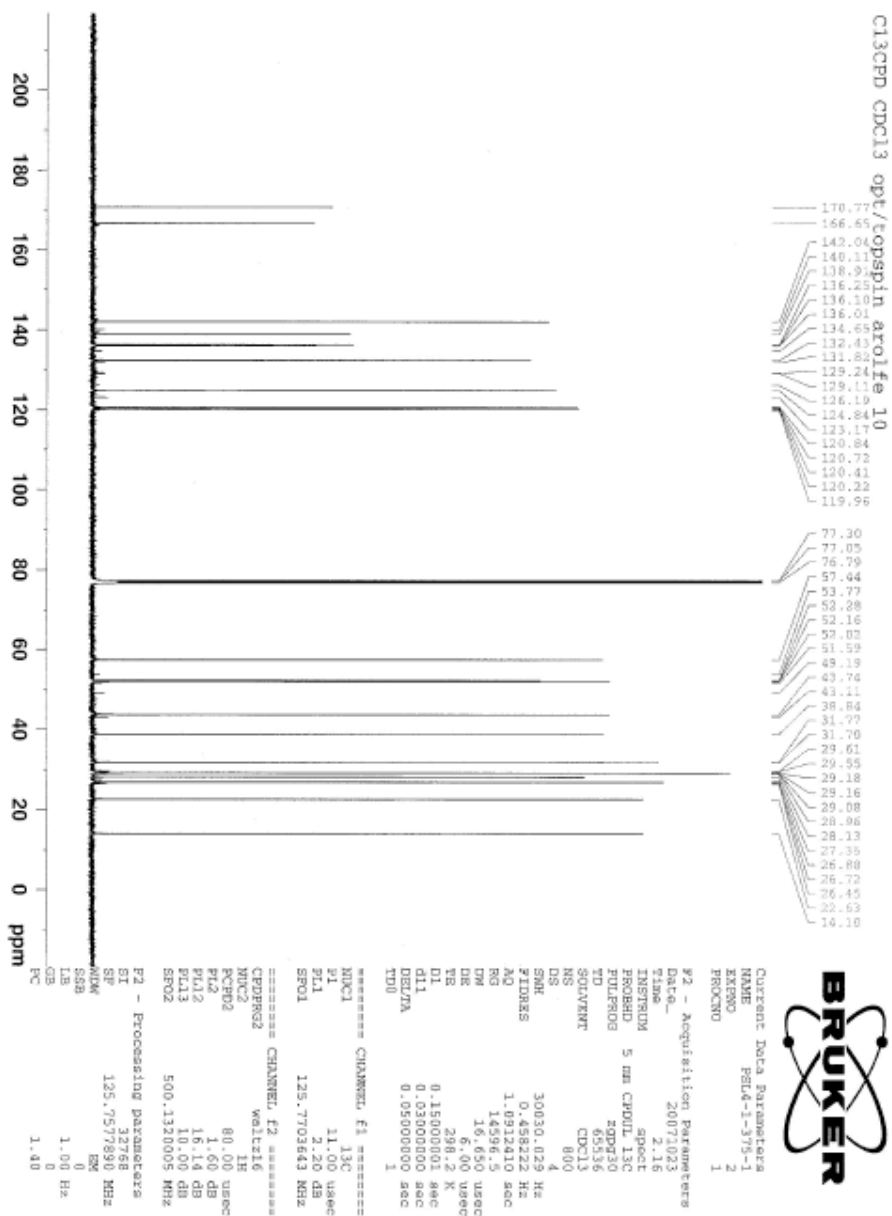
(*E*)-Methyl 3-(1-(2-methoxy-2-oxoethyl)-2-octyl-3-isothiazoline-5-yl)

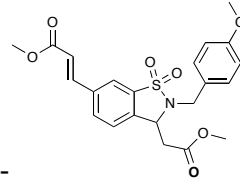
methanoate 1,1' dioxide (17)



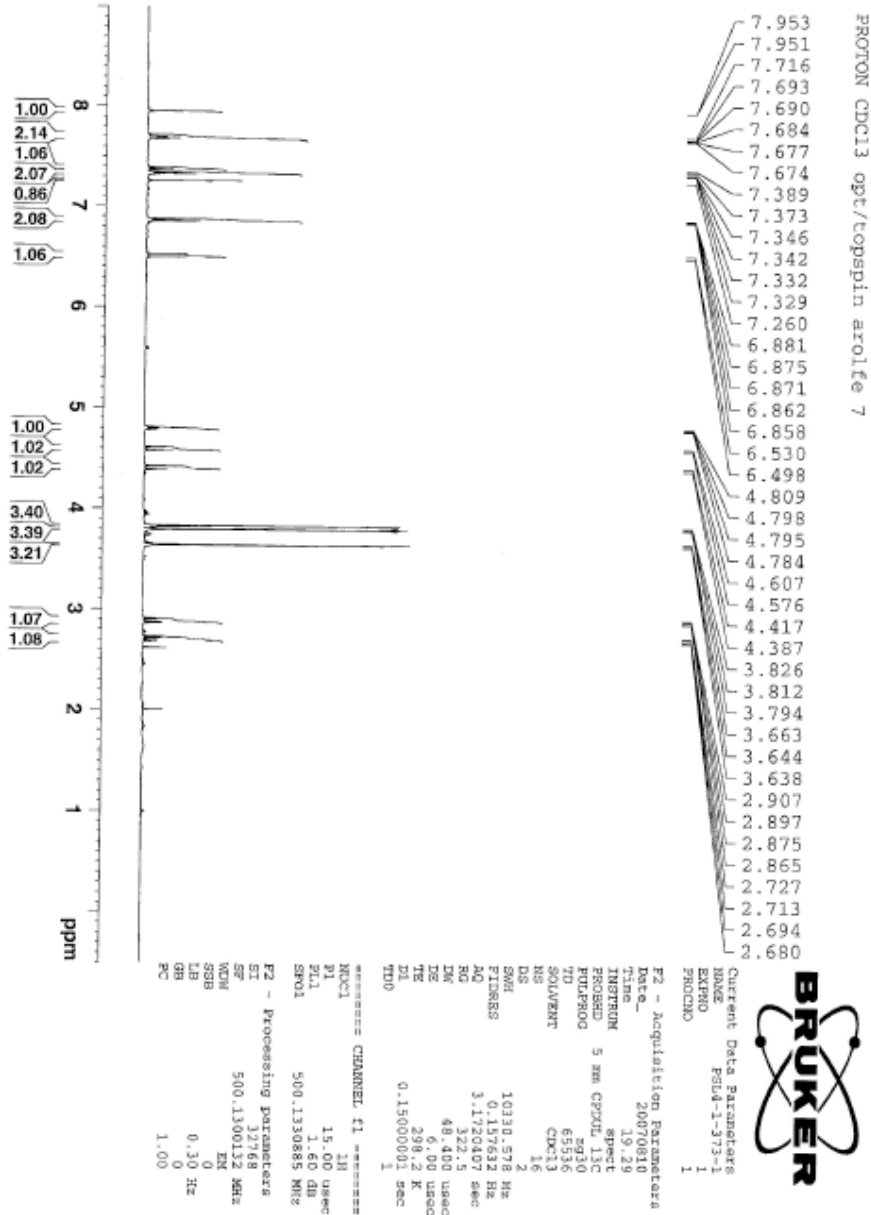


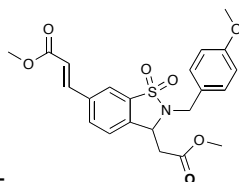
(E)-Methyl 3-(1-(2-methoxy-2-oxoethyl)-2-octyl-3-isothiazoline-5-yl) methanoate 1,1' dioxide (17)



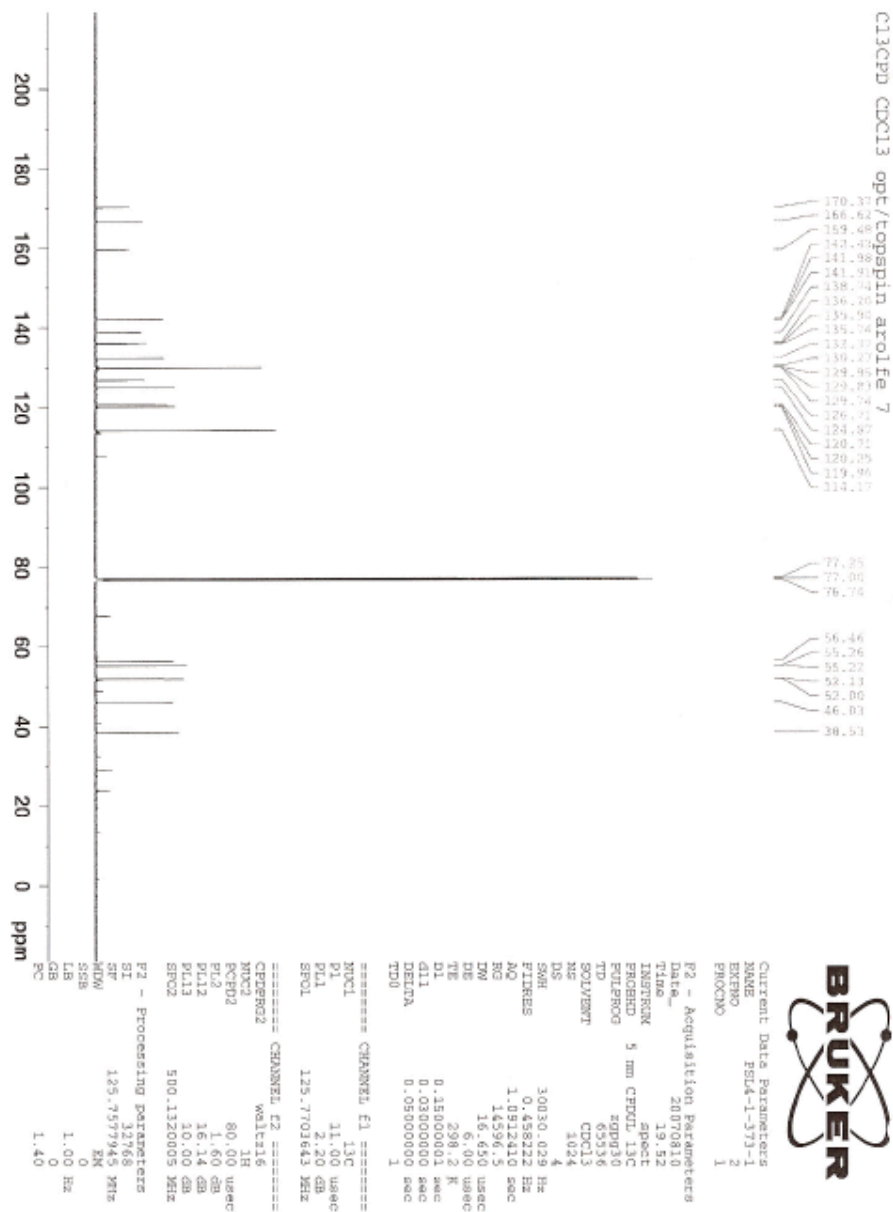


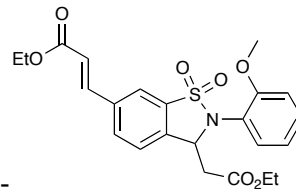
(*E*)-Methyl 3-(1-(2-methoxy-2-oxoethyl)-2-(4-methoxybenzyl)-3-isothiazoline-5-yl)prop-2-enoate (**18**)



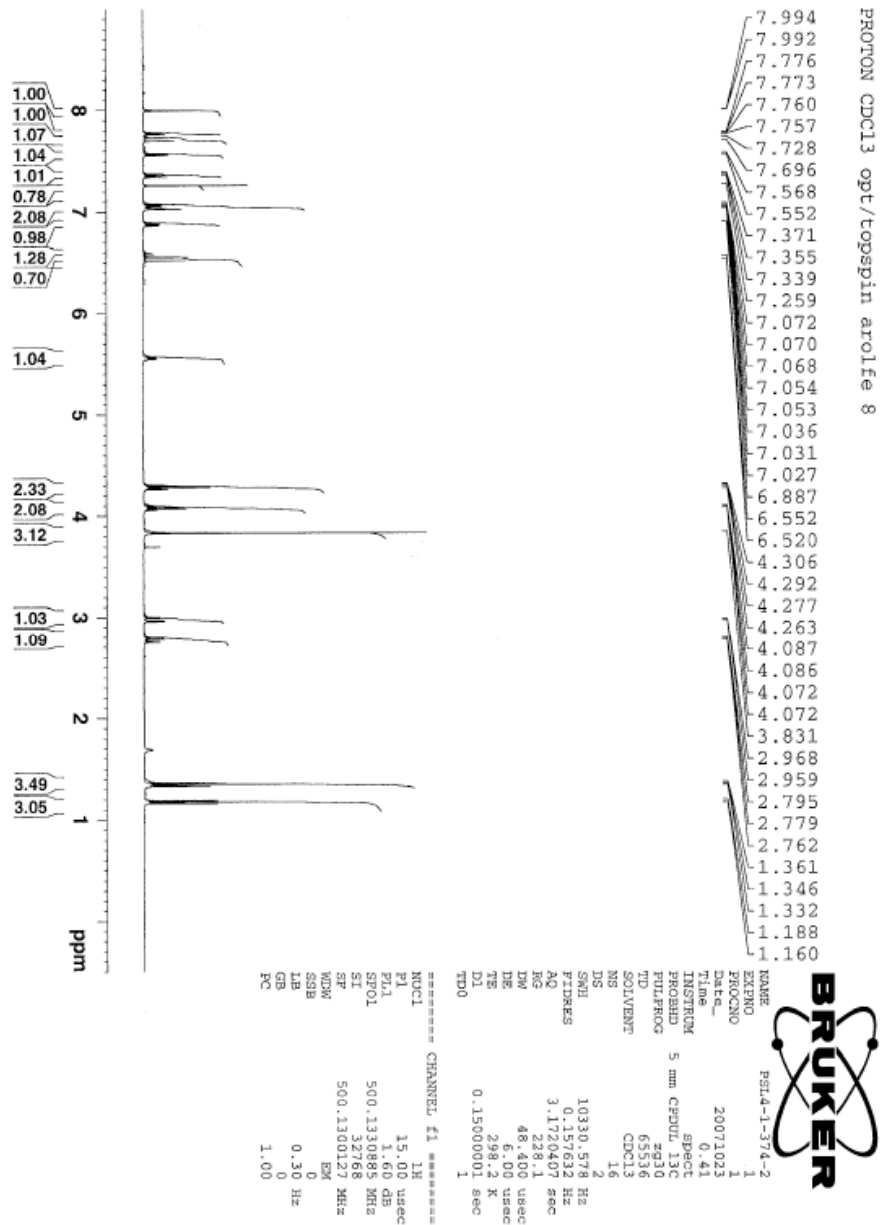


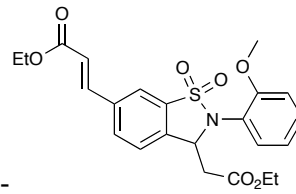
(*E*)-Methyl 3-(1-(2-methoxy-2-oxoethyl)-2-(4-methoxybenzyl)-3-isothiazoline-5-yl)prop-2-enoate (**18**)



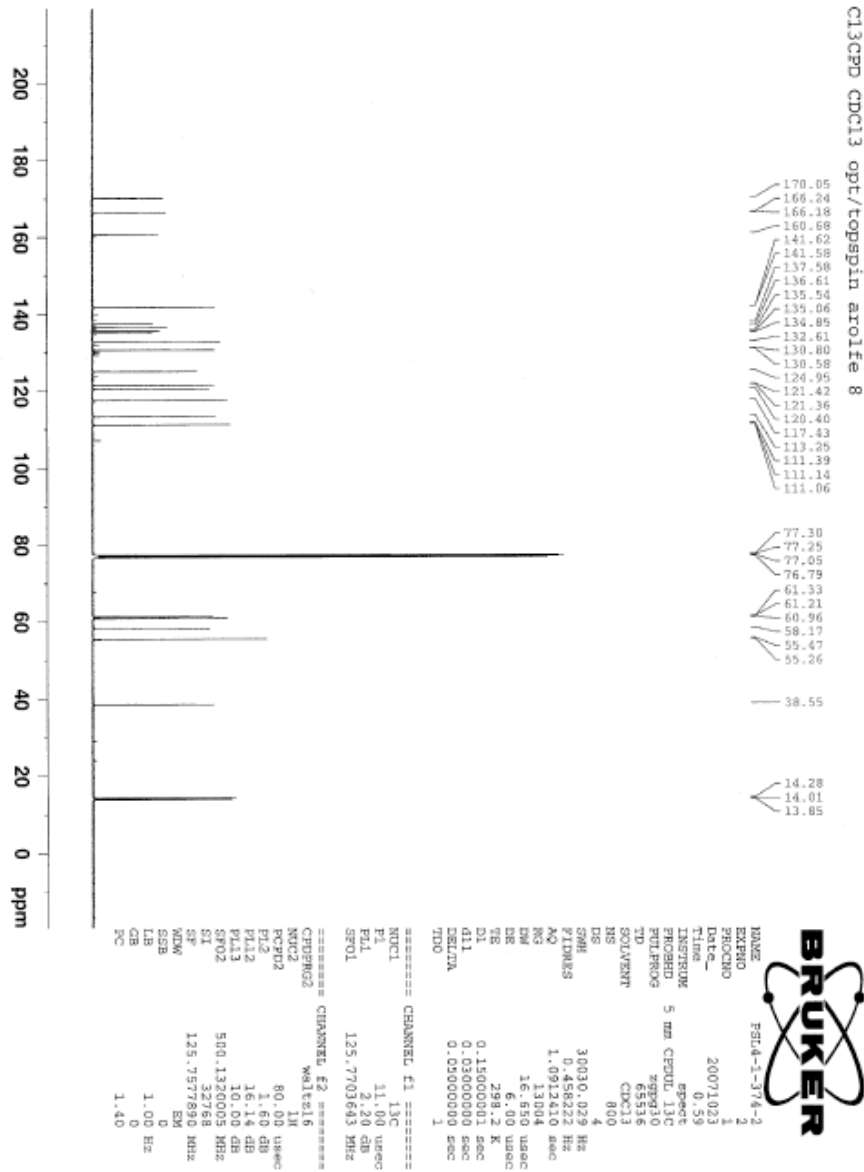


(*E*)-Ethyl 3-(1-(2-ethoxy-2-oxoethyl)-2-(2-methoxyphenyl)-3-isothiazoline-5-yl) prop-2-enoate (**19**)

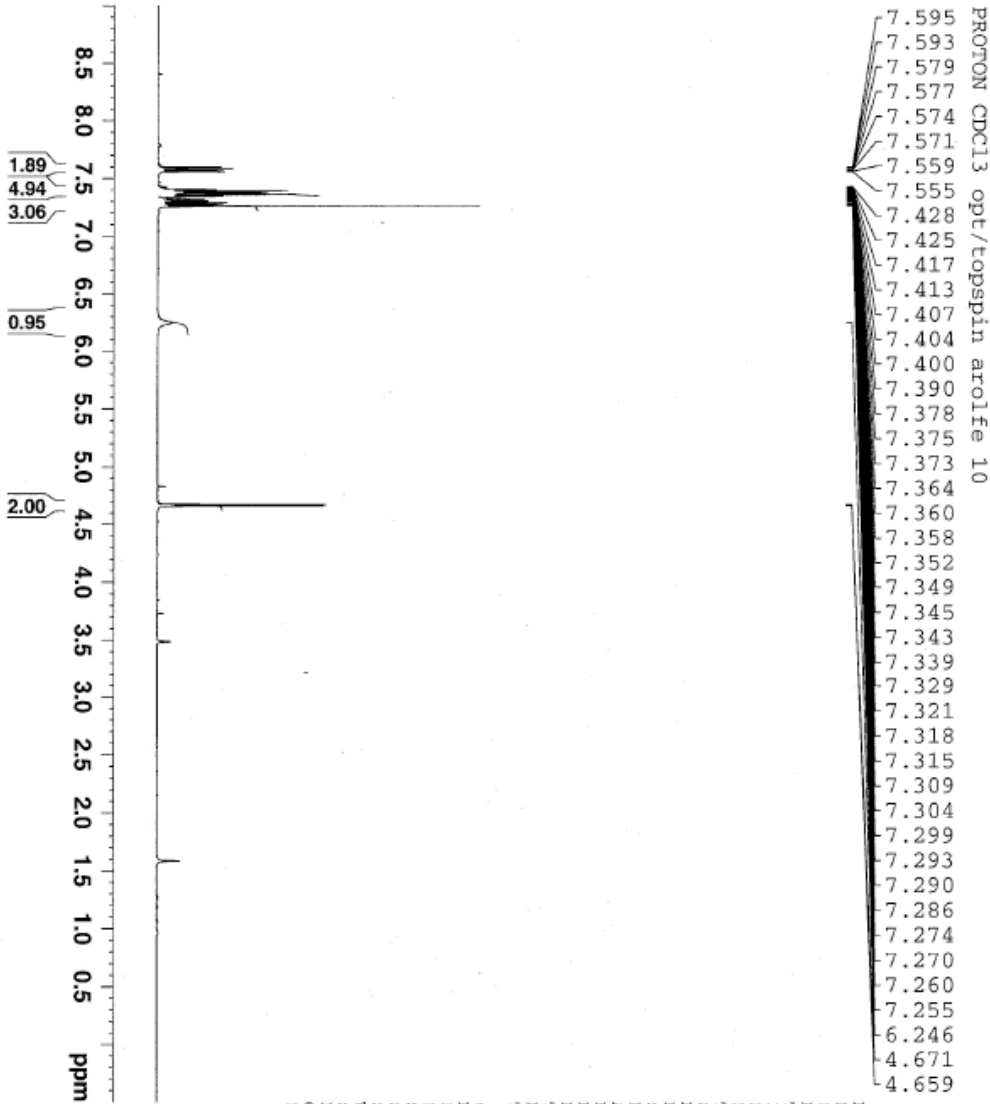
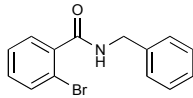




(*E*)-Ethyl 3-(1-(2-ethoxy-2-oxoethyl)-2-(2-methoxyphenyl)-3-isothiazoline-5-yl) prop-2-enoate (**19**)



N-Benzyl-2-bromobenzene (20)



PROTON CDCl3 opt/topspin arolfe 10

7.595
7.593
7.579
7.577
7.574
7.571
7.559
7.555
7.428
7.425
7.417
7.413
7.407
7.404
7.400
7.390
7.378
7.375
7.373
7.364
7.360
7.358
7.352
7.349
7.345
7.343
7.339
7.329
7.321
7.318
7.315
7.309
7.304
7.299
7.293
7.290
7.286
7.274
7.270
7.260
7.255
6.246
4.671
4.659

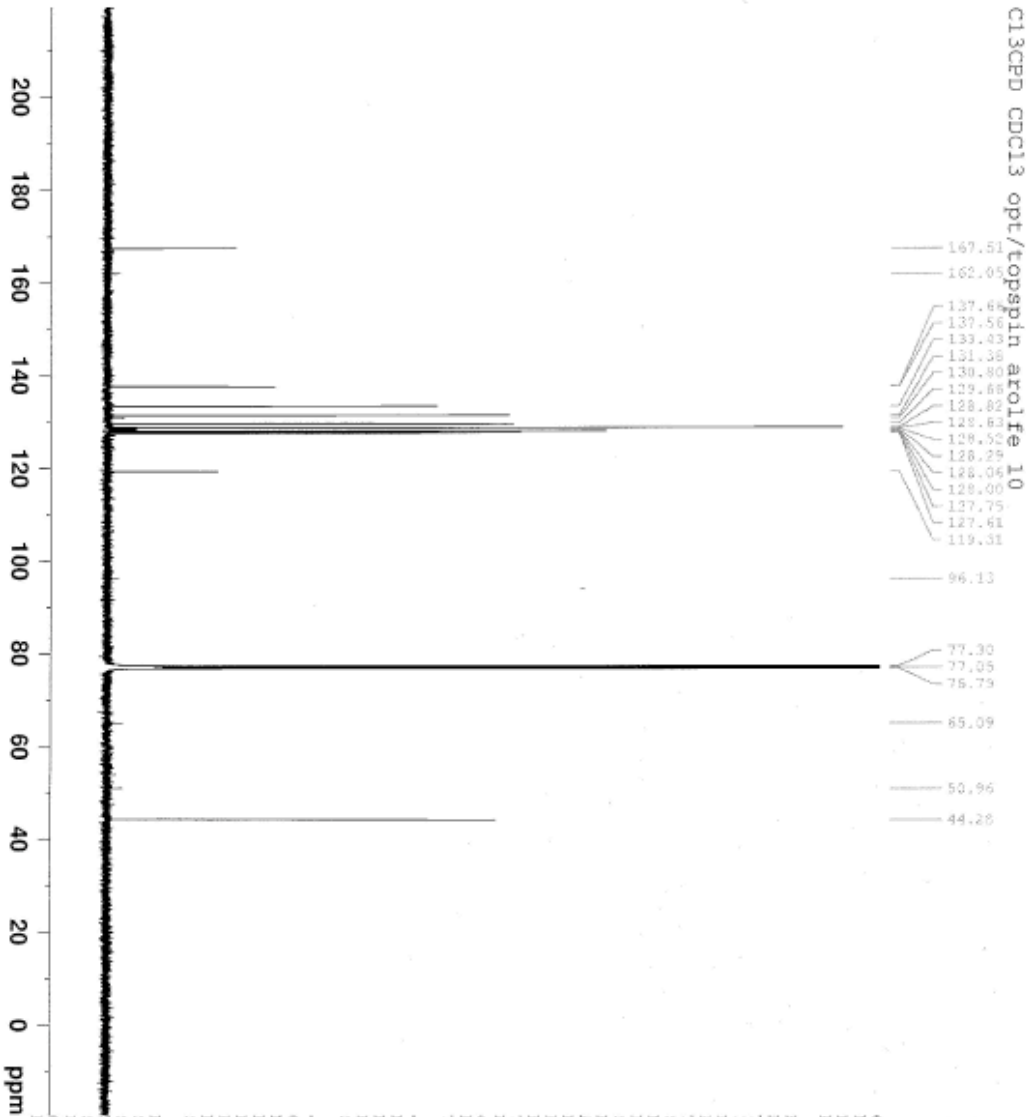
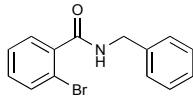


```

NAME          PSL10-642
EXPNO         1
PROCNO        1
Date_         20080521
Time_         17.19
INSTRUM       spect
PROBHD        5 mm CPDUL 13C
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1720407 sec
RG            812.7
WDW           EM
SSB           0
GB            0
PC            1.00

===== CHANNEL f1 =====
NUC1           1H
P1            15.00 usec
PL1           1.60 dB
SFO1          500.130885 MHz
SI            32768
SF            500.1300138 MHz
WDW           EM
SSB           0
GB            0
PC            1.00
    
```

N-Benzyl-2-bromobenzene (20)



C13CPD CDC13 opt/topspin aro1fe 10

- 167.51
- 162.05
- 137.99
- 137.83
- 137.67
- 131.30
- 130.80
- 129.88
- 128.82
- 128.66
- 128.50
- 128.34
- 128.00
- 127.75
- 127.61
- 119.31

- 96.13
- 77.30
- 77.05
- 75.79
- 65.09
- 50.96
- 44.28



Current Data Parameters
 NAME P5110-642
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080521
 Time 17.42

INSTRUM spect
 PROBRD 5 mm CPDUL 13C
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4

SWH 30030.029 Hz
 FIDRES 0.458222 Hz
 AQ 1.0912410 sec
 RG 14596.5
 DW 16.650 usec
 DE 6.00 usec
 TE 299.0 K

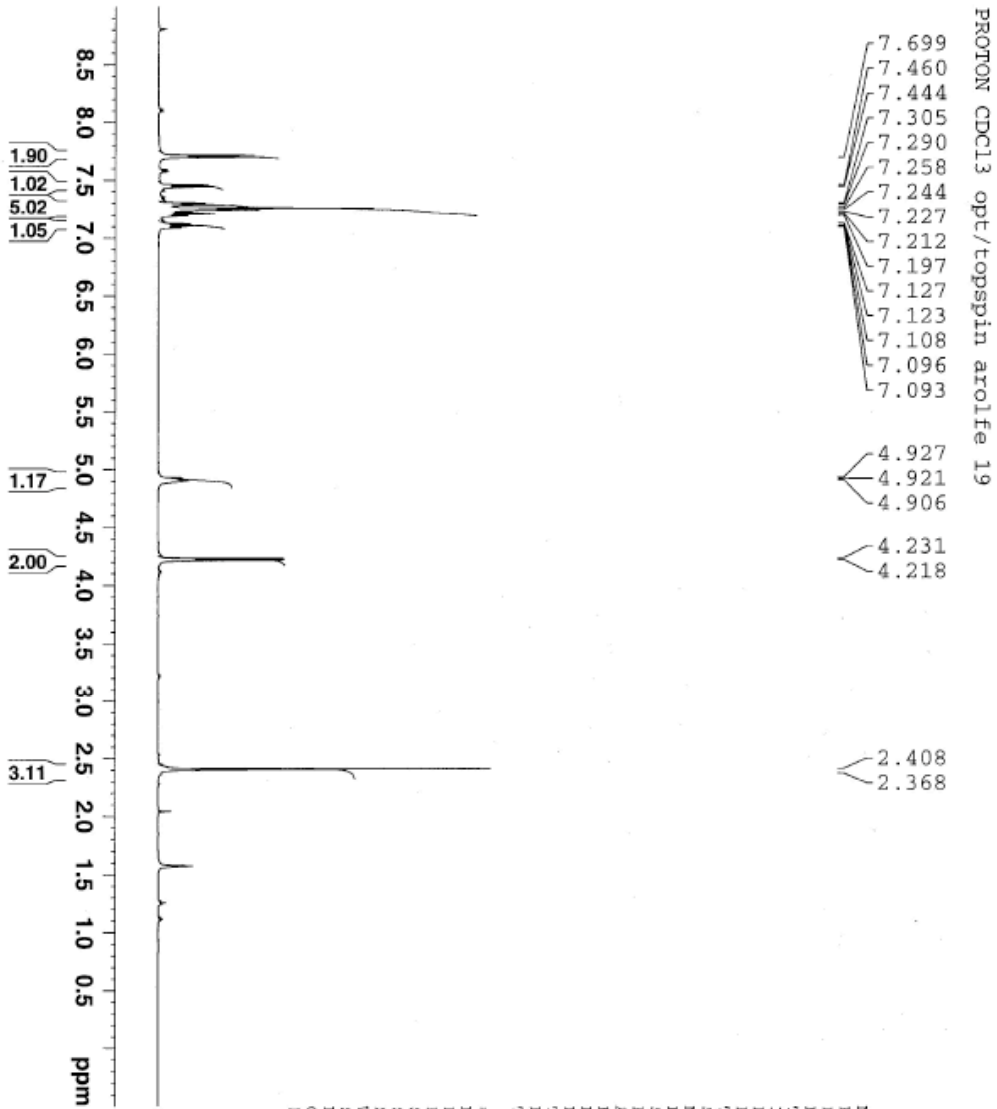
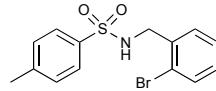
D1 0.15000001 sec
 d11 0.03000000 sec
 DELTA 0.05000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 11.00 usec
 Pl1 2.20 dB
 SFO1 125.7703643 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 1.60 dB
 PL12 16.14 dB
 PL13 10.00 dB
 SFO2 500.1320005 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7577890 MHz
 MDW 0
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

N-(2-Bromobenzyl)-4-methylbenzenesulfonamide (21)



PROTON CDCl3 opt/topspin arolfe 19

7.699
7.460
7.444
7.305
7.290
7.258
7.244
7.227
7.212
7.197
7.127
7.123
7.108
7.096
7.093

4.927
4.921
4.906

4.231
4.218

2.408
2.368

1.90
1.02
5.02
1.05

1.17

2.00

3.11

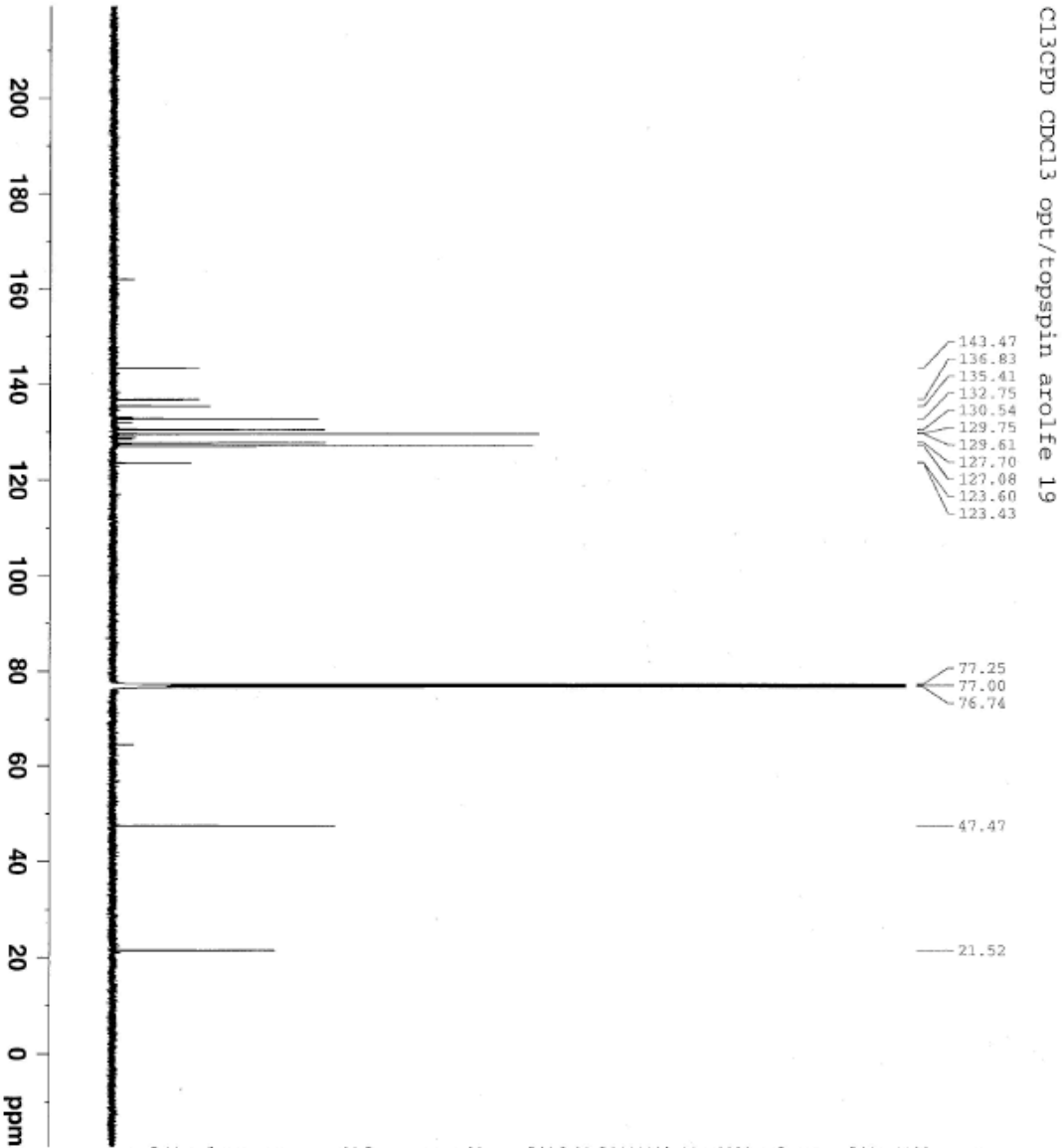
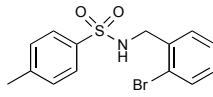


```

NAME          FSI10-633
EXPNO         1
PROCNO        1
Date_         20080520
Time         2.54
INSTRUM       spect
PROBHD        5 mm CPDUL13C
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1720407 sec
RG            812.7
AQ            48.400 usec
DE            6.00 usec
TE            298.1 K
DI            0.15000001 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            15.00 usec
PL1           1.60 dB
SFO1          500.1330885 MHz
SI            32768
SF            500.1300153 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```


N-(2-Bromobenzyl)-4-methylbenzenesulfonamide (21)



143.47
136.83
135.41
132.75
130.54
129.75
129.61
127.70
127.08
123.60
123.43

77.25
77.00
76.74

47.47

21.52



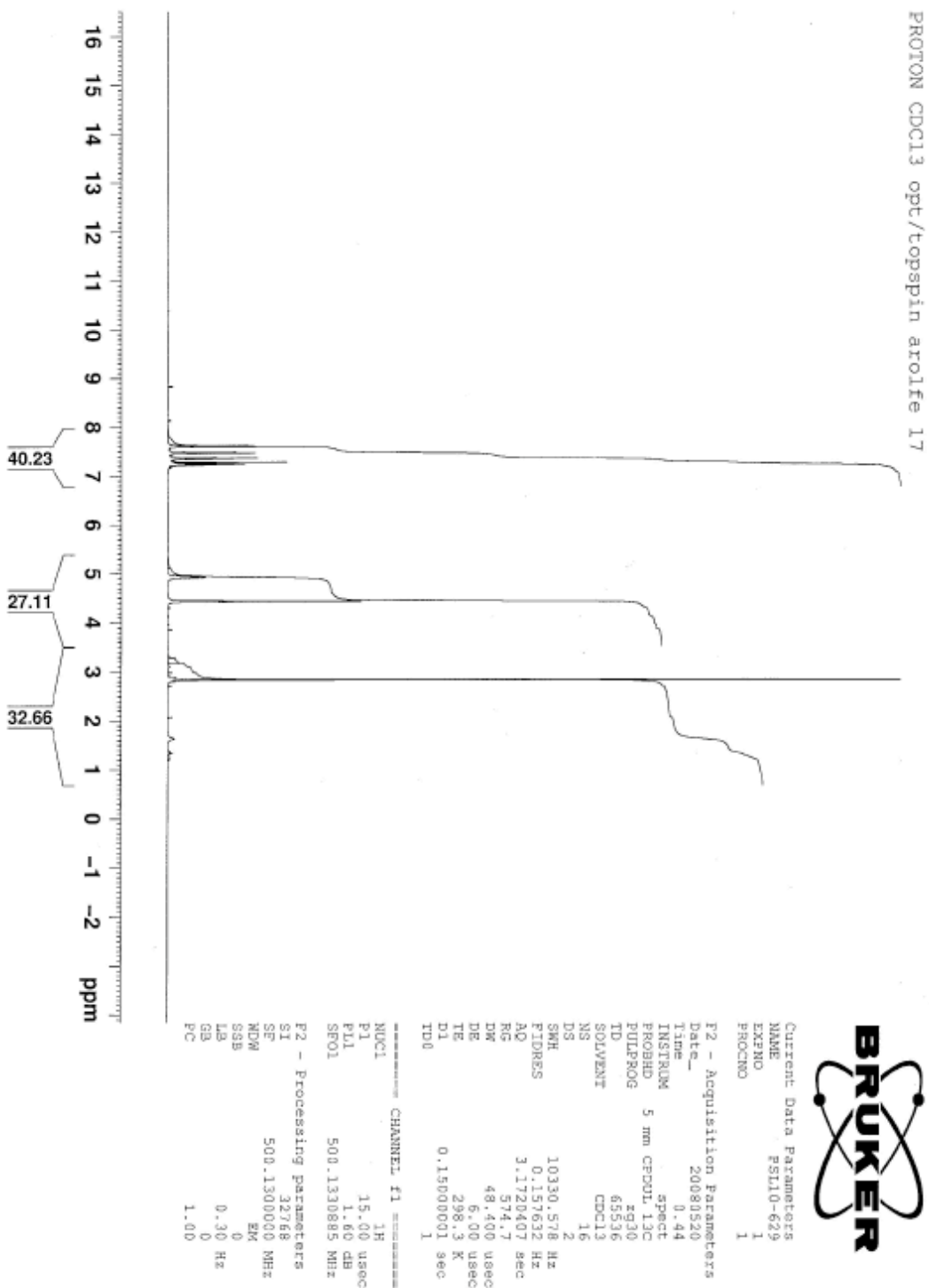
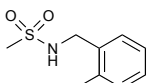
```

NAME          PSL10-633
EXPNO         2
PROCNO        1
Date_         20080520
Time          3.25
INSTRUM       spect
PROBHD        5 mm CPDUL 13C
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            1400
DS            4
SWH           30030.023 Hz
FIDRES        0.458222 Hz
AQ            1.0912410 sec
RG            16394
DE            16.650 usec
TE            298.1 K
D1            0.15000001 sec
d11           0.03000000 sec
DELTA         0.05000000 sec
TD0           1

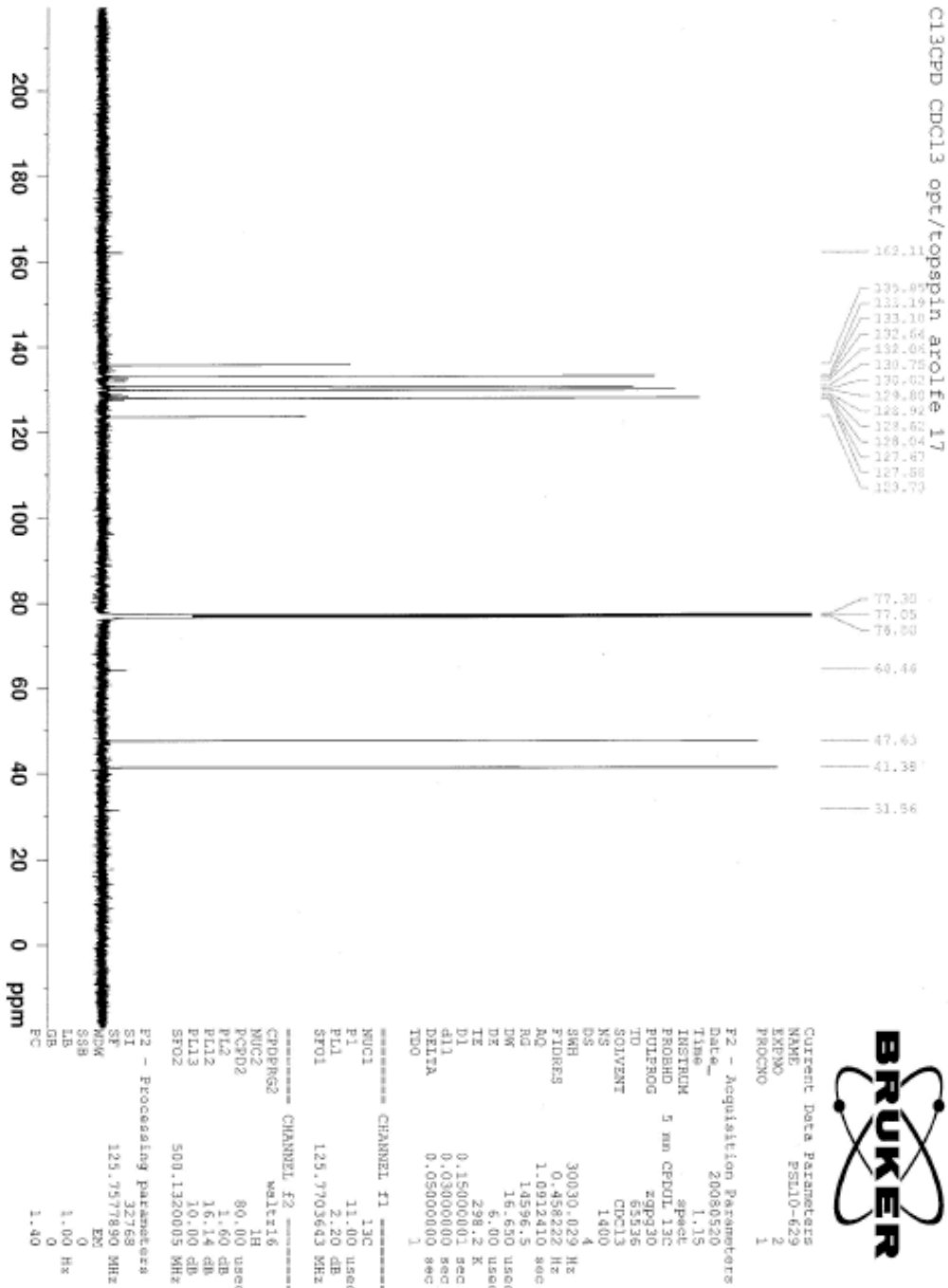
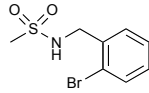
===== CHANNEL f1 =====
NUC1          13C
P1            11.00 usec
PL1           2.20 dB
SFO1         125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2           1.60 dB
PL12         16.14 dB
PL13         10.00 dB
SFO2         500.1320005 MHz
SI           32768
SF           125.7577949 MHz
WDW           EM
SSB           0
LB           1.00 Hz
GB           0
PC           1.40
    
```

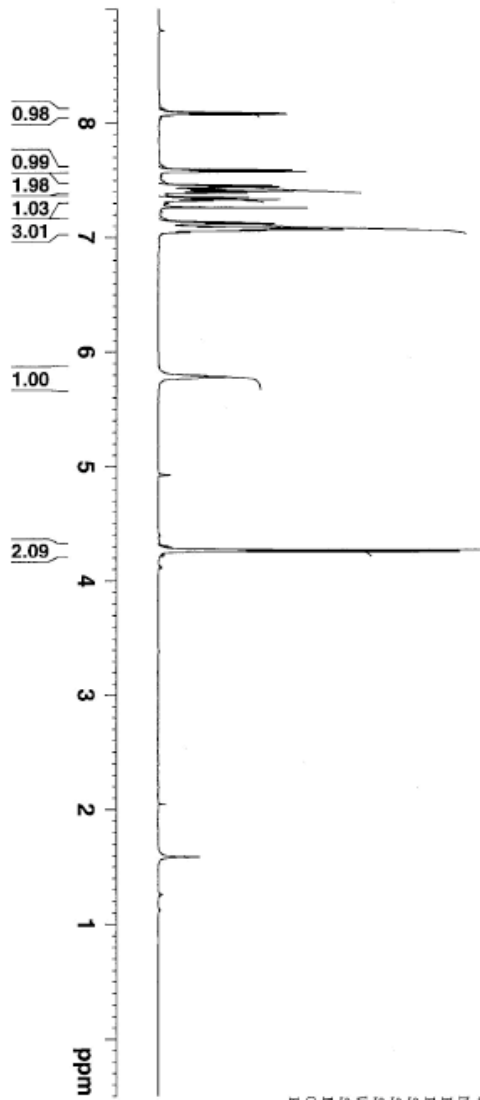
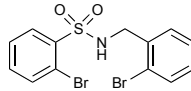
N-(2-Bromobenzyl)methanesulfonamide (22)



N-(2-Bromobenzyl)methanesulfonamide (22)



2-Bromo-N-(2-bromobenzyl)benzenesulfonamide (23)



PROTON CDCl3 opt/topspin aro1fe 18

8.088
8.085
8.073
8.069
7.590
7.587
7.574
7.572
7.456
7.453
7.442
7.438
7.427
7.420
7.418
7.405
7.403
7.390
7.388
7.348
7.345
7.333
7.330
7.318
7.315
7.138
7.133
7.123
7.119
7.098
7.095
7.083
7.080
7.076
7.070
7.065
7.061
7.057
7.047
7.043
5.793
5.783
4.266
4.253

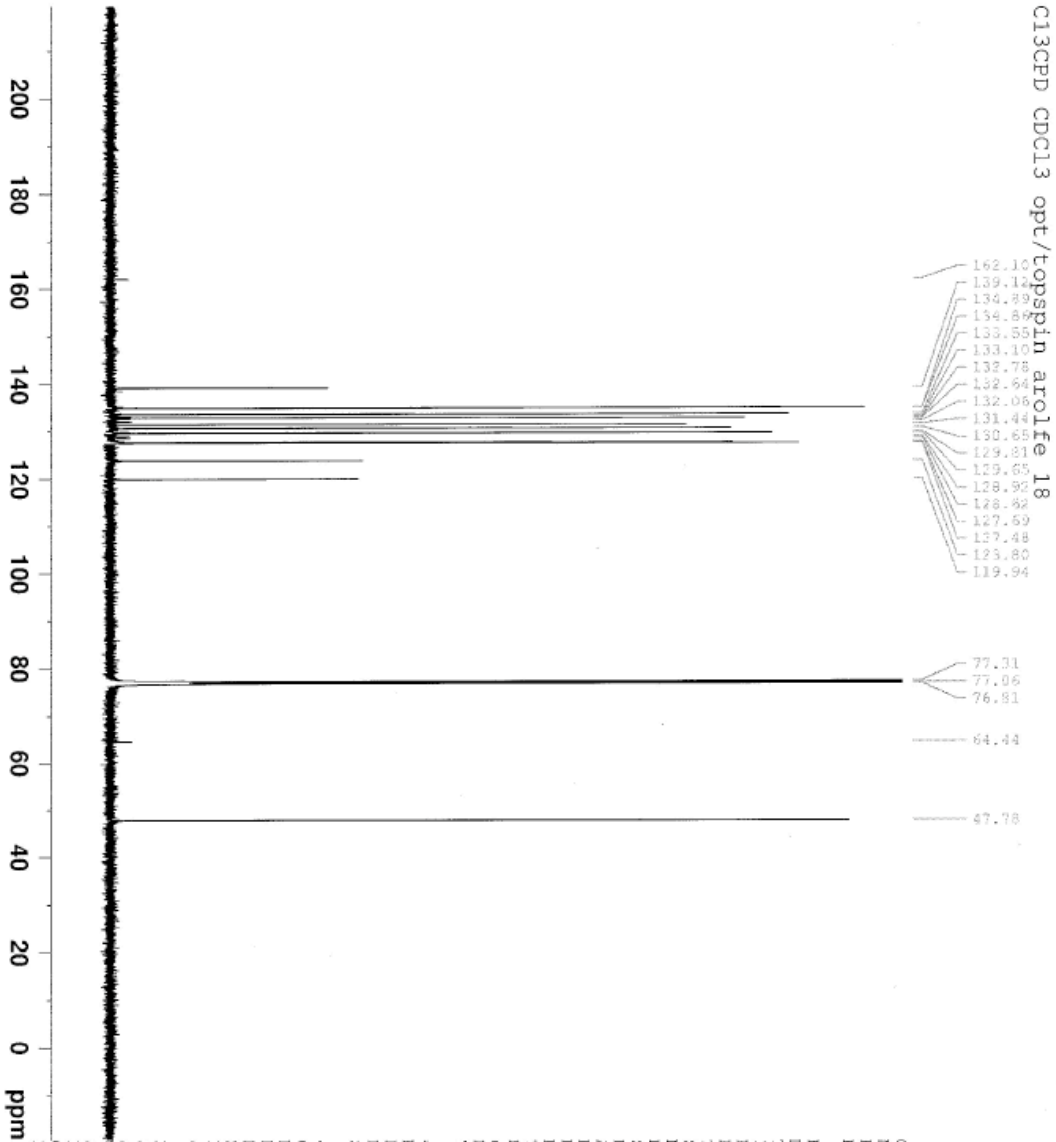
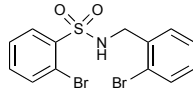


```

NAME          PSL10-630
EXPNO         1
PROCNO        1
Date_         20080520
Time          1.49
INSTRUM       spect
PROBHD        5 mm CPDUL 13C
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           10330.578 Hz
FIDRES        0.157652 Hz
AQ            3.1720407 sec
RG            456.1
DE            48.400 usec
TE            298.2 K
D1            0.15000001 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            15.00 usec
PL1           1.80 dB
SFO1          500.1330885 MHz
SI            32768
SF            500.1300135 MHz
WDW           EM
SSB           0
GB            0
PC            1.00
    
```

2-Bromo-N-(2-bromobenzyl)benzenesulfonamide (23)



C13CPD CDCl3 opt/topspin arolfe 18

- 162.10
- 159.12
- 154.85
- 153.55
- 133.10
- 132.78
- 132.64
- 132.06
- 131.44
- 130.65
- 129.81
- 129.65
- 128.90
- 128.82
- 127.59
- 127.48
- 123.80
- 119.94

- 77.31
- 77.06
- 76.81
- 64.44
- 47.78



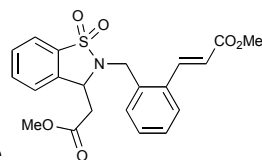
Current Data Parameters
 NAME PS110-630
 EXPNO 2
 F2PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080520
 Time 2.20
 INSTRUM spect
 PROBHD 5 mm CPDUL13C
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1400
 DS 4
 SWH 30030.029 Hz
 FIDRES 0.458222 Hz
 AQ 1.0912410 sec
 RG 14596.5
 DW 16.650 usec
 DE 6.00 usec
 TE 298.2 K
 D1 0.15000001 sec
 d11 0.03000000 sec
 DELTA 0.05000000 sec
 TD0 1

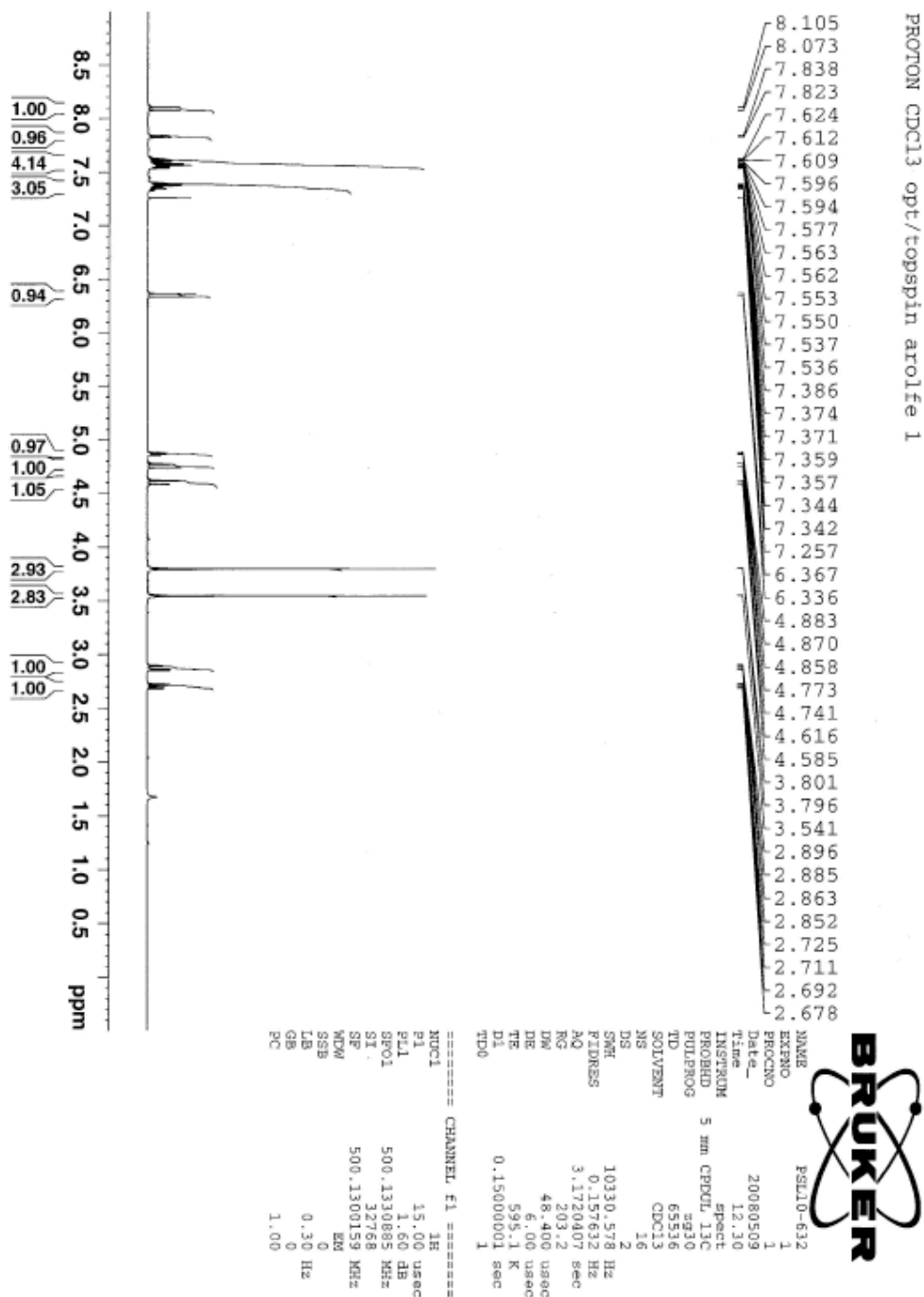
CHANNEL F1
 NUC1 13C
 P1 11.00 usec
 PL1 2.20 dB
 SFO1 125.7703643 MHz

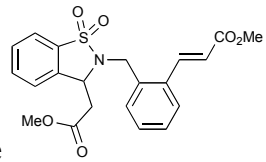
CHANNEL F2
 CPDPRG2 waltz16
 NUC2 1H
 P2 80.00 usec
 PCPD2 1.60 dB
 PL12 16.14 dB
 PL13 10.00 dB
 SFO2 500.1320005 MHz

F2 - Processing parameters
 SI 32768
 SF 125.757890 MHz
 WDW EM
 SSB 0
 GB 0
 PC 1.40

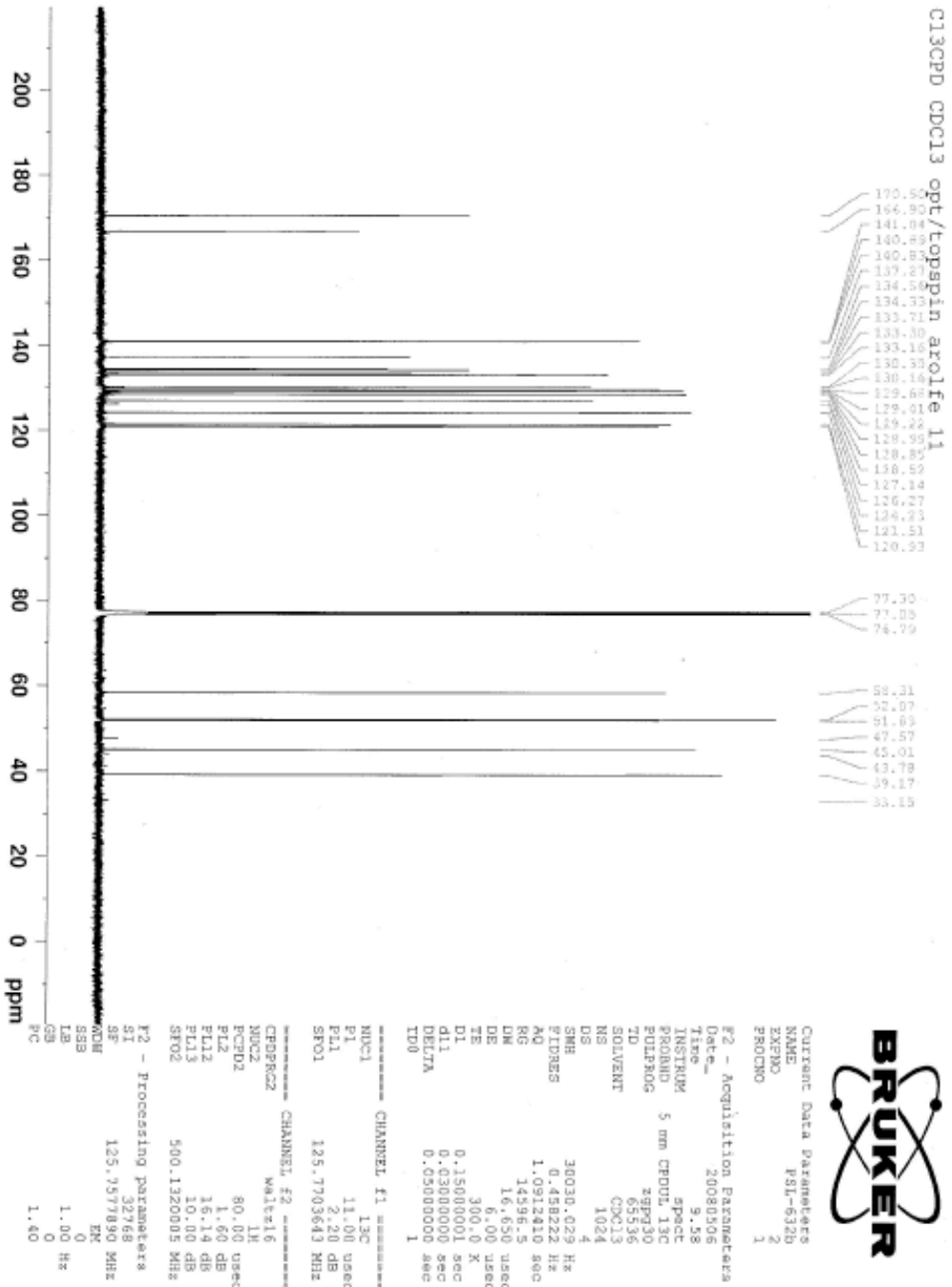


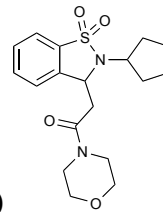
(*E*)-Methyl 3-(2-((1-(2-methoxy-2-oxoethyl)-3-isothiazolin-2-yl)methyl)phenyl)acrylate (24)



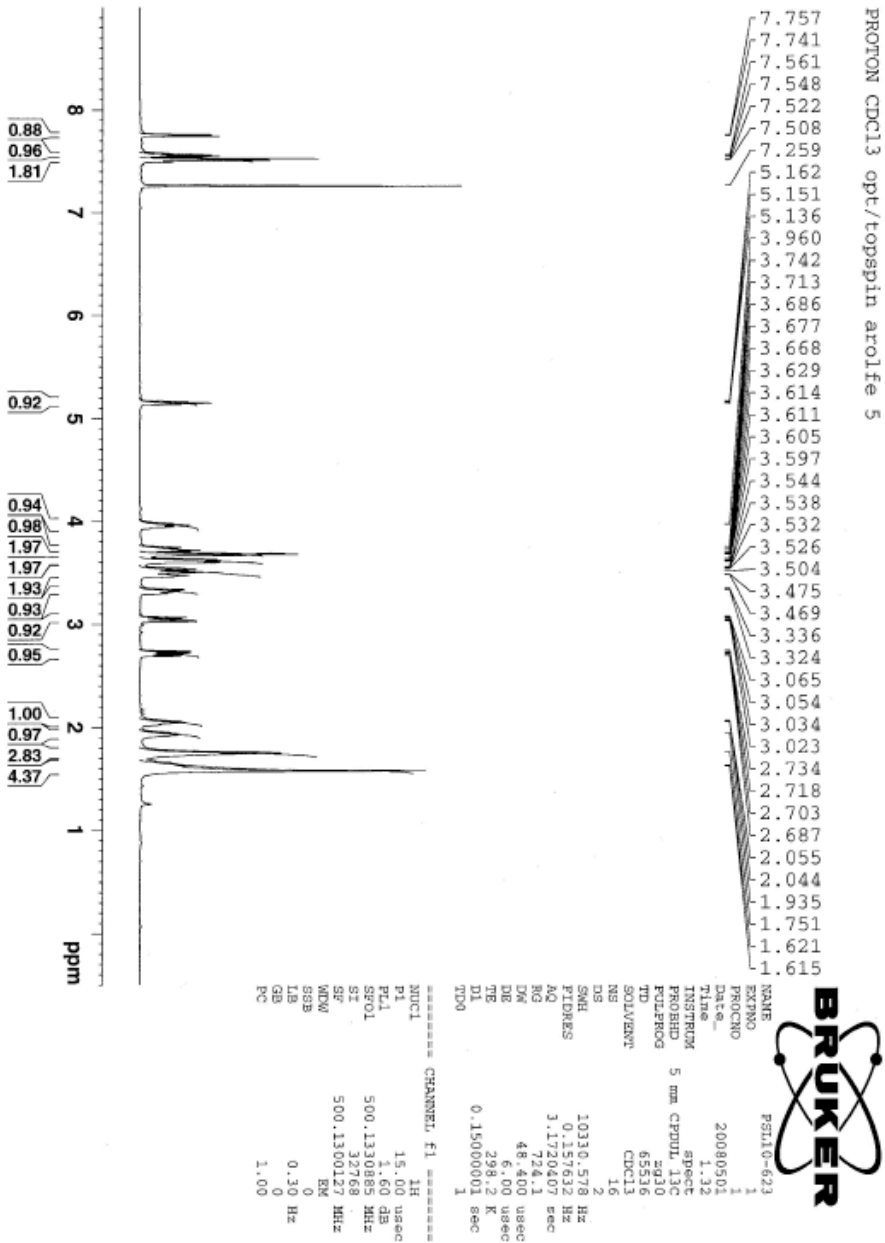


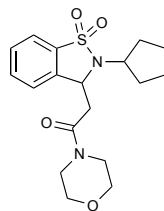
(E)-Methyl 3-(2-((1-(2-methoxy-2-oxoethyl)-3-isothiazolin-2-yl)methyl)phenyl)acrylate (24)



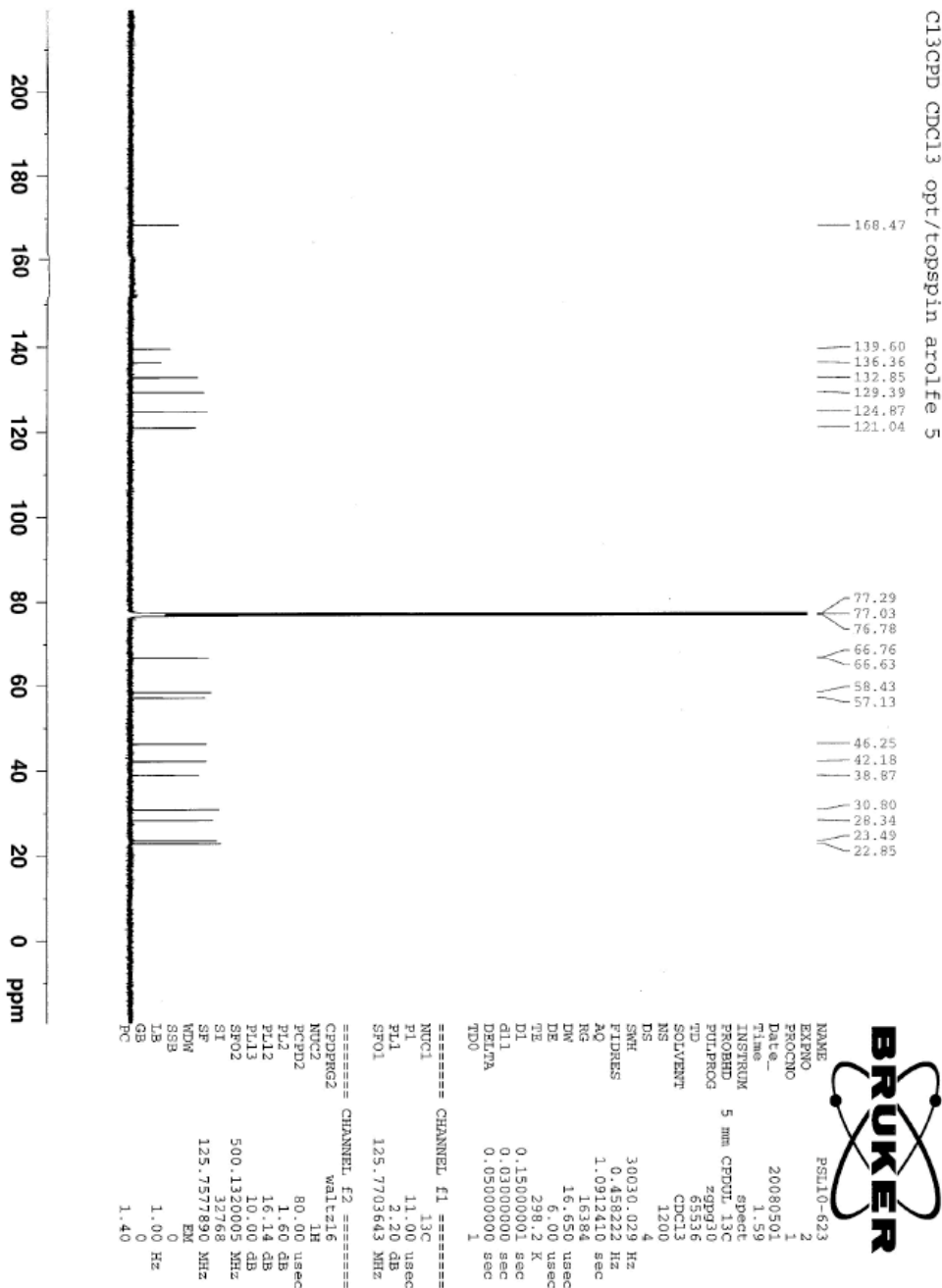


2-Cyclopentyl-3-(2-morpholino-2-oxoethyl)isothiazoline-1-one (29)

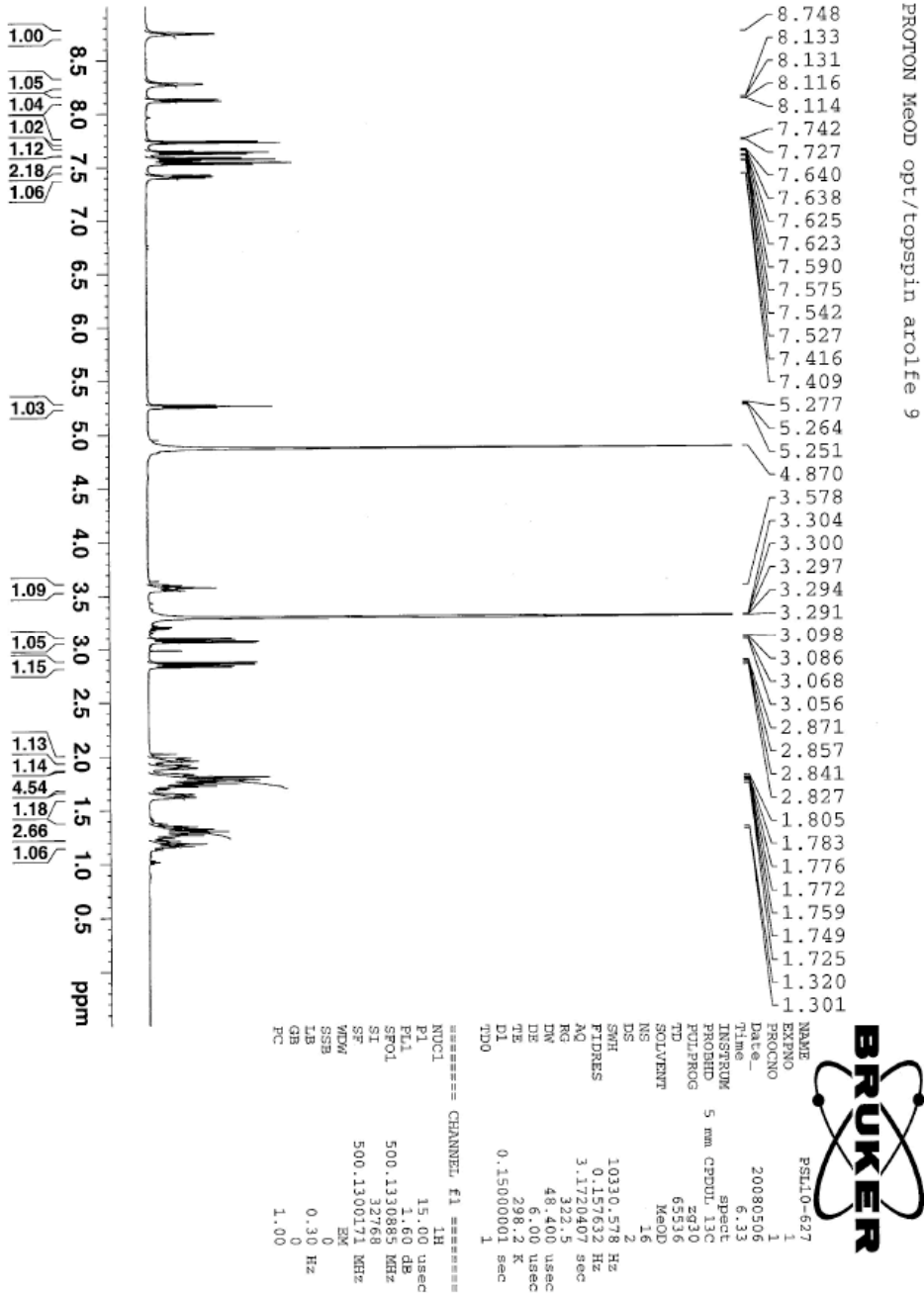
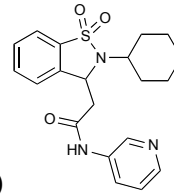


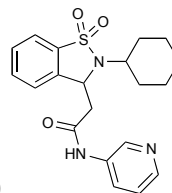


2-Cyclopentyl-3-(2-morpholino-2-oxoethyl)isothiazoline-1-one (29)



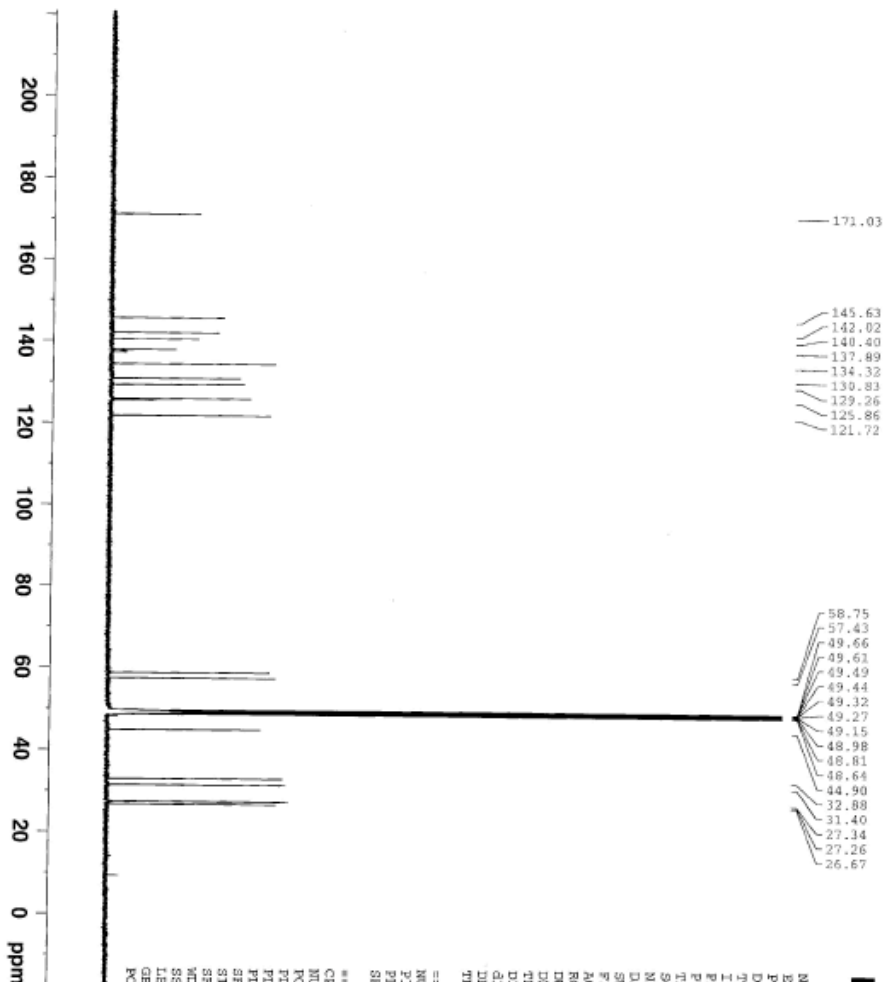
2-(2-Cyclohexyl-3-isothiazoline-1-yl)-N-(pyridin 3yl)acetamide (30)





2-(2-Cyclohexyl-3-isothiazoline-1-yl)-N-(pyridin-3-yl)acetamide (30)

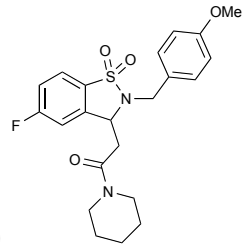
C13CPD MeOD opt/topspin aro1e 9



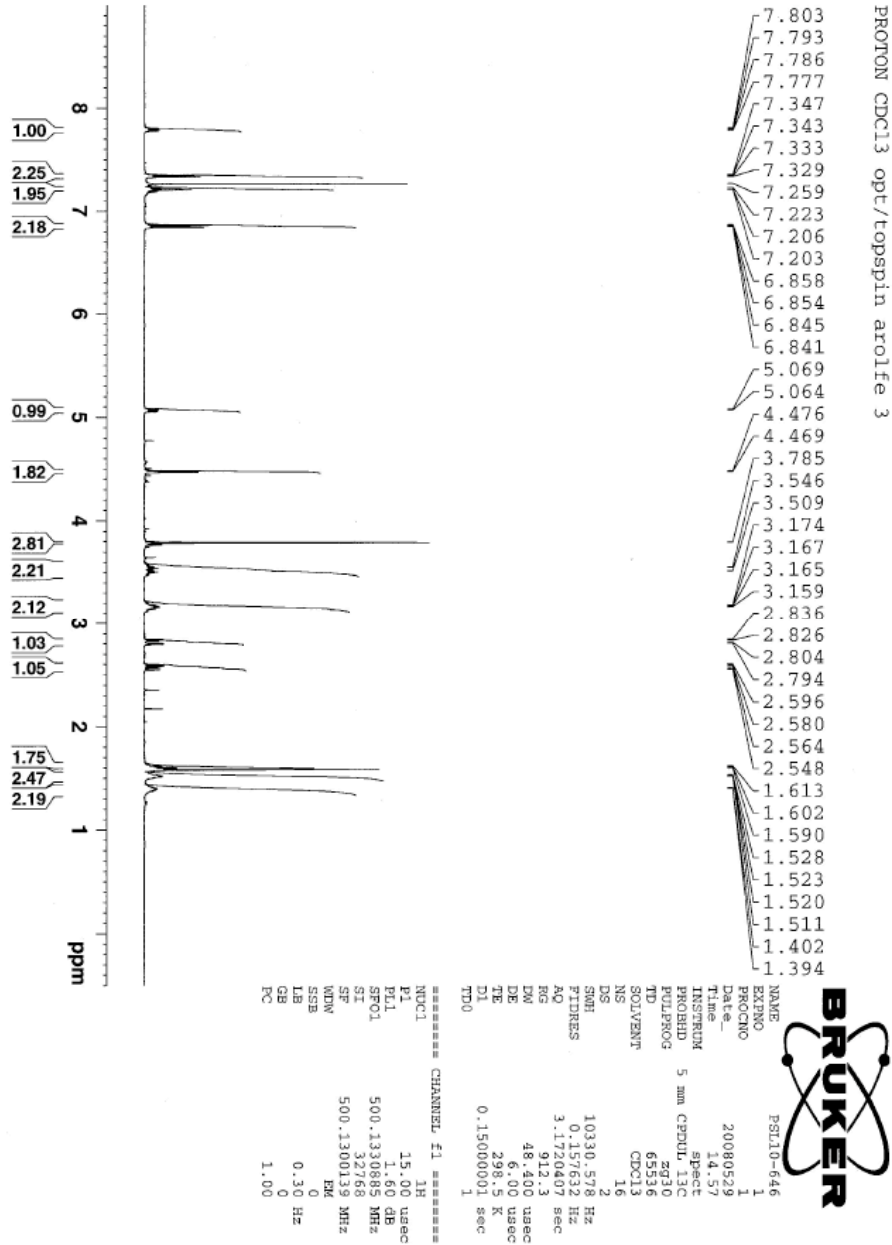
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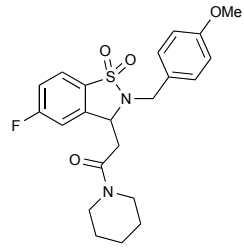
NAME          PSL10-627
EXPNO         2
PROCNO        1
Date_         20080506
Time          7.11
INSTRUM       spect
PROBHD        5 mm CPDUL 13C
PULPROG       zgpg30
TD            65536
SOLVENT       MeOD
NS            1700
DS            4
SWH           30030.029 Hz
FIDRES        0.458222 Hz
AQ           1.0912410 sec
RG           4.3004
DE           18.630 usec
TE           299.2 K
D1           0.1500000 sec
d11          0.0300000 sec
DELTA        0.0500000 sec
TDO          1
===== CHANNEL f1 =====
NUC1          13C
P1           11.00 usec
PL1          2.20 dB
SFO1         125.7703643 MHz
===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2        80.00 usec
PL2          1.60 dB
PL12         16.14 dB
PL13         10.00 dB
SFO2         500.1320005 MHz
SI           32768
SF          125.7575931 MHz
RG           0
KRB          0
NUC3          13C
PCPD3        1.00 Hz
PL3          0
PL4          0
PL5          0
PL6          0
PL7          0
PL8          0
PL9          0
PL10         0
PL11         0
PL12         0
PL13         0
PL14         0
PL15         0
PL16         0
PL17         0
PL18         0
PL19         0
PL20         0
PL21         0
PL22         0
PL23         0
PL24         0
PL25         0
PL26         0
PL27         0
PL28         0
PL29         0
PL30         0
PL31         0
PL32         0
PL33         0
PL34         0
PL35         0
PL36         0
PL37         0
PL38         0
PL39         0
PL40         0
PL41         0
PL42         0
PL43         0
PL44         0
PL45         0
PL46         0
PL47         0
PL48         0
PL49         0
PL50         0
PL51         0
PL52         0
PL53         0
PL54         0
PL55         0
PL56         0
PL57         0
PL58         0
PL59         0
PL60         0
PL61         0
PL62         0
PL63         0
PL64         0
PL65         0
PL66         0
PL67         0
PL68         0
PL69         0
PL70         0
PL71         0
PL72         0
PL73         0
PL74         0
PL75         0
PL76         0
PL77         0
PL78         0
PL79         0
PL80         0
PL81         0
PL82         0
PL83         0
PL84         0
PL85         0
PL86         0
PL87         0
PL88         0
PL89         0
PL90         0
PL91         0
PL92         0
PL93         0
PL94         0
PL95         0
PL96         0
PL97         0
PL98         0
PL99         0
PL100        0
===== CHANNEL f3 =====
CPDPRG3      waltz16
NUC3          13C
PCPD3        80.00 usec
PL3          1.60 dB
PL13         10.00 dB
SFO3         500.1320005 MHz
SI           32768
SF          125.7575931 MHz
RG           0
KRB          0
NUC4          13C
PCPD4        1.00 Hz
PL4          0
PL5          0
PL6          0
PL7          0
PL8          0
PL9          0
PL10         0
PL11         0
PL12         0
PL13         0
PL14         0
PL15         0
PL16         0
PL17         0
PL18         0
PL19         0
PL20         0
PL21         0
PL22         0
PL23         0
PL24         0
PL25         0
PL26         0
PL27         0
PL28         0
PL29         0
PL30         0
PL31         0
PL32         0
PL33         0
PL34         0
PL35         0
PL36         0
PL37         0
PL38         0
PL39         0
PL40         0
PL41         0
PL42         0
PL43         0
PL44         0
PL45         0
PL46         0
PL47         0
PL48         0
PL49         0
PL50         0
PL51         0
PL52         0
PL53         0
PL54         0
PL55         0
PL56         0
PL57         0
PL58         0
PL59         0
PL60         0
PL61         0
PL62         0
PL63         0
PL64         0
PL65         0
PL66         0
PL67         0
PL68         0
PL69         0
PL70         0
PL71         0
PL72         0
PL73         0
PL74         0
PL75         0
PL76         0
PL77         0
PL78         0
PL79         0
PL80         0
PL81         0
PL82         0
PL83         0
PL84         0
PL85         0
PL86         0
PL87         0
PL88         0
PL89         0
PL90         0
PL91         0
PL92         0
PL93         0
PL94         0
PL95         0
PL96         0
PL97         0
PL98         0
PL99         0
PL100        0
===== CHANNEL f4 =====
CPDPRG4      waltz16
NUC4          13C
PCPD4        80.00 usec
PL4          1.60 dB
PL14         10.00 dB
SFO4         500.1320005 MHz
SI           32768
SF          125.7575931 MHz
RG           0
KRB          0
NUC5          13C
PCPD5        80.00 usec
PL5          1.60 dB
PL15         10.00 dB
SFO5         500.1320005 MHz
SI           32768
SF          125.7575931 MHz
RG           0
KRB          0
NUC6          13C
PCPD6        80.00 usec
PL6          1.60 dB
PL16         10.00 dB
SFO6         500.1320005 MHz
SI           32768
SF          125.7575931 MHz
RG           0
KRB          0
NUC7          13C
PCPD7        80.00 usec
PL7          1.60 dB
PL17         10.00 dB
SFO7         500.1320005 MHz
SI           32768
SF          125.7575931 MHz
RG           0
KRB          0
NUC8          13C
PCPD8        80.00 usec
PL8          1.60 dB
PL18         10.00 dB
SFO8         500.1320005 MHz
SI           32768
SF          125.7575931 MHz
RG           0
KRB          0
NUC9          13C
PCPD9        80.00 usec
PL9          1.60 dB
PL19         10.00 dB
SFO9         500.1320005 MHz
SI           32768
SF          125.7575931 MHz
RG           0
KRB          0
NUC10         13C
PCPD10       80.00 usec
PL10         1.60 dB
PL20         10.00 dB
SFO10        500.1320005 MHz
SI           32768
SF          125.7575931 MHz
RG           0
KRB          0
NUC11         13C
PCPD11       80.00 usec
PL11         1.60 dB
PL21         10.00 dB
SFO11        500.1320005 MHz
SI           32768
SF          125.7575931 MHz
RG           0
KRB          0
NUC12         13C
PCPD12       80.00 usec
PL12         1.60 dB
PL22         10.00 dB
SFO12        500.1320005 MHz
SI           32768
SF          125.7575931 MHz
RG           0
KRB          0
NUC13         13C
PCPD13       80.00 usec
PL13         1.60 dB
PL23         10.00 dB
SFO13        500.1320005 MHz
SI           32768
SF          125.7575931 MHz
RG           0
KRB          0
NUC14         13C
PCPD14       80.00 usec
PL14         1.60 dB
PL24         10.00 dB
SFO14        500.1320005 MHz
SI           32768
SF          125.7575931 MHz
RG           0
KRB          0
NUC15         13C
PCPD15       80.00 usec
PL15         1.60 dB
PL25         10.00 dB
SFO15        500.1320005 MHz
SI           32768
SF          125.7575931 MHz
RG           0
KRB          0
NUC16         13C
PCPD16       80.00 usec
PL16         1.60 dB
PL26         10.00 dB
SFO16        500.1320005 MHz
SI           32768
SF          125.7575931 MHz
RG           0
KRB          0
NUC17         13C
PCPD17       80.00 usec
PL17         1.60 dB
PL27         10.00 dB
SFO17        500.1320005 MHz
SI           32768
SF          125.7575931 MHz
RG           0
KRB          0
NUC18         13C
PCPD18       80.00 usec
PL18         1.60 dB
PL28         10.00 dB
SFO18        500.1320005 MHz
SI           32768
SF          125.7575931 MHz
RG           0
KRB          0
NUC19         13C
PCPD19       80.00 usec
PL19         1.60 dB
PL29         10.00 dB
SFO19        500.1320005 MHz
SI           32768
SF          125.7575931 MHz
RG           0
KRB          0
NUC20         13C
PCPD20       80.00 usec
PL20         1.60 dB
PL30         10.00 dB
SFO20        500.1320005 MHz
SI           32768
SF          125.7575931 MHz
RG           0
KRB          0

```



**5-Fluoro-2-(4-methoxybenzyl)-3-(2-oxo-2-(piperidin-1-yl)ethyl)
isothiazoline-1-one (31)**





**5-Fluoro-2-(4-methoxybenzyl)-3-(2-oxo-2-(piperidin-1-yl)ethyl)
isothiazoline-1-one (31)**

