

Supporting Information for

Total Syntheses of the Histone Deacetylase Inhibitors Largazole and 2-*epi*-Largazole: Application of *N*-Heterocyclic Carbene-Mediated Acylations in Complex Molecule Synthesis.

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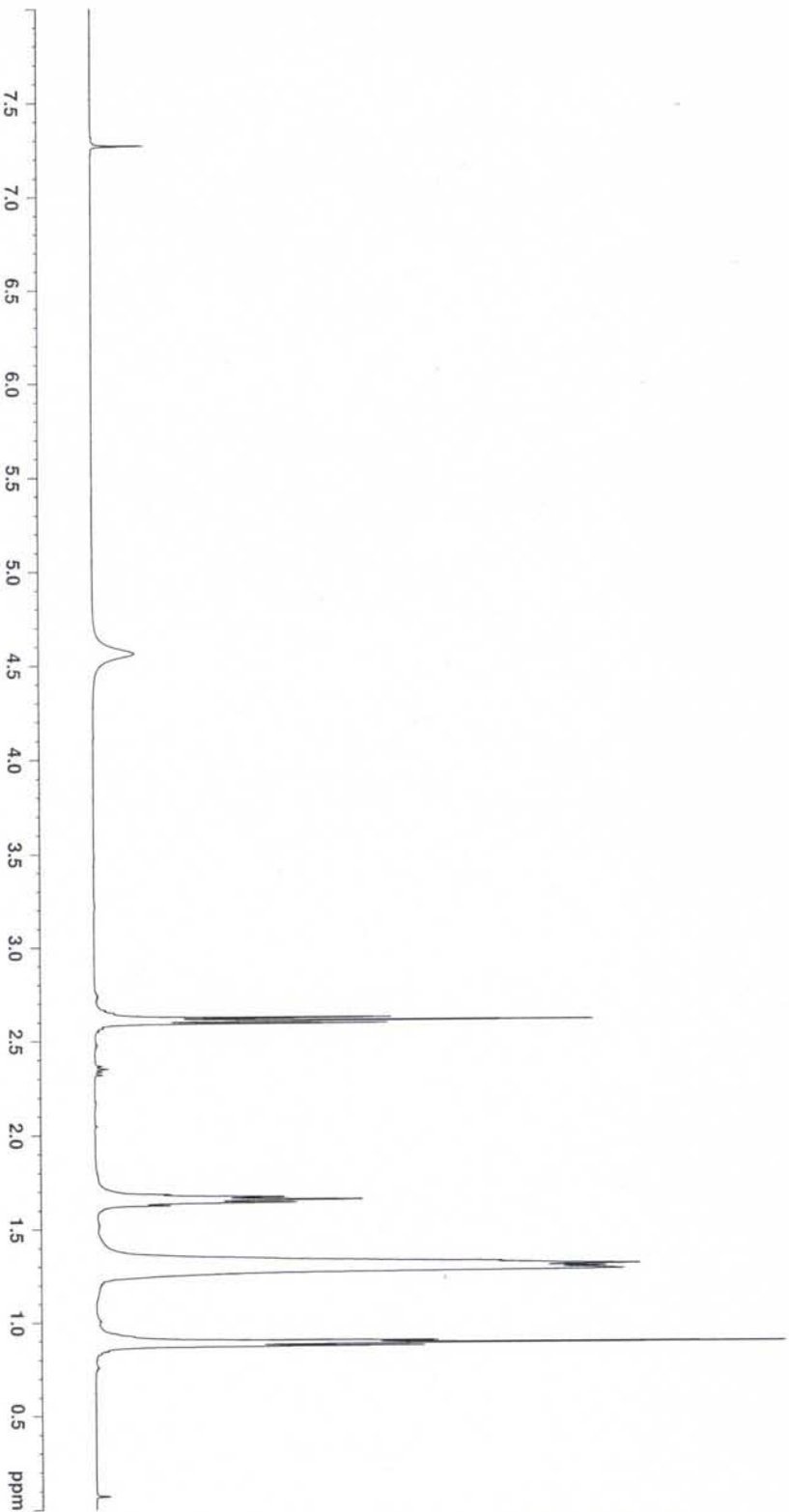
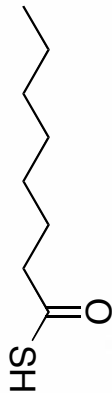
*forsyth@chemistry.ohio-state.edu

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12-22-2010

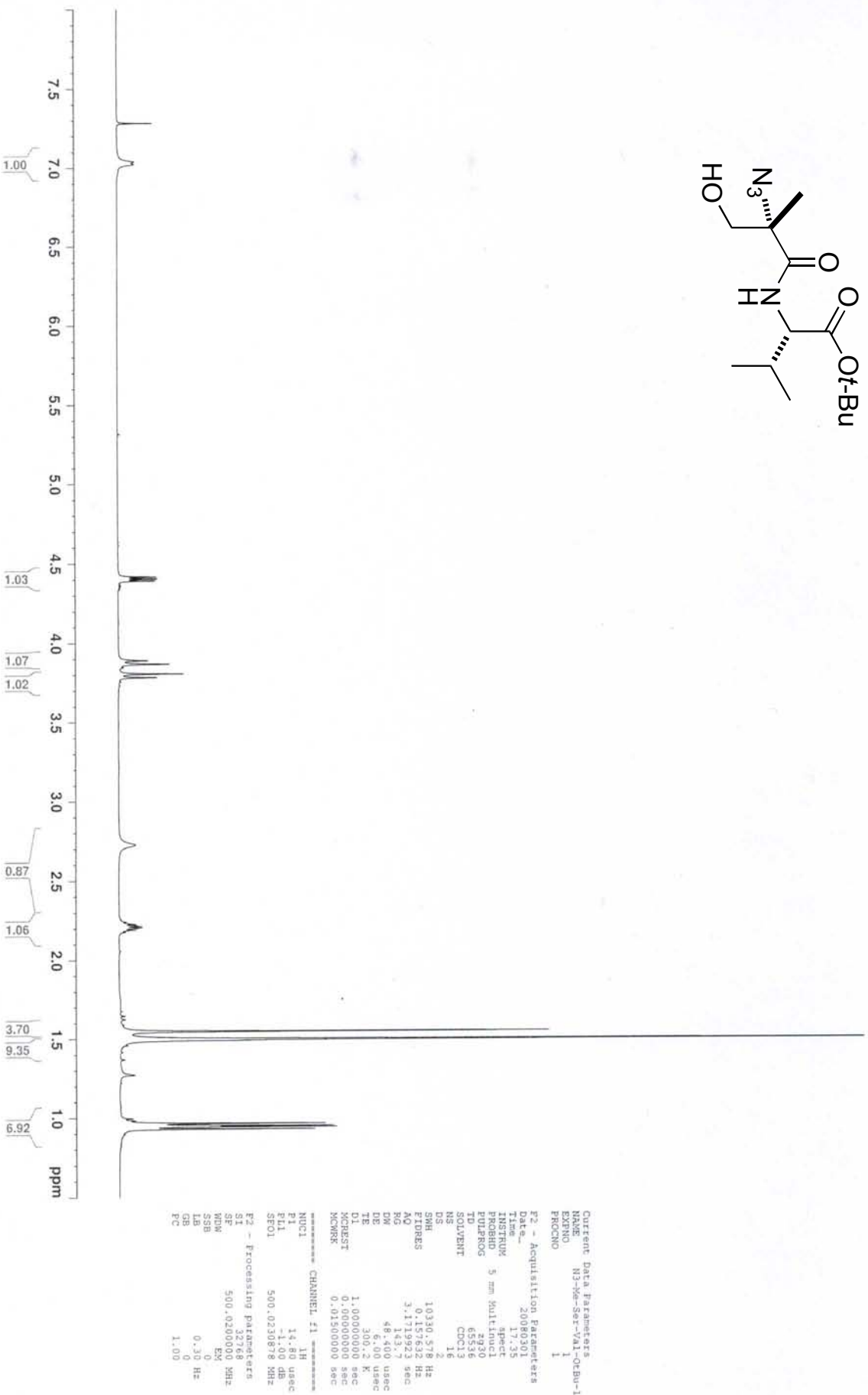
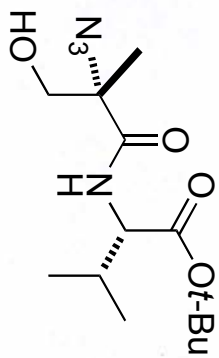
General Method for Synthetic Compounds

All air sensitive reactions were carried out under argon in oven-dried glassware using standard syringe, cannula and septa techniques. Unless otherwise noted, all reactions were carried out at room temperature (20-25 °C). Molecular sieves were activated by heating at 120 °C for 24 h under vacuum. Tetrahydrofuran (THF), methylene chloride (CH₂Cl₂) and toluene were purified with a pressurized alumina column-based solvent purification system. Acetonitrile (CH₃CN) and *N,N*-diisopropylethylamine (*i*-Pr₂NEt) were distilled from CaH₂ under nitrogen. Microwave assisted reactions were performed in sealed-tube mode on a CEM Discover S-class reactor. Dimethyl sulfoxide (DMSO) was dried over 3 Å molecular sieves. Deuterated chloroform (CDCl₃) was neutralized with anhydrous K₂CO₃. Other reagents were used as received from commercial sources. Flash column chromatography was performed using Silicycle SilicaFlash P60. Analytical TLC was performed with Silicycle glassed backed TLC extra hard layer 60 plates and Kieselgel silica gel 60 F_{254s} HPTLC plates visualized by fluorescence upon 254 nm irradiation and/or staining with anisaldehyde reagent (450 mL of 90% ethanol, 25 mL of sulfuric acid, 15 mL of acetic acid, and 25 mL of anisaldehyde) or PMA reagent (phosphomolybdic acid 25 g, cerium(IV) sulfate 7.5 g, H₂O 479 mL, H₂SO₄ 25 mL). Preparative TLC was performed using Kieselgel silica gel 60 F_{254s} HPTLC plates. The solvent combinations for flash column chromatography and TLC are described in volume ratios. ¹H NMR and ¹³C NMR spectra were obtained in CDCl₃ and referenced to the residual CHCl₃ resonances at 7.27 ppm (¹H) and 77.0 ppm (¹³C), or in CD₃OD and referenced to the residual CH₃OH resonances at 3.31 ppm (¹H) and 49.0 ppm (¹³C), using 400 MHz or 500 MHz NMR instruments. Optical rotations were obtained using a polarimeter at the sodium D line (589 nm) using a 3.5 i.d. × 100 mm cylindrical glass cell and were reported in concentration (*c* = g/100 mL) at 22 °C. IR spectra were obtained with an Ir spectrometer. High resolution mass spectrometric analyses were performed with an electrospray ionization-time-of-flight mass spectrometer.

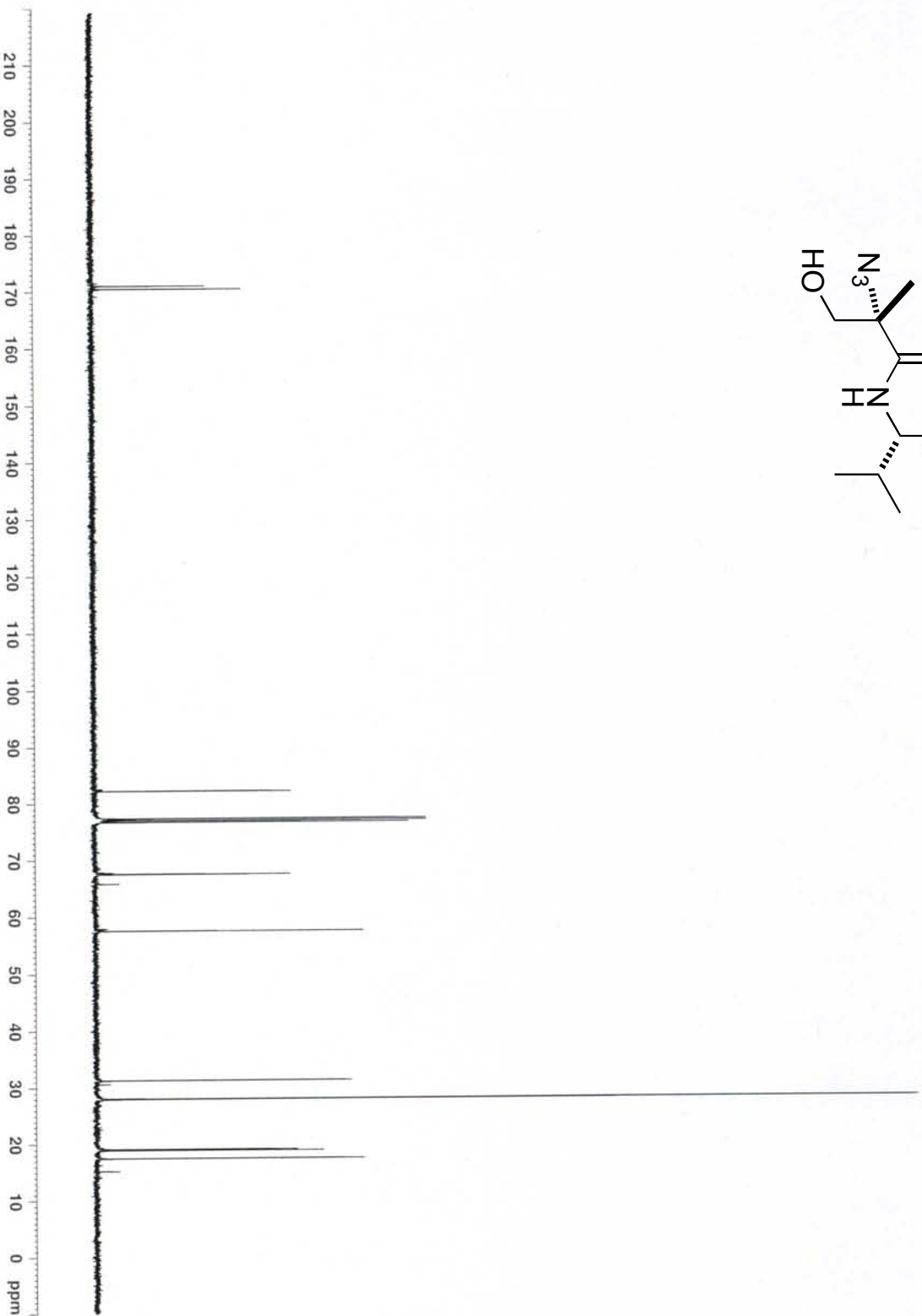
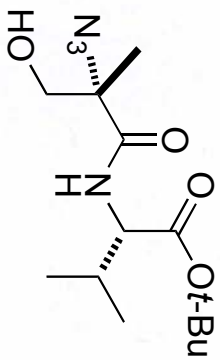


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 PC 40.0 usec
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 TR 298.2 K
 DI 1.000000 sec
 DE 0.010000 sec
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¹H NMR spectrum of thiooctanoic S-acid (500 MHz, CDCl₃)



¹H NMR spectrum for compound **15** (500 MHz, CDCl₃)



```

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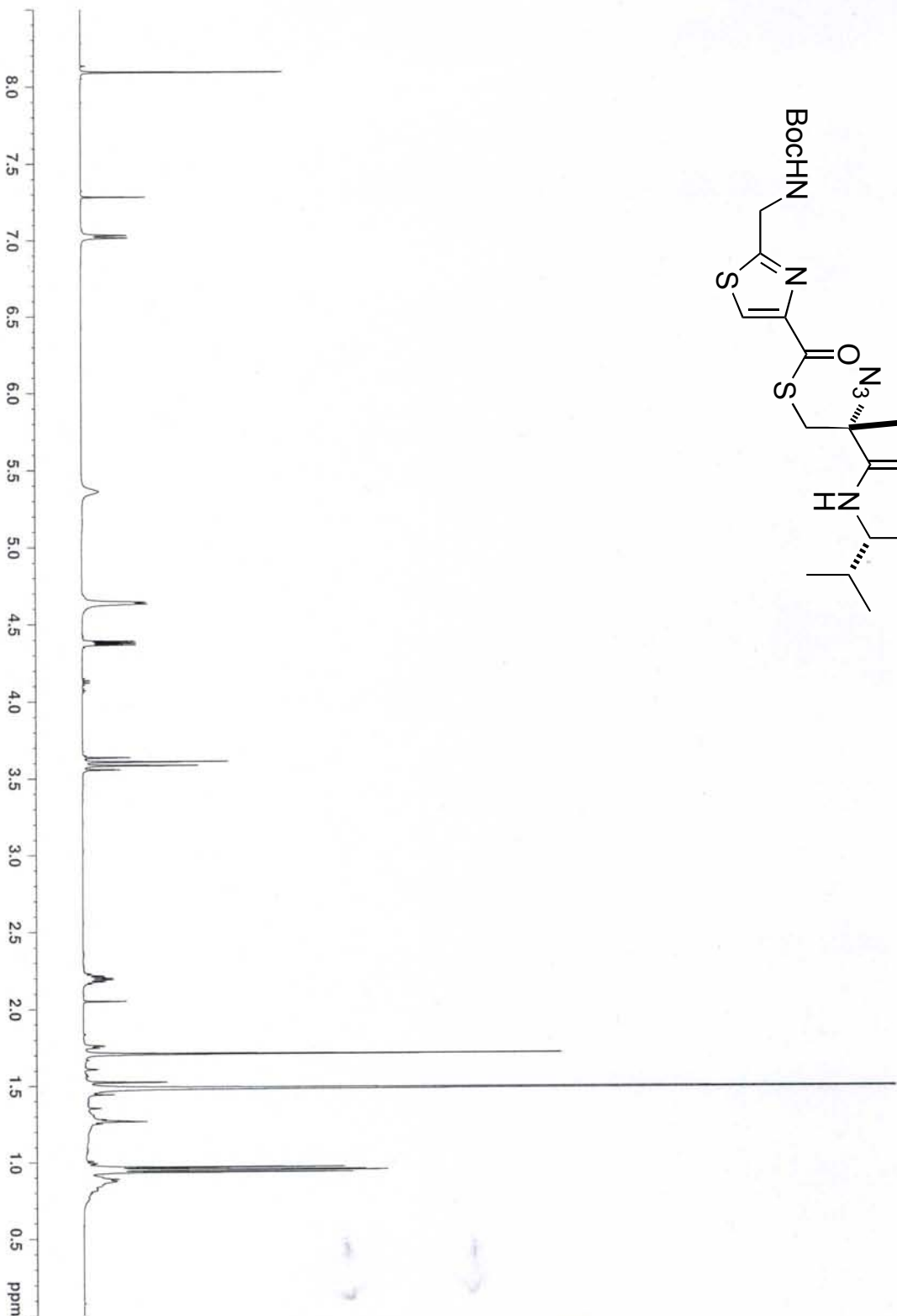
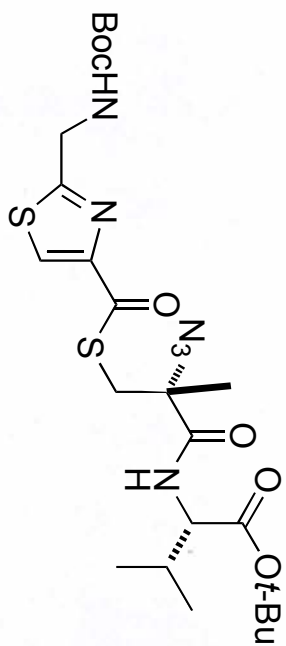
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^{13}C NMR spectrum for compound **15** (500 MHz, CDCl_3)



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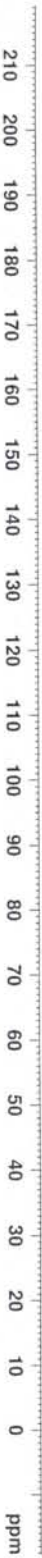
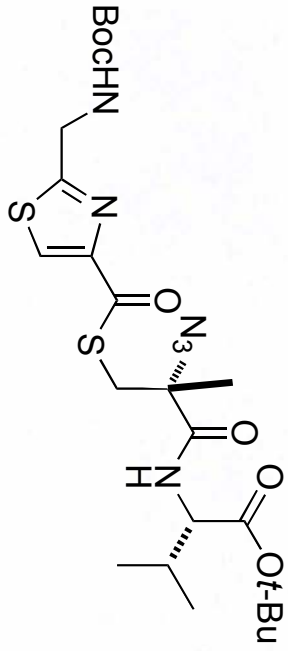
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EXPNO     1
PROCNO    1

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^1H NMR spectrum for compound **19** (500 MHz, CDCl_3)



^{13}C NMR spectrum for compound **19** (500 MHz, CDCl_3)

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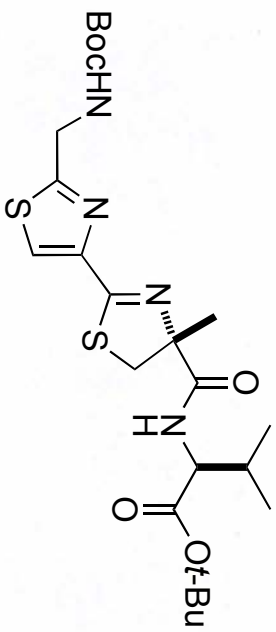
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DS           4
SWH          30039.522 Hz
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RG           1.032244 sec
RG           4096
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DL           0.030000000 sec
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P2           100.00 usec
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P12          18.60 dB
P13          22.50 dB
SFO2         500.0220001 MHz

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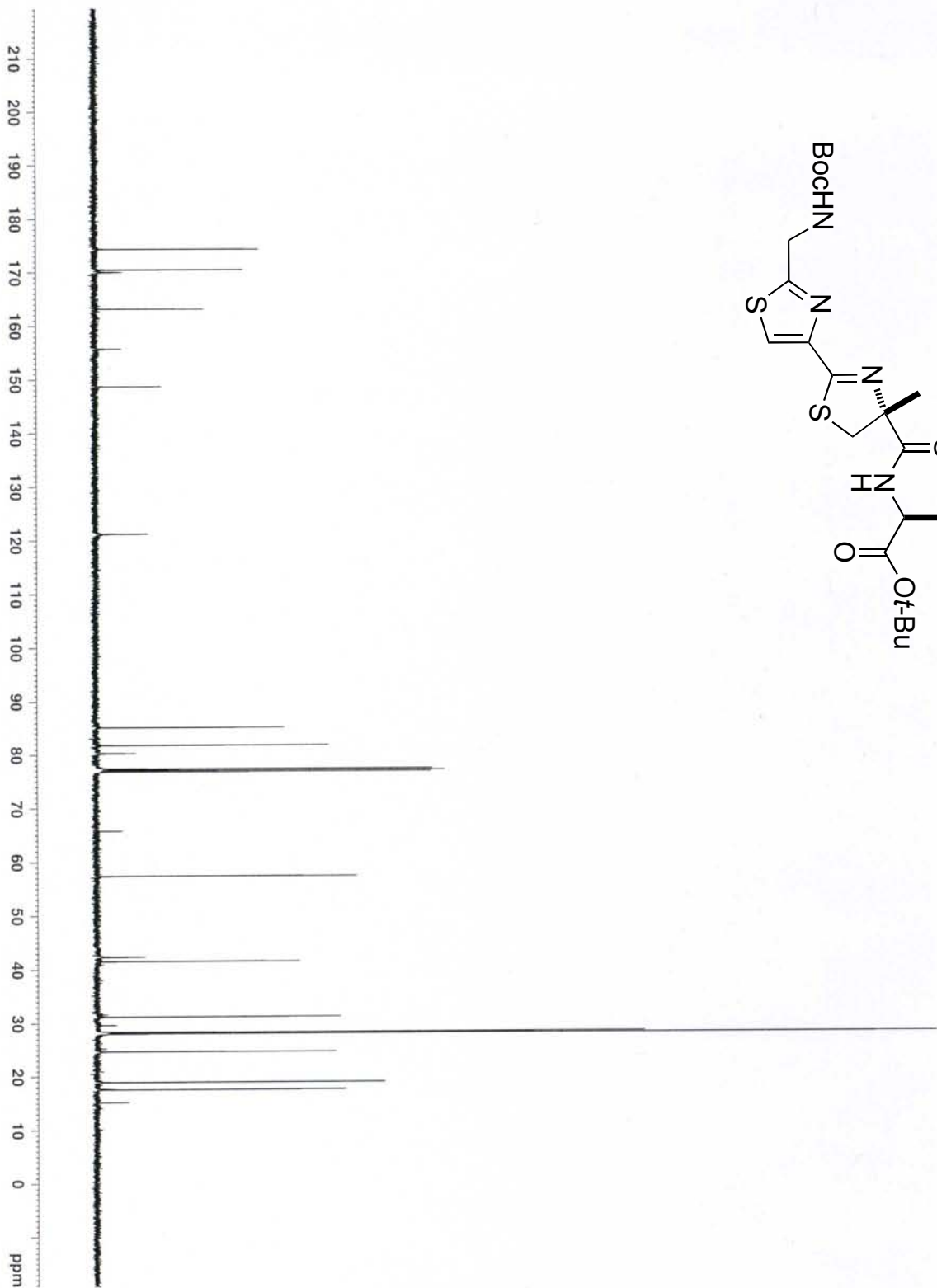
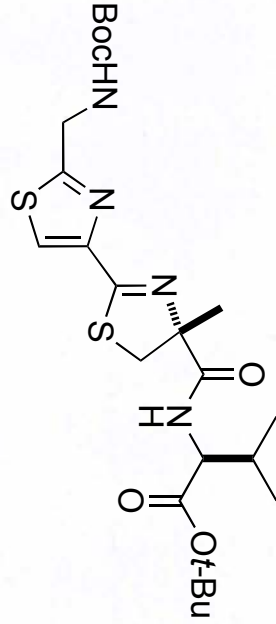



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SFO: 500.136
SF: 500.136188
WDW: EM
SSB: 0
GB: 0
EC: 0
RO: 0
DE: 0
TE: 300.2
FIDRES: 0.1416000
AQRES: 0.0002000
RG1: 1024
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RG3: 1024
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WDW: EM
SSB: 0
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EC: 0
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TE: 300.2
FIDRES: 0.1416000
AQRES: 0.0002000
RG1: 1024
RG2: 1024
RG3: 1024
SAMPLN: 65536
SAMPLF: 11.700000
SI: 32768
SF: 500.136188
WDW: EM
SSB: 0
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EC: 0
RO: 0
DE: 0
TE: 300.2
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SAMPLF: 11.700000

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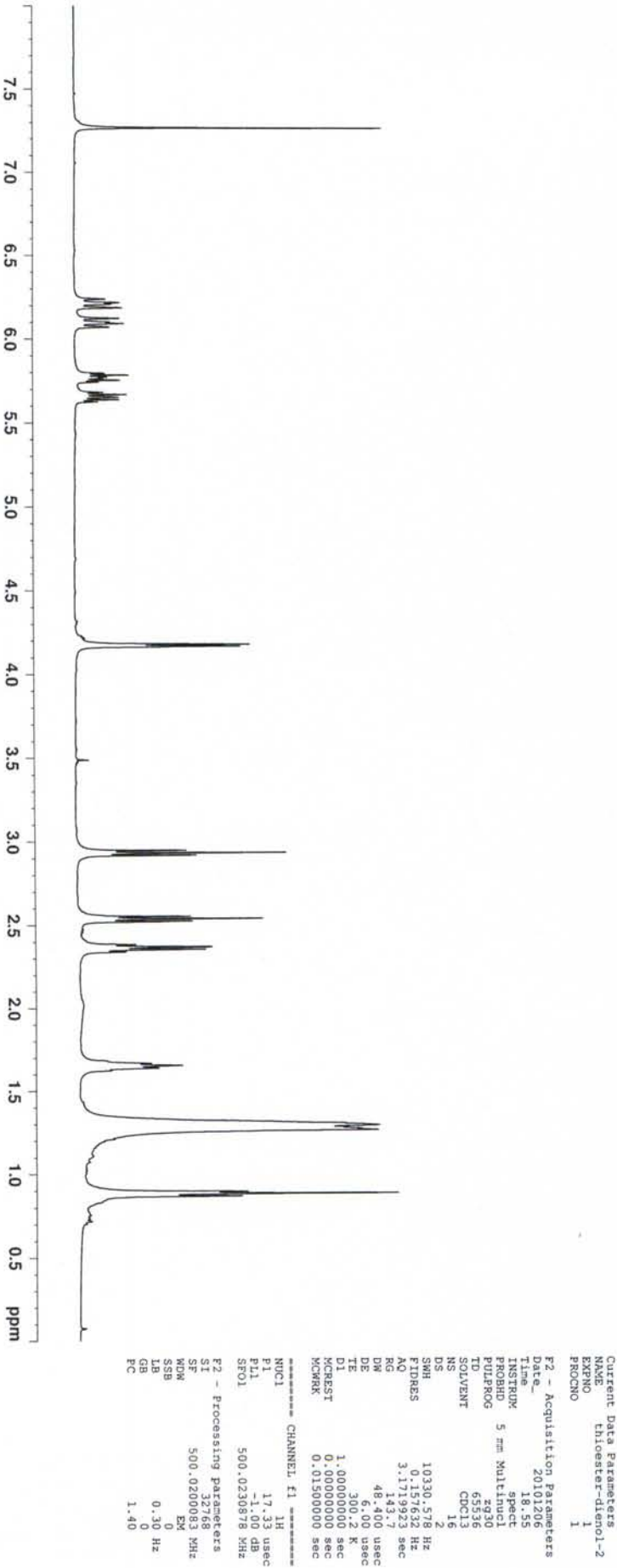
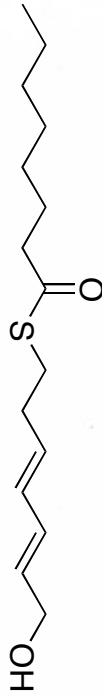
¹H NMR spectrum for compound 5 (500 MHz, CDCl₃)



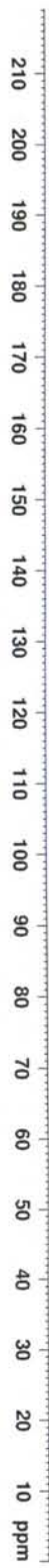
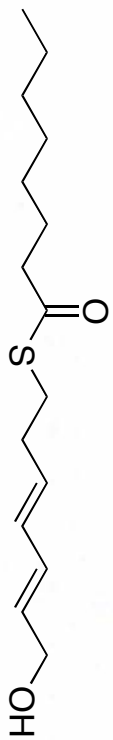
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P1       12.00
P2       12.00
P3       123.762500 MHz
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P3       123.762500 MHz
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PULPROG  zgpg30
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WDW      EM
SSB      0
GB       0
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NUC1     13C
P1       12.00
PC       218.00
===== Processing parameters =====
SI       32768
SF       125.762500 MHz
WDW      EM
SSB      0
GB       0
PC       218.00
=====
  
```

¹³C NMR spectrum for compound **5** (500 MHz, CDCl₃)



¹H NMR spectrum for compound **21** (500 MHz, CDCl₃)



Current Data Parameters
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 EXPNO 1
 PROCNO 1

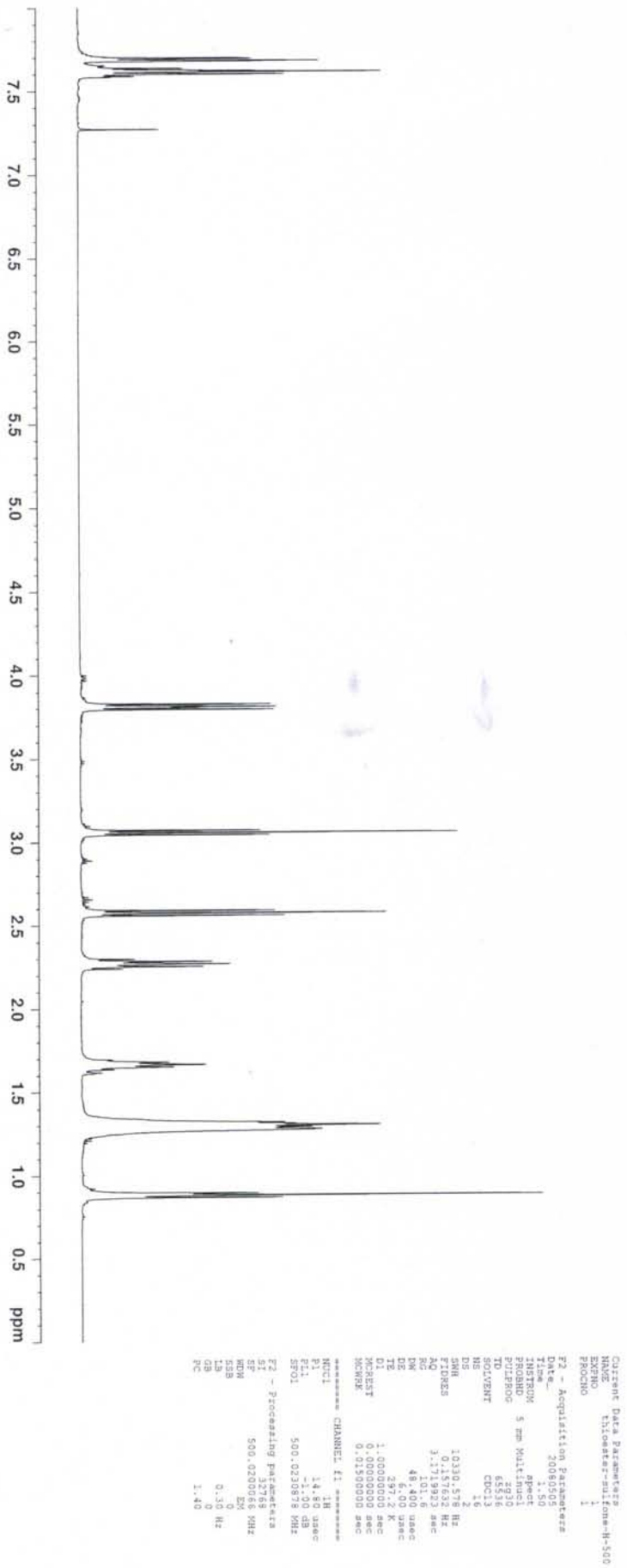
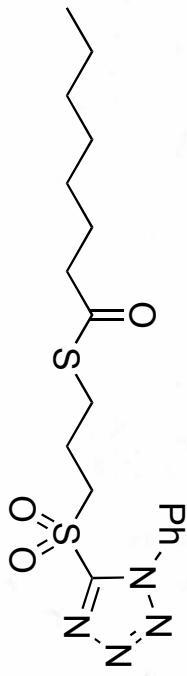
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 SOLVENT CDCl3
 NS 573
 DS 4
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 FIDRES 0.458222 Hz
 AQ 1.0912244 sec
 RG 3251
 DW 16.650 usec
 DE 12.400 usec
 TE 300.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
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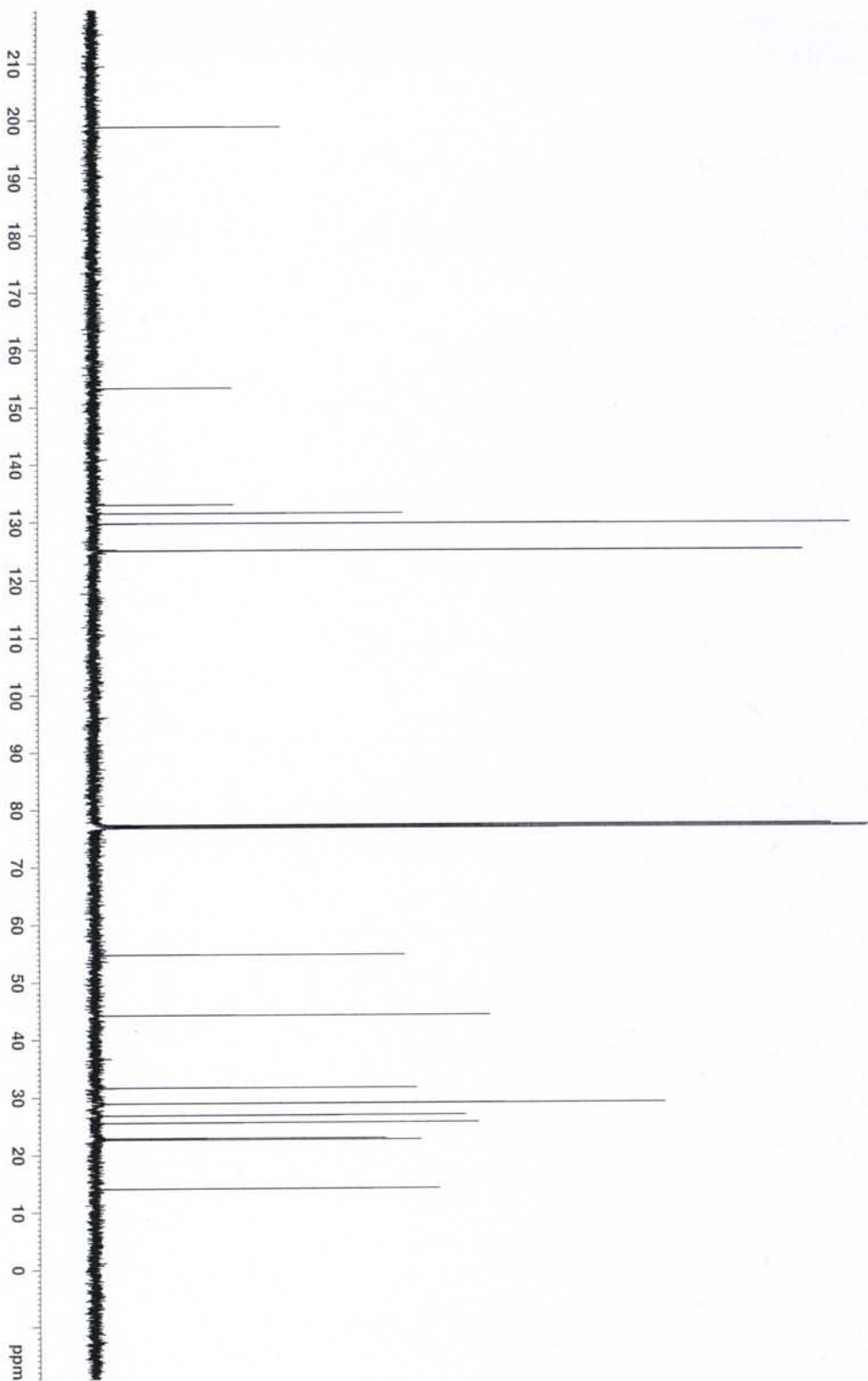
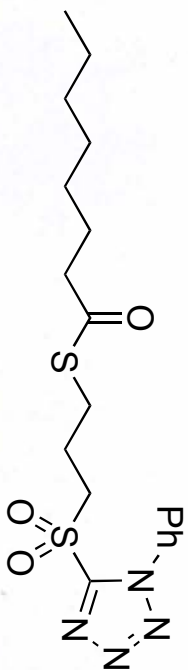
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 PL13 22.50 dB
 SFO2 500.0220001 MHz

F2 - Processing parameters
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 SF 125.7301290 MHz
 MDW EX 0
 SSB 0
 LB 1.00 Hz
 GB 0
 FC 1.40

¹³C NMR spectrum for compound 21 (500 MHz, CDCl₃)



¹H NMR spectrum for compound **24** (500 MHz, CDCl₃)



^{13}C NMR spectrum for compound **24** (500 MHz, CDCl_3)

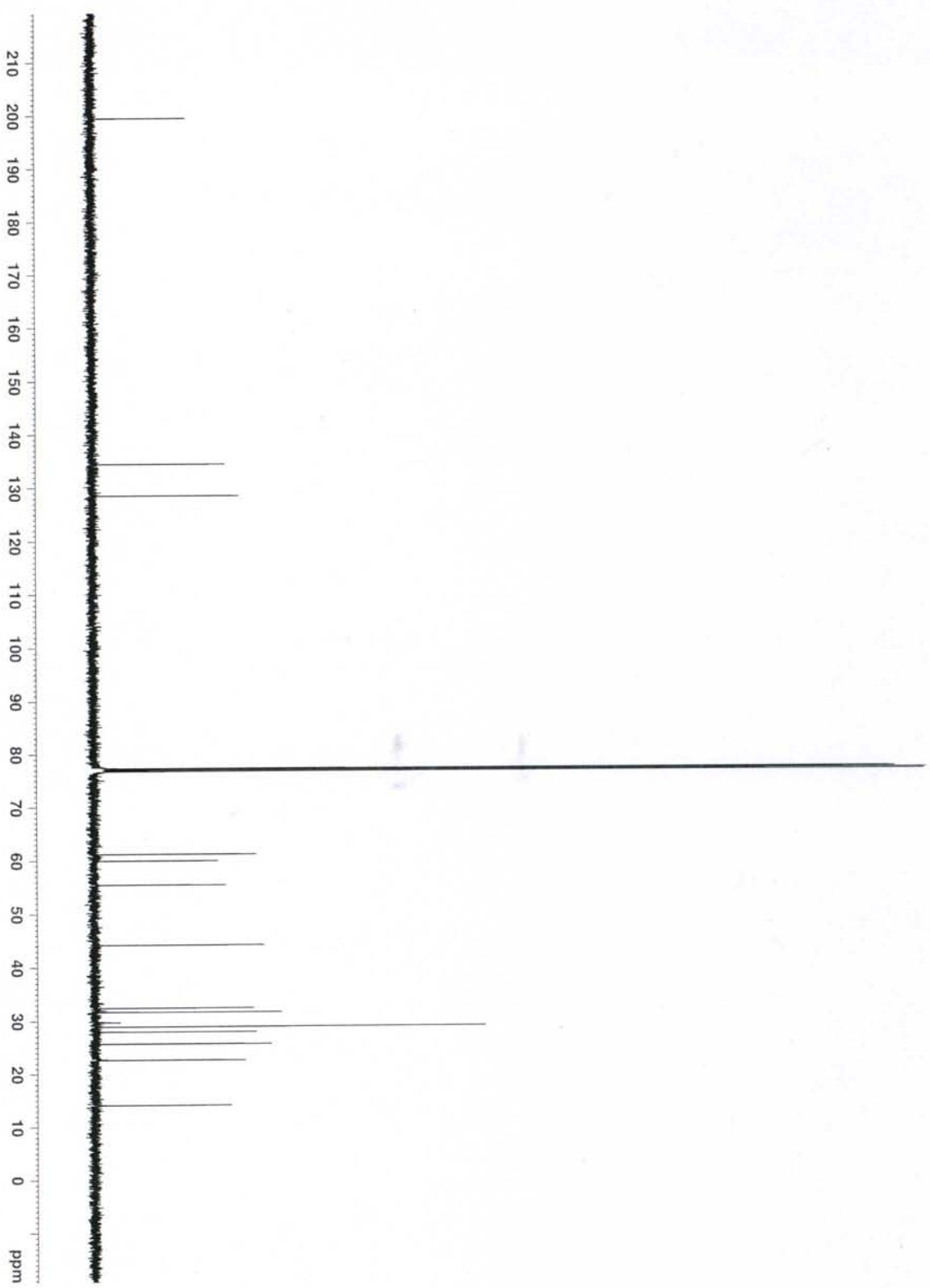
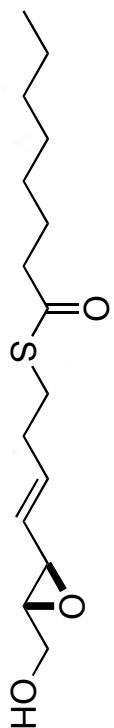
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SOLVENT  DMS-d6
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DS        4
SWH       6058.834 Hz
FWHM      0.6322 Hz
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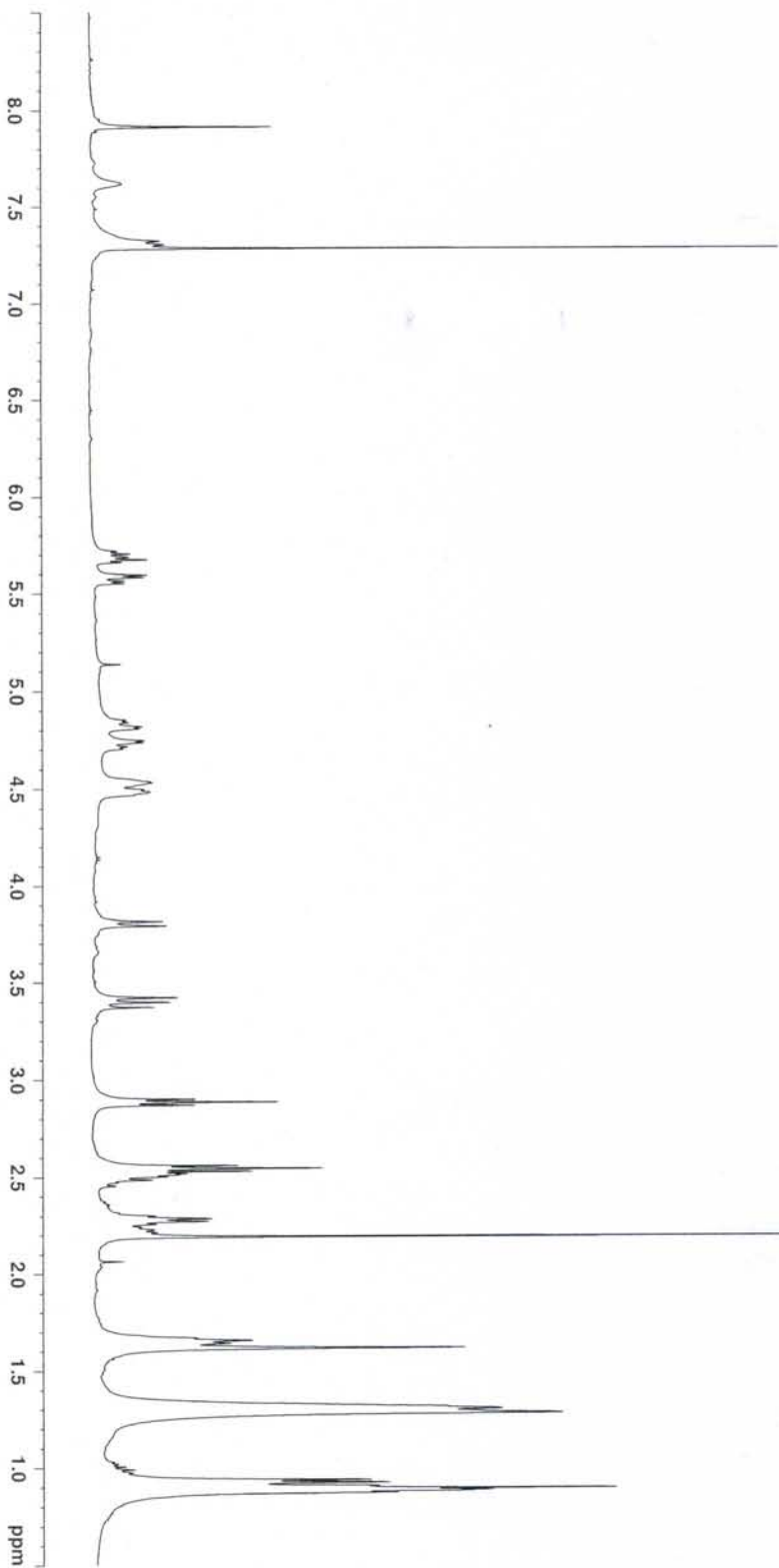
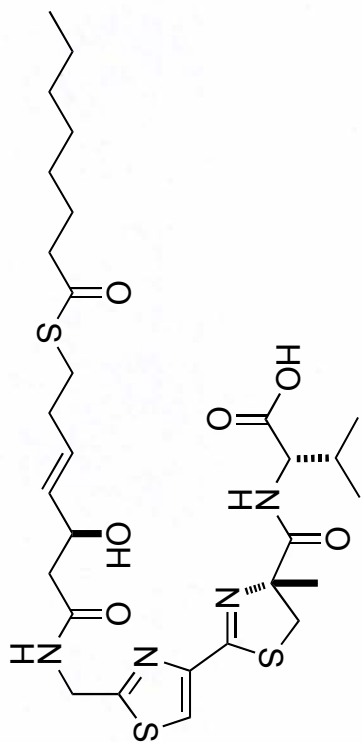
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PL13       22.50 dB
SFO2      500.0218001 MHz

F2 - Processing parameters
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SOLVENT   DMS
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DS         1.40
  
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 TD 65536
 SFO 299.13
 CHANID 13C
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¹³C NMR spectrum for compound **22** (500 MHz, CDCl₃)



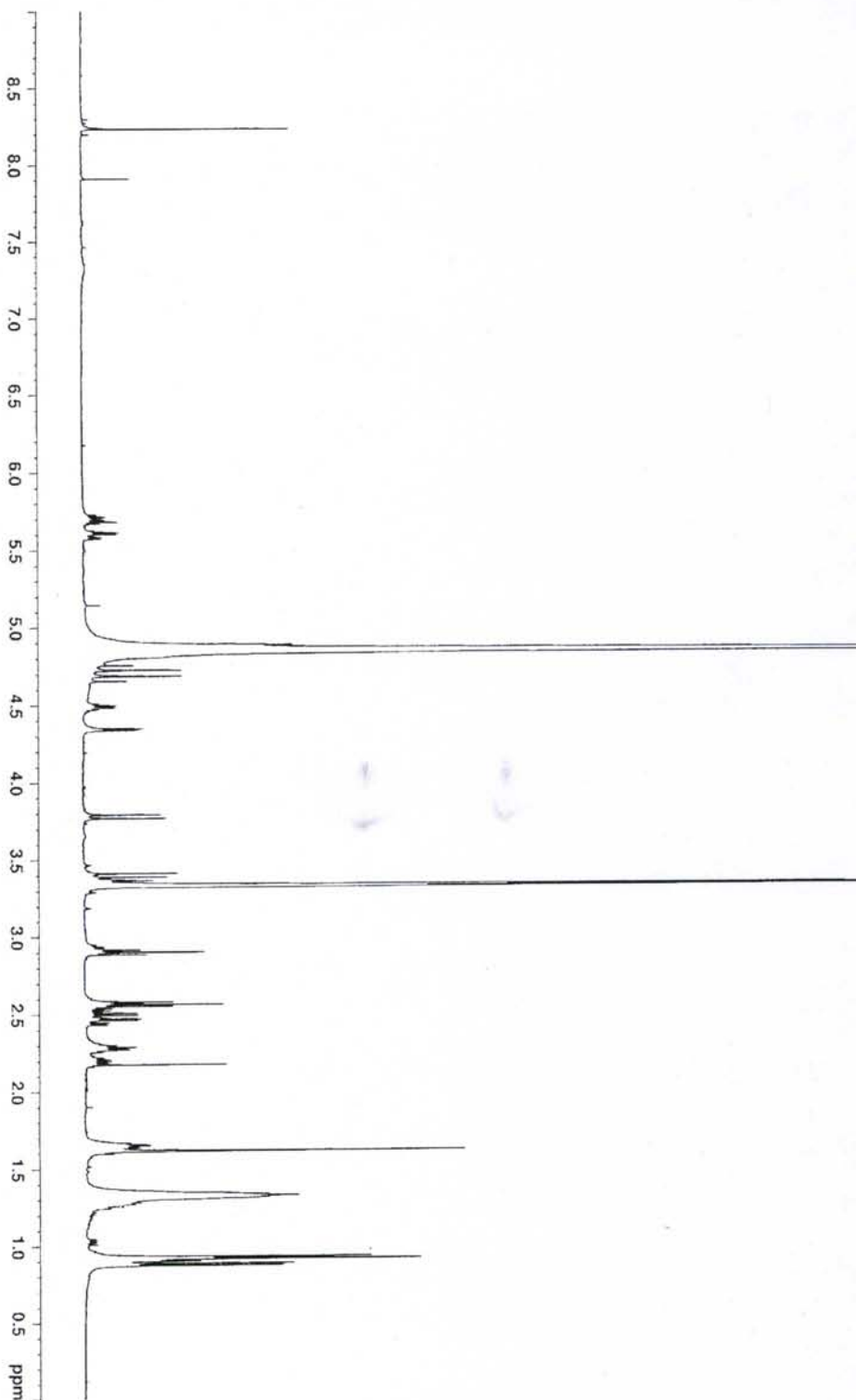
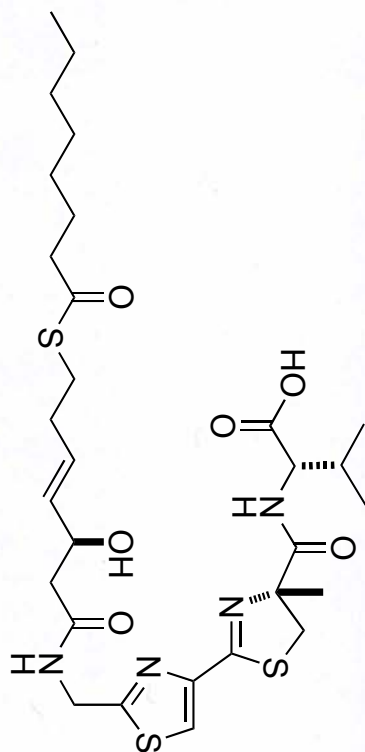
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 RG 362
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 ACQRES 0.01500000 sec
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 PL 0.00 dB
 SFO1 500.0230978 MHz

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¹H NMR spectrum for compound 2 (500 MHz, CDCl₃)

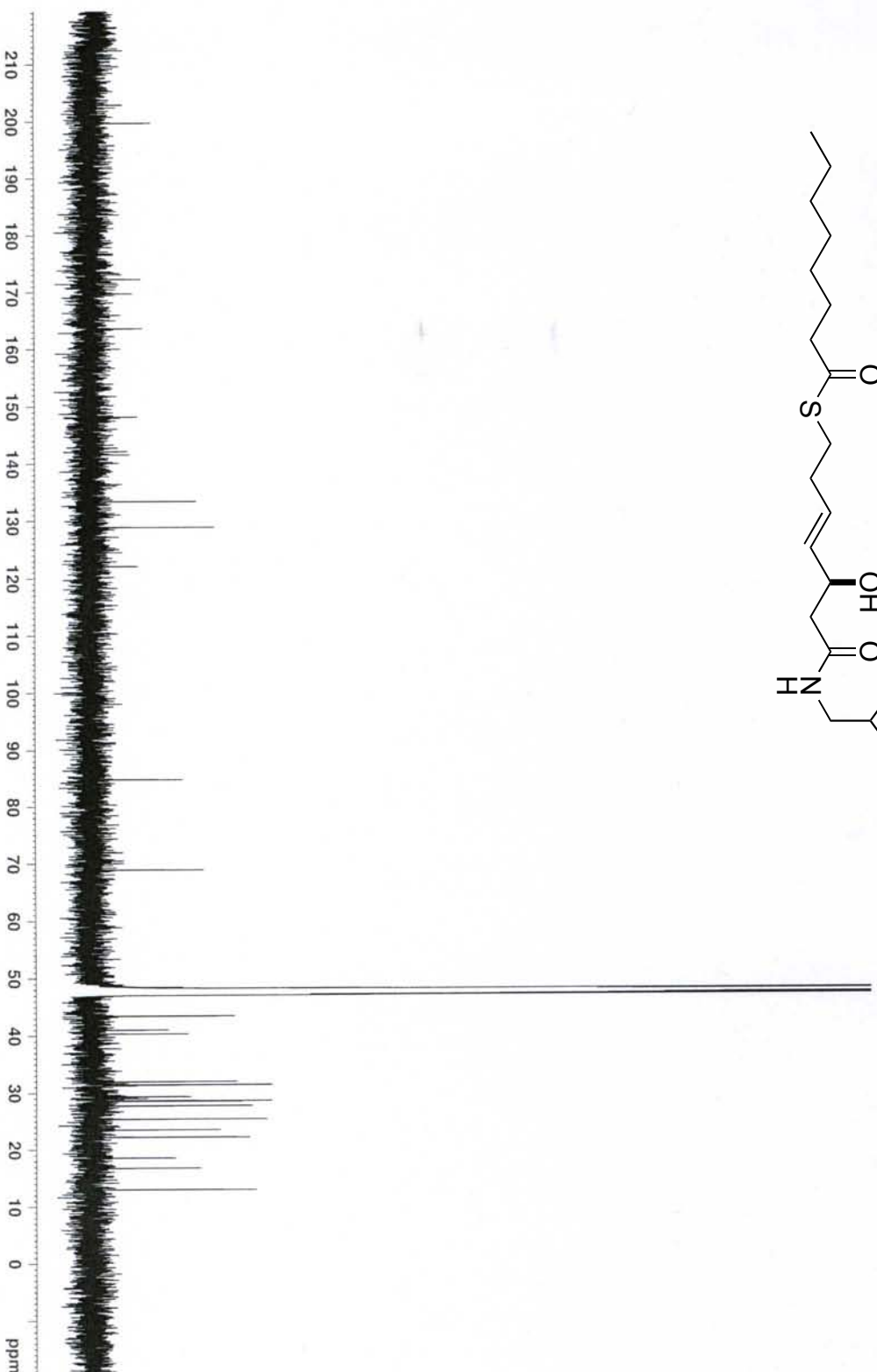
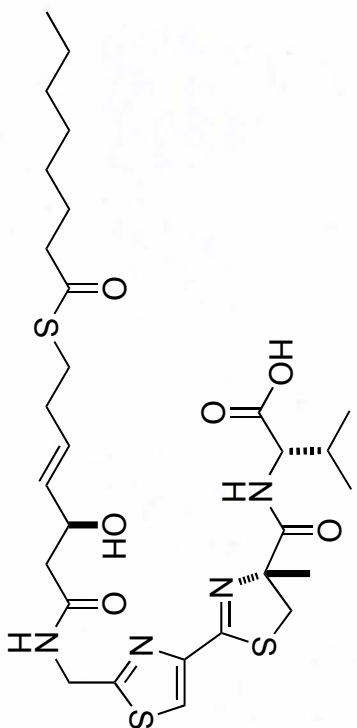


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SI  32768
SF  499.999
SOLVENT  Me2O
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DS  2
SWH  10352.074 Hz
FIDRES  0.177632 Hz
AQ  2.119205 sec
RG  400.0000
AC  48.400000
DE  6.000000
TE  300.2 K
WVFREQ  1.000000000 MHz
WWTXST  0.000000000 sec
WOMXK  0.015000000 sec
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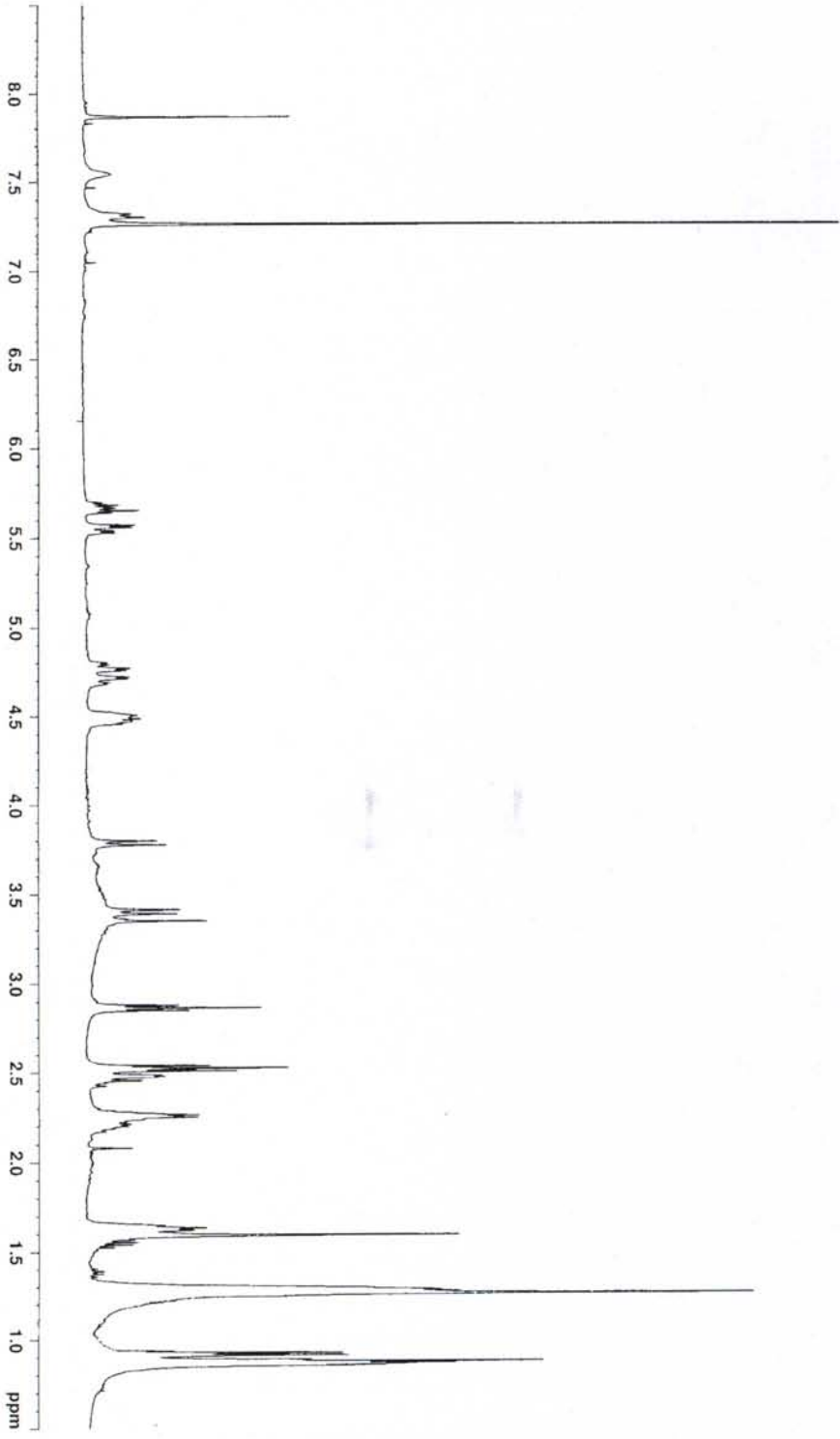
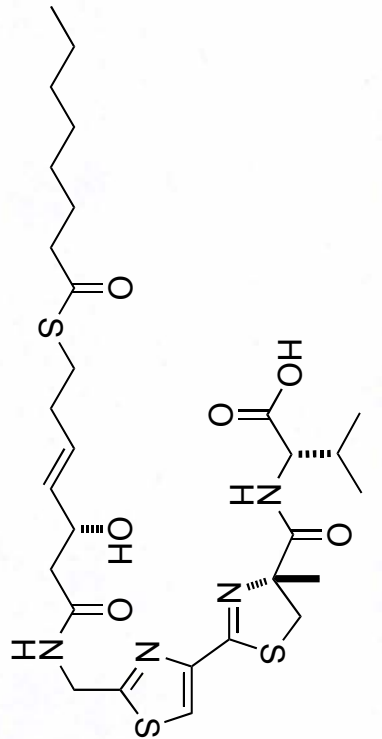
¹H NMR spectrum for compound 2 (500 MHz, CD₃OD)



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d113      0.00000000 sec
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PL1        0.00 dB
SFO1      125.760380 MHz
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NUC2       13C
P2         100.000 usec
PL2        -1.00 dB
SFO2      590.0220001 MHz
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P3         125.760380 MHz
PL3        0.00 dB
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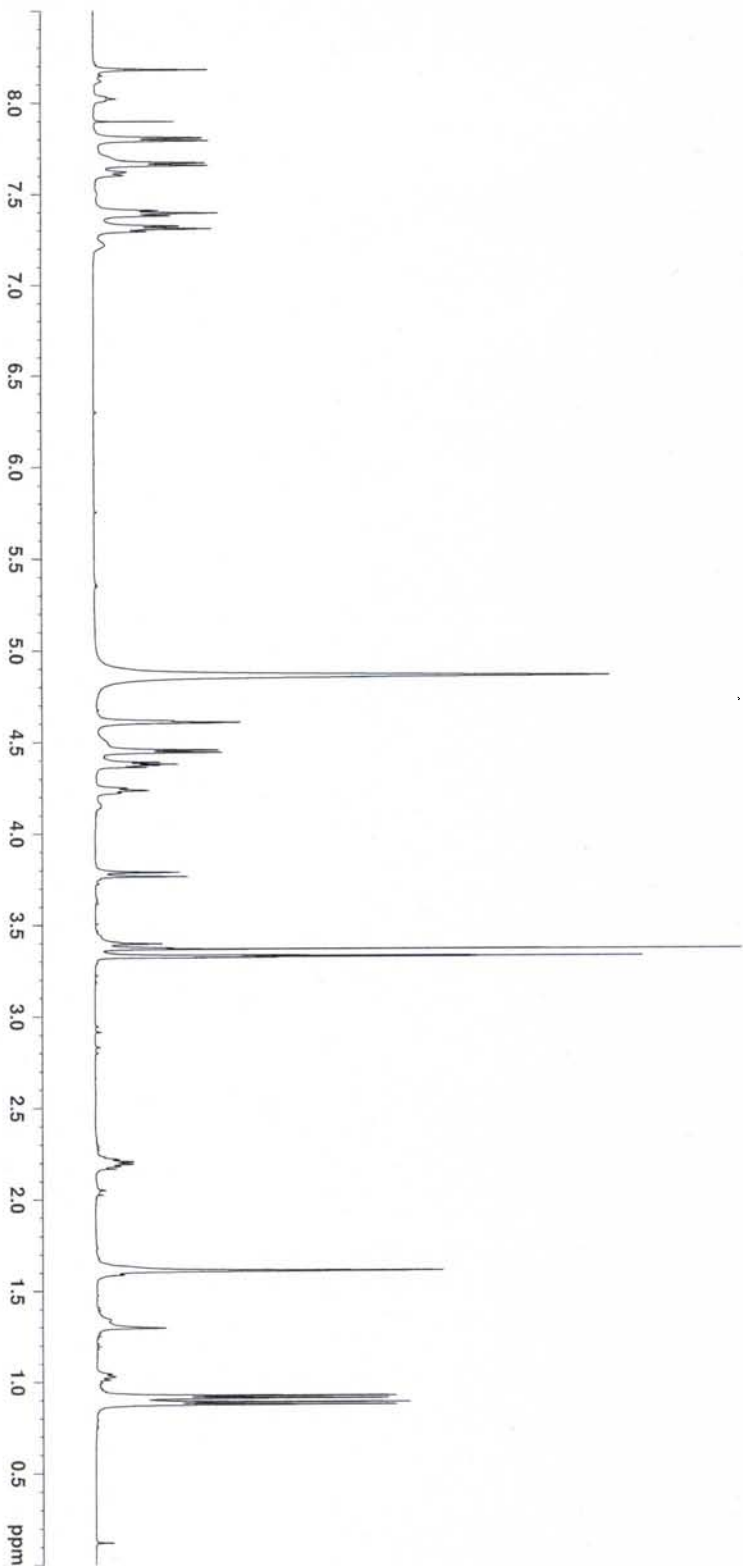
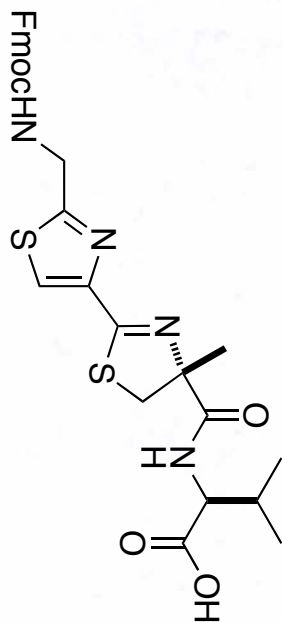
¹³C NMR spectrum for compound 2 (500 MHz, CD₃OD)



```

Current Data Parameters
NAME: 17-epi-2
EXPNO: 1
PROCNO: 1
F2 - Acquisition Parameters
Date_ 20040418
Time 20.48
INSTRUM spect
PROBHD 5mm
PULPROG zgpg30
TD 65536
SFO 500.136188
WDW EM
SSB 0
RG 320.0
AQ 0.187500
FIDRES 0.001278 Hz
AQRES 0.001278 Hz
NUC1 13C
NUC2 13C
PC 1.00
===== CHANNEL f1 =====
NUC1 13C
P1 14.40 usec
PL1 -1.00 dB
F1 500.136188 MHz
SFO 500.136188 MHz
===== Processing parameters =====
SI 32768
SF 500.136188 MHz
WDW EM
SSB 0
RG 320.0
AQ 0.187500
PC 1.00
  
```

¹H NMR spectrum for compound 17-*epi*-2 (500 MHz, CDCl₃)

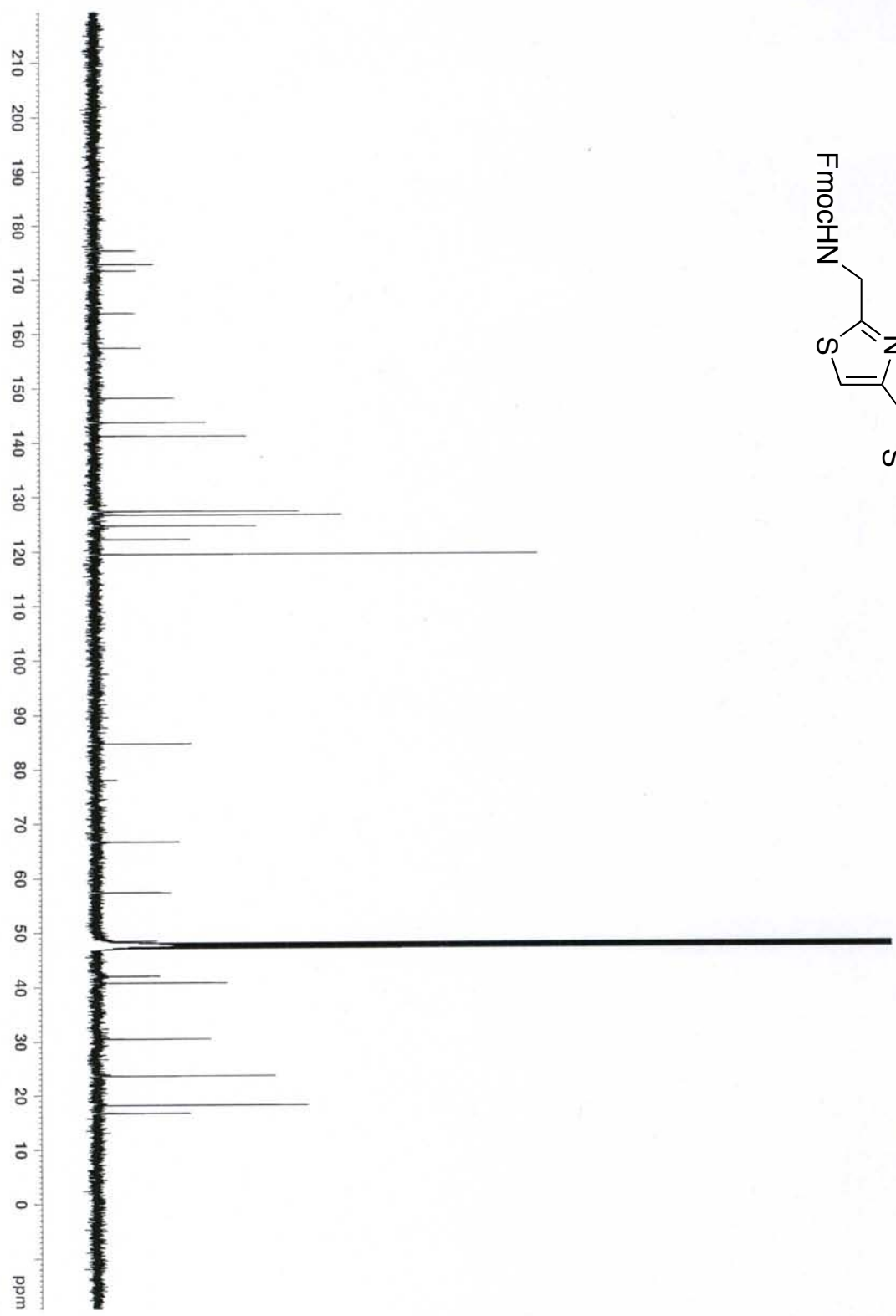
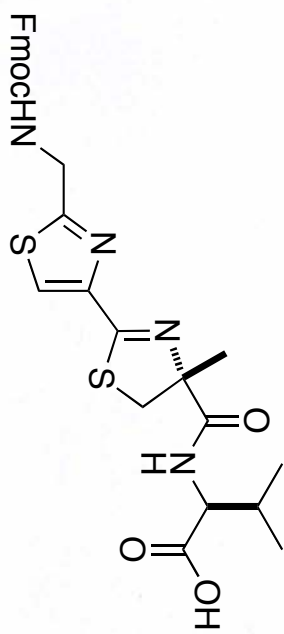


```

Compound 30a: 3-oxo-2-((S)-2-hydroxypropanoylamino)ethyl-1-methyl-1H-thiazole-4-carboxamide
NAME: 30a
EXPNO: 1
PROCNO: 1
F2 - Acquisition Parameters
Date_UTC: 20111220
Time: 20.12
INSTRUM: spect
PROBHD: 5 mm BBIKQNP1
PULPROG: zgpg30
TD: 65536
AQ: 0.020000000
RG: 327.500
AQ: 0.020000000
SOLVENT: H2O
NUC1: 13C
NUC2: 13C
NUC3: 13C
NUC4: 13C
NUC5: 13C
NUC6: 13C
PC: 1.000000000
TE: 300.2 K
FIDRES: 0.151562 Hz
AQRES: 0.000000000
SFO: 125.761 MHz
SF: 125.761 MHz
WDW: EM
SSB: 0
GB: 0
PC: 1.00
===== CHANNEL f1 =====
NUC1: 13C
P1: 14.00
PL1: 0 dB
F1: 125.761 MHz
SFO1: 125.761 MHz
===== CHANNEL f2 =====
NUC1: 13C
P1: 14.00
PL1: 0 dB
F1: 125.761 MHz
SFO1: 125.761 MHz
F2 - Processing parameters
SI: 327.500
SF: 500.1363000 MHz
WDW: EM
SSB: 0
GB: 0
PC: 1.00

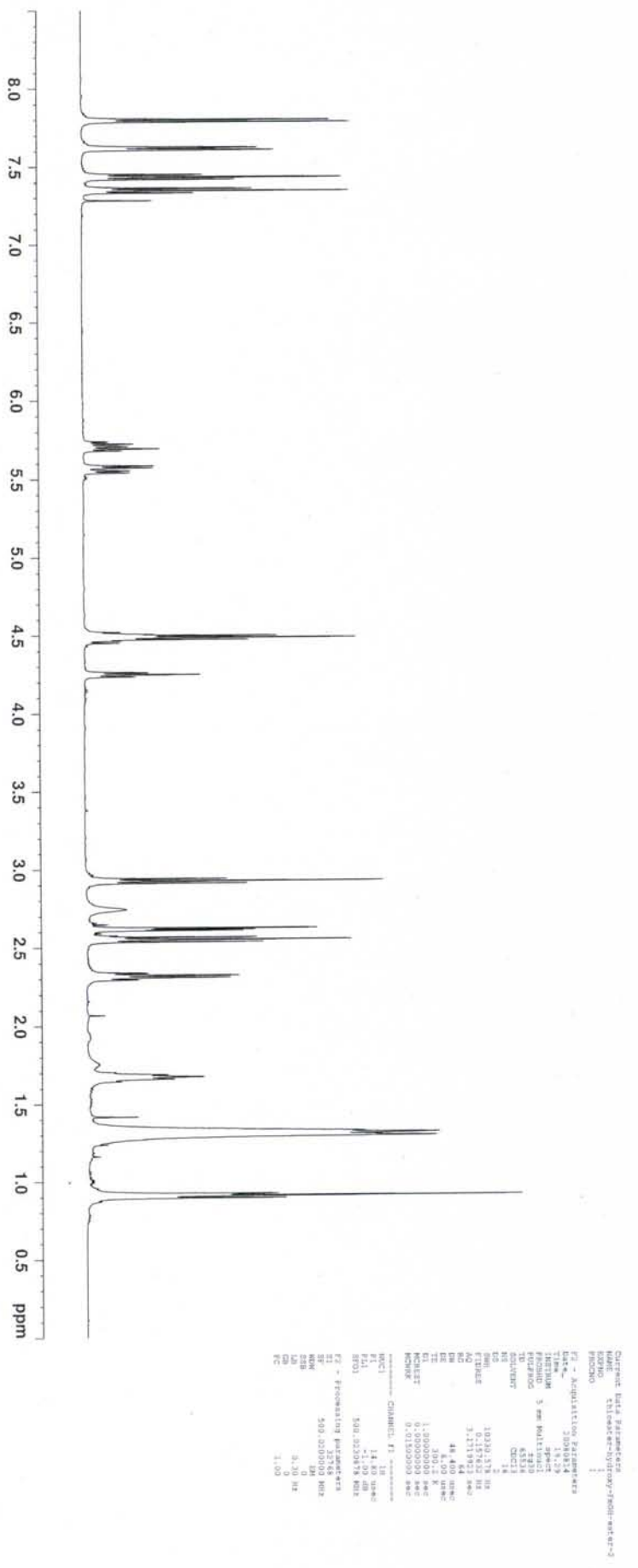
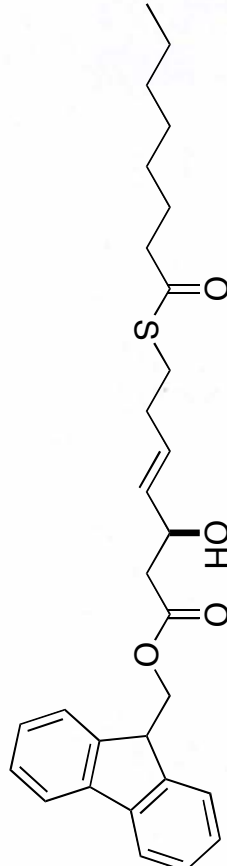
```

¹H NMR spectrum for compound **30** (500 MHz, CDCl₃)

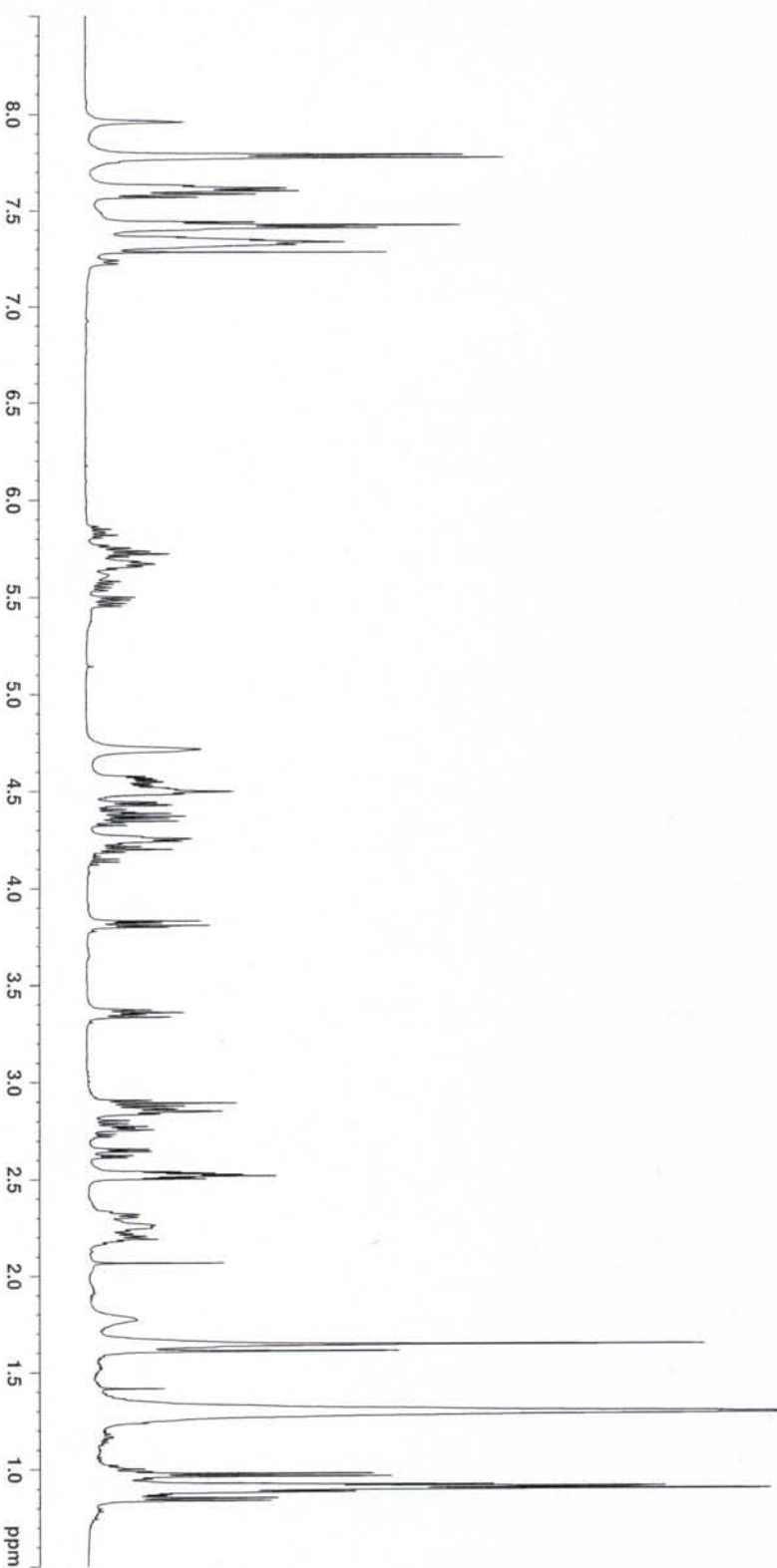
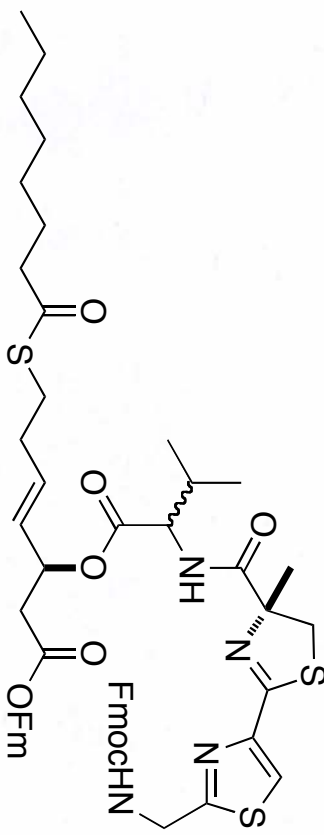


Output: Data Parameters
 Name: FmocMethylthiazolone-1-1
 P1: Acquisition Parameters
 Date_ : 2018
 Time : 12:50:00
 Instrument: spect
 Processor: 402316
 F2: 125.762175 MHz
 Solvent: CDCl3
 DE: 90.000000 Hz
 TE: 300.2 K
 F1: 500.136260 MHz
 AQ: 1.00000000 sec
 FT: 32768
 SFO: 125.762175 MHz
 PC: 14.000000 sec
 F2 - Processing parameters
 Date_ : 2018
 Time : 12:50:00
 Instrument: spect
 Processor: 402316
 F2: 125.762175 MHz
 Solvent: CDCl3
 DE: 90.000000 Hz
 TE: 300.2 K
 F1: 500.136260 MHz
 AQ: 1.00000000 sec
 FT: 32768
 SFO: 125.762175 MHz
 PC: 14.000000 sec

¹³C NMR spectrum for compound 30 (500 MHz, CDCl₃)



¹H NMR spectrum for compound **31** (500 MHz, CDCl₃)



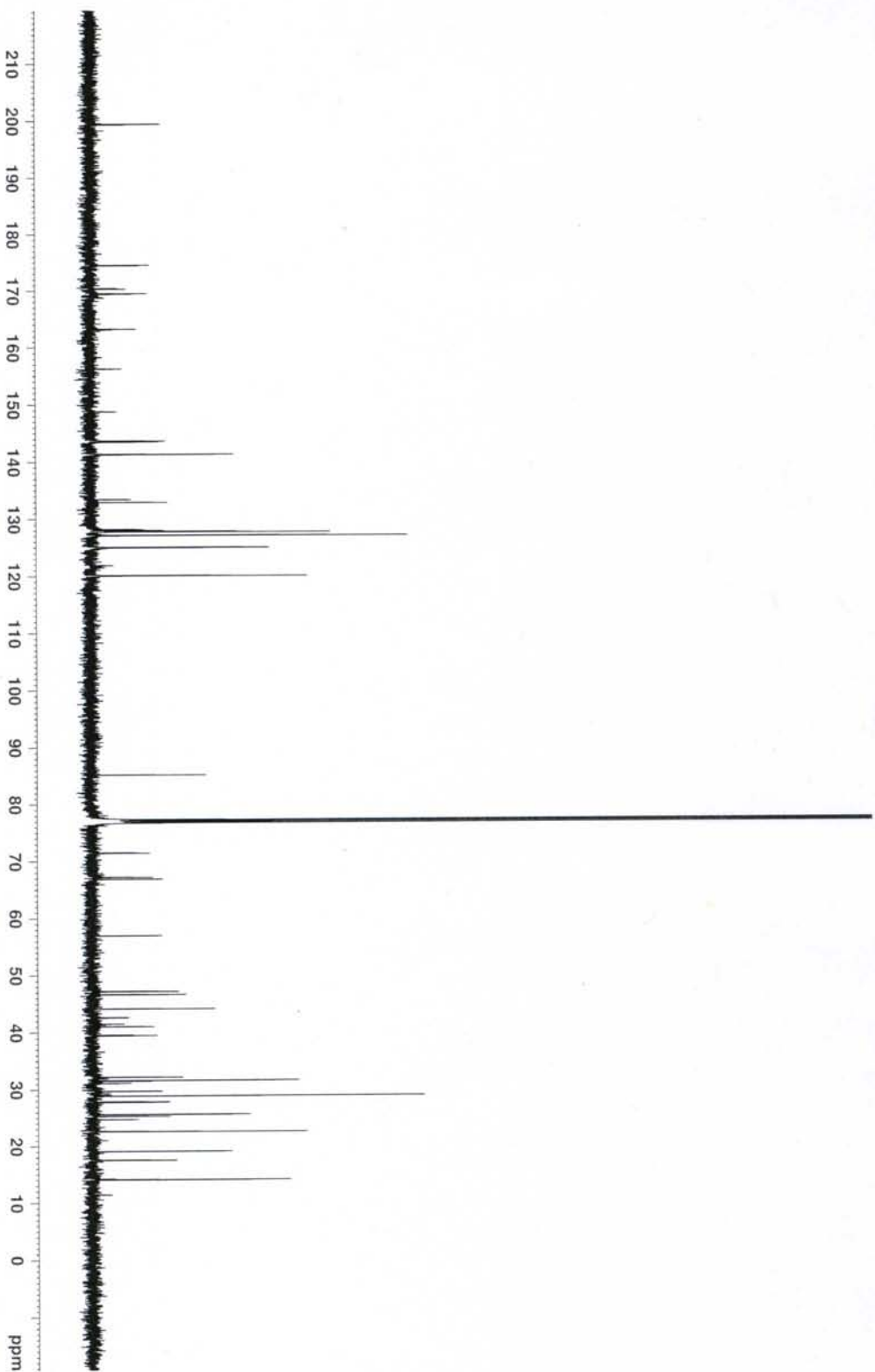
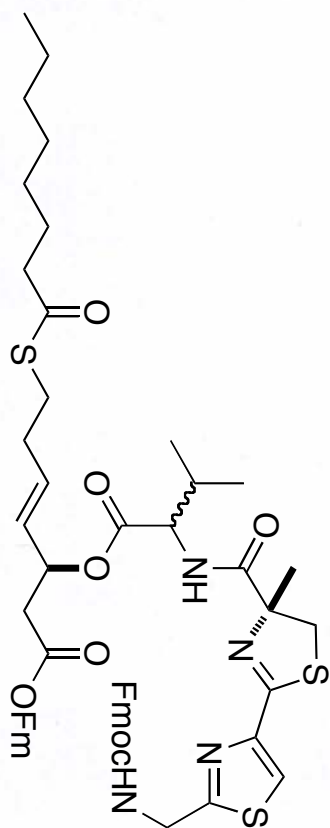
Current Data Parameters
 NAME FmocHN-FmocHeater-1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080915
 Time 19:21
 INSTRUM spect
 PROBRD 5 mm MSL1000
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SMR 10330.578 Hz
 FIDRES 0.117622 Hz
 AQ 3.117928 sec
 RQ 2.2228 sec
 DE 48.400 usec
 TE 300.2 K
 D1 1.00000000 sec
 MCREST 0.00000000 sec
 MCWRR 0.01500000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 14.50 usec
 PL1 -1.00 dB
 SFO1 500.0230879 MHz

F2 - Processing parameters
 SI 32768
 SF 500.0200000 MHz
 MDW 0
 EX 0
 SSB 0.30 Hz
 GB 0
 PC 1.40

¹H NMR spectrum for compound **32** (2R : 2S = 1.5 : 1, 500 MHz, CDCl₃)



^{13}C NMR spectrum for compound **32** ($2R : 2S = 1.5 : 1$, 500 MHz, CDCl_3)

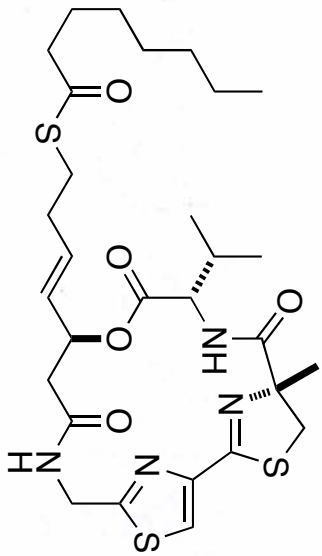
Current Data Parameters
 NAME FmocNH-FmocHeater-I-C
 EXNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20090919
 Time 11:05:03
 INSTRUM spect
 PROBRD 5 mm Multinuc1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SFO 300.10 029 Hz
 FIDRES 0.458322 Hz
 AQ 1.0912344 sec
 RG 7298.2
 DW 16.650 usec
 DE 12.00 usec
 TE 300.2 K
 AT 2.000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 MCREST 0.00000000 sec
 MCMRFX 0.01500000 sec

===== CHANNEL #1 =====
 NUC1 13C
 P1 12.00 usec
 PL 3.00 dB
 SFO1 125.7427020 MHz

===== CHANNEL #2 =====
 CPDPRG2 waltz16
 NUC2 1H
 P2 100.00 usec
 PL2 -1.00 dB
 PL12 18.80 dB
 PL13 22.50 dB
 SFO2 500.0220001 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7304296 MHz
 MDI EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00



```

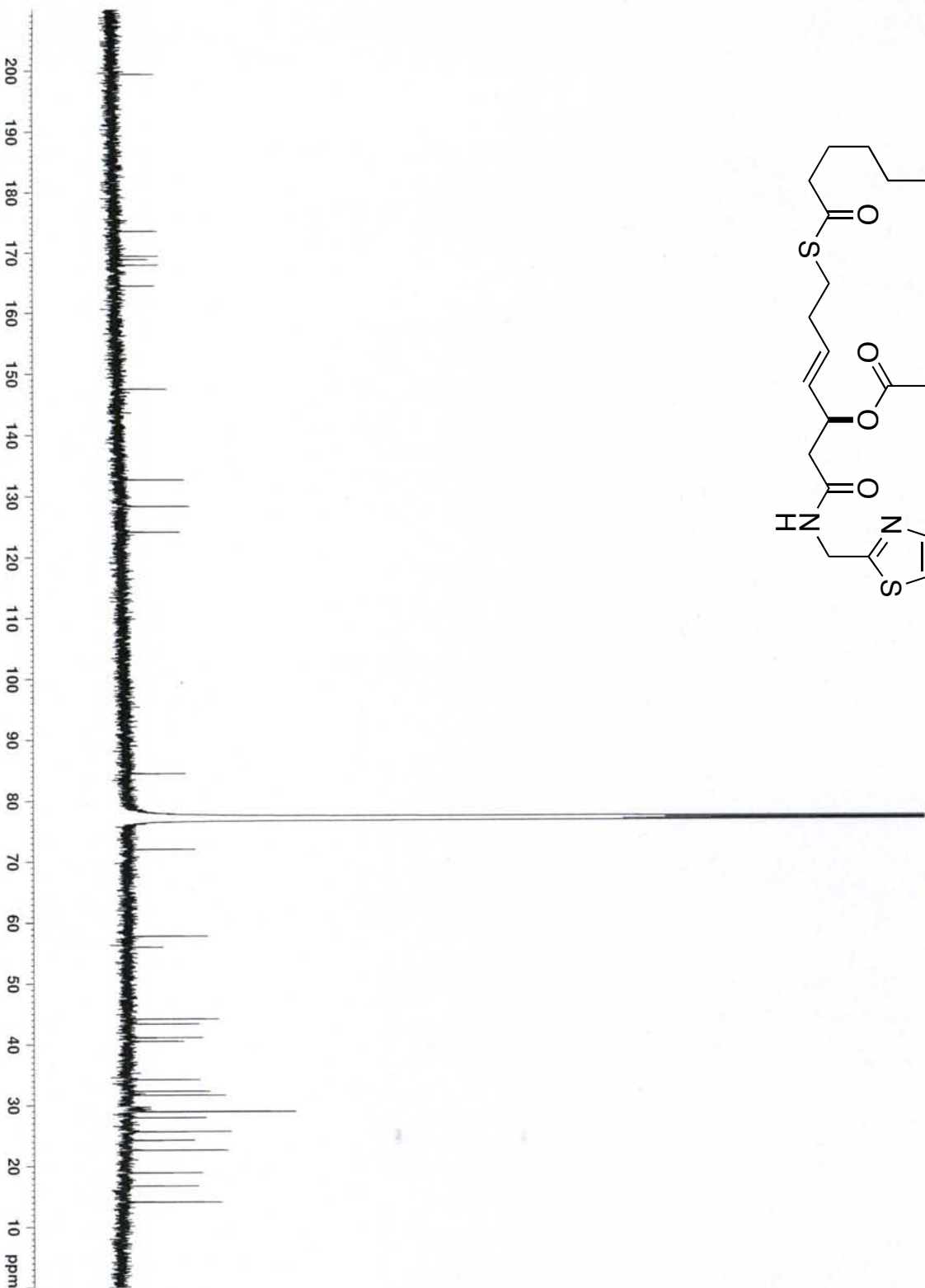
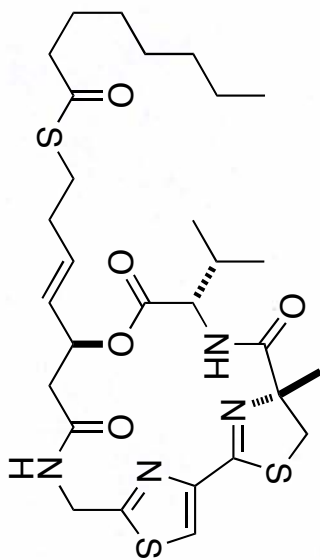
Current Data Parameters
NAME      11-largetazole-4
EXPNO     1
PROCNO    1

F2 - Acquisition Parameters
Date_     20091116
Time      12:40
INSTRUM   spect
PROBHD    5 mm Multinucl
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        10330.578 Hz
FIDRES     0.119824 Hz
AQ         3.119824 sec
RG         645.1
DW         48.400 usec
DE         6.00 usec
TE         300.2 K
D1         1.00000000 sec
D2         0.00000000 sec
D3         0.00000000 sec
D4         0.01500000 sec
MCREST    0.01500000 sec
KCMRKR

===== CHANNEL f1 =====
NUC1       1H
P1         14.80 usec
PL1        -1.00 dB
SFO1       500.0230878 MHz

F2 - Processing parameters
SI         32768
SF         500.0200000 MHz
RG         645.1
WDW        EM
SSB        0
GB         0
PC         1.00
  
```

¹H NMR spectrum for largetazole (**1**) (500 MHz, CDCl₃)



Current Data Parameters
 NAME 11-largazole-S-C
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20081121
 Time 23:10
 INSTRUM spect
 PROBRD 5 mm Maltinnici
 F2P1 200930
 F2P2 200930
 F2P3 200930
 T0 200930
 SOLVENT CDCl3
 NS 49033
 DS 4

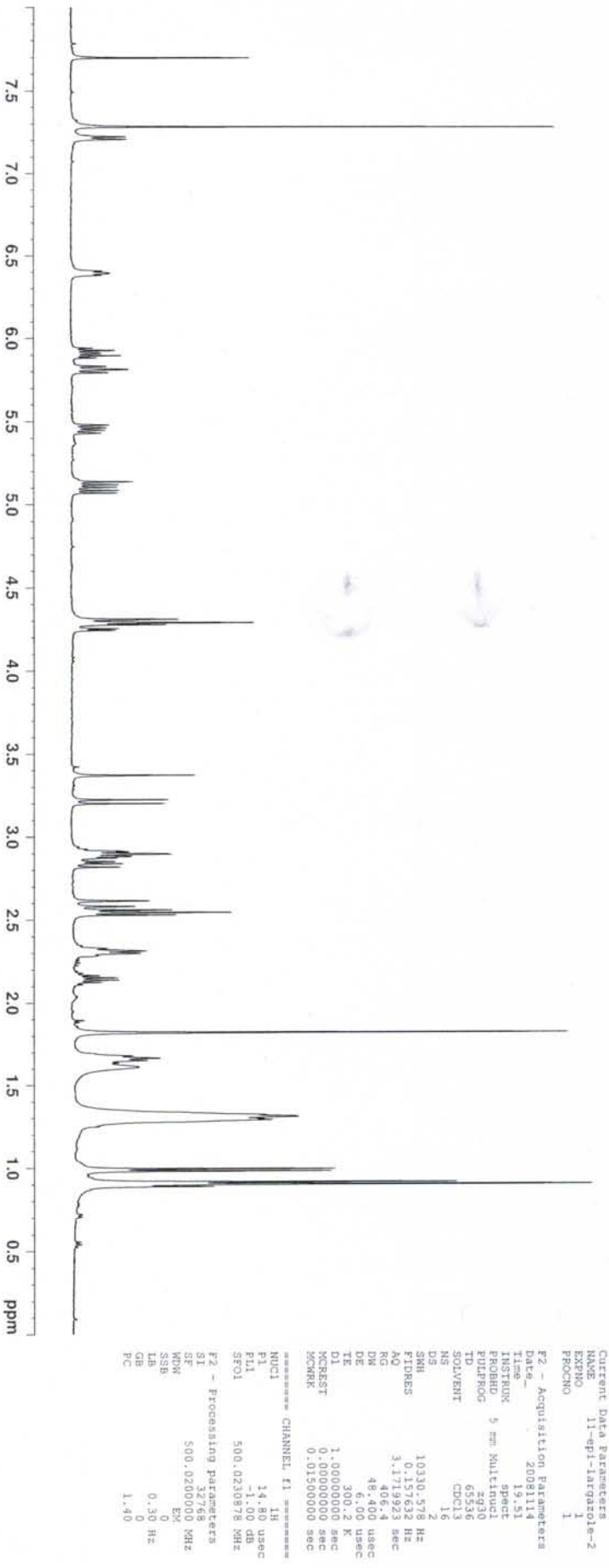
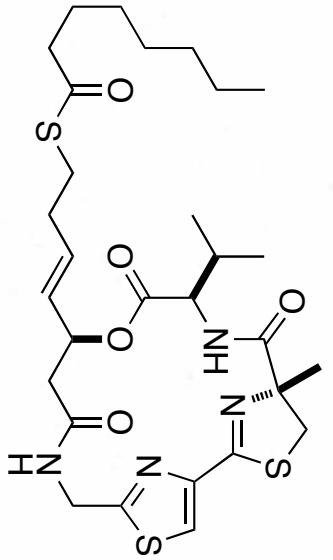
SWH 30030.029 Hz
 FIDRES 0.458222 Hz
 AQ 1.0912244 sec
 RG 3251
 DM 14.650 usec
 DE 360.0 usec
 TE 300.2 K
 FI 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 XCFREST 0.00000000 sec
 XCMRCK 0.01500000 sec

===== CHANNEL F1 =====
 NUCL 13C
 P1 12.00 usec
 PL1 3.00 dB
 SFO1 125.7427020 MHz

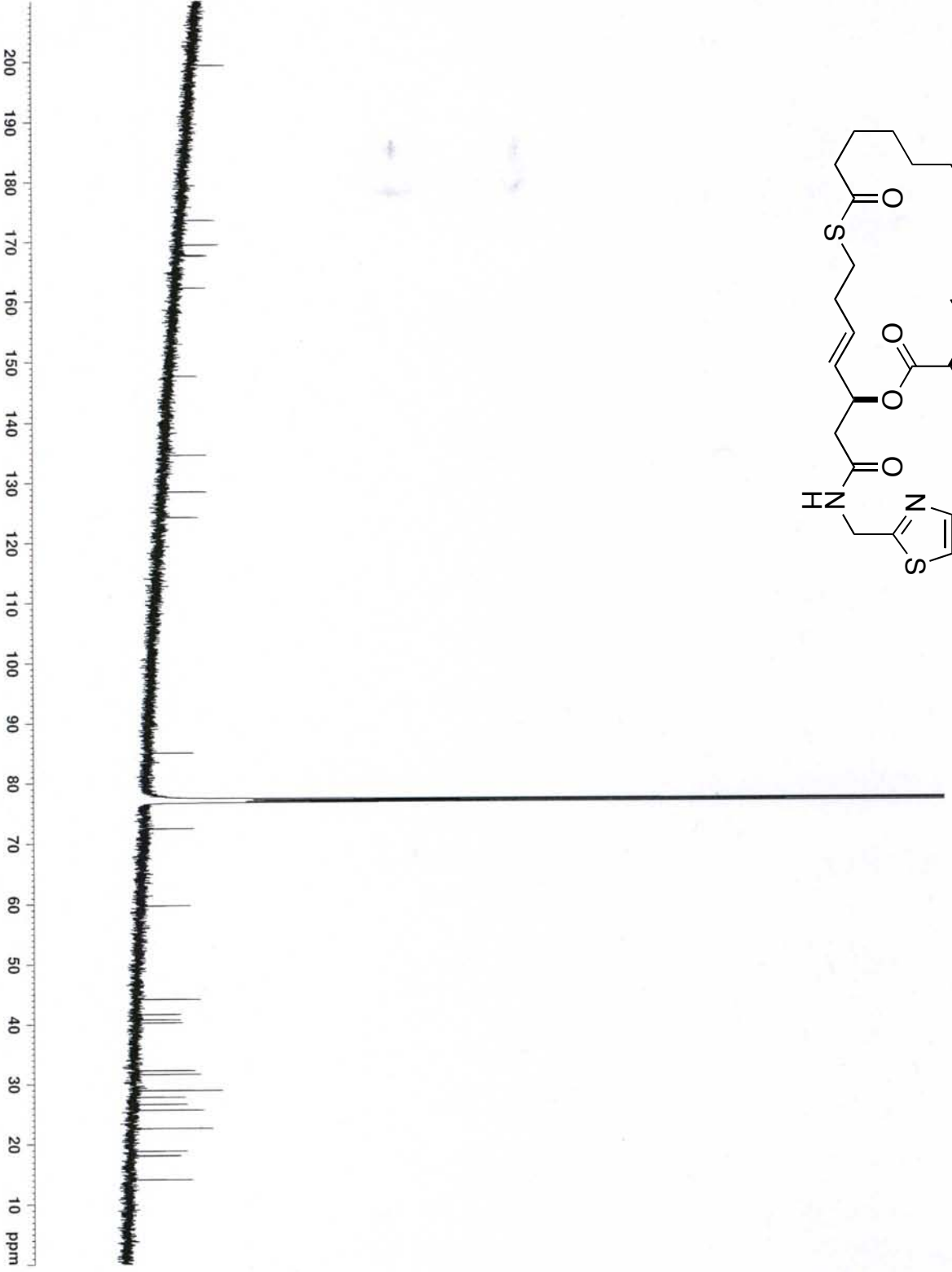
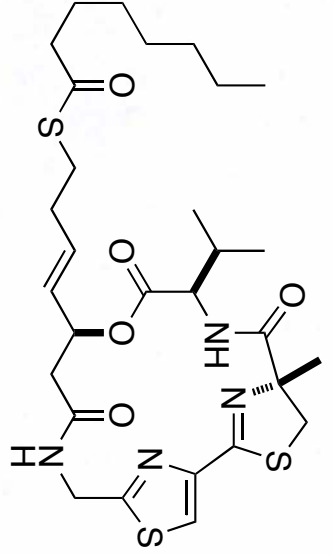
===== CHANNEL F2 =====
 CHDRG2 waltz16
 NUCL2 1H
 PCPD2 100.00 usec
 PL2 1.00 dB
 PL12 15.39 dB
 PL13 15.39 dB
 SFO2 500.0220001 MHz

F2 - Processing parameters
 SI 32768
 SF 125.7301290 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 FC 1.40

^{13}C NMR spectrum for largazole (1) (500 MHz, CDCl_3)



¹H NMR spectrum for 2-*epi*-largazole (**33**) (500 MHz, CDCl₃)



^{13}C NMR spectrum for 2-*epi*-largazole (**33**) (500 MHz, CDCl_3)

```

Current Data Parameters
NAME      11-epi-largazole-3-C
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20081116
Time     18.14
INSTRUM  spect
PROBHD   5 mm Multinuc1
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       4094
DS       4
SFO      30030.029 Hz
FIDRES   0.458222 Hz
AQ       1.091244 sec
RG       1625.5
DW       16.650 usec
DE       16.00 usec
TE       300.2 K
D1       2.00000000 sec
d11      0.03000000 sec
DELTA    1.89999998 sec
MCREST   0.00000000 sec
MCWRRK   0.01500000 sec

===== CHANNEL f1 =====
NUC1      13C
P1        13.00 usec
PL        3.00 dB
SFO1     125.7627020 MHz

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      13C
PCPD2     100.00 usec
PL2       -1.00 dB
PL12      15.59 dB
PL13      22.50 dB
SFO2     500.0220001 MHz

F2 - Processing parameters
SFO      125.7301290 MHz
SF       32766
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.00
  
```