

Stereoselective Syntheses of the C(1)-C(9) Fragment of Amphidinolide C

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Supporting Information

Selected ¹H NMR and ¹³C NMR Spectra for Key Intermediates

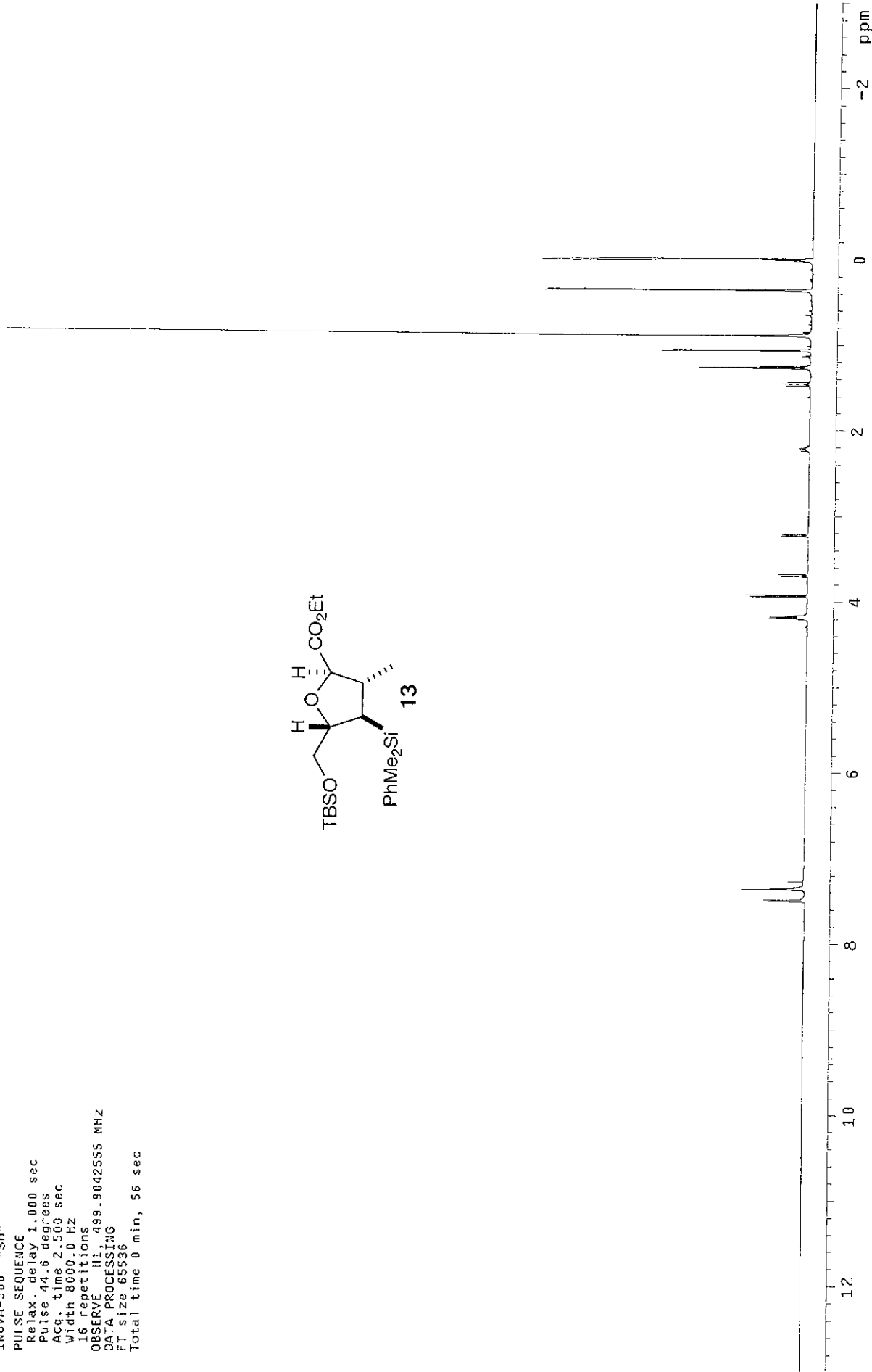
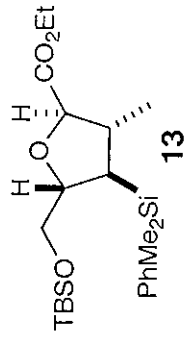
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STANDARD PROTON PARAMETERS

SI-3

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
INOVA-500 "Sn"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 44.6 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE Hz: 499.9042555 MHZ
DATA PROCESSING
F1 size 65536
Total time 0 min, 56 sec



STANDARD CARBON PARAMETERS

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

User: 1-14-87

NOVA-500 "Sn"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 39.7 degrees

Acq. time 1.300 sec

Width 29896.3 Hz

480 repetitions

OBSERVE C13, 125.7010316 MHz

DECOUPLE H1, 499.9067532 MHz

Power 32 dB

continuously on

WALTZ-16 modulated

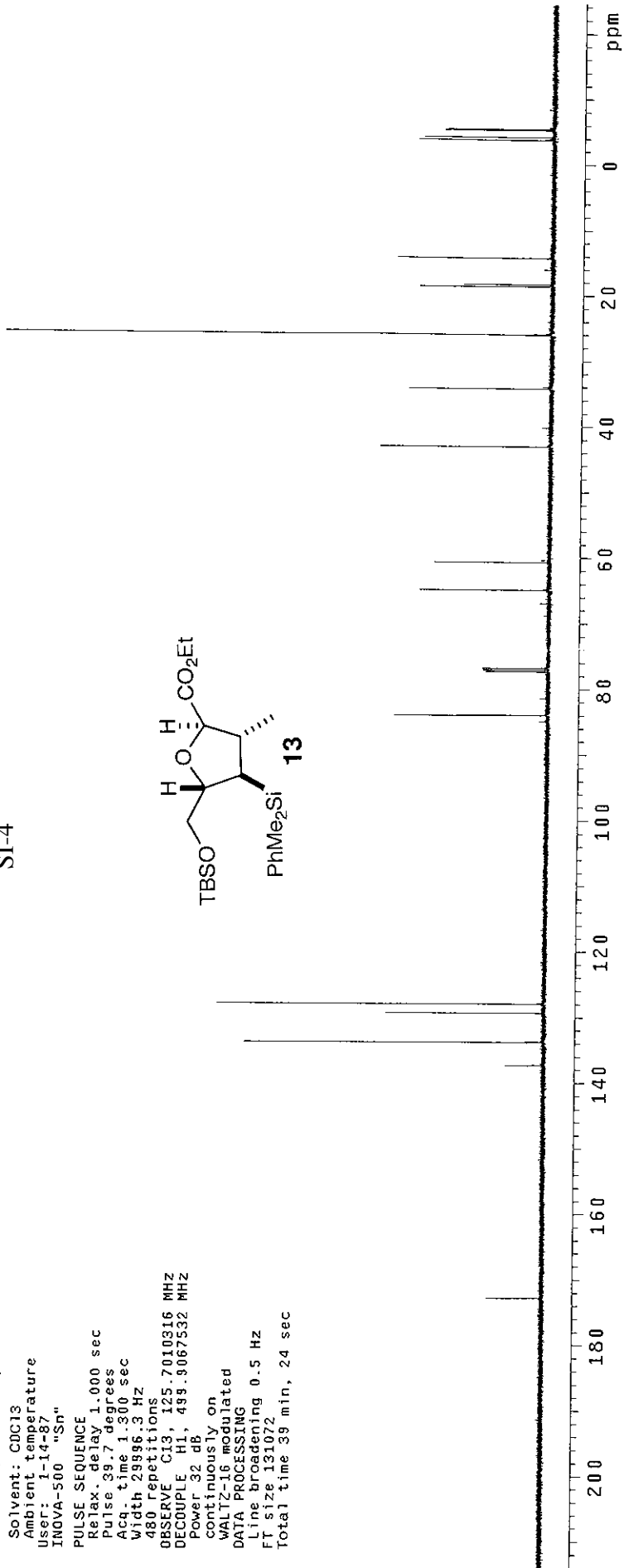
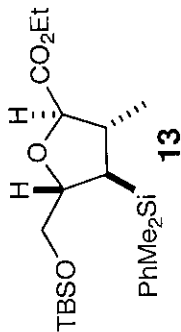
DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

Total time 39 min, 24 sec

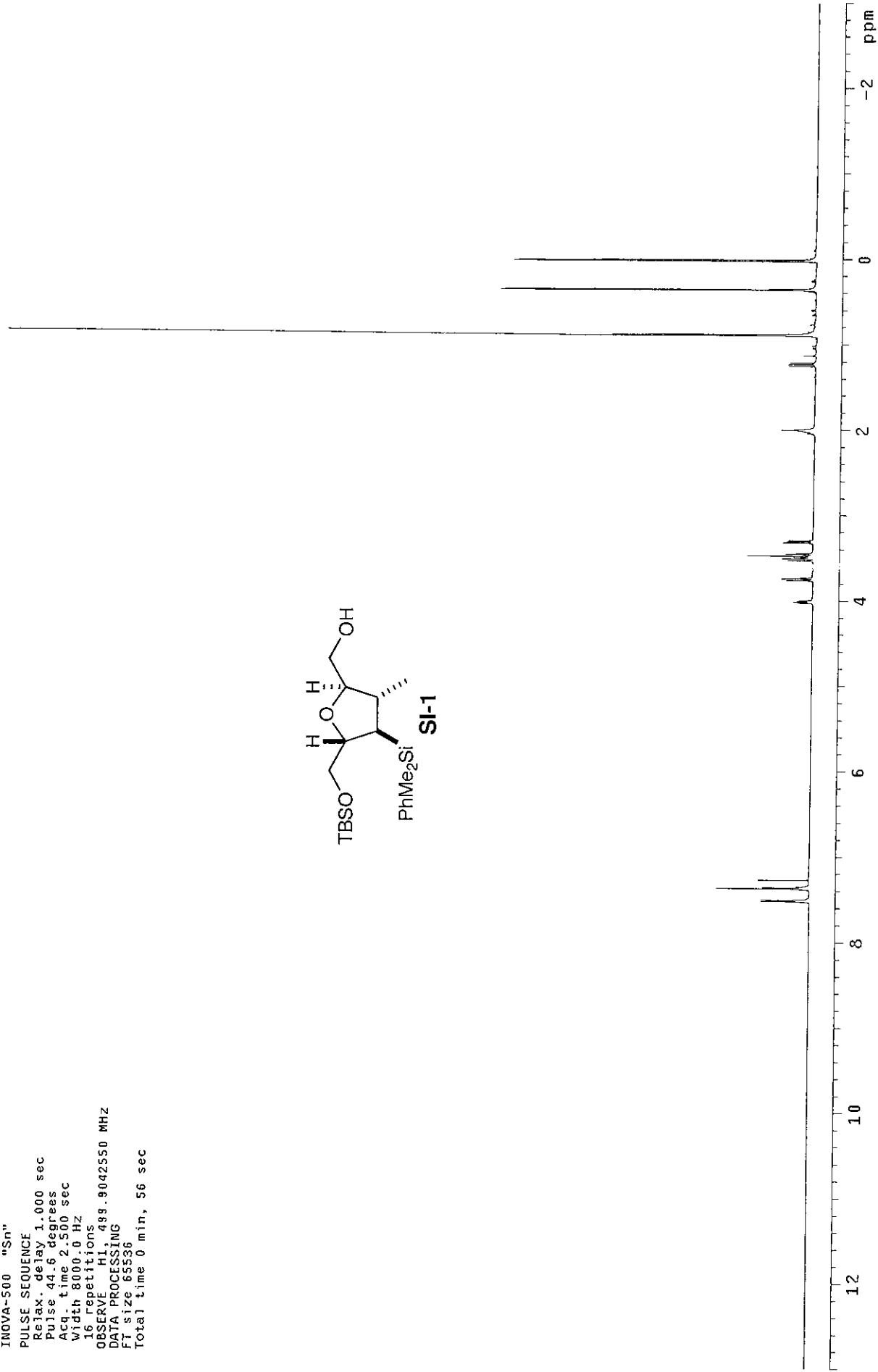
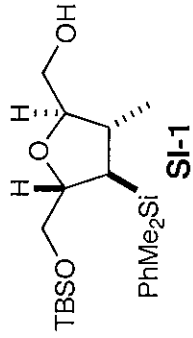
SI-4



STANDARD PROTON PARAMETERS

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
INOVA-500 "Sn"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 41.6 degrees
Acq. time 2.500 sec
width 8000.0 Hz
16 repetitions
OBSERVE HI, 499.9042550 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 56 sec

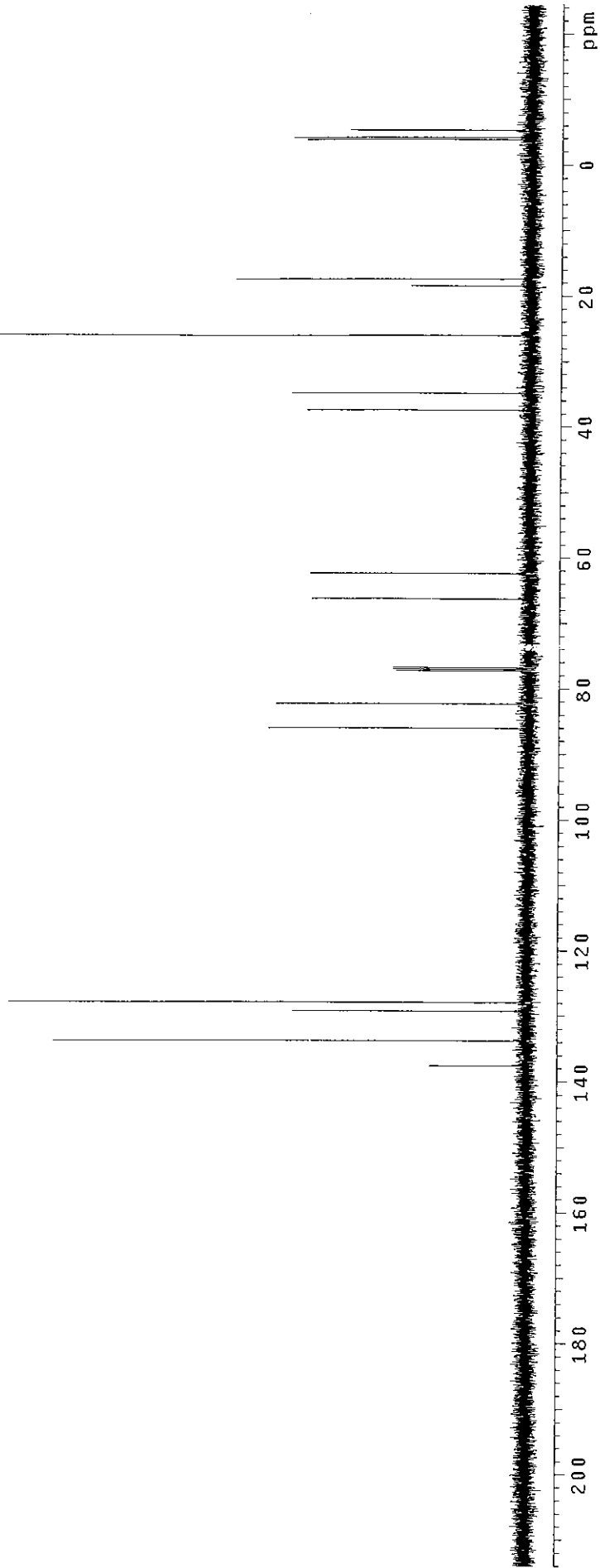
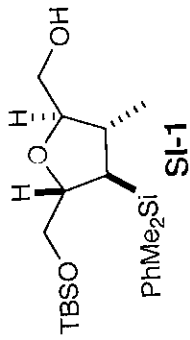
SI-5



STANDARD CARBON PARAMETERS

Pulse Sequence: s2bu1
 Solvent: CDCl3
 Ambient temperature
 User: i-14-87
 INOVA-500 "Sn"
 PULSE SEQUENCE
 Relax delay 1.000 sec
 Pulse 39.7 degrees
 Acq. time 1.300 sec
 Width 29986.3 Hz
 64 repetitions
 OBSERVE C13, 125.7010307 MHZ
 DECOUPLE H1, 499.9067532 MHZ
 Power 32 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072
 Total time 39 min, 24 sec

SI-6



STANDARD PROTON PARAMETERS

Pulse Sequence: s2pu1

Solvent: CDC13

Ambient temperature

INOVA-500 "Sn"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 44.6 degrees

Acq. time 2.500 sec

Width 8000.0 Hz

8 repetitions

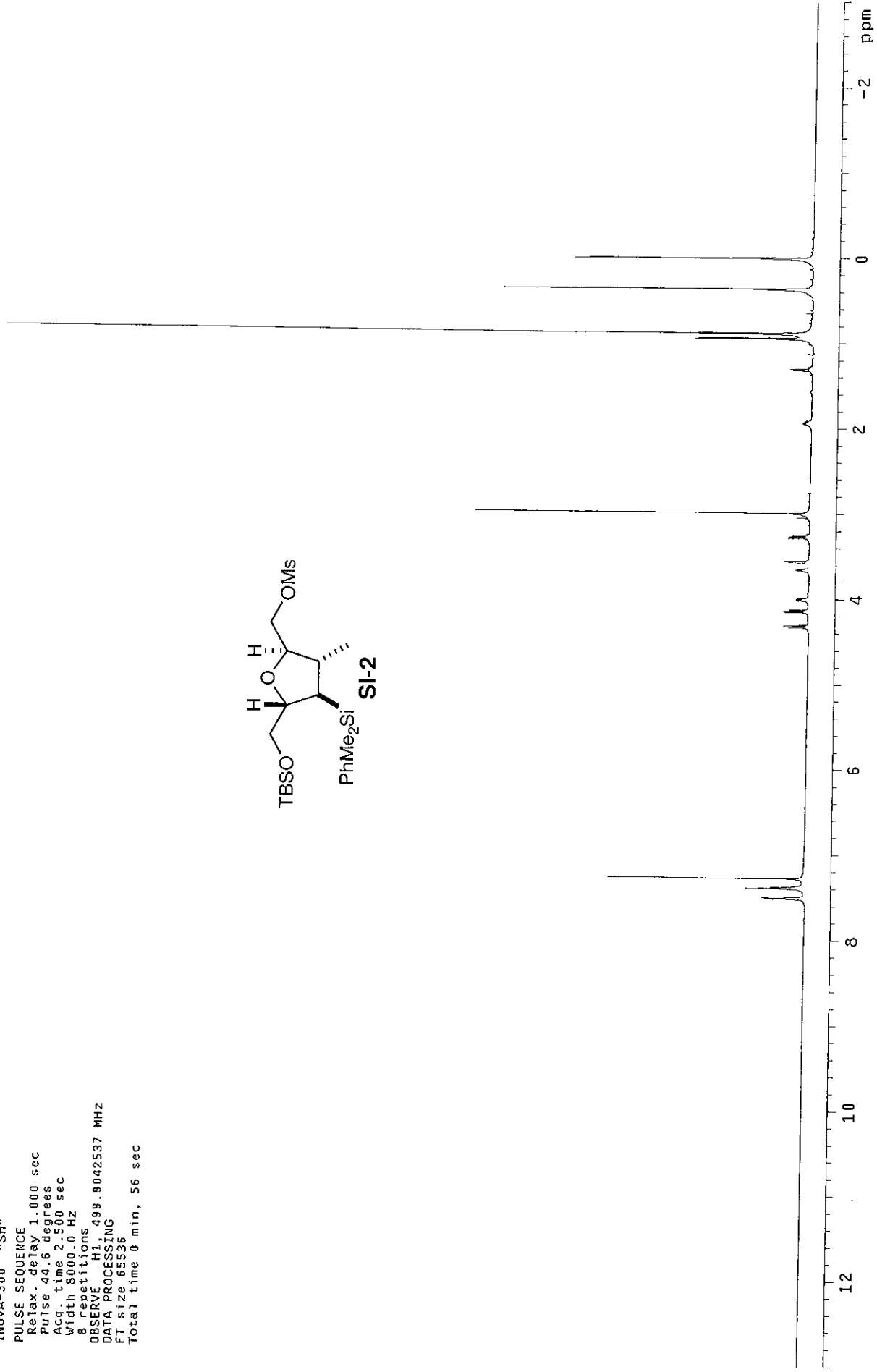
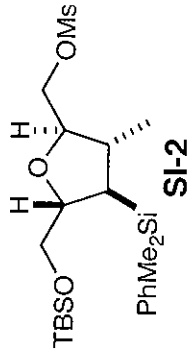
RESERVE H1, 499.9042537 MHZ

DATA PROCESSING

FT size 65536

Total time 0 min, 56 sec

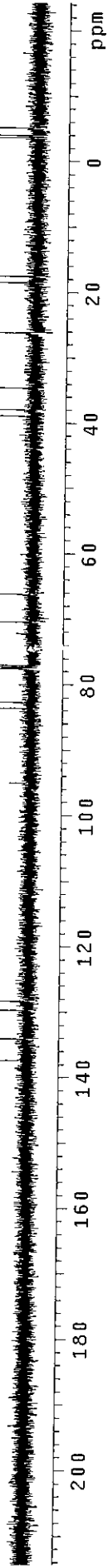
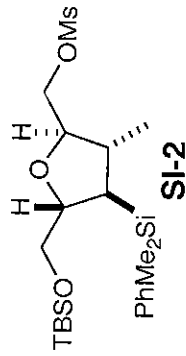
SI-7



STANDARD CARBON PARAMETERS

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
User: 1-14-B7
IROVA-500 "Sn"
PULSE SEQUENCE
Relax delay 1.000 sec
Pulse 39.7 degrees
Acq. time 1.500 sec
Width 29996.3 Hz
160 repetitions
OBSERVE C13, 125.7009946 MHz
DECOUPLE H1, 499.9067532 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 331072
Total time 39 min, 24 sec

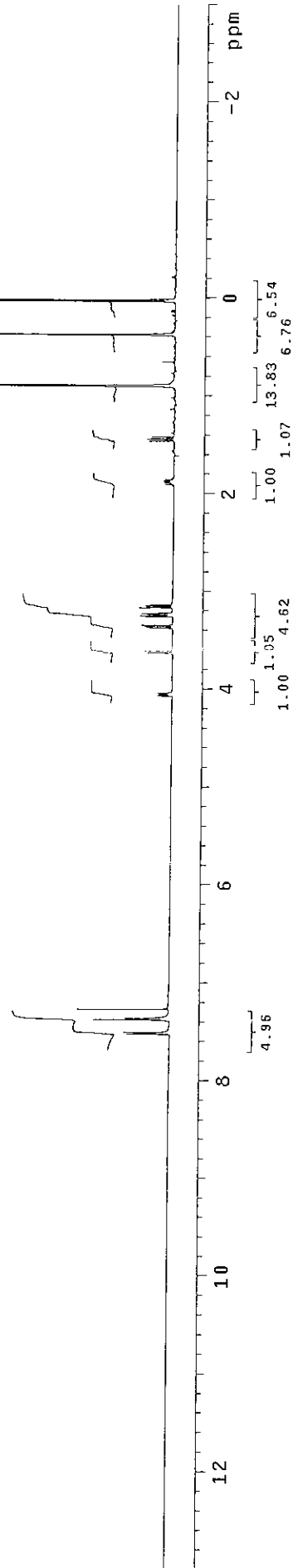
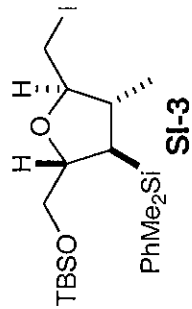
SI-8



STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul
 Solvent: CDCl₃
 Ambient temperature
 INOVA-500 "Si"
 PULSE SEQUENCE
 Relax delay 1.000 sec
 Pulse 44.6 degrees
 Acq time 2.500 sec
 Width 8000.0 Hz
 16 repetitions
 OBSERVE H1, 499.9042553 MHZ
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 56 sec

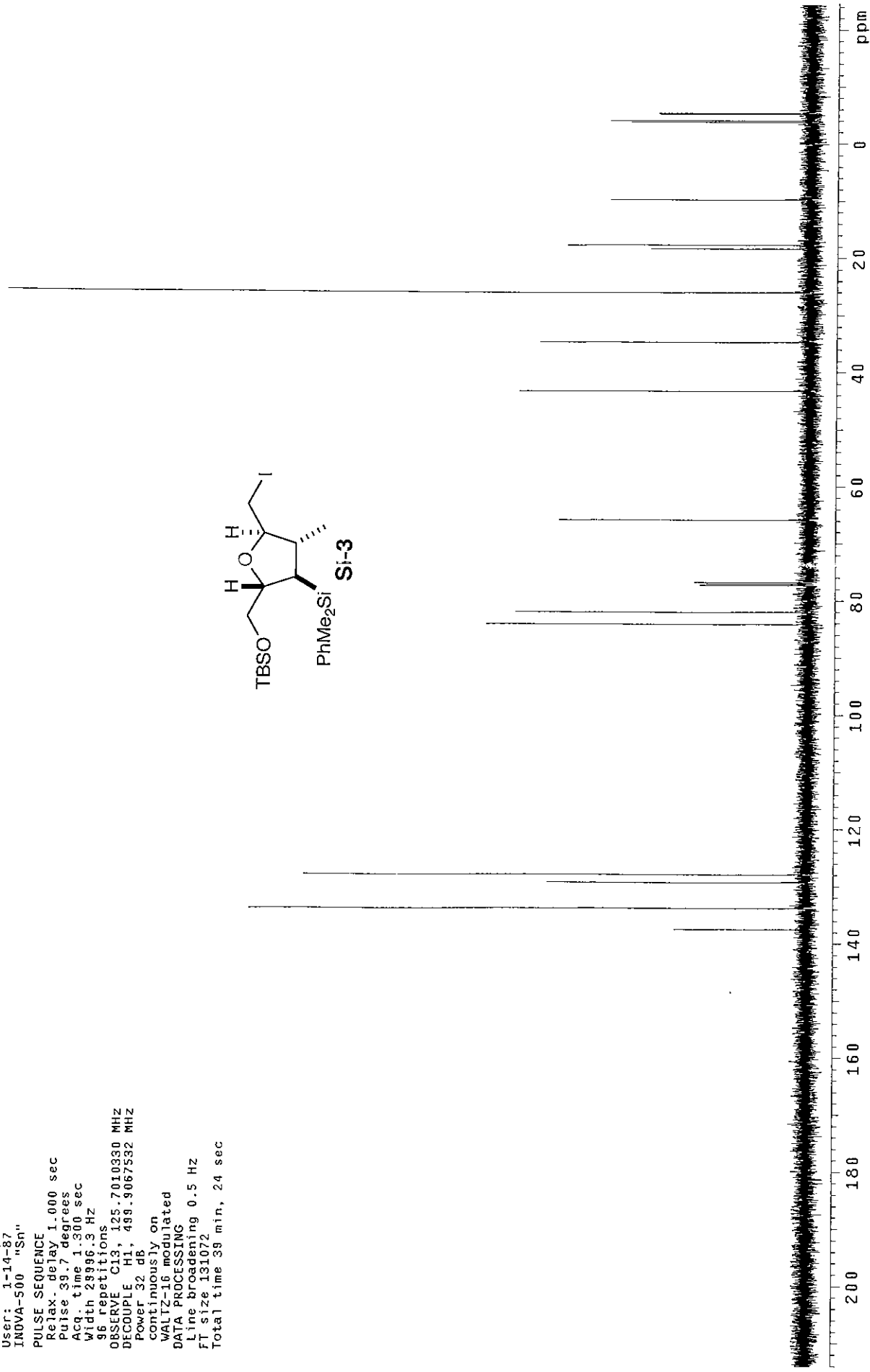
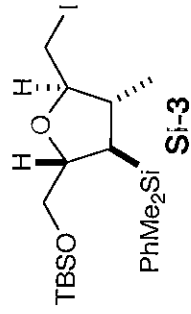
SI-9



STANDARD CARBON PARAMETERS

Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 User: i-14-87
 INOVA-500 "Sn"
 PULSE SEQUENCE
 Relax delay 1.000 sec
 Pulse 39.7 degrees
 Acq. time 1.300 sec
 Width 29986.3 Hz
 96 repetitions
 OBSERVE C13, 125.7010330 MHZ
 DECOUPLE H1, 499.9067532 MHZ
 Power 32 dB
 continuously on
 WAIT-16 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072
 Total time 39 min, 24 sec

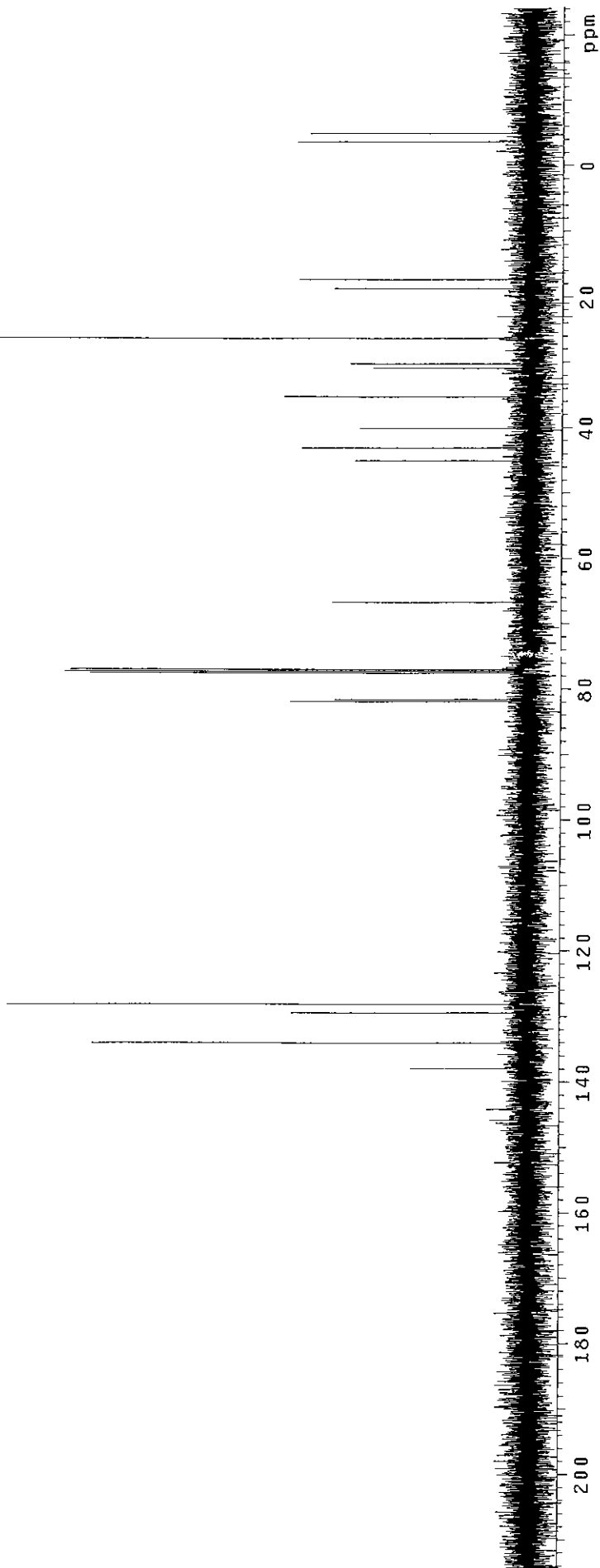
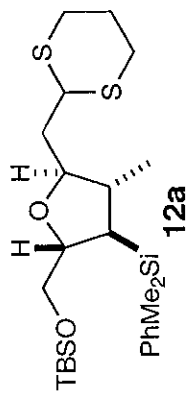
SI-10



STANDARD CARBON PARAMETERS

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INDVA-500 "Sn"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 39.7 degrees
Acq. time 1.300 sec
Width 29996.3 Hz
256 repetitions
OBSERVE C13, 125.7009946 MHZ
DECOUPLE H1, 499.9067532 MHZ
Power 32 dB,
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 39 min, 24 sec

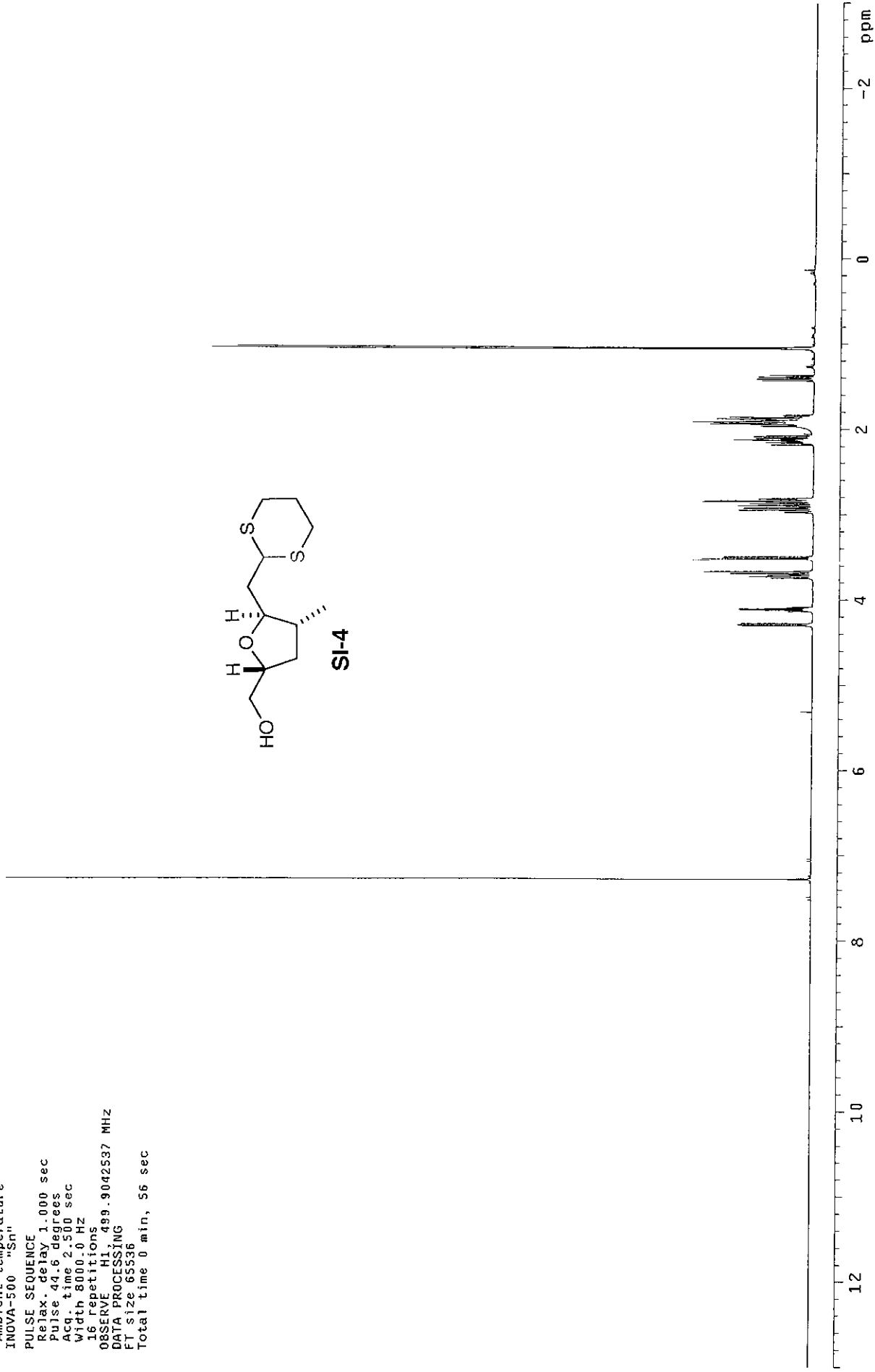
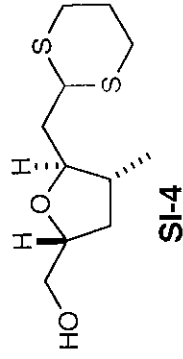
SI-12



STANDARD PROTON PARAMETERS

Pulse Sequence: s2pu1
Solvent: CDC13
Ambient temperature
INOVA-500 "Sp"
PULSE SEQUENCE
Relax delay 1.000 sec
Pulse 44.6 degrees
Acq time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.9042537 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 56 sec

SI-13



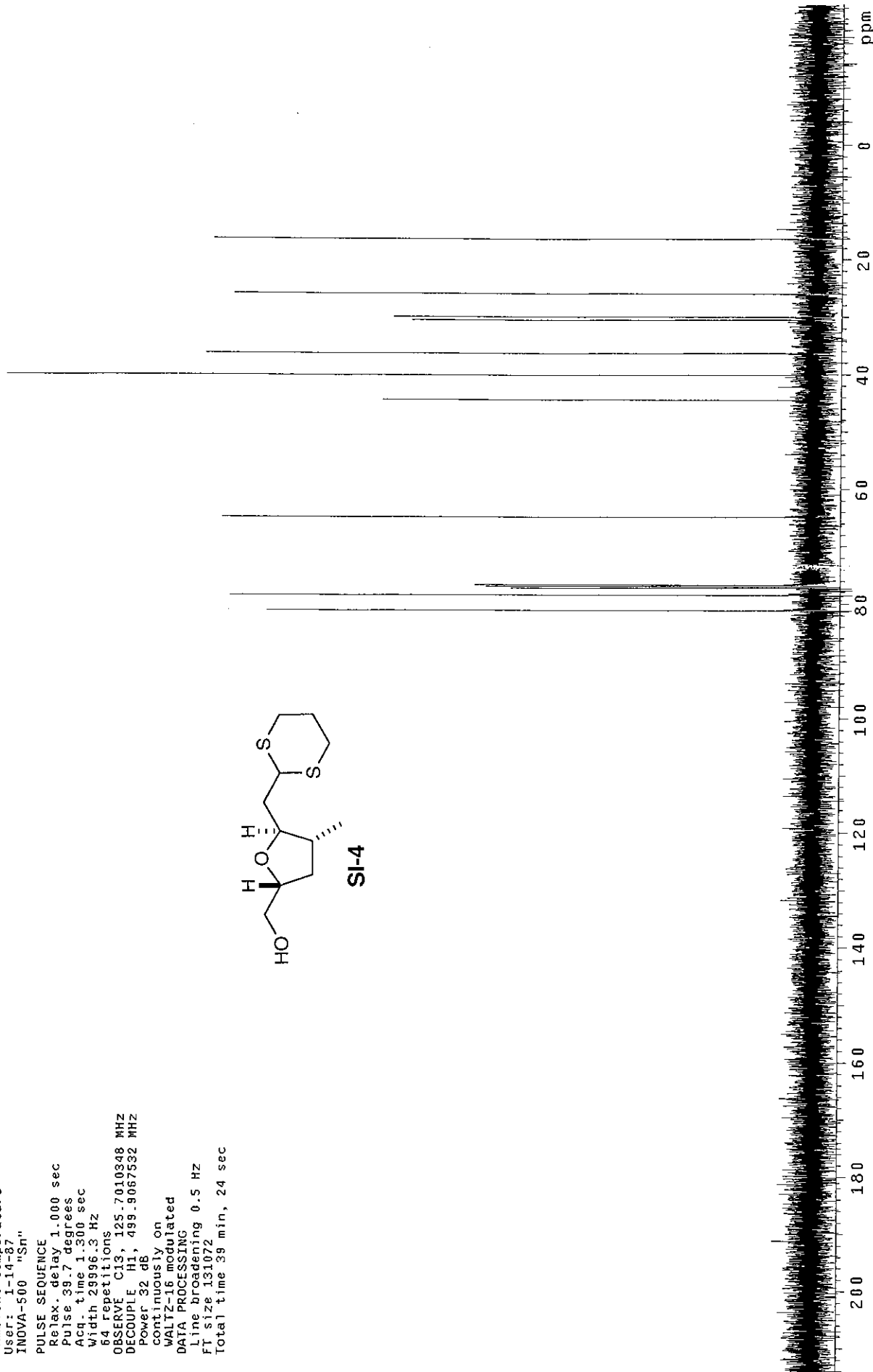
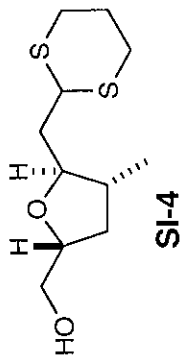
STANDARD CARBON PARAMETERS

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "S"n"

PULSE SEQUENCE

Relax. delay 1.000 sec
Pulse 39.7 degrees
Acq. time 1.300 sec
Width 29996.3 Hz
64 repetitions
OBSERVE C13, 125.7010348 MHz
DECOUPLE H1, 499.9067532 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
F1 size 131072
Total time 39 min, 24 sec

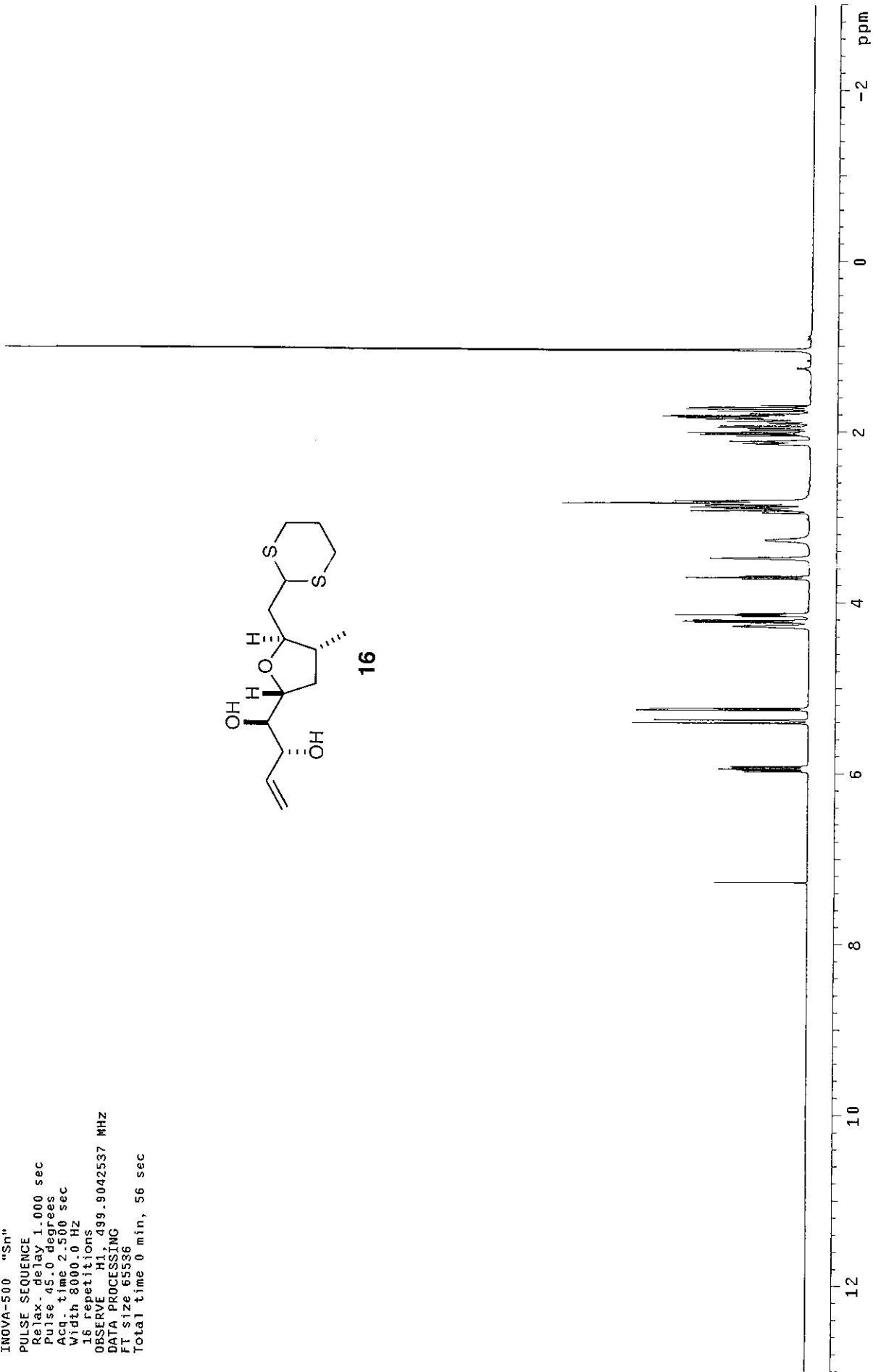
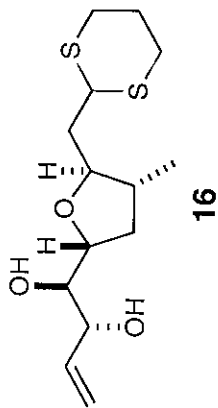
SI-14



STANDARD PROTON PARAMETERS

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
INOVA-500 "Sh"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
16 repetitions
OBSERVE HI, 499.9042537 MHZ
DATA PROCESSING
F1 size 65536
Total time 0 min, 56 sec

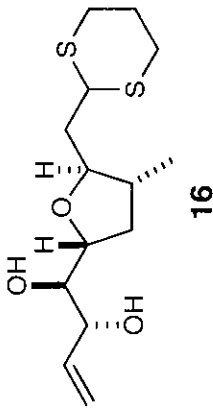
SI-15



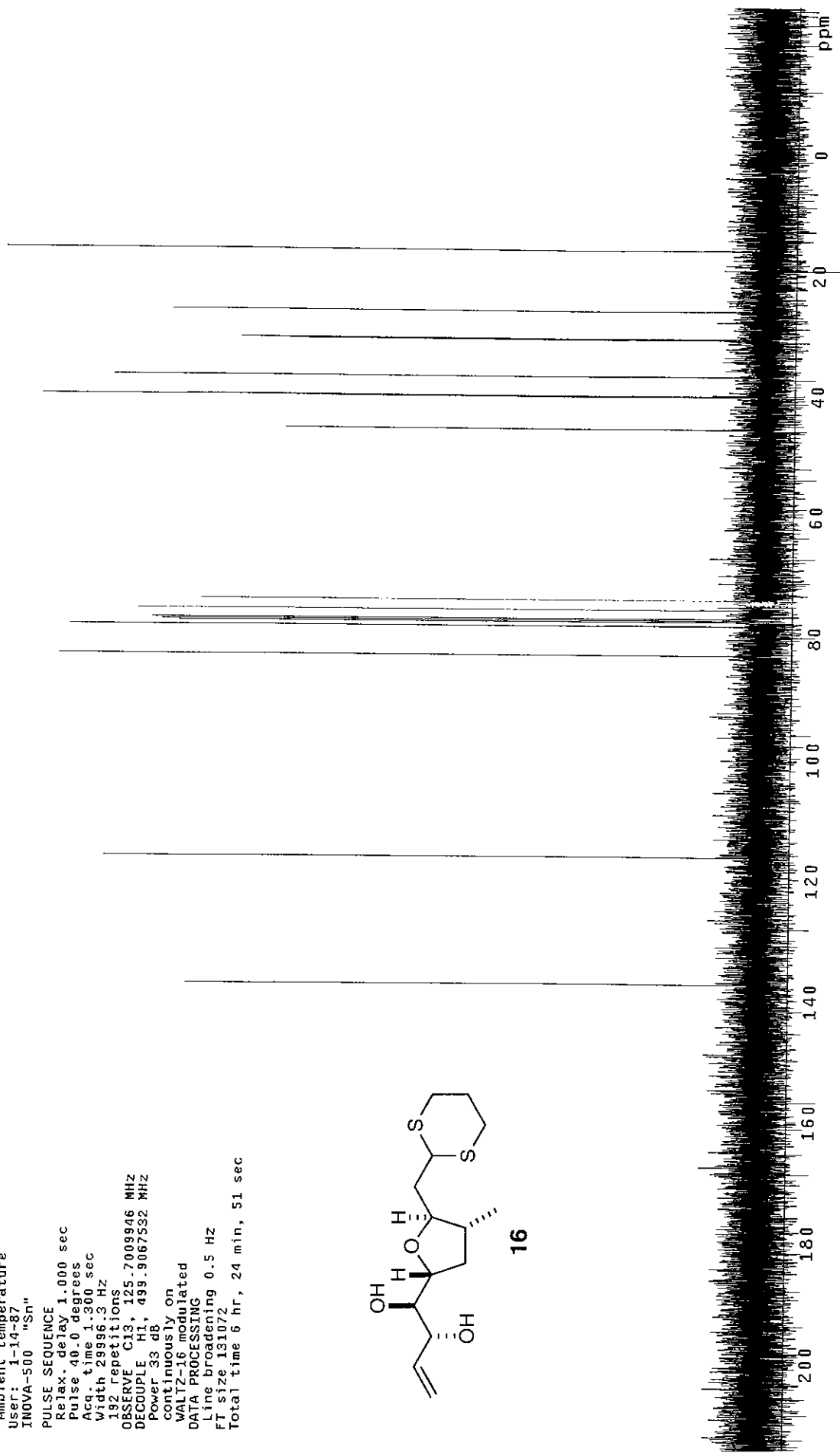
STANDARD CARBON PARAMETERS

Pulse Sequence: s2pu1
 Solvent: CDC13
 Ambient temperature
 User: 1-14-87
 INOVA-500 "Sn"

PULSE SEQUENCE
 Relax. delay 1.000 sec
 Pulse 40.0 degrees
 Acq. time 1.300 sec
 Width 29996.3 Hz
 192 repetitions
 OBSERVE C13, 125.7009946 MHZ
 DECOUPLE H1, 499.9067532 MHZ
 Power 33 dB,
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FT size 131072
 Total time 6 hr, 24 min, 51 sec



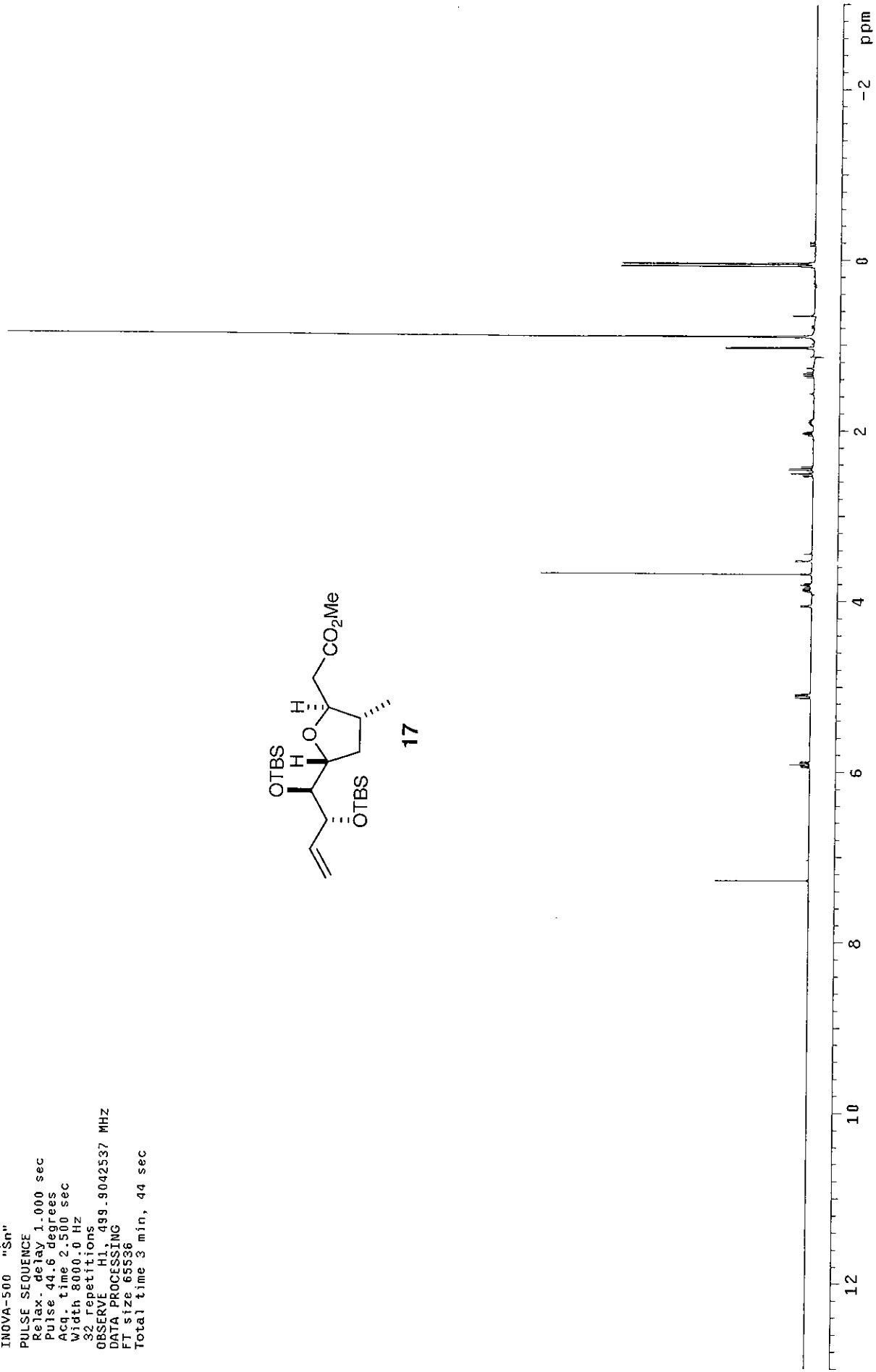
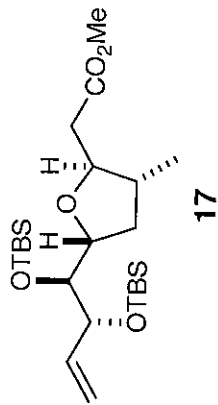
SI-16



STANDARD PROTON PARAMETERS

Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 INOVA-500 "Sn"
 PULSE SEQUENCE
 Relax delay 1.000 sec
 Pulse 44.6 degrees
 Acq. time 2.500 sec
 Width 8000.0 Hz
 32 repetitions
 OBSERVE H1, 499.9042537 MHZ
 DATA PROCESSING
 FT size 65536
 Total time 3 min, 44 sec

SI-17



STANDARD CARBON PARAMETERS

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

User: 1-14-87

File: char13CII-188

INOVA-500 "Kf"

PULSE SEQUENCE

Relax. delay 1.000 sec

Pulse 39.7 degrees

Acq. time 1.300 sec

Width 29996.3 Hz

1408 repetitions

OBSERVE C13, 125.7009846 MHZ

DECUPLE H1, 499.9067532 MHZ

Power 32 dB

continuously on

WALTZ-16 modulated

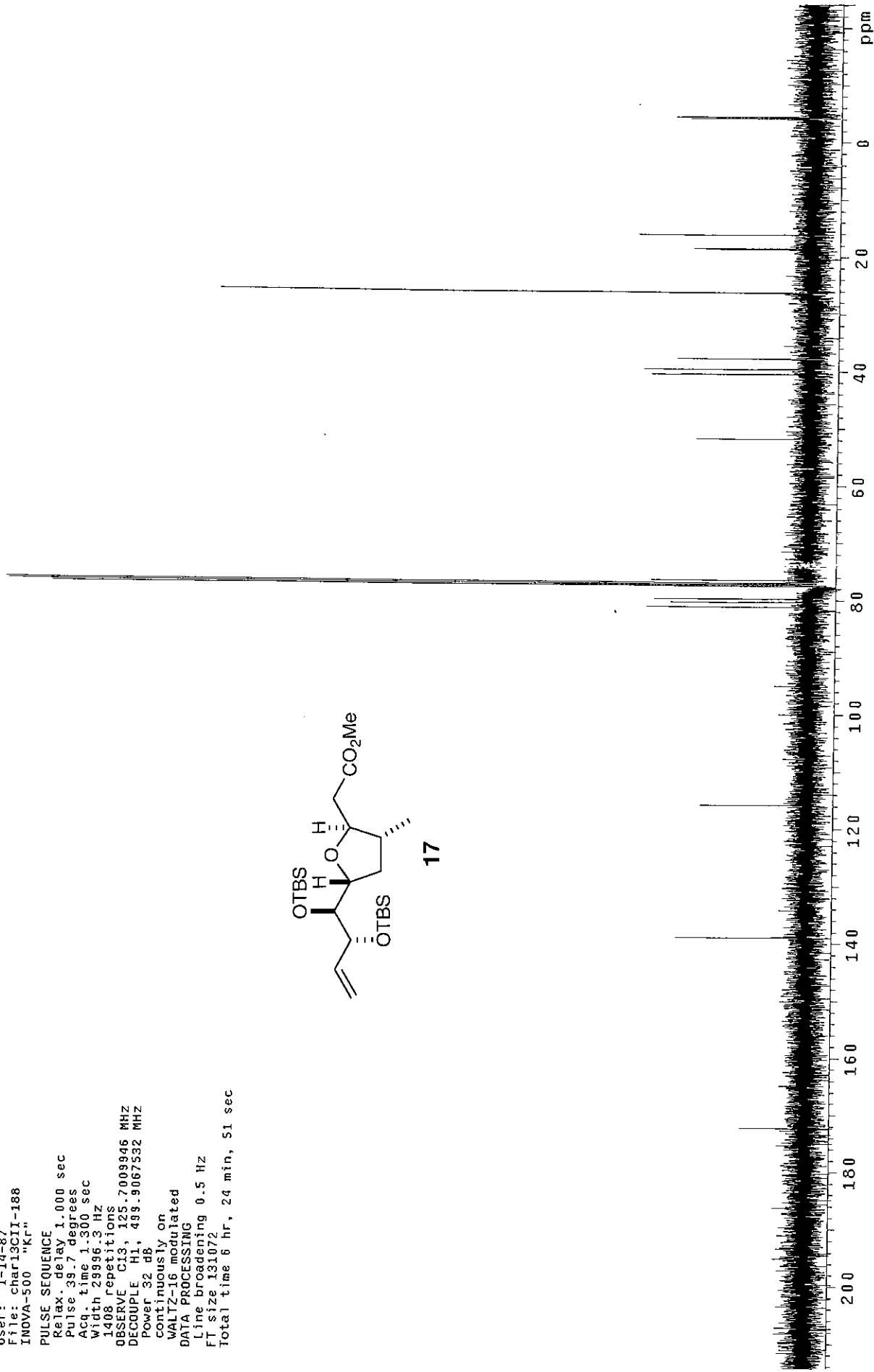
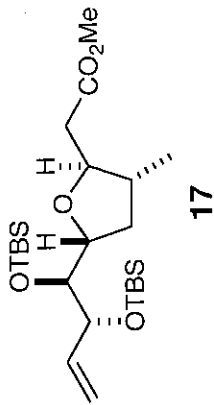
DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

Total time 6 hr, 24 min, 51 sec

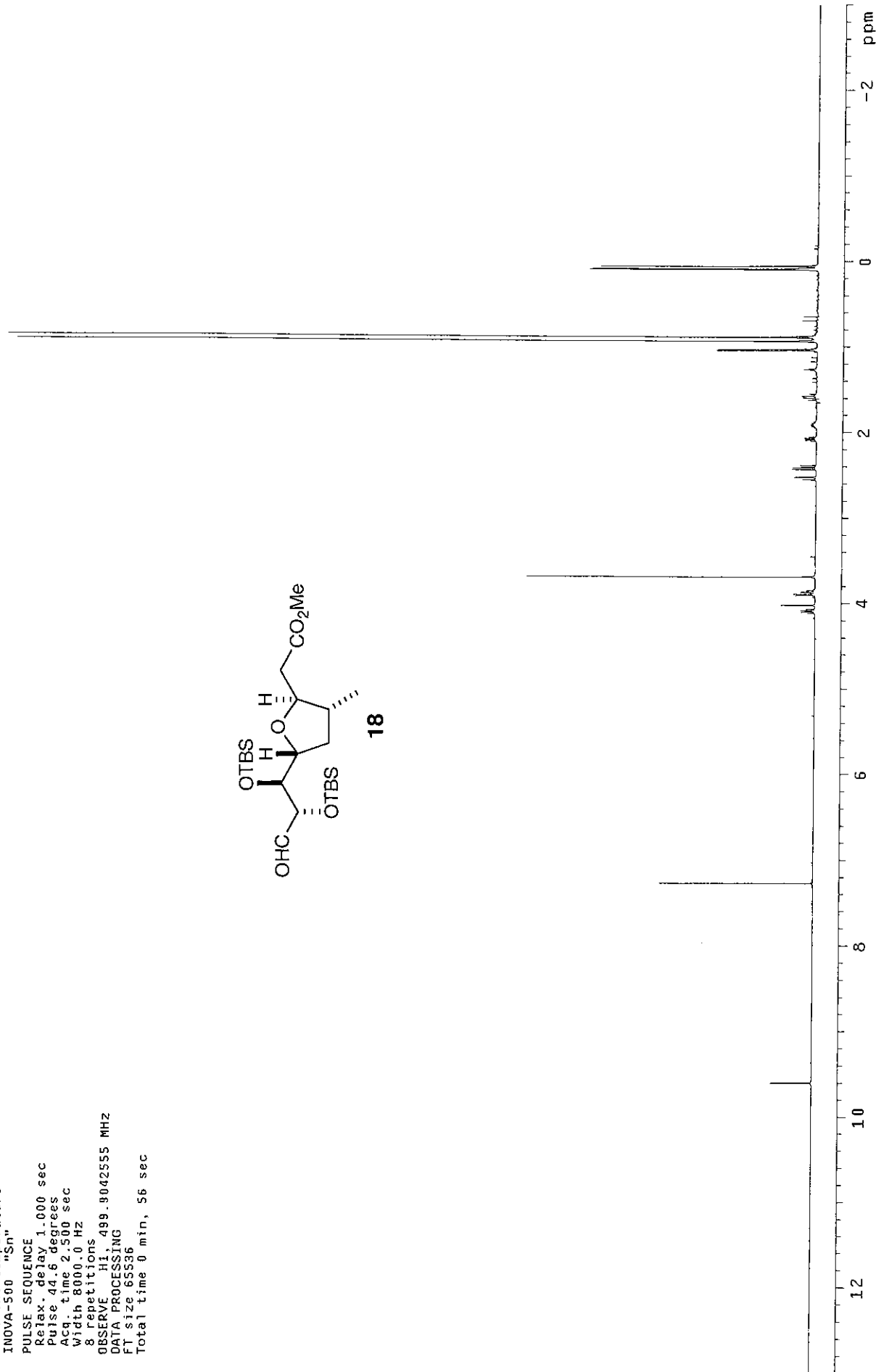
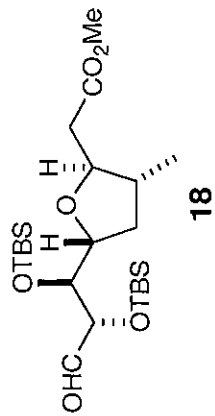
SI-18



STANDARD PROTON PARAMETERS

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
INOVA-500 "Sn"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 44.6 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.9042555 MHZ
DATA PROCESSING
FT size 65536
Total time 0 min, 56 sec

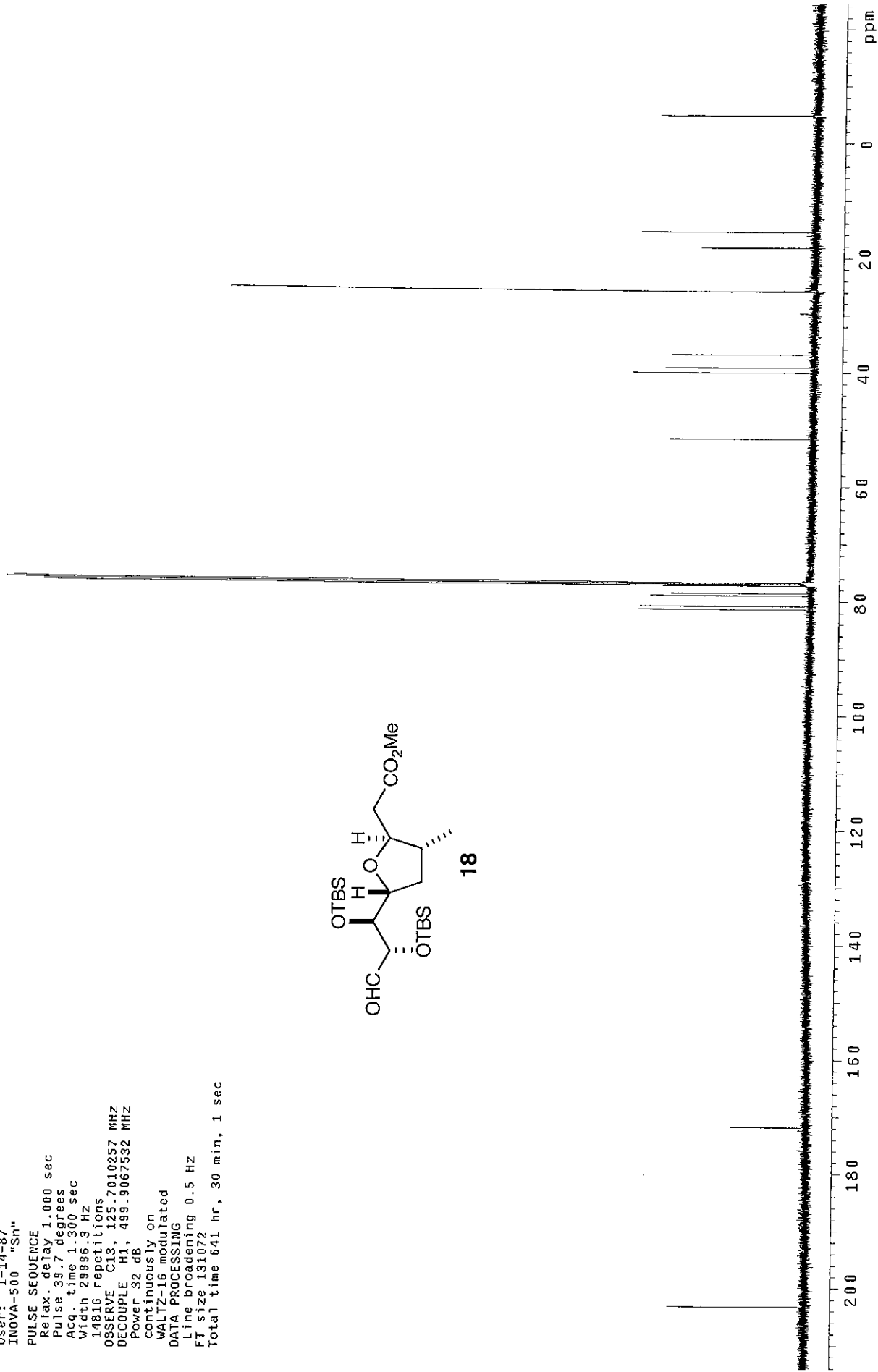
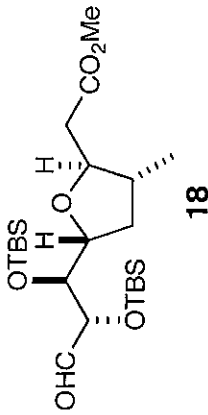
SI-19



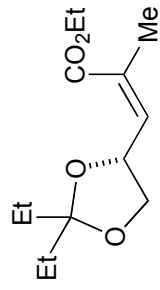
STANDARD CARBON PARAMETERS

Pulse Sequence: s2pu1
 Solvent: CDC13
 Ambient temperature
 User: i-14-87
 INOVA-500 "Sn"
 PULSE SEQUENCE
 Relax delay 1.000 sec
 Pulse 39.7 degrees
 Acq. time 1.300 sec
 Width 29996.3 Hz
 14816 repetitions
 OBSERVE C13, 125.7010257 MHz
 DECOUPLE H1, 499.9067532 MHz
 Power 32 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 0.5 Hz
 FI size 131072
 Total time 641 hr, 30 min, 1 sec

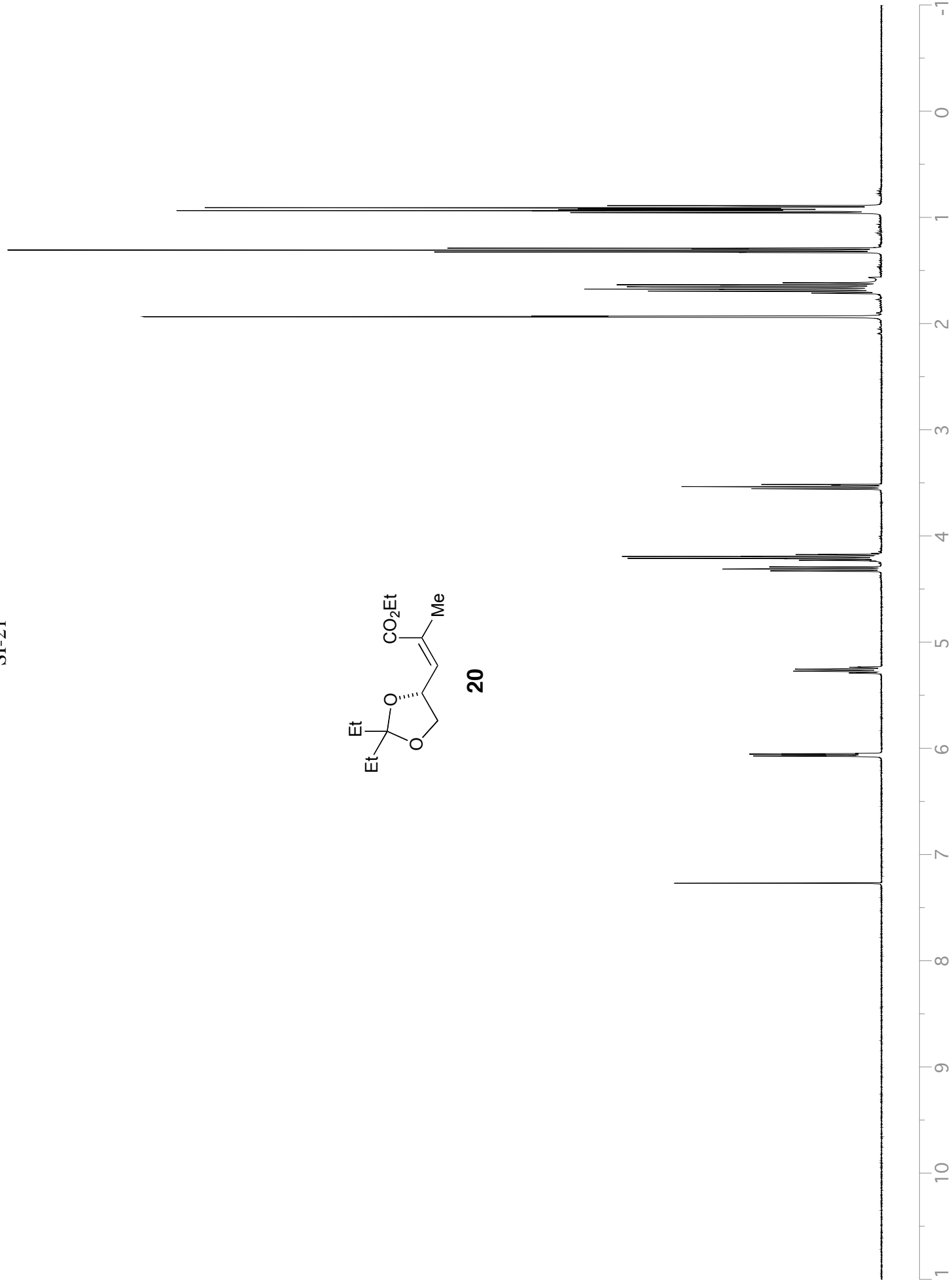
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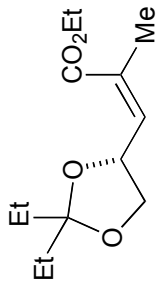
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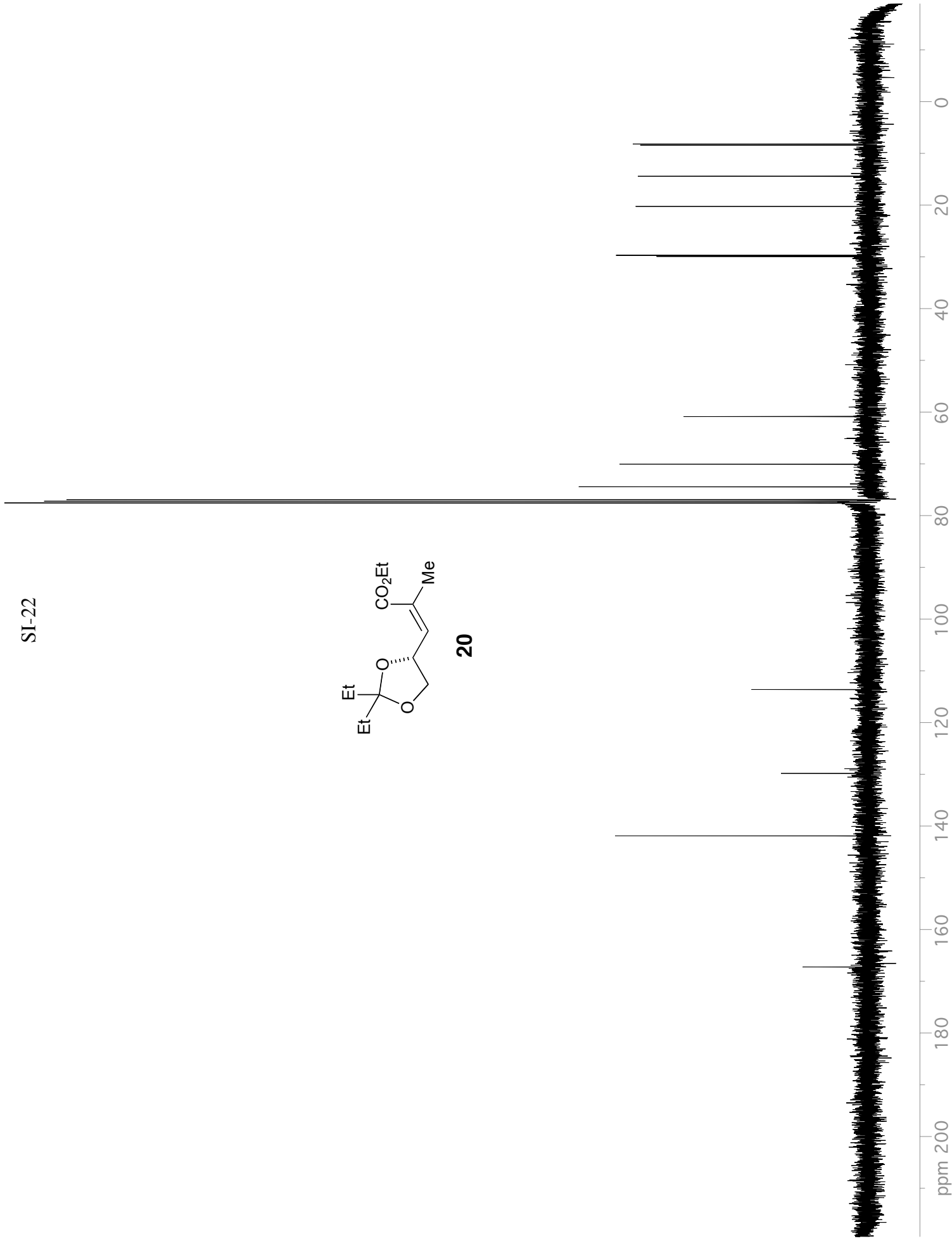
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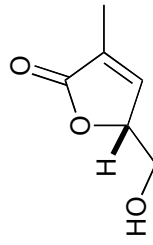
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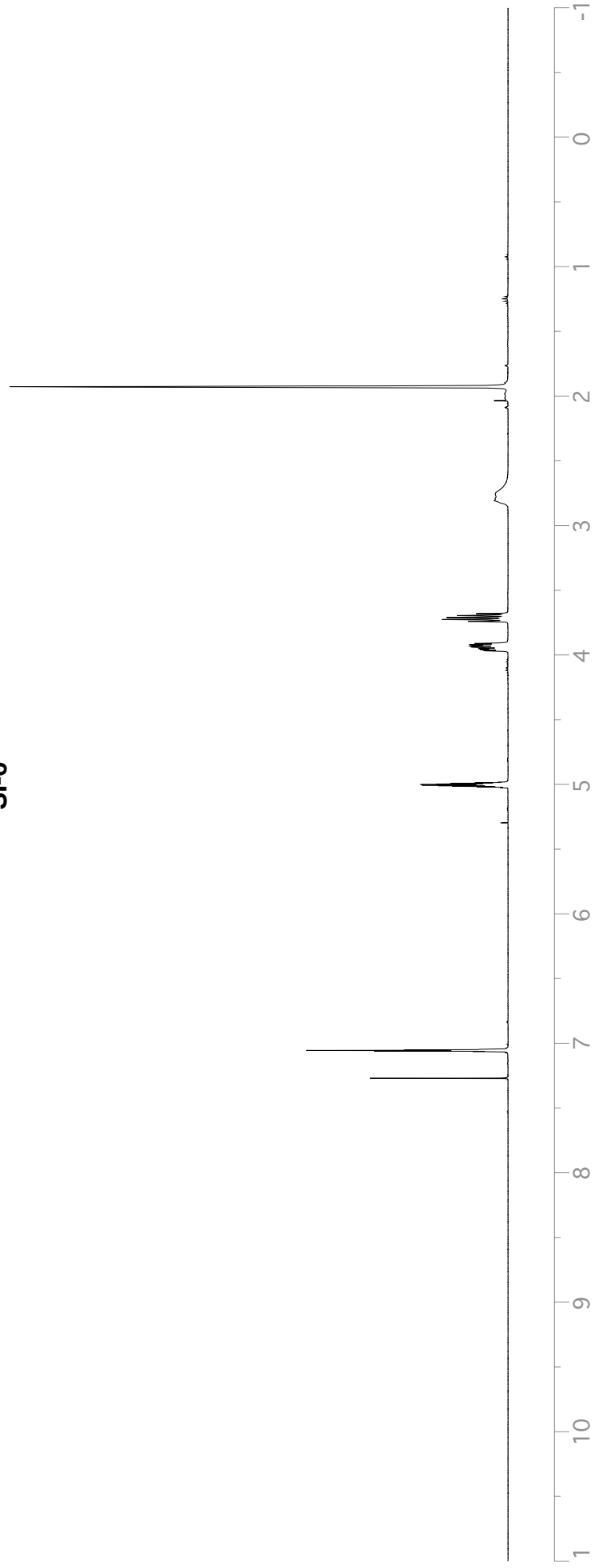
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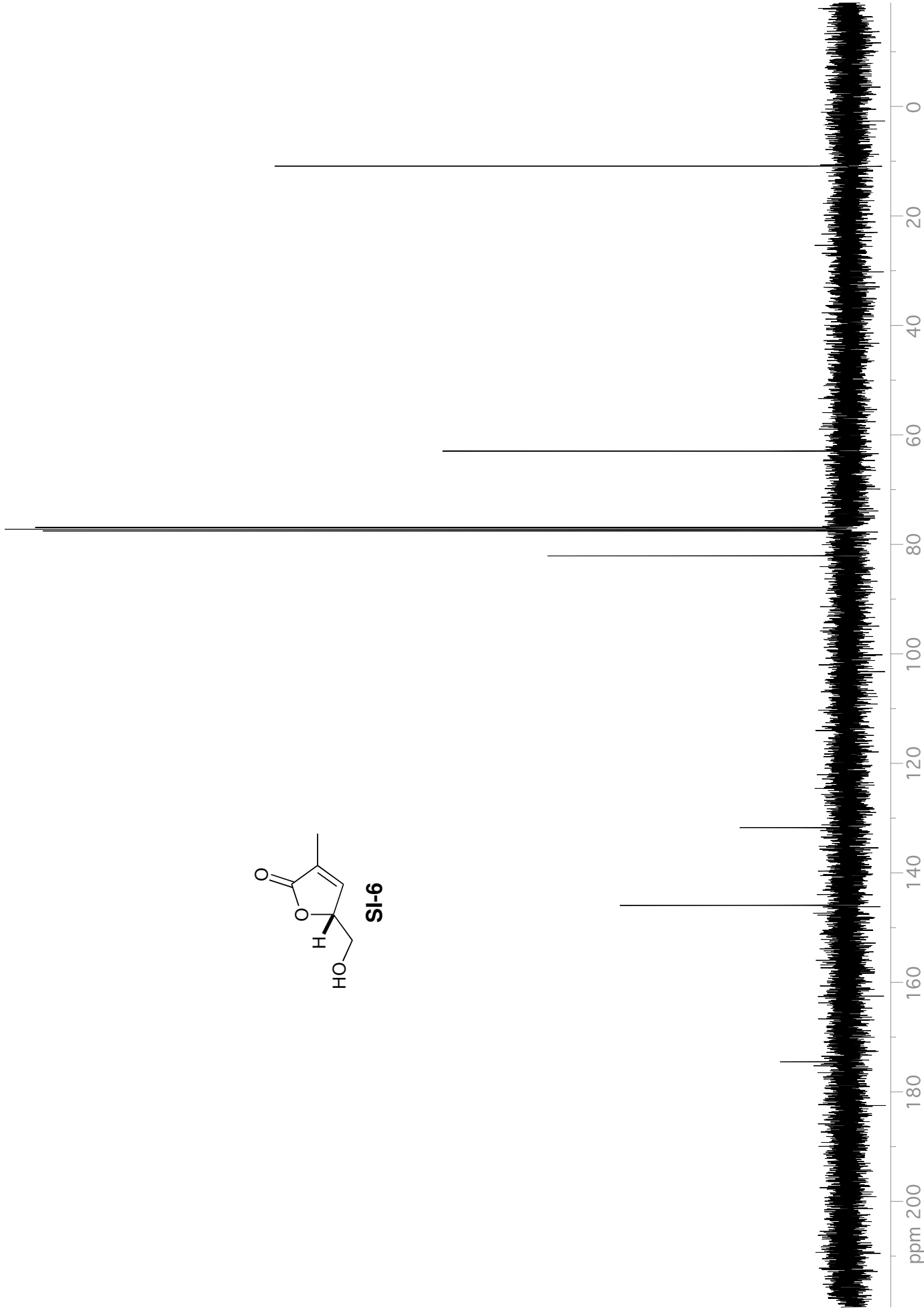
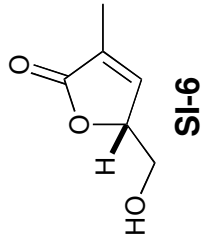
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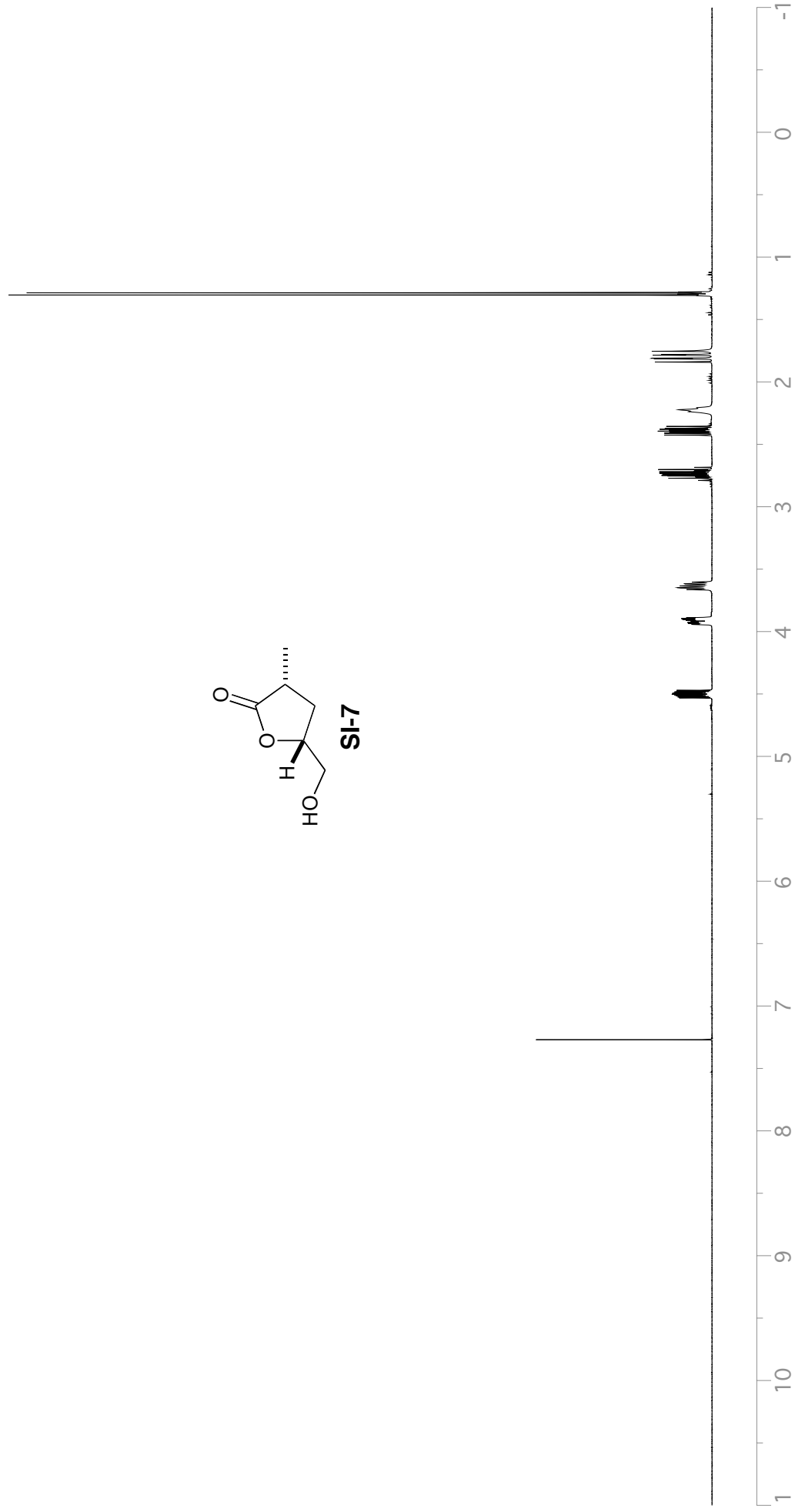
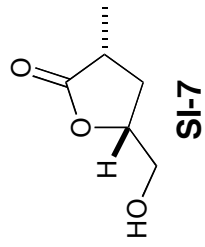
SI-6



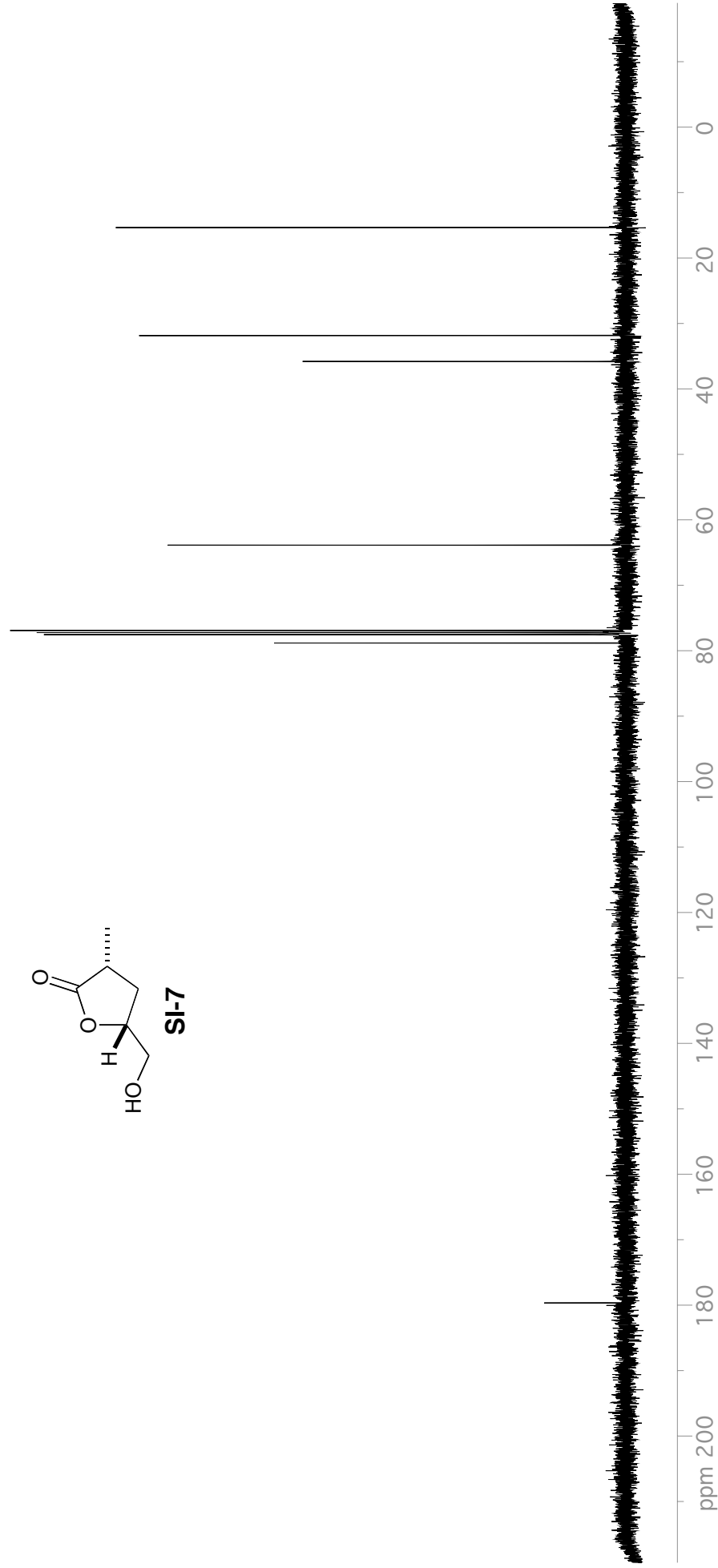
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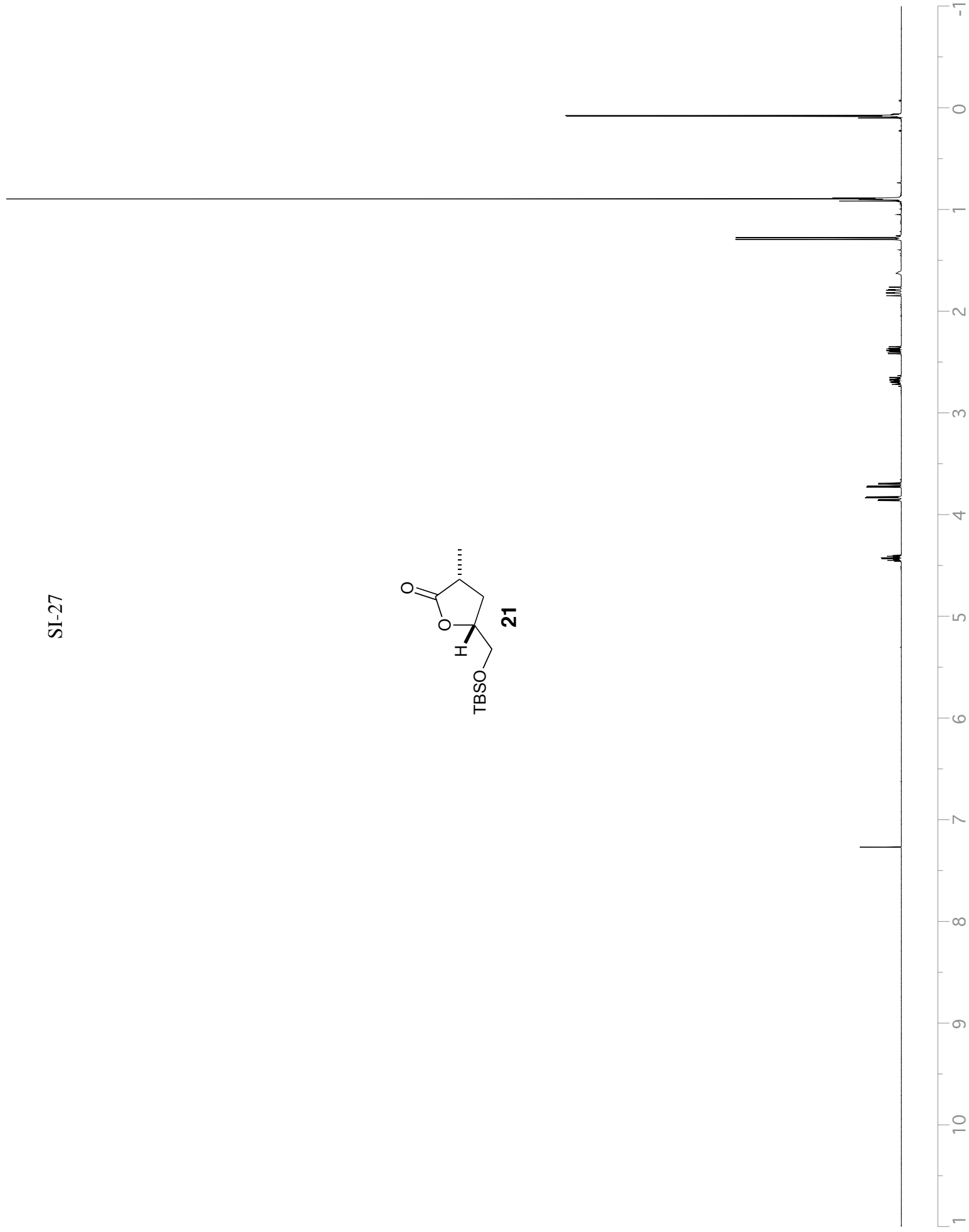
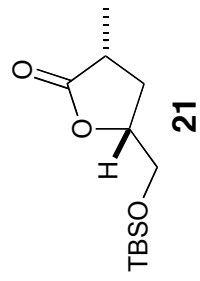
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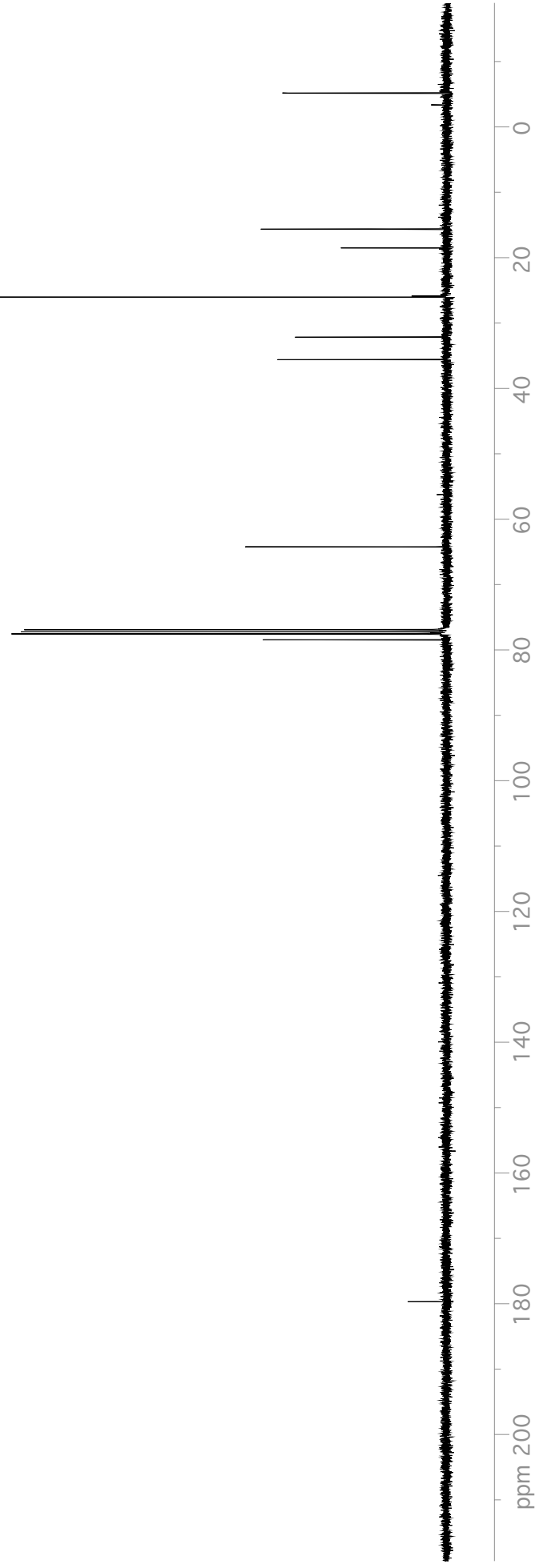
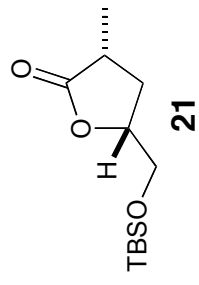
SI-26



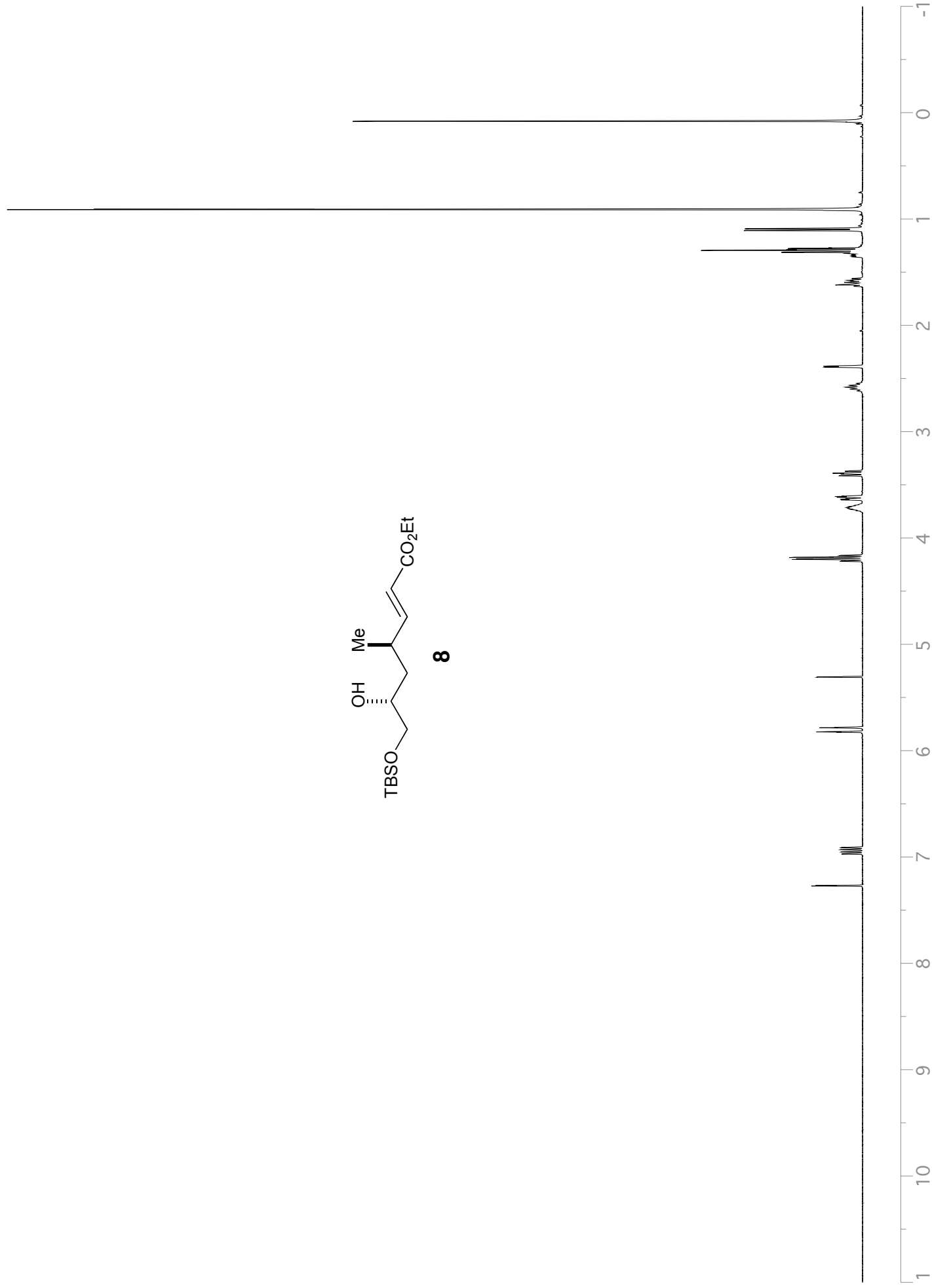
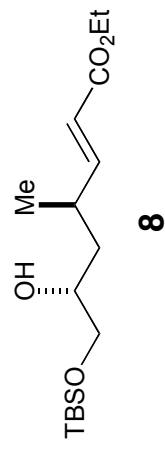
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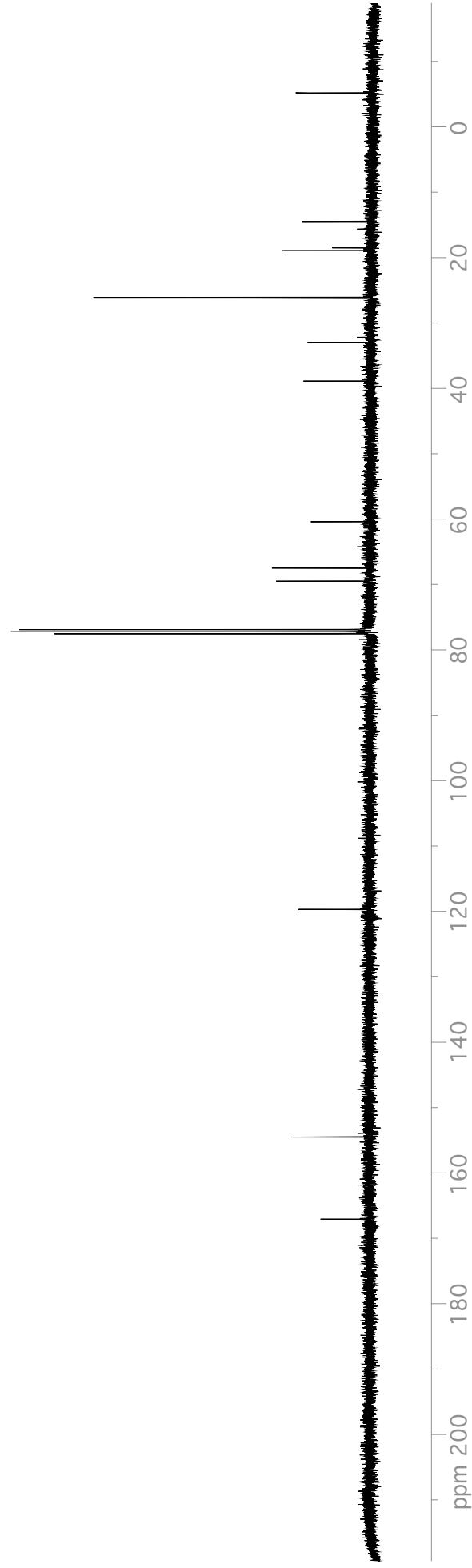
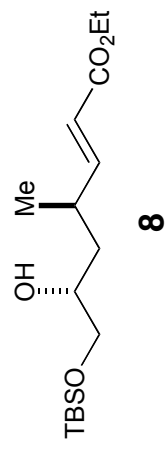
SI-28



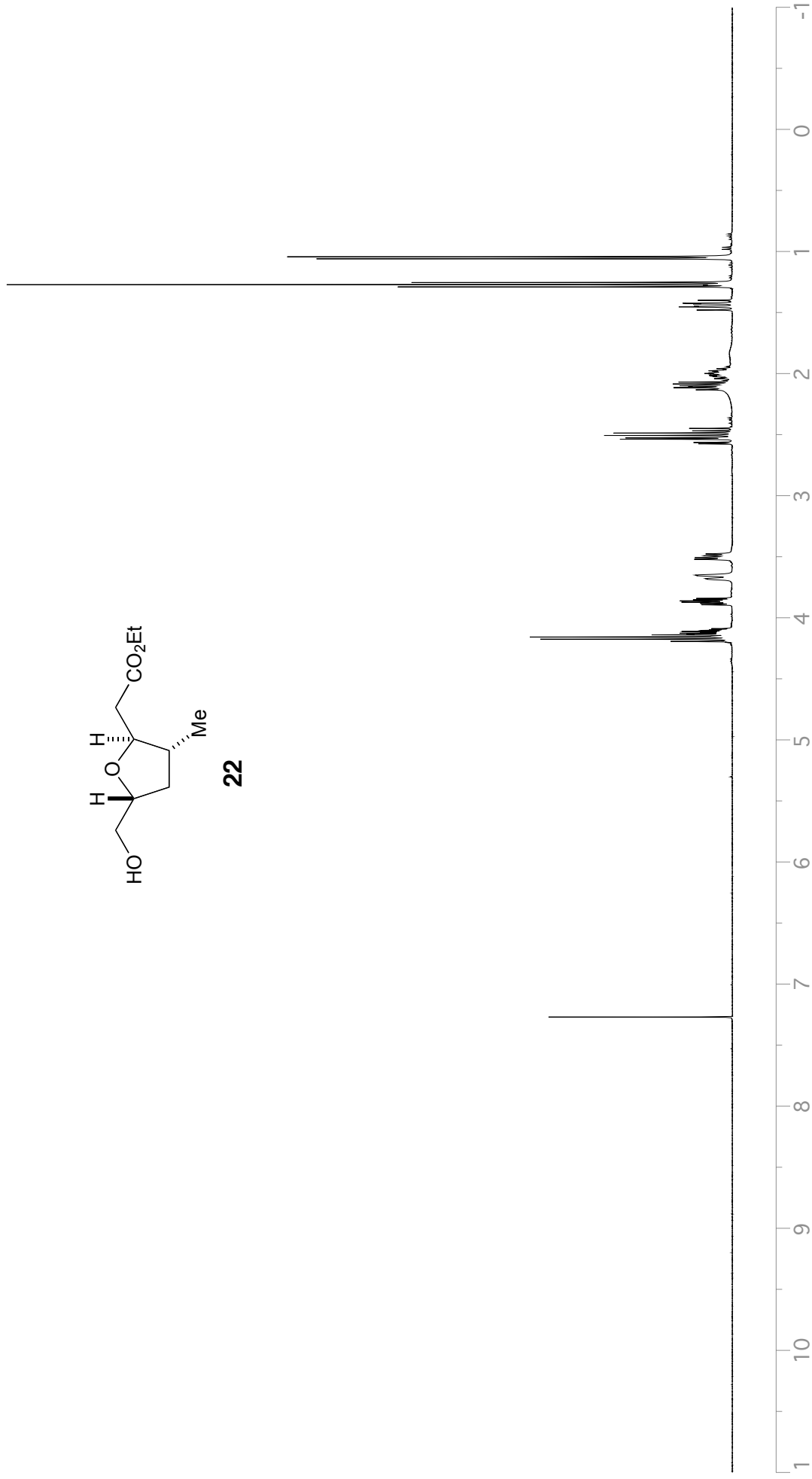
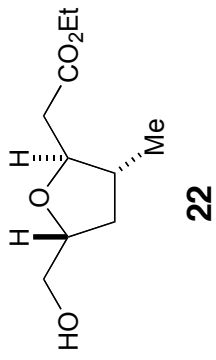
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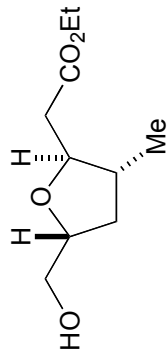
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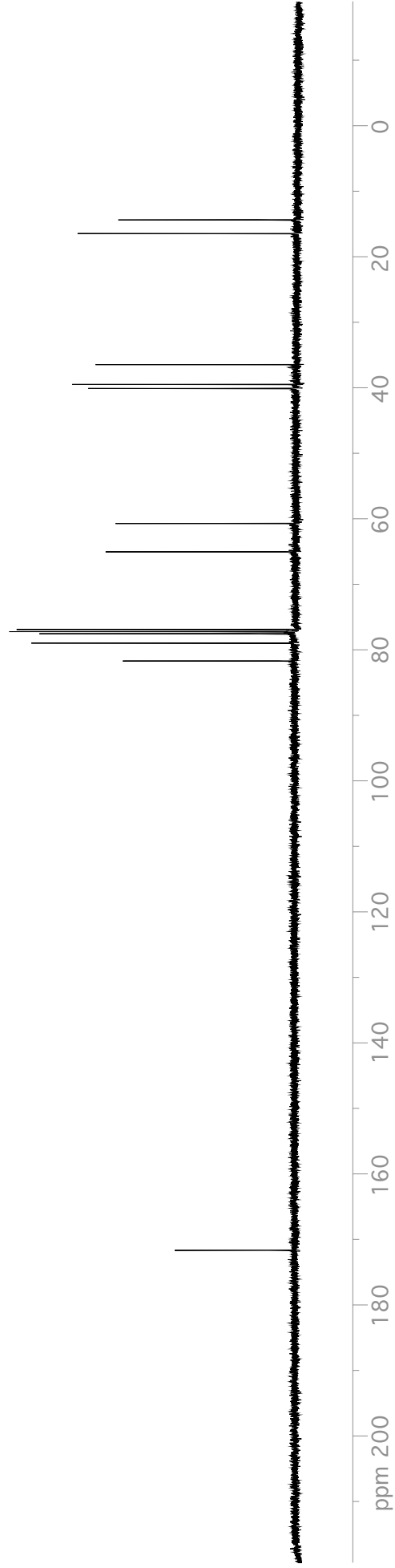
SI-31



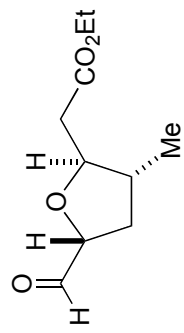
SI-32



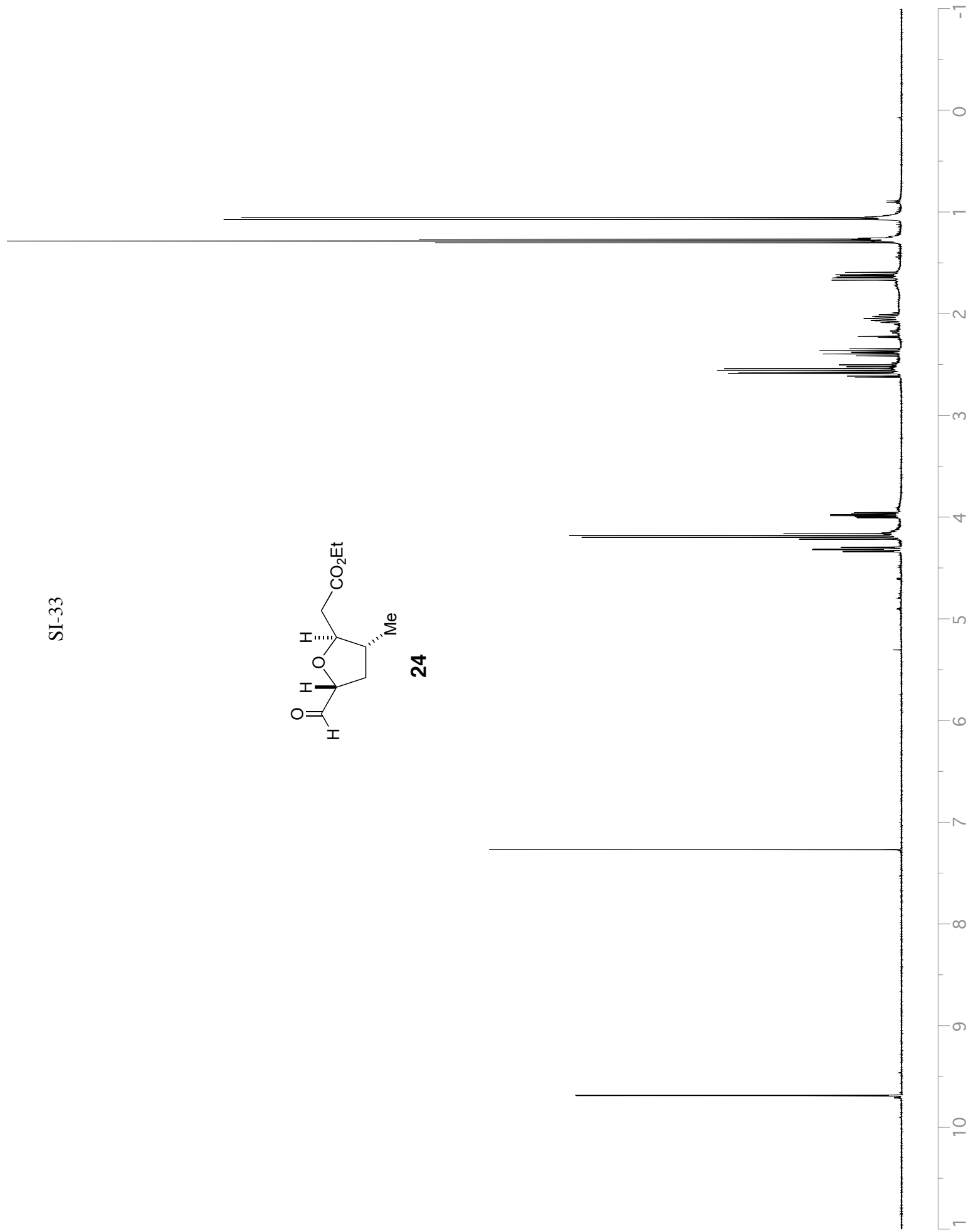
22



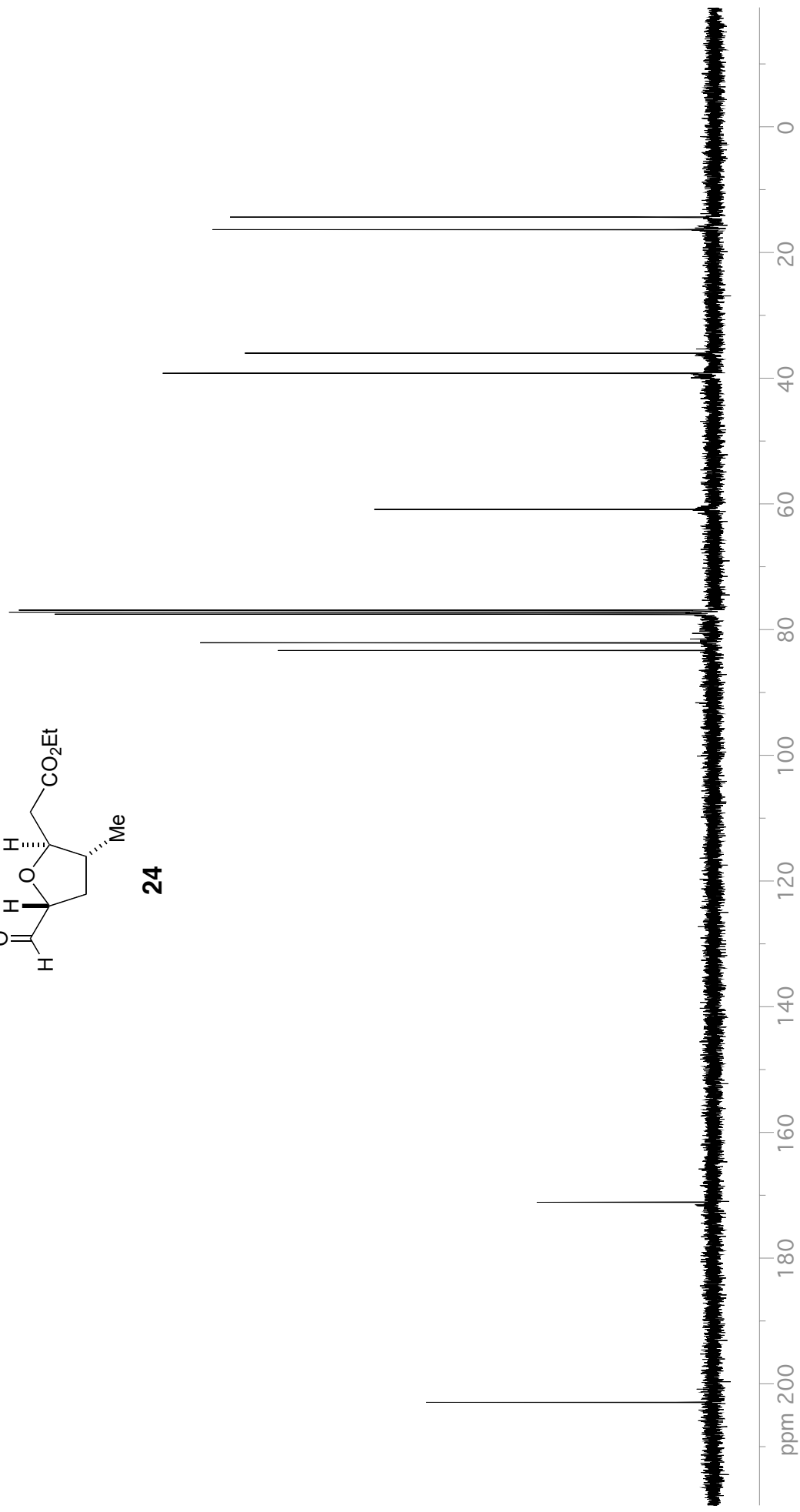
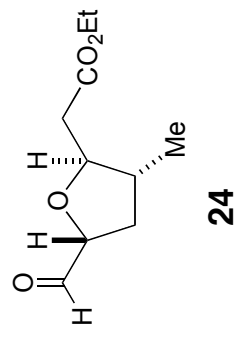
SI-33



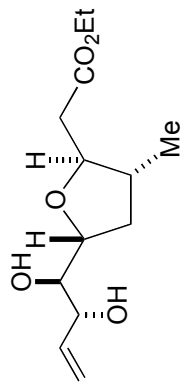
24



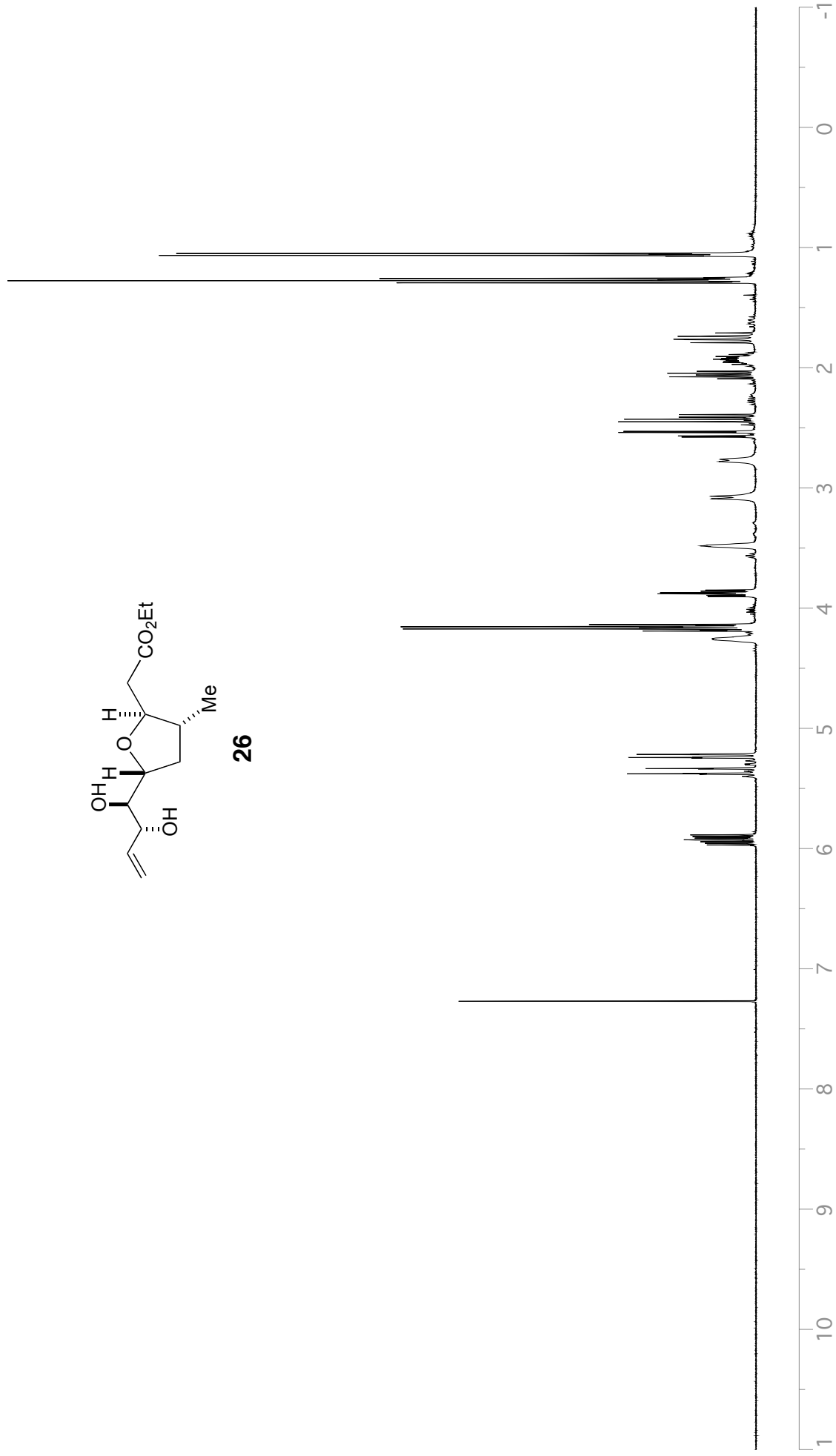
SI-34



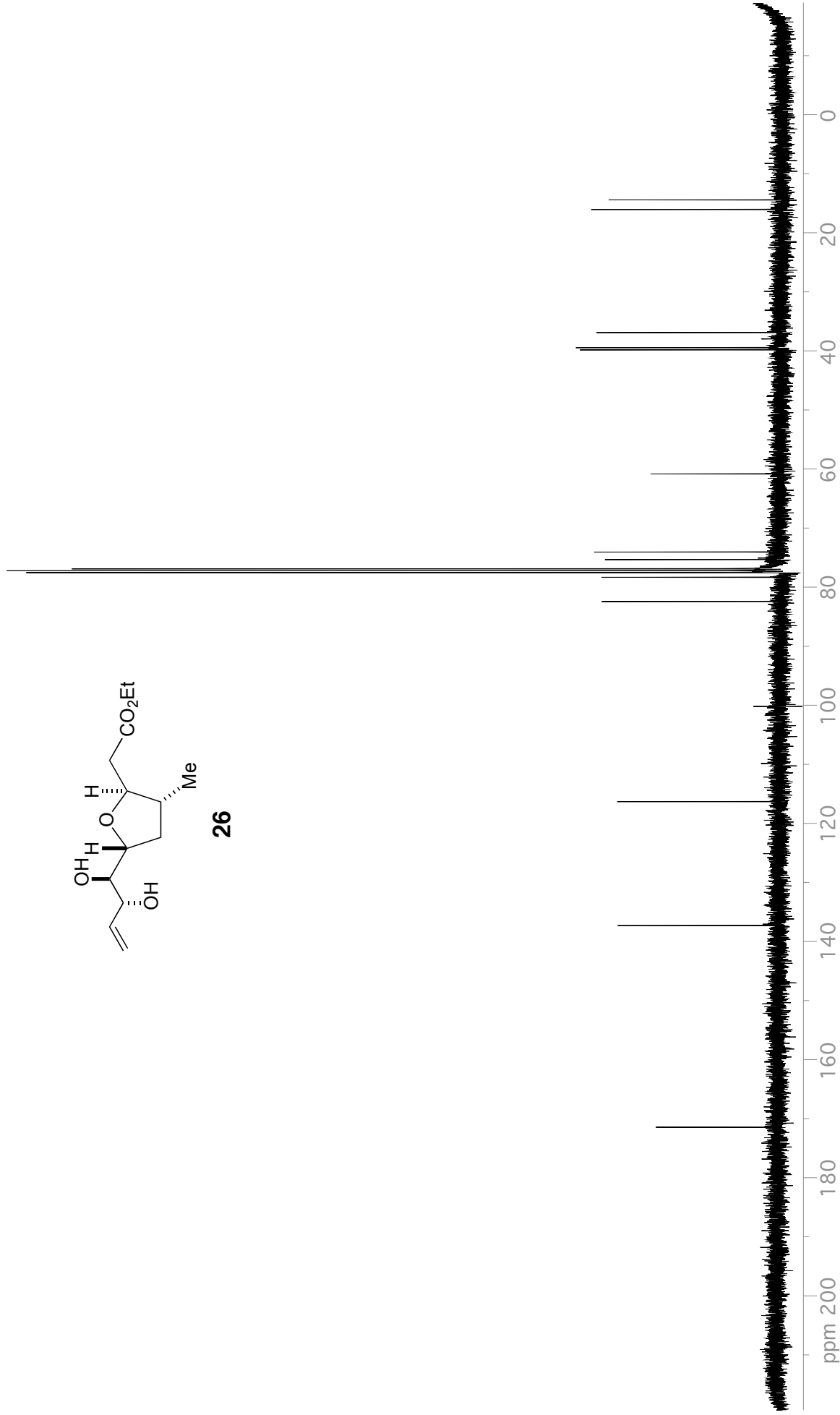
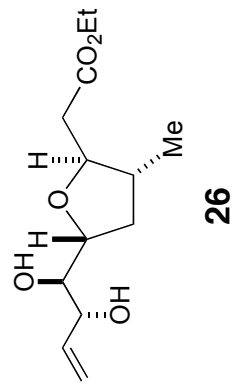
SI-35



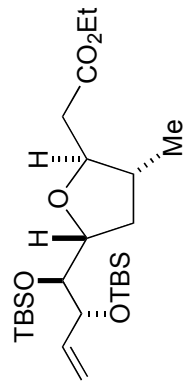
26



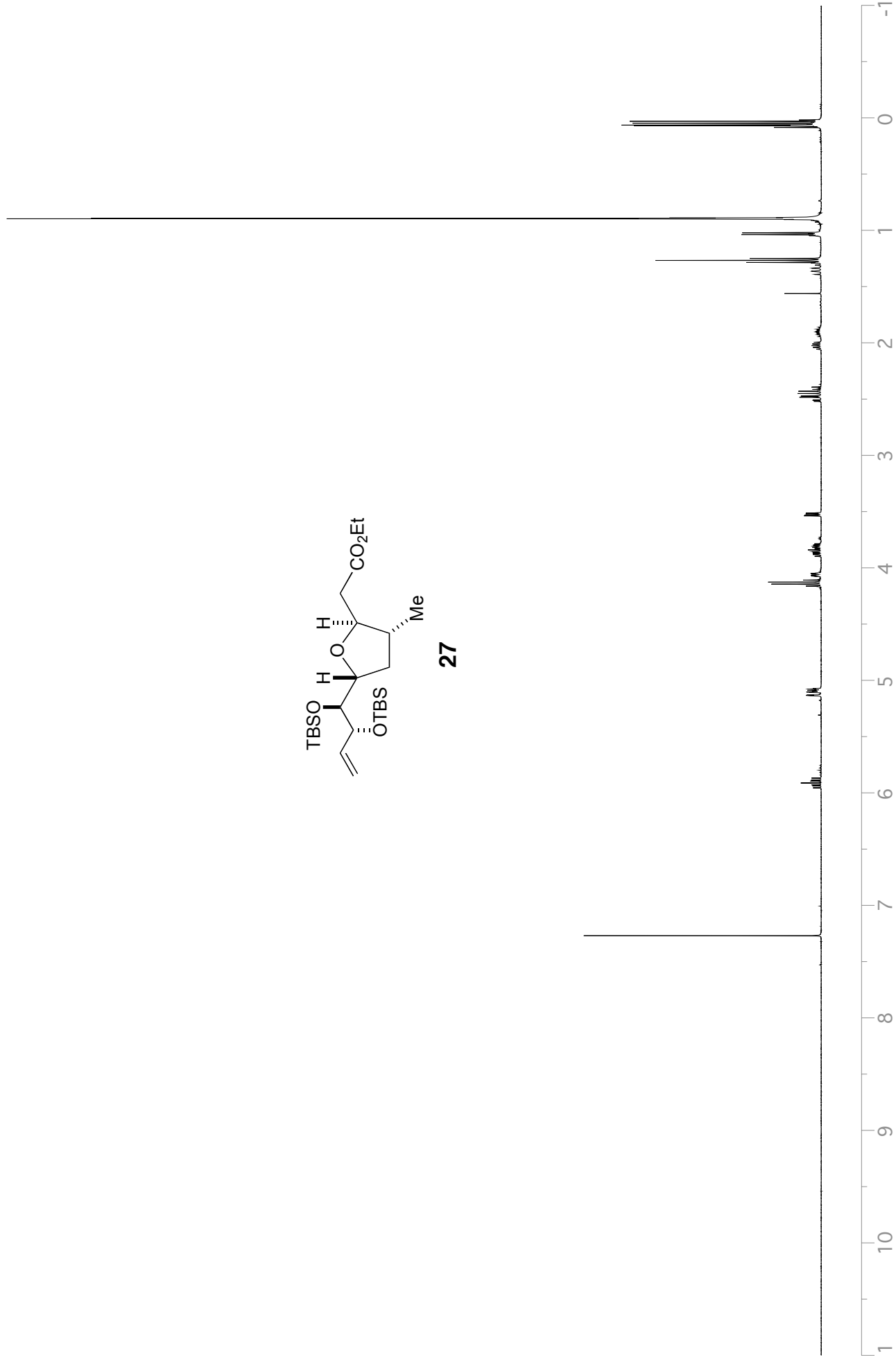
SI-36

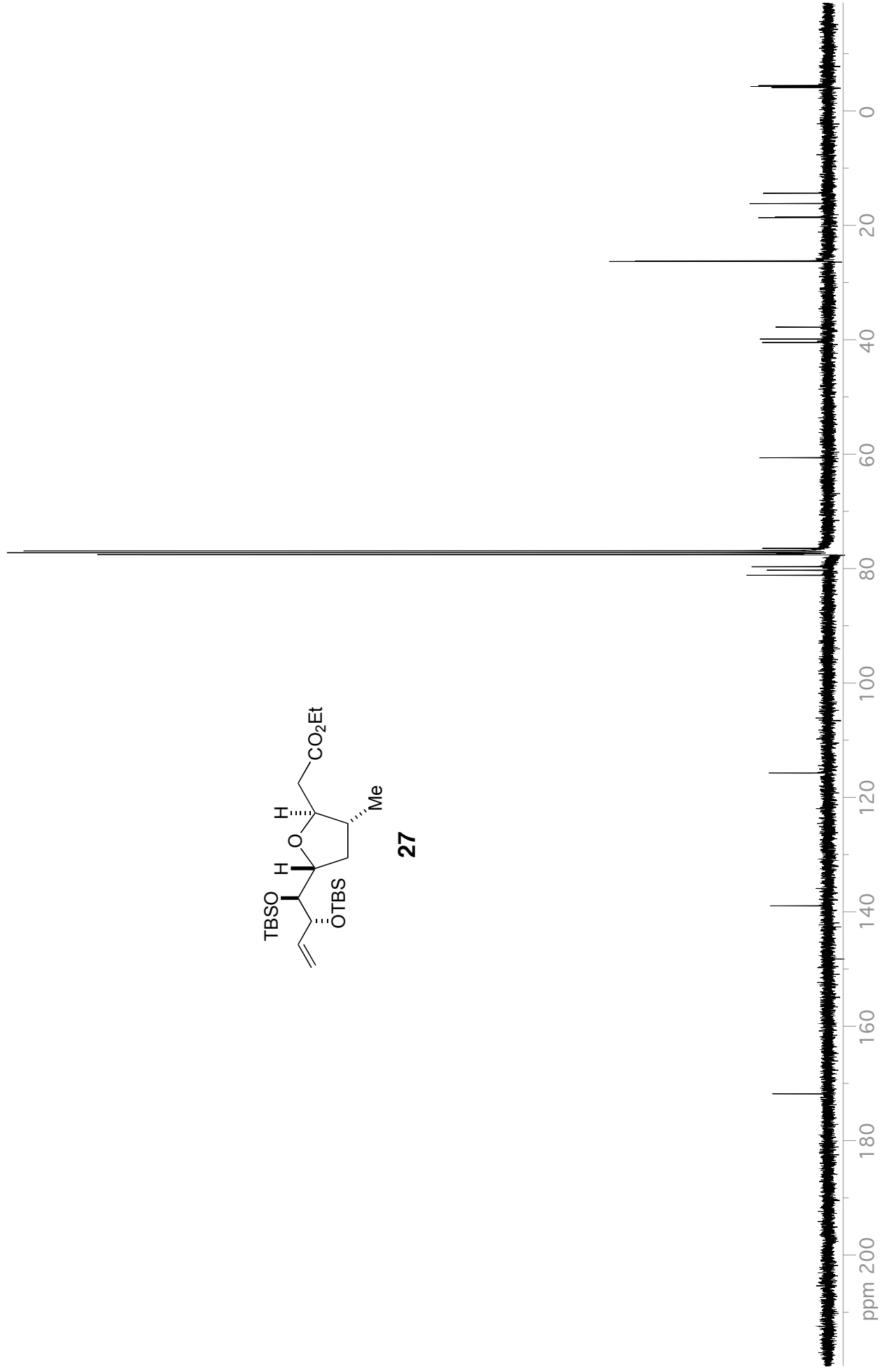
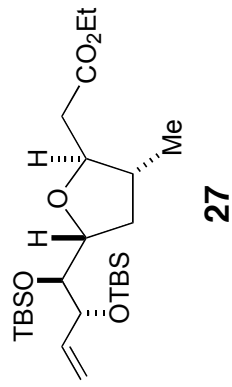


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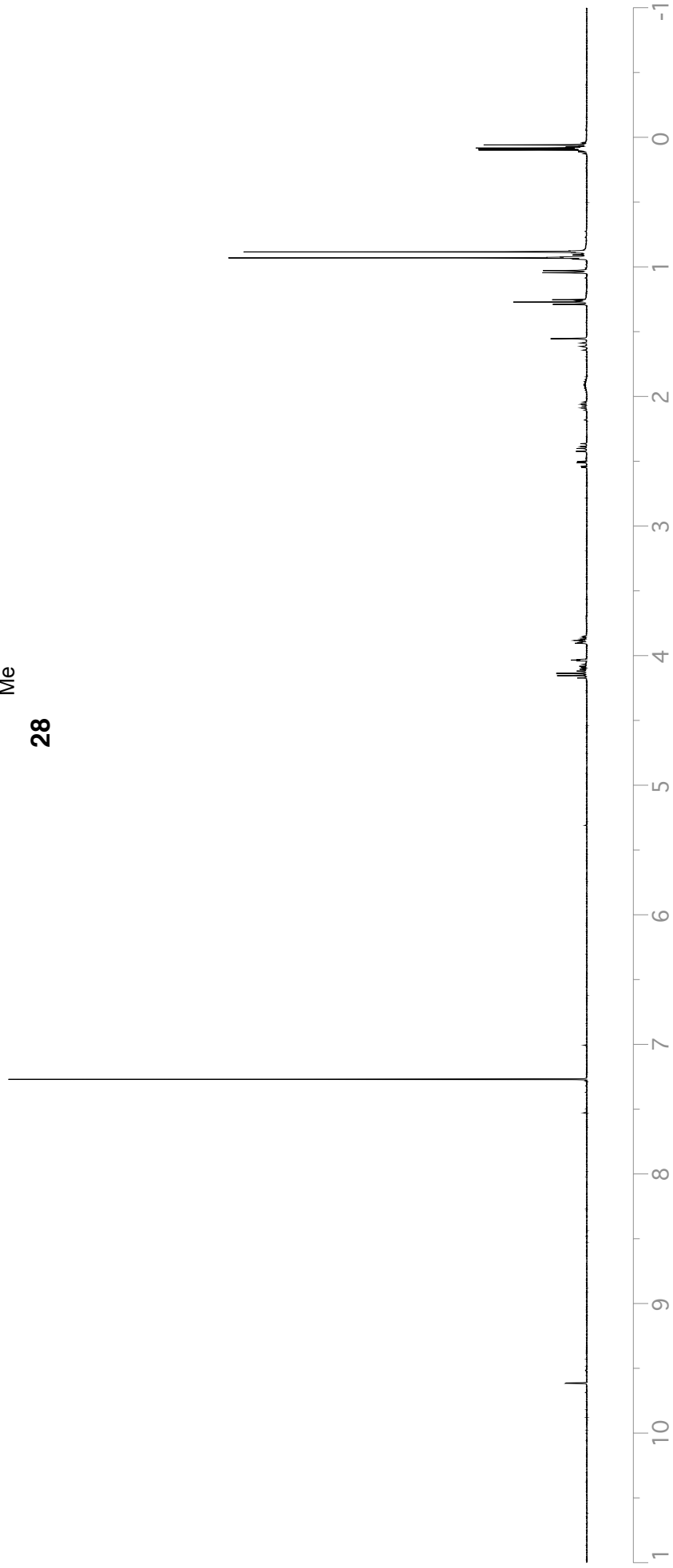
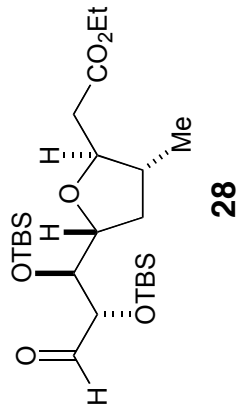


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