

1,5- α -D-Mannoseptanosides, Ring-size Isomers That Are Impervious to α -Mannosidase-catalyzed Hydrolysis

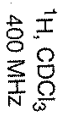
Matthew A. Boone, Frank E. McDonald,* Joseph Lichter, Stefan Lutz, Rui Cao, and
Kenneth I. Hardcastle

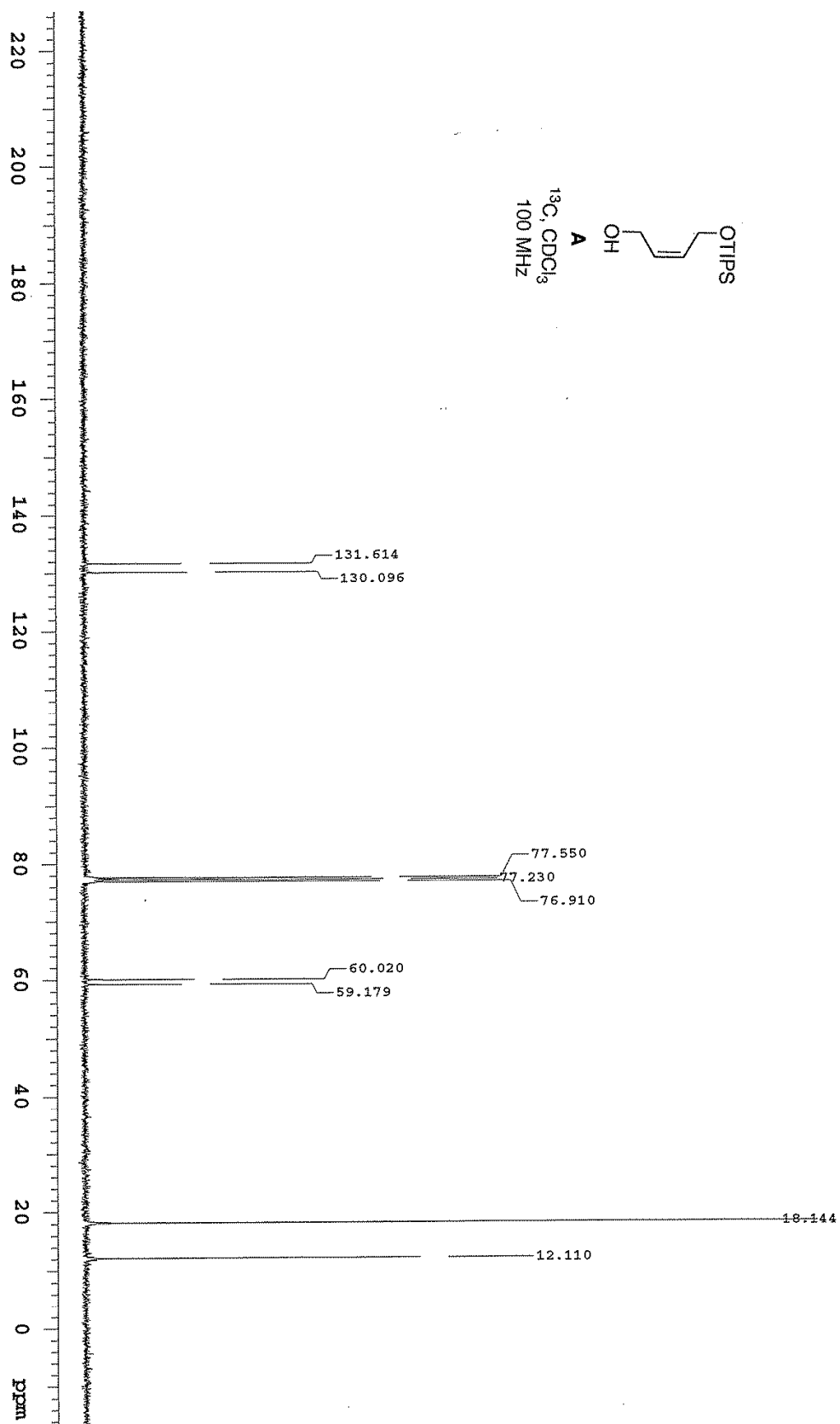
Department of Chemistry, Emory University, Atlanta, GA 30322

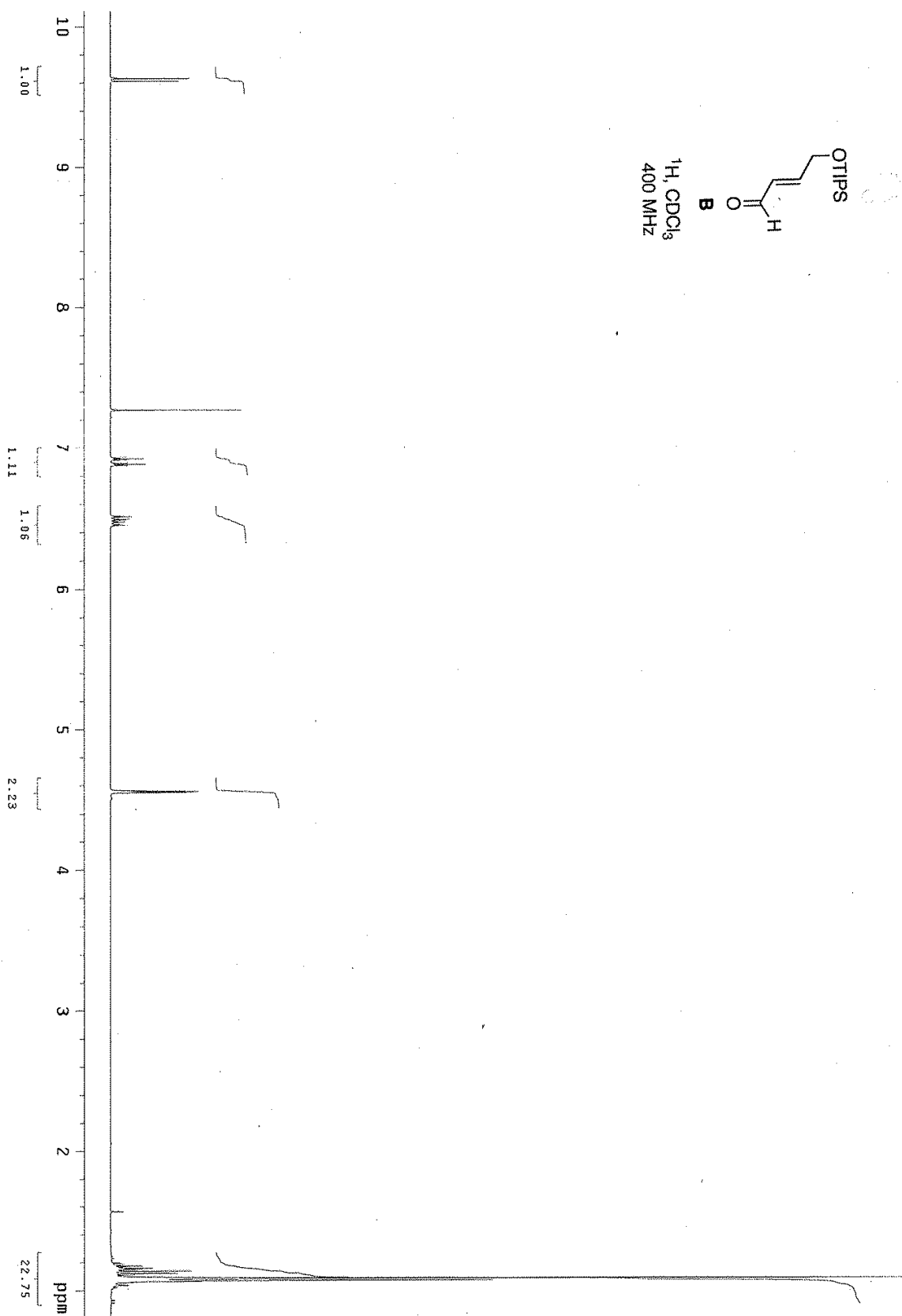
Supporting Information - part 2

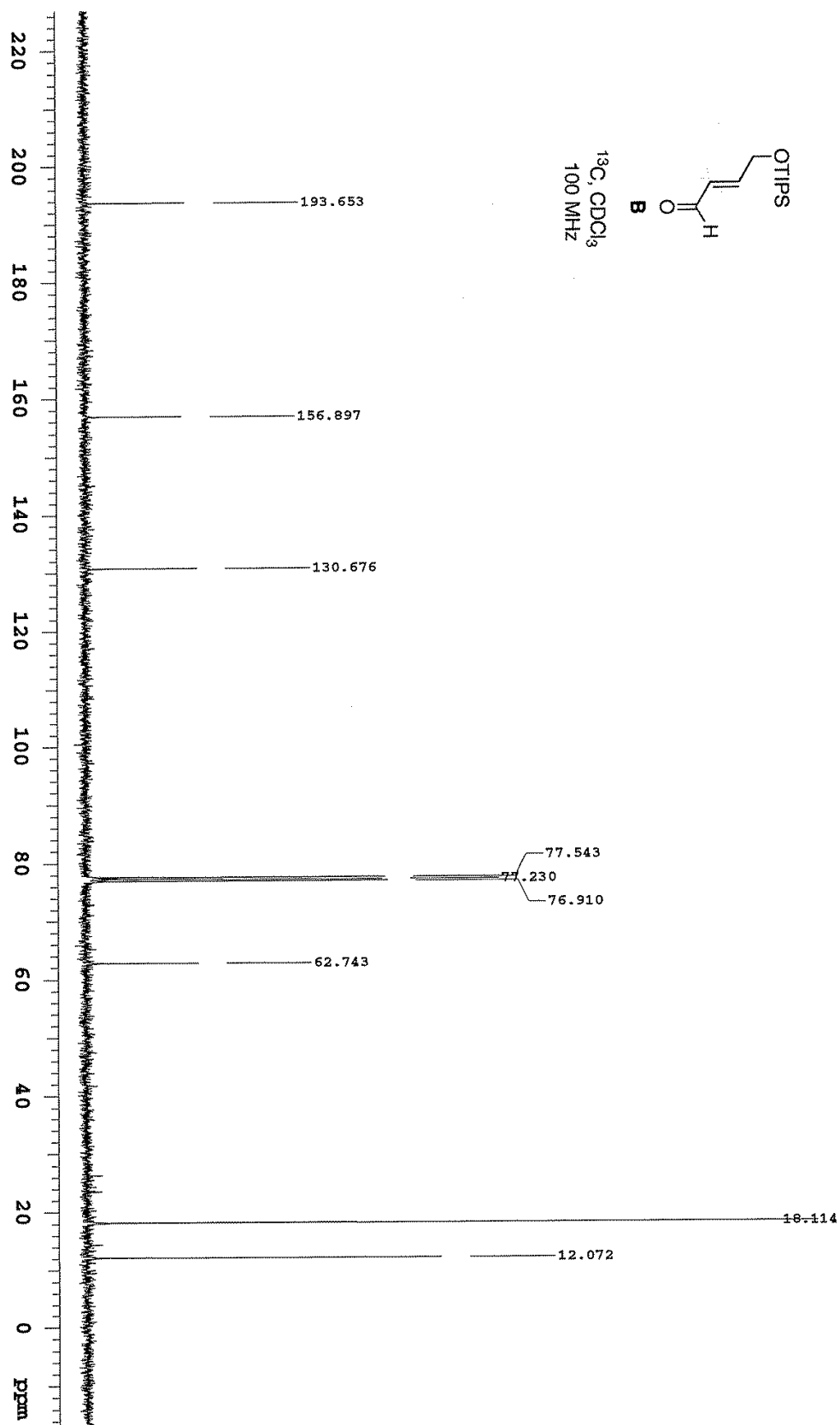
^1H and ^{13}C NMR spectra for new compounds

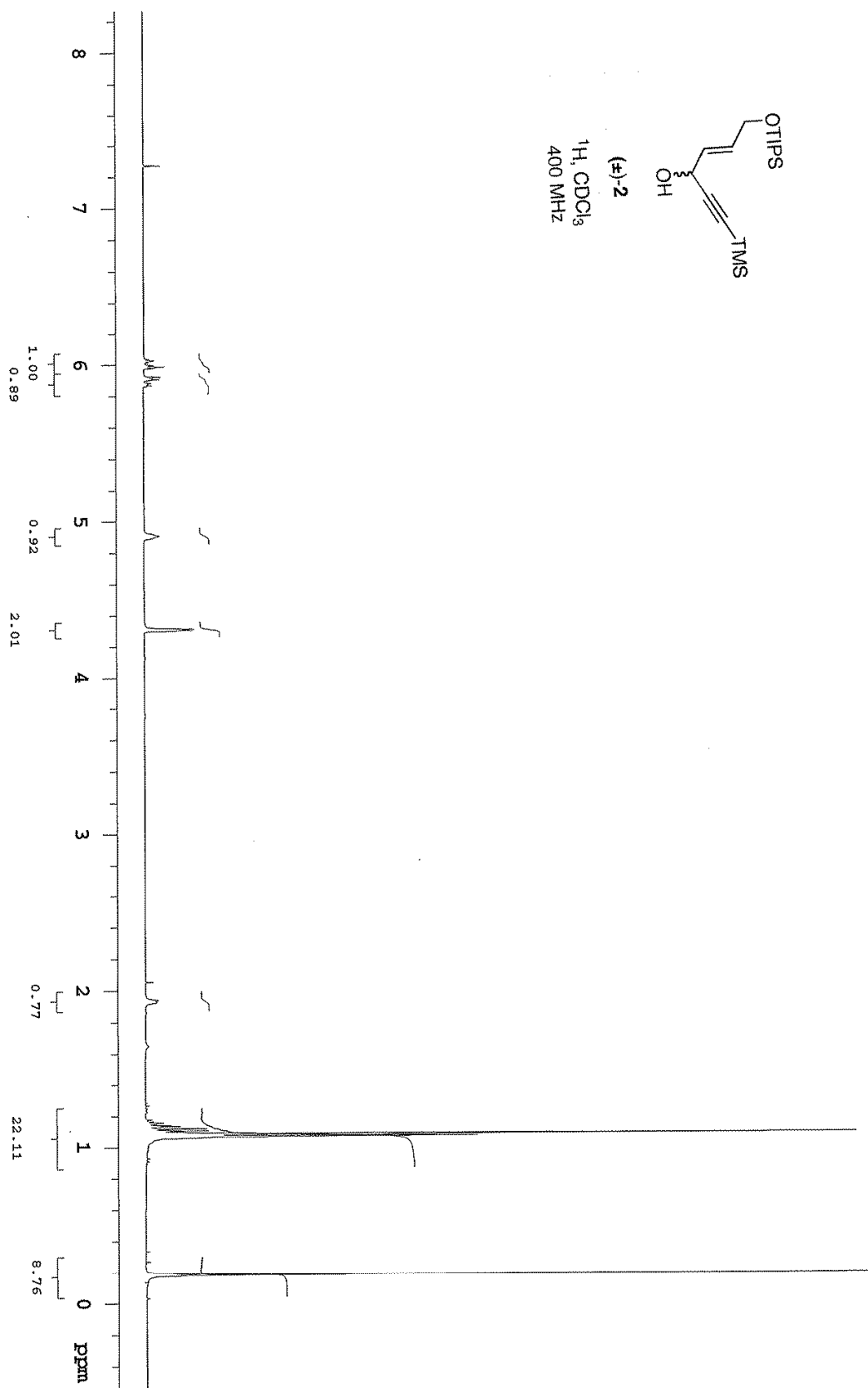
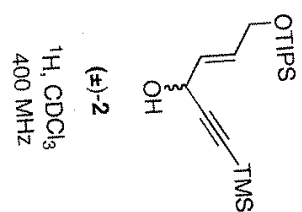
Synthesis of D-arabinoseptanose glycals 9 - 13	61 - 92
Preparation of various D-mannoseptanoside derivatives 16 and 21 - 25	93 - 122
Synthesis of D-mannoseptanosyl di- and trisaccharides 31 - 33	123 - 142





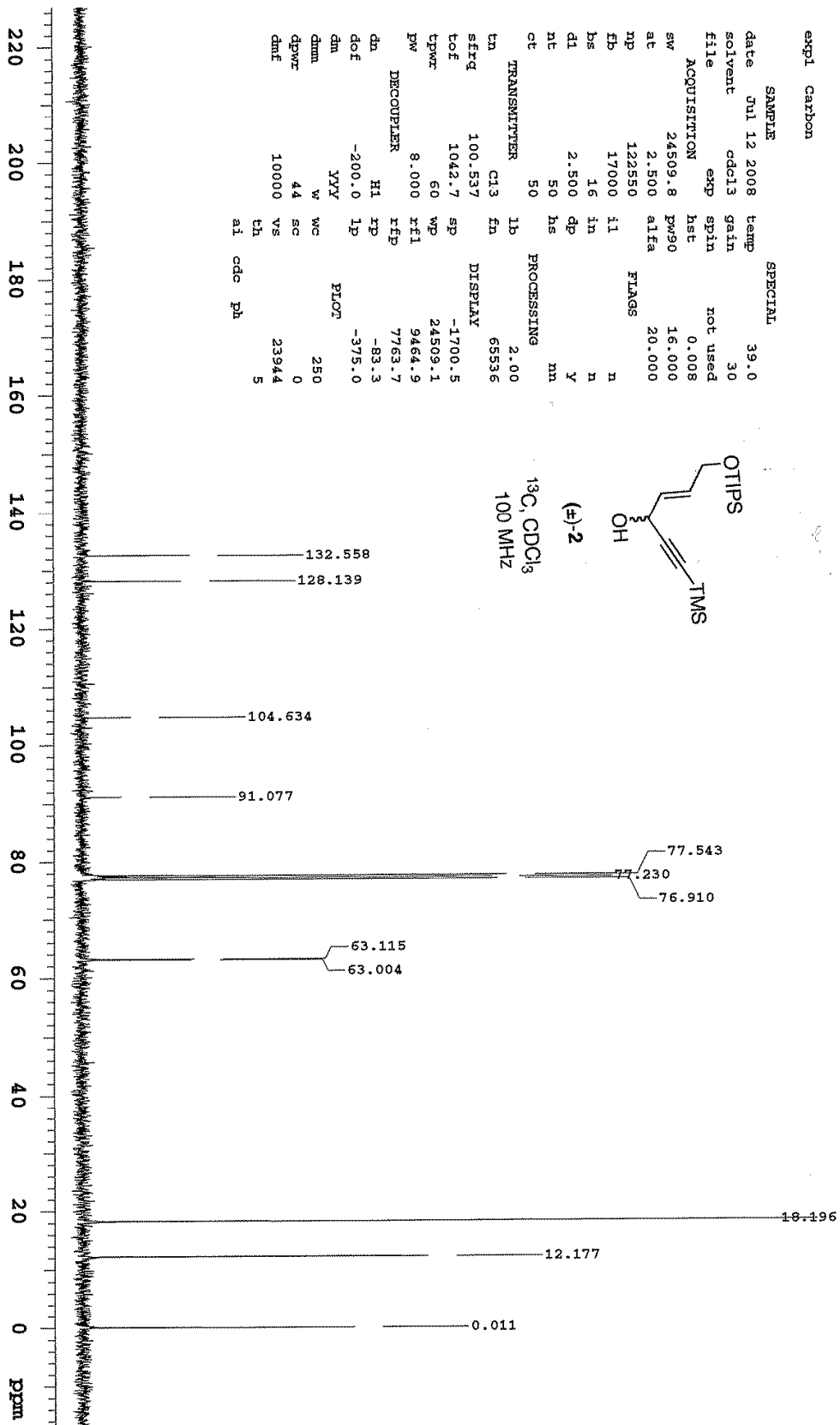
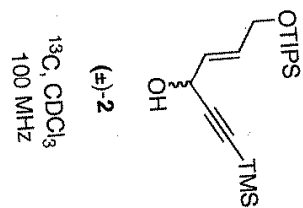


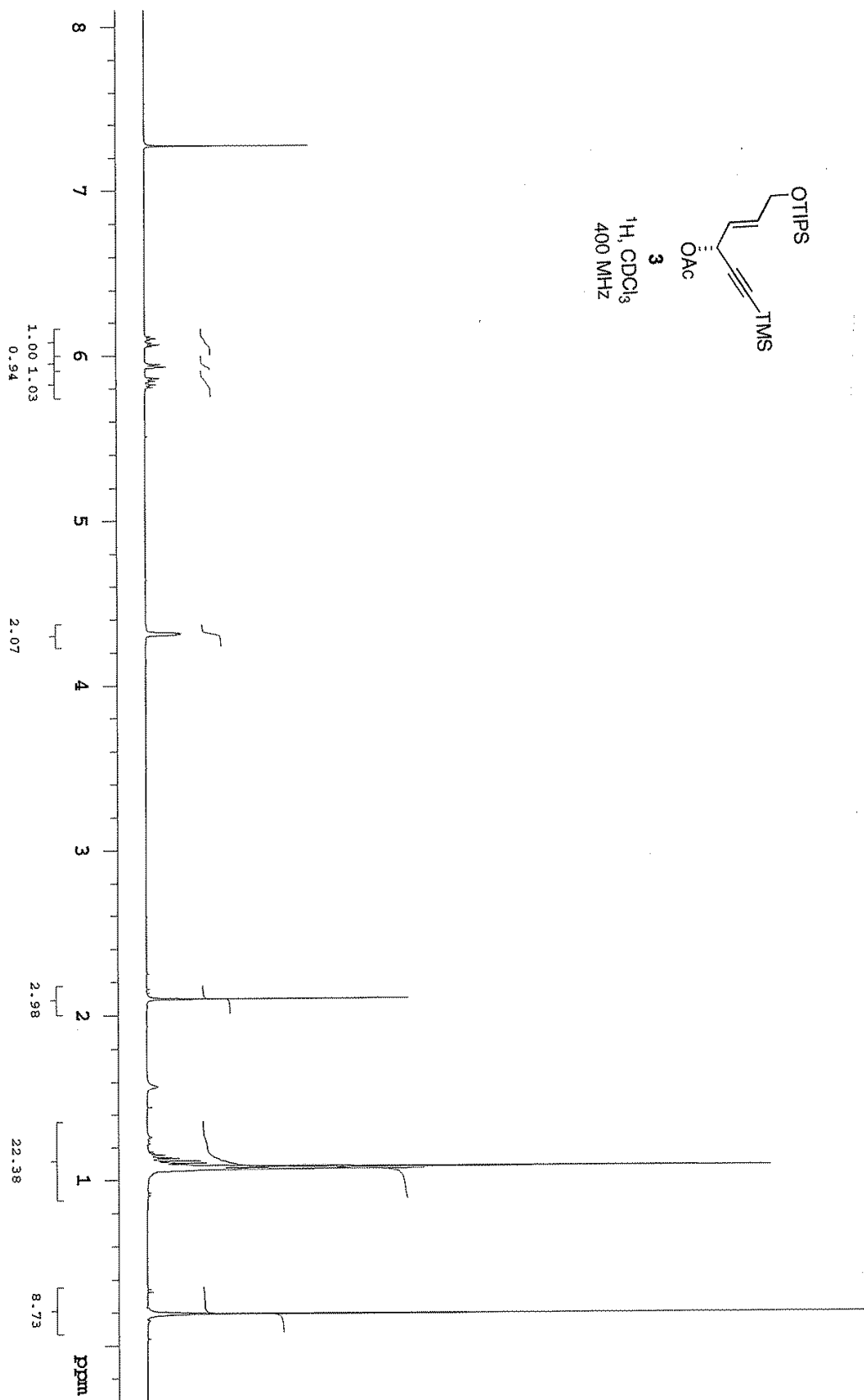


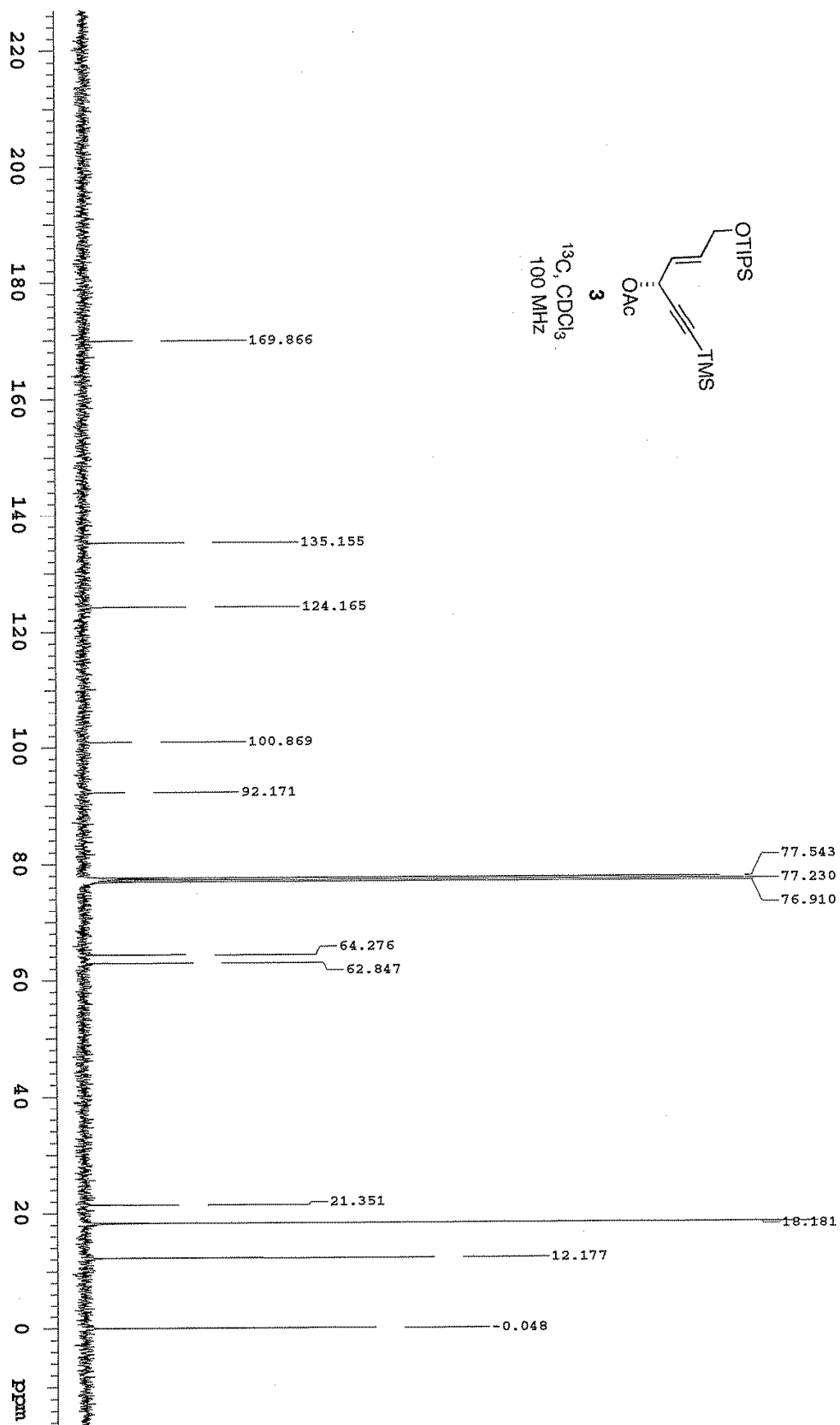


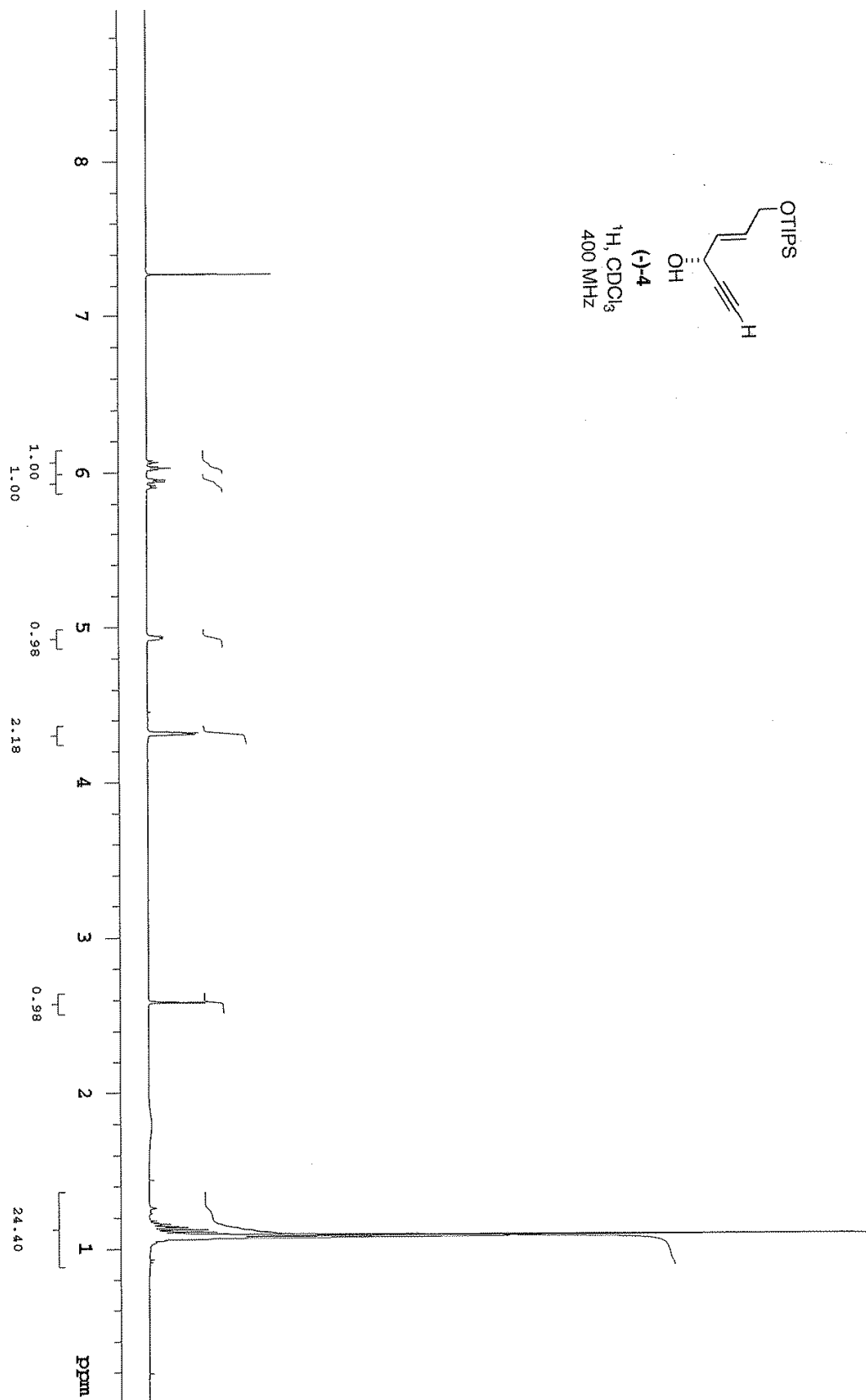
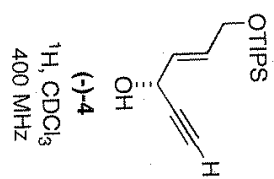
expt Carbon

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at 2.500 alfa 20.000
np 122550 FLAGS
fb 17000 il n
bs 16 in n
dl 2.500 dp y
nt 50 hs nm
ct 50
TRANSMITTER lb
tn c13 fn 65536
sfrq 100.537 sp -1700.5
tof 1042.7 xfl 24509.1
tpwr 60 wp 9464.9
pw 8.000 rfp 7763.7
DECOUPLER H1 ip -83.3
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dnf YYY PLOT 250
dmu w wc
dpm 44 sc 0
dpr 10000 vs 23944
dmf th 5
al cdc ph





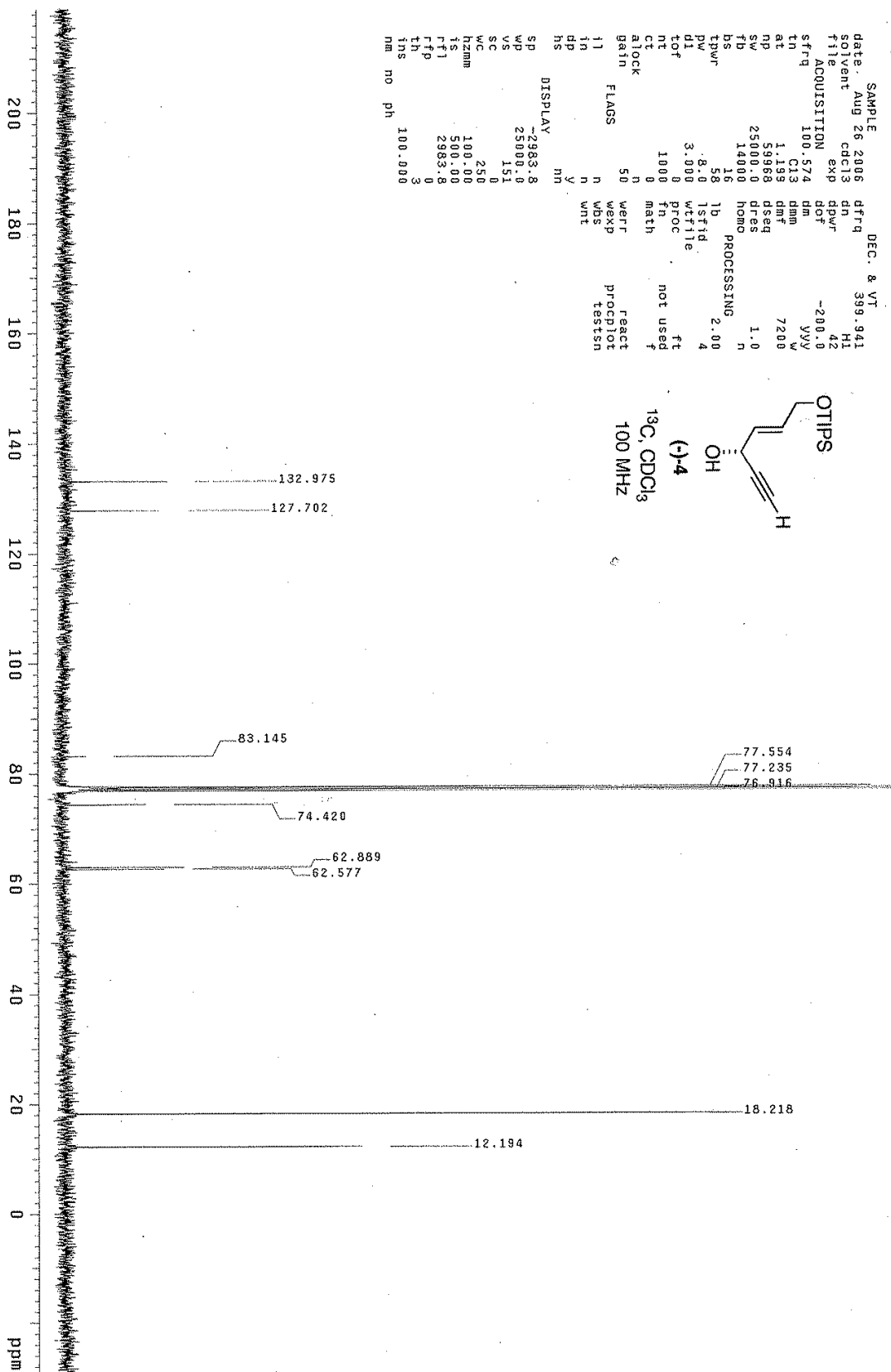
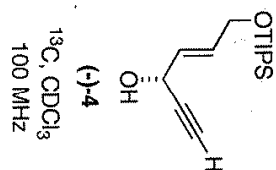


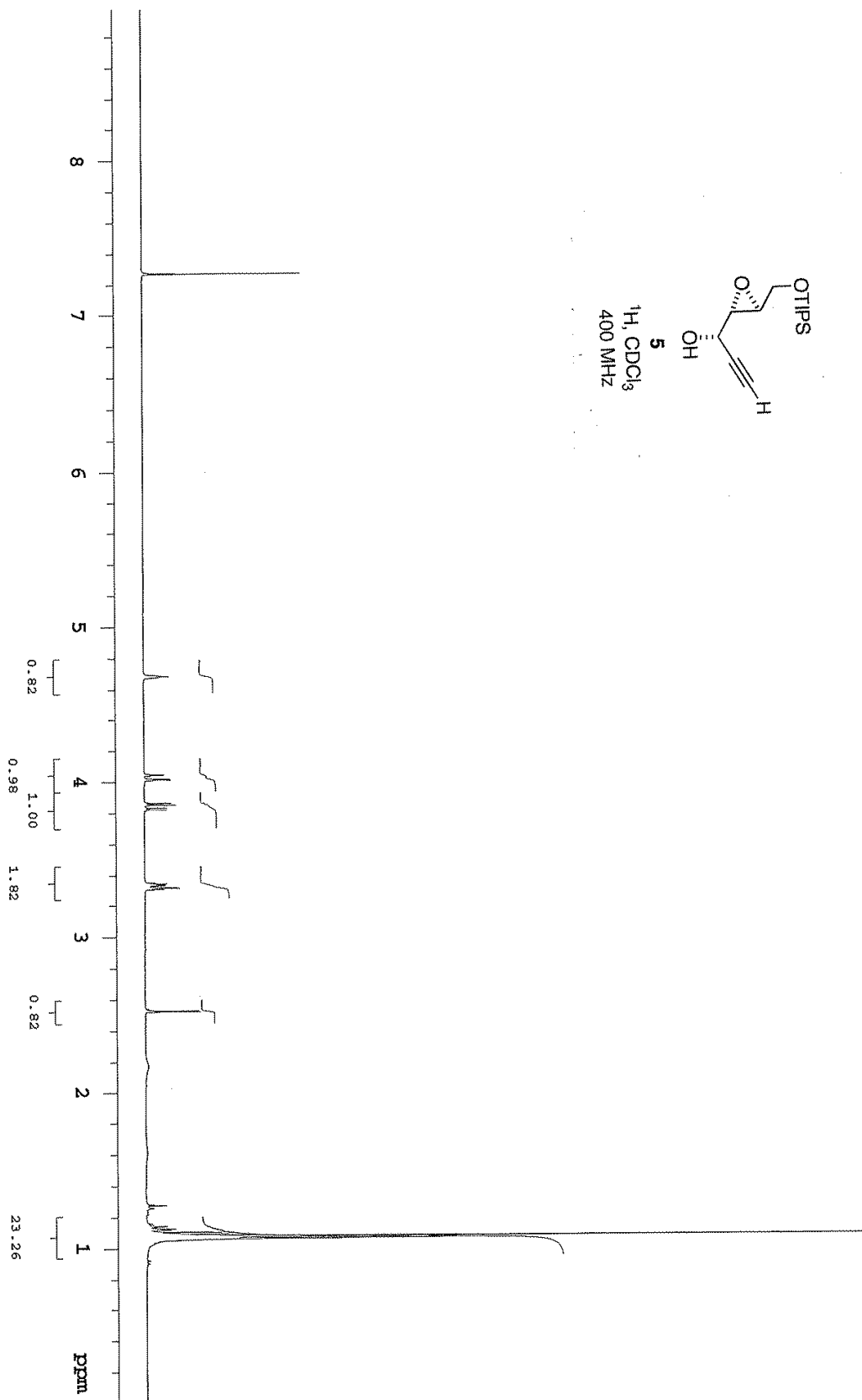
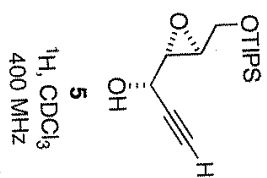


13C OBSERVE

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sfrq 100.574 dm yyy 7200
in C13 dam w
at 1.199 dmf 1.0
np 59968 dseg
sw 25000.0 dres
fb 14000 homo
bs 16
tpwr 58 1b PROCESSING 2.00
pw -8.0 1sfid 4
dl 3.000 wfile ft
tof 0 proc not used
nt 1000 fn math f
ct 0
alock n
gain 50
FLAOS n
il n
in n
dp n
hs y
DISPLAY
sp -2983.8
wp 2500.0
vs 151
sc 0
wc 250
hzmm 100.00
is 500.00
rf1 2983.8
rfp 0
th 3
ins nm
no ph 100.000

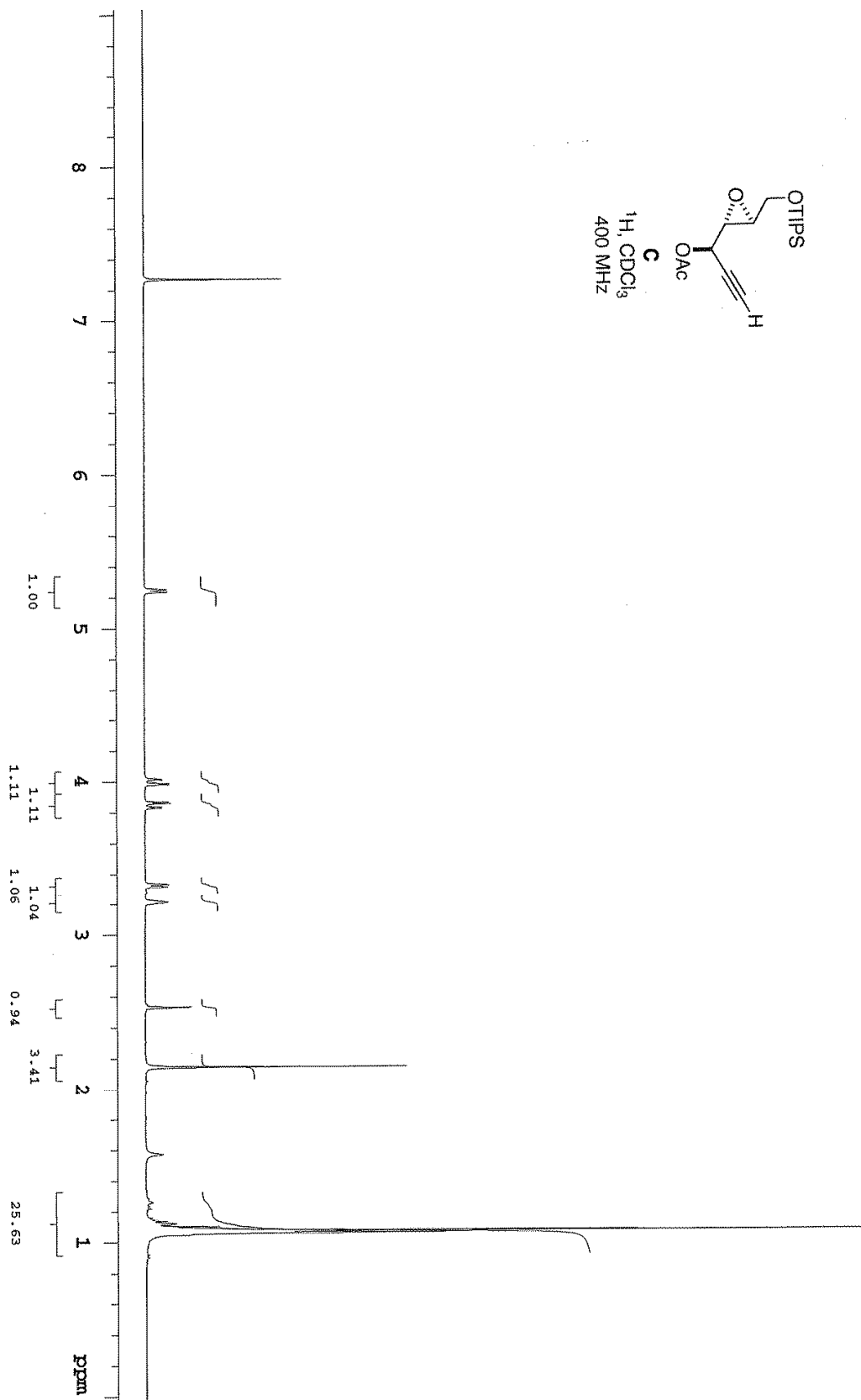
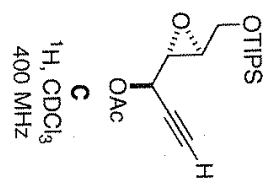


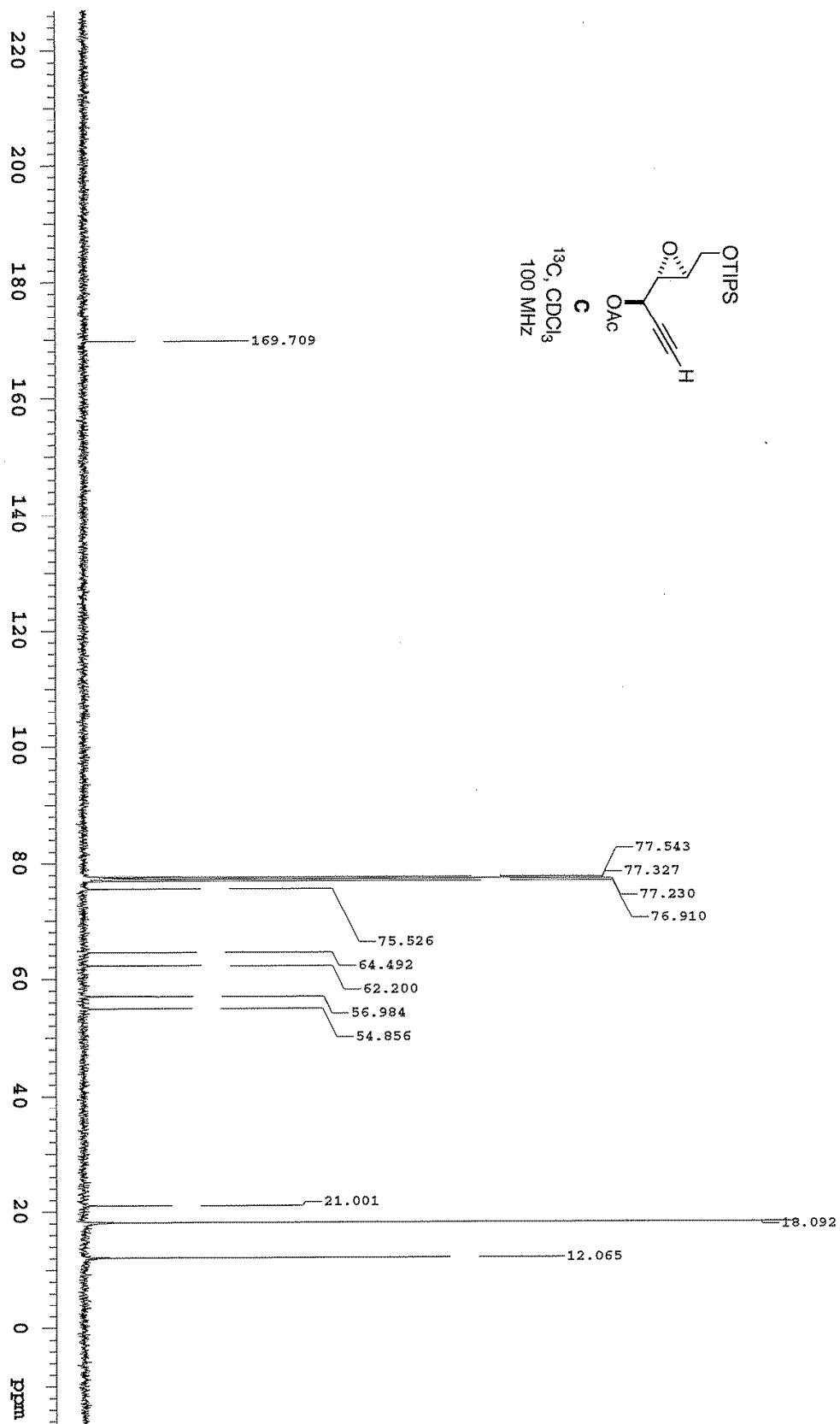


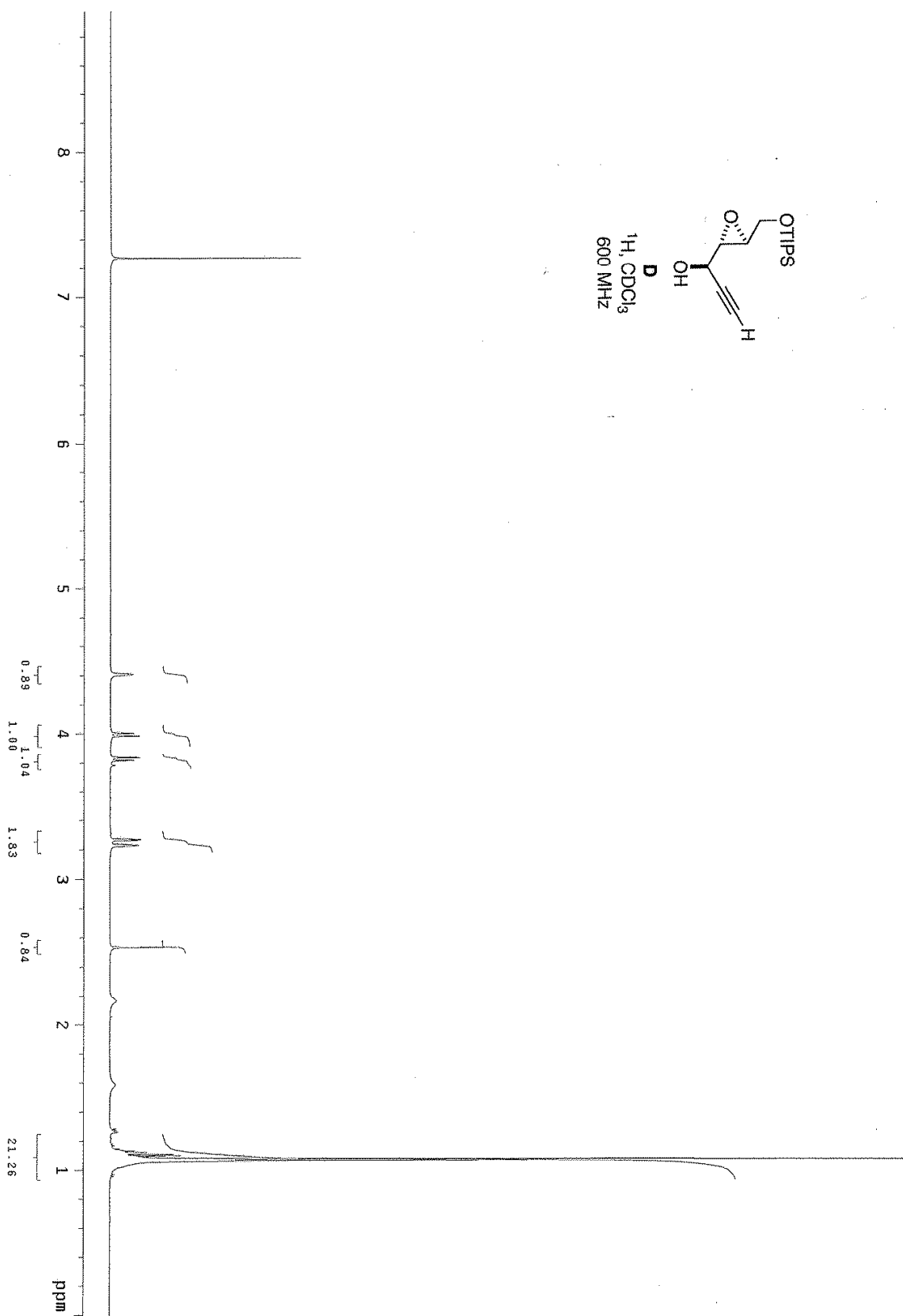
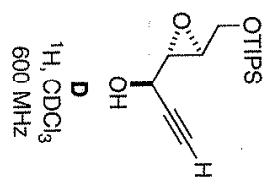
exp1 std13c

¹³C, CDCl₃
 100 MHz
5





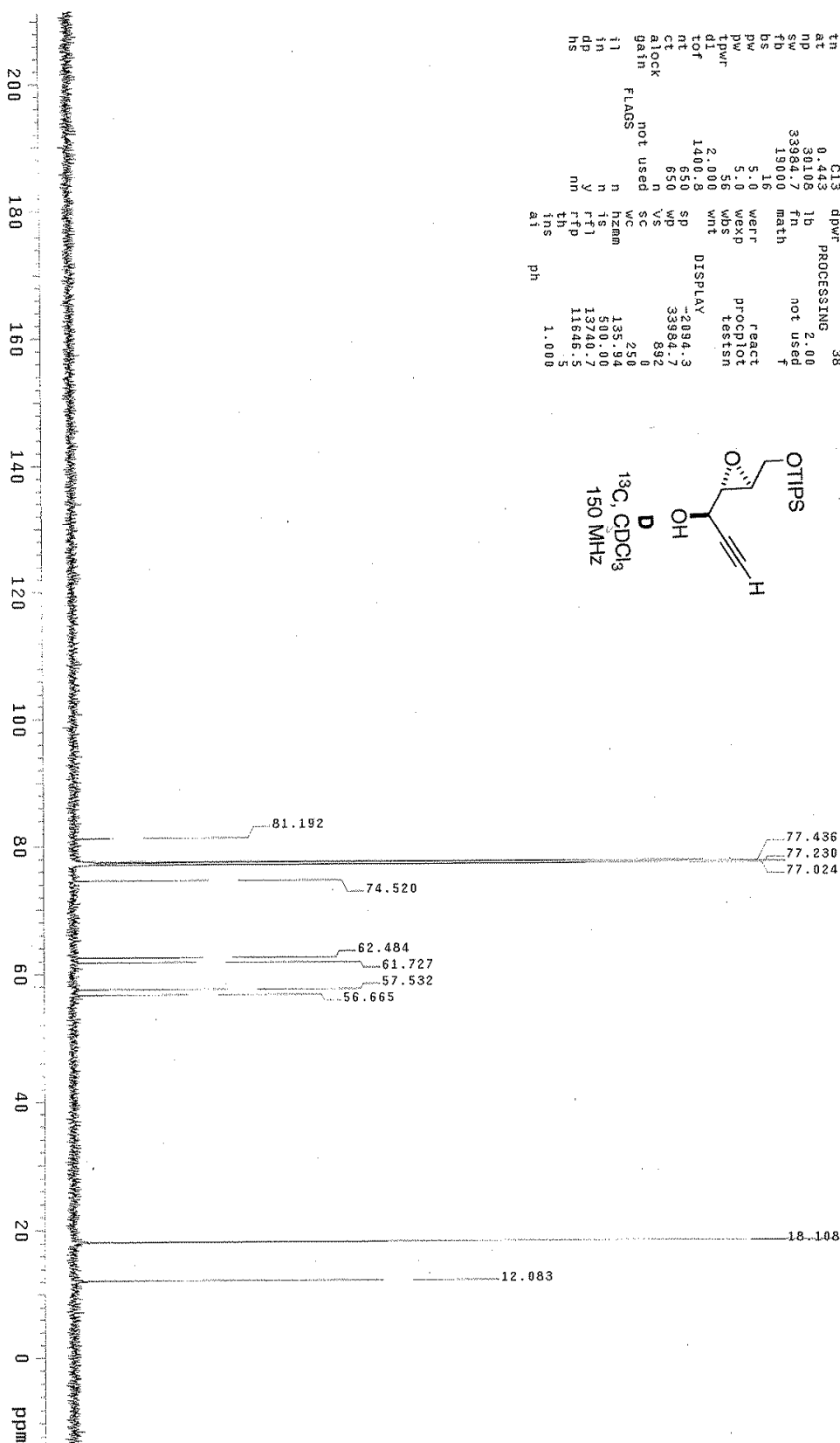
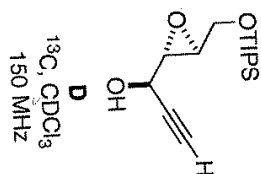




13C SENSITIVITY
ASTM SAMPLE

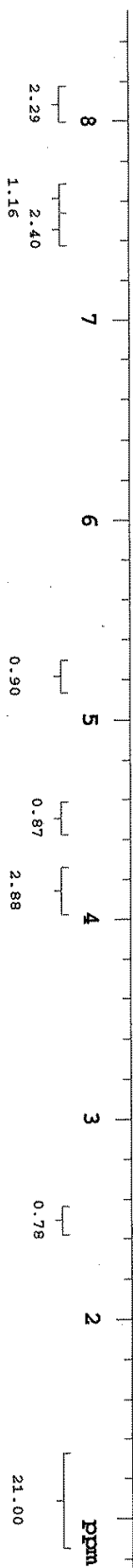
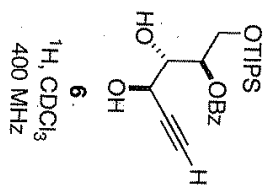
exp2 s2pul

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file	exp	dm	yy
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sfrq	150.817	dnt	38
at	0.413	dpwr	2.00
np	30108	fn	not used
sw	33984.7	math	f
td	15000	wert	react
bs	15000	wexp	proclot
pw	5.0	wbs	testis
tpwr	56	wnt	DISPLAY
tpwr	2.000	sp	-2094.3
tpwr	1400.8	wd	33984.7
tpwr	650	vs	832
tpwr	650	sc	0
tpwr	650	hzm	135.34
tpwr	650	ts	500.00
tpwr	650	rfl	13740.7
tpwr	650	rfl	11646.5
tpwr	650	th	5
tpwr	650	ai	1.000
tpwr	650	ph	



expt1 Proton

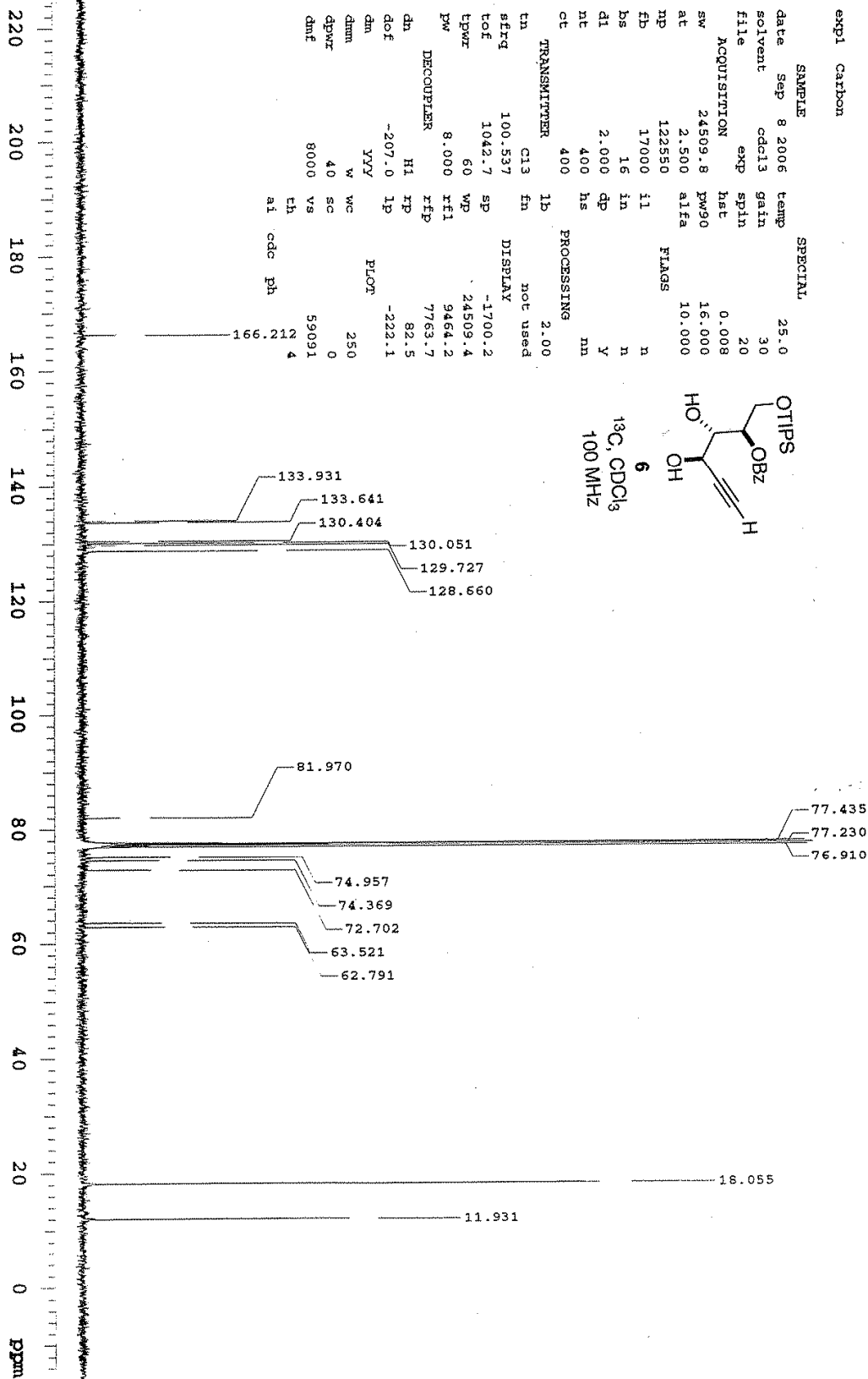
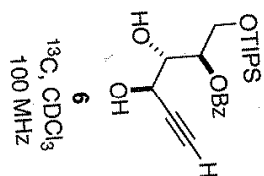
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file exp hst 20
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np 26264 flags 6.600
fb 4000 il n
bs 16 in n
dl 1.500 dp y
nt 8 hs nn
ct 8
TRANSMITTER lb 0.20
tn H1 fn 32768
sfreq 399.790 DISPLAY 289.8
tof 399.4 sp 3132.0
tpwr 60 wp 3702.0
pw 4.750 rfl 2906.5
DECOUPLER C13 rfp 168.0
dn C13 rp -25.1
dof 0 lp
dm nmnm PLOT 250
dmu c wc 0
dmu 37 sc 515
dpr 29412 vs 12
dmf th
ai cdc ph

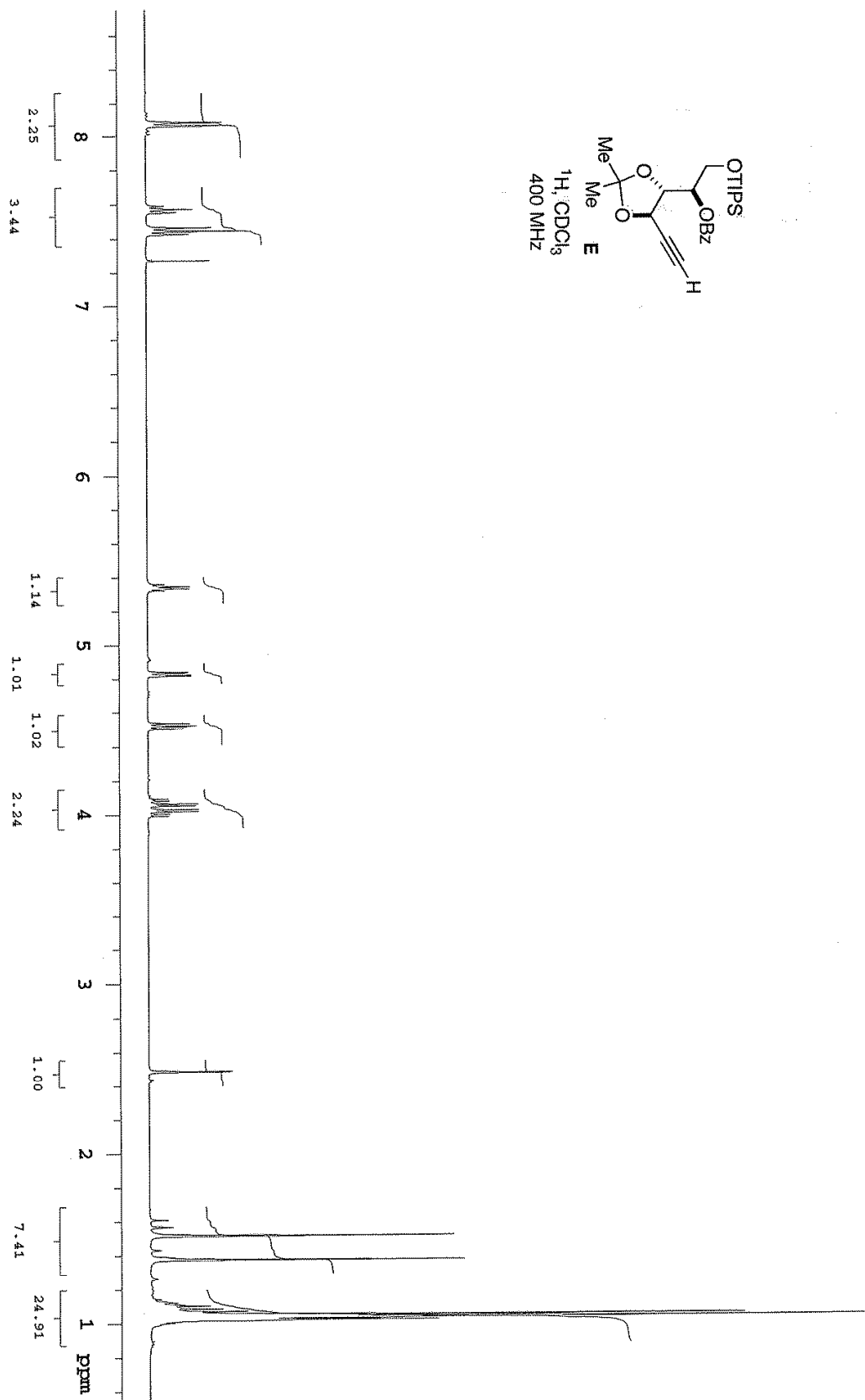
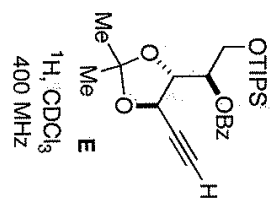


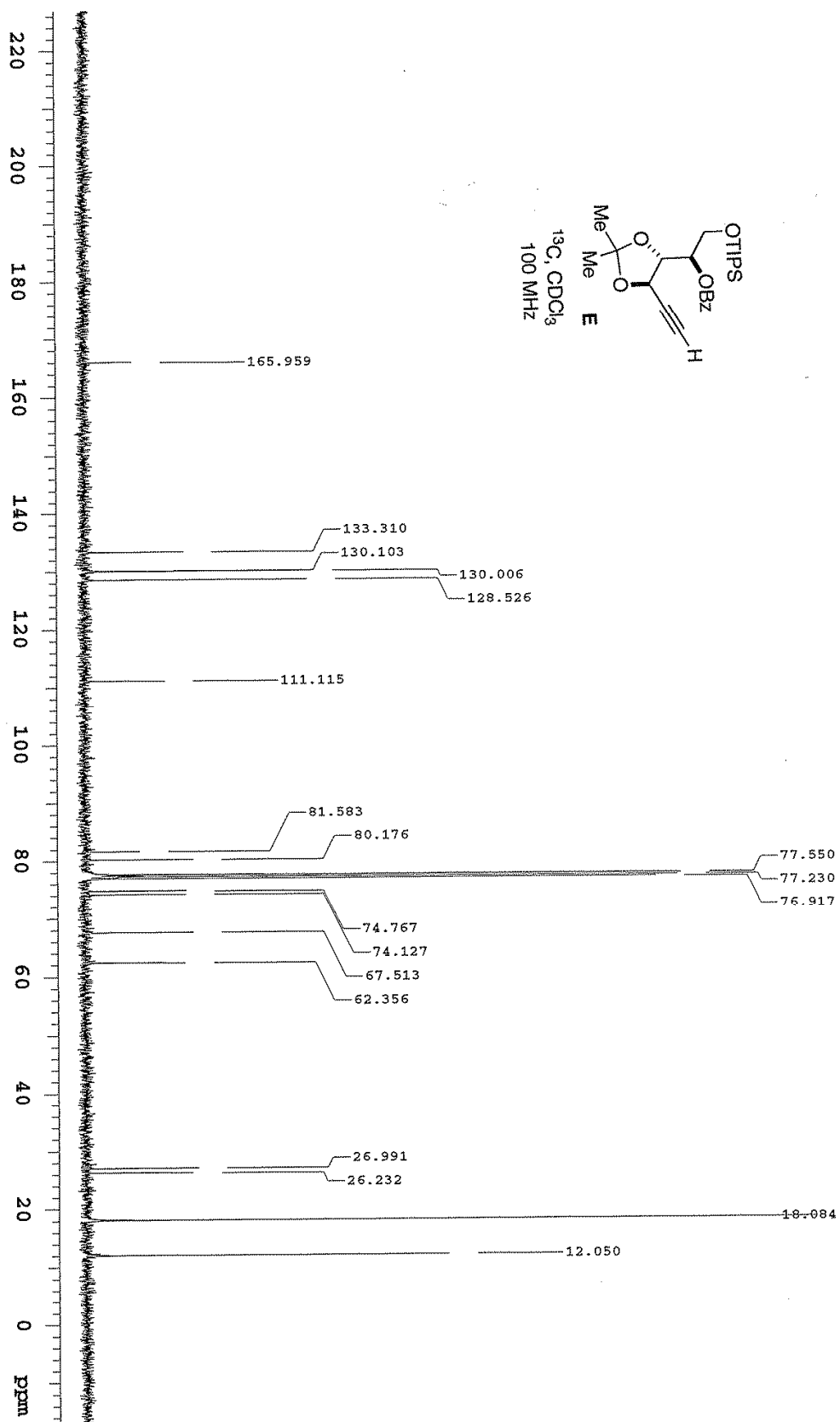
expt Carbon

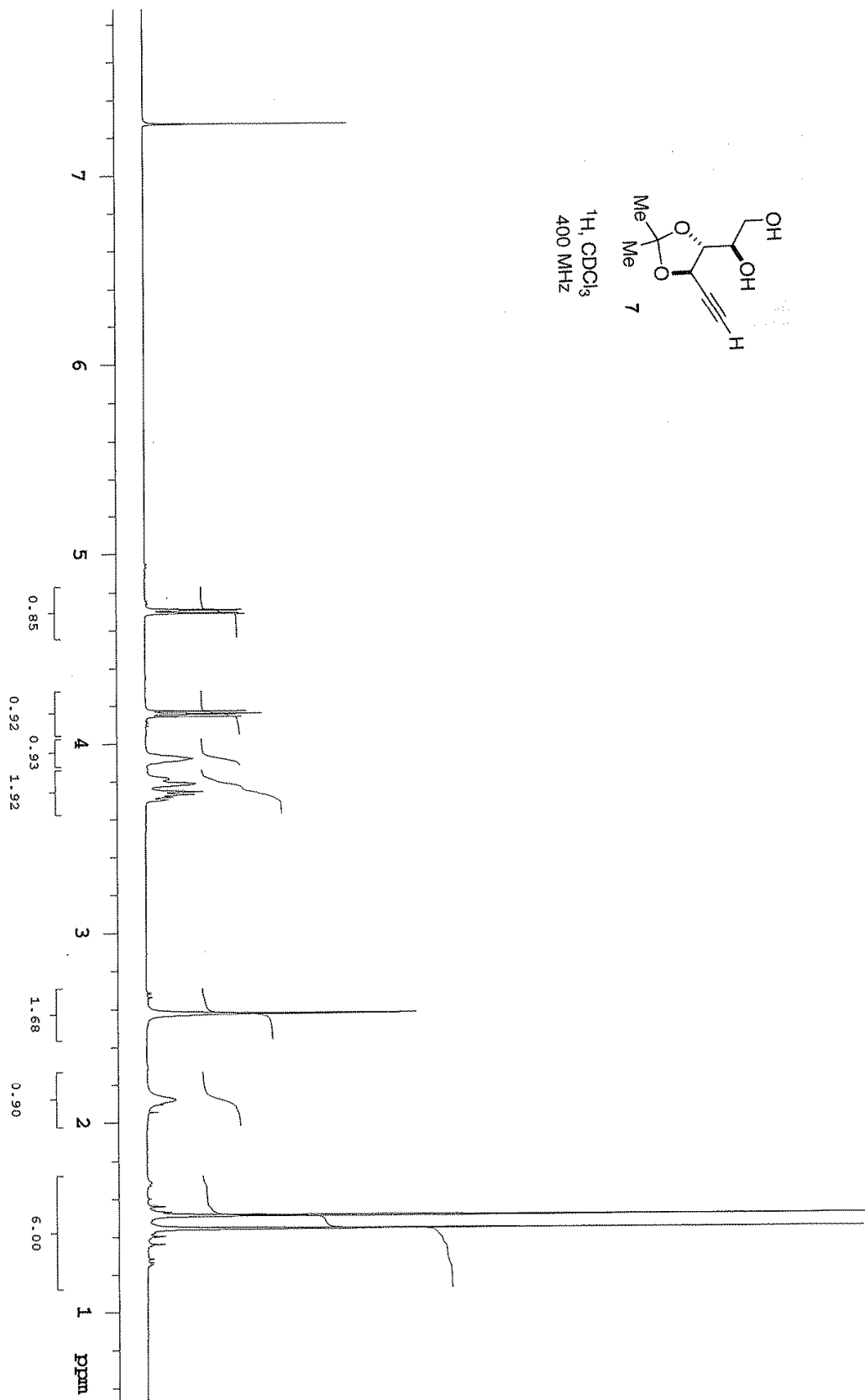
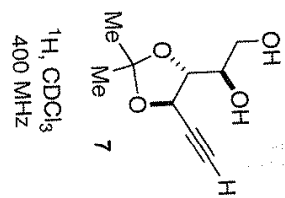
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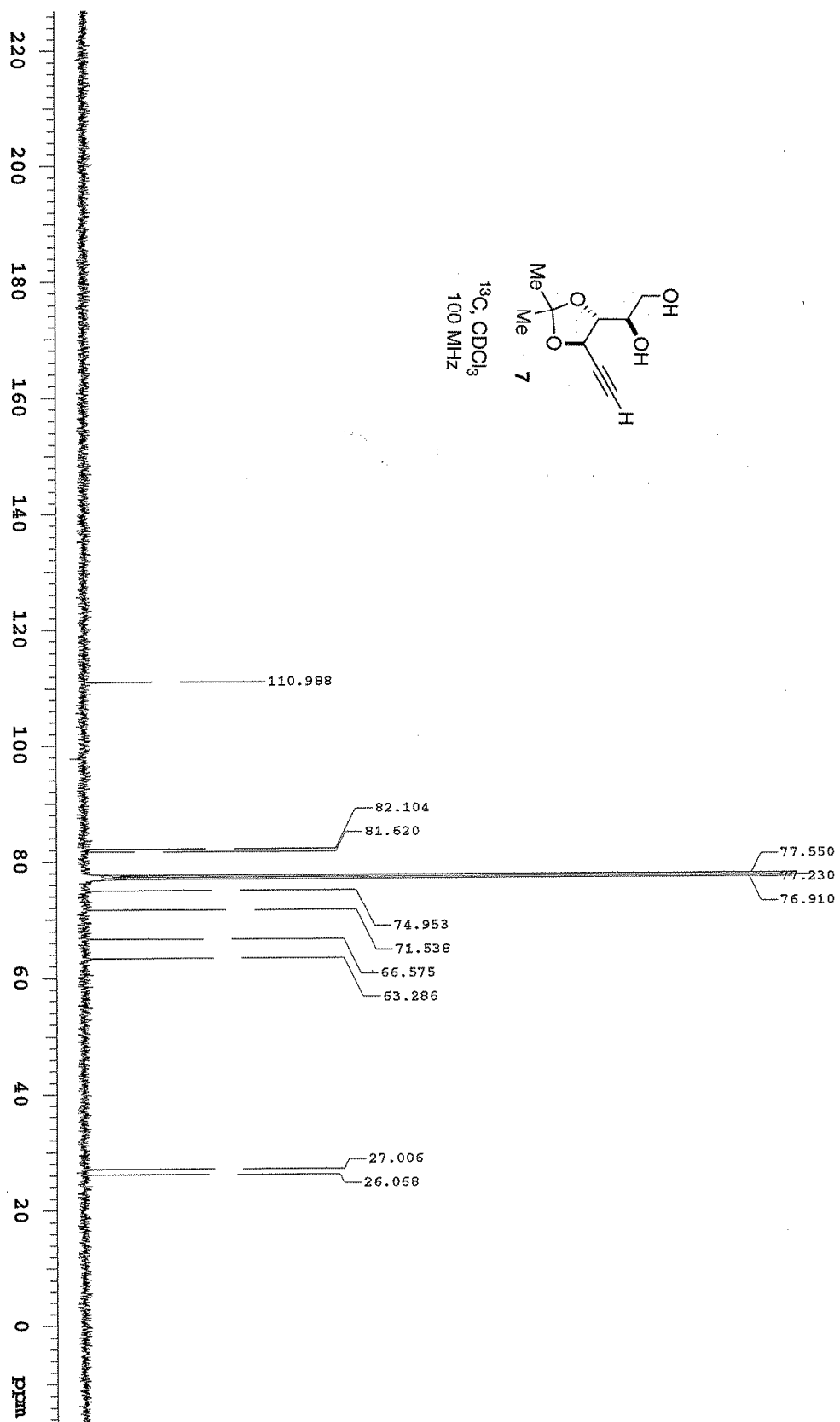
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np 122550 flags
fb 17000 il n
bs 16 in n
dl 2.000 dp y
nt 400 hs nm
ct 400
TRANSMITTER lb 2.00
tn c13 fn not used
sfrq 100.537 DISPLAY
tof 1042.7 sp -1700.2
lpwr 60 wp 24509.4
pw 8.000 rfi 9464.2
DECOUPLER rfp 7763.7
dn h1 rp 82.5
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ai cdc ph 4
166.212
166.212

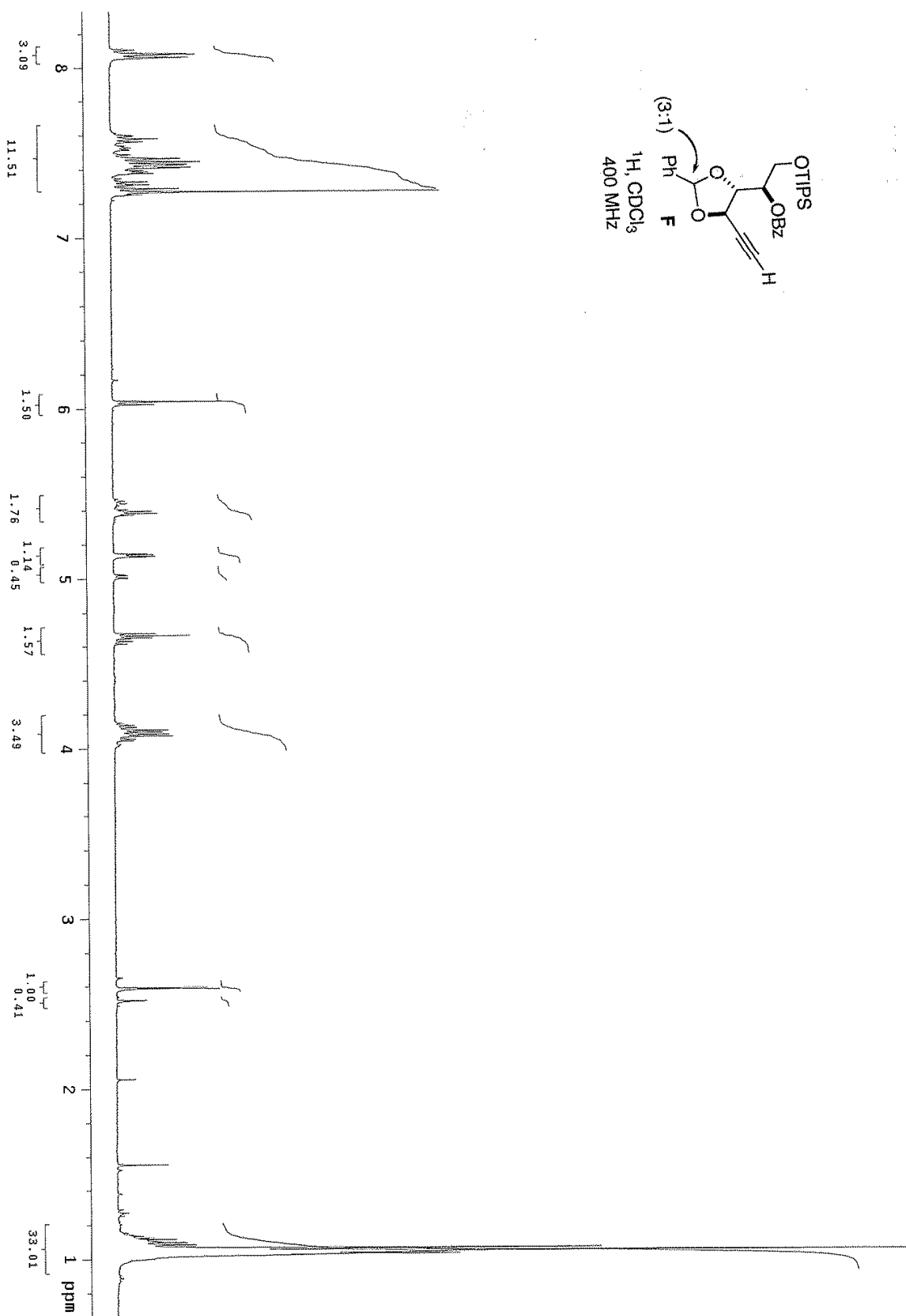
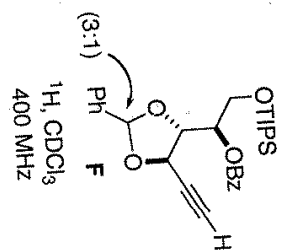


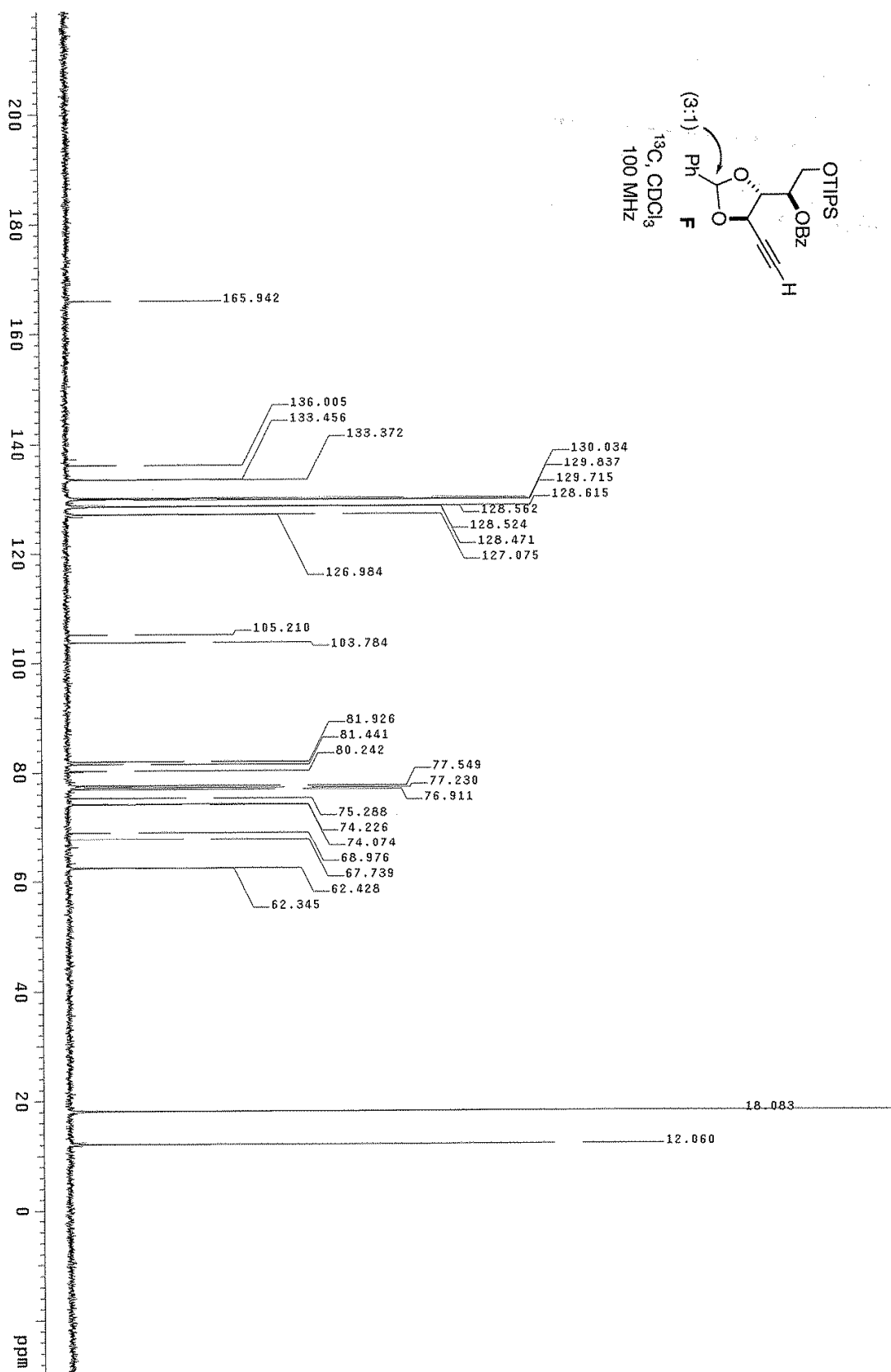


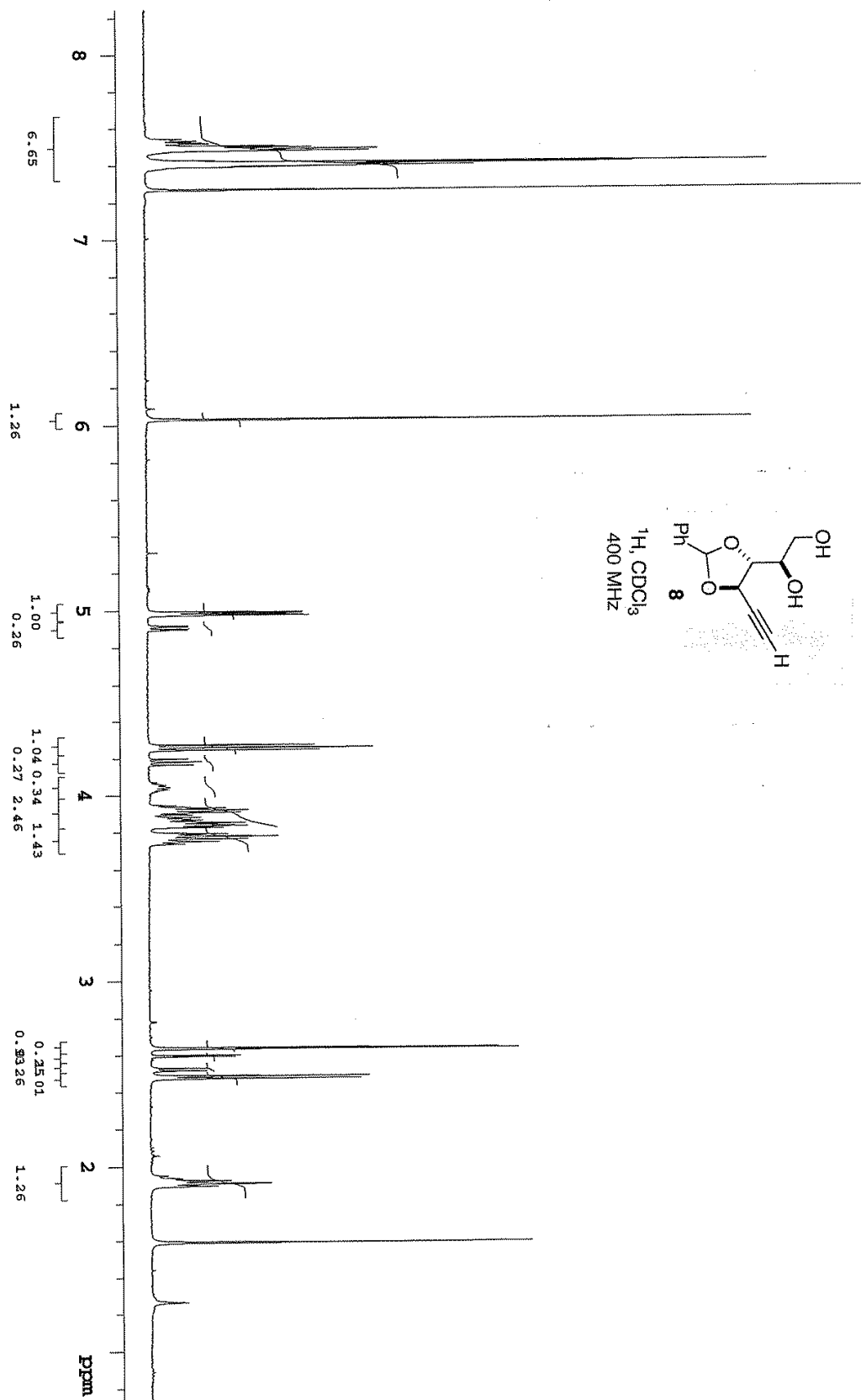






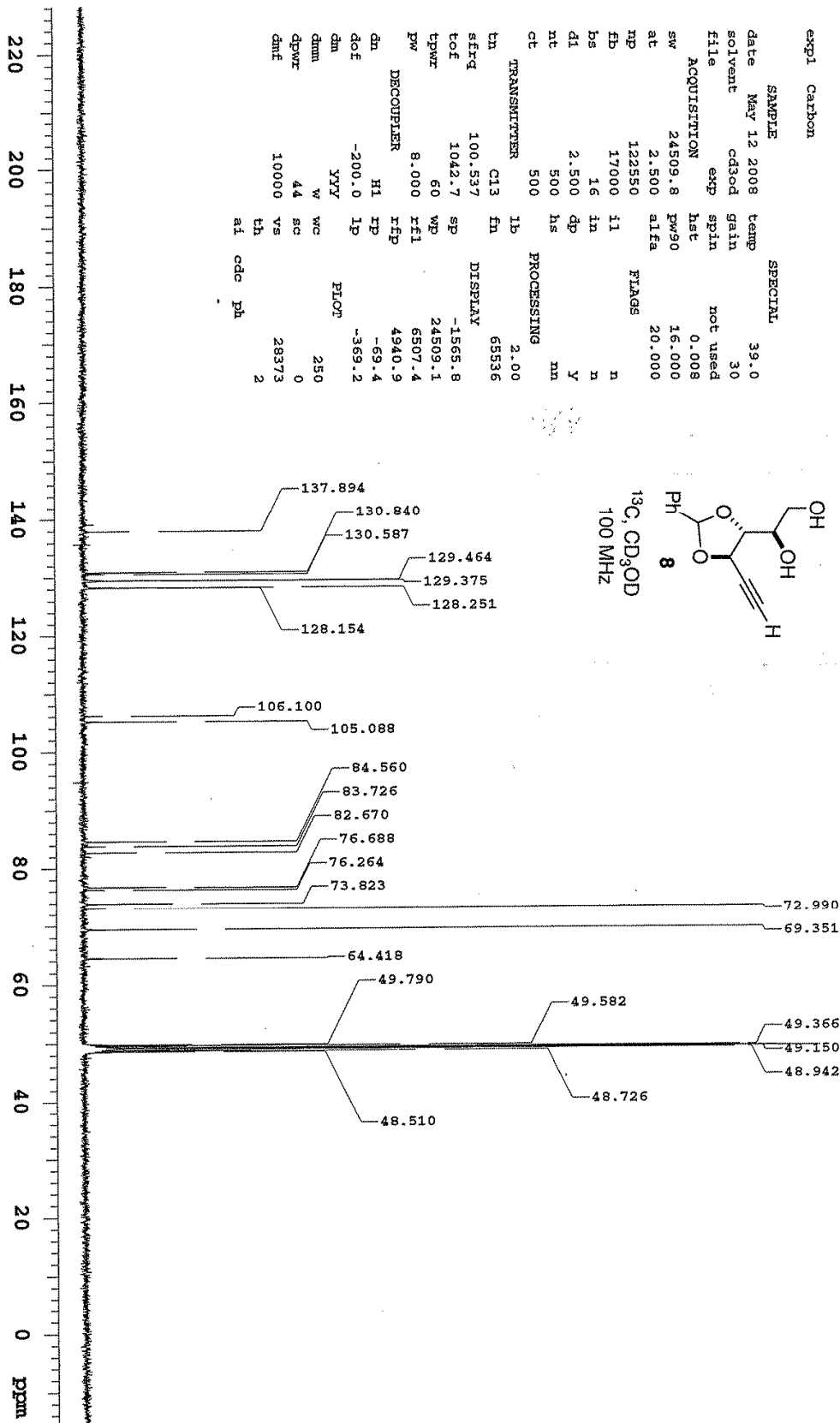
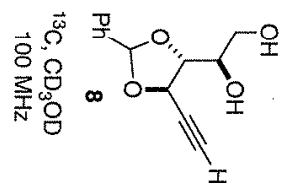


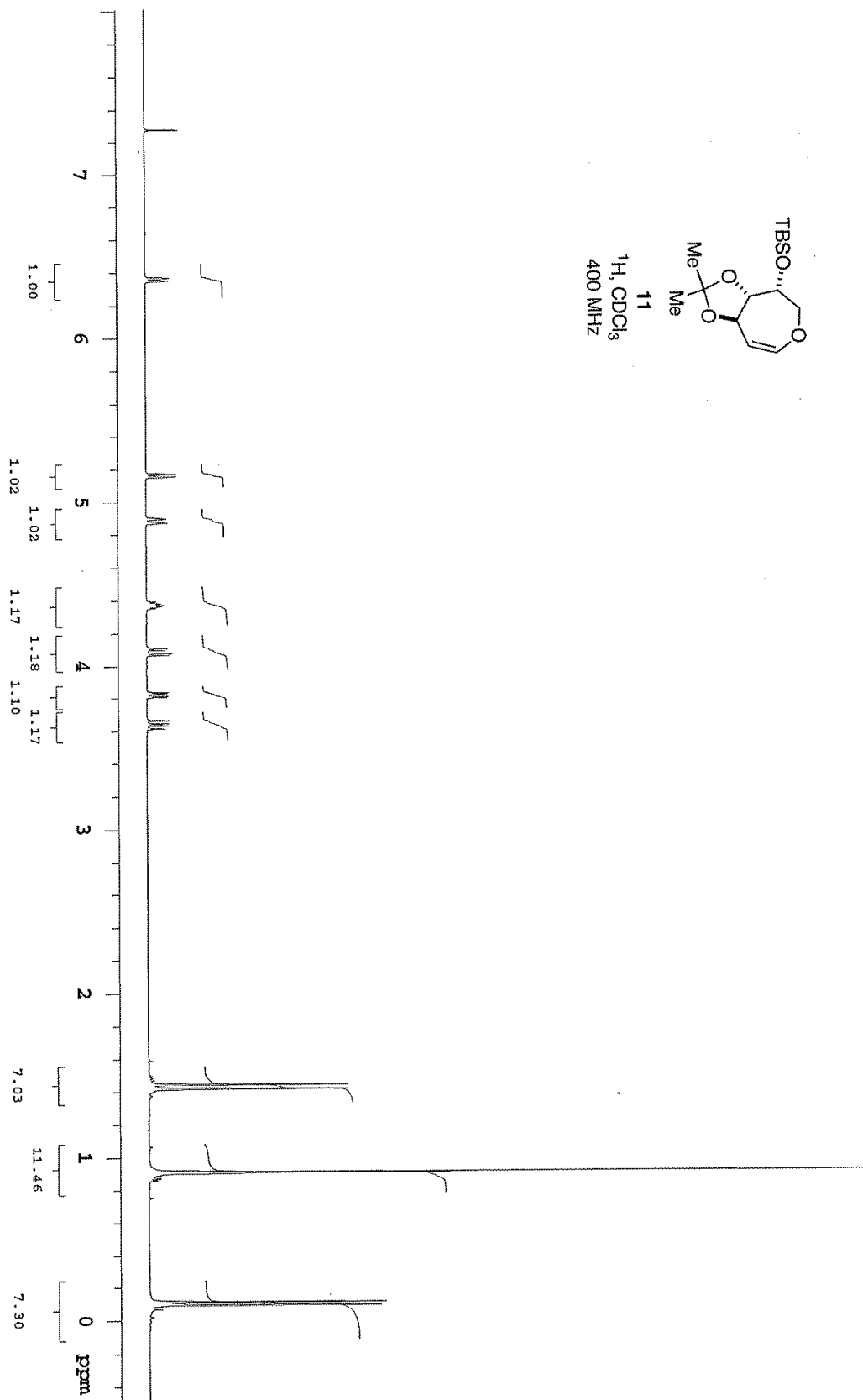
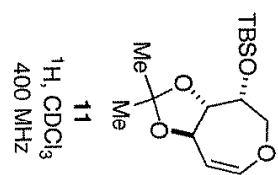


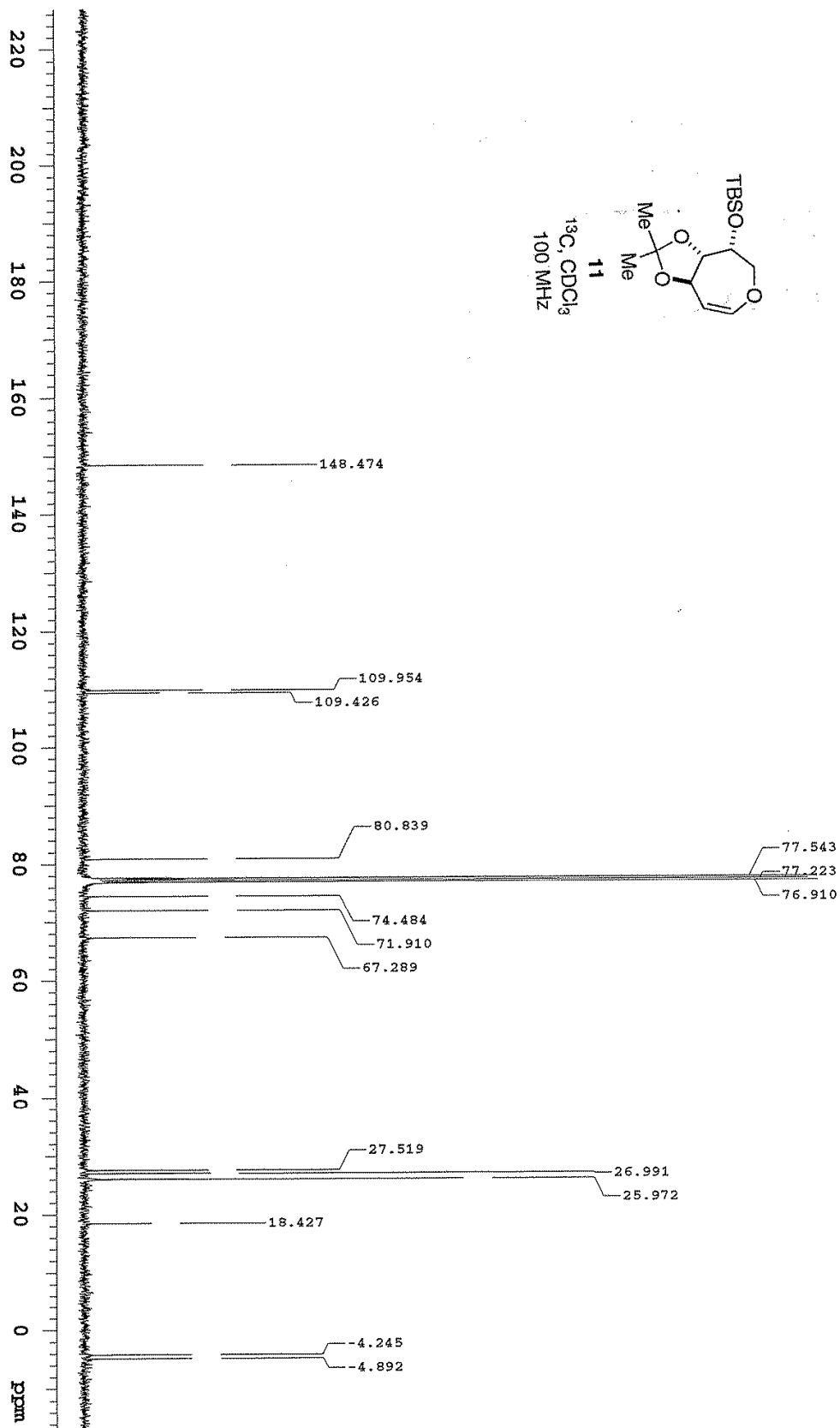


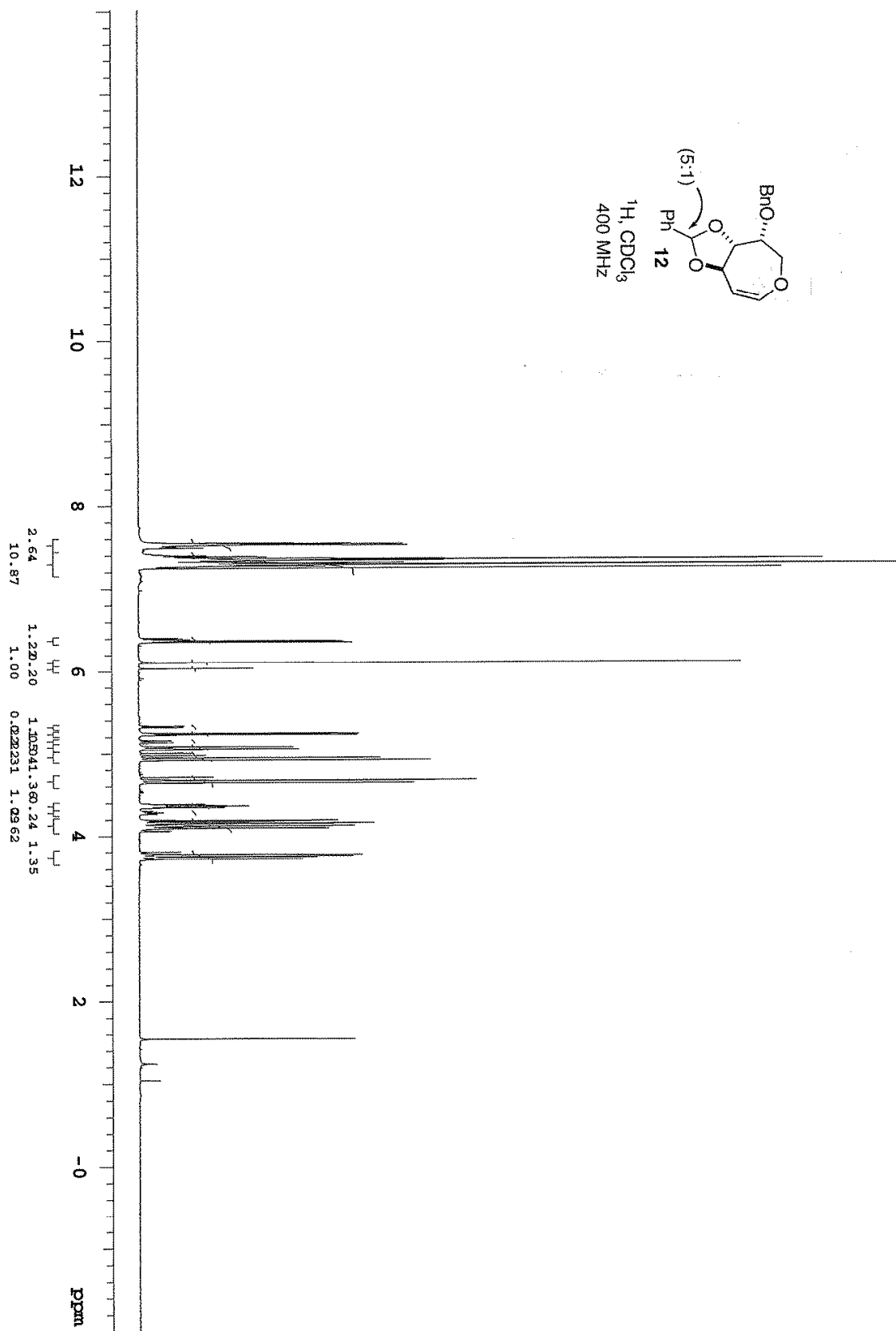
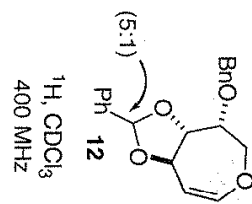
exp1 Carbon

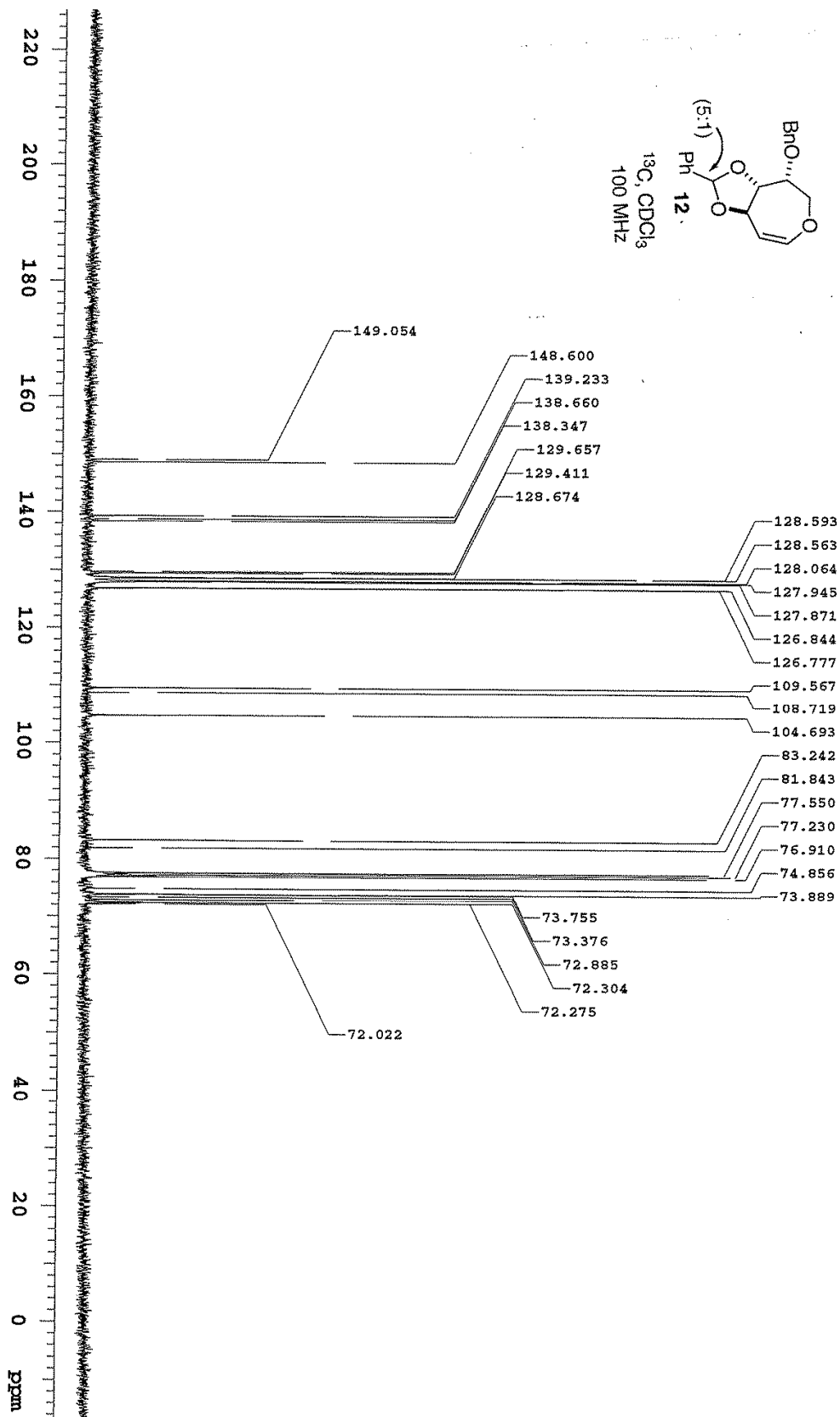
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ACQUISITION
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at 2.500 alfa 20.000
np 122550
fb 17000 il n
bs 16 in y
dl 2.500 dp Y
nt 500 hs nm
ct 500
TRANSMITTER
tn C13 fb 65536
sfreq 100.537
tof 1042.7 sp -1565.8
tpwr 60 wp 24509.1
pw 8.000 rfi 6507.4
DECOUPLER
dn H1 rfp 4940.9
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dm YYY FLOC 250
dmm v wc 0
dpr 44 sc 28373
dmf 10000 vs 2
th al cdc ph

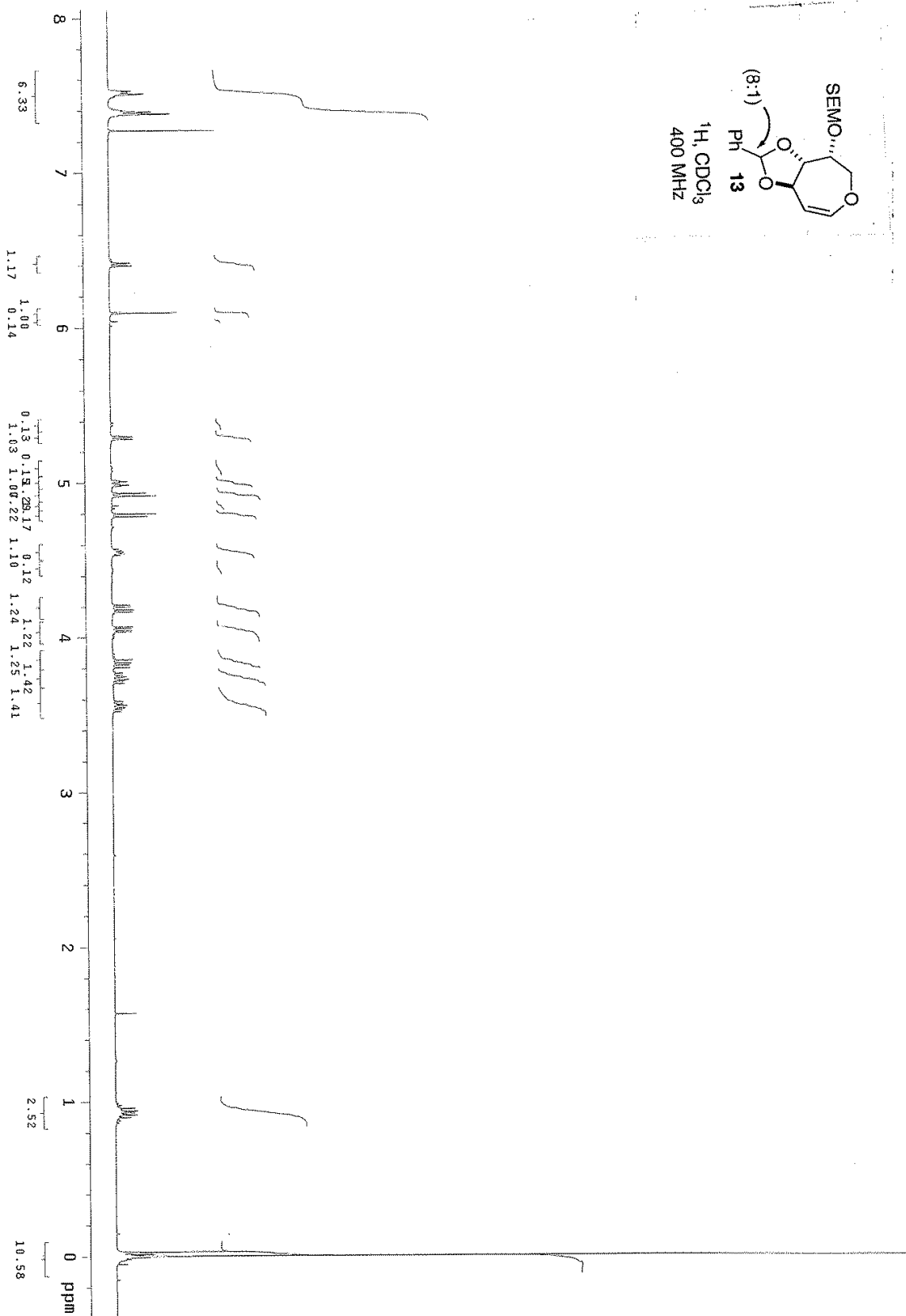
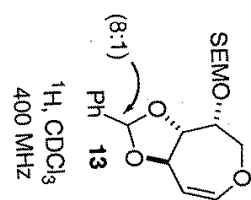








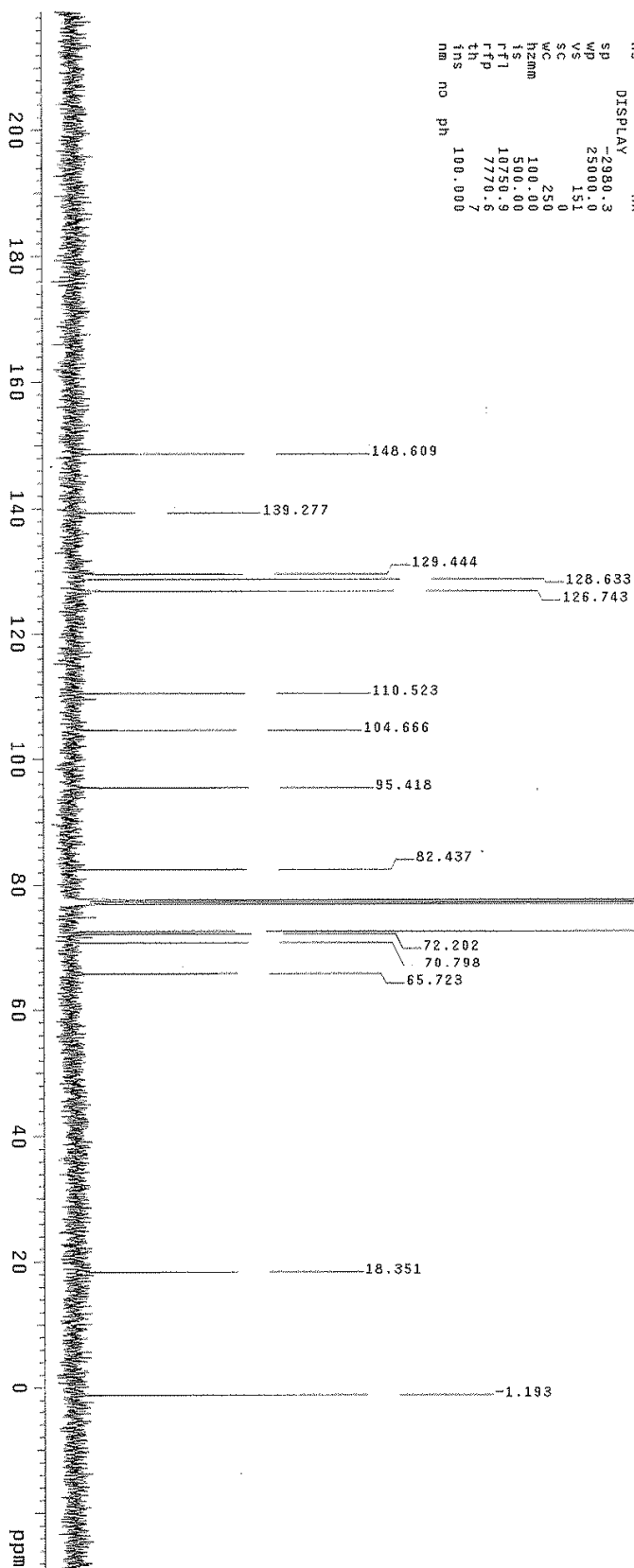
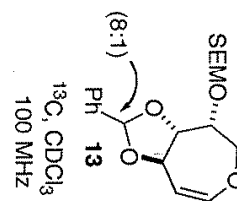


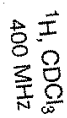


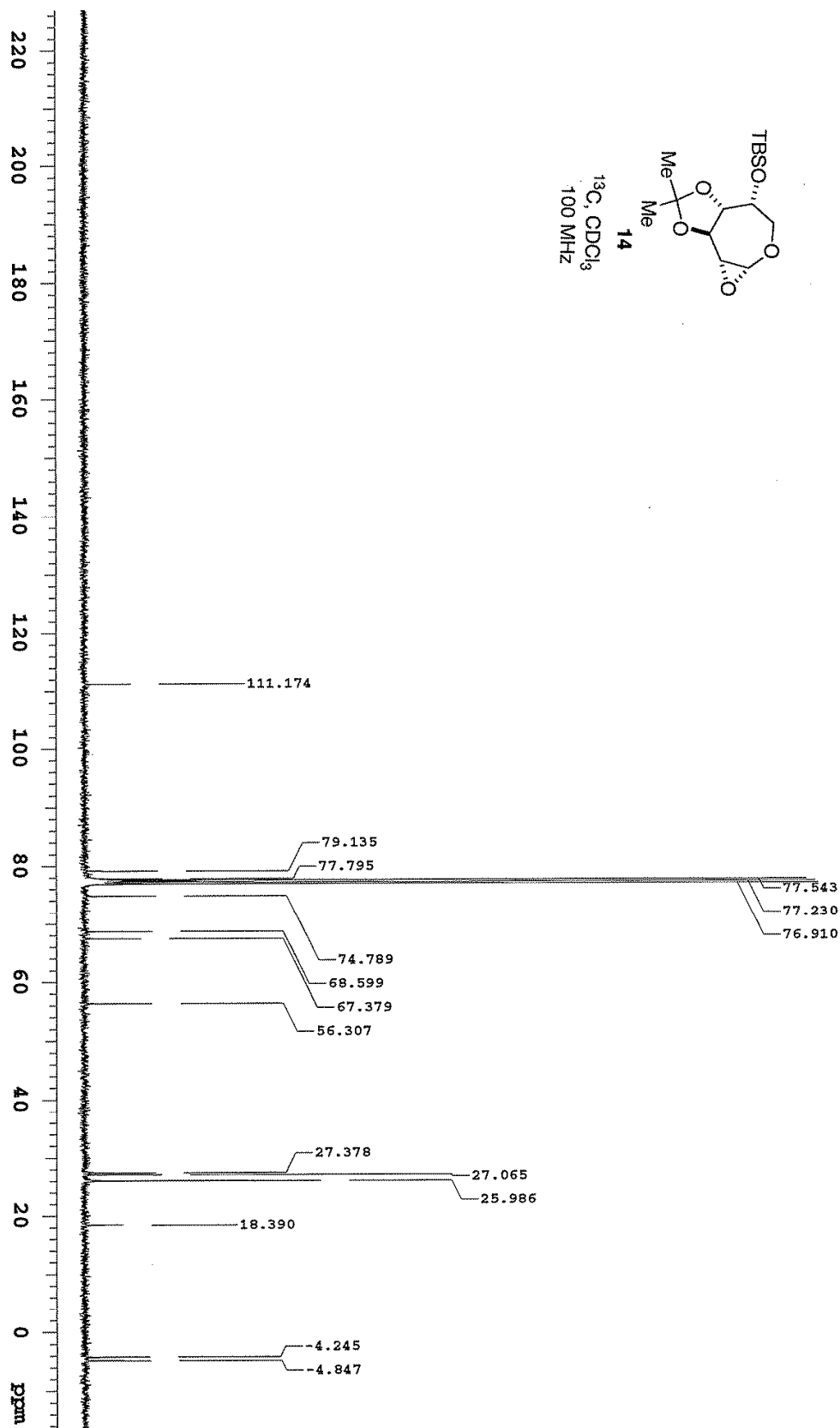
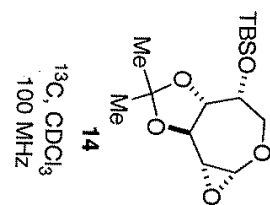
13C OBSERVE

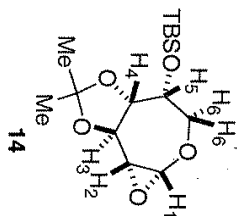
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tn 1.033 dm 7200
at 1.033 dm 7200
np 5.033 dm 7200
sw 25000 dres 1.0
fd 1400 homo n
ls 15
tpwr 58 1b PROCESSING 2.00
pw 8.0 15fid 4
st 3.000 wfttle ft
nt 600 proc not used f
ct 0 math
gain 50 warr fract
j1 n wepp wexp
j2 n wbs testn
j3 n wnt
hs n
DISPLAY
sp -2980.3
wp 25000.0
vs 151
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wc 250
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is 500.00
rf1 10750.9
rfp 7770.6
th 7
ins 100.000
nm no ph

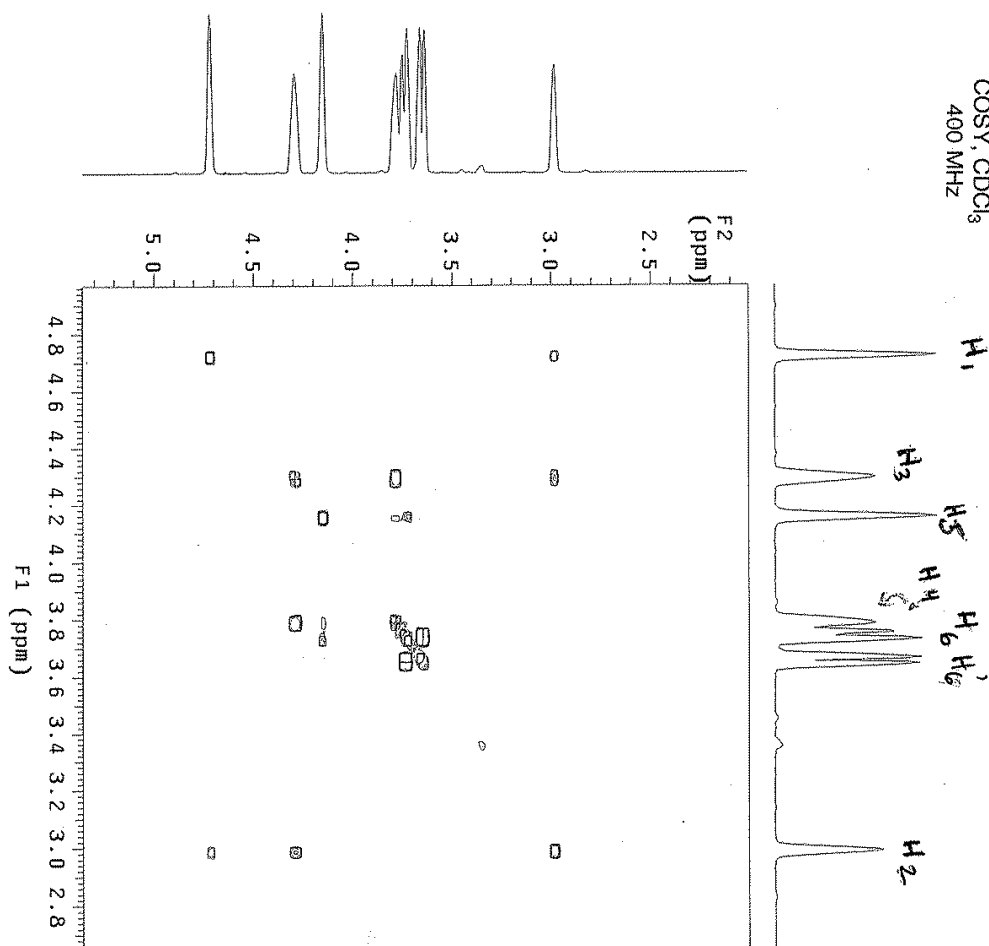


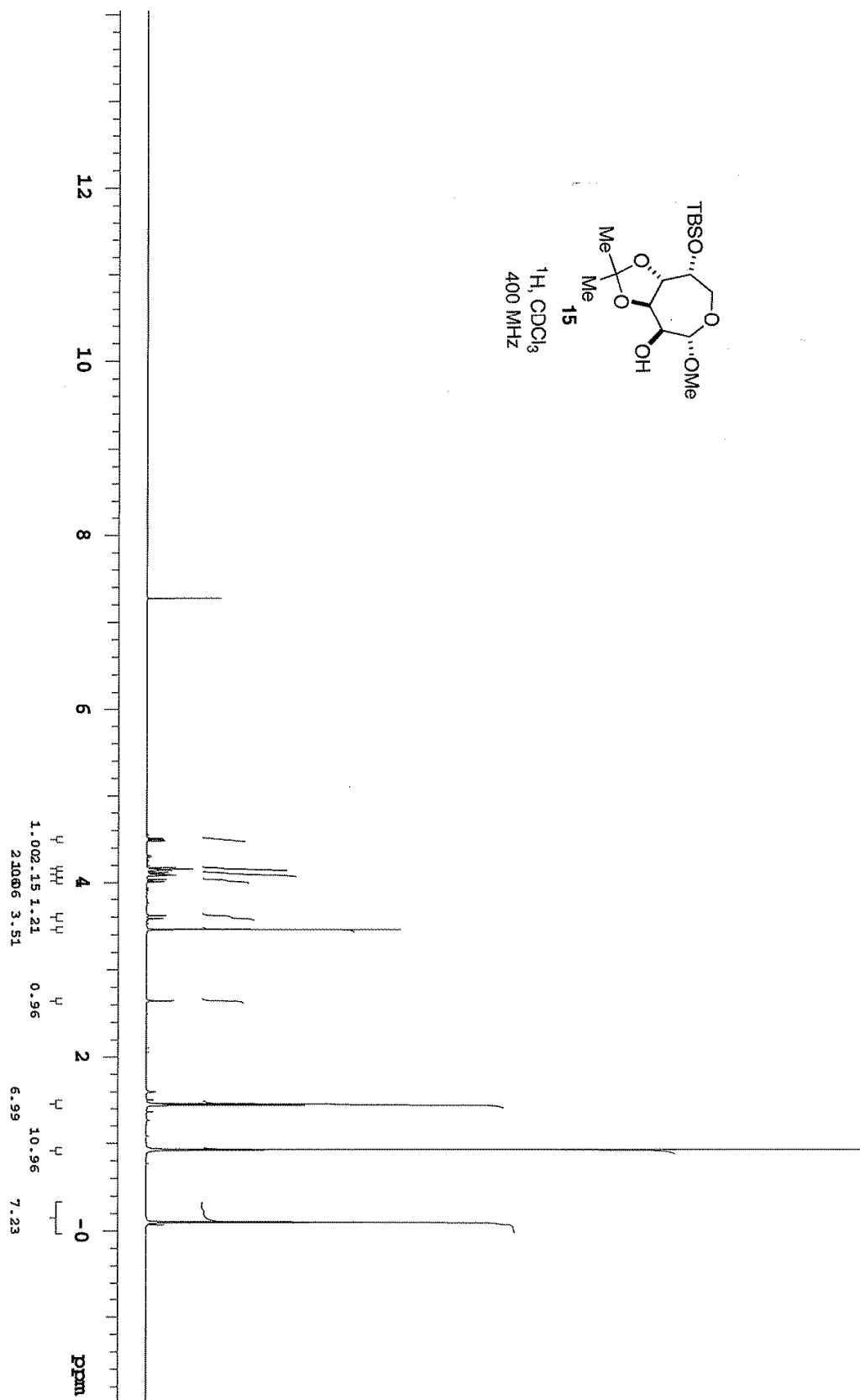
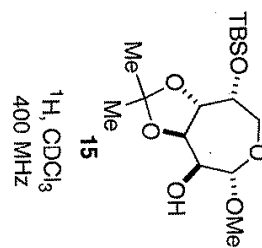


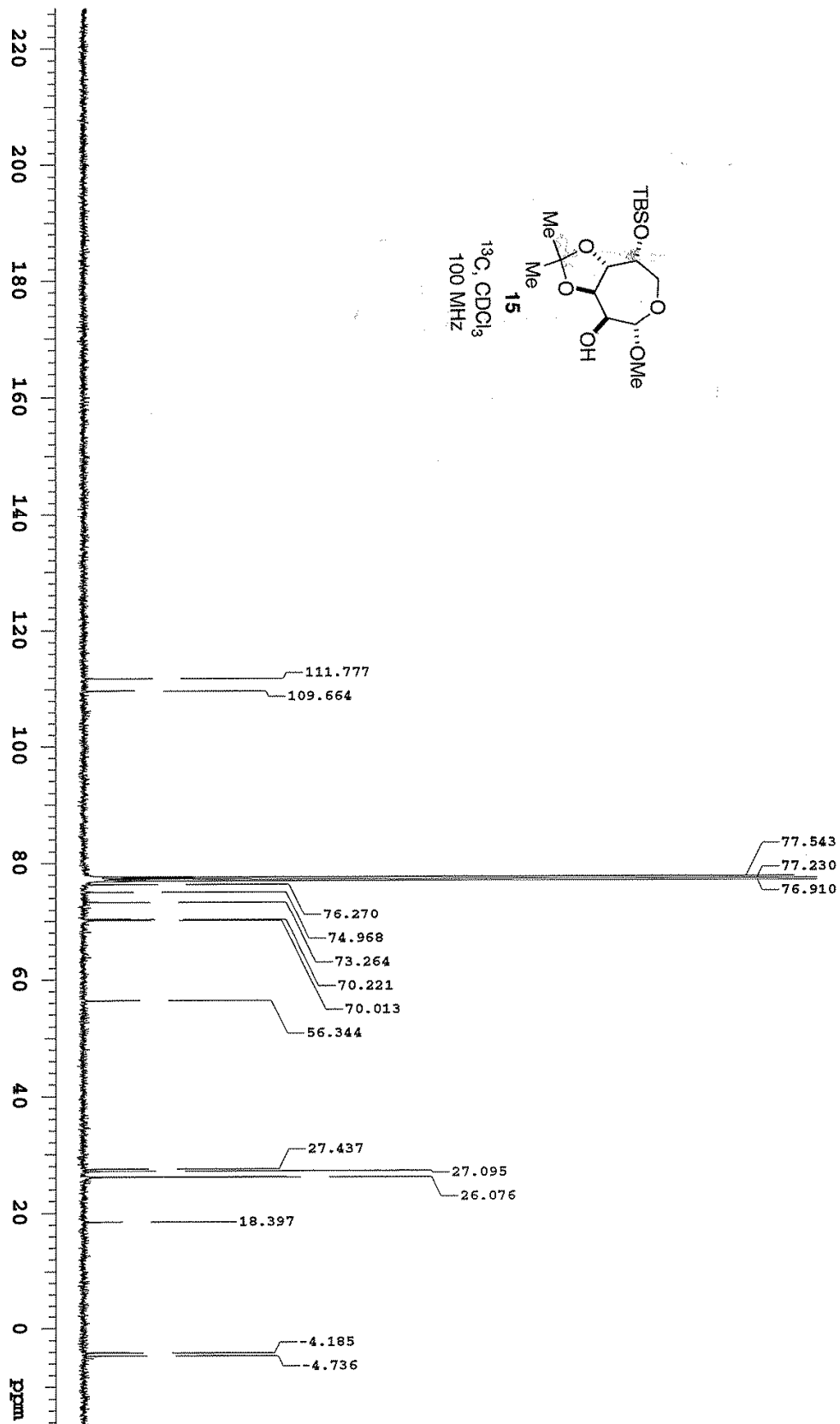


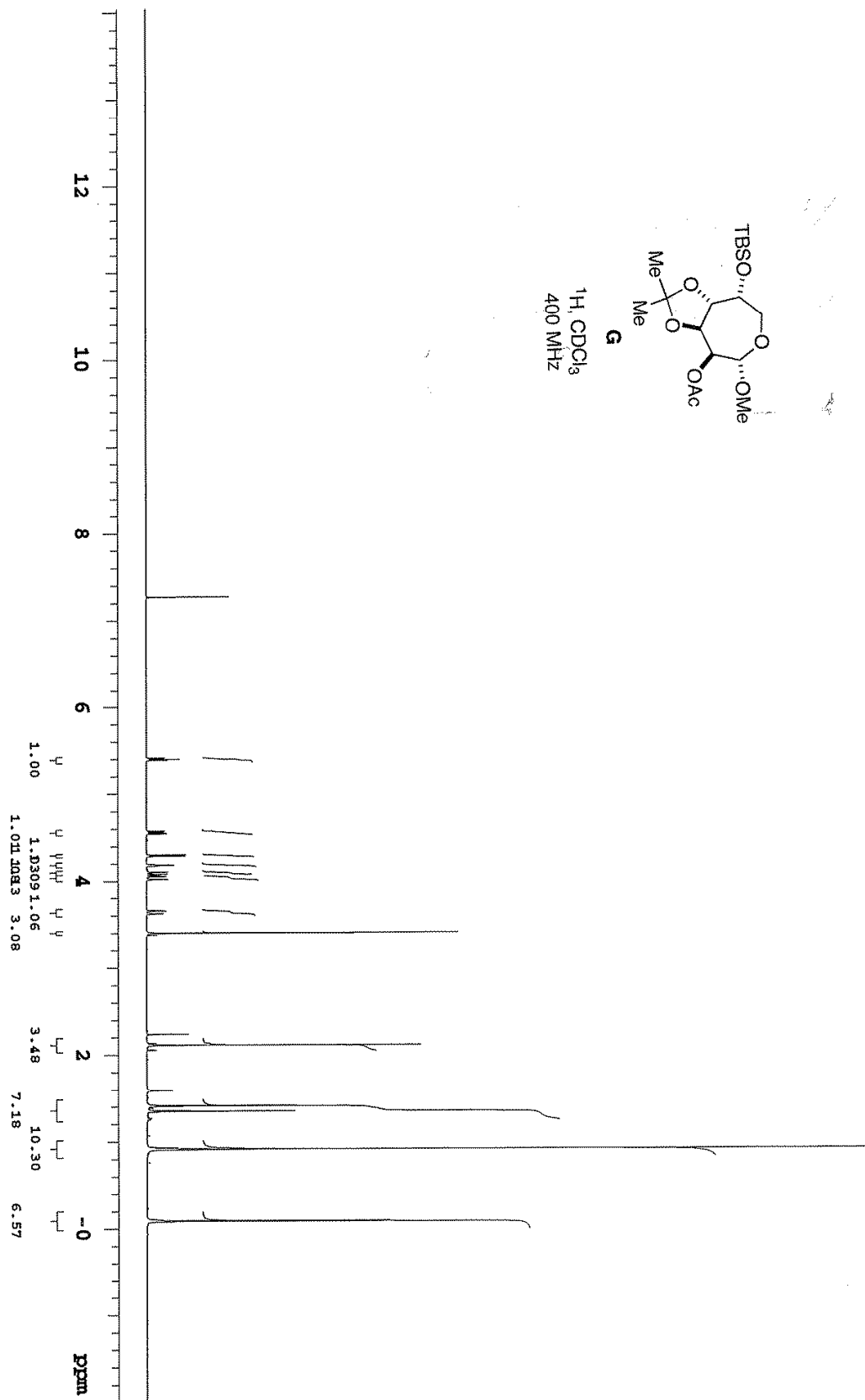
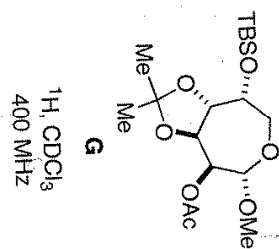


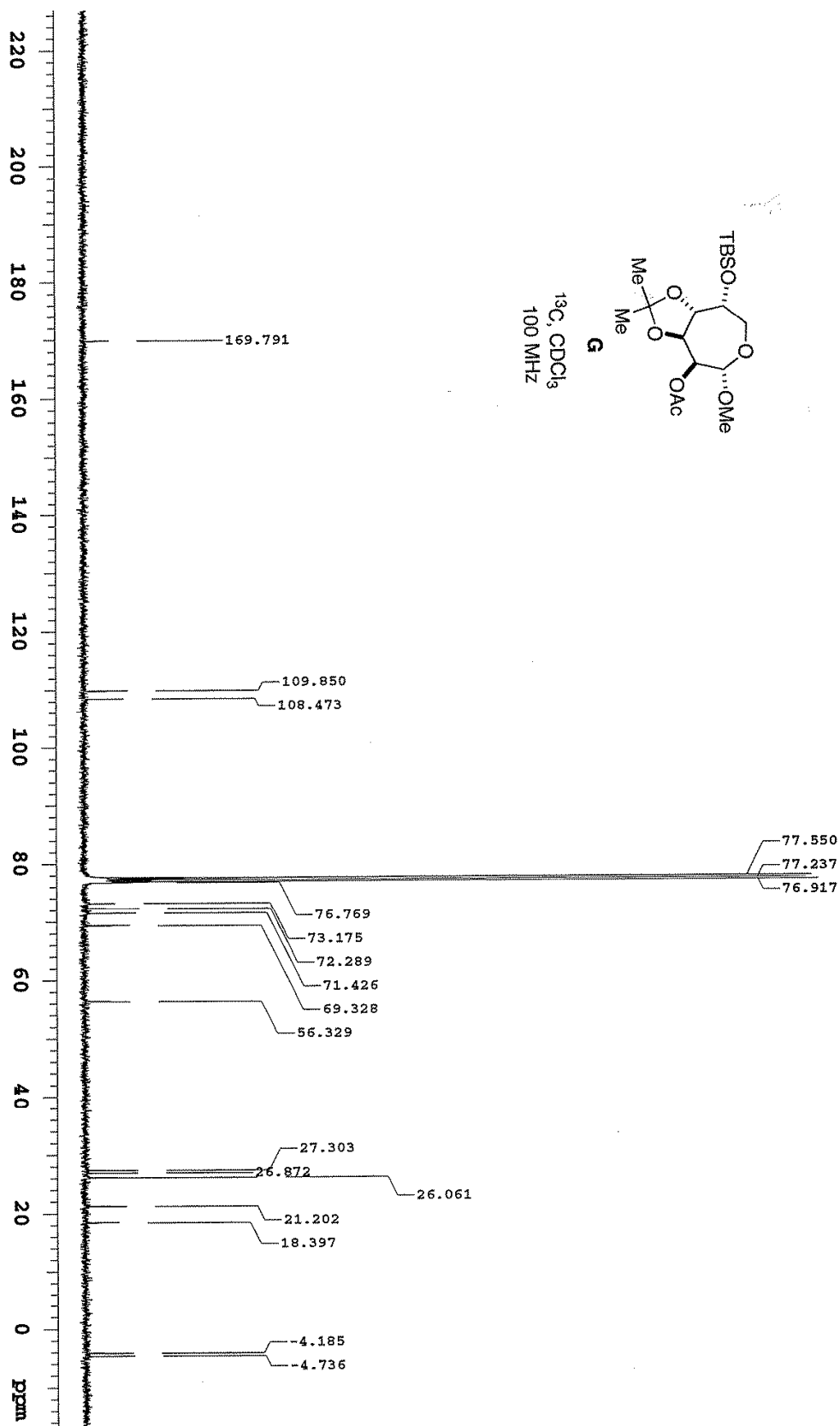
COSY, CDCl₃
400 MHz

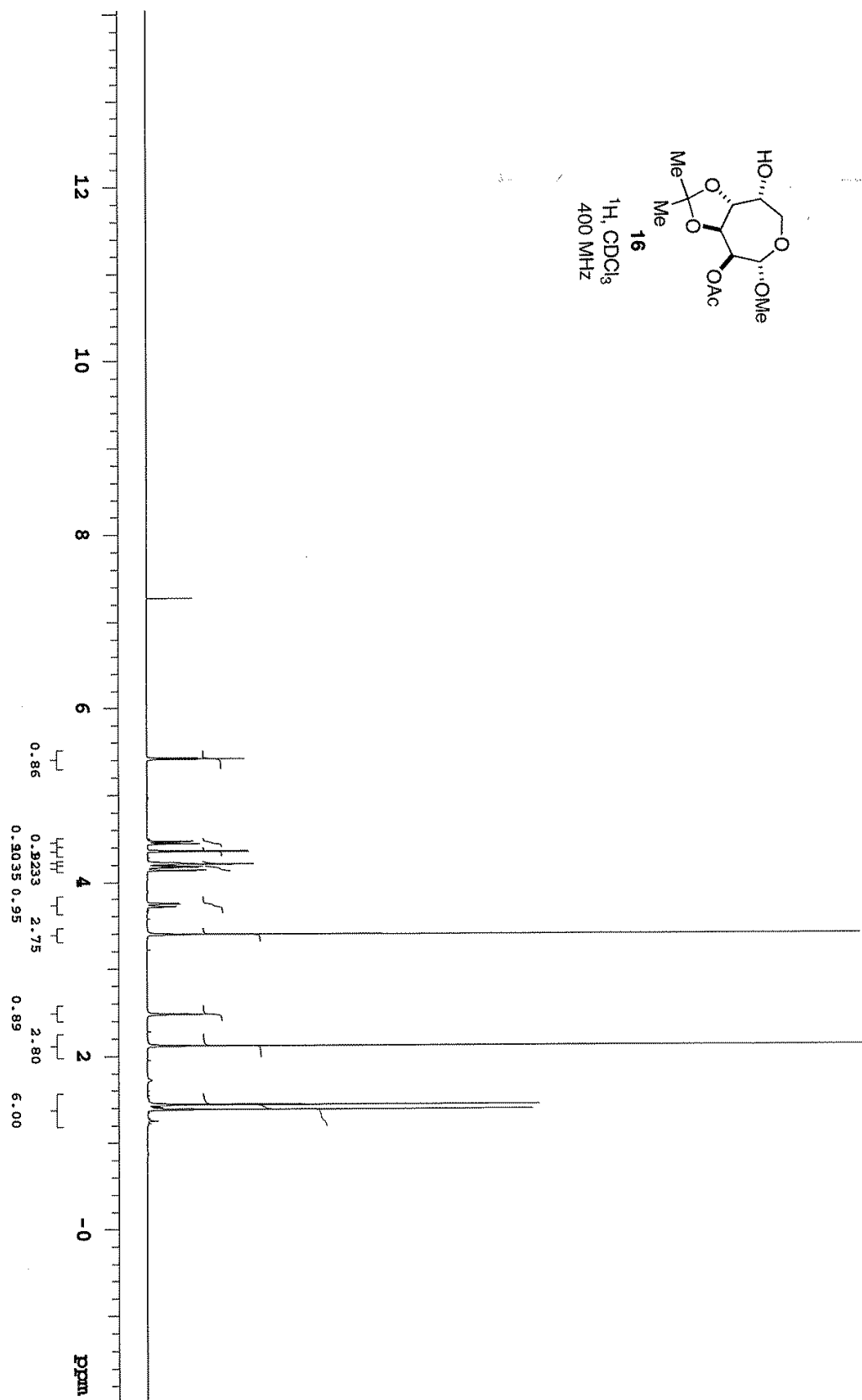
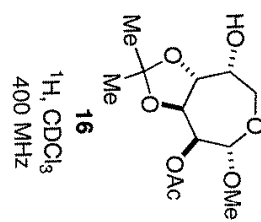


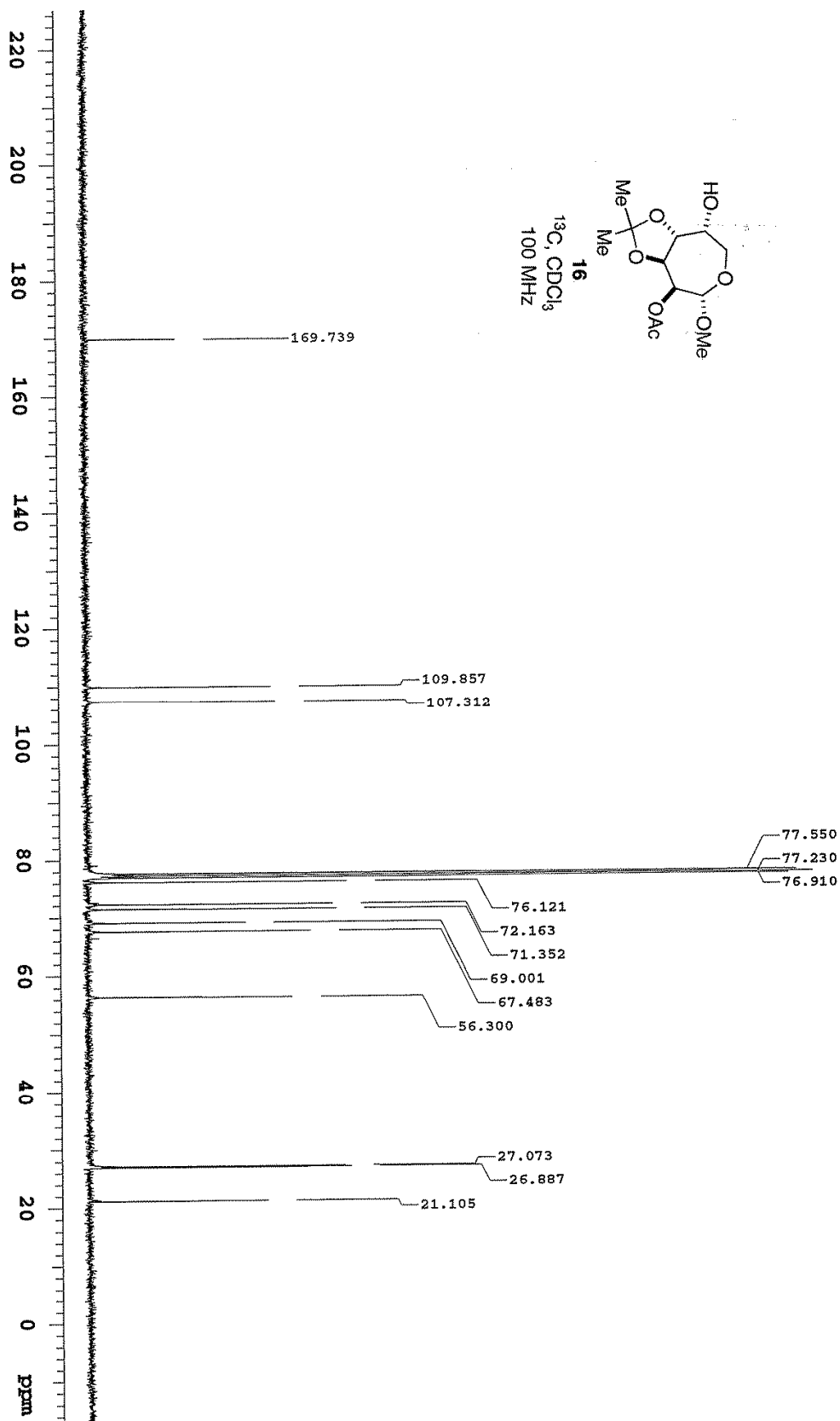


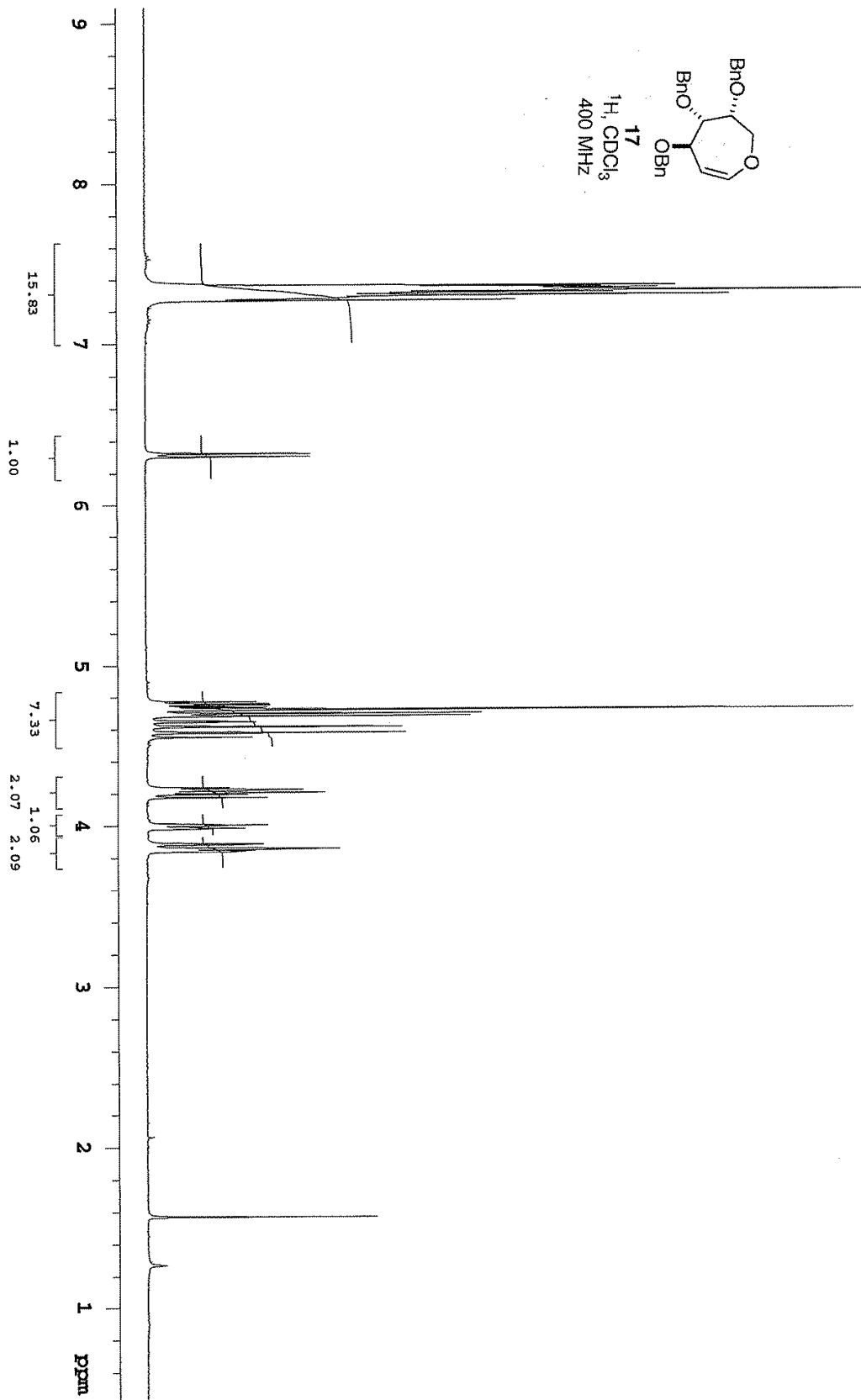
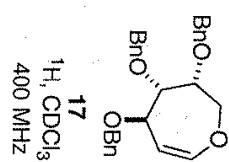


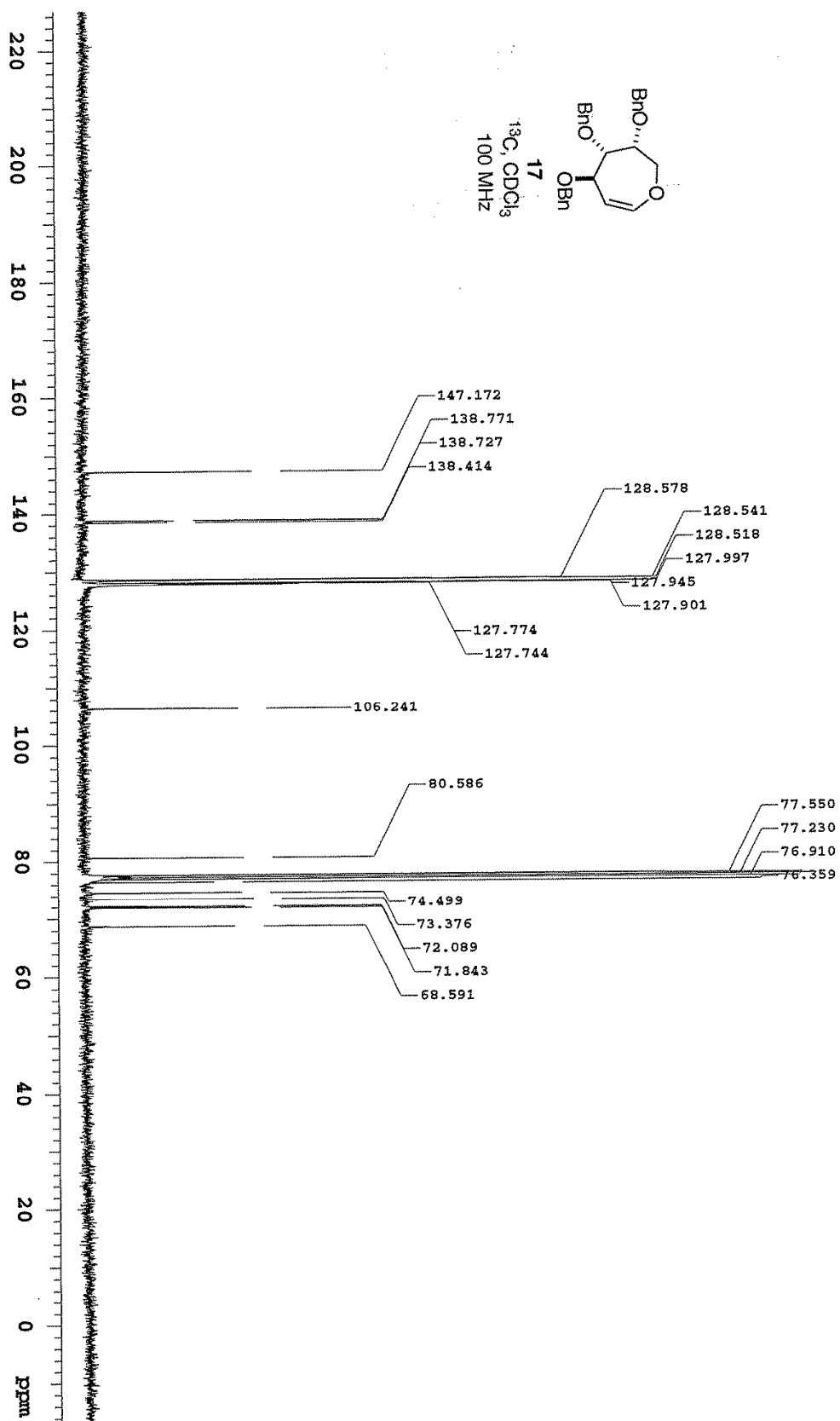


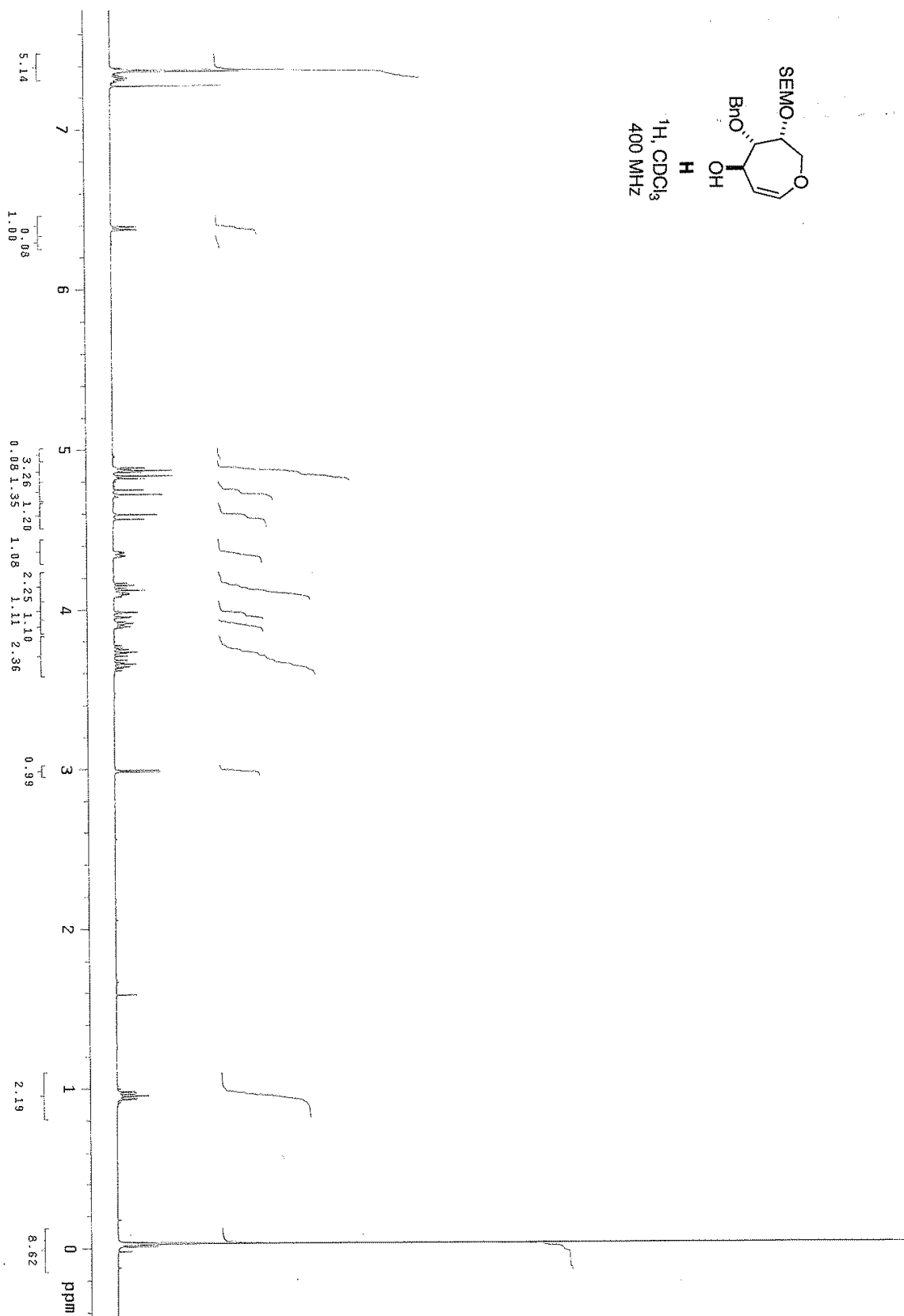
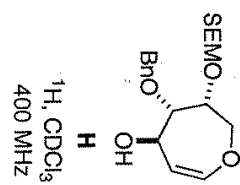








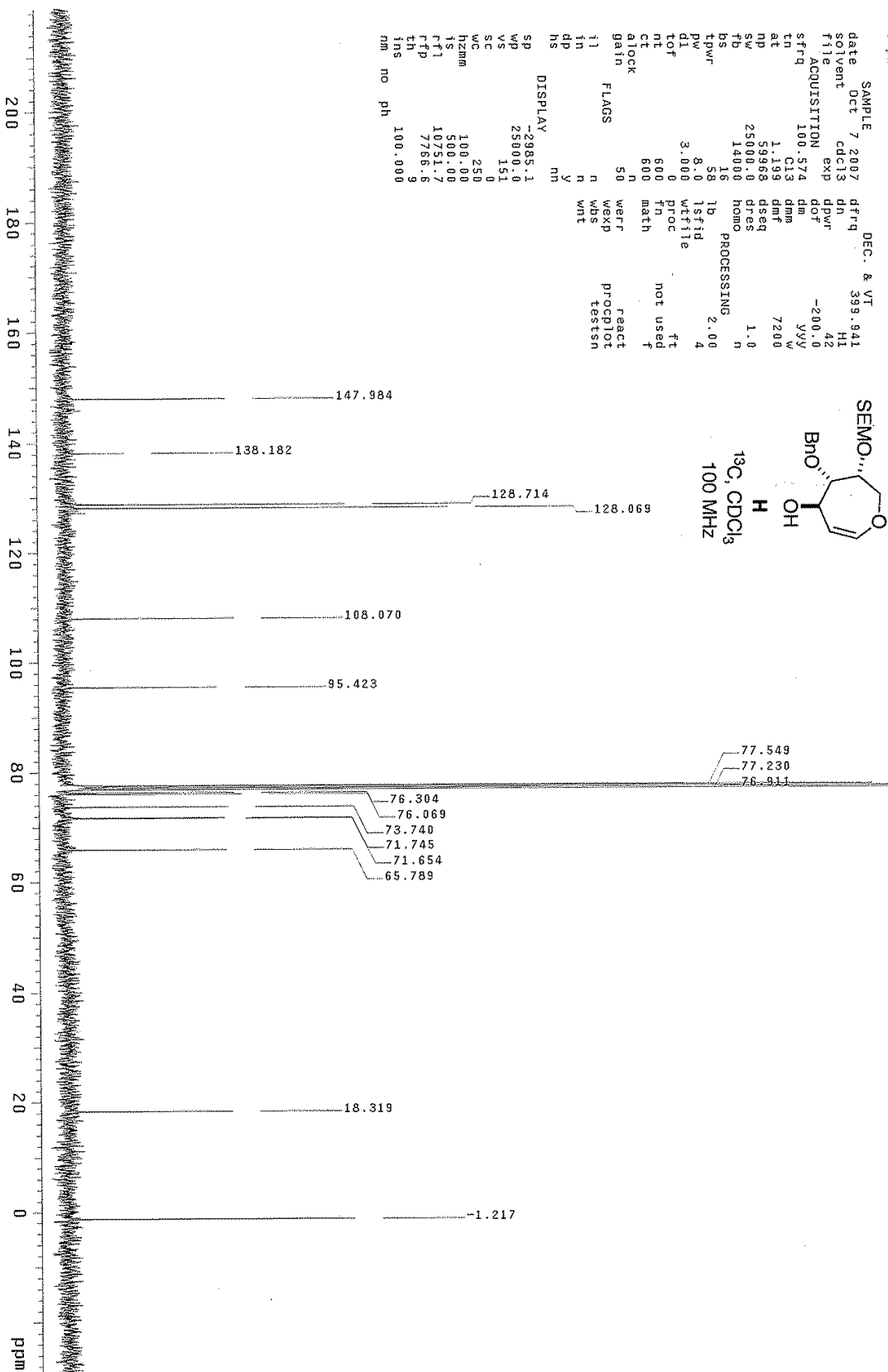
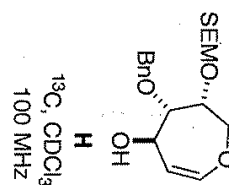


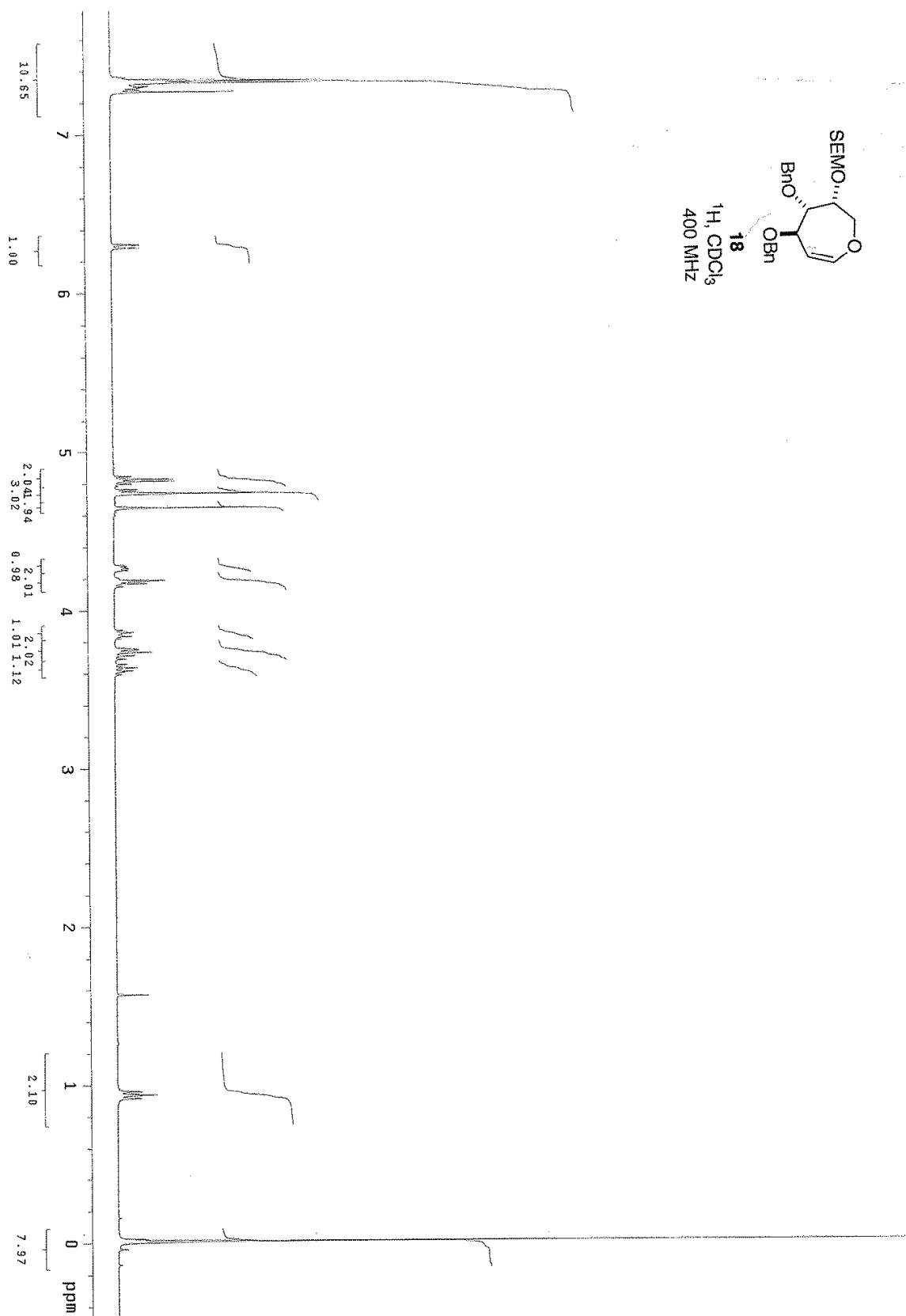
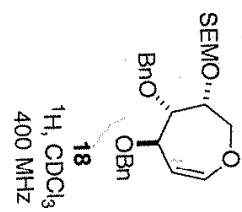


13C OBSERVE

exptl std13c

SAMPLE DEC. & VT
date Oct 7 2007 dfrq 399.941
solvent cdc13 dn H1
file exp 42
ACQUISITION dpr -200.0
sfrq 100.574 dm yyy
tn C13 dnm w
at 1.139 det 7200
np 139368 dseq 1.0
sw 25000.0 dres
fb 14000 homo n
bs 56 PROCESSING 2.00
tpwr 8.00 lb 1s fid 4
pw 3.000 wfile ft
d1 0 proc not used
tof 0 fn
nt 600 math
ct 600
clock n
gain 50 weff react
flags n wexp procplot
ij n wbs testn
in y
dp v
hs DISPLAY nh
sp -2985.1
wp 25000.0
vs 151
sc 0
wc 250
hzm 100.00
is 500.00
rfl 10751.7
rfp 7766.6
th 9
ins 100.000
nm no ph

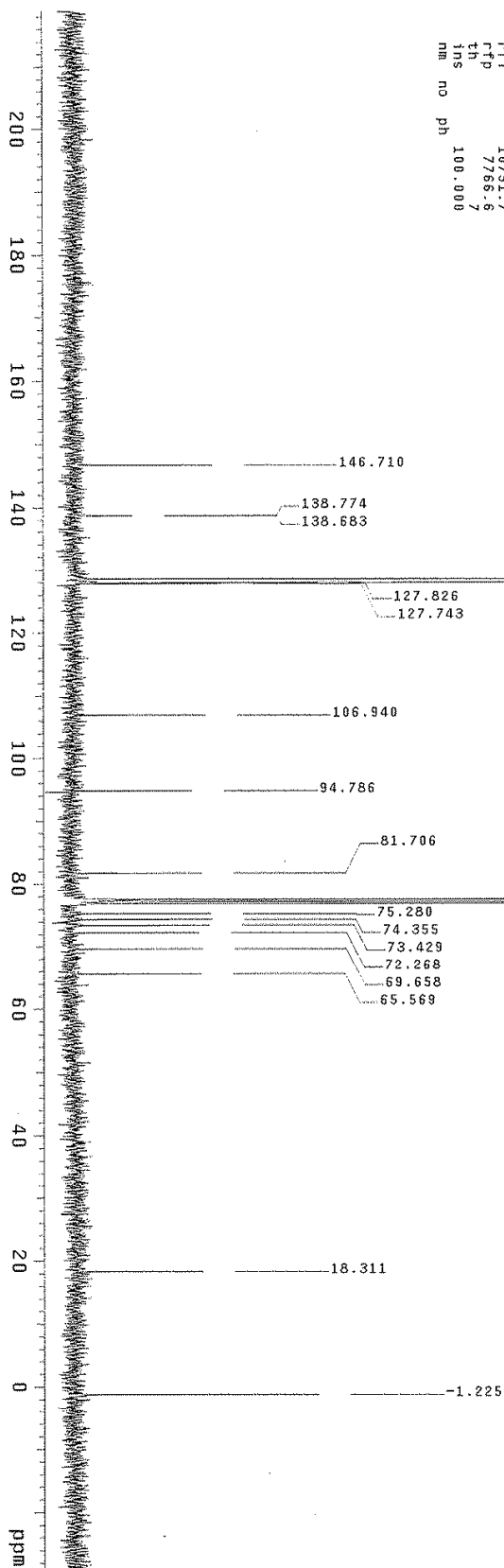
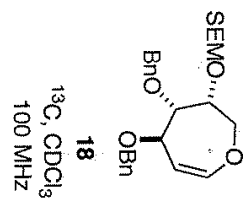


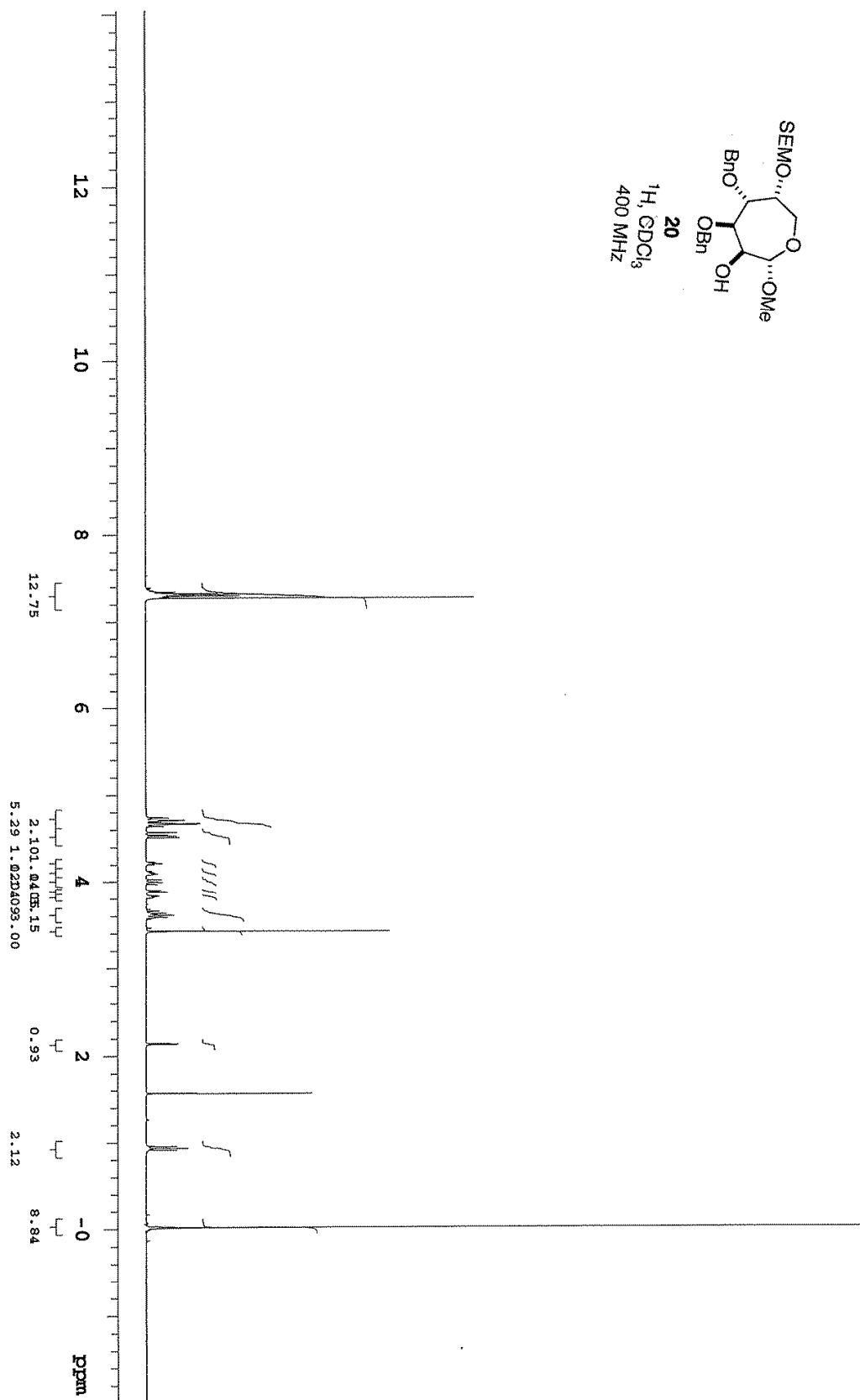
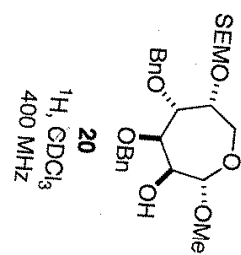


13C OBSERVE

exp1 std13c

SAMPLE DEC. & VT
date Oct 9 2007 dfrq 399.941
solvent cdc13 dn H1
file cdc13 exp 42
ACQUISITION exp dof -200.0
sfrq 100.574 dm yyy
in G13 dnm w
at 1.199 dmf 7200
np 59568 dseq
sw 25000.0 dres 1.0
fb 14000 homo n
bs 16 PROCESSING 2.00
tpwr 38 1b fid 4
pw 8.0 wtitle ft
dl 3.000 0 proc
tof 0 600 math not used
ct 600 math f
clock n
gain 50 wexp react
il n wht proclot
in n wht testm
hs y
DISPLAY nm
sp -2985.1
wp 25000.0
vs 151
sc 0
wc 250
hzm 100.00
is 500.00
rf1 10751.7
rfp 7766.6
th 7
ins 100.000
nm no ph





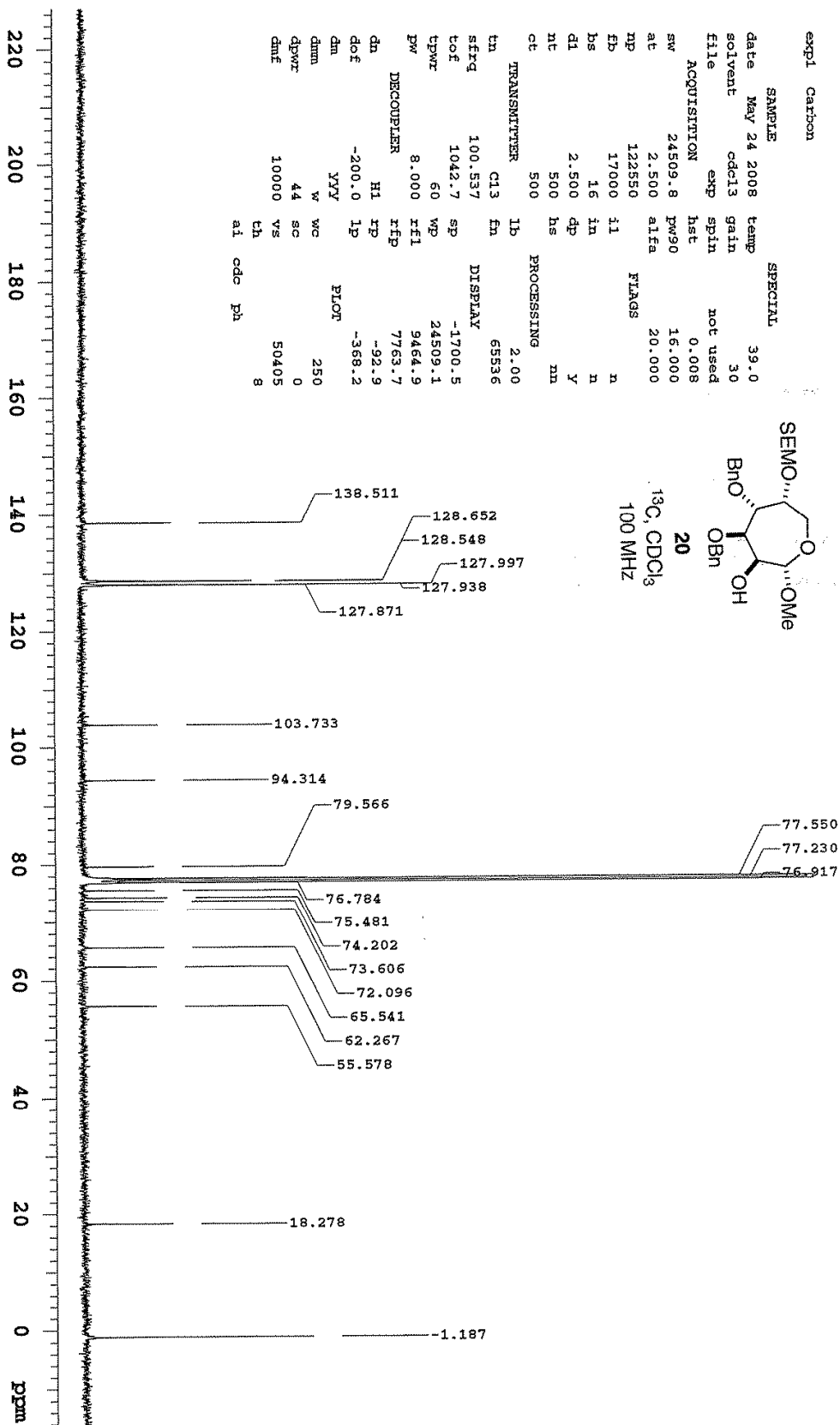
expt Carbon

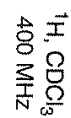
SAMPLE
date May 24 2008 temp 39.0
solvent cdc13 gain 30
file exp spin not used
ACQUISITION
sw 24509.8 hst 0.008
at 2.500 pwr 16.000
np 122550 alfa 20.000
fb 17000 fl
bs 16 in n
dl 2.500 dp y
nt 500 hs mn
ct 500
TRANSMITTER
tn C13 lb 2.00
sfreq 100.537 fn 65536
tof 1042.7 sp -1700.5
tpwr 60 wp 24509.1
pw 8.000 xfl 9464.9
DECOUPLER
dm H1 xfp 7763.7
dof -200.0 lp -92.9
dm YYY FLOT
dum w wc 250
dpr 44 sc 0
dmf 10000 vs 50405
al cdc ph 8

SEMO
BnO
OBn
OH
OMe

20

¹³C, CDCl₃
100 MHz

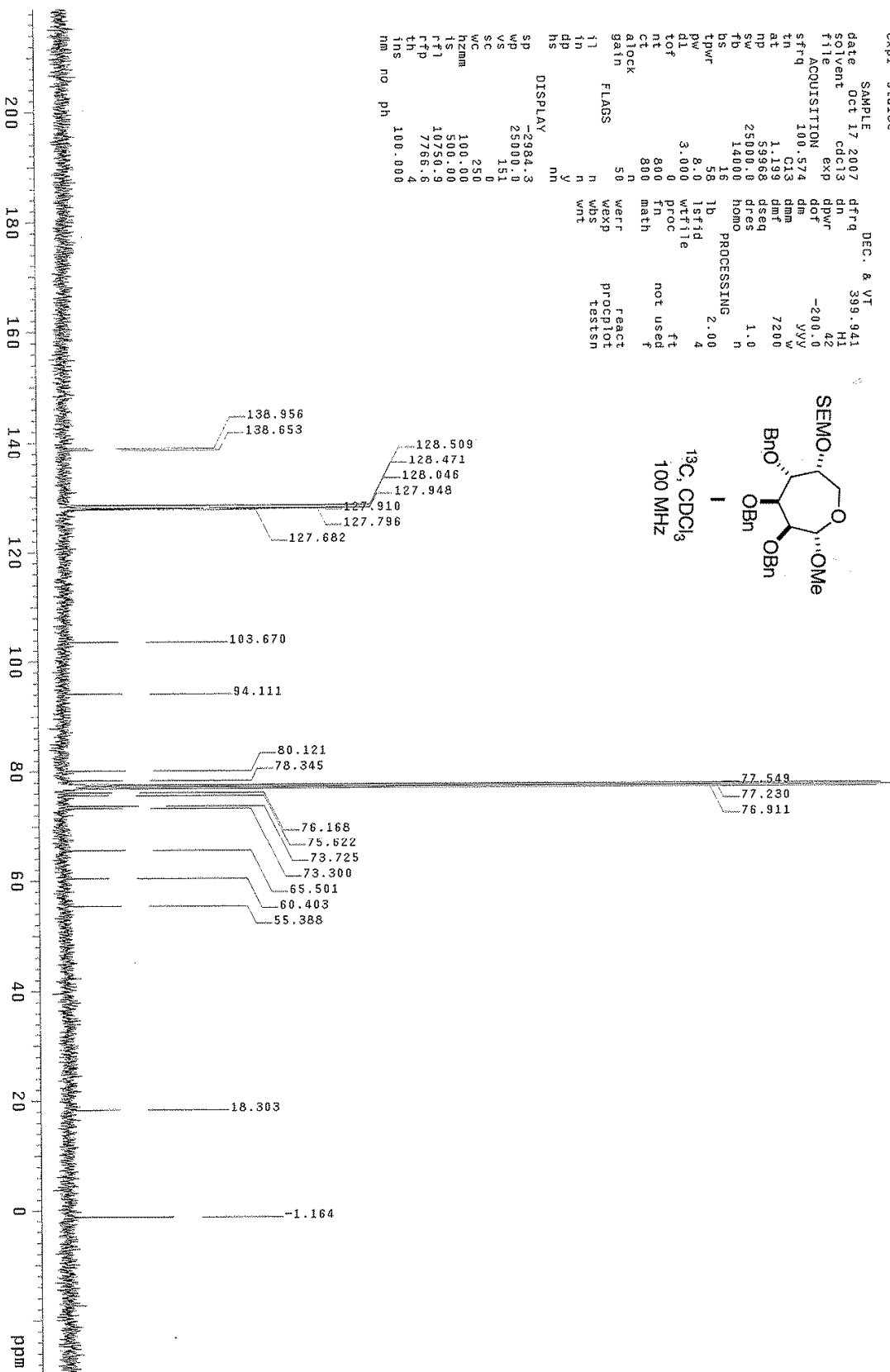
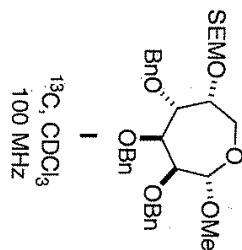


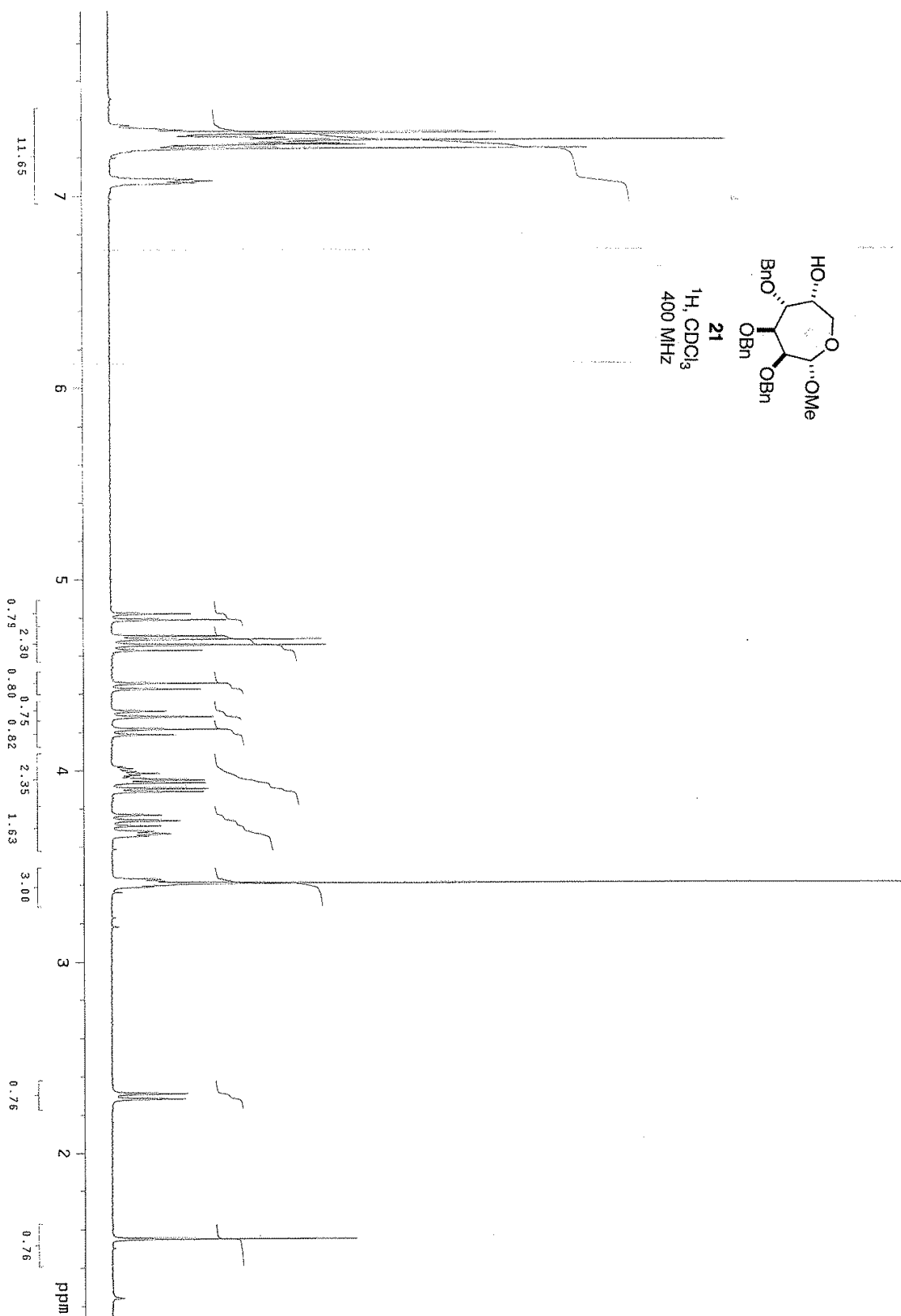


13C OBSERVE

expi sid13c

SAMPLE DEC. & VT
 date Oct 17 2007 dfrq 399.941
 solvent cdc13 dn H1
 file exp 42
 ACQUISITION dpr -200.0
 sfreq 100.574 dm yyy
 tn 1.143 gnm w
 at 5.358 dseg 7200
 np 25900.0 des 1.0
 sw 14000 homo n
 ds 58 1b PROCESSING 2.00
 tpwr 8.0 1s fid 4
 pl 3.000 wfile ft
 tof 0 proc not used
 nt 800 fn math f
 ct 800 n
 clock 50 warr react
 gain 50 wexp procplot
 j1 n wbs testn
 jn y
 dp n
 hs y
 DISPLAY
 sp -2984.3
 wp 25000.0
 vs 151
 sc 0
 wc 250
 hzmm 100.00
 is 500.00
 rfi 10750.9
 rfp 7765.6
 th 4
 ins 100.000
 nm no ph

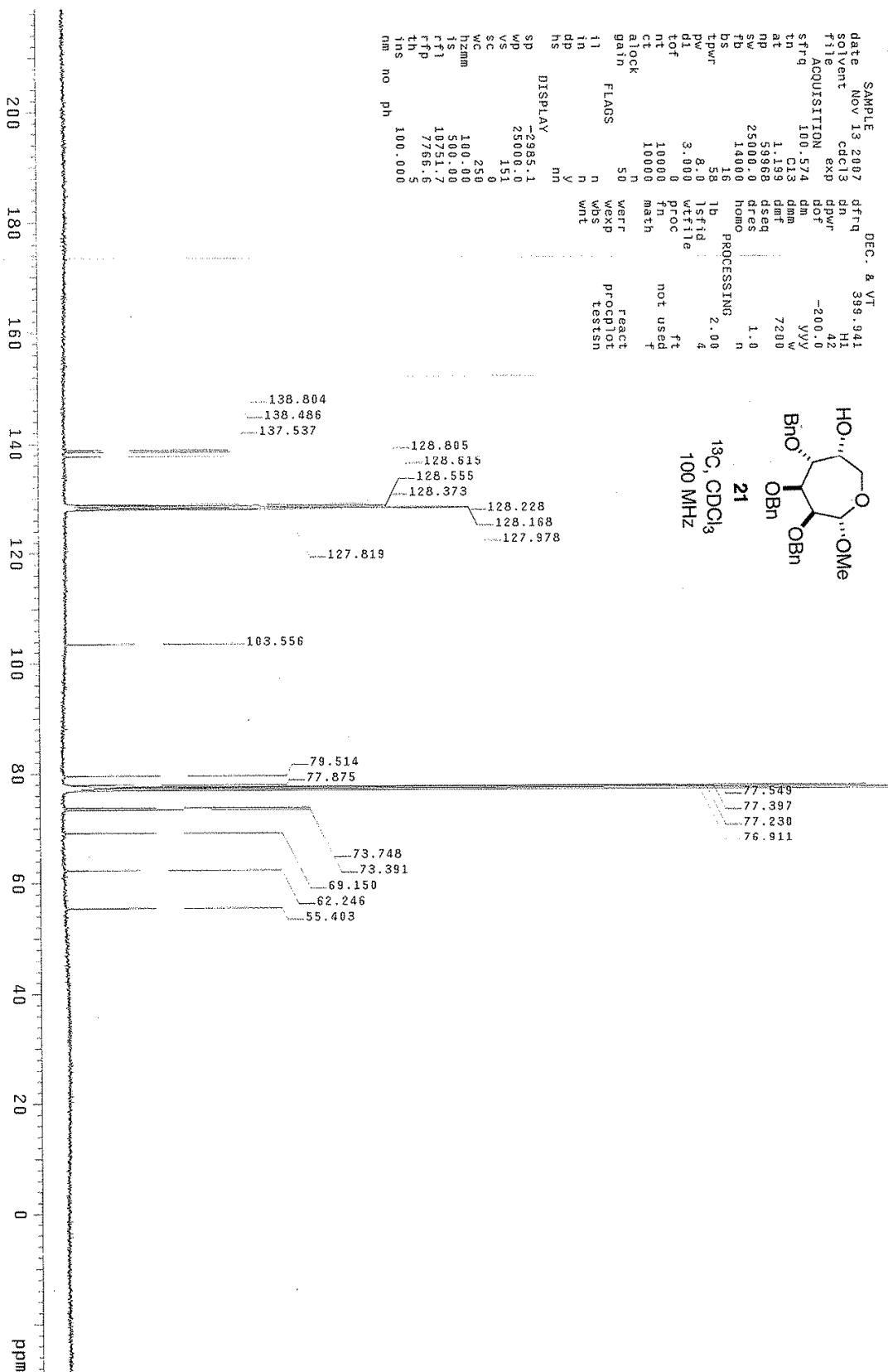
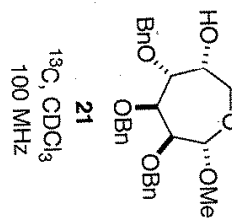


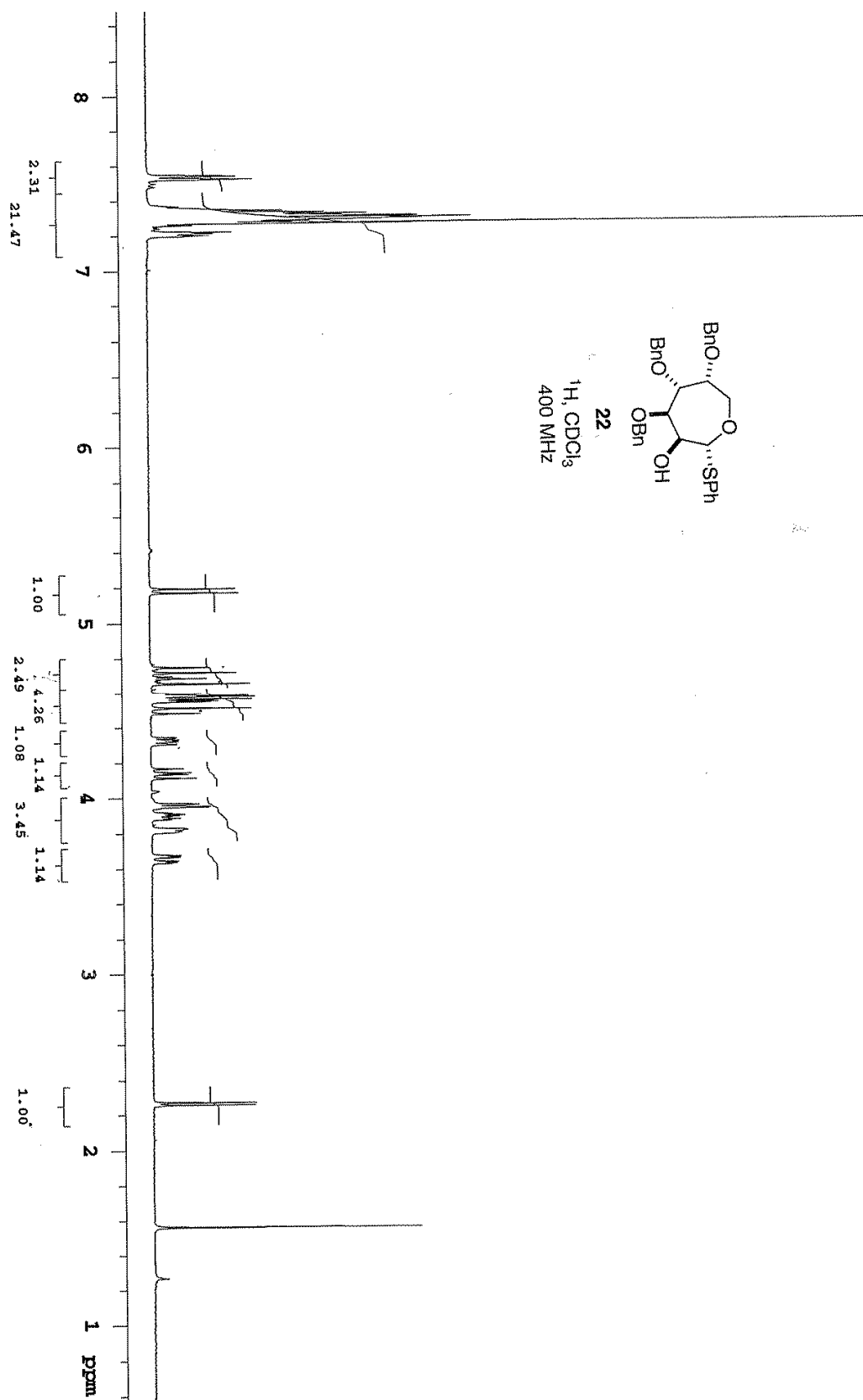


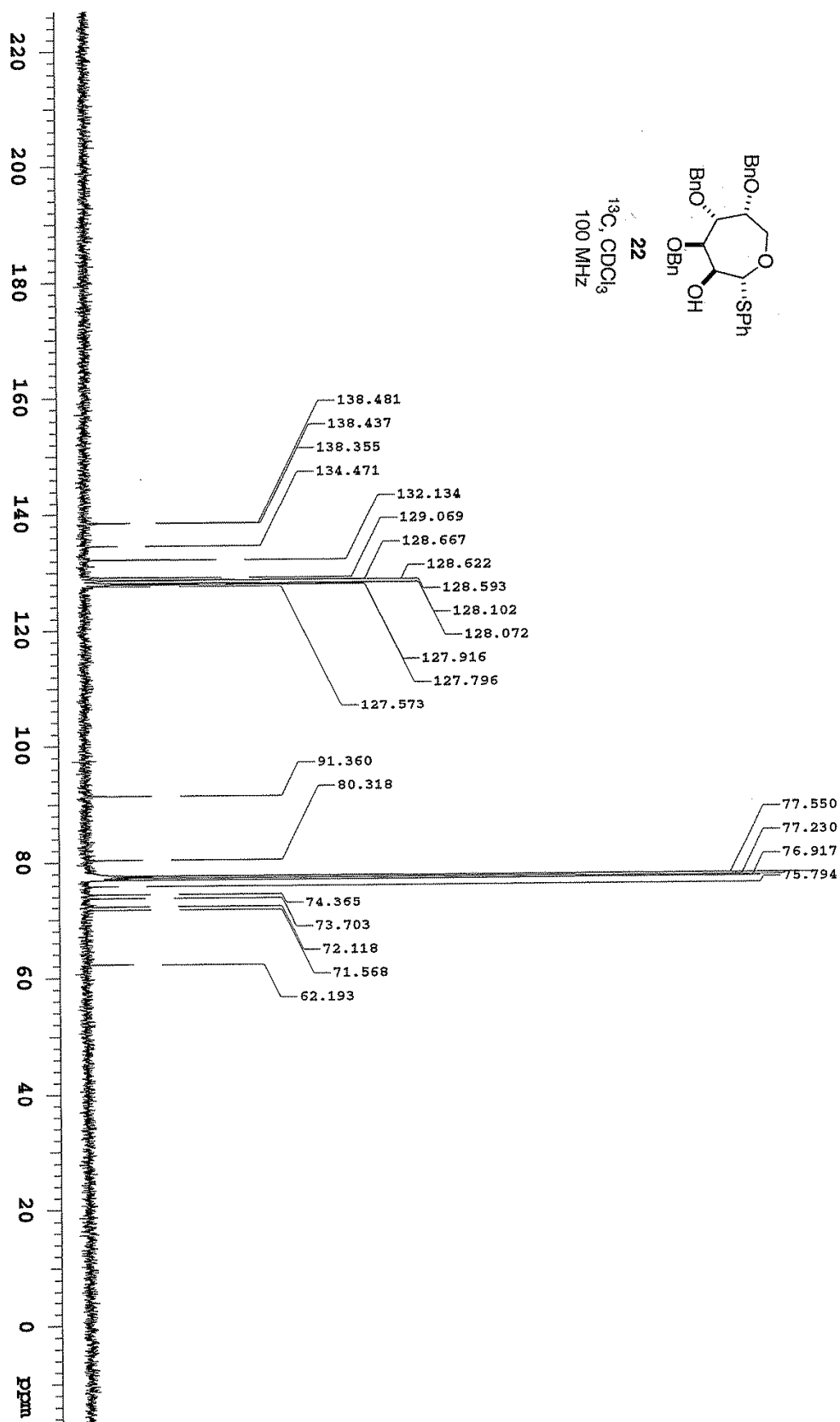
13C OBSERVE

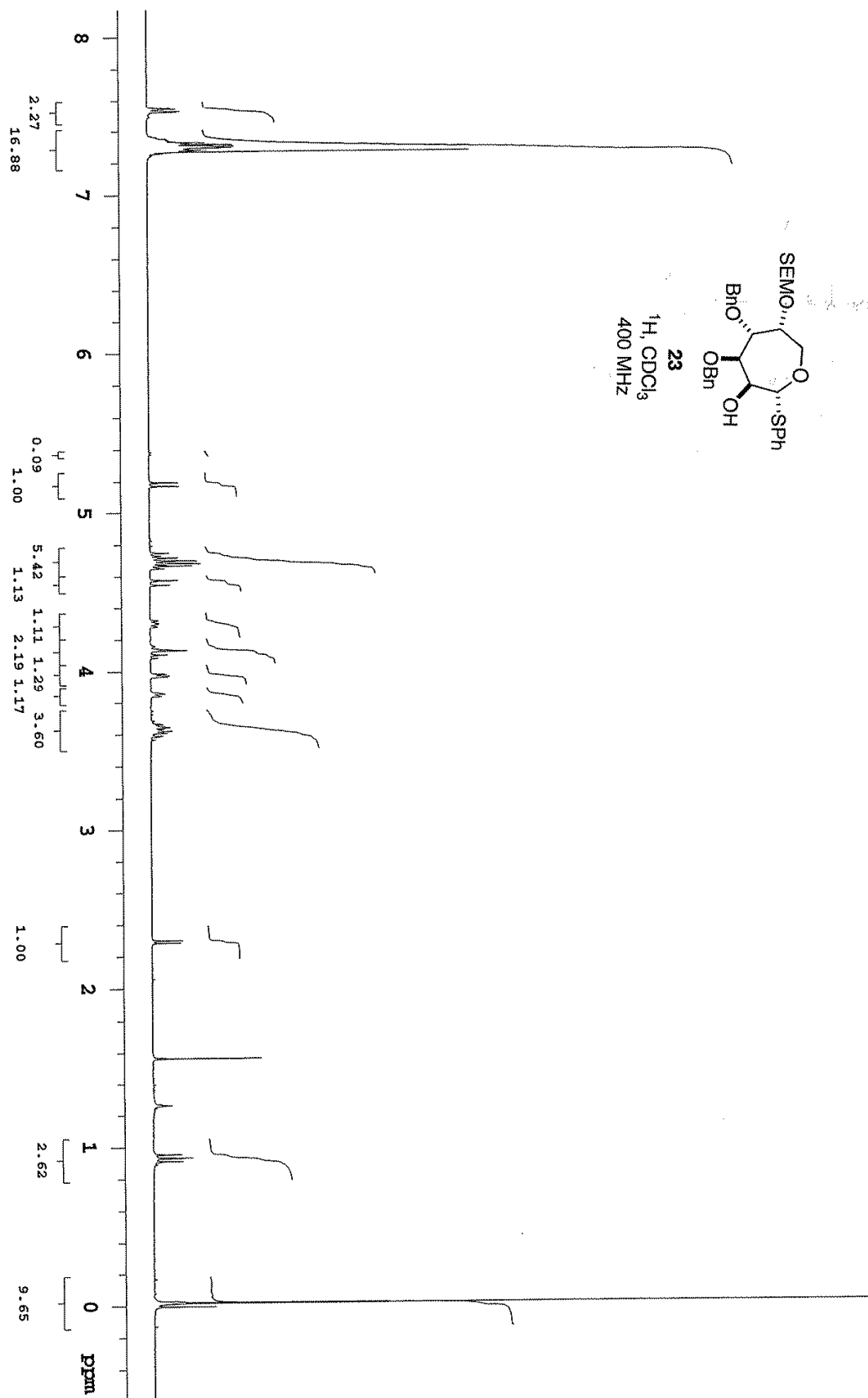
exptl std13c

SAMPLE DEC. & VT
date Nov 13 2007 dfrq 389.941
solvent cdcl3 dn H1
file exp 42
ACQUISITION
strq 100.574 dm
in C13 dnm
at 1.159 dnt
np 59968 dseq
sw 25000.0 dres
fb 14000 homo
bs 16
lpwr 58 1b PROCESSING 2.00
pw 8.0 1stid 4
di 3.000 write
tof 0 proc
nt 10000 tn not used
ct 10000 math
clock n
gain 50 weff react
fls n wexp procpot
in n wnt testn
dp n
ns y
DISPLAY
sp -2985.1 nm
wp 25000.0
vc 151
wc 0
h2mm 100.00
is 500.00
rfi 10751.7
rfp 7765.6
ins 5
no 100.000
ph



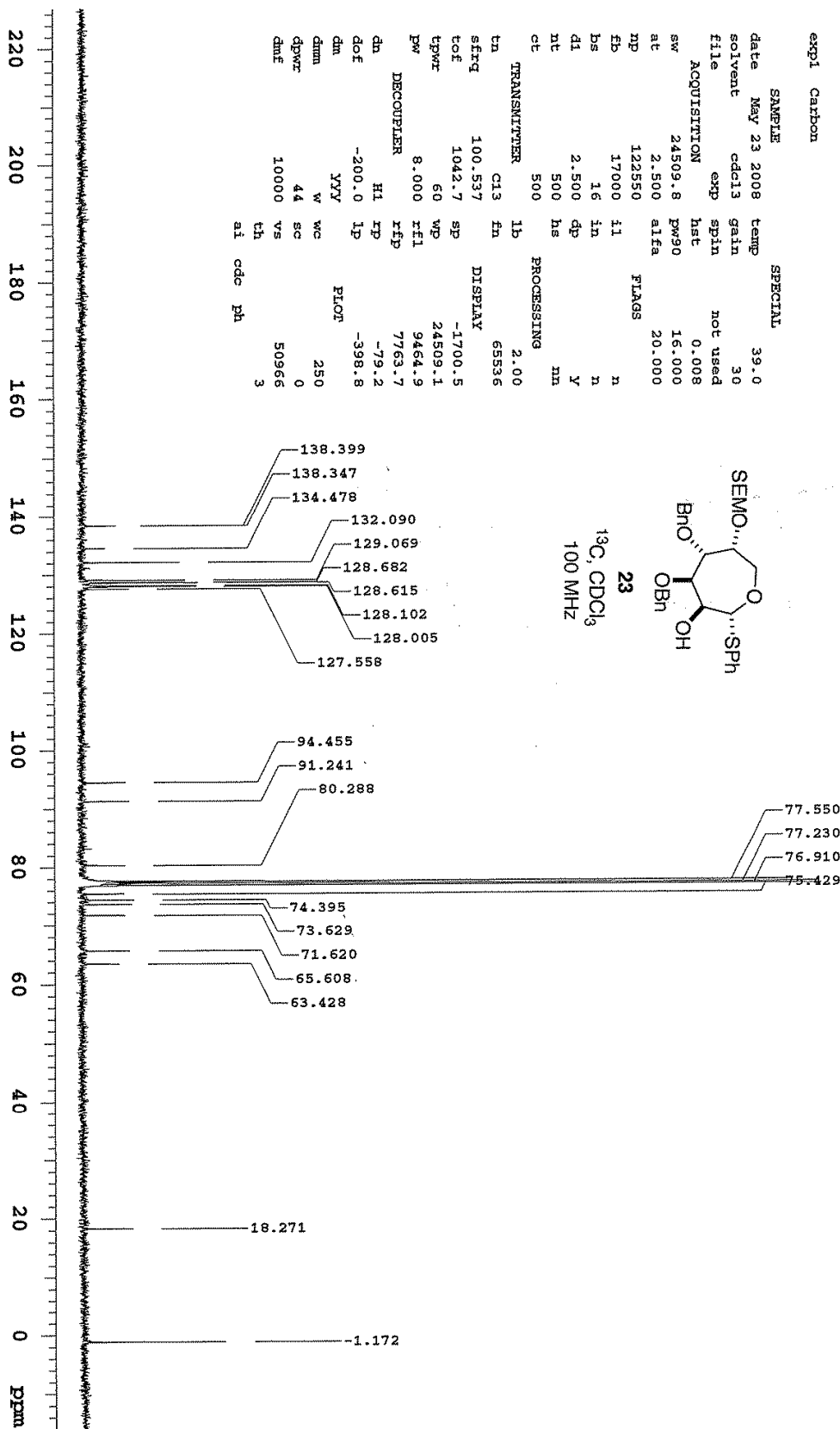
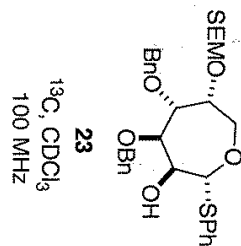


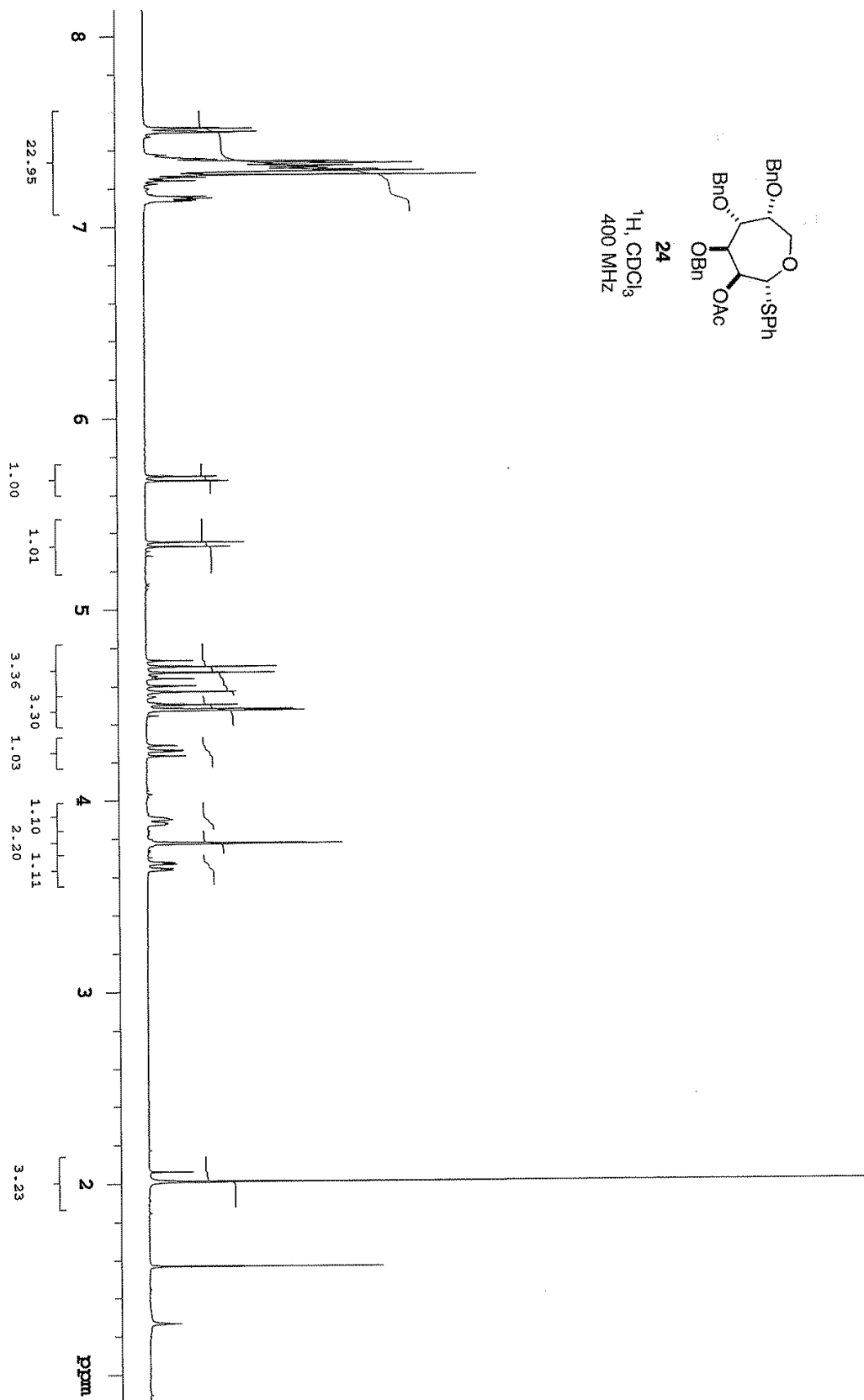
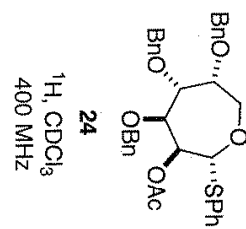


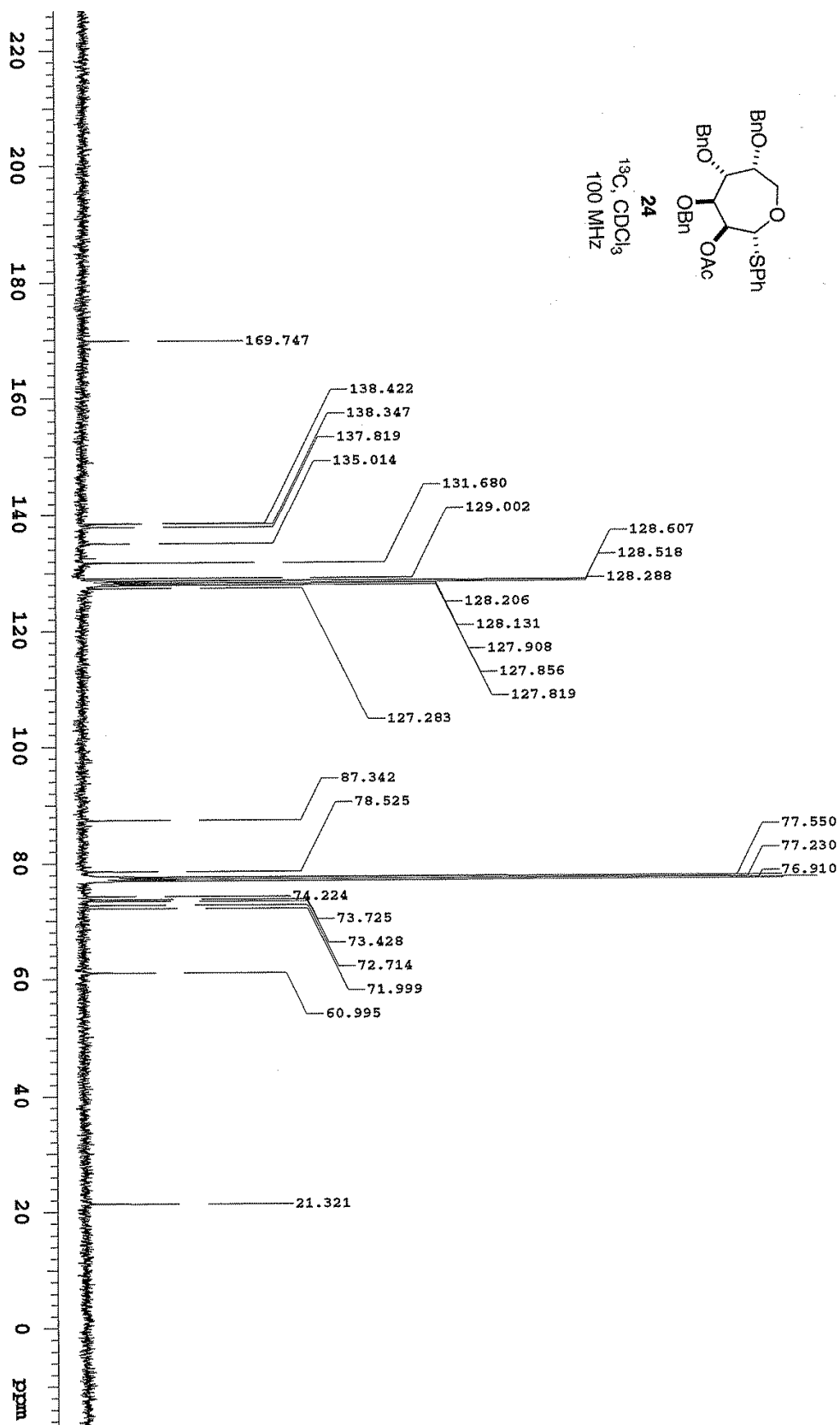


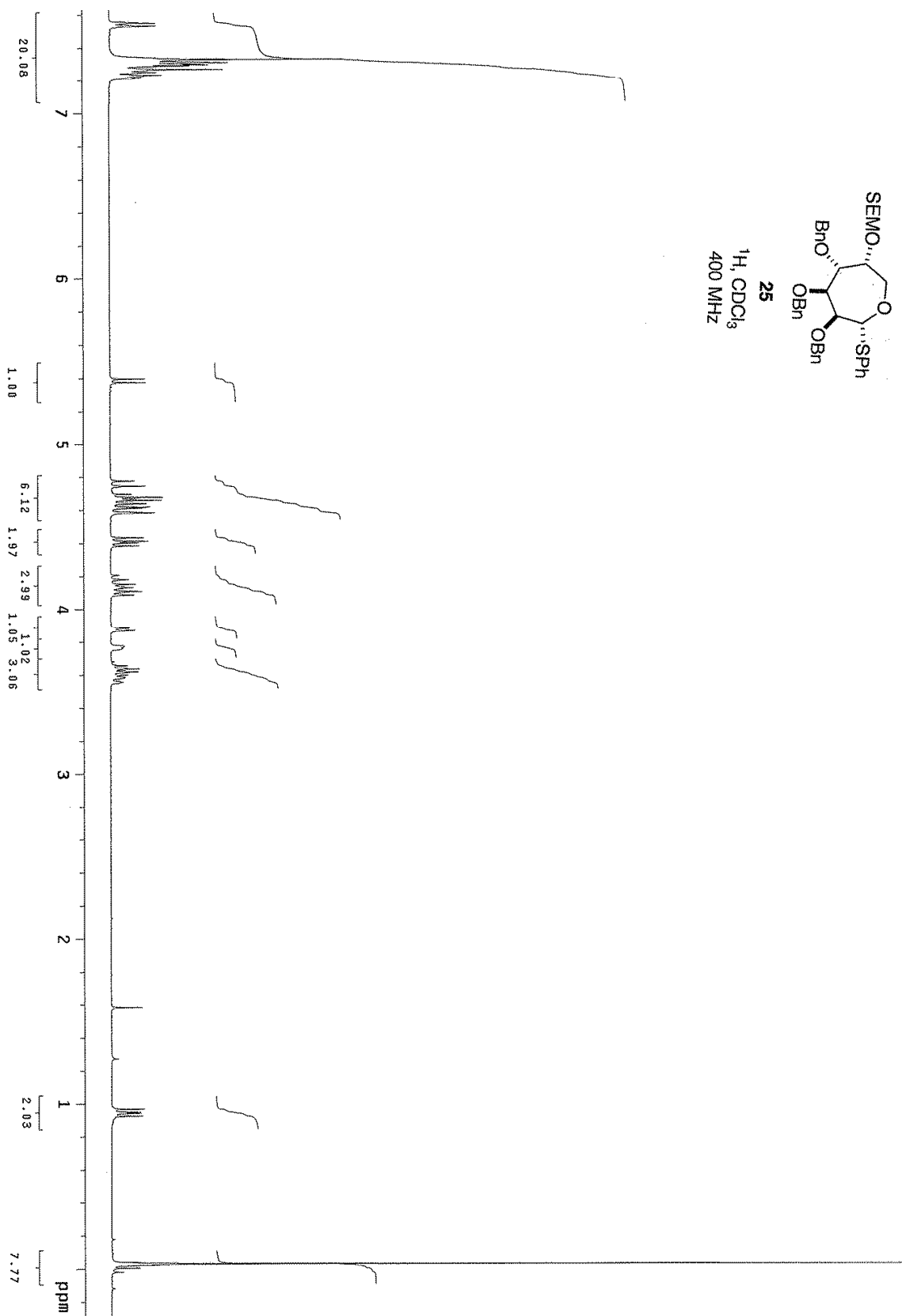
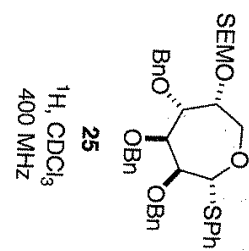
expt Carbon

SAMPLE SPECIAL
date May 23 2008 temp 39.0
solvent cdc13 gain 30
file exp spin not used
ACQUISITION hst 0.008
sv 24509.8 pw90 16.000
at 2.500 alfa 20.000
np 122550 FLAGS
fb 17000 il n
bs 16 in n
dl 2.500 dp y
nt 500 hs na
ct 500 PROCESSING
TRANSMITTER lb 2.00
tn c13 fn 65536
sfrq 100.537 DISPLAY
tof 1042.7 sp -1700.5
tpwr 60 wp 24509.1
pw 8.000 rfl 9464.9
DECOUPLER rfp 7763.7
dn H1 lp -79.2
dof -200.0 lp -398.8
dm YYY FLOT
dmm w wc 250
dpmr 44 sc 0
dmf 10000 vs 50966
al cdc ph 3



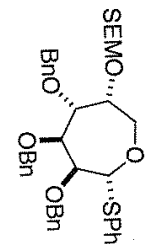






13C OBSERVE

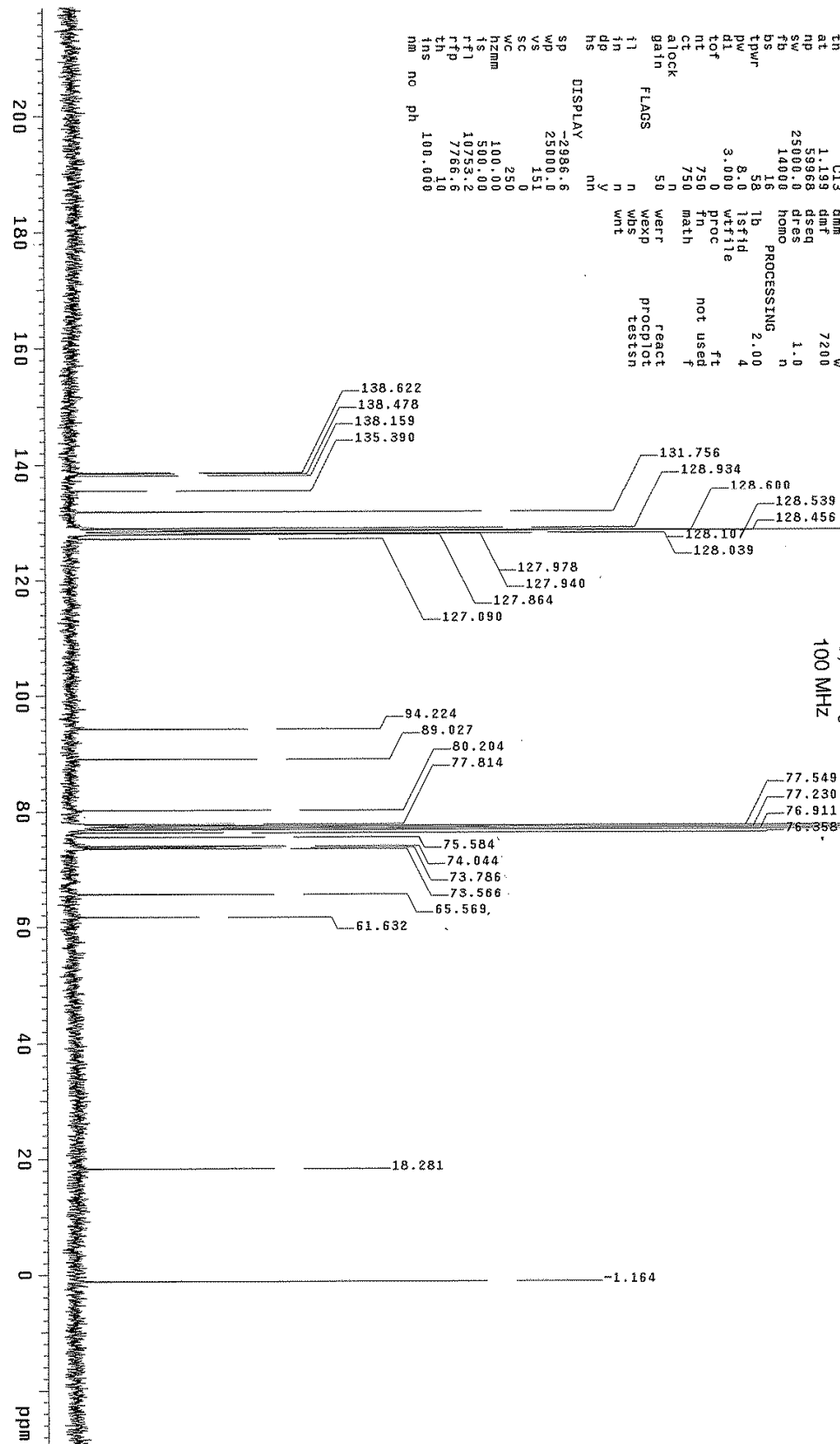
exp4 std13c

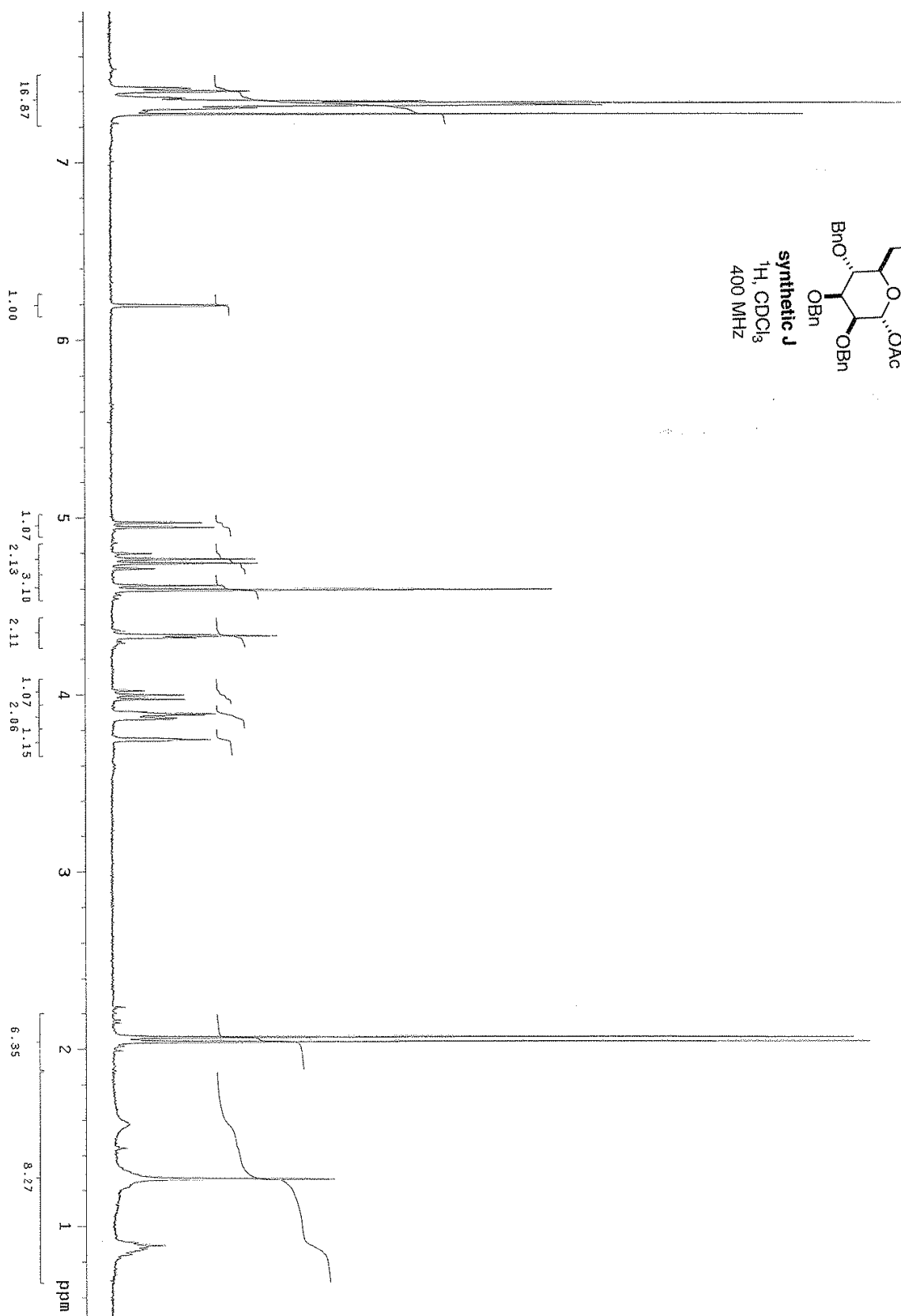
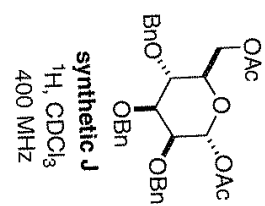


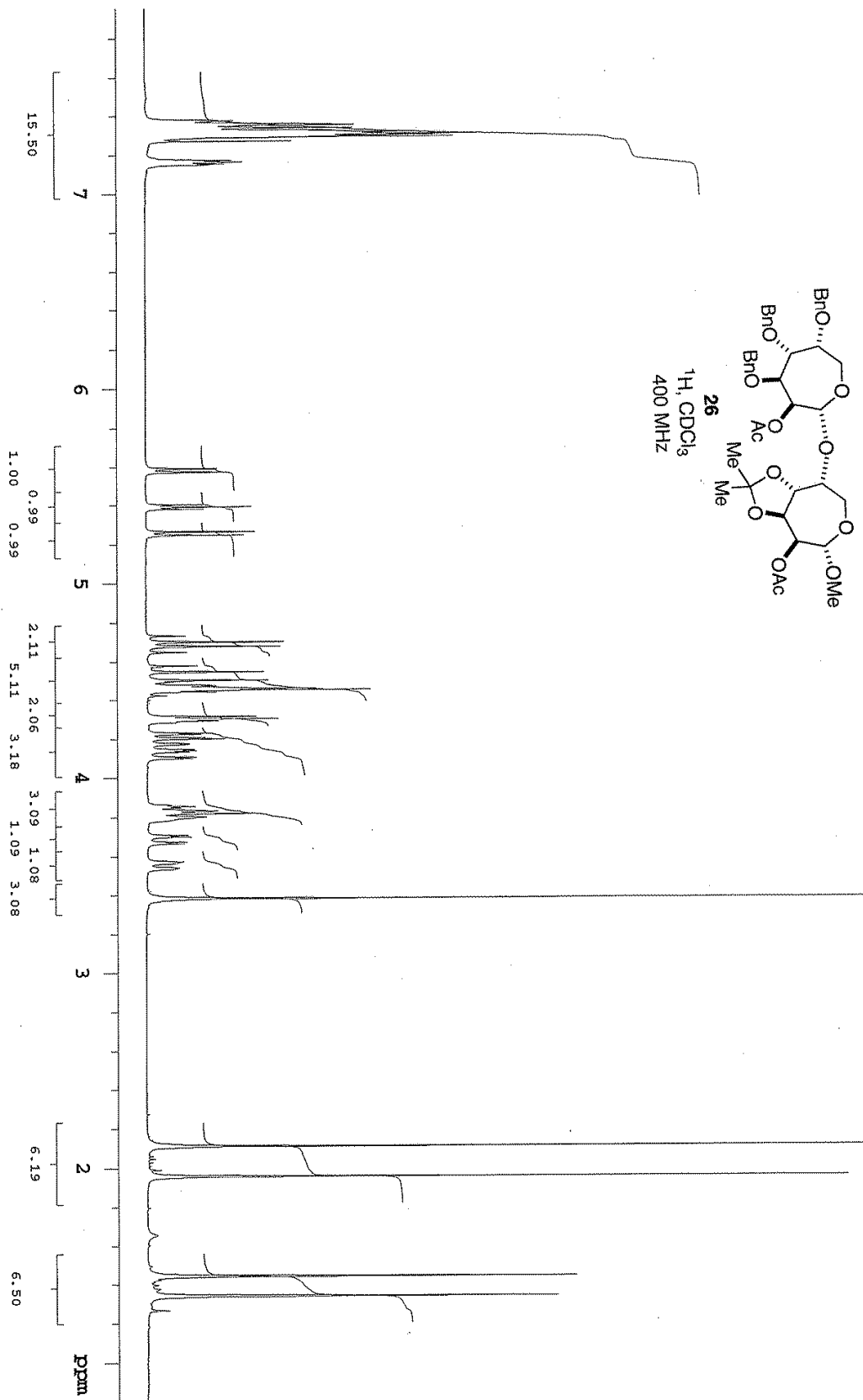
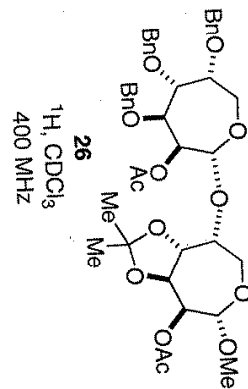
25

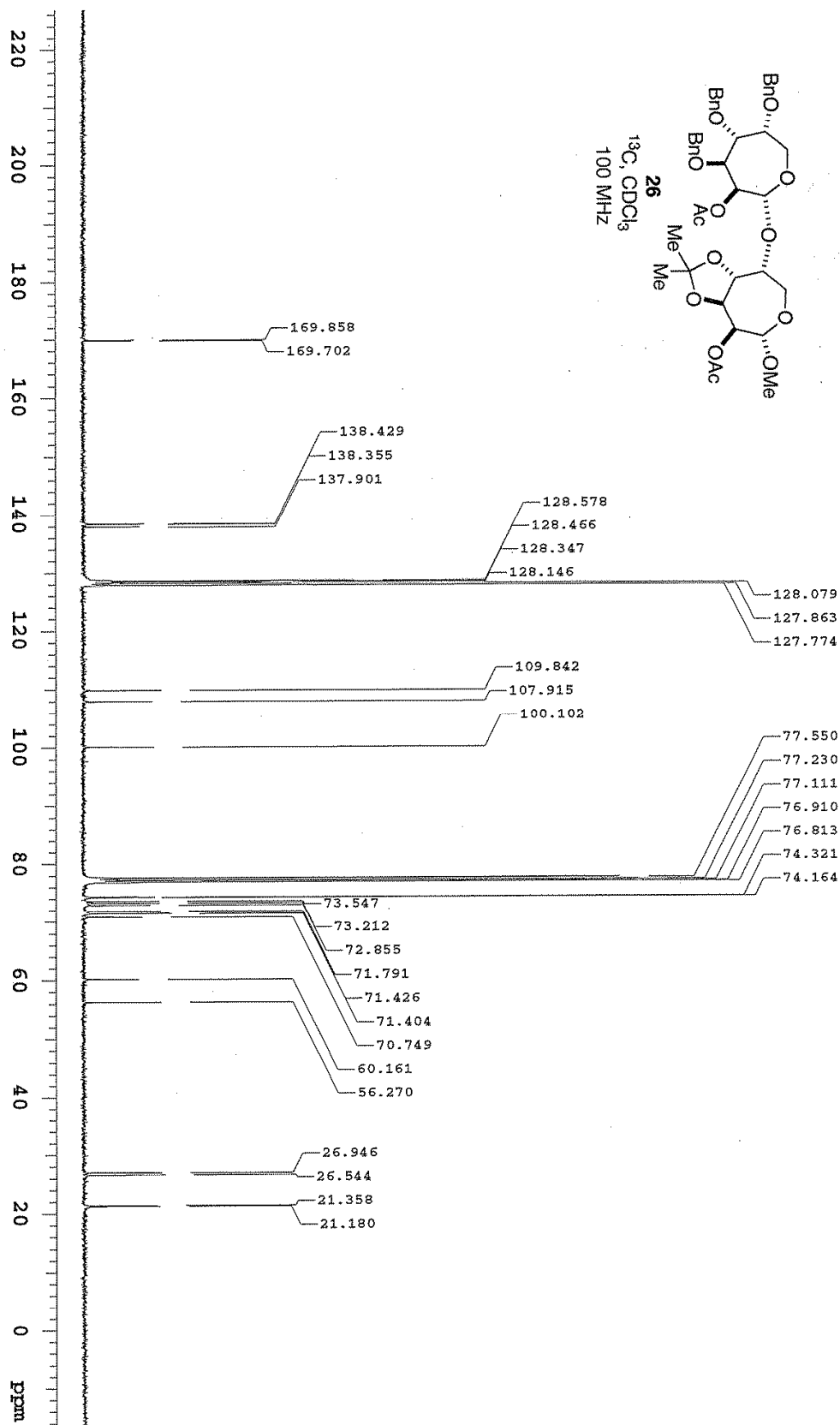
¹³C, CDCl₃
100 MHz

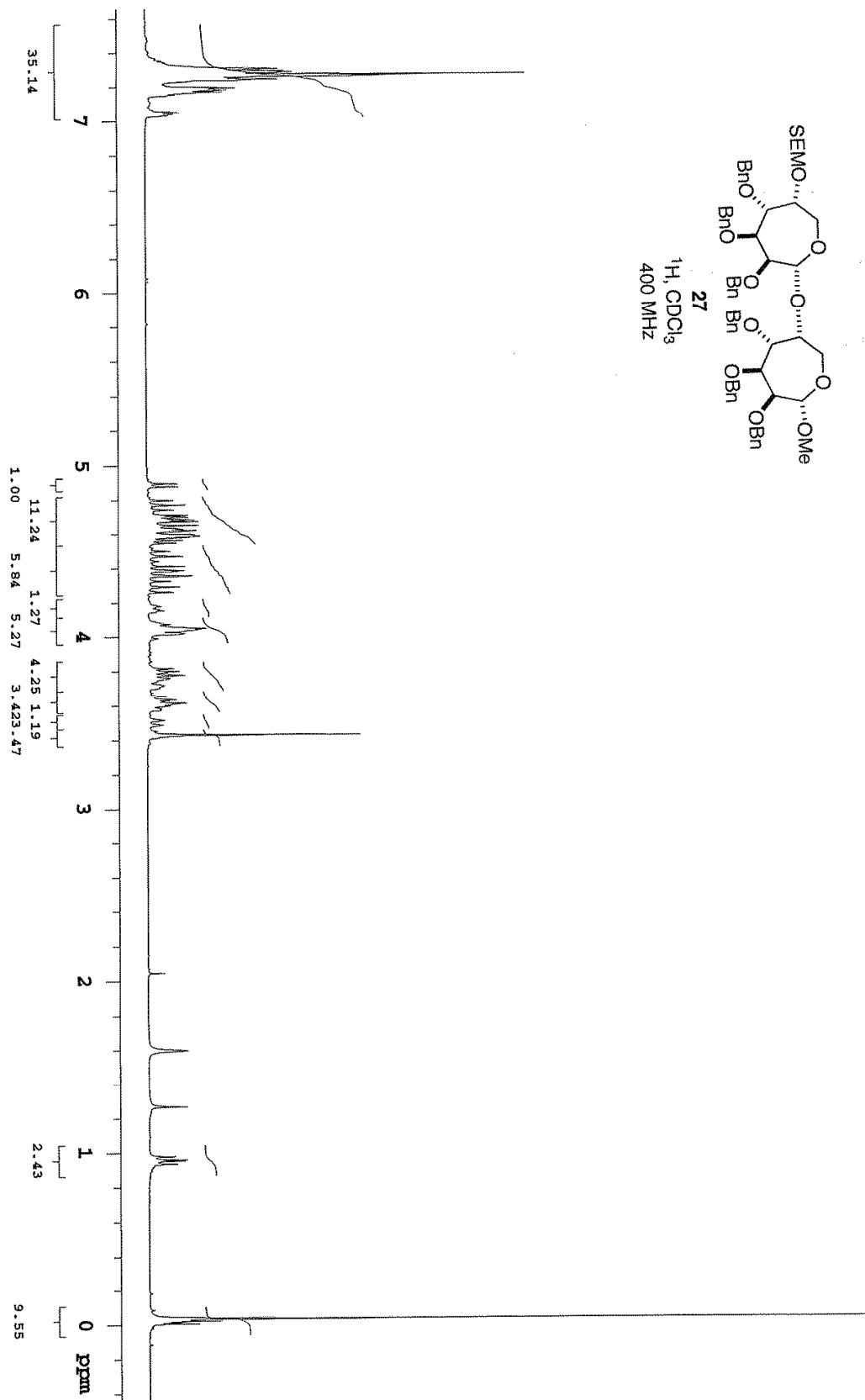
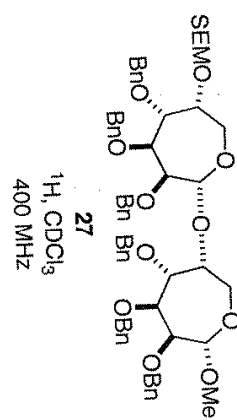
SAMPLE DEC. & VT
date Nov 29 2007 dfrq 399.941
solvent cdcl3 dn hl
file exp 42
ACQUISITION exp 42
sfrq 100.574 dm
th C13 dmm
at 1.199 dnt
np 33968 dresq
sw 25000.0 dres 1.0
fb 14000 homo
ds 16
tpwr 1b
pw 8.0 1sfid 4
d 3.00 wfile
tcf 750 proc
nt 750 ft
c 750 math
a lock 50 wef
gain n wexp
f1 n wbs
f2 n wnt
hs n
DISPLAY
sp -2986.6
wd 25000.0
vs 151
sc 0
wc 250
hmm 100.000
ts 500.000
rf1 10753.2
rfp 7766.6
th 10
ins 100.000
nm no ph











expt Carbon

SAMPLE
date Jan 17 2008 temp 31.0
solvent cdcl3 gain 50
file /home/nmruser/spin not used
/mcdonald/Matt/SEM-hst 0.008
_perbenzyl_manno_d- pw90 16.000
isaccharide_c13.fi- alfa 10.000

SPECIAL

ACQUISITION

FLAGS

PROCESSING

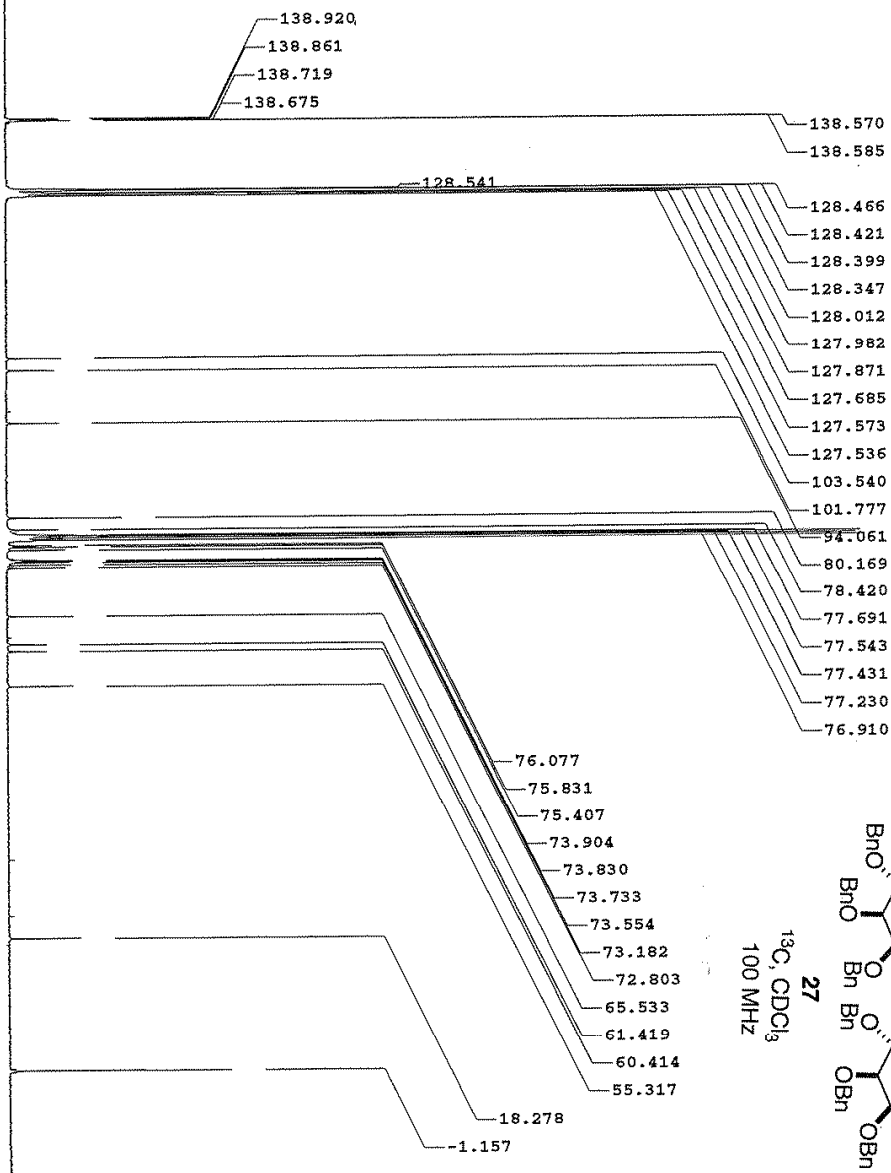
DISPLAY

TRANSMITTER

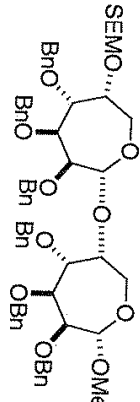
DECOUPLER

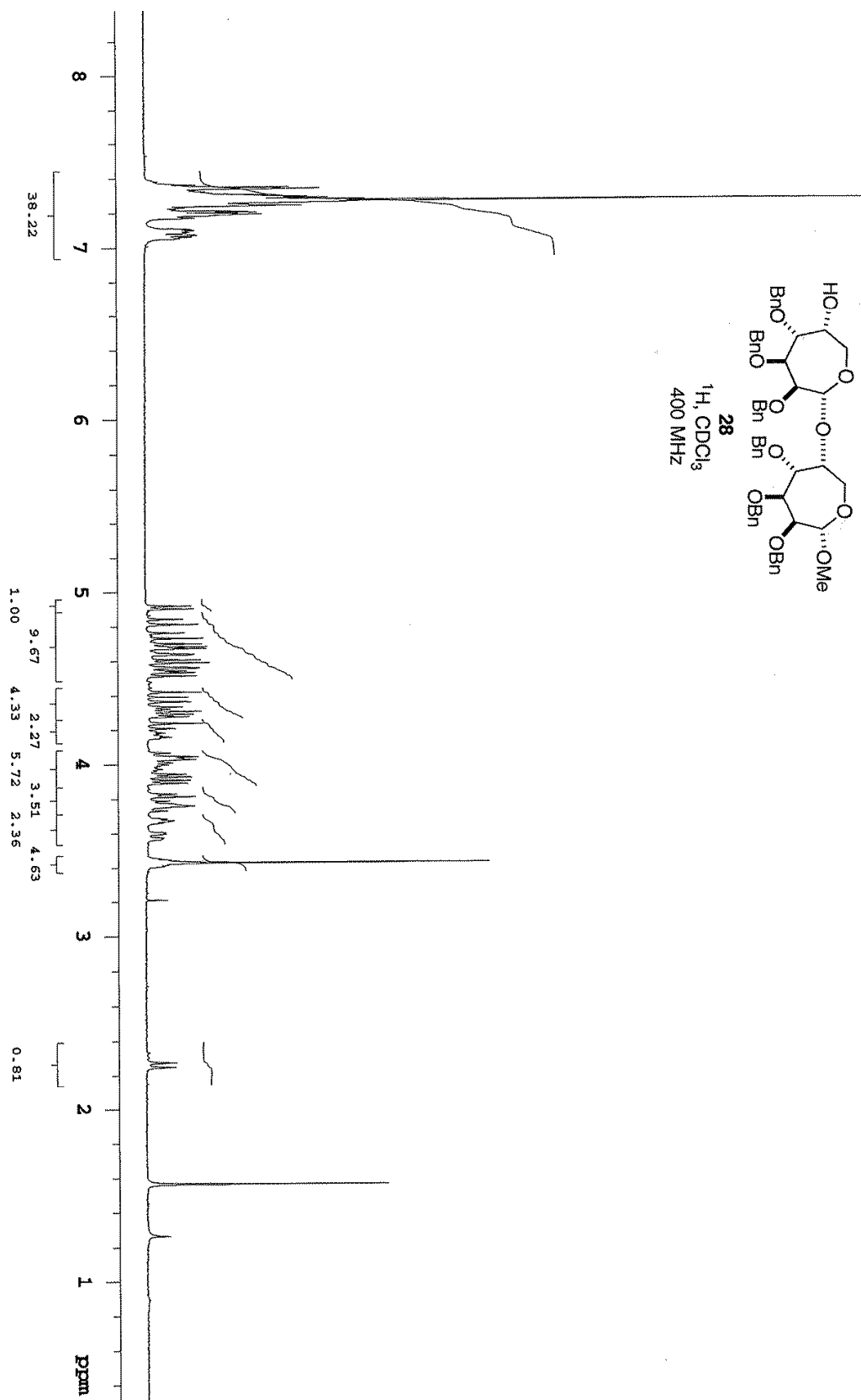
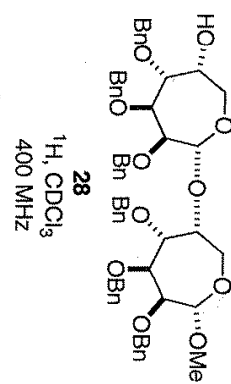
sw 24509.8 in n
at 2.500 dp y
np 12250 hs nm
fb 17000
bs 16 lb 2.00
dl 2.500 fm 65536
nt 10750
ct 10750 sp -1702.8
tn C13 xfl 24509.1
sfrq 100.537 xfp 9467.2
tof 1042.7 xp 7763.7
tpwr 60 lp 4.2
pw 7.250 plot -206.1
wv wc 250
dn H1 sc 0
dof -200.0 vs 5248
dm YYY th 3
dmm w al cdc ph
dpmr 44
dmf 10000

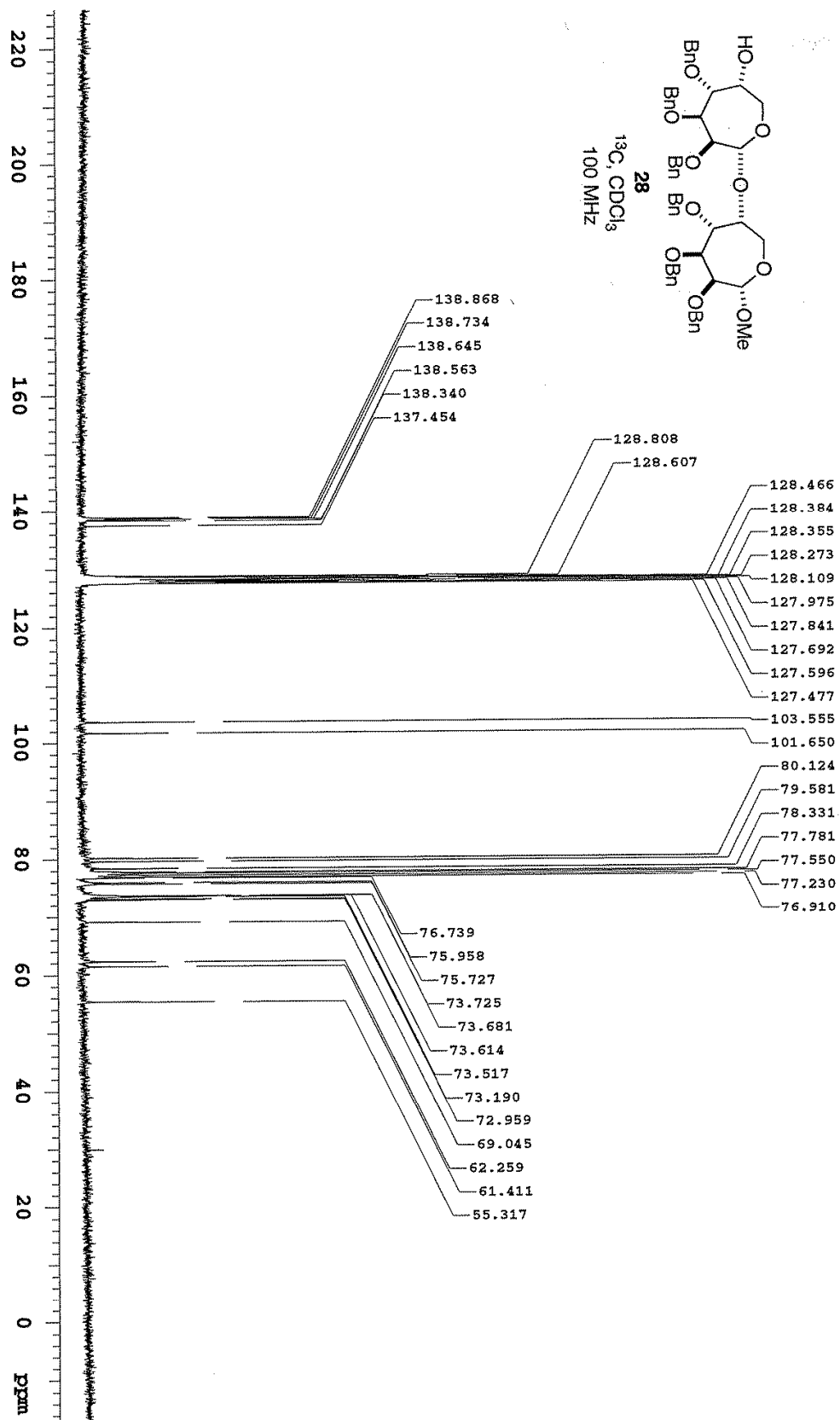
220 200 180 160 140 120 100 80 60 40 20 0 ppm

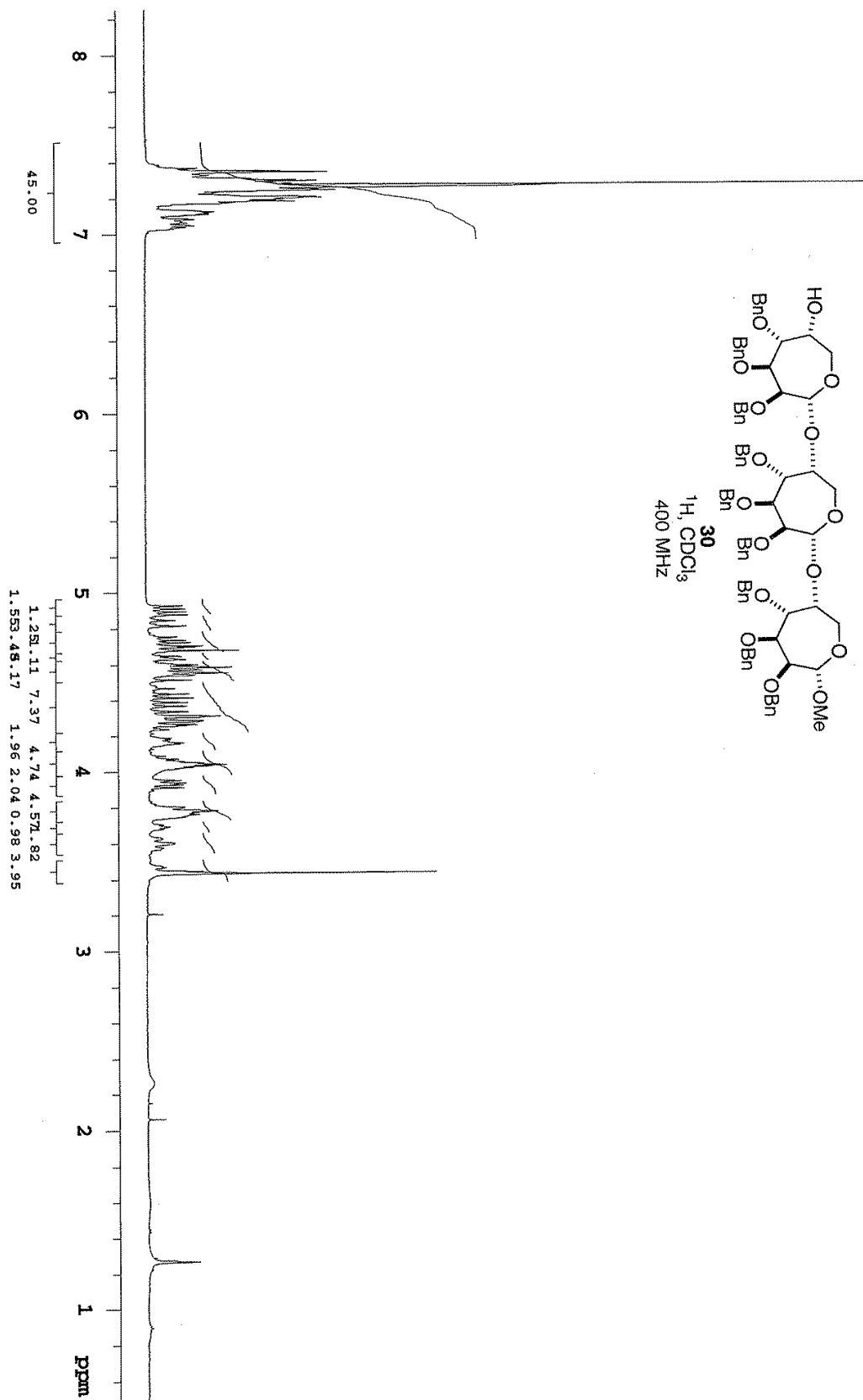
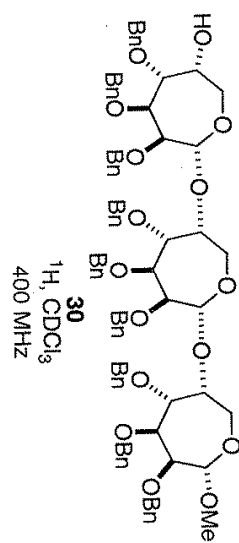


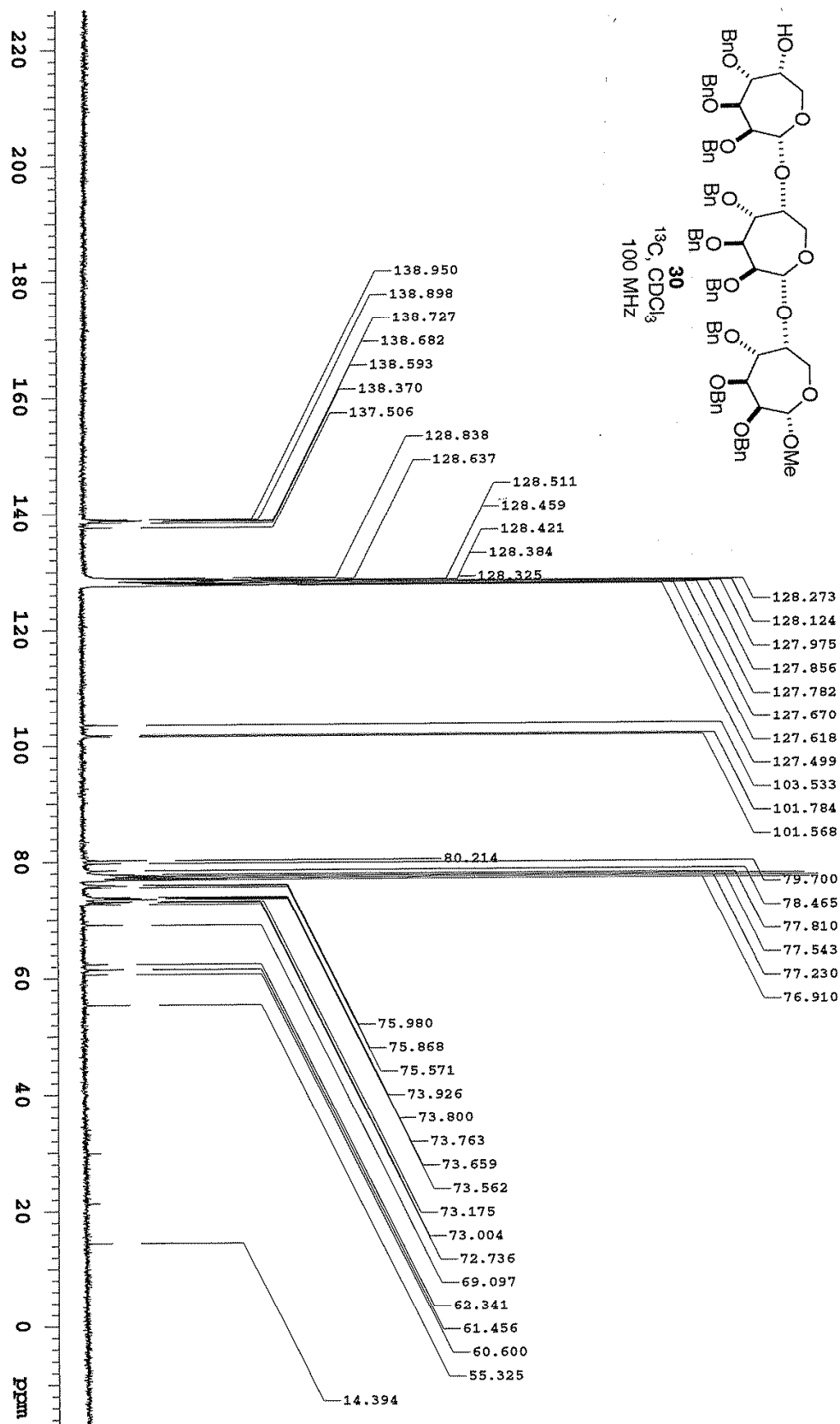
13C, CDCl3
100 MHz

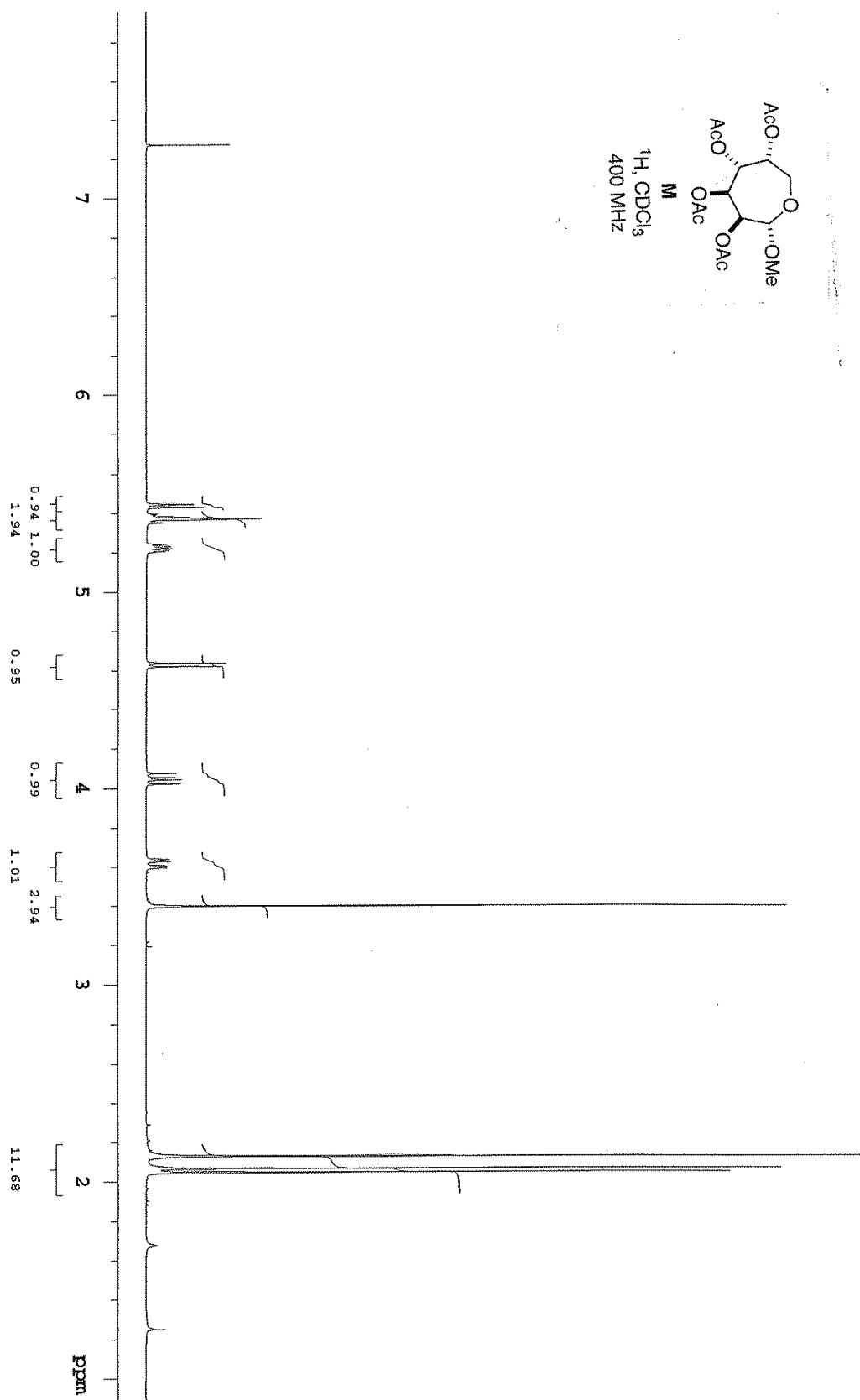
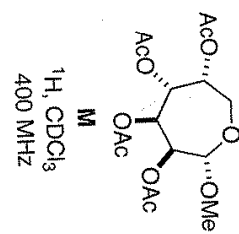


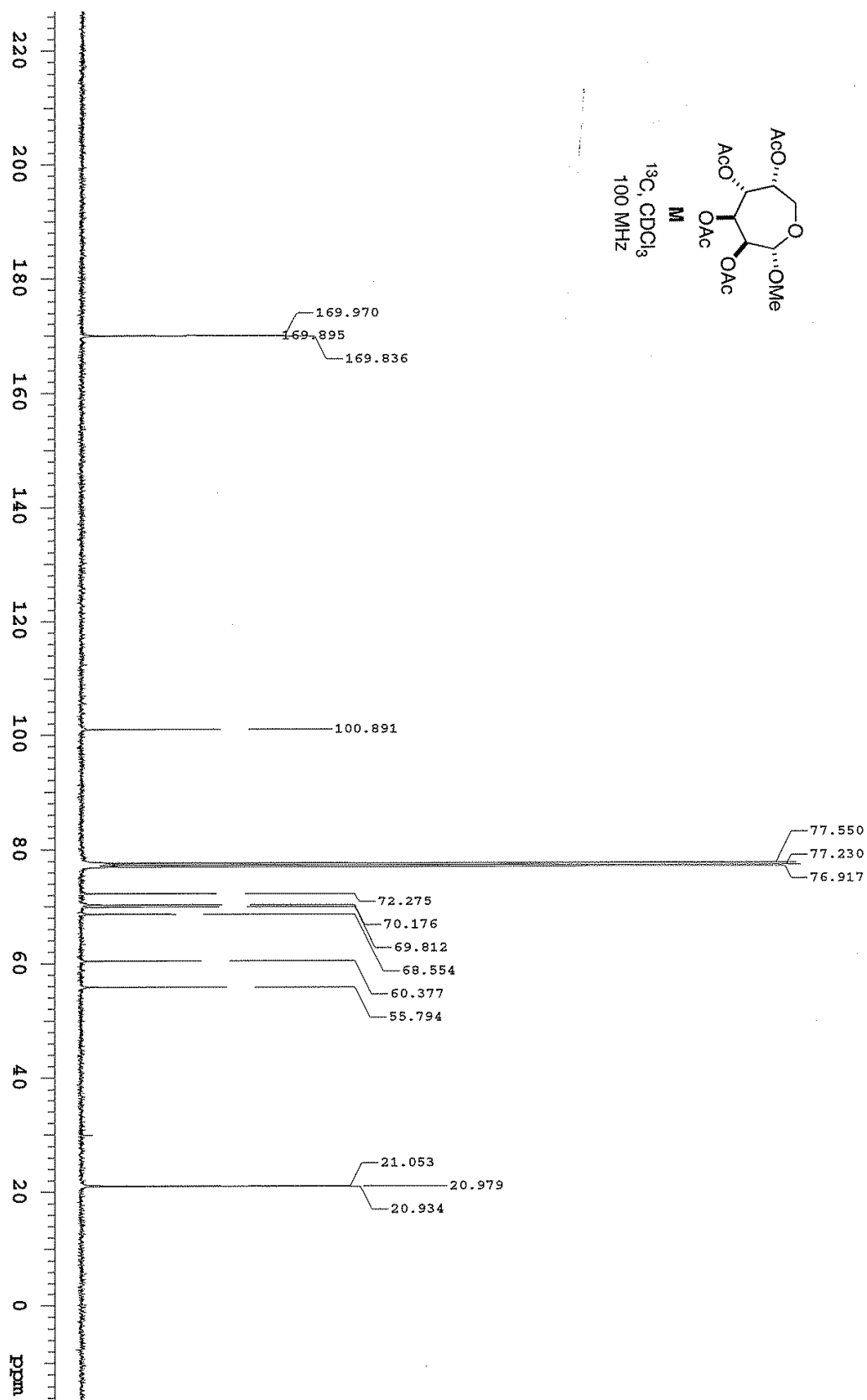


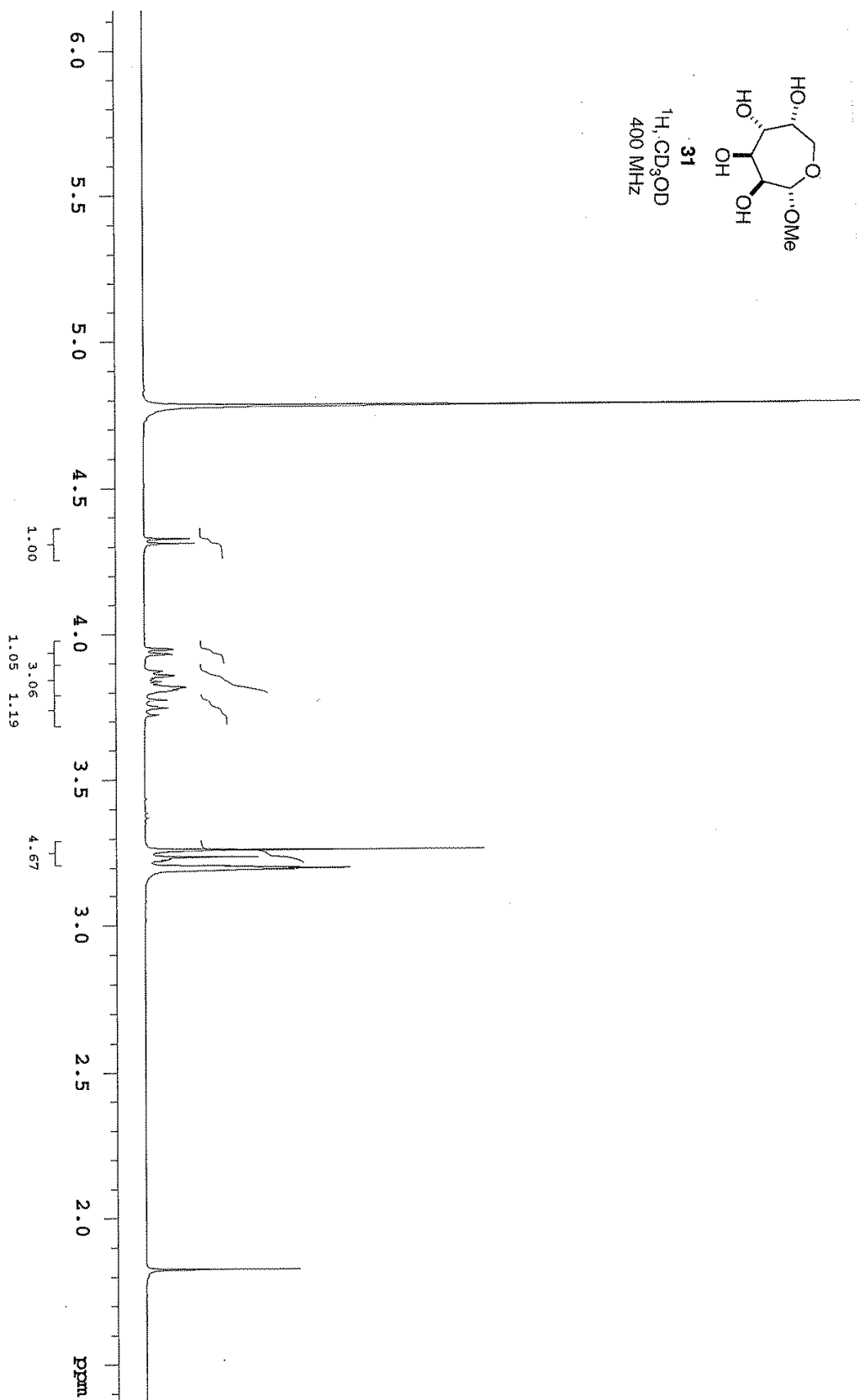
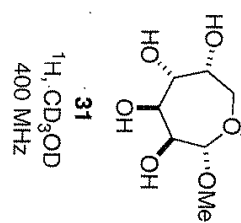


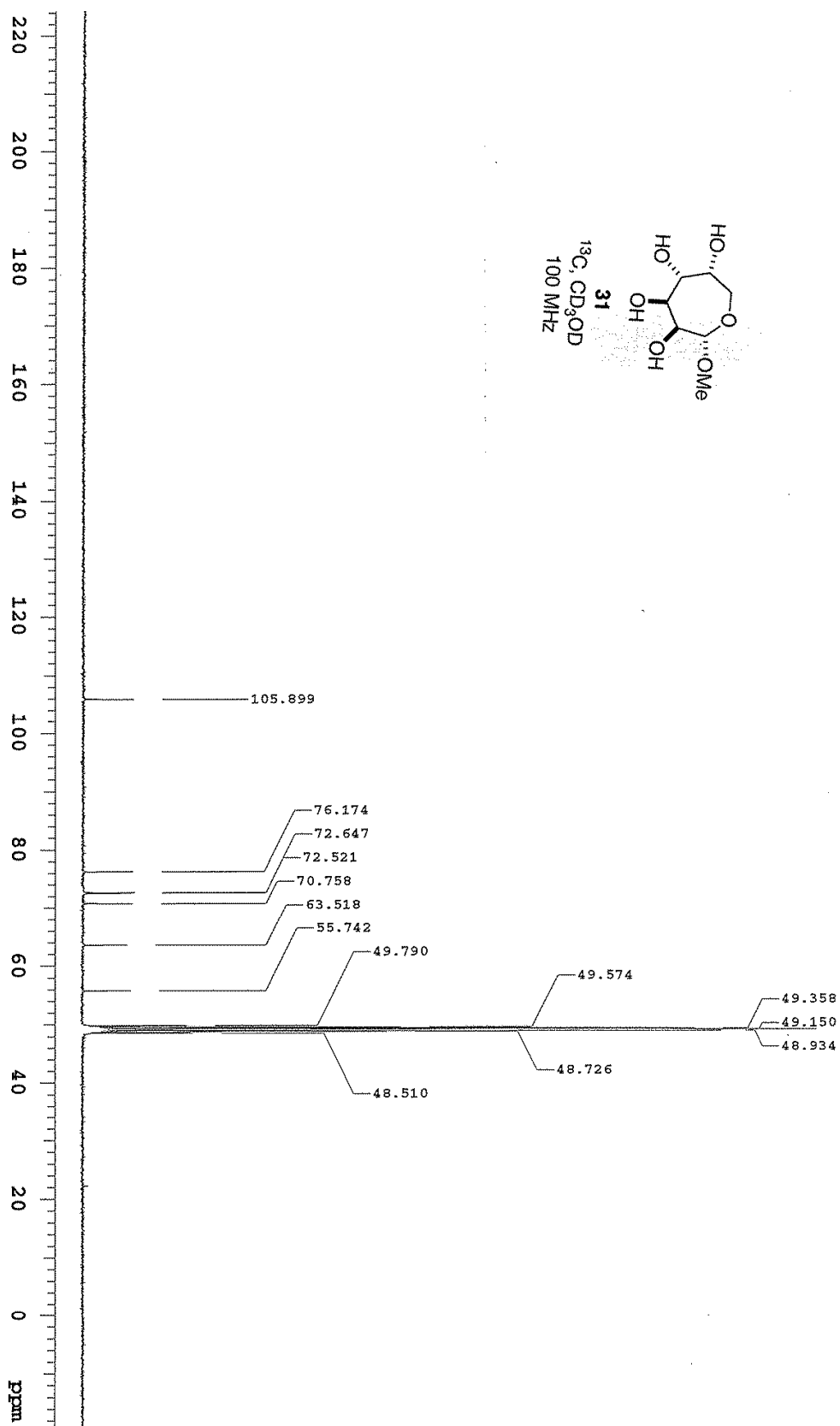


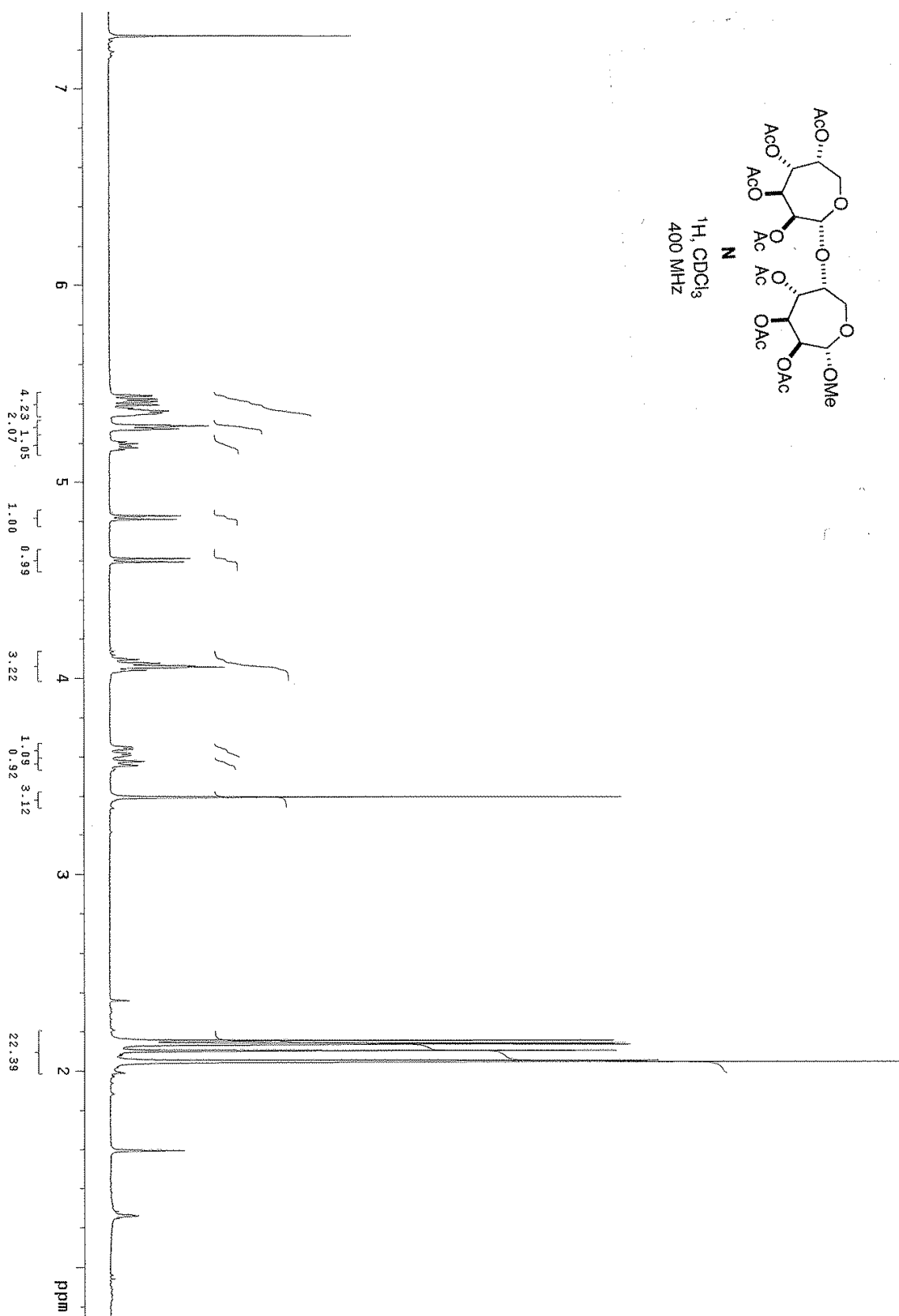
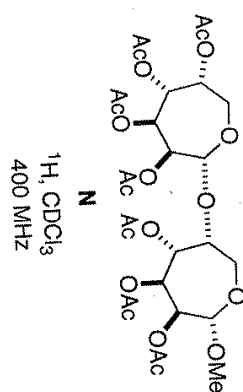


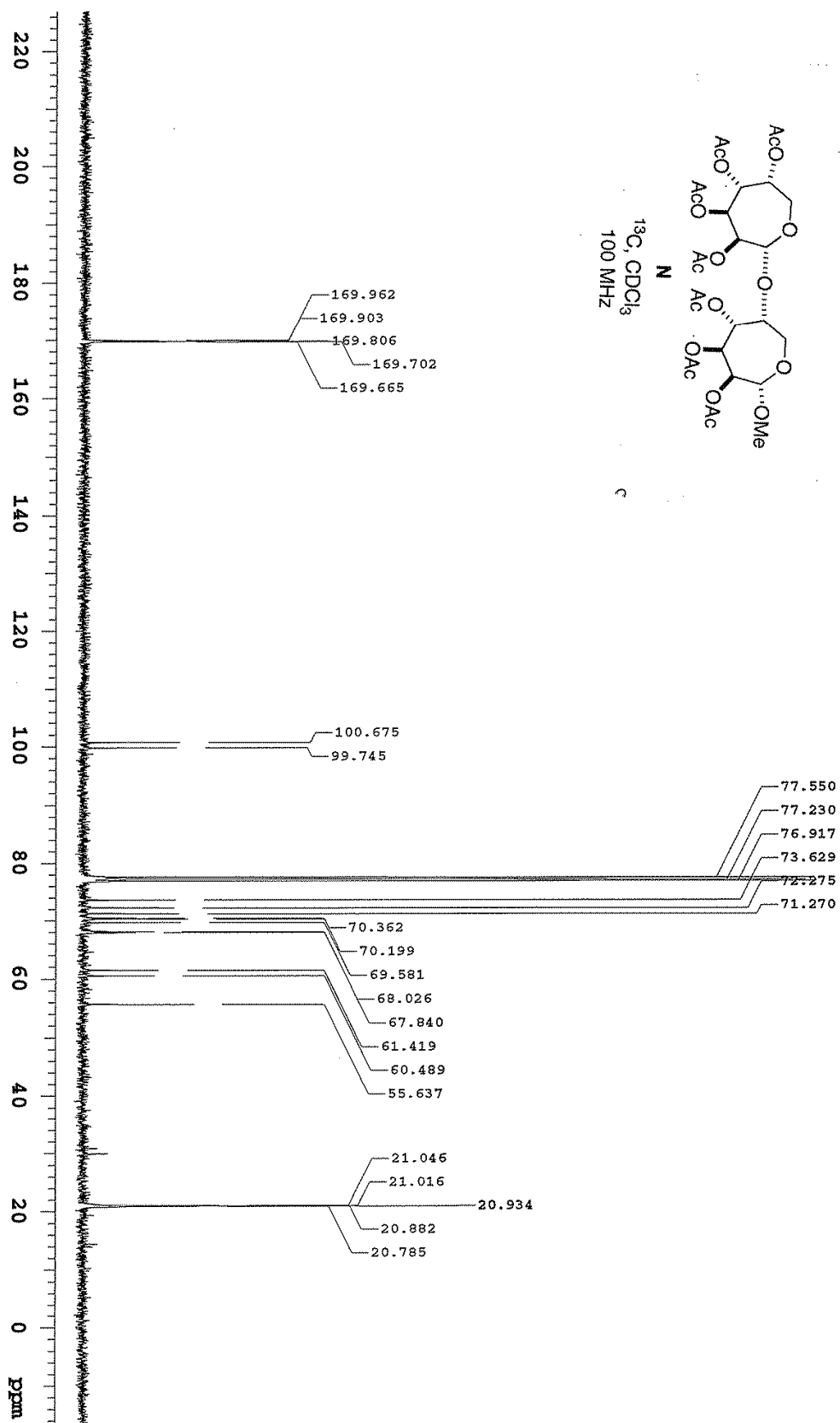












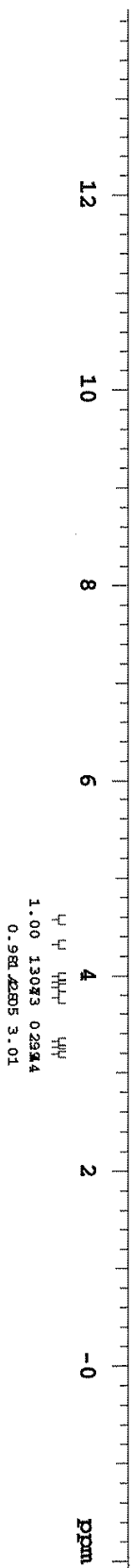
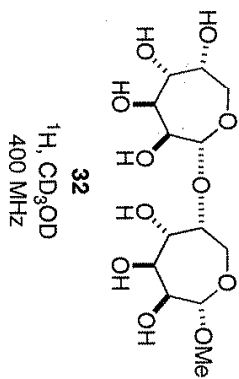
expt1 Proton

SAMPLE
date Apr 11 2008 temp 39.0
solvent cd3od gain not used
file /home/nmruser/spin 20
/mcdonald/Matt/poi-hst 0.008
yol mannodisacchar-pw90 11.000
ide_CD3OD_h1.fid alfa 6.600

ACQUISITION
sw 6410.3 il 11
at 2.300 in n
np 29488 dp y
fb 4000 hs nn
bs 16
d1 1.500 lb 0.20
nt 8 fn 32768
ct 8

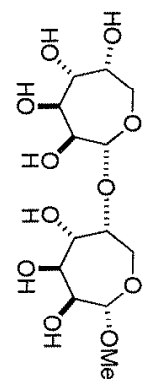
PROCESSING
DISPLAY
-845.0
tn H1 wp 6409.9
stfq 399.791 rfi 2756.4
tof 399.5 rfp 1911.0
lpwr 60 rp -175.8
pw 5.500 lp -31.3

DECOUPLER
dn C13 wc 250
dof 0 sc 0
dm mn vs 52
dmm c th 9
dpwr 42 ai cdc ph
dmf 32000



exptl Carbon

SAMPLE
date Apr 11 2008
solvent cd3od
file exp
ACQUISITION
sw 24509.8
at 2.500
mp 122550
fb 17000
bs 16
dl 2.500
nt 11500
ct 11500
TRANSMITTER
tn C13
sfreq 100.537
tof 1042.7
tpwr 60
pw 8.000
DECOUPLER
dn H1
dof -200.0
dm YYY
dmm W
dpr 44
dmf 10000
SPECIAL
temp 39.0
gain 50
exp spin not used
hst 0.008
pwr90 16.000
alfa 10.000
FLAGS
il n
in n
dp y
hs mn
PROCESSING
lb 2.00
fn 65536
DISPLAY
sp -1563.5
wp 24509.1
rfl 6505.2
rfp 4940.9
rp -63.0
lp -198.5
PLOT
wc 250
sc 0
vs 3010
ch 2
ai cdc ph



32
¹³C, CD₃OD
100 MHz

