

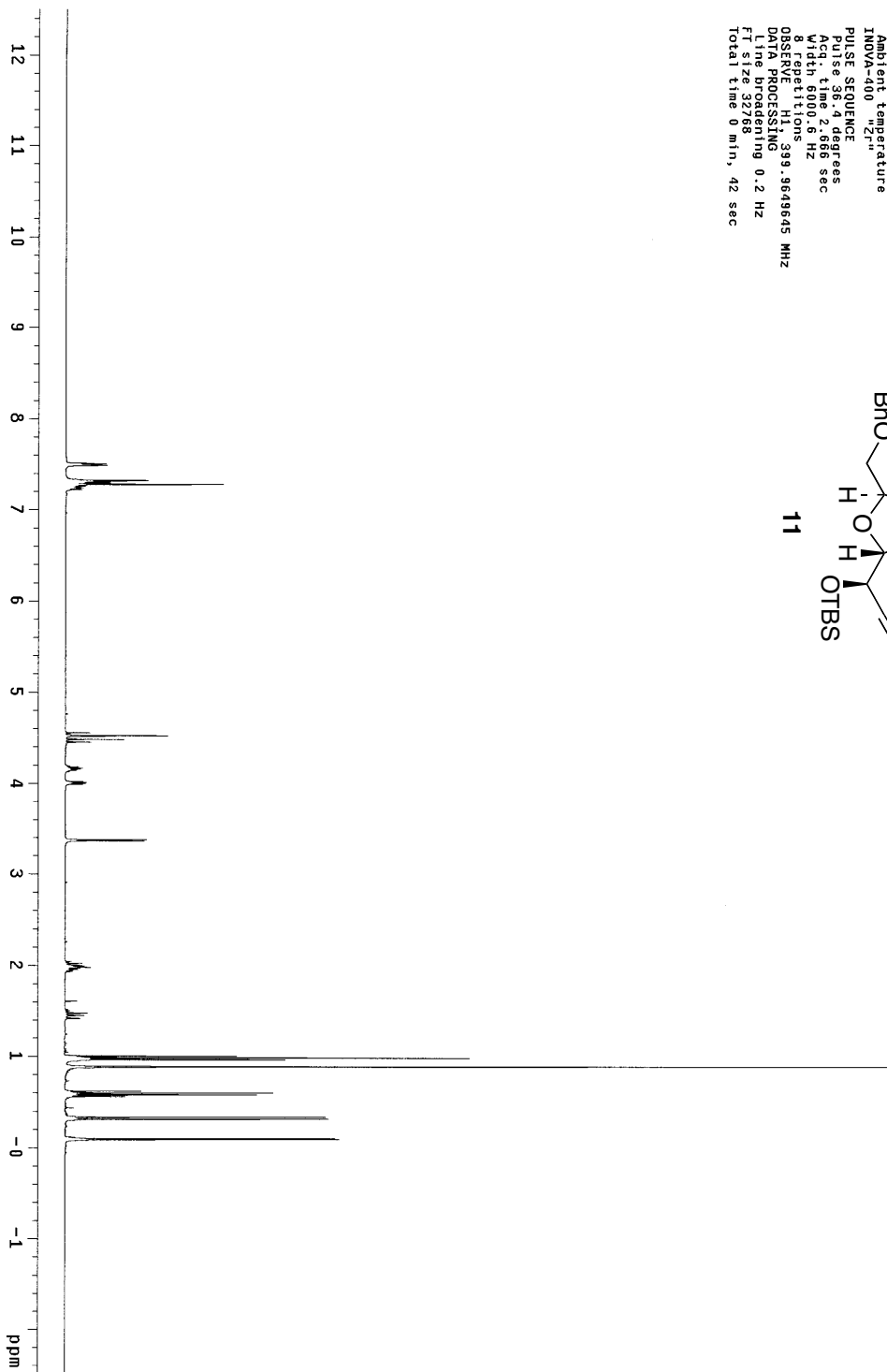
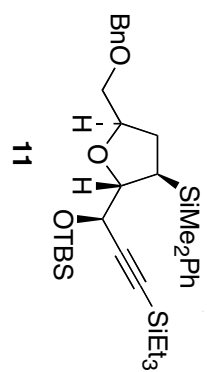
**Efficient Protodesilylation of Unactivated C(sp³)-SiMe₂Ph Bonds using
Tetrabutylammonium Fluoride**

Cheryl L. Heitzman, William T. Lambert, Eric Mertz, J. Brad Shotwell, Jennifer M. Tinsley,
Porino Va and William R. Roush*

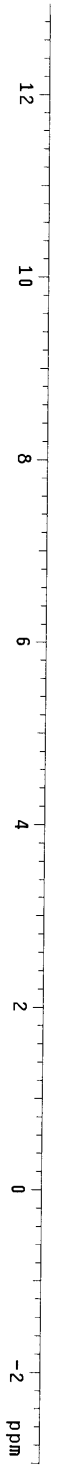
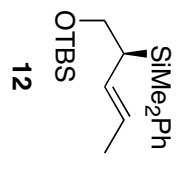
Department of Chemistry, University of Michigan, Ann Arbor, MI, 48109-1055
Email: roush@umich.edu

Scanned Spectra to Accompany Supporting Information

STANDARD 1H OBSERVE
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
INOVA-400 "Zr"
PULSE SEQUENCE
Pulse: 36.4 degrees
Acq. time: 2.566 sec
Width: 6000.6 Hz
OBSERVED FID: 399.9649645 MHz
DATA PROCESSING: 0.2 Hz
Line broadening: 0.2 Hz
FT size: 32768
Total time: 0 min, 42 sec



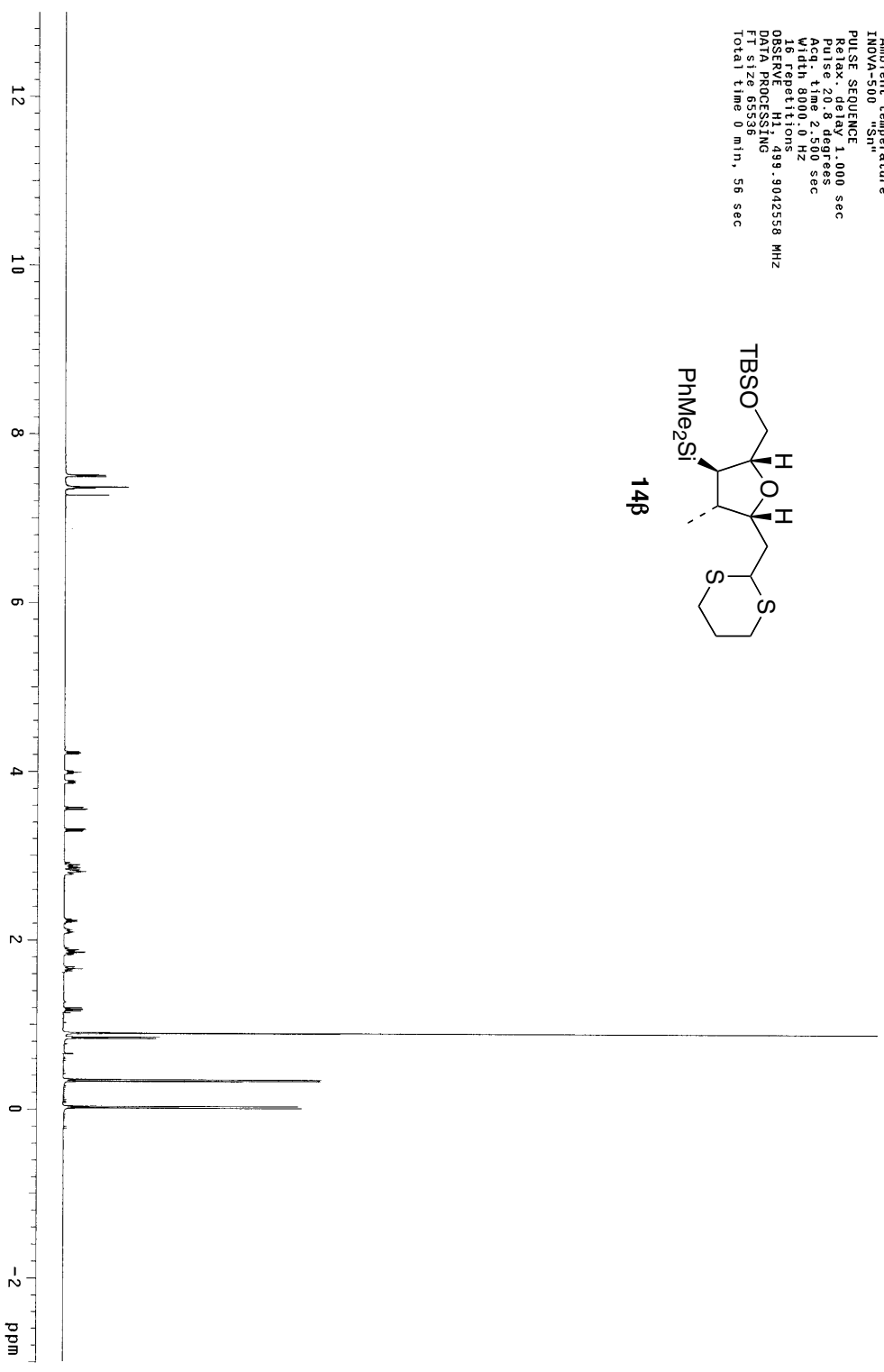
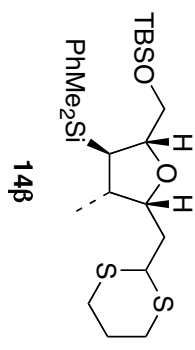
STANDARD PROTON PARAMETERS
Pulse Sequence: szpu1
Solvent: CDCl3
Ambient temperature
INDYA-500 "Sn"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
8 Repetitions 499.9042558 MHz
OBSERVED F1 F2
FT size 65536
Total time 0 min, 56 sec



II-163

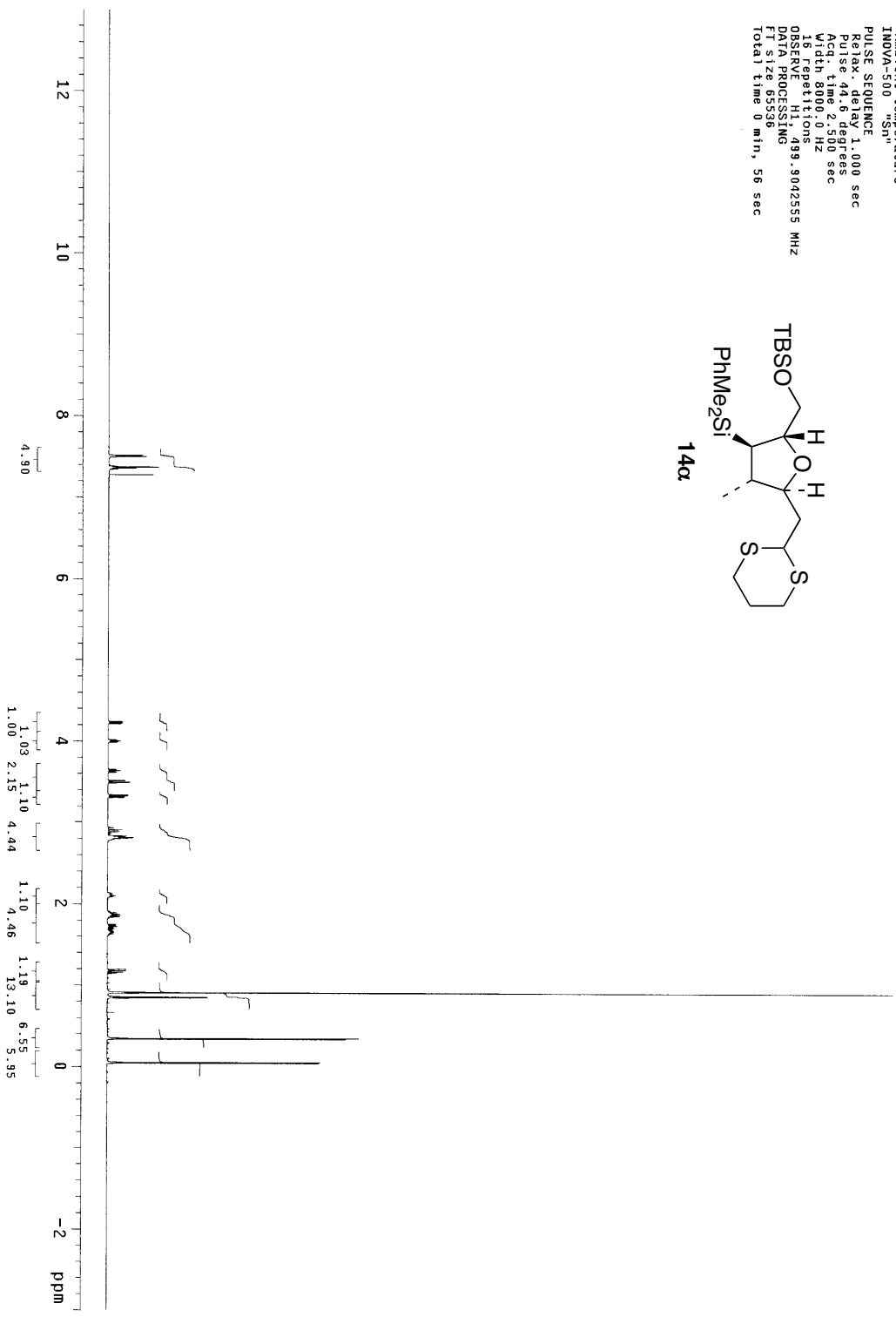
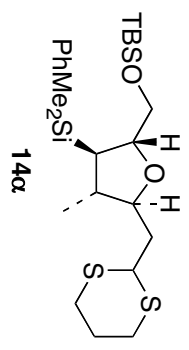
STANDARD PROTON PARAMETERS

Pulse Sequence: szpu1
Solvent: CDCl3
Ambient temperature
INOVA-500 "Si"
PULSE SEQUENCE
R1: 1.000 sec
Pulse: 20.8 degrees
Acq. time: 2.500 sec
Width: 3000.0 Hz
16 repetitions
OBSERVE H1: 499.3042558 MHz
DATA PROCESSING
F1 size: 65536
Total time: 0 min, 56 sec



STANDARD PROTON PARAMETERS

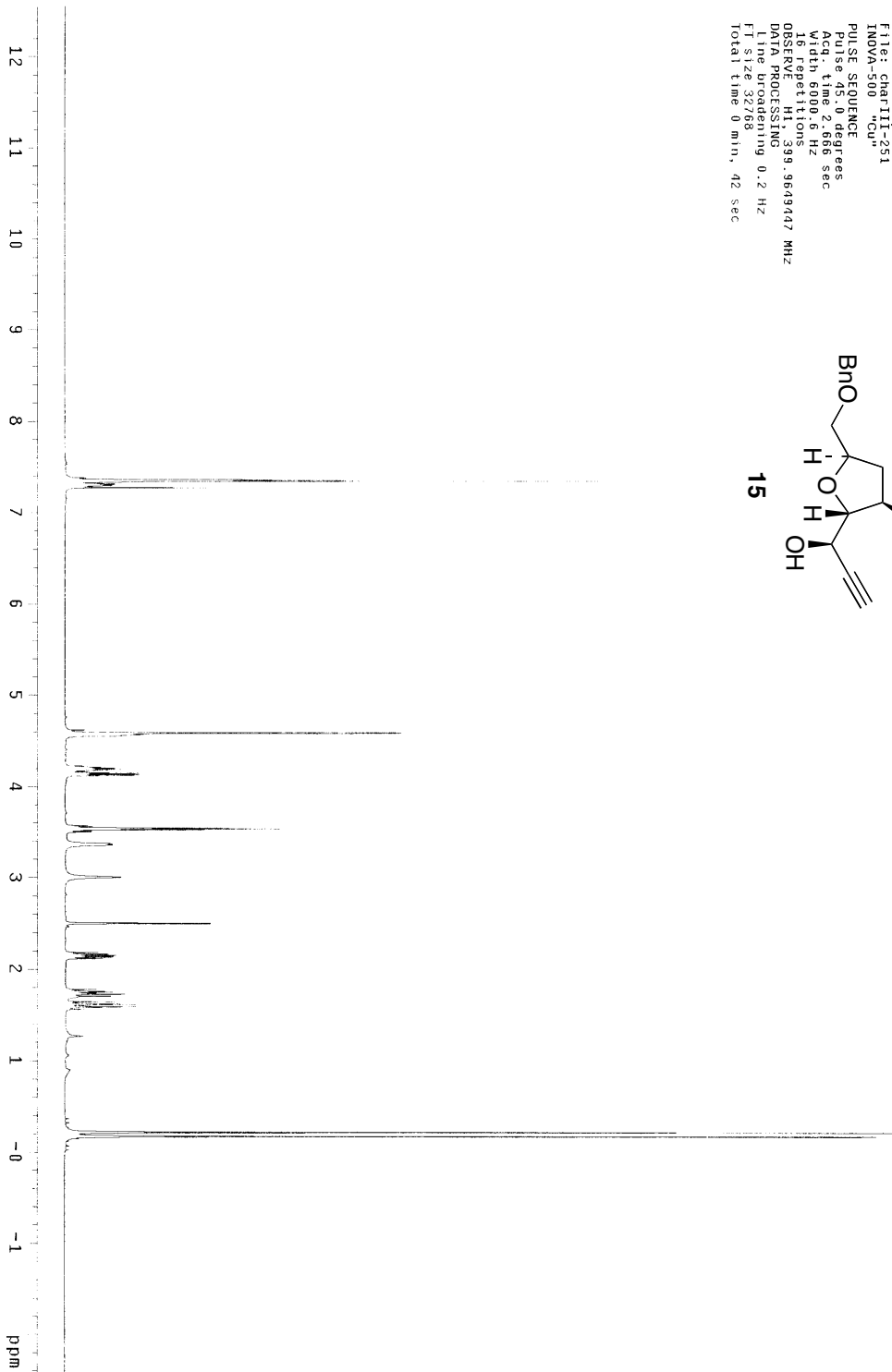
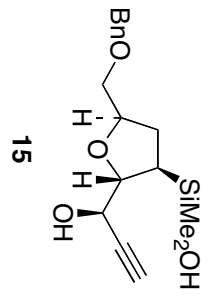
Pulse Sequence: szpul
 Solvent: CDCl3
 Ambient temperature
 INOVA-500 5H
 PULSE SEQUENCE
 Relax. delay 1.000 sec
 Pulse 41.6 degrees
 Width 800 Hz
 16 repetitions
 OBSERVE H1 499.9042555 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 56 sec



U-144

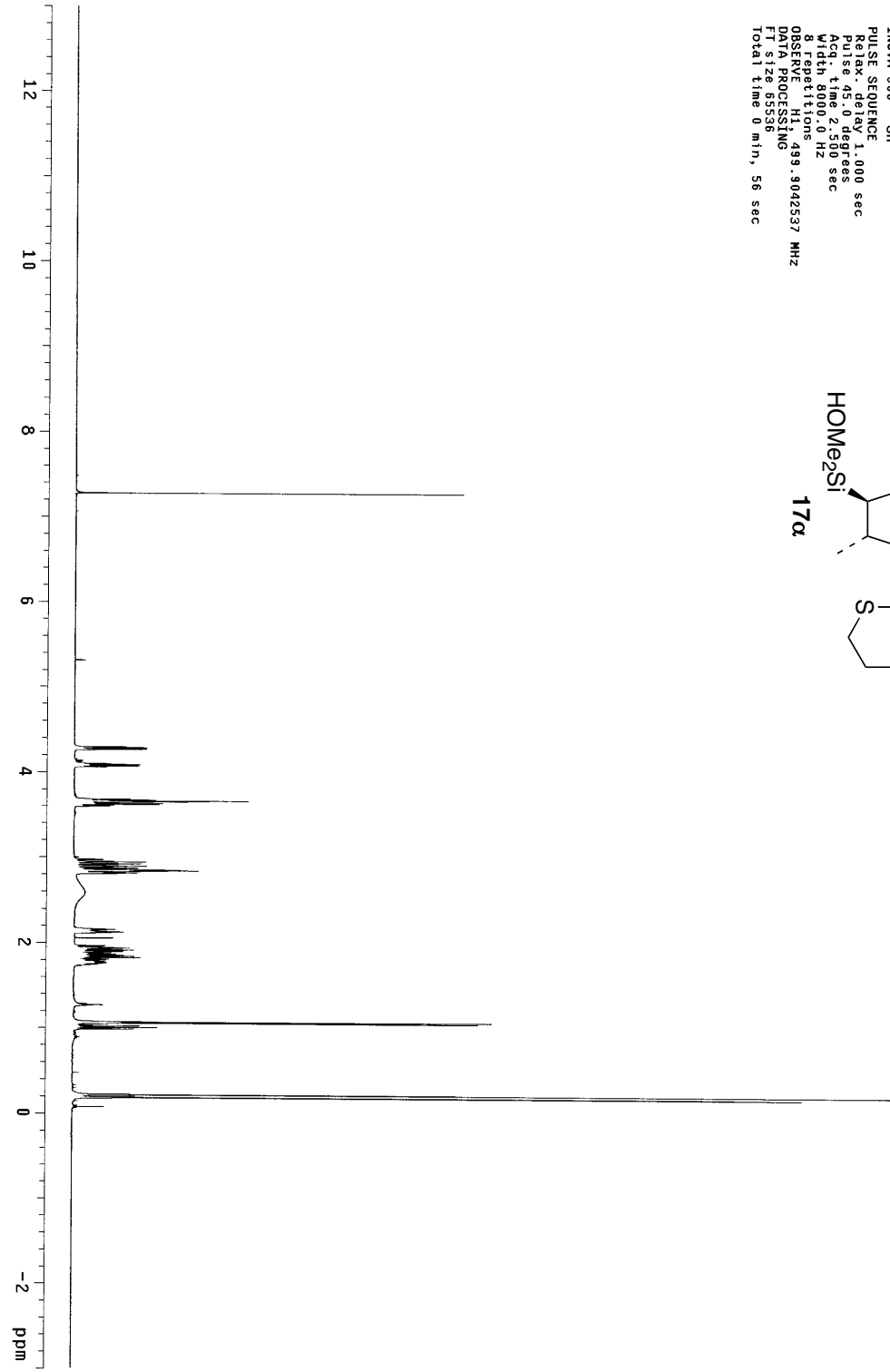
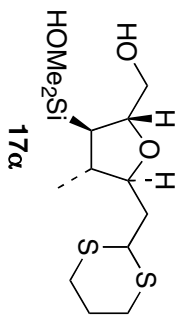
III-251

Jbs111-251on400MHz
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
File: Char111-251
INOVA-500 ¹³Cu
PULSE SEQUENCE
Pulse 45.0 degrees
Acq. time 2.656 sec
Width 6009.6 Hz
Spectral width 9.9643447 MHz
Observed H ions 43
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 0 min, 42 sec



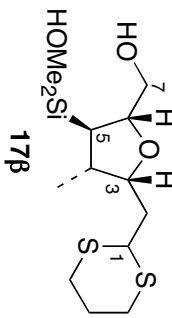
STANDARD PROTON PARAMETERS

Pulse Sequence: szpu1
Solvent: CDCl3
Acquire temperature: INOVA-500
PULSE SEQUENCE: 1, 000 sec
Pulse sequence: 45.00 sec
Acq. time: 2.500 sec
Width: 8000.0 Hz
8 repetitions
OBSERVE: H1, 499.9042537 MHz
DATA PROCESSING
F1 size: 85536
Total time: 0 min, 56 sec



STANDARD PROTON PARAMETERS

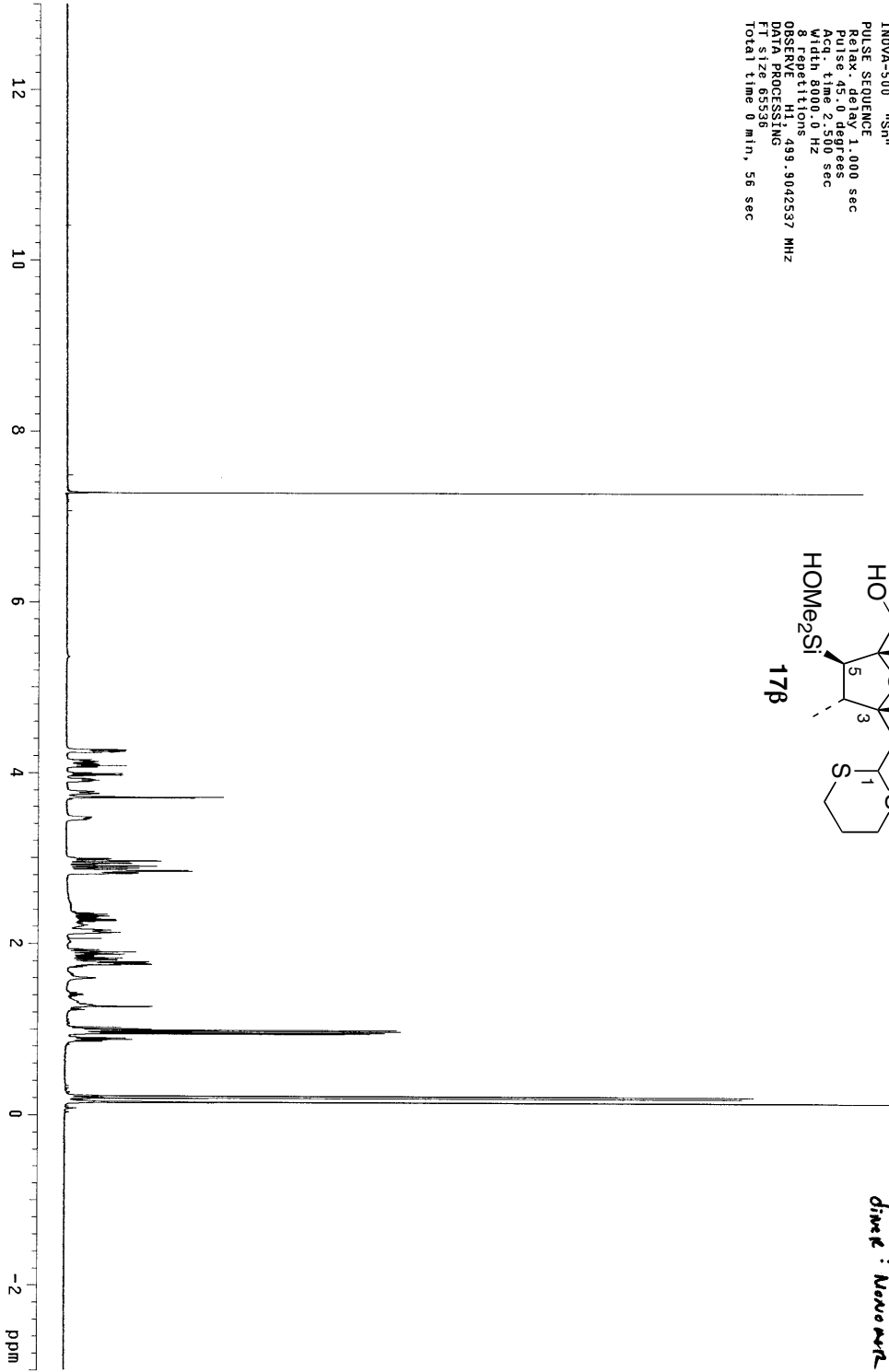
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 MHz
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Width 3000.0 Hz
8 Repetitions
OBSERVE H1, 499.9042537 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 56 sec



II-269

Ca 1:1

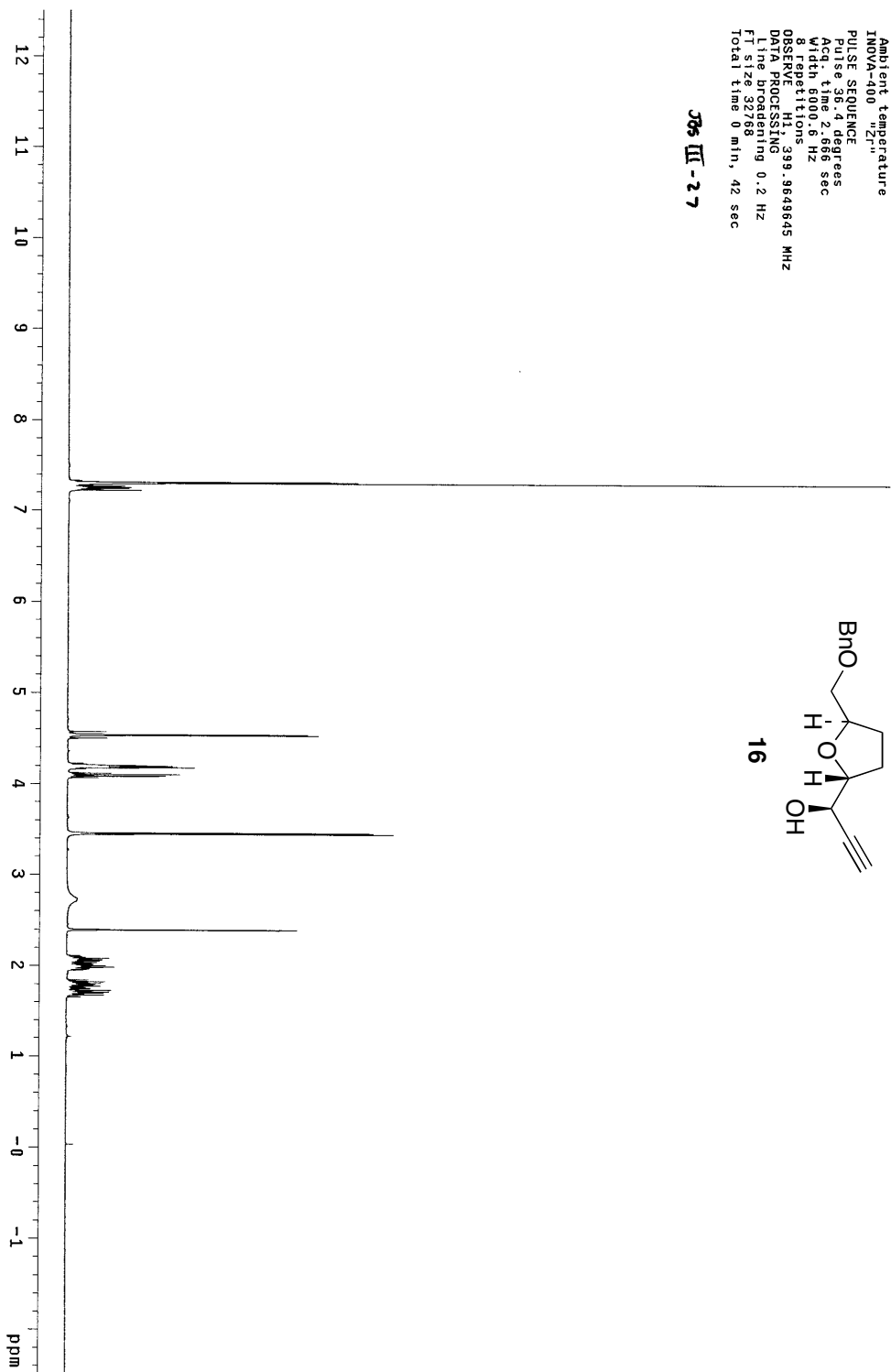
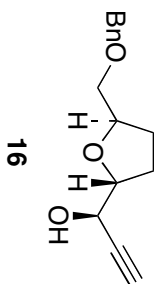
dimethyl Novec



STANDARD 1H OBSERVE

Pulse Sequence: sZpu1
 Solvent: CDCl3
 Ambient Temperature
 INOVA-400 "Zr"
 PULSE SEQUENCE
 Pulse: 38.4 degrees
 Acq. time: 2.666 sec
 Width: 6000.6 Hz
 Offset: 100.625 MHz
 Observed: 99.9649645 MHz
 DATA PROCESSING
 Line broadening: 0.2 Hz
 FI size: 32768
 Total time: 0 min, 42 sec

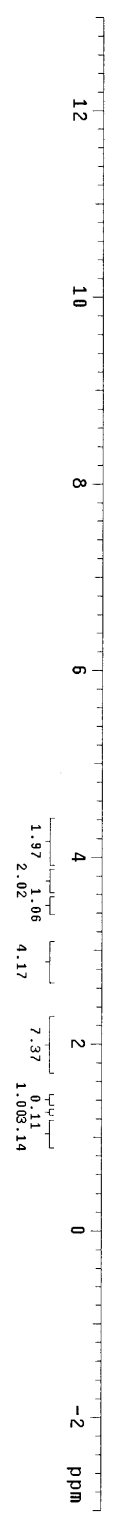
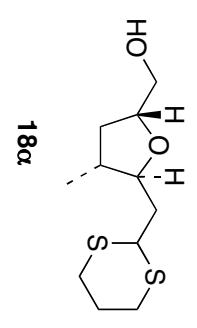
306 III - 2 7



II-130

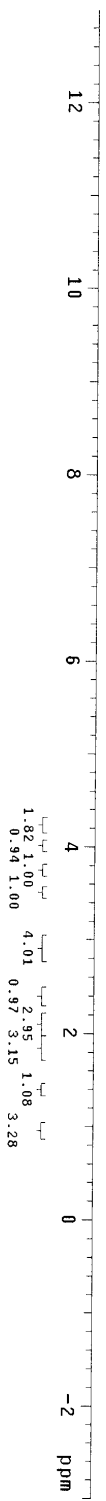
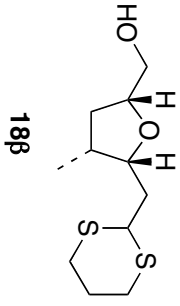
STANDARD PROTON PARAMETERS

Pulse Sequence: szpu1
Solvent: CDCl3
Ambient temperature
INOVA-500 "5H"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 49.6 degrees
Acq. time 2.500 sec
16
15
OBSERVE: H1 on 499.9042537 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 56 sec



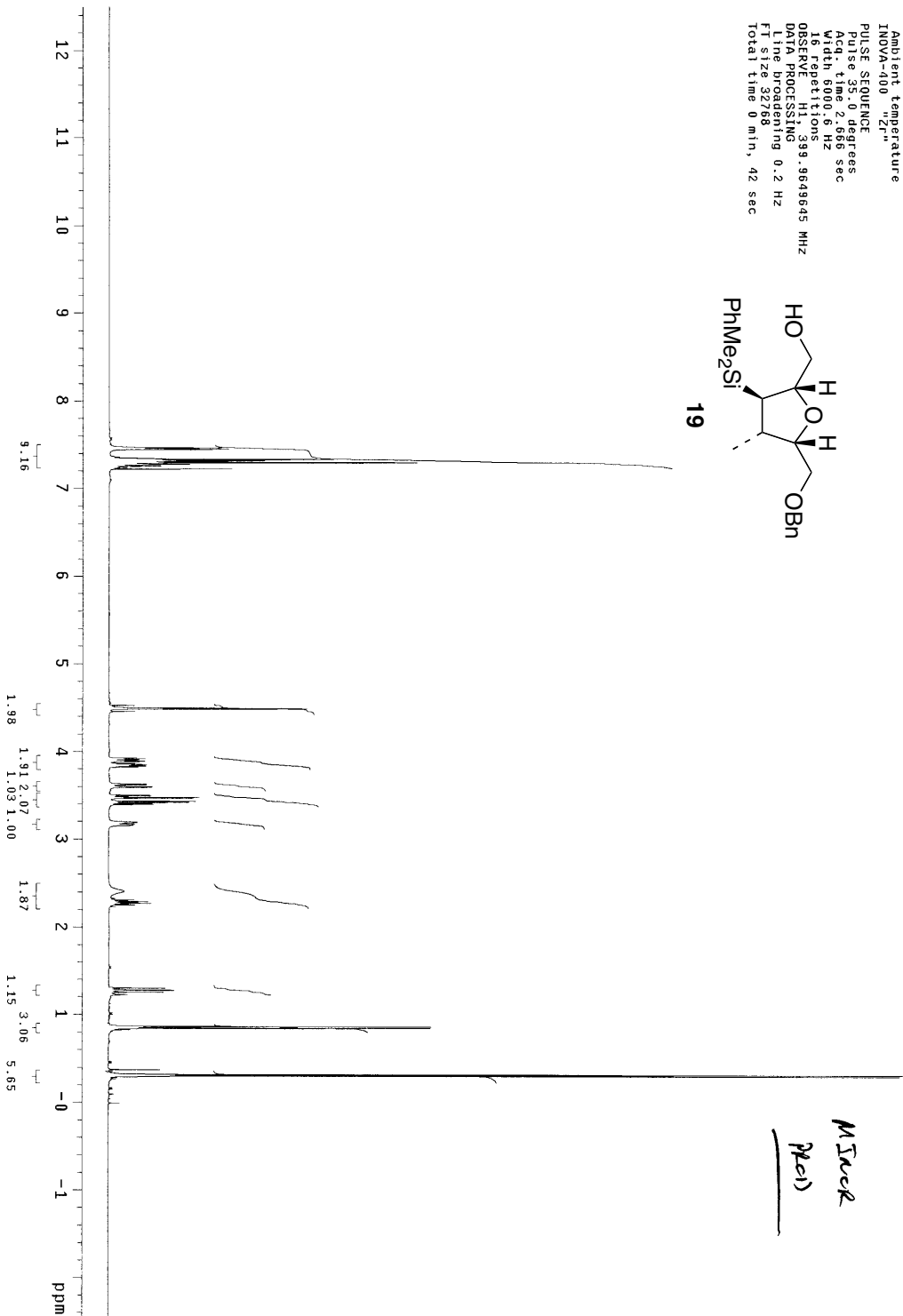
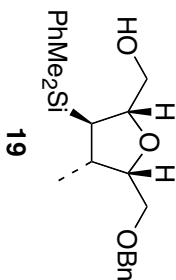
STANDARD PROTON PARAMETERS

Pulse Sequence: szpu1
Solvent: CDCl3
Ambient temperature
INOVA-500 130MHz
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 41.6 degrees
Acq. time 4.500 sec
Width 800 Hz
16 repetitions
OBSERVE H1: 499.9042555 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 56 sec



STANDARD 1H OBSERVE

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
INOVA-400 "Zr"
PULSE SEQUENCE
Pulse 35.0 degrees
Acq. time 2.666 sec
Width 6000.6 Hz
OBSERVE H1 399.9649645 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 0 min, 42 sec



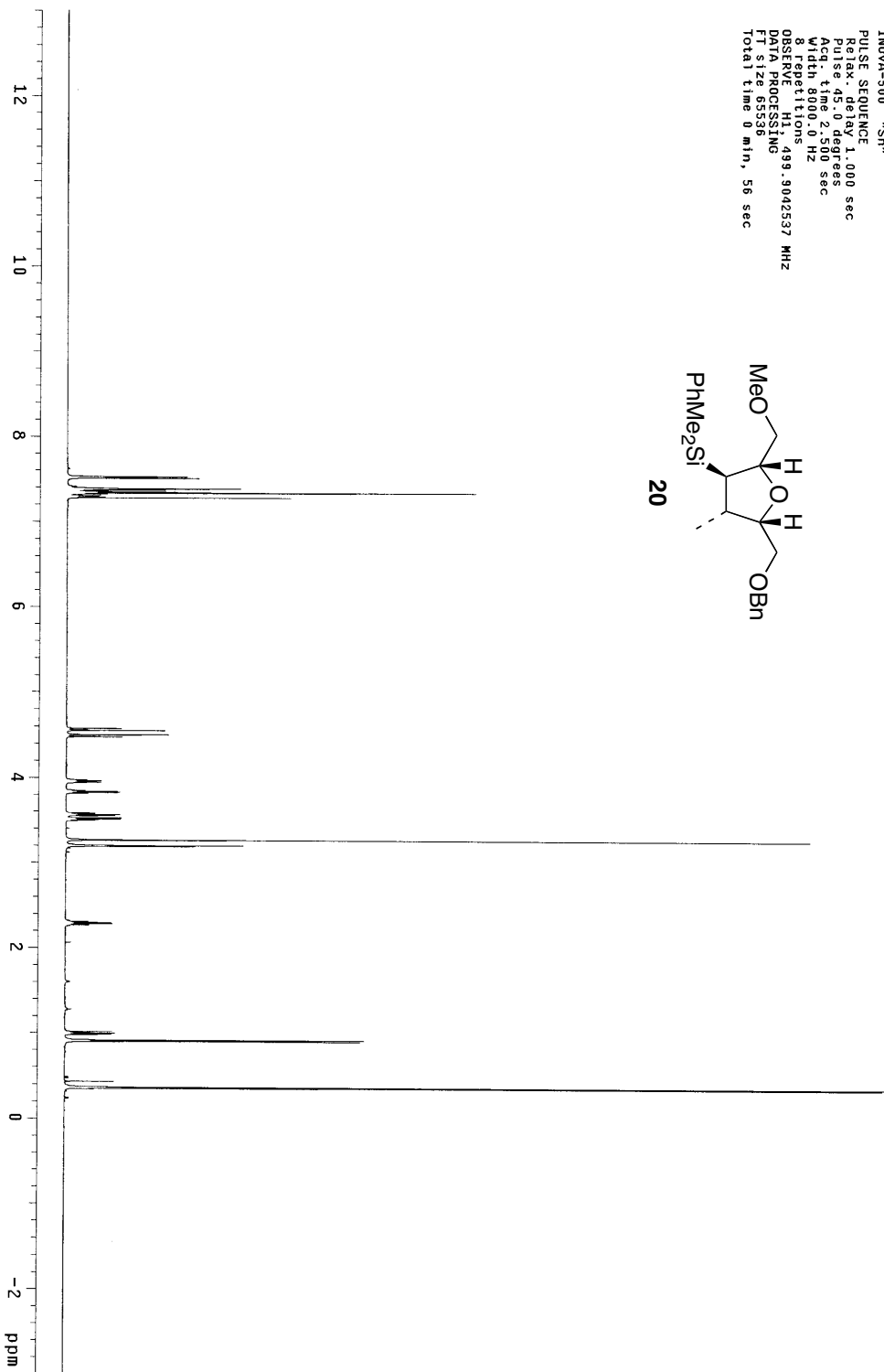
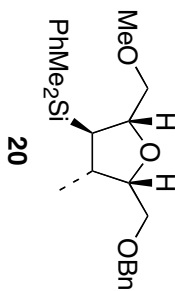
II-55

Mack

Med

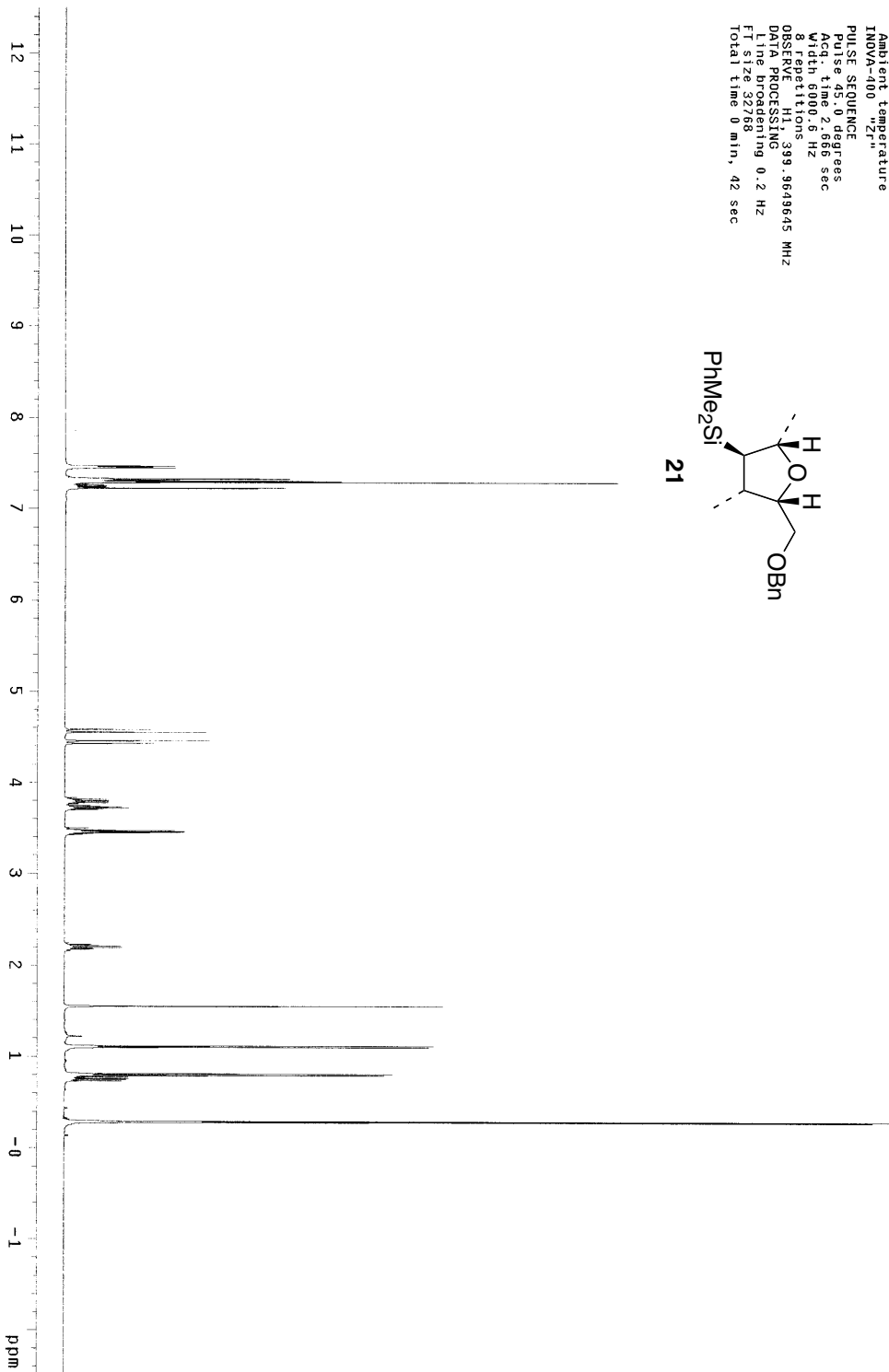
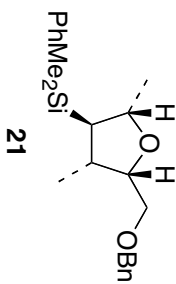
STANDARD PROTON PARAMETERS

Pulse Sequence: s2pu1
Solvent: CDCl3
Temp: 23.0 C / 296.1 K
INOVA-500 "Srf"
PULSE SEQUENCE
Relax. delay: 1.000 sec
Pulse: 45.0 degrees
Acq. time: 2.500 sec
Width: 8000.0 Hz
8 Repetitions: 499.9042537 MHz
OBSERVED F1: 99.9042537 MHz
P1: 12.00000000 sec
P1 size: 65536
Total time: 0 min, 56 sec



STANDARD 1H OBSERVE

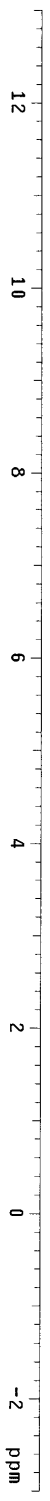
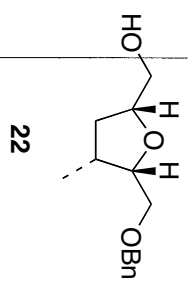
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
INOVA-400 "ZPL"
PULSE SEQUENCE
Pulse 45.0 degrees
Acq. time 2.665 sec
8
P1 len 8900.8 Hz
OBSERVE: H1 399.9649645 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 0 min, 42 sec



HPLC

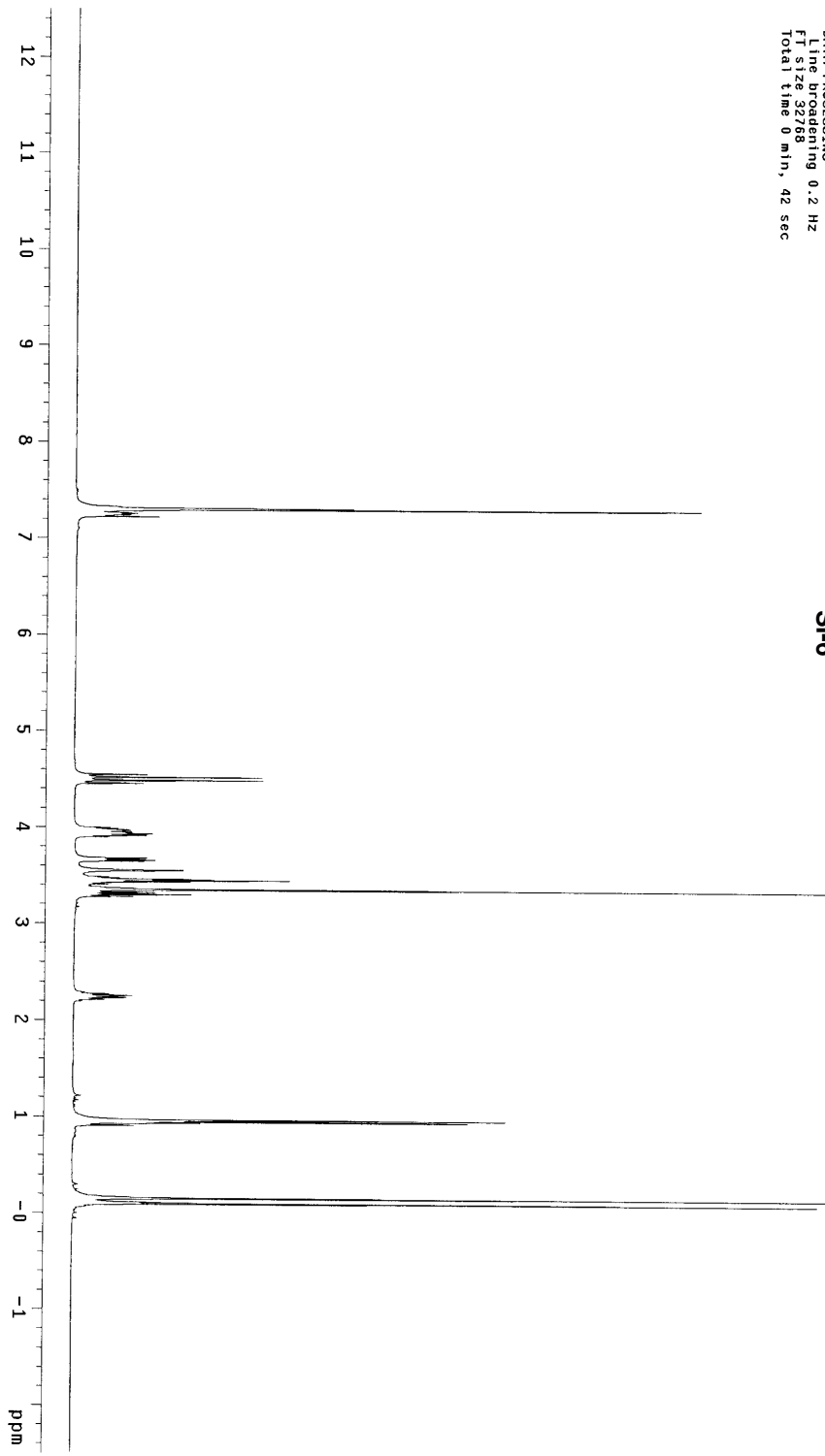
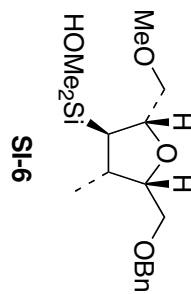
STANDARD PROTON PARAMETERS

Pulse Sequence: s2pu1
Solvent: CDCl3
Temp: 23.0 C / 296.1 K
INOVA-500 "1H"
PULSE SEQUENCE:
Relax: delay 1.000 sec
Pulse: 45.0 degrees
Acq: time 2.500 sec
SFO: 300.0 Hz
NUC1: 1H
OBSERVE: 1H
P1: 12.00
PR: 1.00
DATA PROCESSING
FT size 65536
Total time 0 min, 56 sec



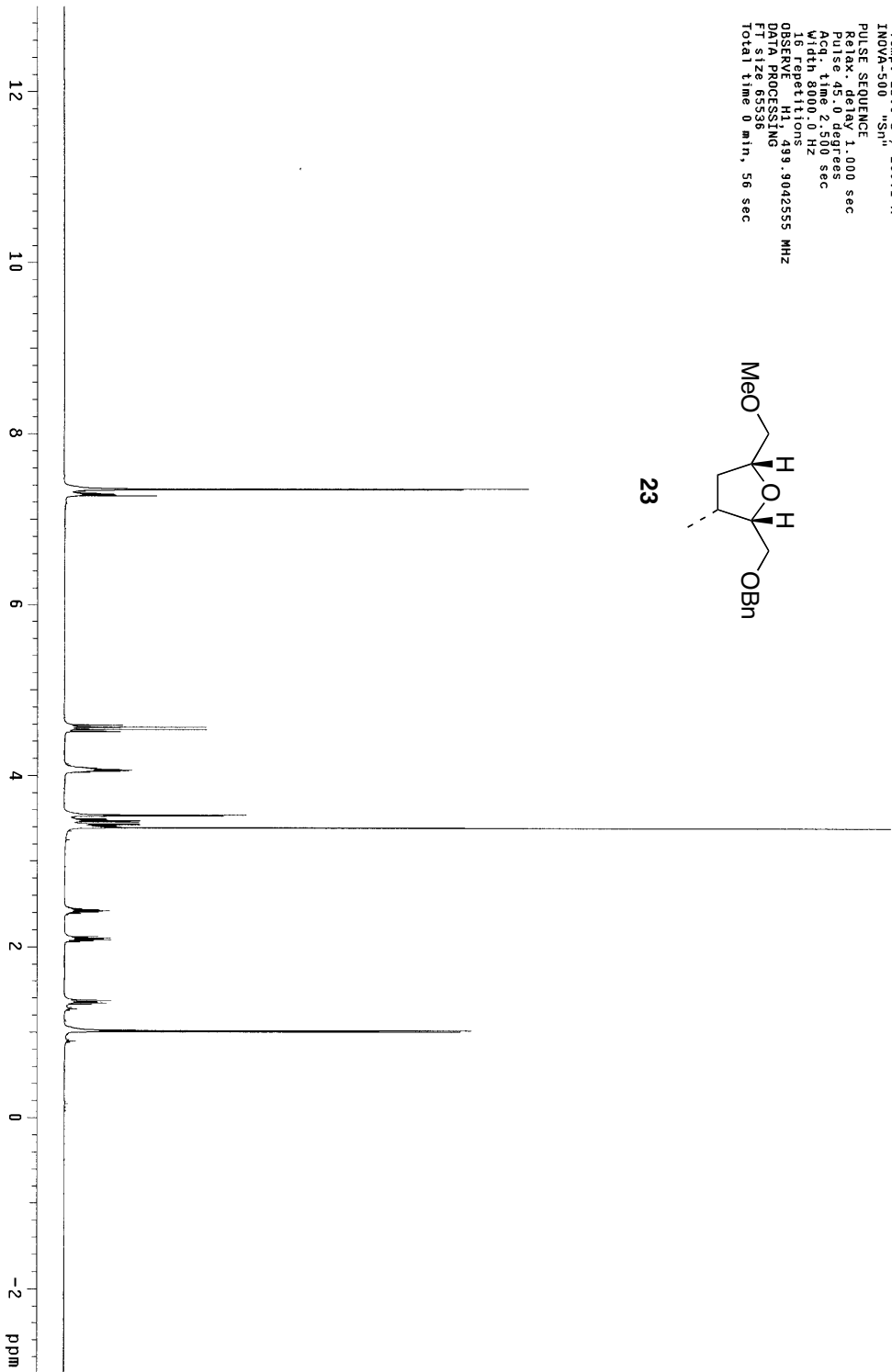
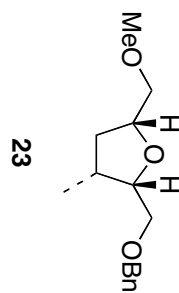
STANDARD 1H OBSERVE

Pulse Sequence: szpu1
Solvent: CDCl3
Acquisition Temperature: 300.2 K
INOVA-400
PULSE SEQUENCE
Pulse 1: 2.0000000000000000 sec
Acquisition: 2.0000000000000000 sec
Width: 6000.6 Hz
16 repetitions
OBSERVE: H1, 399.9649645 MHz
DATA PROCESSING
Line broadening: 0.2 Hz
FT size: 32788
Total time: 0 min, 42 sec



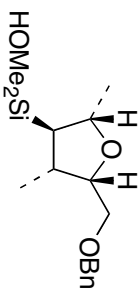
STANDARD PROTON PARAMETERS

Pulse Sequence: szpul
Solvent: CDCl3
Temp: 23.0 C / 296.1 K
INOVA-500 5H
PULSE SEQUENCE
Relax: delay 1.000 sec
Pulse: 49.0 degrees
Width: 8000.0 Hz
16 repetitions
OBSERVE: H1, 499.902555 MHz
DATA PROCESSING
FT: size 65536
Total time 0 min, 56 sec

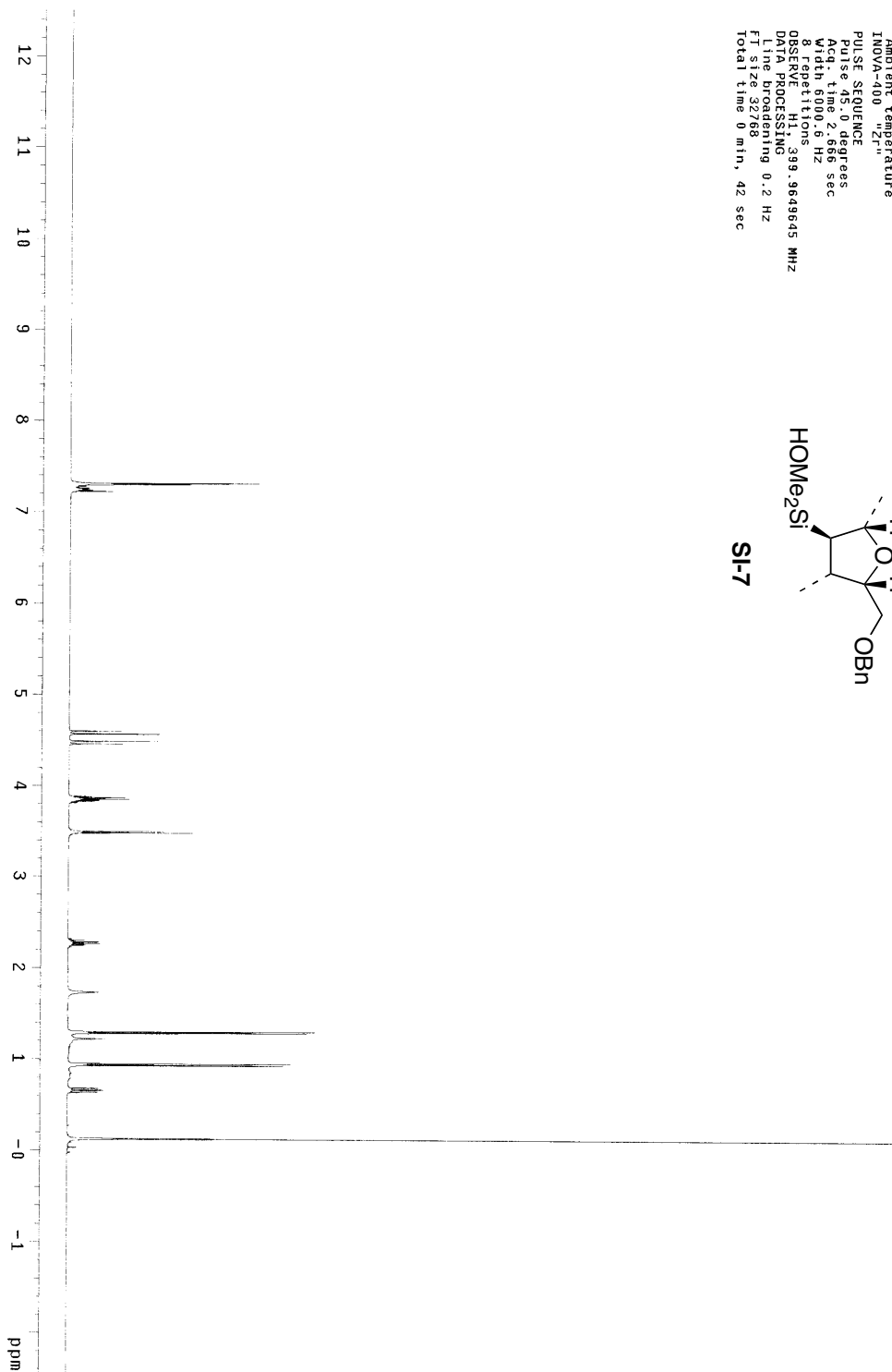


STANDARD 1H OBSERVE

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
INOVA-400 "Zr"
PULSE SEQUENCE
Pulse 45.0 degrees
Acq. time 2.666 sec
Width 6000.6 Hz
Sweep rate 399.9649645 MHz
OBSERVE: 1H
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 0 min, 42 sec



SI-7



STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

Solvent: CDCl3

Applic: zgpg30

Temperature

INOVA-500 "K"

PULSE SEQUENCE

Pulse 45.0 degrees

Acq. time 2.666 sec

Width 6000.6 Hz

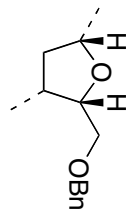
16 repetitions

OBSERVE H1, 399.9649845 MHz

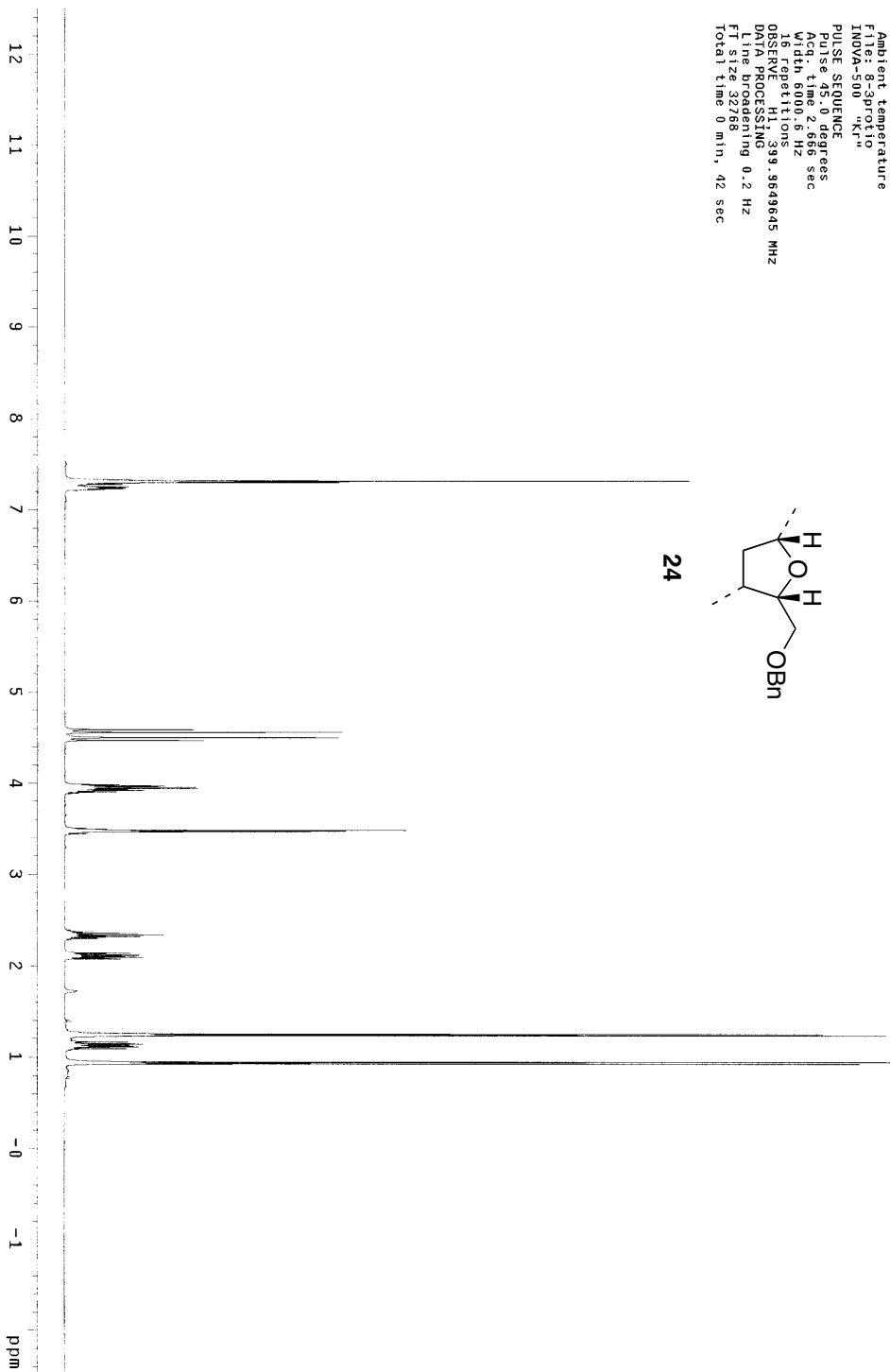
DATA PROCESSING

FT 24.32768

Total time 0 min, 42 sec

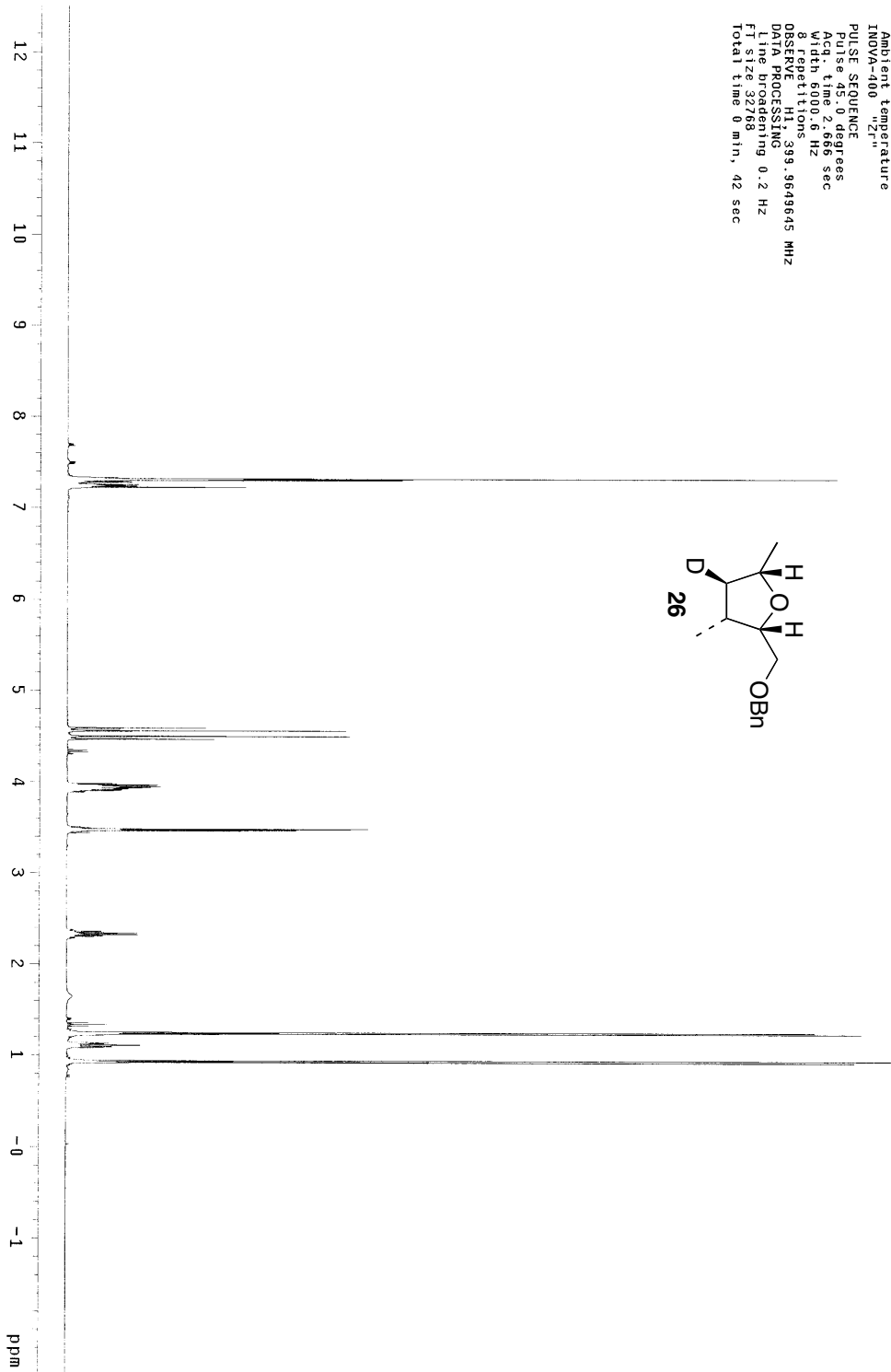
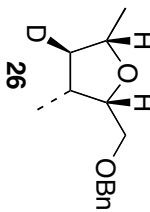


24

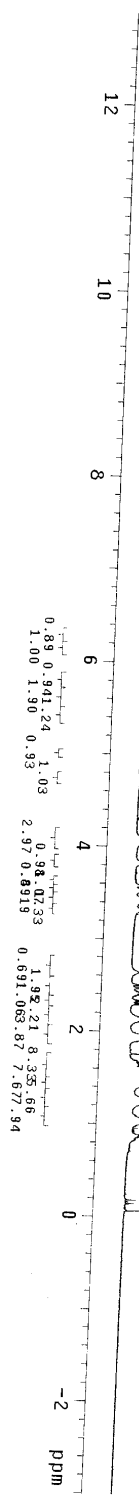
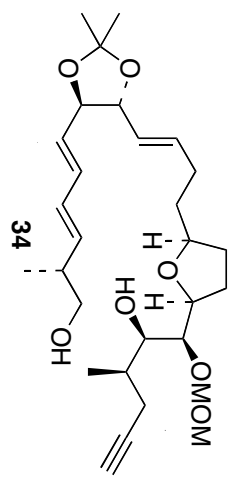


STANDARD 1H OBSERVE

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
INDVA-400 "Zr"
PULSE SEQUENCE
Pulse 45.0 degrees
Acq. time 2.666 sec
8
P1 den 810.8 Hz
OBSERVE H1 399.9649645 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 0 min, 42 sec



pv-III-31p
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 INOVA-500 "SN"
 PULSE SEQUENCE
 Relax. delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.500 sec
 Width 1297.0 Hz
 8 Repetitions
 OBSERVE H1 199.9042605 MHz
 DATA PROCESSING
 FT size 65536
 Total time 0 min, 28 sec



PV-II-175P

Pulse Sequence: s2pul1

Solvent: CDCl3

Acq. Temp: Temperature

INOVA-400

PULSE SEQUENCE

Pulse Seq: s2, degrees

Acq: 172.450 sec

Width: 6000.6 Hz

8 repetitions

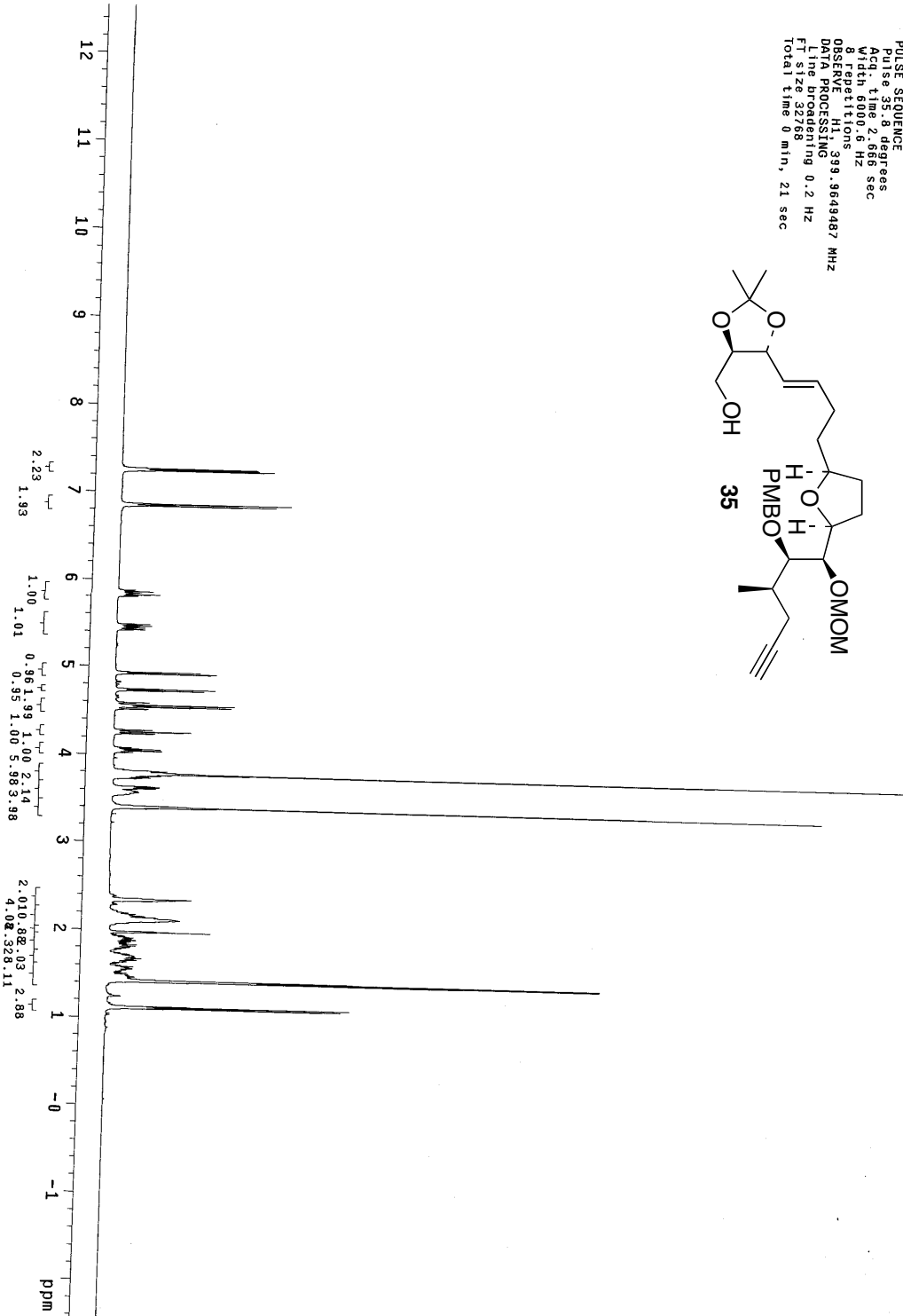
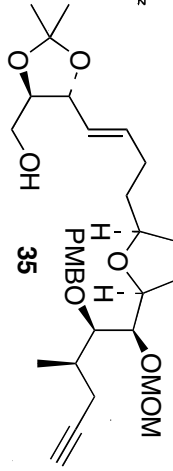
OBSERVE: H1, 399.9649487 MHz

DATA PROCESSING

Line Broadening 0.2 Hz

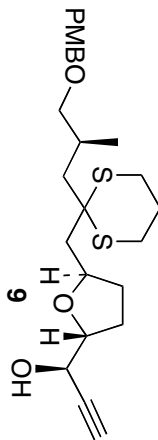
File Size 32788

Total Time 0 min, 21 sec

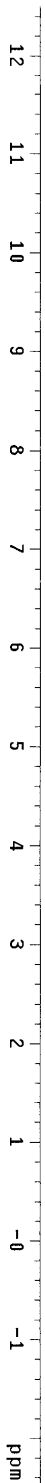


STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-400
"Zr"
PULSE SEQUENCE
Pulse 35.0 degrees
Acq. time 2.656 sec
Width 6000.6 Hz
Repetitions 99.9643645 MHz
Data Processing
Line broadening 0.2 Hz
FT size 32768
Total time 0 min, 42 sec

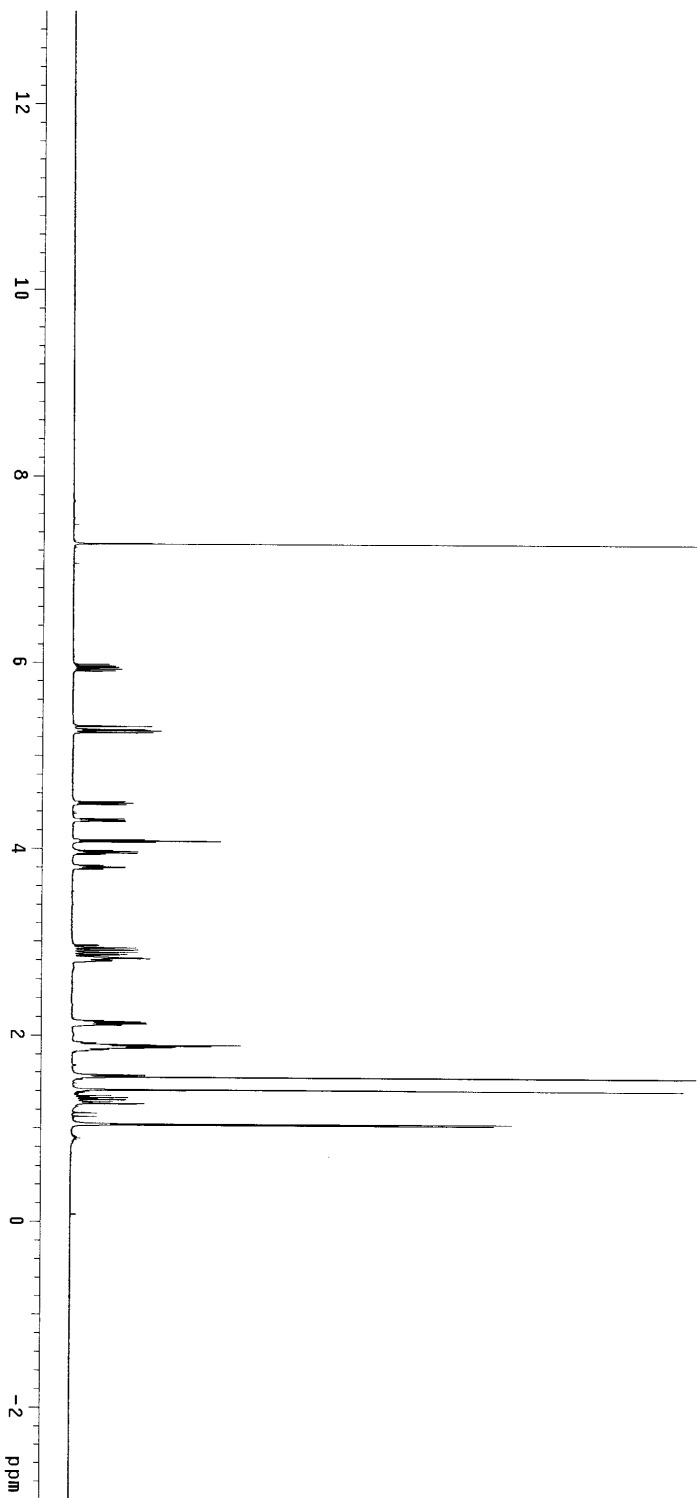
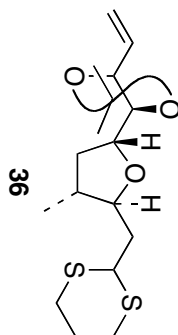


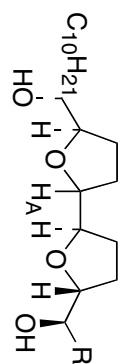
0w 400 MHz



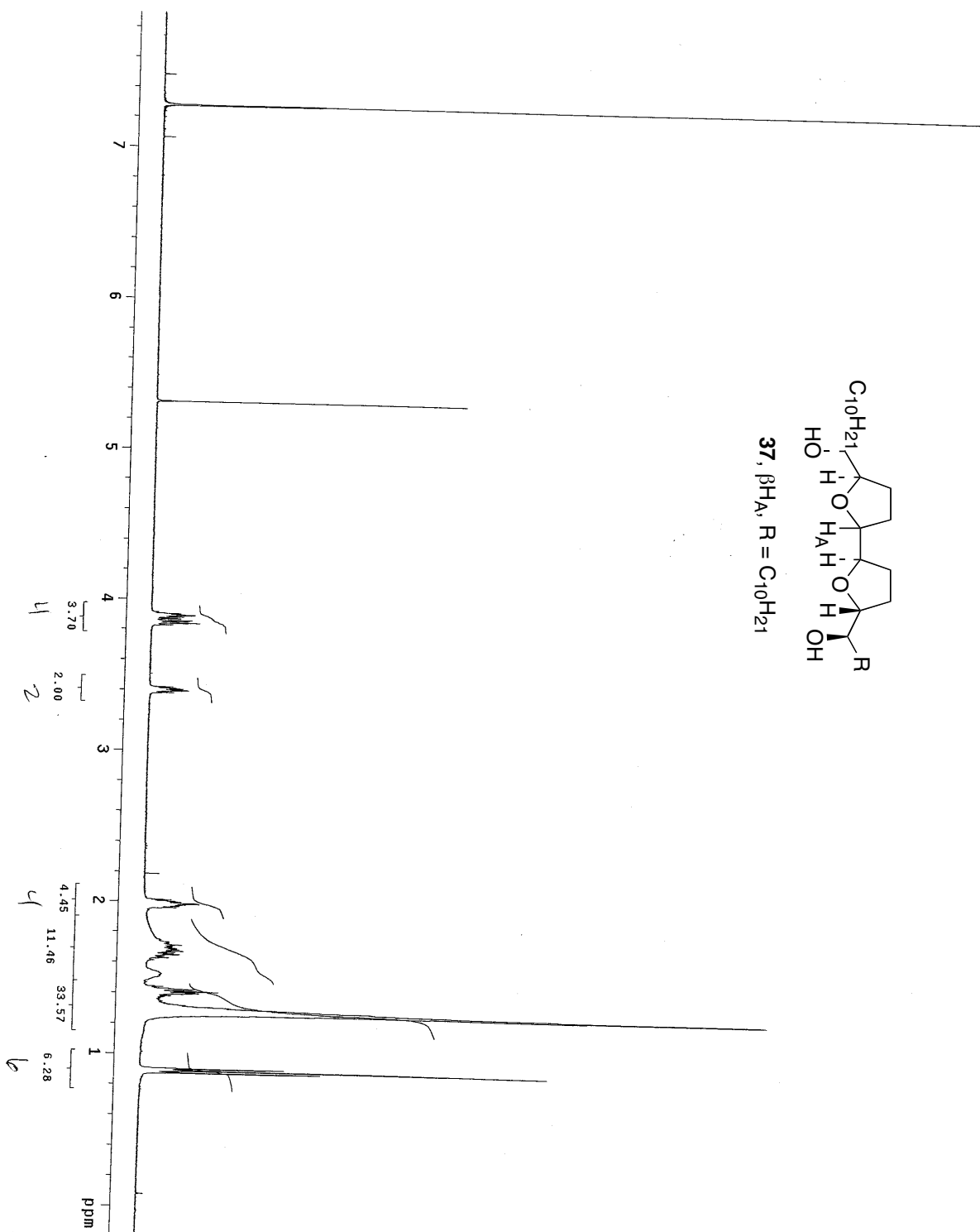
STANDARD PROTON PARAMETERS

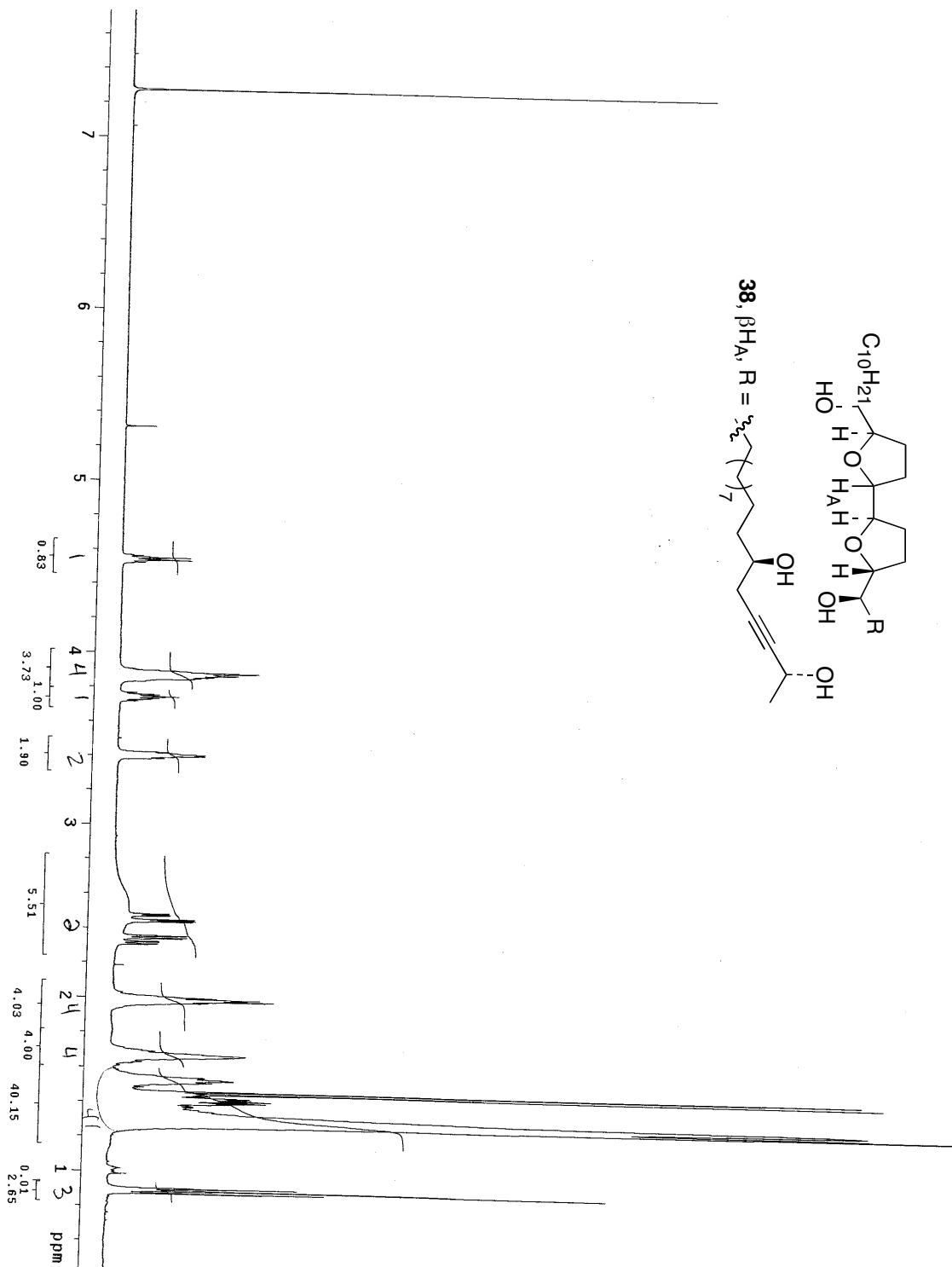
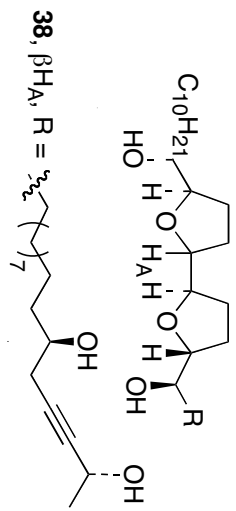
Pulse Sequence: szpu1
Solvent: CDCl3
Ambient temperature
File: jbs111-247
INOVA-500 "K1"
PULSE SEQUENCE
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.500 sec
Width 8000.0 Hz
Spectral width 99.9042555 MHz
OBSERVED
DATA PROCESSING
FT size 65536
Total time 0 min, 56 sec





37, βH_A , $\text{R} = \text{C}_{10}\text{H}_{21}$

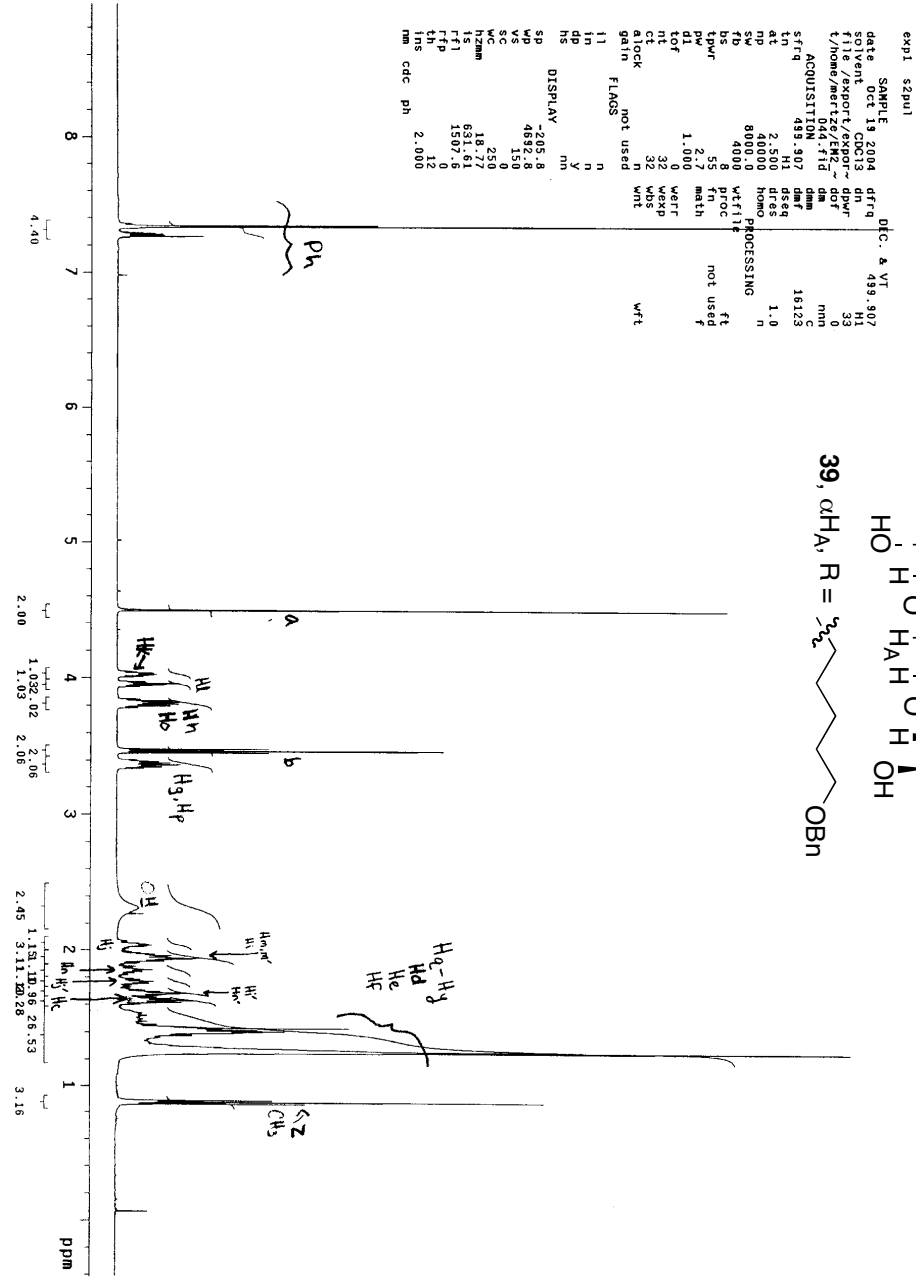
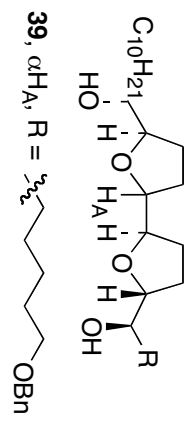




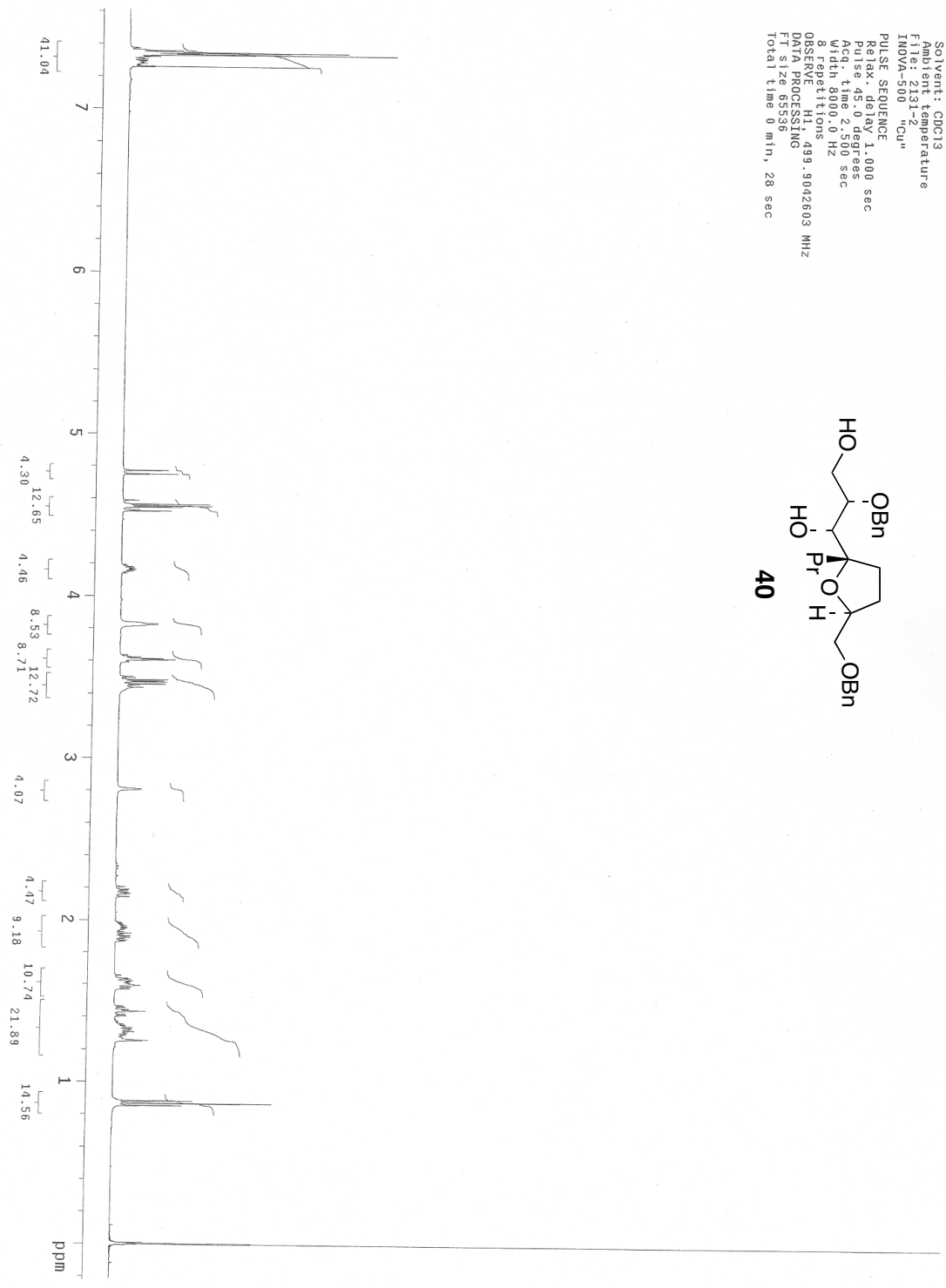
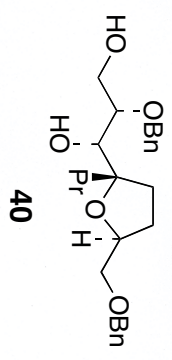
STANDARD PROTON PARAMETERS

```

SAMPLE
date Oct 19 2004      dfrq 499.907
solvent CDCl3         dn
file /export/export-  dprv
/home/mrl244/111     dm
ACQUISITION          dnm
sfrq 499.907         daf 16123
an 2.50             dss 1.0
ap 40000            homo
sw 8000.0           wffile
hd 4000             wffile
tpwr 55             fn
pw 2.7              math
d1f 1.000          uort
n1f 32             wds
ct 32              wnt
clock not used
gain not used
fl1 n
fl2 n
fl3 n
fl4 n
fl5 n
fl6 n
fl7 n
fl8 n
fl9 n
fl10 n
fl11 n
fl12 n
nm cdc ph 2.000
  
```



2131-1
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 File: 2131-2
 INOVA-500 "C"u"
 PULSE SEQUENCE
 Relax delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.500 sec
 Width 8000.0 Hz
 8 repetitions
 OBSERVE H1, 499.9042603 MHz
 DATA PROCESSING
 F1 size 65536
 Total time 0 min, 28 sec



2131-1 after HPLC 11mg
 Pulse Sequence: s2pu1
 Solvent: CDCl3
 Ambient temperature
 INOVA-500 "Sn"
 PULSE SEQUENCE
 Relax: delay 1.000 sec
 Pulse 45.0 degrees
 Acq. time 2.500 sec
 Width 8000.0 Hz
 8 Repetitions
 00SRF0004.H1: 499.9042613 MHz
 D1: 6.128065536
 FT: 6128065536
 Total time 0 min, 28 sec

