

Supporting Information: Spectra

Synthesis of Complex Allylic Esters via C—H Oxidation vs C—C Bond Formation

Nicolaas A. Vermeulen, Jared H. Delcamp and M. Christina White
Roger Adams Laboratory, Department of Chemistry, University of Illinois, Urbana, Illinois 61801

STANDARD PROTON PARAMETERS

Pulse Sequence: s2pul1

Solvent: CDCl3
Ambient temperature
INOVA-500 "u1500hp"

Relax. delay 10.000 sec
Pulse 90.0 degrees
Acq. time 4.096 sec
Width 8000.0 Hz

4 repetitions

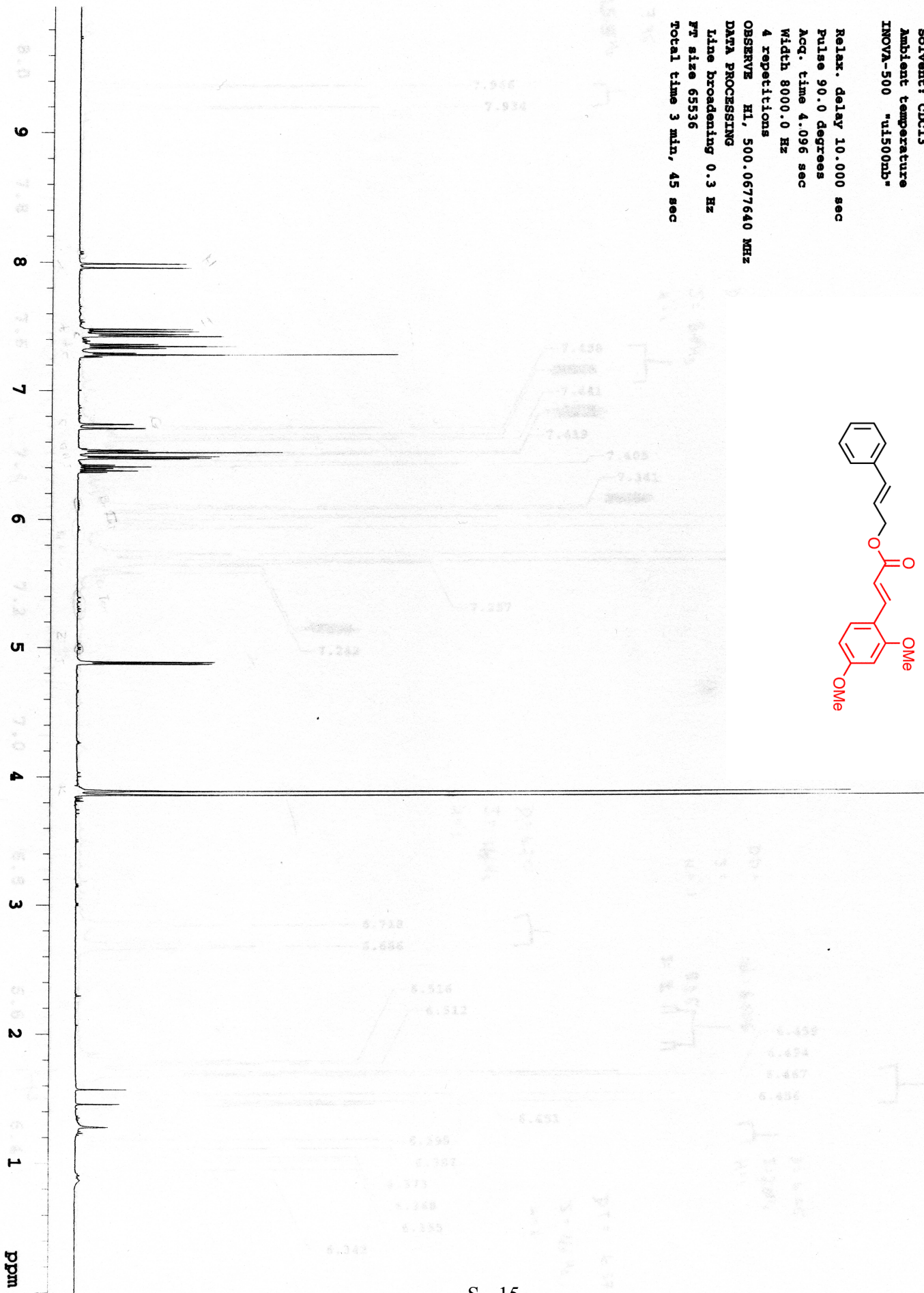
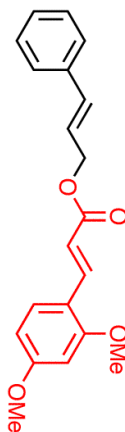
OBSERVE H1, 500.0677640 MHz

DATA PROCESSING

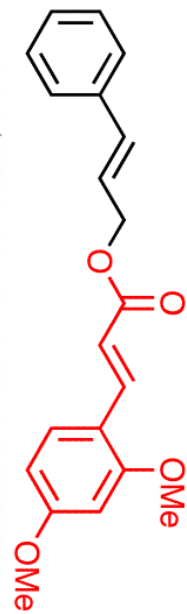
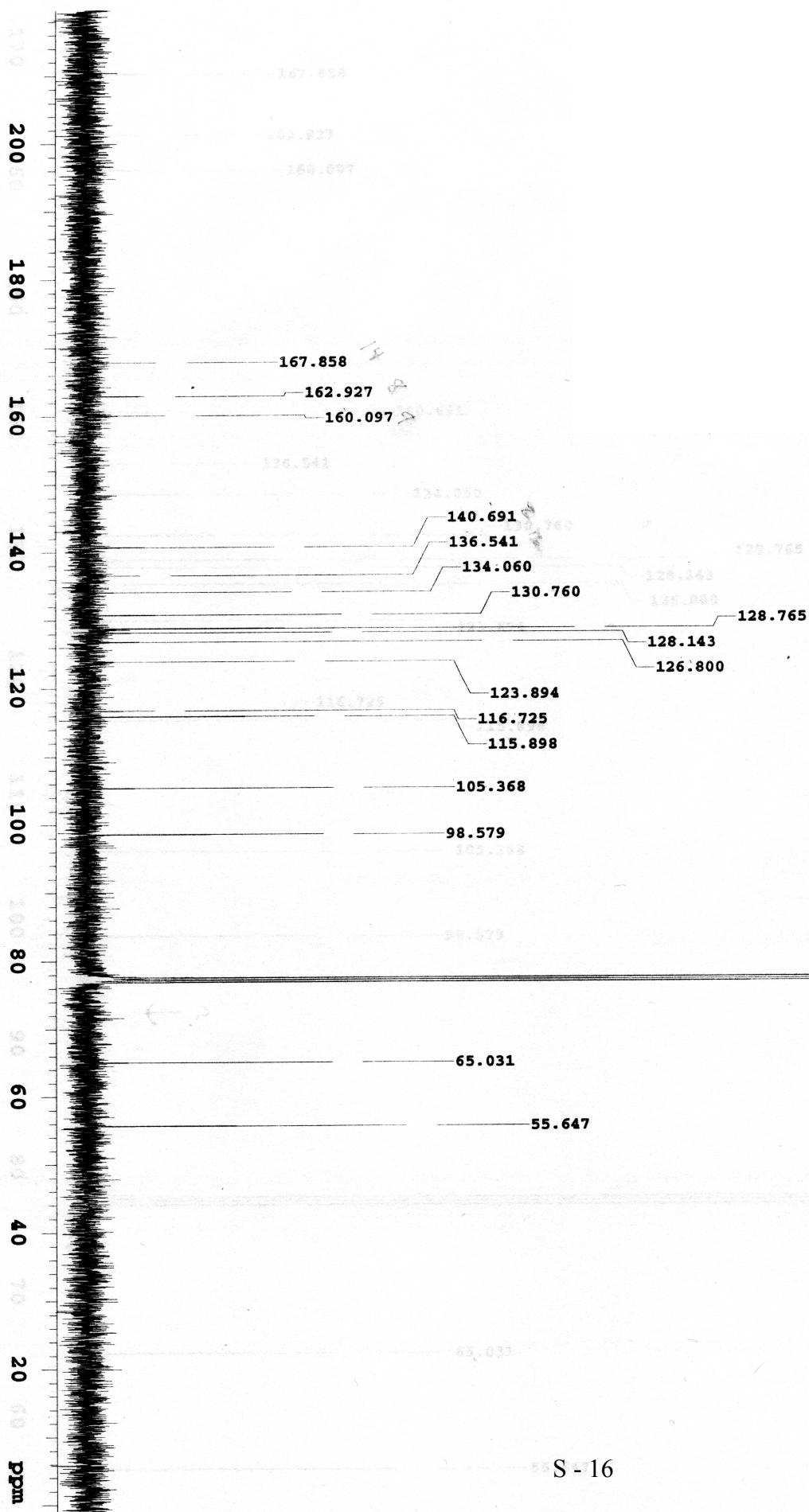
Line broadening 0.3 Hz

FT size 65536

Total time 3 min, 45 sec



Filename: -



Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

UNITY-400 "4400"

Relax. delay 5.000 sec

Pulse 45.0 degrees

Acq. time 4.096 sec

Width 8000.0 Hz

13 repetitions

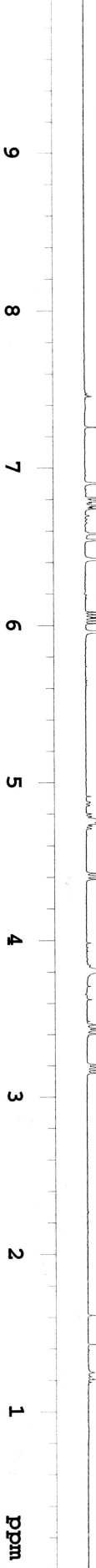
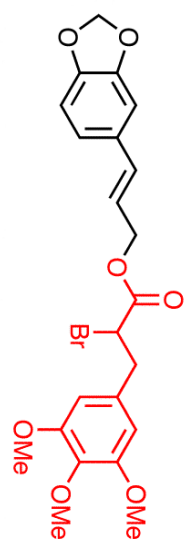
OBSERVE H1, 399.9486787 MHz

DATA PROCESSING

Line broadening 0.3 Hz

Fr size 65536

Total time 9 min, 42 sec



Pulse Sequence: s2pul

Solvent: CDC13
Ambient temperature
UNIT4-400 "u400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.311 sec
Width 25000.0 Hz

256 repetitions

OBSERVE C13, 100.5671512 MHz

DECOUPLE H1, 399.9500118 MHz

Power 48 dB

continuously on

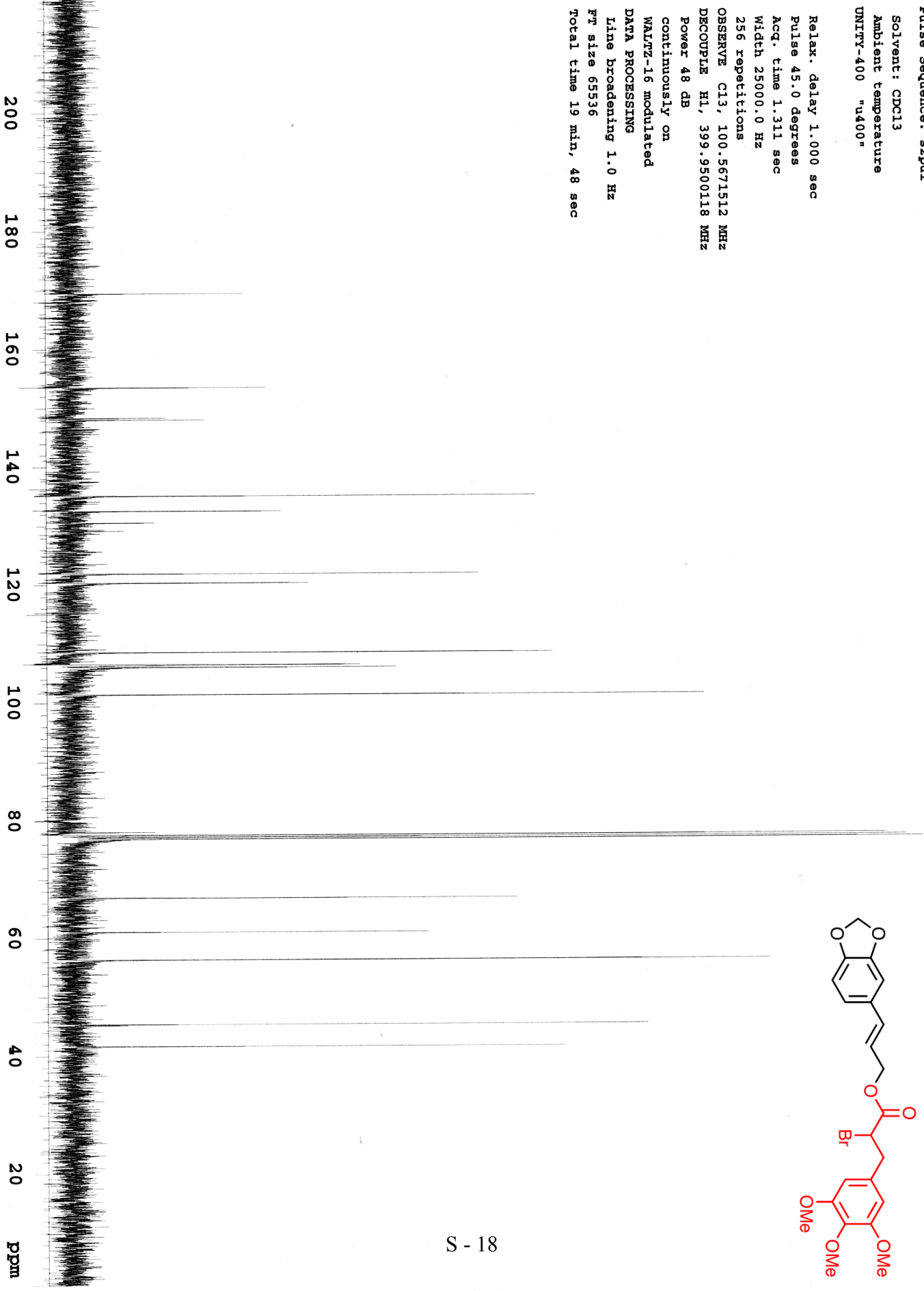
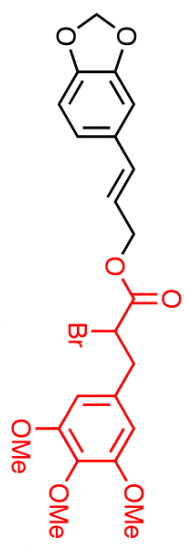
WALTZ-16 modulated

DATA PROCESSING

line broadening 1.0 Hz

FT size 65536

Total time 19 min, 48 sec



NVX136.colummed

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

Operator: userld

INOVA-400 "u400"

Relax. delay 10.000 sec

Pulse 49.2 degrees

Acq. time 4.096 sec

Width 8000.0 Hz

5 repetitions

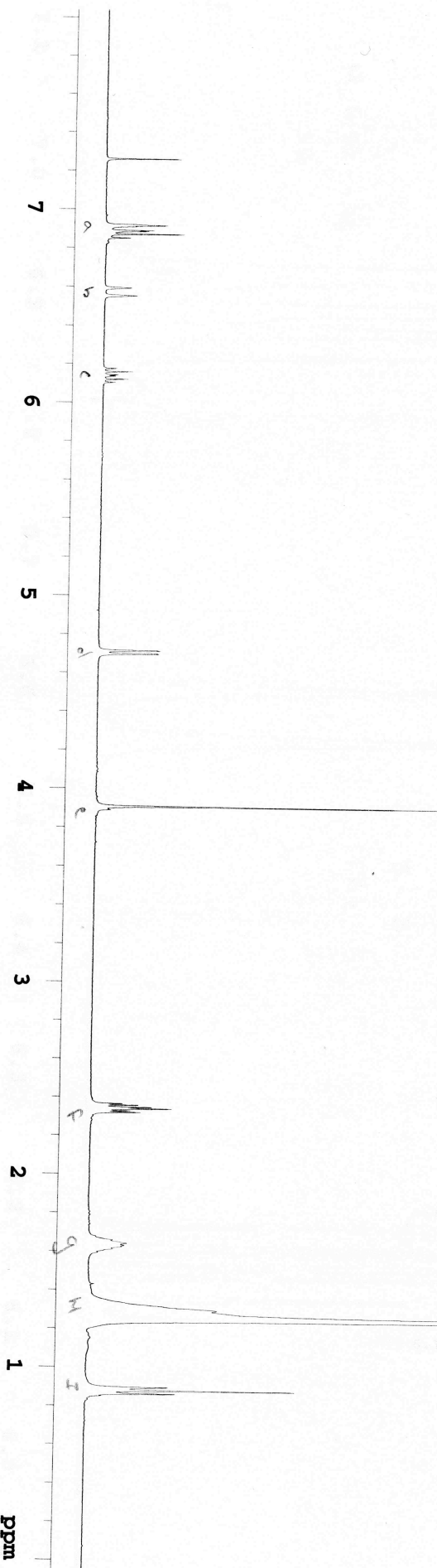
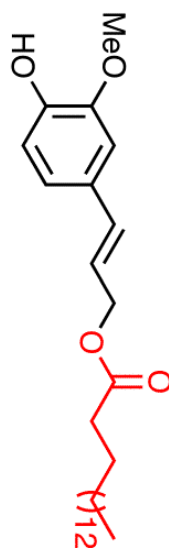
OBSERVE H1, 399.9454252 MHz

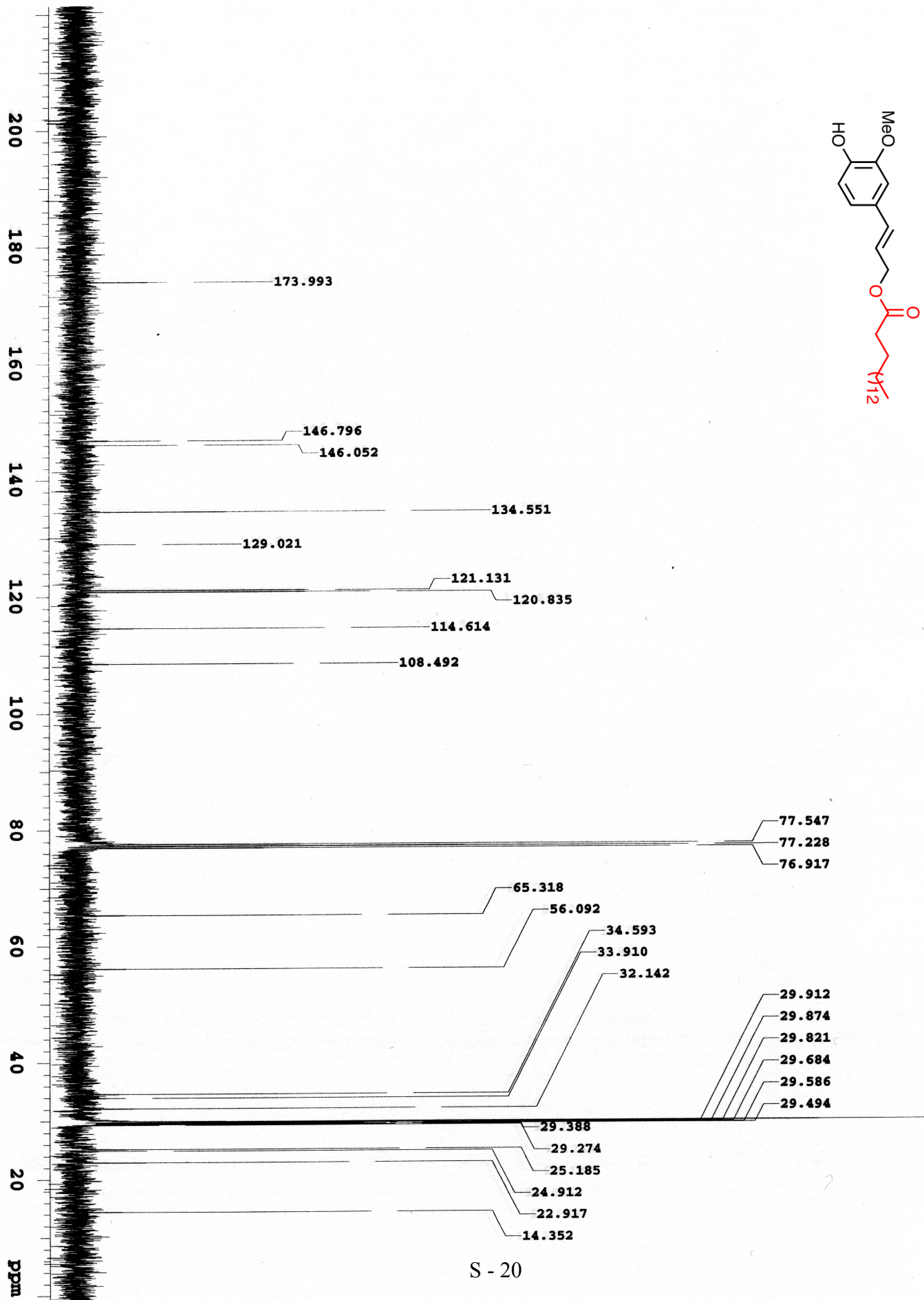
DATA PROCESSING

Line broadening 0.3 Hz

FT size 65536

Total time 7 min, 31 sec





Pulse Sequence: s2pul1

Solvent: CDCl3

Ambient temperature

INOVA-500 "ui500nb"

Relax. delay 10.000 sec

Pulse 90.0 degrees

Acq. time 4.096 sec

Width 8000.0 Hz

4 repetitions

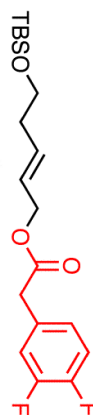
OBSERVE H1, 500.0677728 MHz

DATA PROCESSING

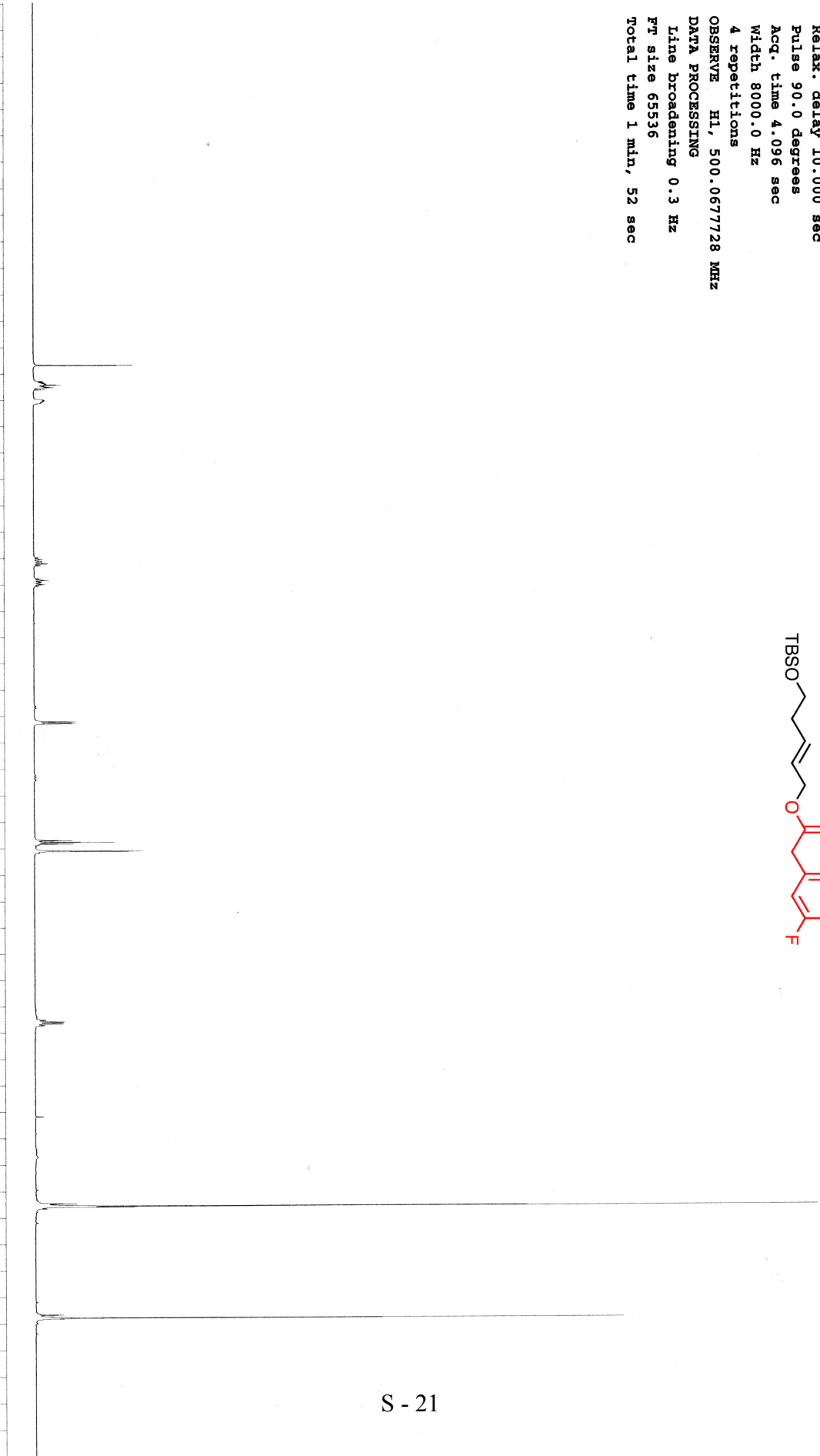
Line Broadening 0.3 Hz

FT size 65536

Total time 1 min, 52 sec



9 8 7 6 5 4 3 2 1 -0 ppm



Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

User: 1-14-87

UNITY-500 "u500"

Relax. delay 3.000 sec

Pulse 63.5 degrees

Acq. time 1.086 sec

Width 30165.9 Hz

448 repetitions

OBSERVE C13, 125.6487514 MHz

DECOUPLE H1, 499.6981123 MHz

Power 40 dB

continuously on

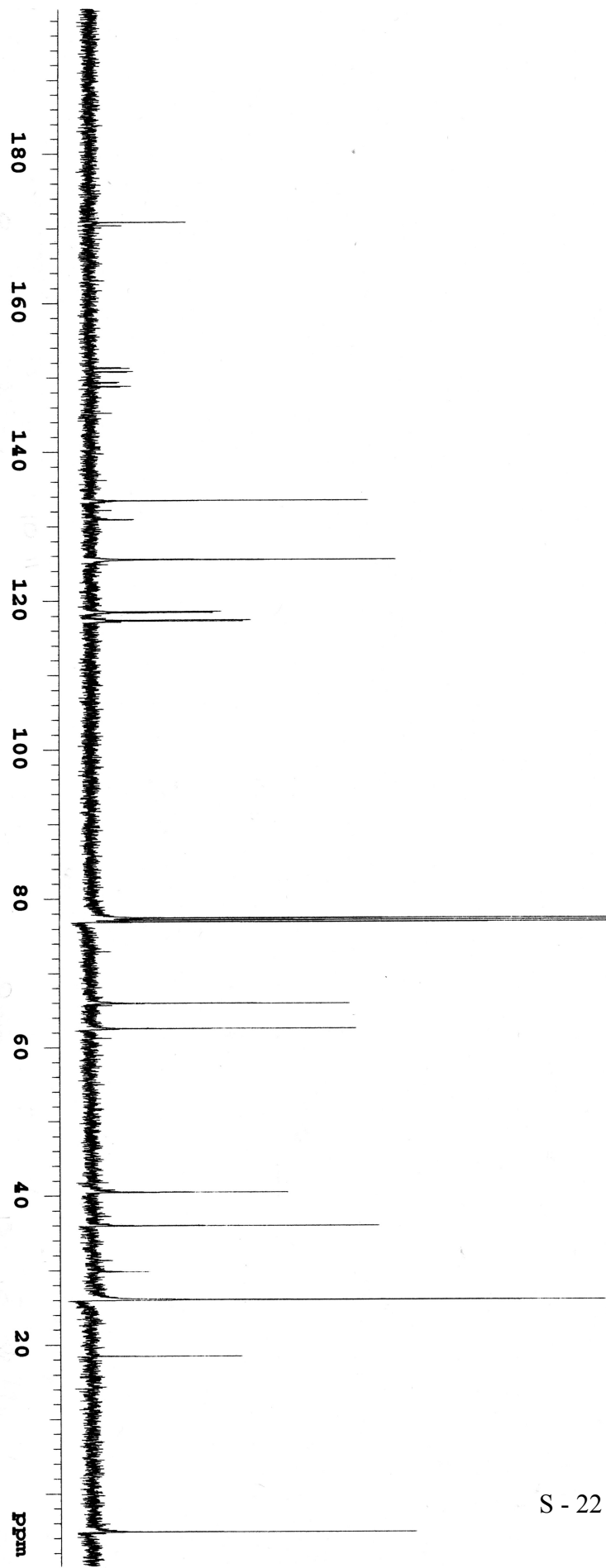
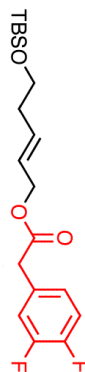
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

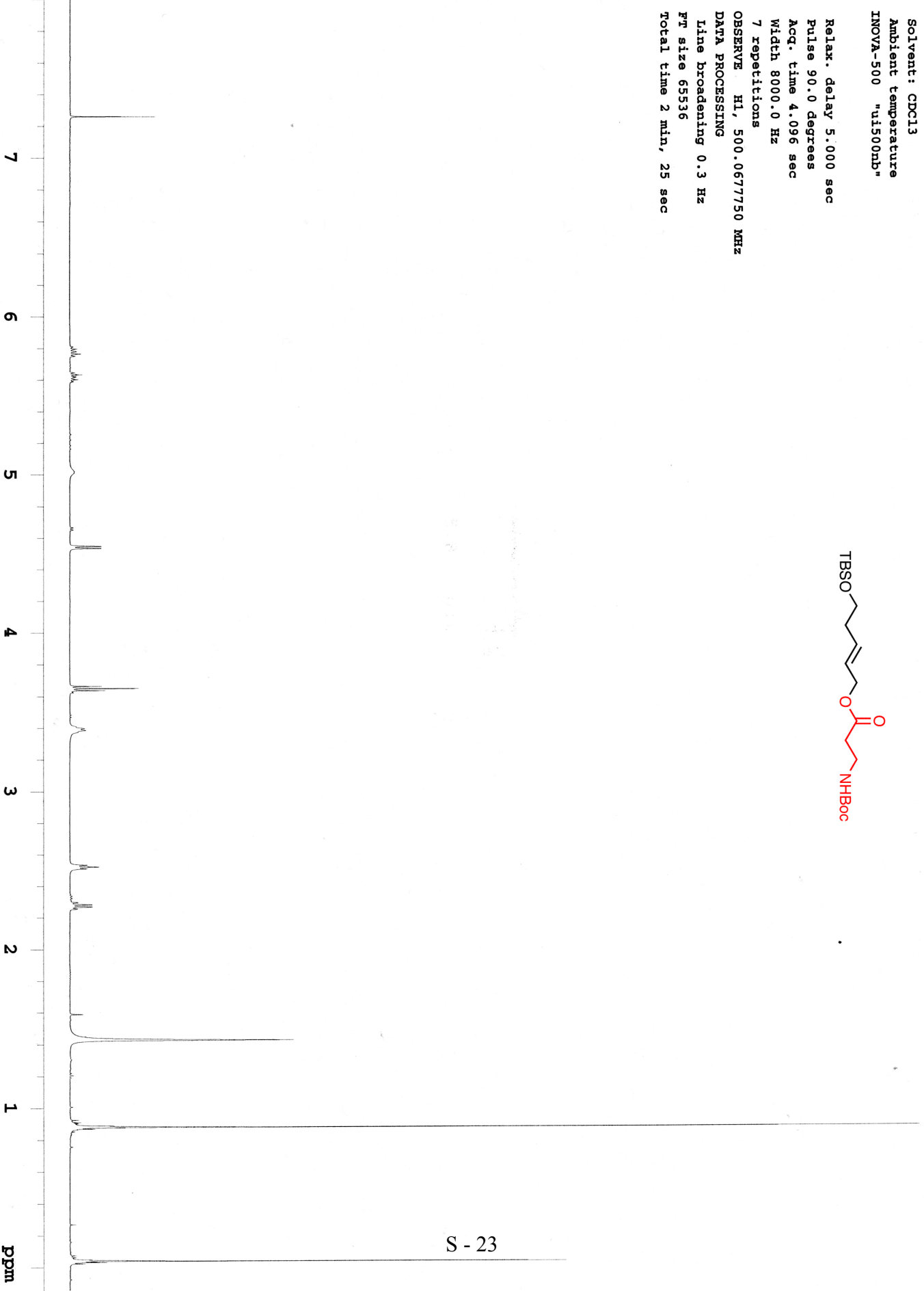
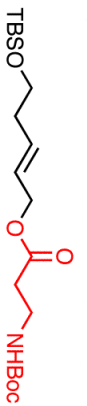
FT size 65536

Total time 35 min, 1 sec



Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "ui500nb"

Relax. delay 5.000 sec
Pulse 90.0 degrees
Acq. time 4.096 sec
Width 8000.0 Hz
7 repetitions
OBSERVE H1, 500.0677750 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 2 min, 25 sec



13C OBSERVE

Filename: -

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
UNITRY-400 "1400"

Relax. delay 4.000 sec

Pulse 60.0 degrees

Acq. time 1.311 sec

Width 25000.0 Hz

656 repetitions

OBSERVE C13, 100.5671512 MHz

DECOUPLE H1, 399.9500118 MHz

Power 48 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

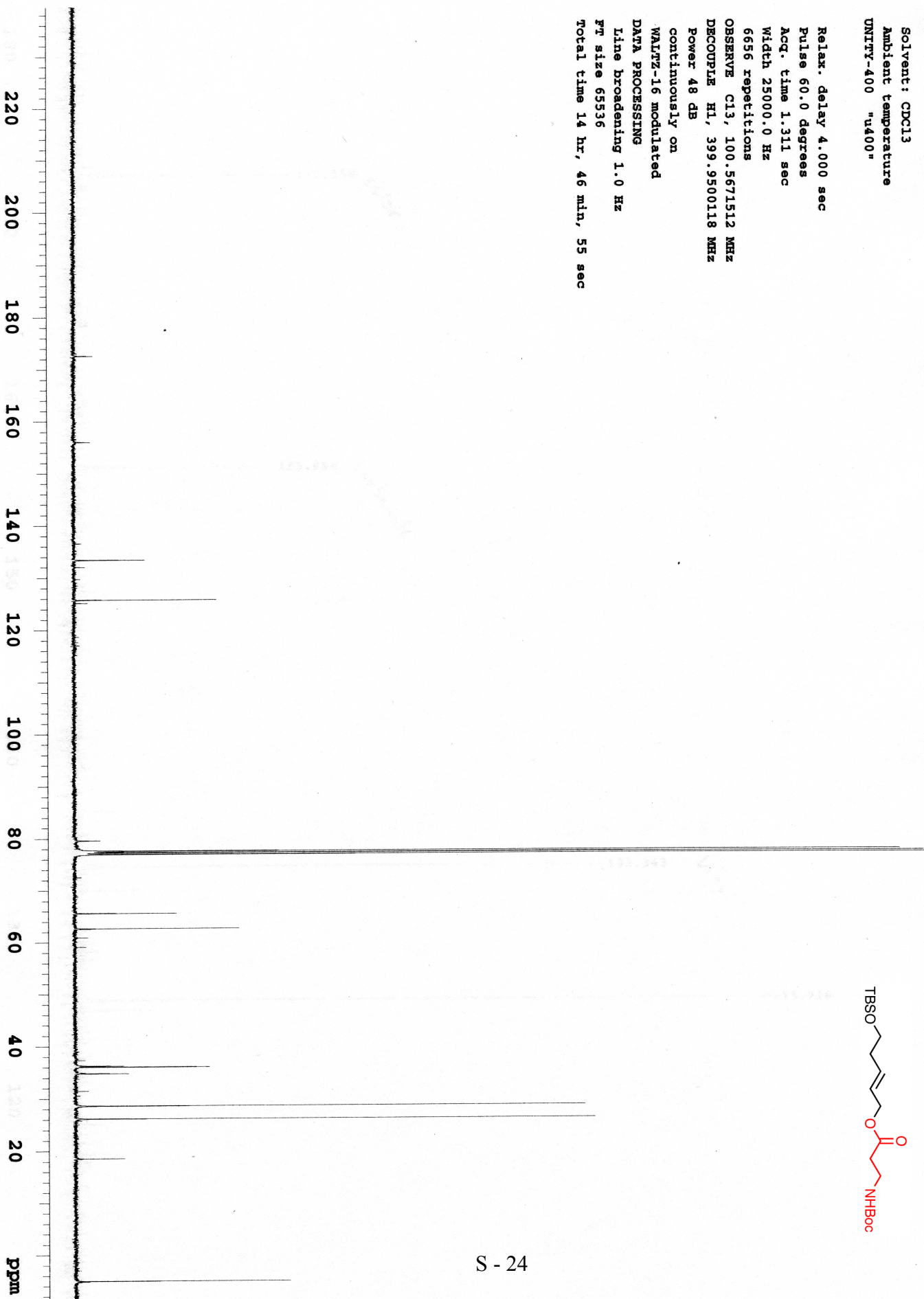
Line broadening 1.0 Hz

PT size 65536

Total time 14 hr, 46 min, 55 sec



S - 24



Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

UNITTY-400 "u400"

Relax. delay 5.000 sec

Pulse 45.0 degrees

Acq. time 4.096 sec

Width 8000.0 Hz

3 repetitions

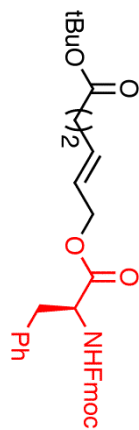
OBSERVE H1, 399.9486787 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FW size 65536

Total time 2 min, 25 sec



9 8 7 6 5 4 3 2 1 ppm

date Feb 28 2008 dn H1
 solvent CDC13 dof 0
 file /export/home/~ dm nm
 data/u400/White/ve- dmm c
 rmeulen/NVTI.Subs- dmf 200
 trale.B.Version2.f~ dpwr 20

ACQUISITION id lb PROCESSING
 sfreq 399.951 wfFile 0.30

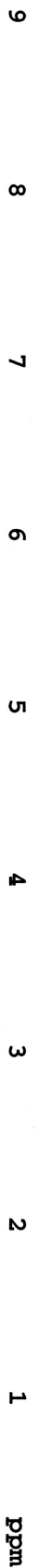
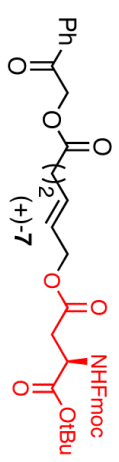
tn H1 proc ft
 at 4.096 fn not used
 np 65536 math f

sw 8000.0 weir
 fb 4400 wepp
 bs 1 wepp
 tpr 63 wbs

pw 7.8 wnt
 dl 10.000 DISPLAY
 tof -425.7 sp -13.8

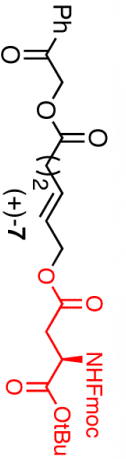
nt 32 wp 4027.8
 ct 32 vs 151
 alock n sc 0

gain not used wc 250
 FLAGS hzmm 16.11
 il n is 390.94
 in n rfl 4903.2
 dp y rfp 2903.6
 hs nm th 24
 ins 1.000
 nm ph



13C OBSERVE

Filename: -



Pulse Sequence: s2pul1

Solvent: CDCl3

Ambient temperature

UNITY-400 "u400"

Relax. delay 2.000 sec

Pulse 60.1 degrees

Acq. time 1.311 sec

Width 25000.0 Hz

944 repetitions

OBSERVE C13, 100.5671512 MHz

DECOUPLE H1, 399.9500118 MHz

Power 48 dB

continuously on

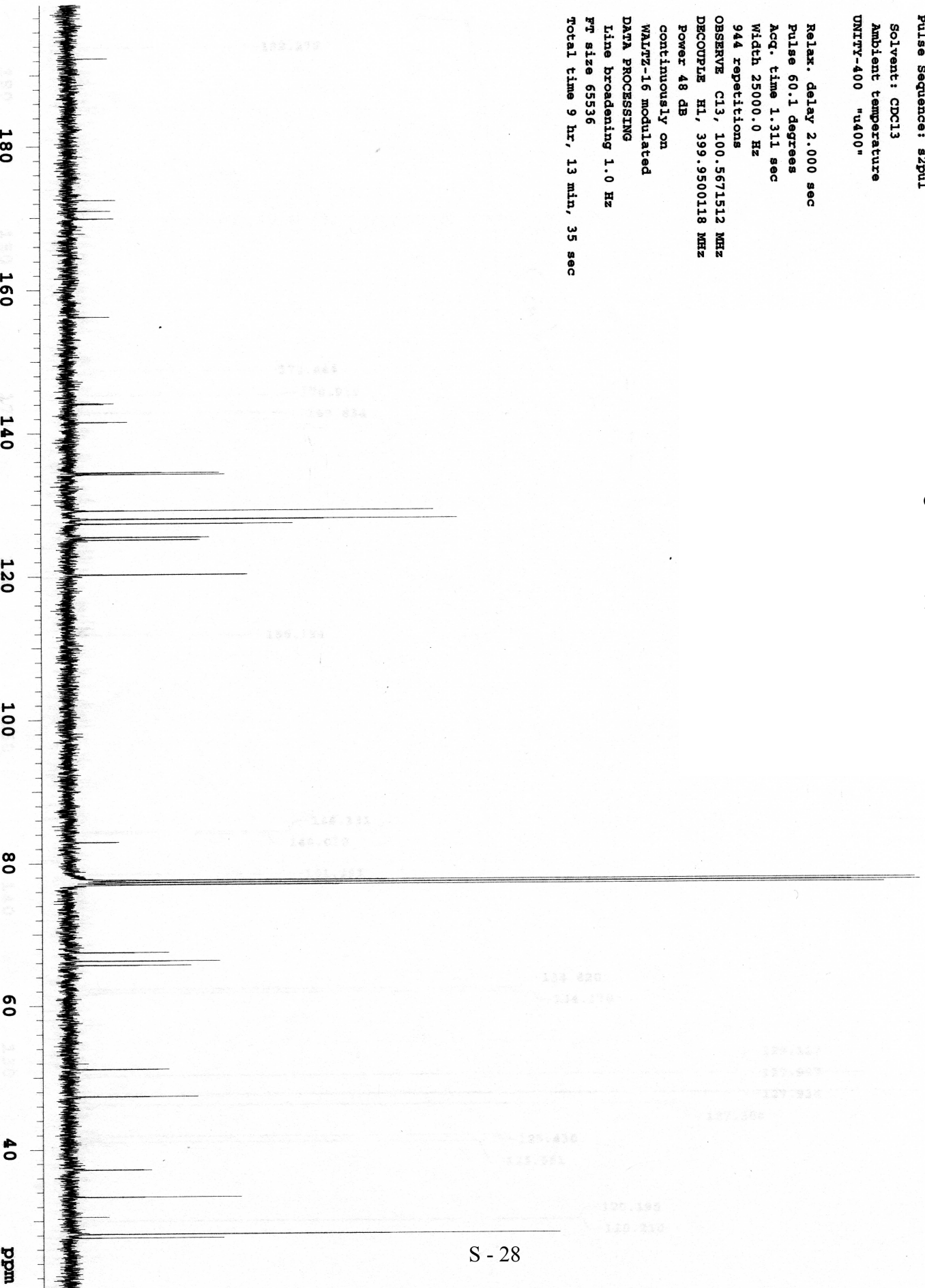
WALTZ-16 modulated

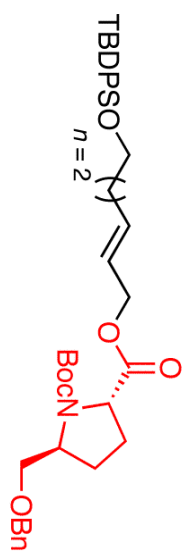
DATA PROCESSING

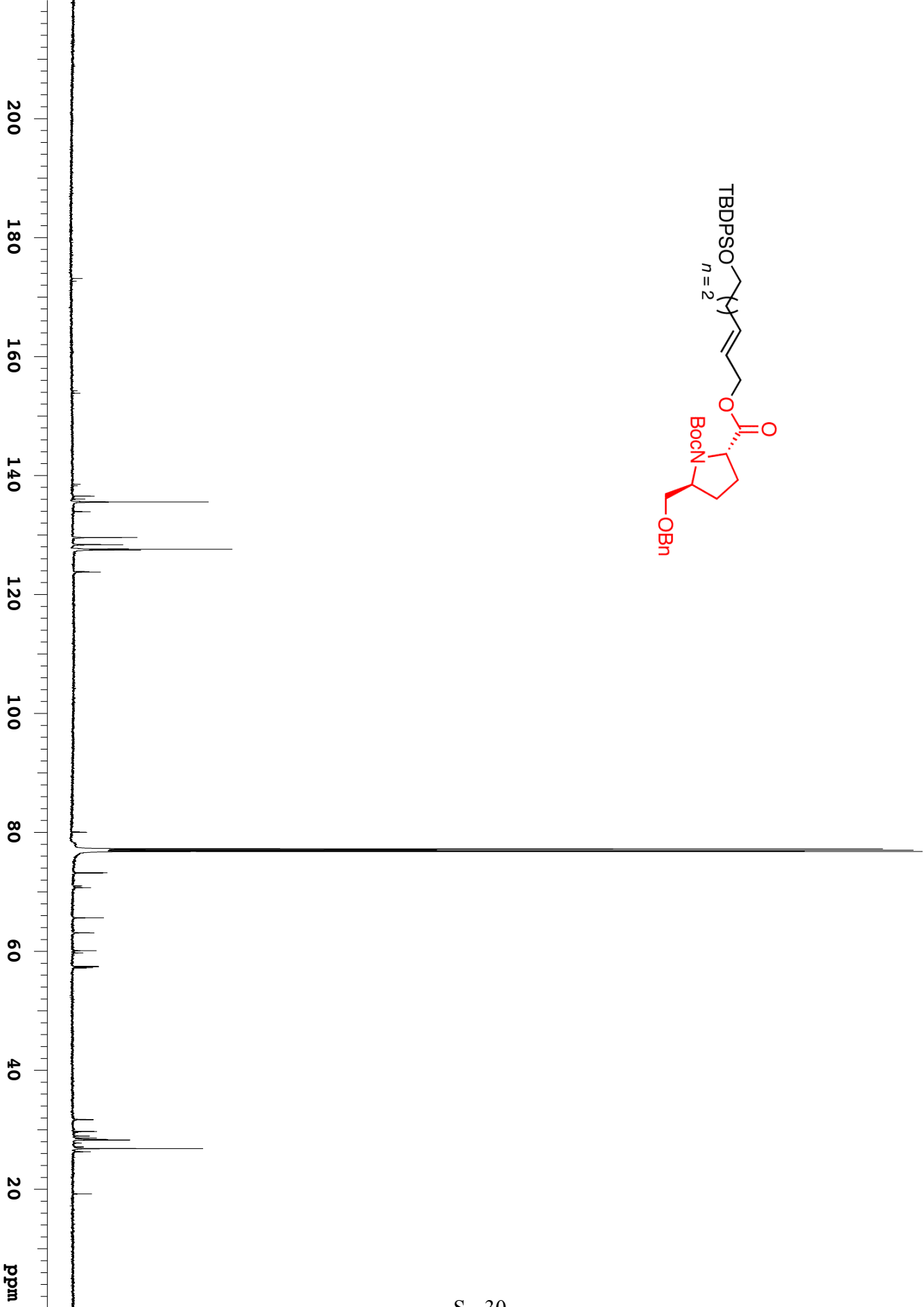
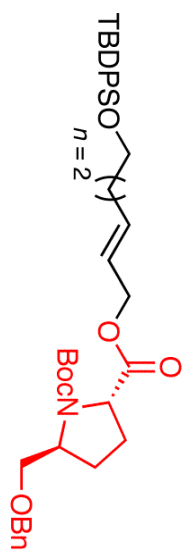
Line broadening 1.0 Hz

FT size 65536

Total time 9 hr, 13 min, 35 sec







Pulse Sequence: s2pul1

Solvent: D2O

Ambient temperature

UNITTY-400 "u400"

Relax. delay 10.000 sec

Pulse 45.0 degrees

Acq. time 4.096 sec

Width 8000.0 Hz

24 repetitions

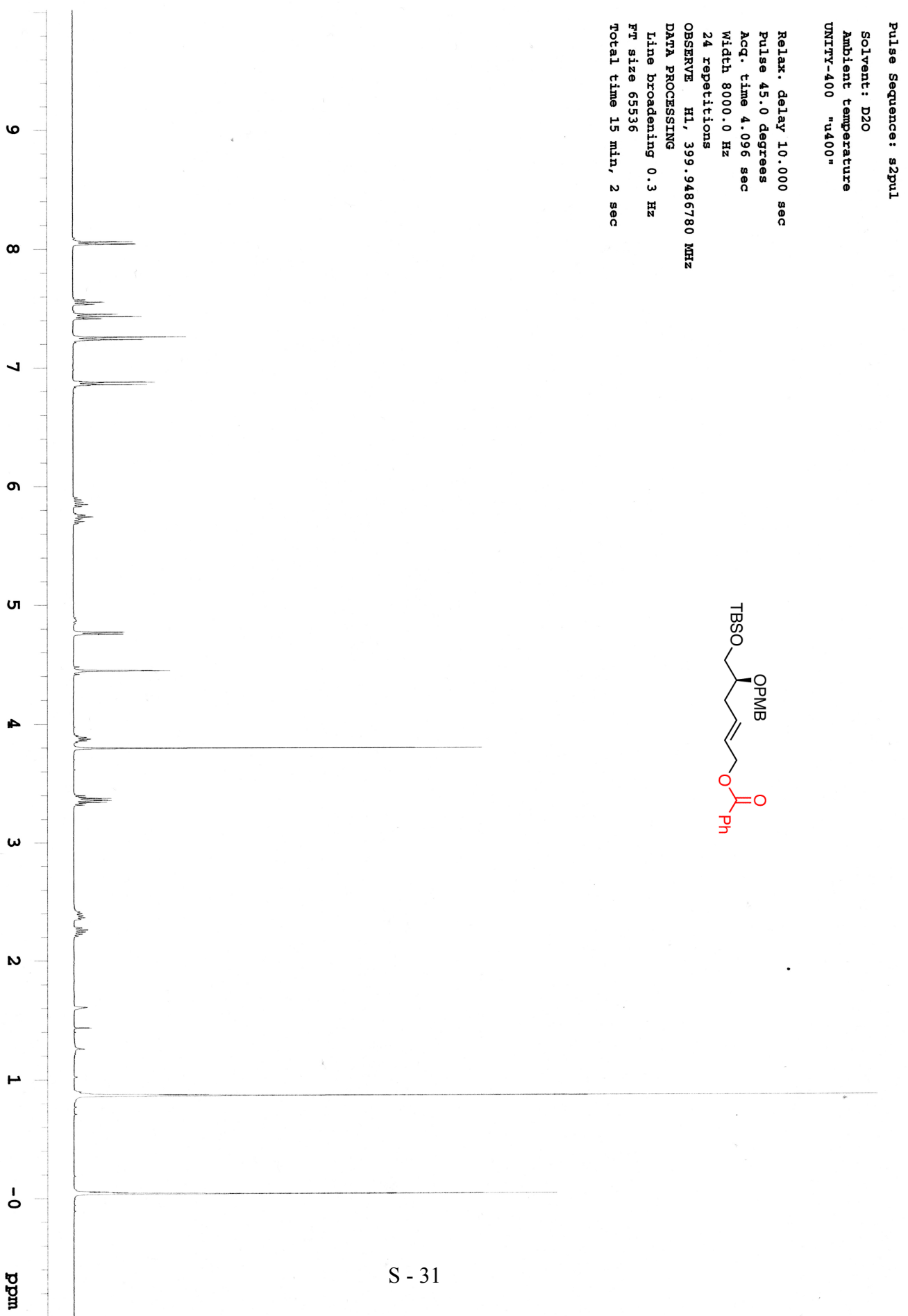
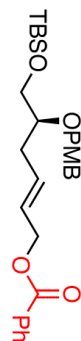
OBSERVE H1, 399.9486780 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FW size 65536

Total time 15 min, 2 sec



Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
UNITRY-400 "u400"

Relax. delay 3.000 sec

Pulse 45.0 degrees

Acq. time 1.311 sec

Width 25000.0 Hz

192 repetitions

OBSERVE C13, 100.5671512 MHz

DECOUPLE H1, 399.9500118 MHz

Power 48 dB

continuously on

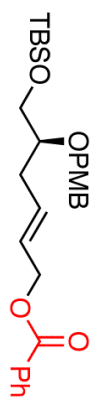
WALTZ-16 modulated

DATA PROCESSING

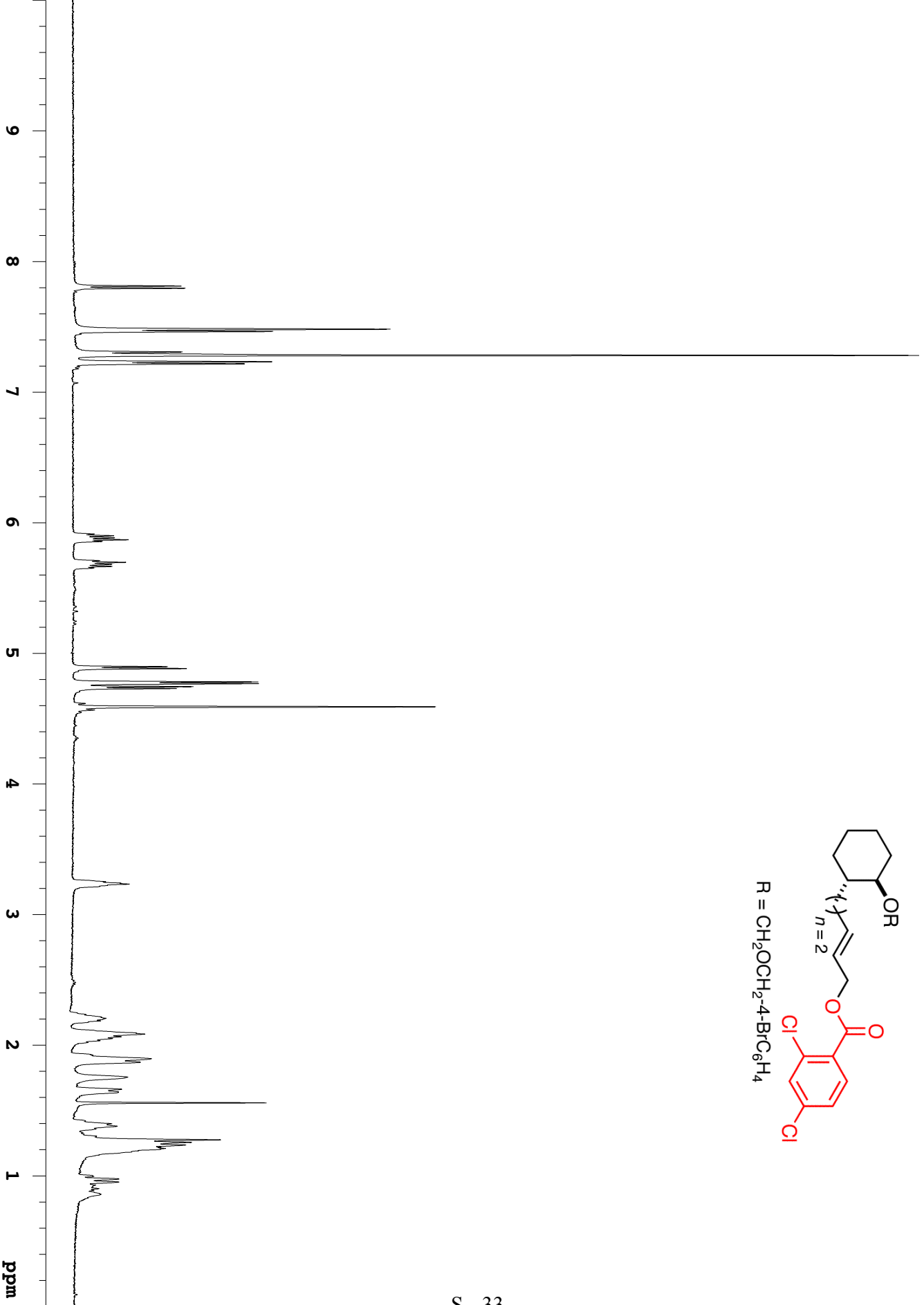
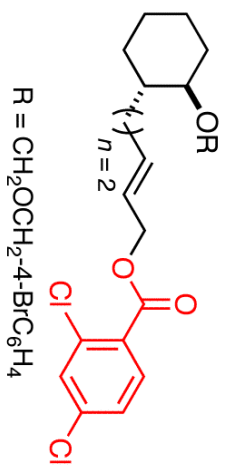
Line broadening 1.0 Hz

FT size 65536

Total time 1 hr, 13 min, 45 sec



220 200 180 160 140 120 100 80 60 40 20 ppm



NVT1140. carbon. sub. 2

Pulse Sequence: s2pu1

Solvent: CDCl3

Ambient temperature

UNITY-400 "n400"

Relax. delay 2.000 sec

Pulse 60.0 degrees

Acq. time 1.311 sec

Width 25000.0 Hz

7552 repetitions

OBSERVE C13, 100.5671512 MHz

DECOUPLE H1, 399.9500118 MHz

Power 48 dB

continuously on

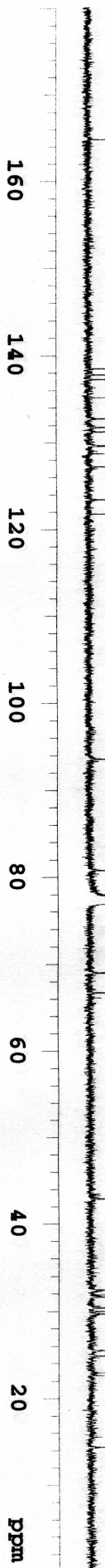
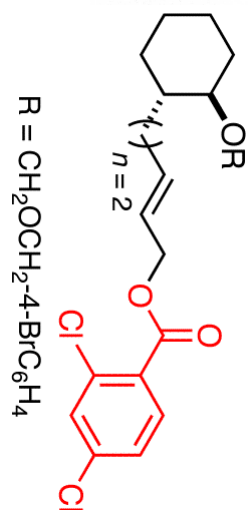
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 9 hr, 13 min, 35 sec



date Mar 21 2008 dfrq 500.070
 solvent CDC13 dn HI
 file /export/home/~ dpwr 18
 data/ui500nb/White~ dof 0
 /vermeulen/NVVI.m~ dm nmn
 acrocycle.proton.f~ dmm c
 id dmf 200

ACQUISITION

sfreq 500.070 dres 1.0
 tn HI homo n
 at 4.096 DEQ2
 np 65536 dfrq2 0
 sw 8000.0 dn2
 fb 4000 dpwr2 1
 bs 1 dof2 0
 tpwr 55 dm2 n
 pw 7.7 dmm2 c
 dl 10.000 dmf2 200

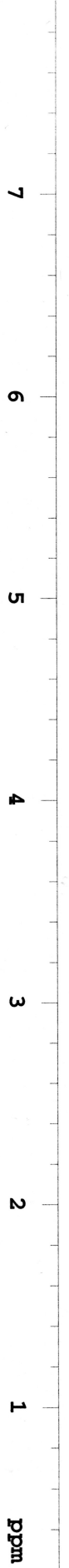
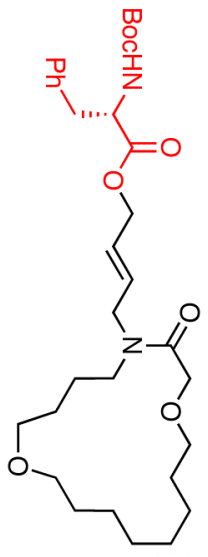
tof 0 dseq2
 nt 16 dres2 1.0
 ct 6 homo2 n
 alock n PROCESSING
 gain not used lb 0.30

FLAGS

il n proc wfile ft
 in n fn not used
 dp y math f
 hs nm

DISPLAY

sp 91.3 wepr
 wp 3889.2 wbs
 vs 131 wnt
 sc 0
 wc 250
 hzmm 15.56
 is 33.57
 rfl 1499.6
 rfp 0
 th 7
 ins 100.000
 ai ph



SAMPLE

DEC. & VT

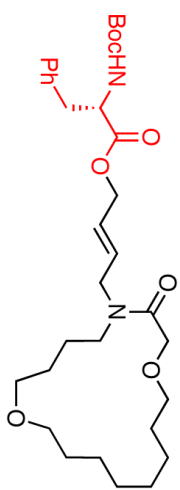
H1

date Mar 21 2008 dn
 solvent CDC13 dof -1092.3
 file /export/home/~ dm
 data/u400/white/ve- dmm YYY
 rmeulen/NVVirimacro- dmf W
 13889
 cycle.carbon.fid dpwr 48

ACQUISITION

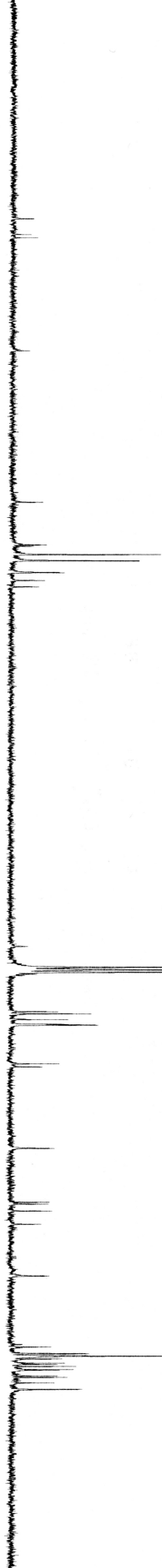
PROCESSING

sfrq 100.579 lb 1.00
 tn C13 wtfile
 at 1.311 proc ft
 np 65536 fn not used
 sw 25000.0 math F
 fb 13800
 bs 16 weirr
 tpwr 54 wexp
 pw 8.7 wbs
 dl 1.000 wnt
 tof 1966.4 DISPLAY 142.7
 nt 100000 sp 19918.8
 ct 14960 wp 162
 alock n vs
 gain not used sc 0
 FLAGS wc 250
 il n Inzmm 79.68
 in n is 500.00
 dp y rfl 910.1
 hs nm rfp 0
 th 20
 lns 100.000
 mm ph



S-36

180 160 140 120 100 80 60 40 20 ppm



Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

INOVA-500 "v1500nb"

Relax. delay 10.000 sec

Pulse 90.0 degrees

Acq. time 4.096 sec

Width 8000.0 Hz

4 repetitions

OBSERVE H1, 500.0677750 MHz

DATA PROCESSING

Line broadening 0.3 Hz

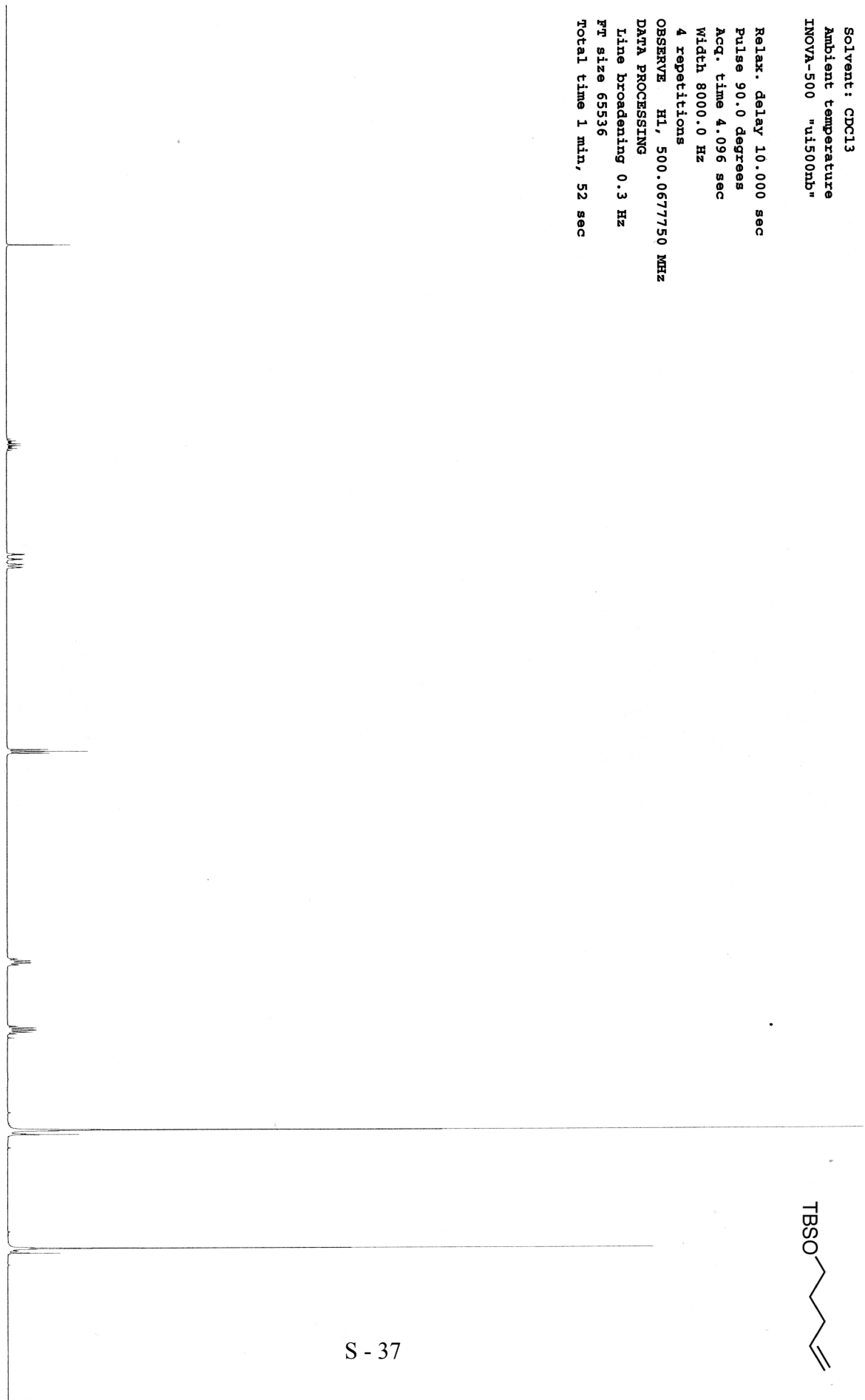
FT size 65536

Total time 1 min, 52 sec



S - 37

8 7 6 5 4 3 2 1 -0 ppm



Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

UNITY-400 "v4.00"

Relax. delay 5.000 sec

Pulse 45.0 degrees

Acq. time 4.096 sec

Width 8000.0 Hz

15 repetitions

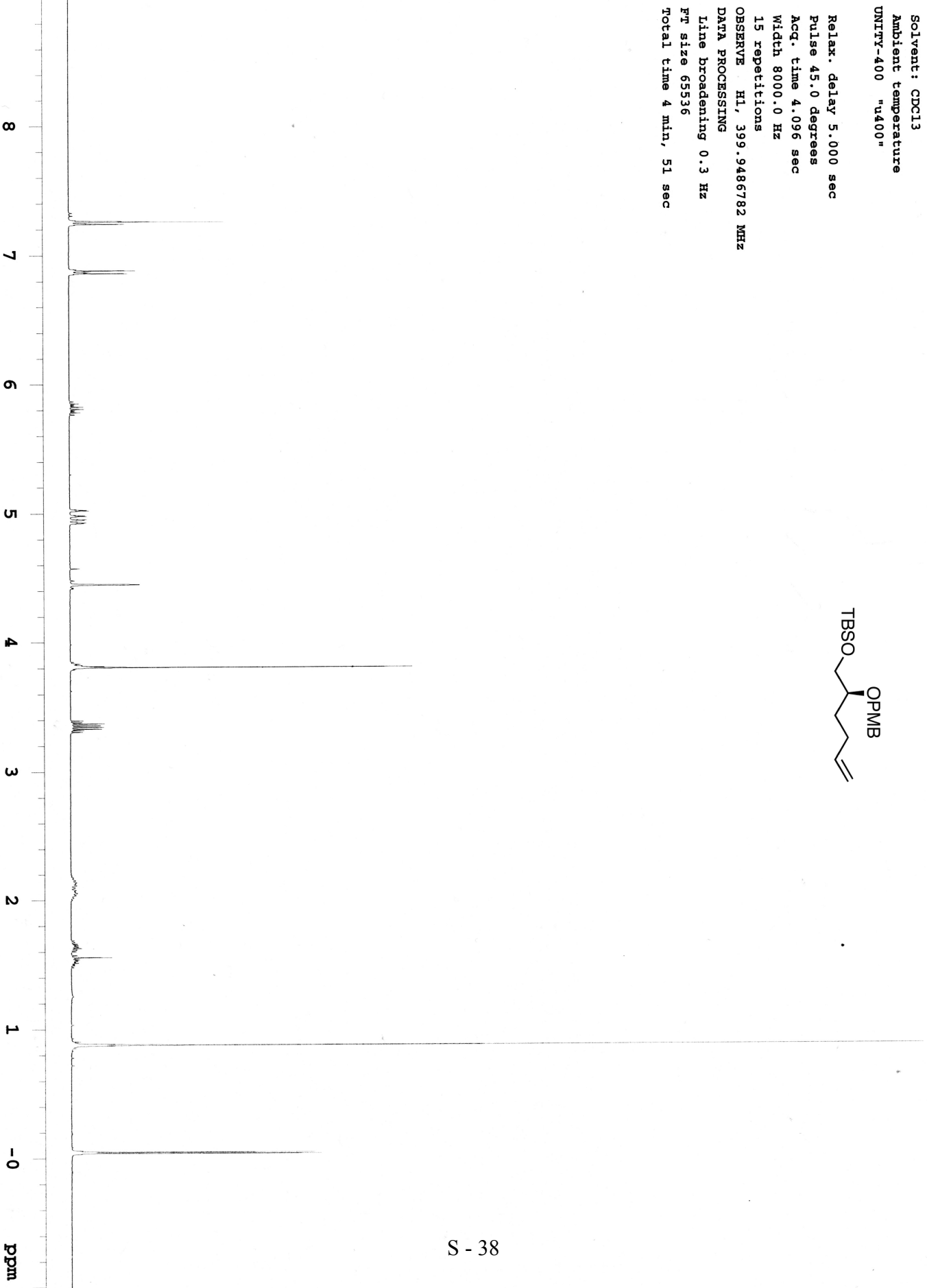
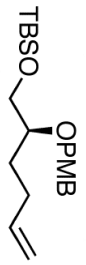
OBSERVE H1, 399.9486782 MHz

DATA PROCESSING

Line broadening 0.3 Hz

Ft size 65536

Total time 4 min, 51 sec



Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

UNITY-400 "u400"

Relax. delay 10.000 sec

Pulse 45.0 degrees

Acq. time 4.096 sec

Width 8000.0 Hz

7 repetitions

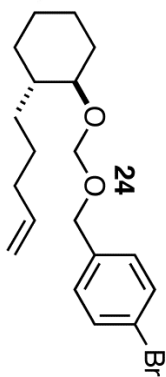
OBSERVE H1, 399.9486780 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 65536

Total time 7 min, 31 sec



Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

UNITY-400 "u400"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.311 sec

Width 25000.0 Hz

550 repetitions

OBSERVE C13, 100.5671512 MHz

DECOUPLE H1, 399.9500118 MHz

Power 48 dB

continuously on

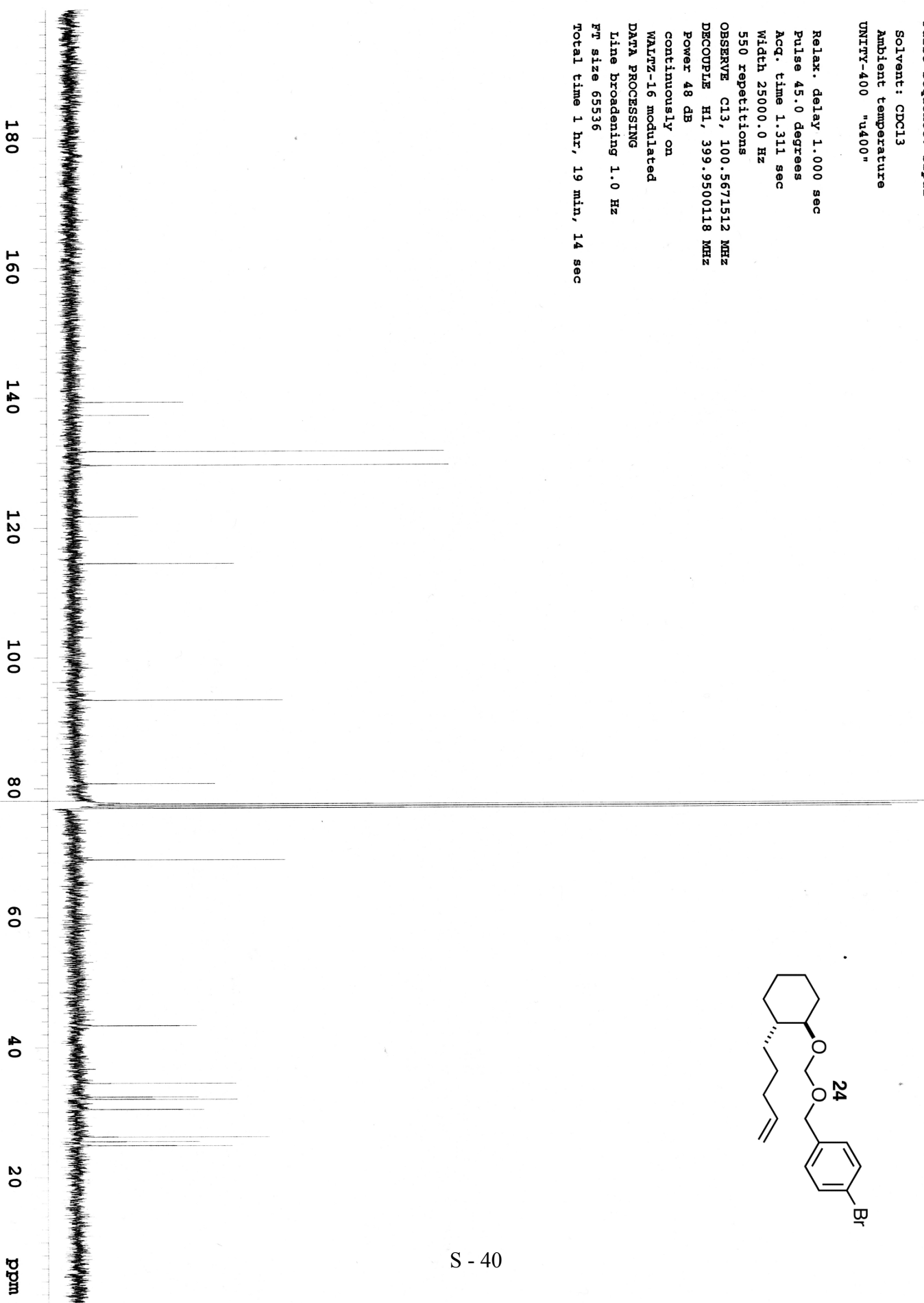
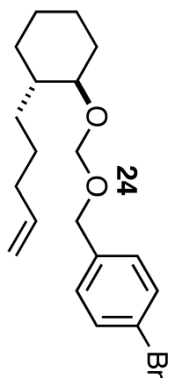
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FW size 65536

Total time 1 hr, 19 min, 14 sec

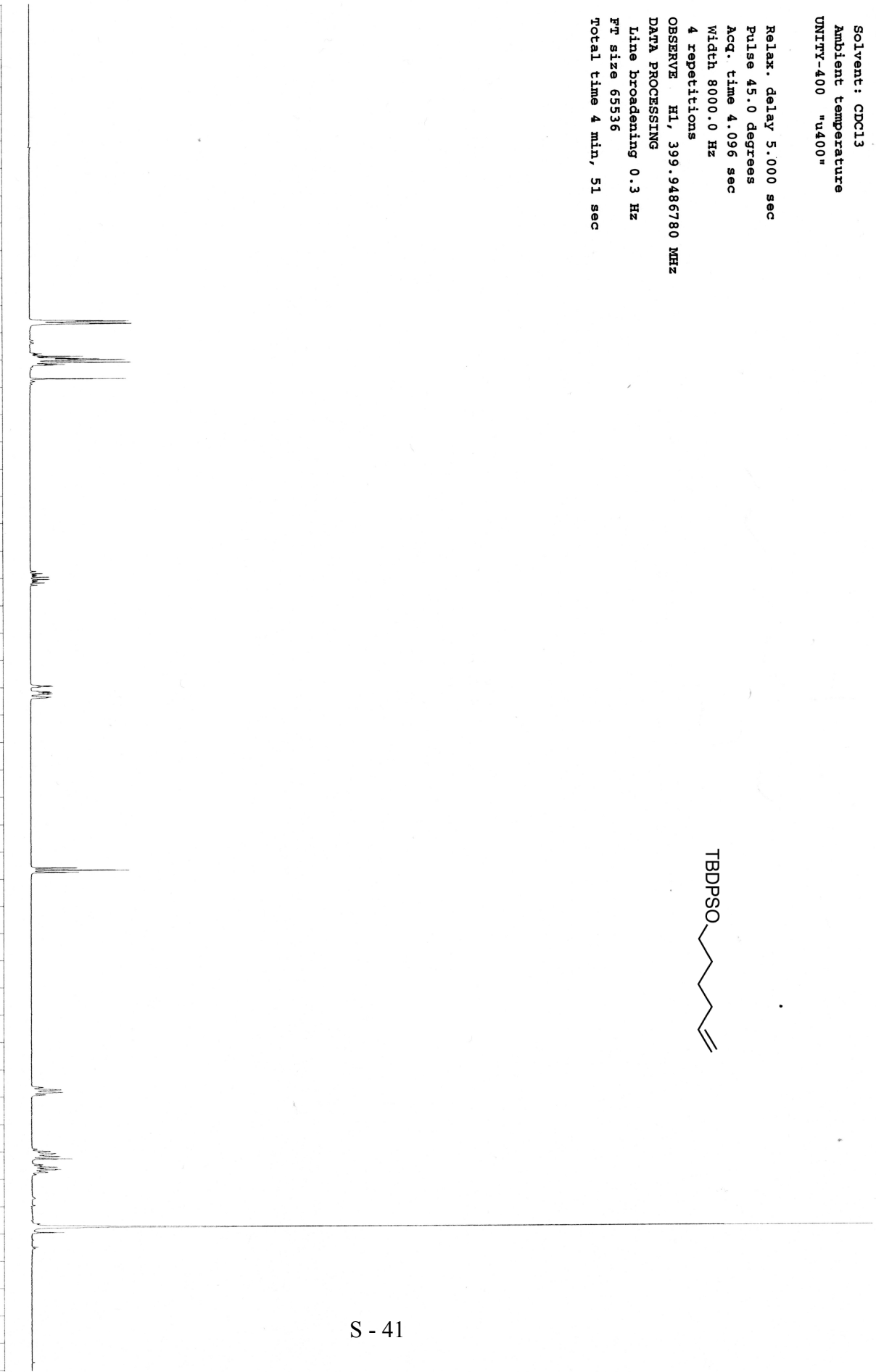


Solvent: CDCl3
Ambient temperature
UNITX-400 "u400"

Relax. delay 5.000 sec
Pulse 45.0 degrees
Acq. time 4.096 sec
Width 8000.0 Hz
4 repetitions
OBSERVE H1, 399.9486780 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 4 min, 51 sec



9
8
7
6
5
4
3
2
1
ppm



Pulse Sequence: s2pul
Solvent: CDC13
Ambient temperature
UNITRY-400 "v400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.311 sec
Width 25000.0 Hz

32 repetitions

OBSERVE C13, 100.5671512 MHz
DECOUPLE H1, 399.9500118 MHz

Power 48 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

Ft size 65536

Total time 19 min, 48 sec



Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

INOVA-500 "vkr500"

Relax. delay 5.000 sec

Pulse 60.0 degrees

Acq. time 4.096 sec

Width 8000.0 Hz

16 repetitions

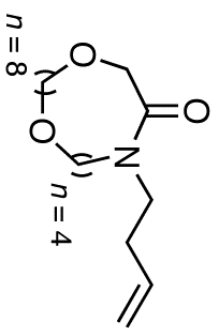
OBSERVE H1, 499.4298995 MHz

DATA PROCESSING

Line broadening 0.3 Hz

FT size 65536

Total time 4 min, 51 sec



9
8
7
6
5
4
3
2
1
ppm

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

User: 1-14-87

INOVA-500 "vxr500"

Relax. delay 1.000 sec

Pulse 60.0 degrees

Acq. time 1.024 sec

Width 32000.0 Hz

1232 repetitions

OBSERVE C13, 125.5817166 MHz

DECOUPLE H1, 499.4315638 MHz

Power 46 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 5 hr, 38 min, 49 sec

