

Solid Phase *versus* Solution Phase Synthesis of Heterocyclic Macrocycles

Seong Jong Kim¹ and Shelli R. McAlpine^{1,*}

¹ School of Chemistry, University of New South Wales, Sydney, NSW 2052 Australia

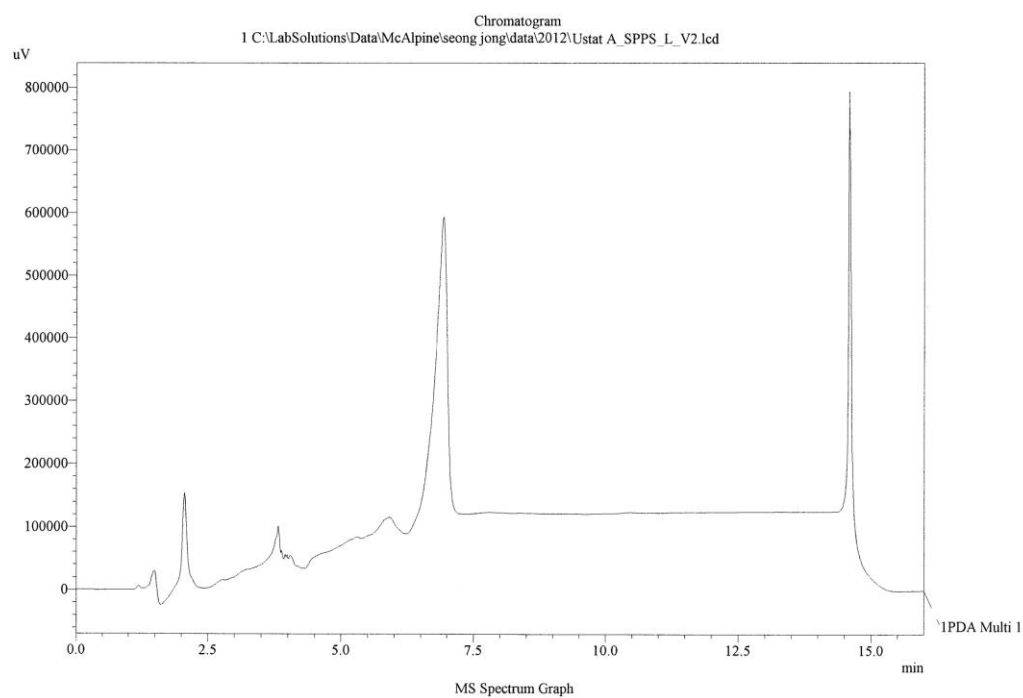
E-Mail: s.mcalpine@unsw.edu.au (S.R. McAlpine)

Supporting Information Available

Spectra of compounds

Linier precursor (2).....	S2
LCMS (ESI)	
HRMS (ESI)	
¹ H NMR	
¹³ C NMR	
Cyclized compound (3).....	S6
LCMS (ESI)	
HRMS (ESI)	
¹ H NMR	
¹³ C NMR	
Oxazoles contained macrocycle (4).....	S10
LCMS (ESI)	
HRMS (ESI)	
¹ H NMR	
¹³ C NMR	

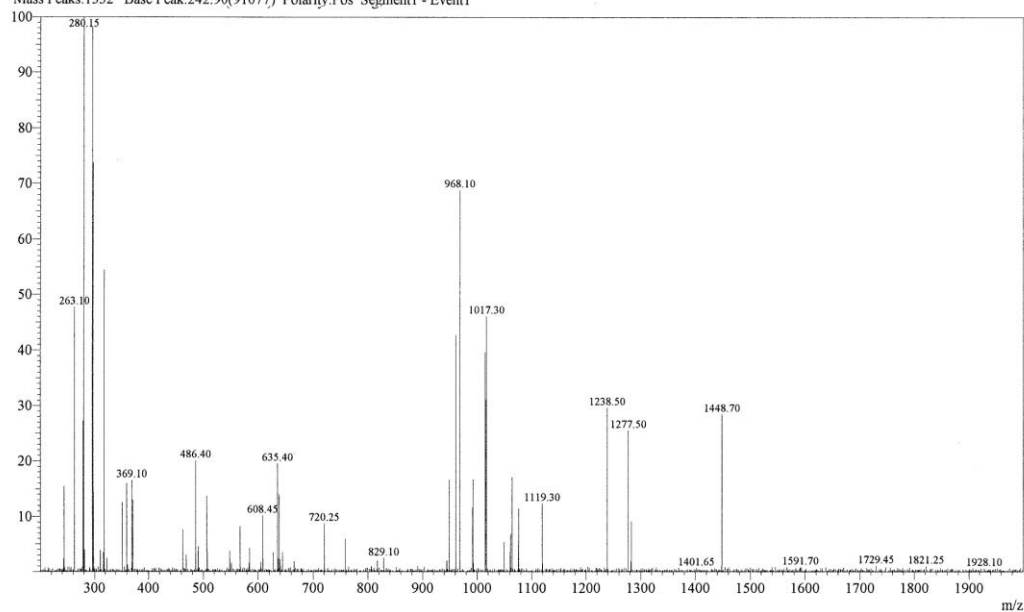
==== Shimadzu LCMSSolution Analysis Report ====



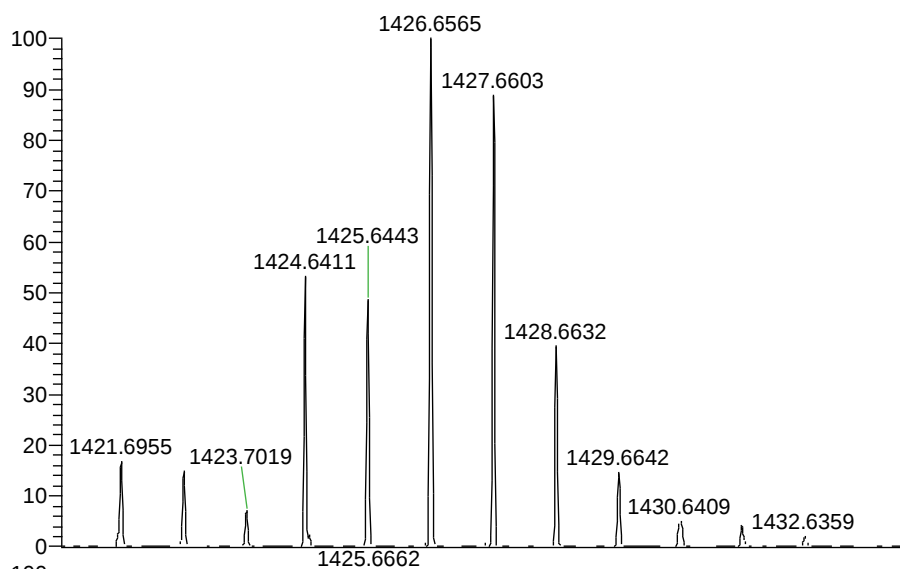
Ret Time: 6.467 (Scan#: 383)

BG Mode: ?

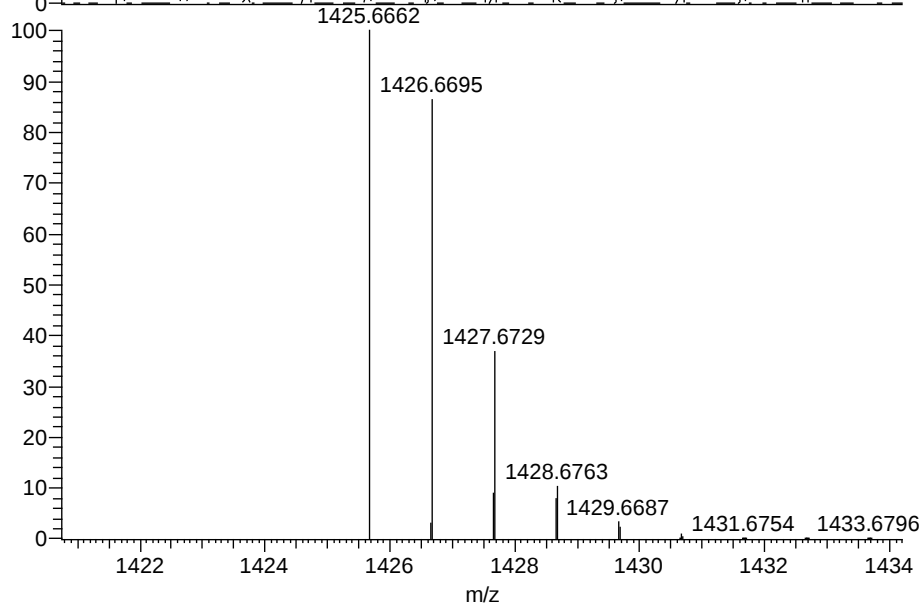
Mass Peaks: 1332 Base Peak: 242.90 (91077) Polarity: Pos Segment 1 - Event 1



Linier precursor (2)_HRMS (ESI)



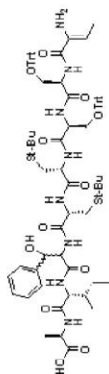
NL:
2.50E6
WT_Sample12#1-17
RT: 0.01-0.48 AV: 17 T:
FTMS + p NSI Full ms
[150.00-2000.00]



NL:
3.55E5
C80H96N8O12S2 +H+
C80H97N8O12S2
pa Chrg 1

Linier precursor(2) - ¹H NMR

Supervisor Shelli McAlpine
Ustat A SPPS L_V2
1H CDCl3 F:\sjk 55

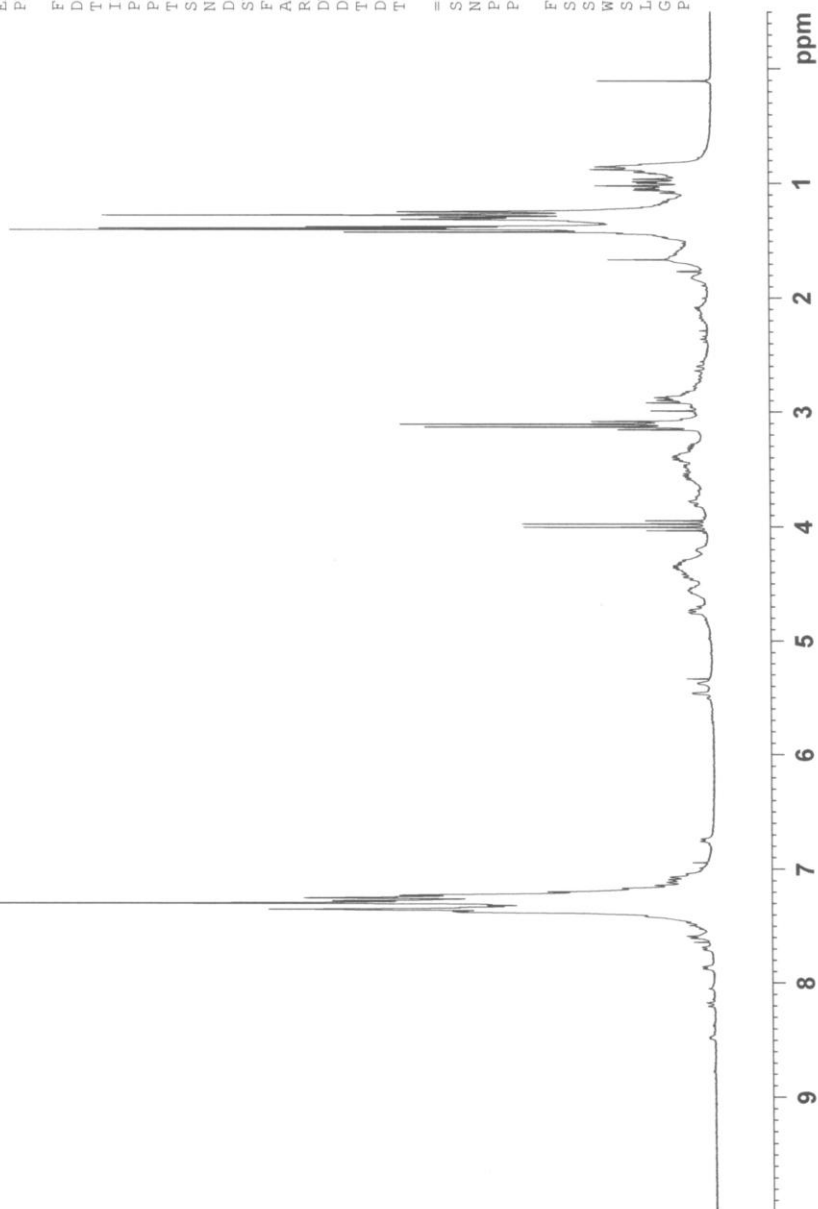


Current Data Parameters
NAME 120807-sjk
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120807
Time_ 23.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 128
DS 0
SWH 4801.537 Hz
FIDRES 0.146531 Hz
AQ 3.4122410 sec
RG 203
DW 104.133 usec
DE 10.47 usec
TE 298.0 K
D1 5.00000000 sec
TD0 1

===== CHANNEL f1 =====
SF01 300.1719511 MHz
NUC1 1H
P1 14.95 usec
PLW1 8.19999981 W

F2 - Processing parameters
SI 131072
SF 300.1700000 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



Linier precursor(2)¹³C NMR



```

Current Data Parameters
NAME      120706-sjk
EXPNO     2
PROCNO    1

F2 - Acquisition Parameters
Date_     20120708
Time      4.03
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         6144
DS         2
SWH        18028.846 Hz
FIDRES     0.275098 Hz
AQ         1.8175317 sec
RG         203
DE         27.733 usec
TE         298.0 K
D1         1.0000000 sec
D11        0.0300000 sec
TD0        6

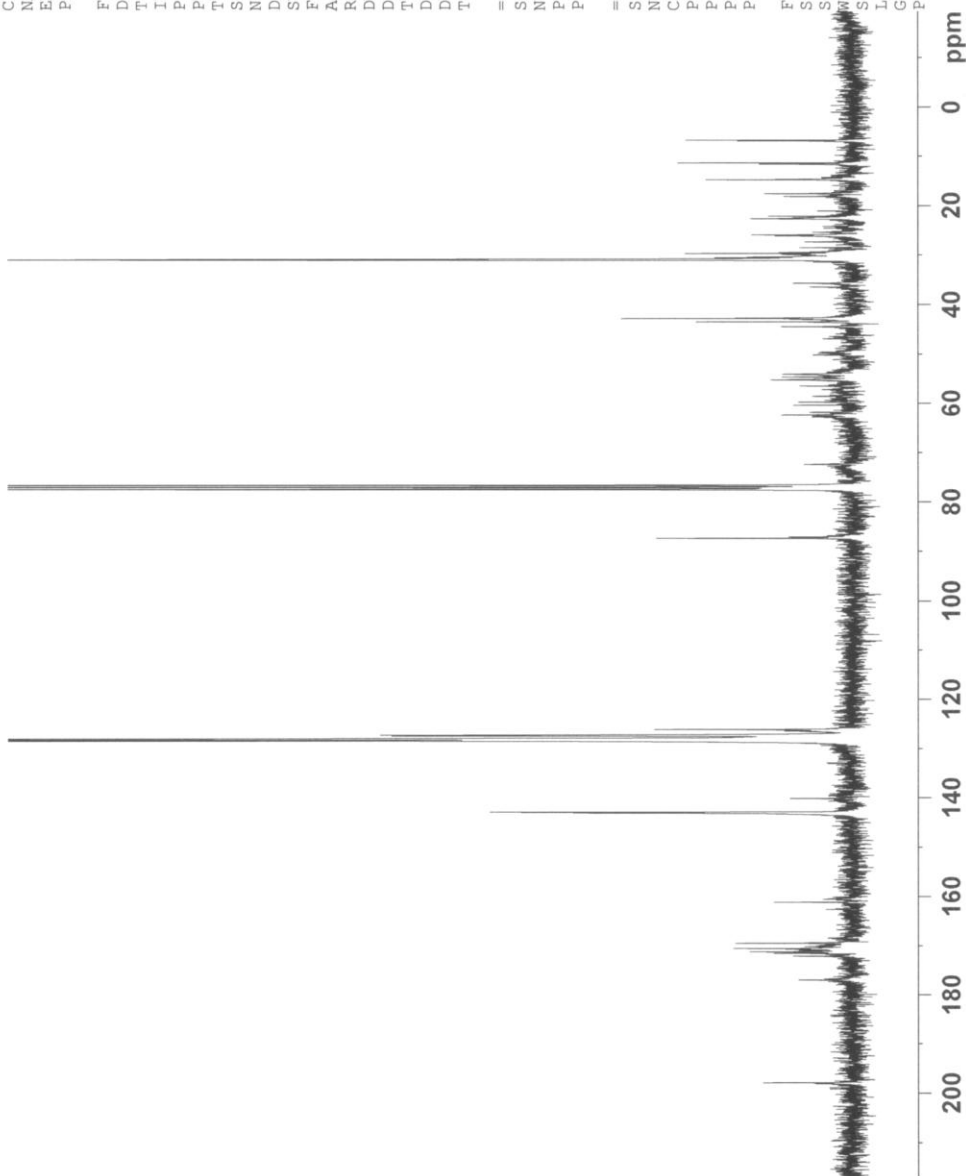
===== CHANNEL f1 =====
SFO1      75.4853543 MHz
NUC1       13C
P1         9.90 usec
PLW1      33.0000000 W

===== CHANNEL f2 =====
SFO2      300.1712007 MHz
NUC2       1H
CPDPRG2   bi_waltz65_256
PCPD2     90.00 usec
PLW2      8.20349979 W
PLW12     0.22635999 W
PLW13     0.18335000 W

F2 - Processing parameters
SI         32768
SF         75.4778070 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

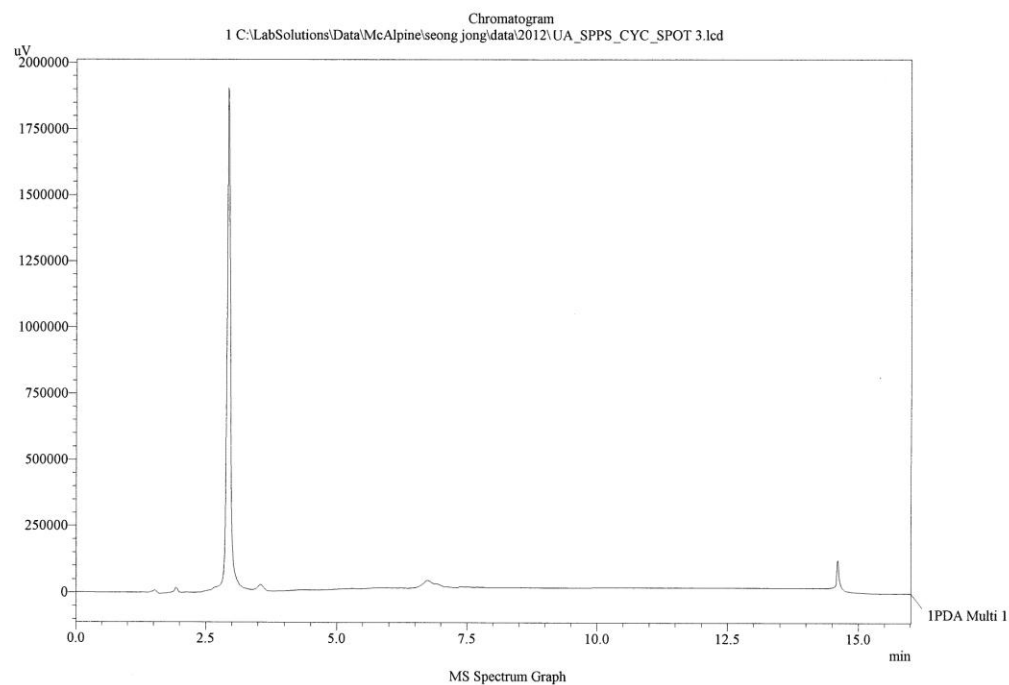
```

supervisor Shelli McAlpine
Istat A L_SPPS
3C.night CDCl3 F:\ sjk 34
  
```



Cyclized compound(3)_LCMS (ESI)

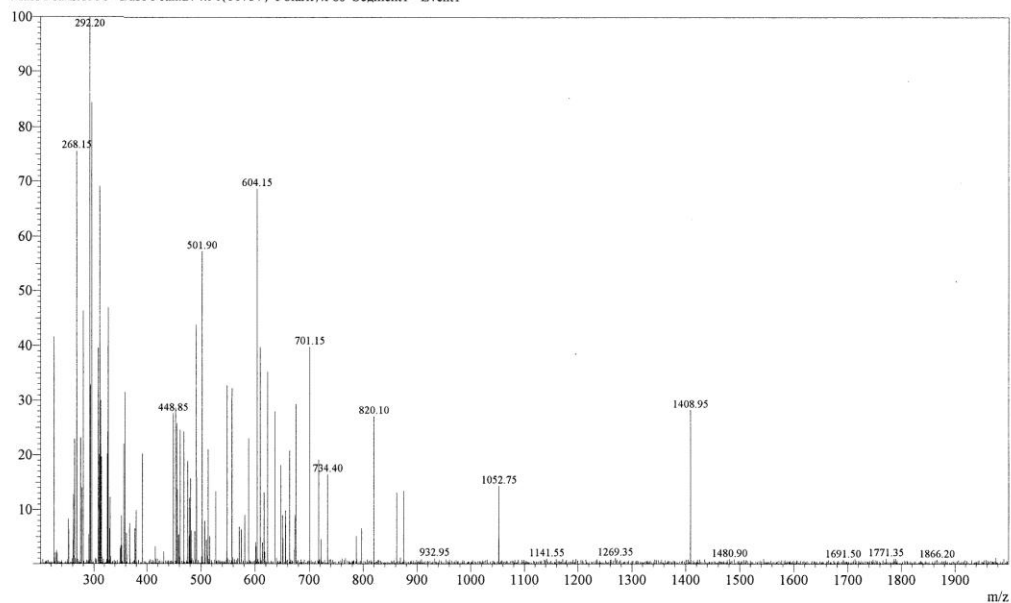
==== Shimadzu LCMSsolution Analysis Report ====



Ret.Time:3.100(Scan#:181)

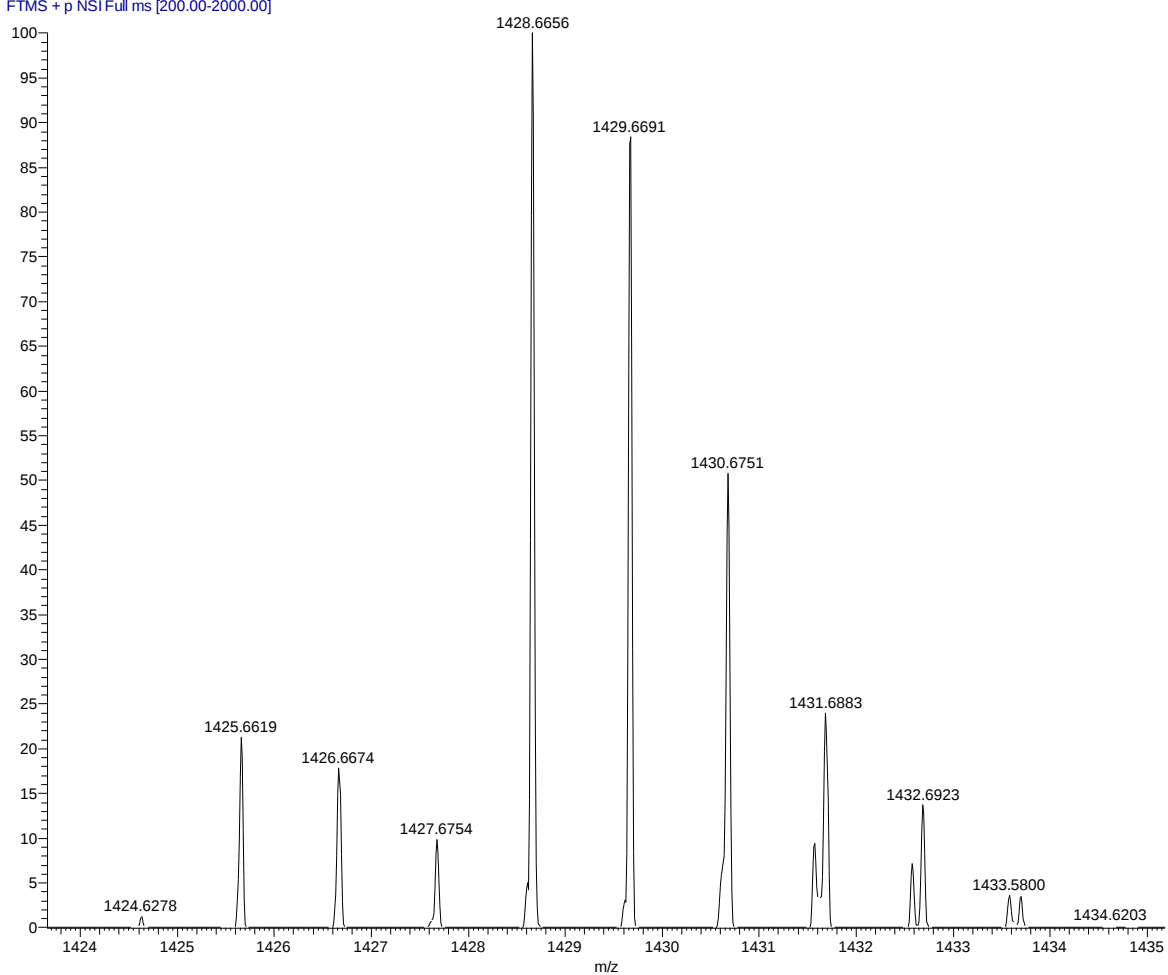
BG Mode:?

Mass Peaks:1338 Base Peak:274.90(60757) Polarity:Pos Segment1 - Event1



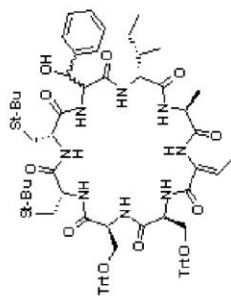
Cyclized compound(3) _ HRMS (ESI)

Chun 2 Pos Full #2-33 RT: 0.03-0.48 AV: 32 NL: 6.85E5
T: FTMS + p NSI Full ms [200.00-2000.00]



Cyclized compound(3) ^1H NMR

Supervisor Shelli McAlpine
IA_SPPS_CYC spot 3
H₂O CDCl₃ F:\sjk 8



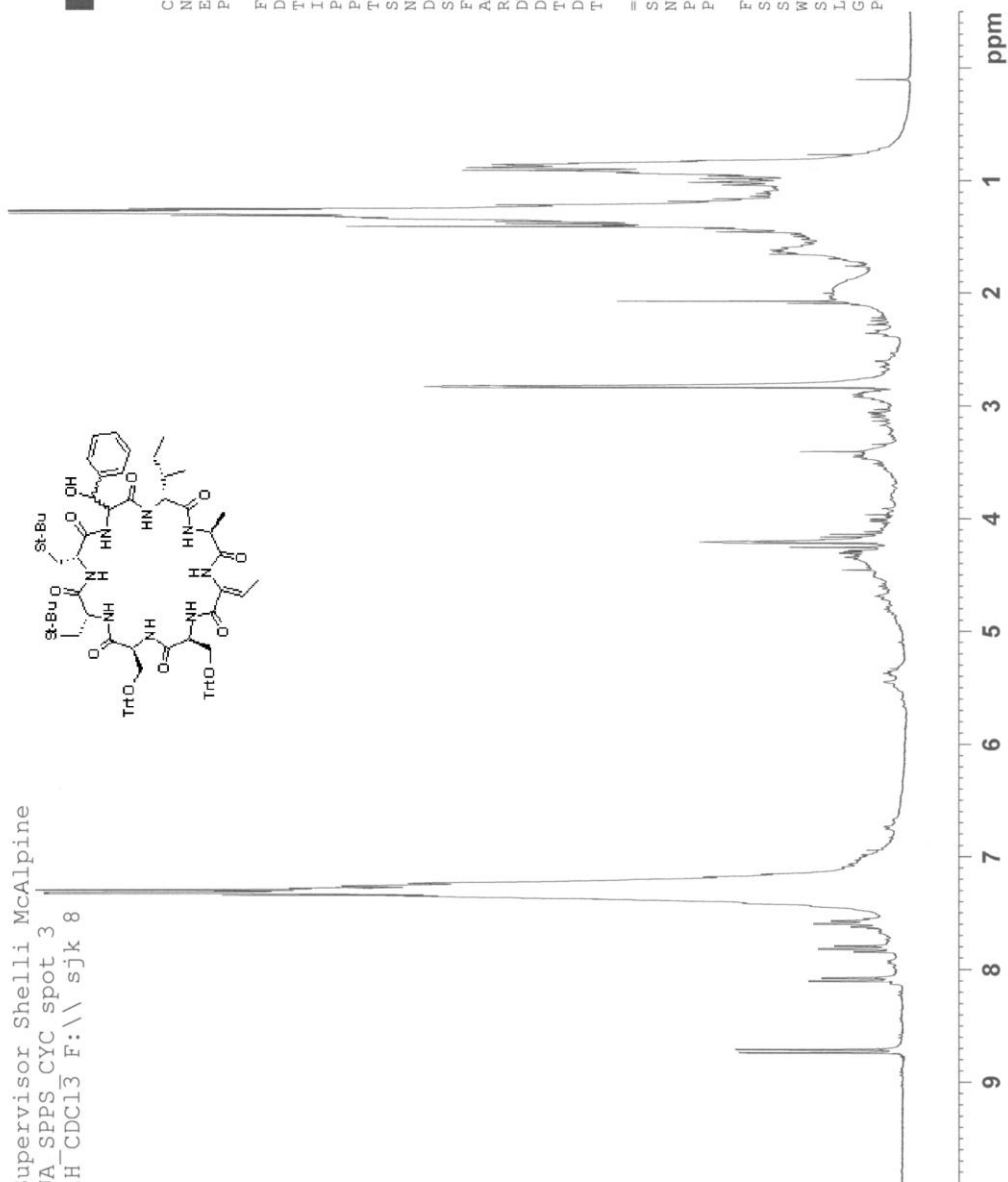
BRUKER

Current Data Parameters
NAME 120824-sjk
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20120825
Time_ 14.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl₃
NS 64
DS 0
SWH 4801.537 Hz
FIDRES 0.146531 Hz
AQ 3.4122410 sec
RG 90.5
DW 104.133 usec
DE 10.47 usec
TE 298.0 K
D1 5.00000000 sec
TD0 1

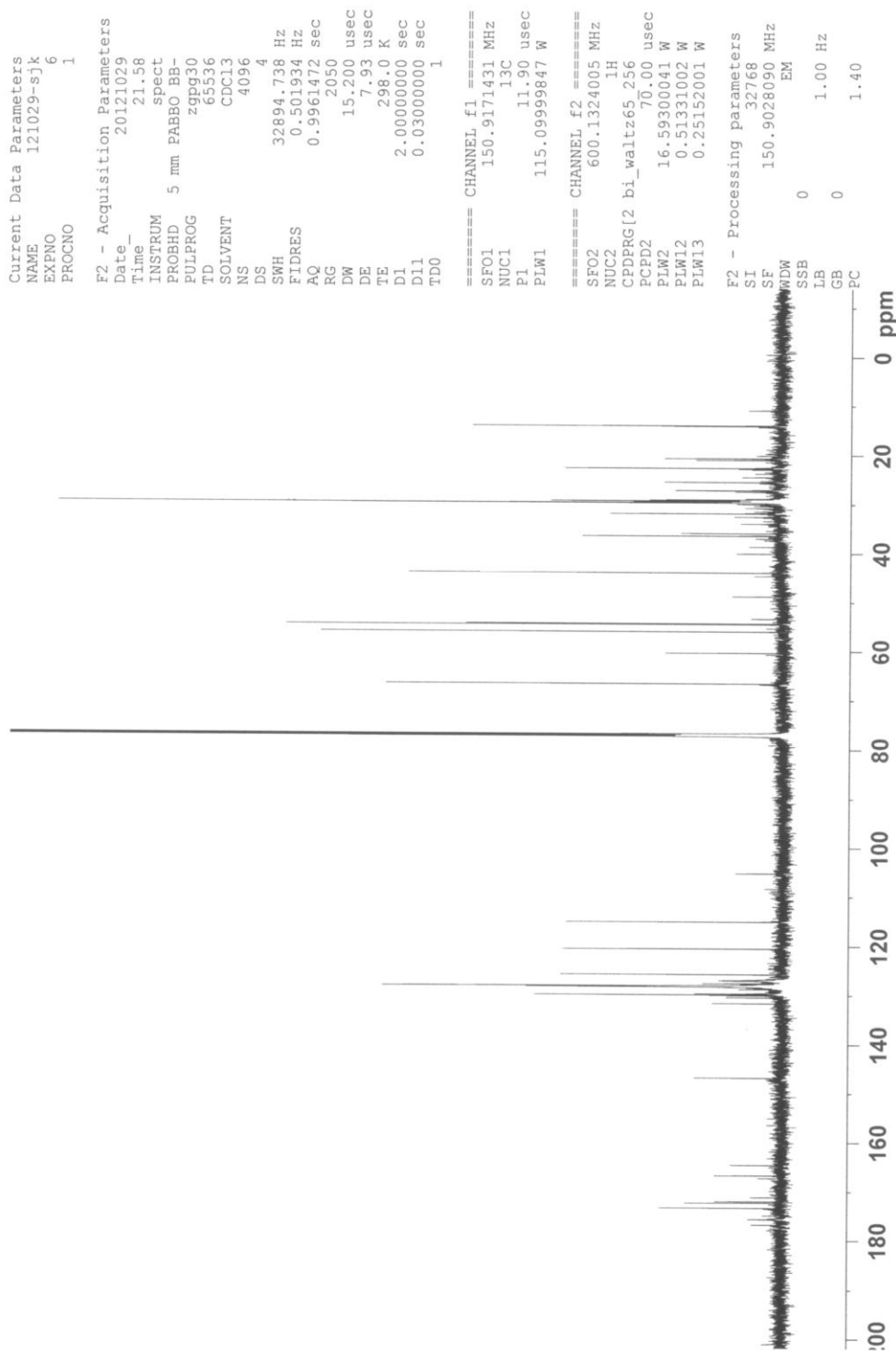
===== CHANNEL f1 =====
SFO1 300.1719511 MHz
NUC1 1H
P1 14.95 usec
PLW1 8.1999981 W

F2 - Processing parameters
SI 131072
SF 300.1700000 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00



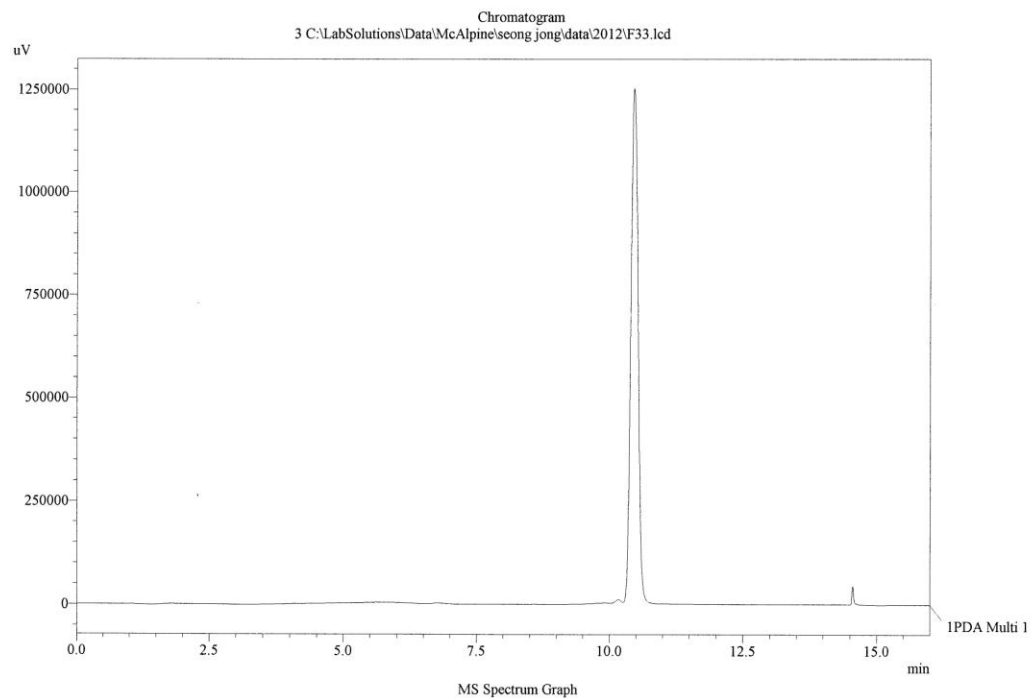
Cyclized compound(3) _ ^{13}C NMR

Supervisor Shelli McAlpine
Istst_SPPS_CYC13C
.3C{1H} CDC13 F:\ .sjk 15



Oxazoles contained macrocycle (4)_LCMS (ESI)

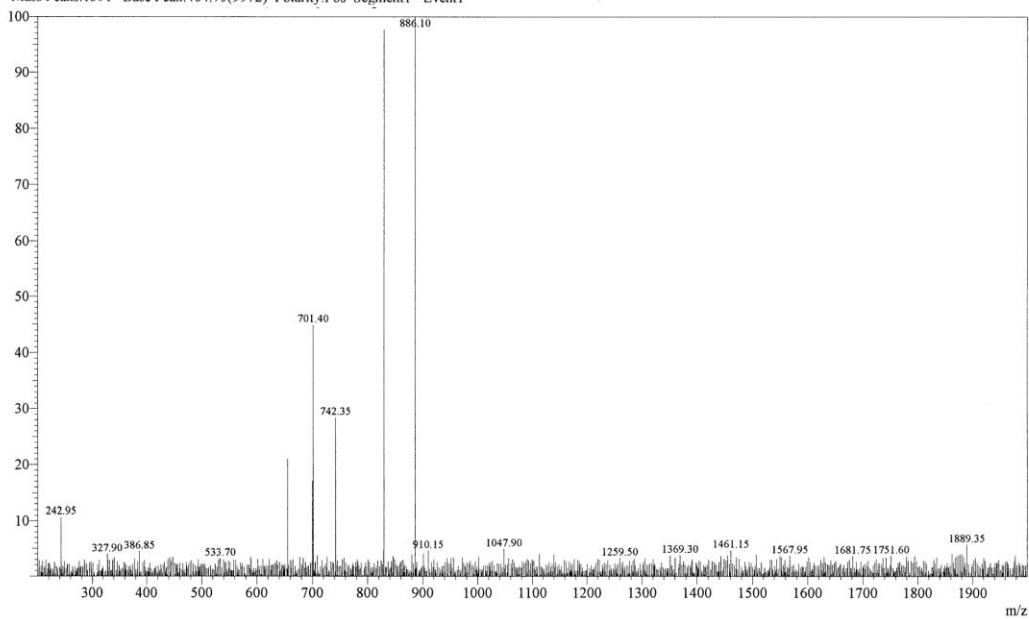
==== Shimadzu LCMSsolution Analysis Report ====



Ret. Time: 10.500(Scan#: 625)

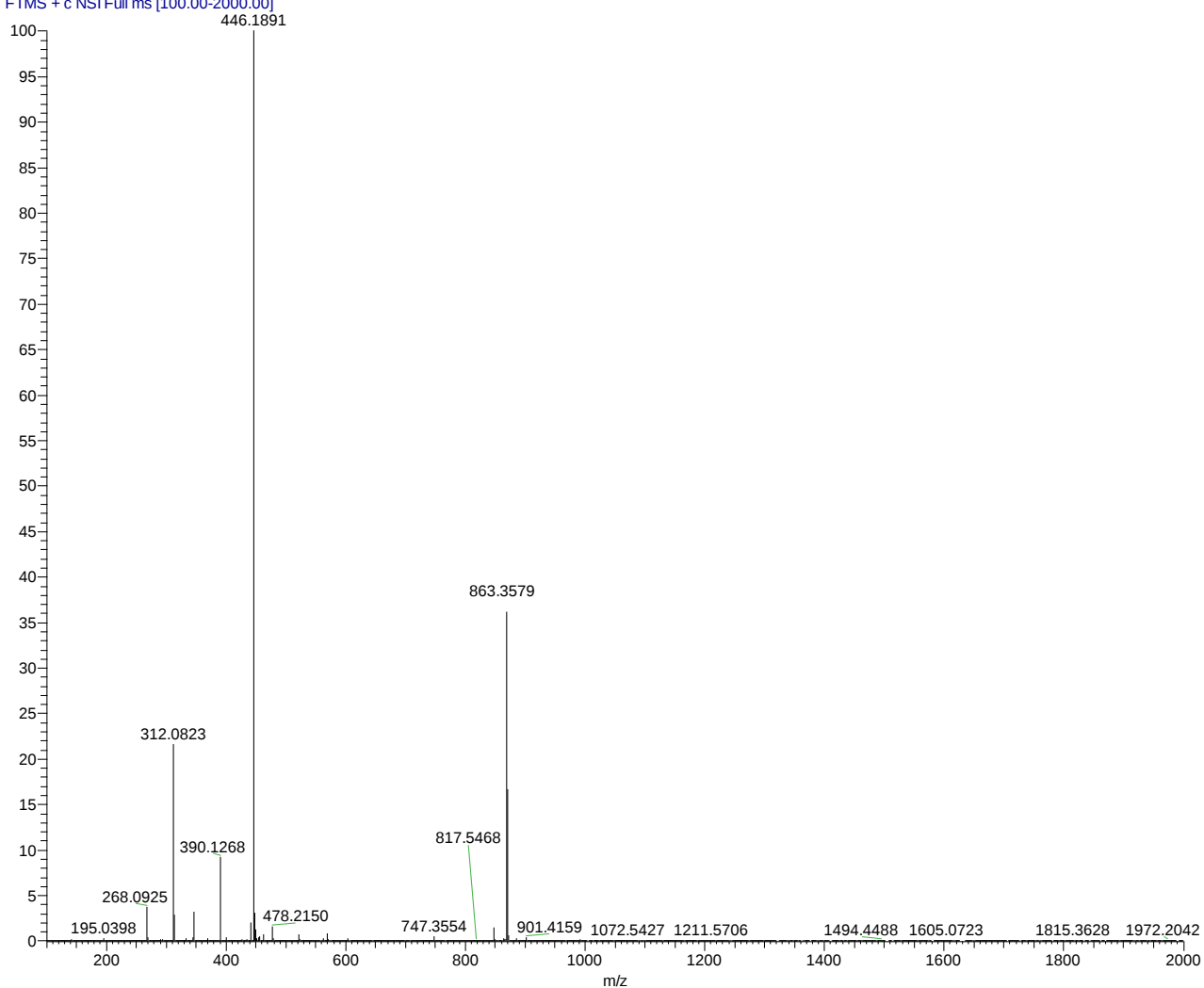
BG Mode: ?

Mass Peaks: 1391 Base Peak: 464.75(9972) Polarity: Pos Segment1 - Event1

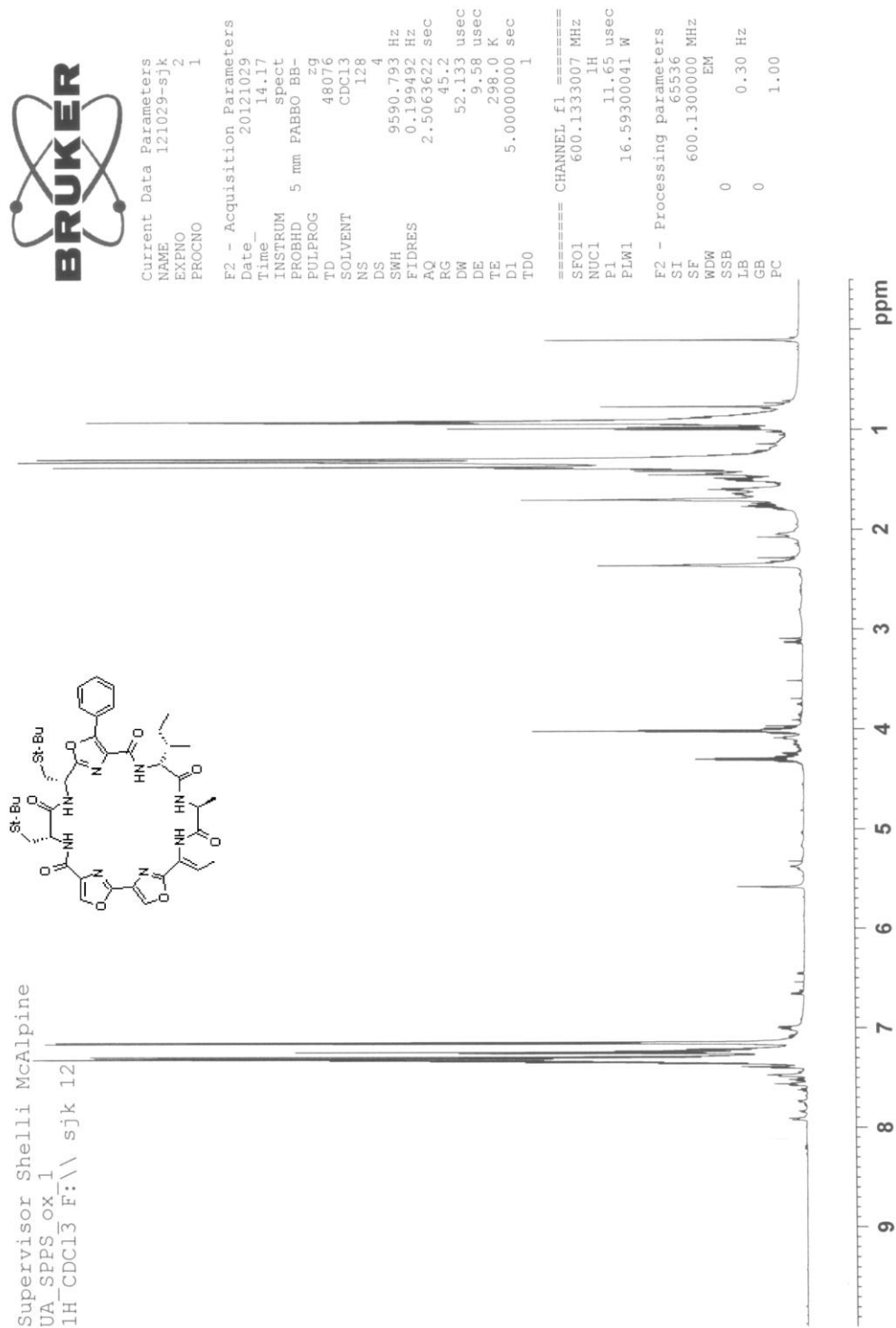


Oxazoles contained macrocycle (4) _ HRMS (ESI)

7_Pos_Full #1-34 RT: 0.01-0.48 AV: 34 NL: 5.13E8
T: FTMS + c NSI Full ms [100.00-2000.00]



Oxazoles contained macrocycle (4)₁H NMR



Oxazoles contained macrocycle (4)₁₃C NMR

Supervisor Shelli McAlpine
Istat SPSS oxazole
.3C{1H} CDCl3 F:\V\sjk 16

