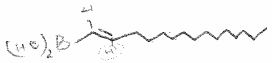
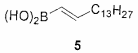
Table 1, Compound 5

Standard Proton Parameters

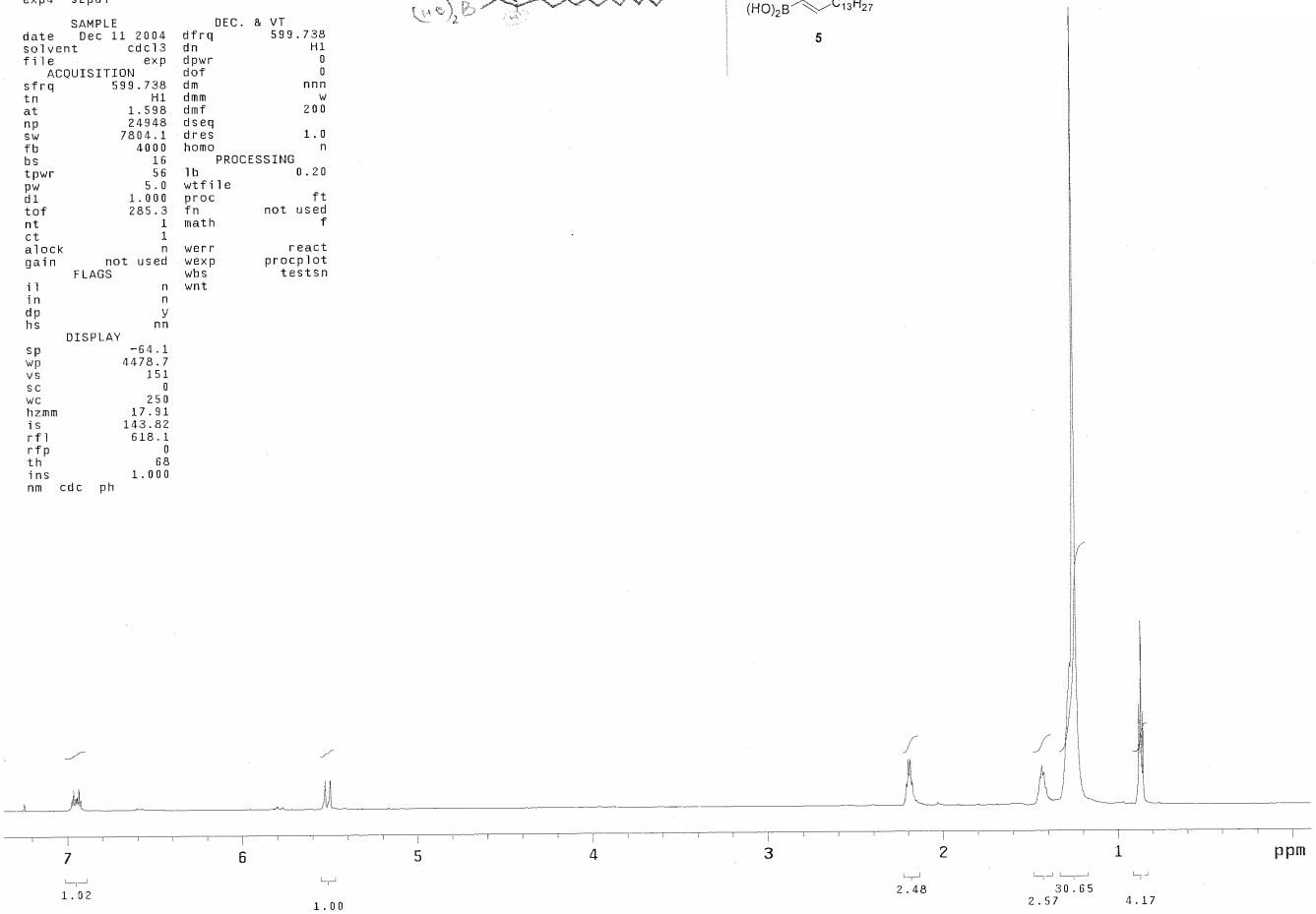
exp4 s2pul

SAMPLE		DEC. & VT	
date	Dec 11 2004	dfrq	599.738
solvent	cdcl3	dn	H1
file	exp	dpwr	0
ACQUISITION		dof	0
sfrq	599.738	dm	nnn
tn	H1	dmm	w
at	1.598	dmf	200
np	24948	dseq	
sw	7804.1	dres	1.0
fb	4000	homo	n
bs	16	PROCESSING	
tpwr	56	lb	0.20
pw	5.0	wtfile	
d1	1.000	proc	ft
tof	285.3	fn	not used
nt	1	math	f
ct	1		
alock	n	werr	react
gain	not used	wexp	procplot
FLAGS		wbs	testsn
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-64.1		
wp	4478.7		
vs	151		
sc	0		
wc	250		
hzmm	17.91		
is	143.82		
rfl	618.1		
rfp	0		
th	68		
ins	1.000		
nm cdc ph			





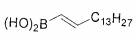
5



13C OBSERVE

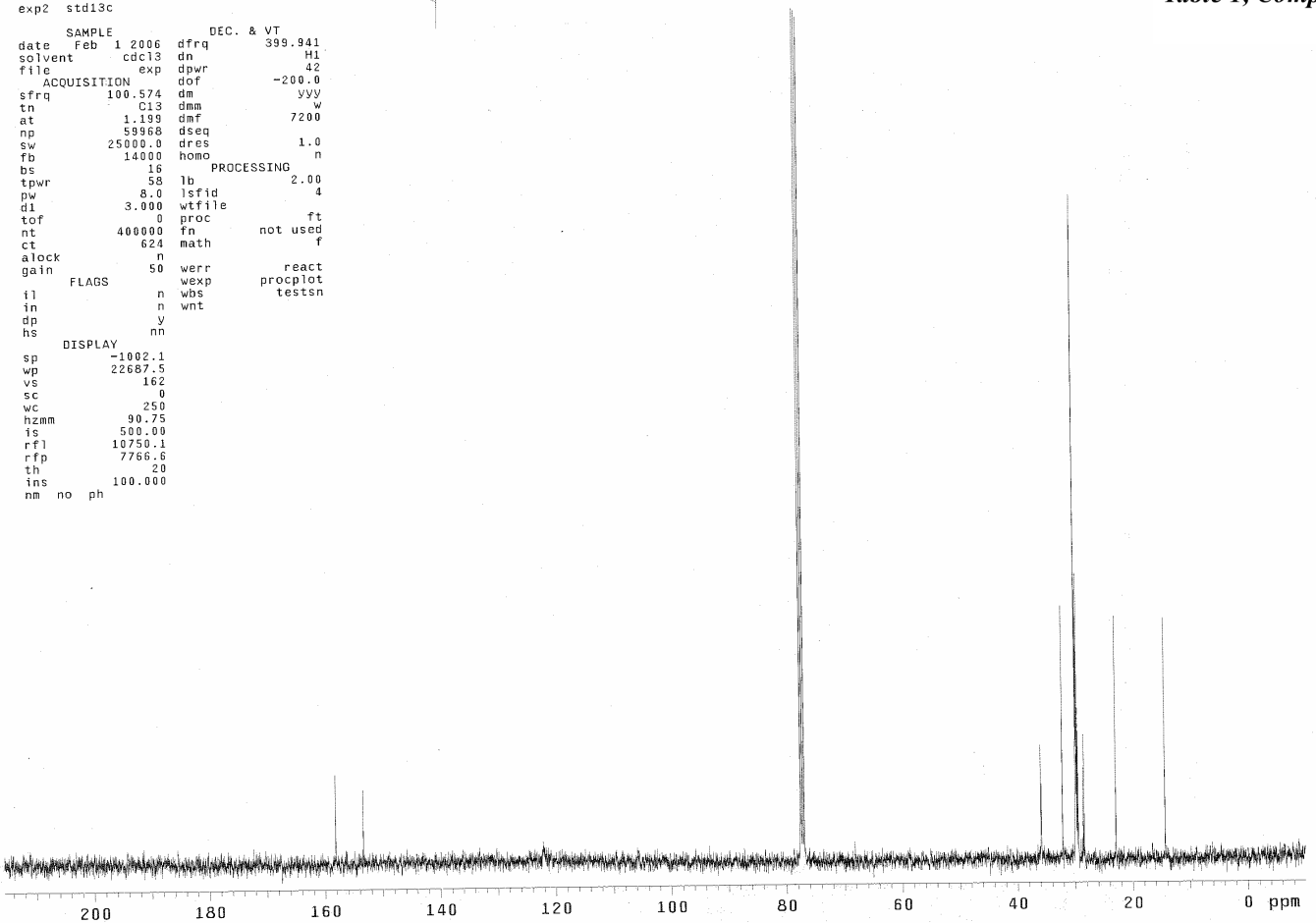
exp2 std13c

SAMPLE		DEC. & VT	
date	Feb 1 2006	dfrq	399.941
solvent	cdcl3	dn	H1
file	exp	dpwr	42
ACQUISITION		dof	-200.0
sfrq	100.574	dm	yyy
tn	C13	dmm	w
at	1.199	dmf	7200
np	59968	dseq	
sw	25000.0	dres	1.0
fb	14000	homo	n
bs	16	PROCESSING	
tpwr	58	lb	2.00
pw	8.0	lsfid	4
d1	3.000	wtfile	
tof	0	proc	ft
nt	400000	fn	not used
ct	624	math	f
alock	n		
gain	50	werr	react
FLAGS		wexp	procplot
il	n	wbs	testsn
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-1002.1		
wp	22687.5		
vs	162		
sc	0		
wc	250		
hzmm	90.75		
is	500.00		
rfl	10750.1		
rfp	7766.6		
th	20		
ins	100.000		
nm no ph			



5

Table 1, Compound 5



HY400NCOC15H31SerineSPh

Pulse Sequence: s2pul

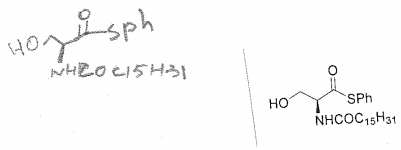
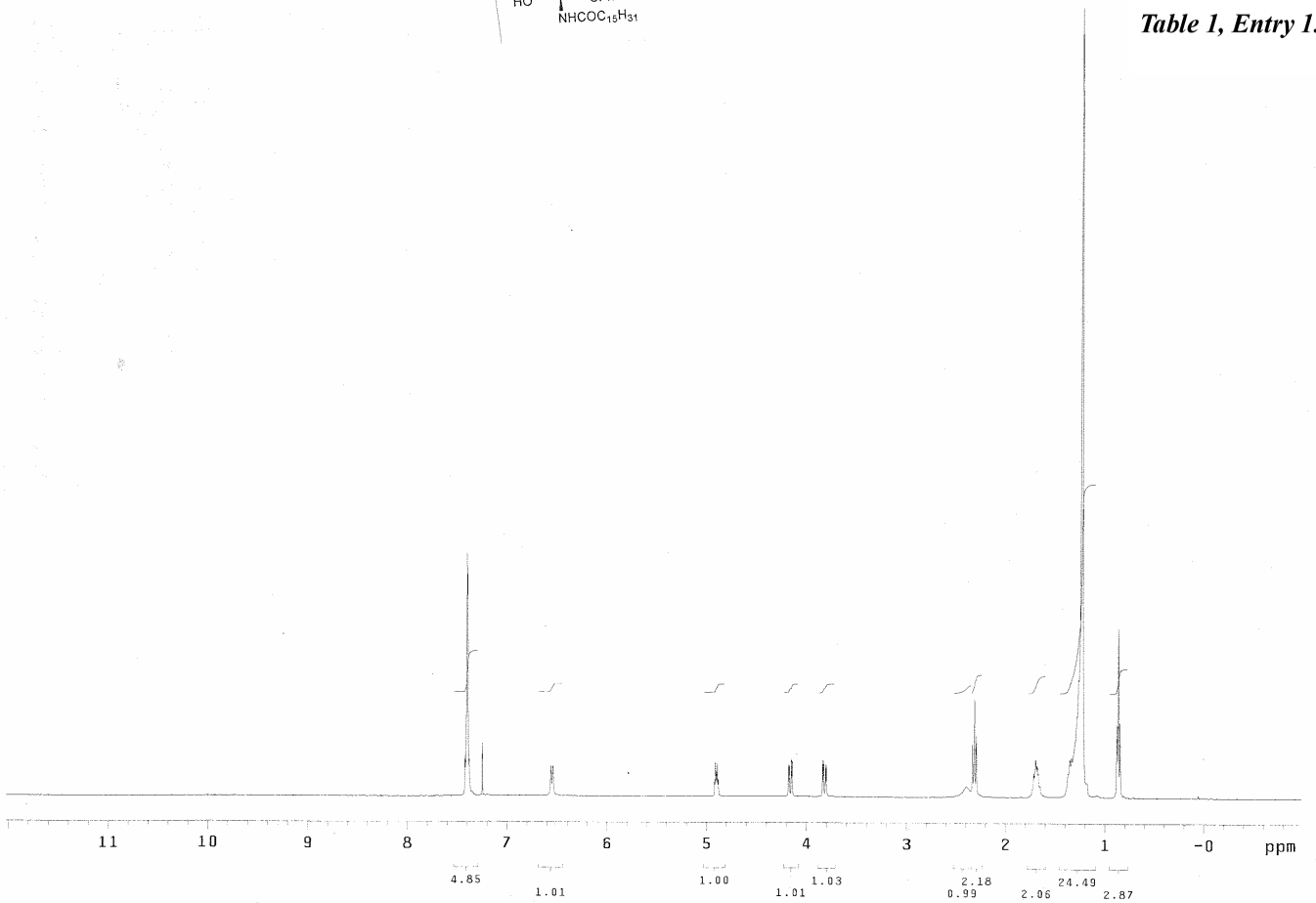


Table 1, Entry 1. R^1=H



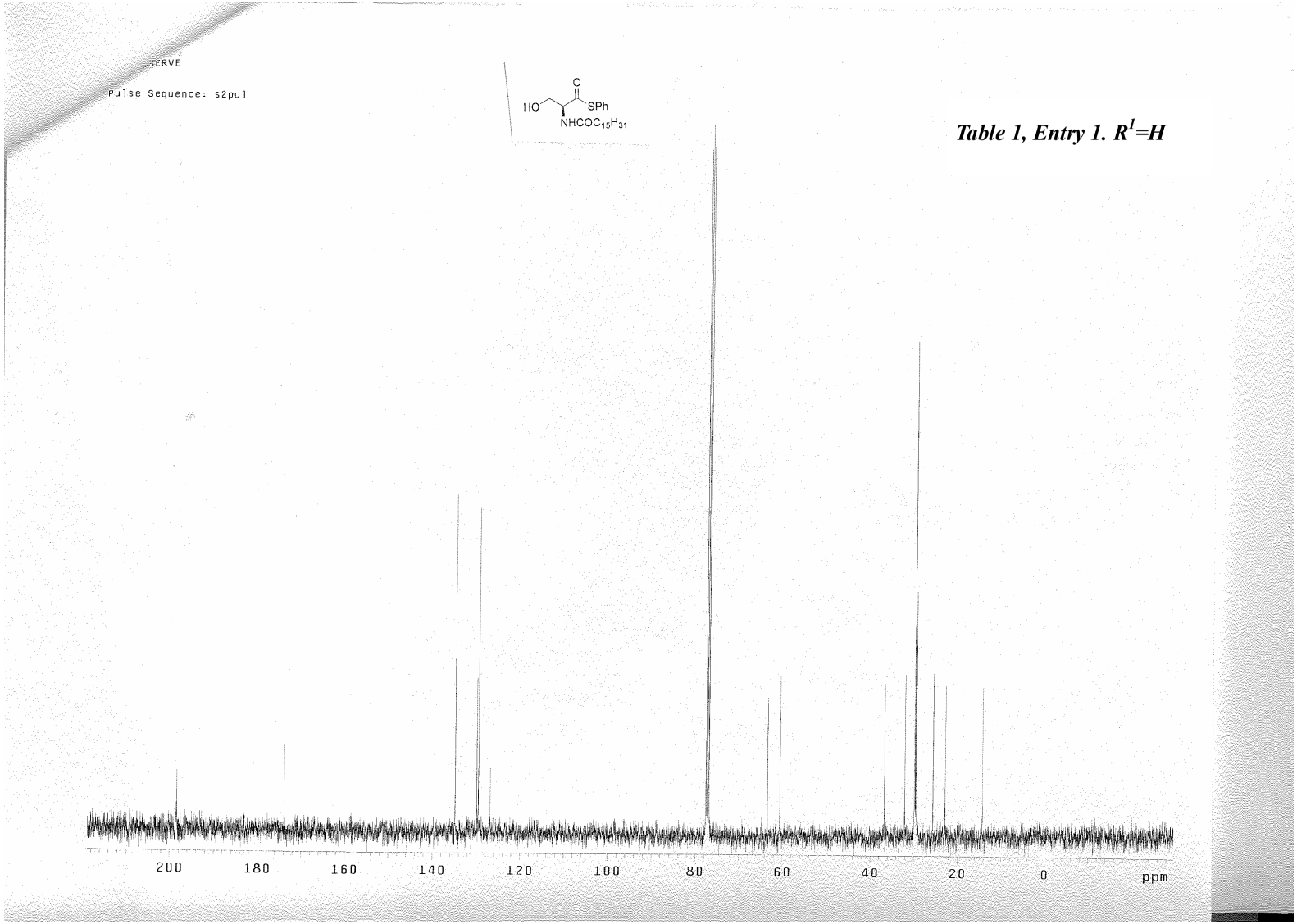


Table 1, Entry 1. R^1=H

HYN15Serinene13

File : xp
Sample id : tmpstudy

Pulse Sequence: s2pul

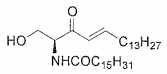
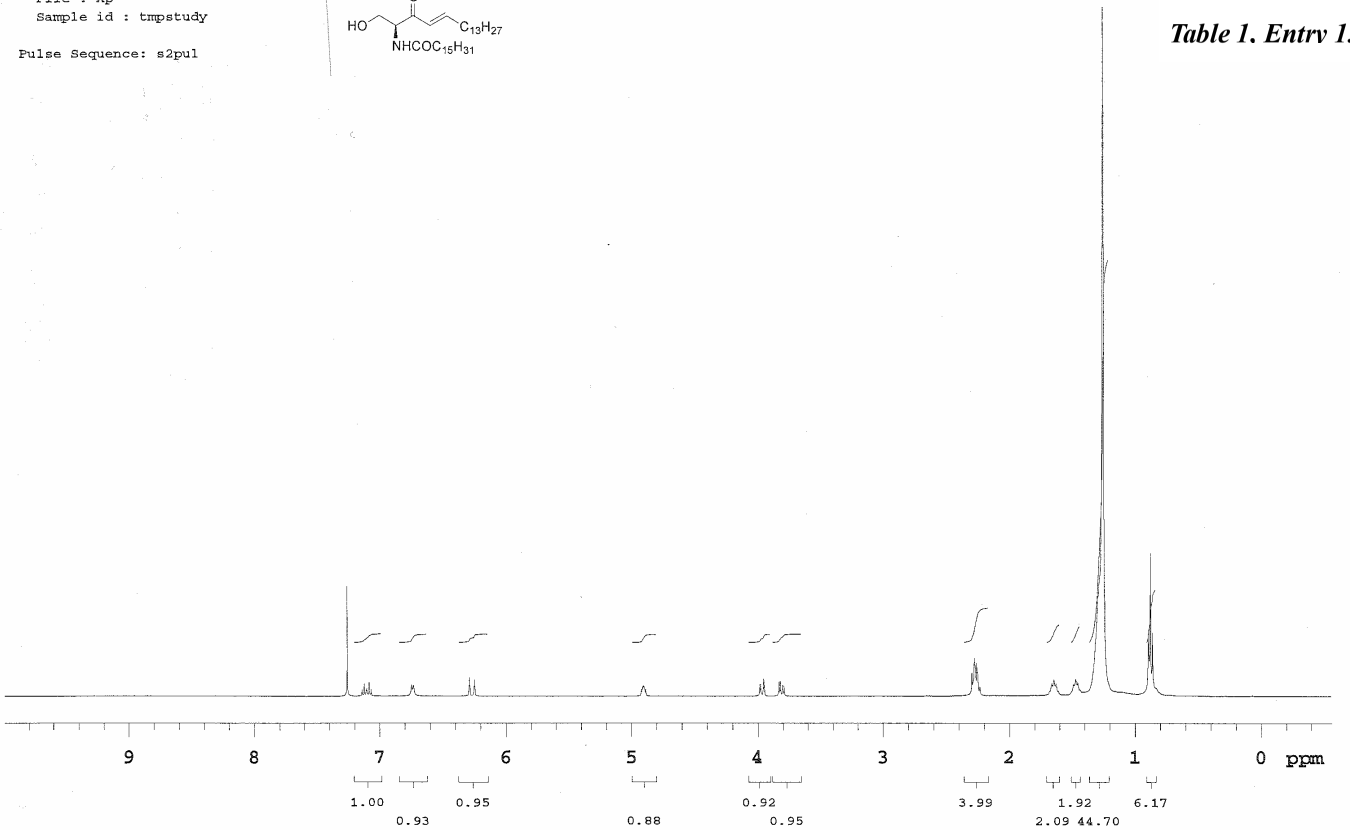


Table 1. Entry 1. R^1=H



13C OBSERVE

```
expl  std13c

      SAMPLE
date   Nov 20 2006
solvent       cdcl3
file            exp
   ACQUISITION
sfrq        100.574
tn              C13
at            1.199
np            59968
sw          25000.0
fb            14000
bs               16
tpwr             58
pw              8.0
dl            3.000
tof               0
nt            50000
ct             1632
alock              n
gain              50
      FLAGS
il                 n
in                 n
dp                 y
hs                nn
     DISPLAY
sp          -2983.8
wp          25000.0
vs              162
sc                0
wc              250
hzmm         100.00
is           500.00
rfl          2983.8
rfp               0
th               20
ins         100.000
nm  no  ph

      DEC. & VT
dfrq           39
dn
dpwr
dof
dm              yyy
dmm               w
dmf            7200
dseq
dres            1.0
homo              n
    PROCESSING
lb             2.00
lsfid             4
wtfile
proc             ft
fn        not used
math              f

werr          react
wexp       procplot
wbs          testsn
wnt
```

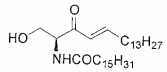


Table 1, Entry 1. R^1=H

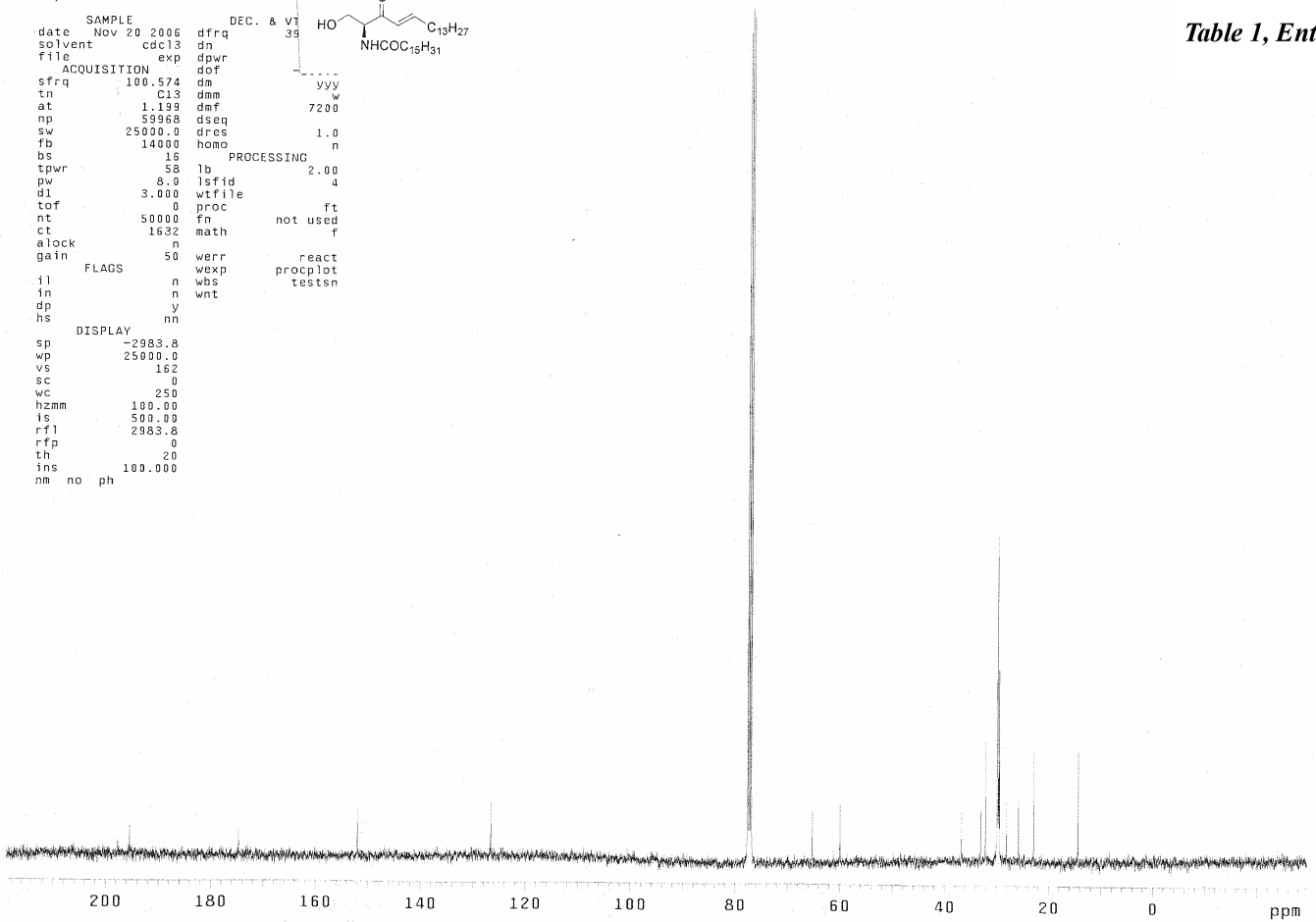
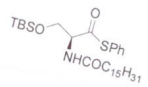


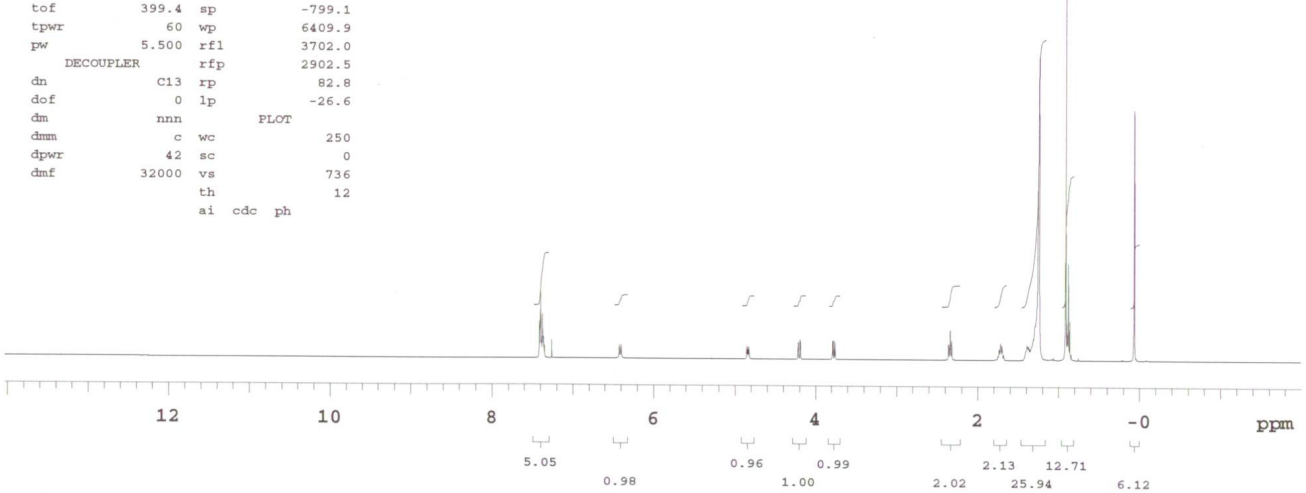
Table 1, Entry 1. R^1=TBDS

HYTBSN15SerineSPh

exp1 Proton

SAMPLE		SPECIAL	
date	Nov 19 2006	temp	25.0
solvent	cdcl3	gain	2
file	exp	spin	20
ACQUISITION		hst	0.008
sw	6410.3	pw90	11.000
at	2.300	alfa	6.600
np	29488	FLAGS	
fb	4000	il	n
bs	16	in	n
d1	1.500	dp	y
nt	5	hs	nn
ct	5	PROCESSING	
TRANSMITTER		lb	0.20
tn	H1	fn	32768
sfrq	399.790	DISPLAY	
tof	399.4	sp	-799.1
tpwr	60	wp	6409.9
pw	5.500	rf1	3702.0
DECOUPLER		rfp	2902.5
dn	C13	rp	82.8
dof	0	lp	-26.6
dm	nnn	PLOT	
dmm	c	wc	250
dpwr	42	sc	0
dmf	32000	vs	736
		th	12
		ai cdc ph	



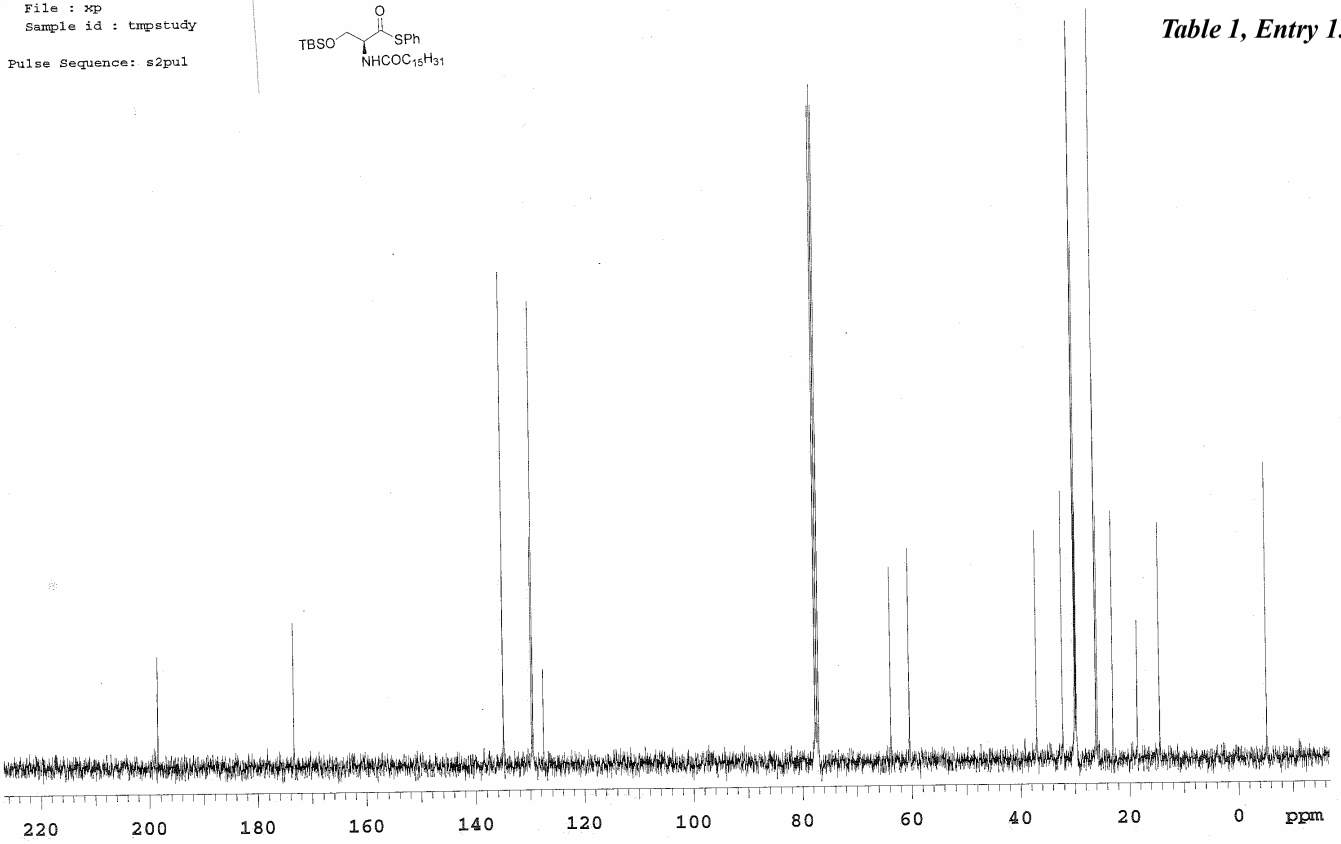


HYTBSN15SerineSPh

File : xp
Sample id : tmpstudy

Pulse Sequence: s2pul

Table 1, Entry 1. R^1=TBDMS

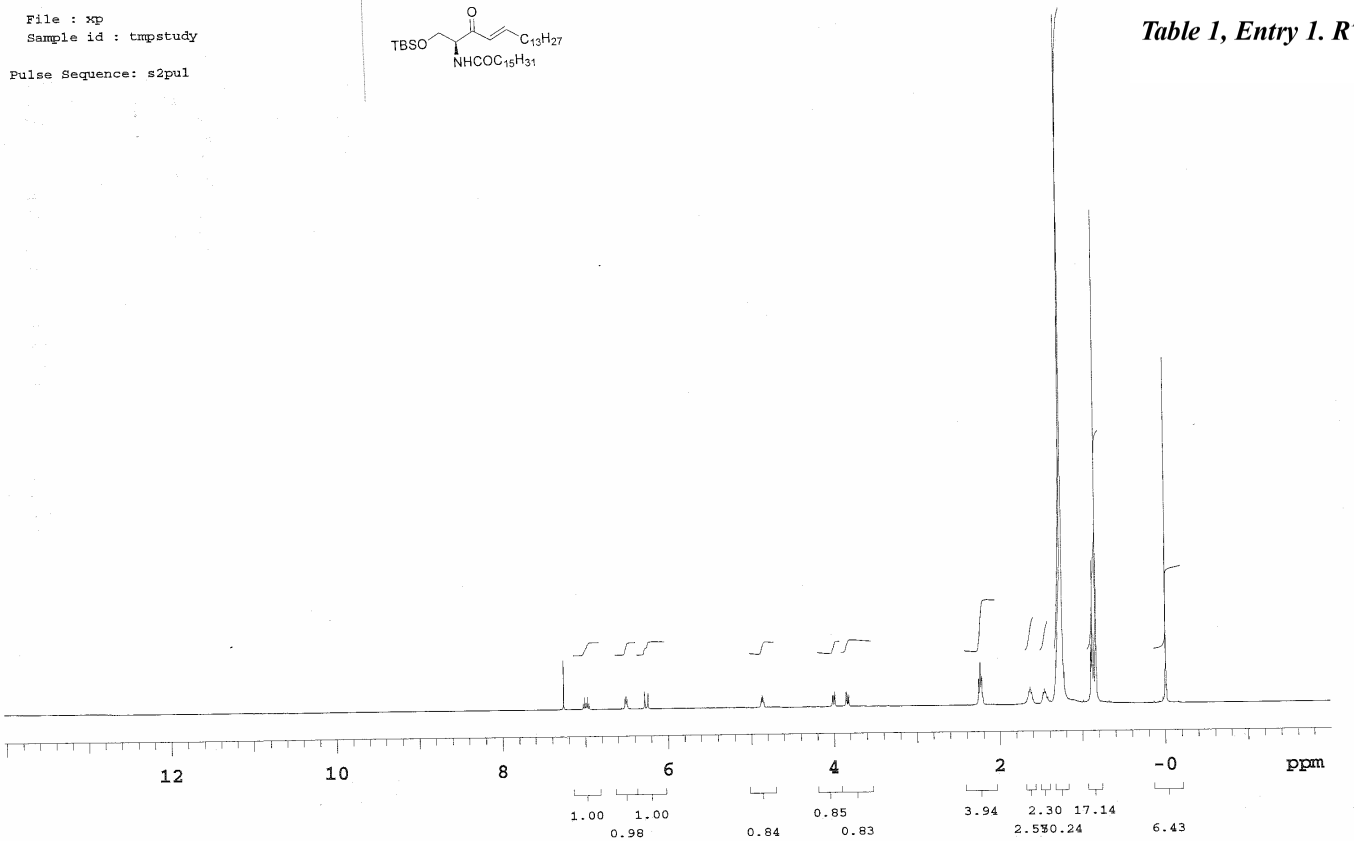


TBSN15SerineneC13H27

File : xp
Sample id : tmpstudy

Pulse Sequence: s2pul

Table 1, Entry 1. R^1=TBDMS



TBSN15SerineneC13H27

File : xp
Sample id : tmpstudy

Pulse Sequence: s2pul

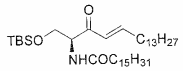
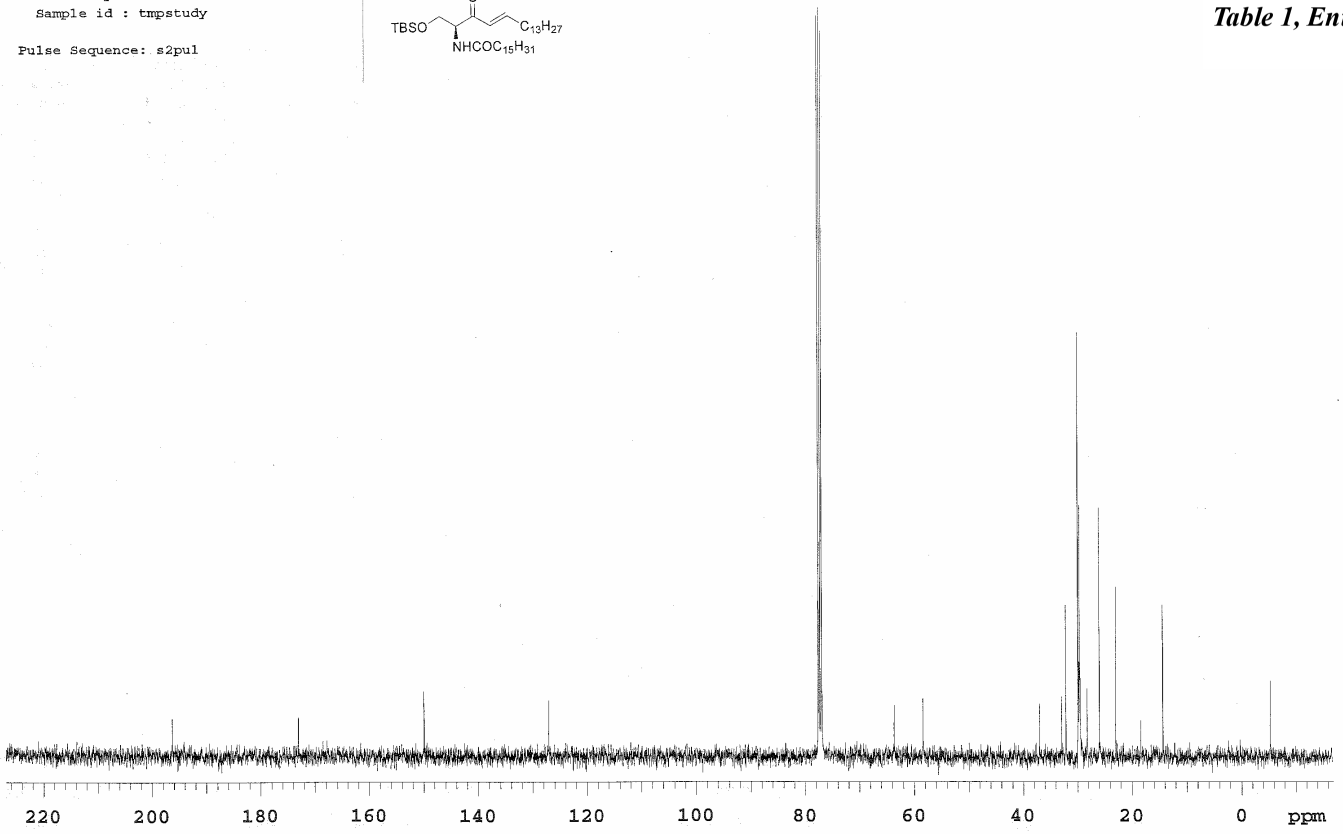


Table 1, Entry 1. R^1=TBDMS



HY-400-ZserSph

File : xp
Sample id : tmpstudy

Pulse Sequence: s2pul

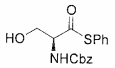
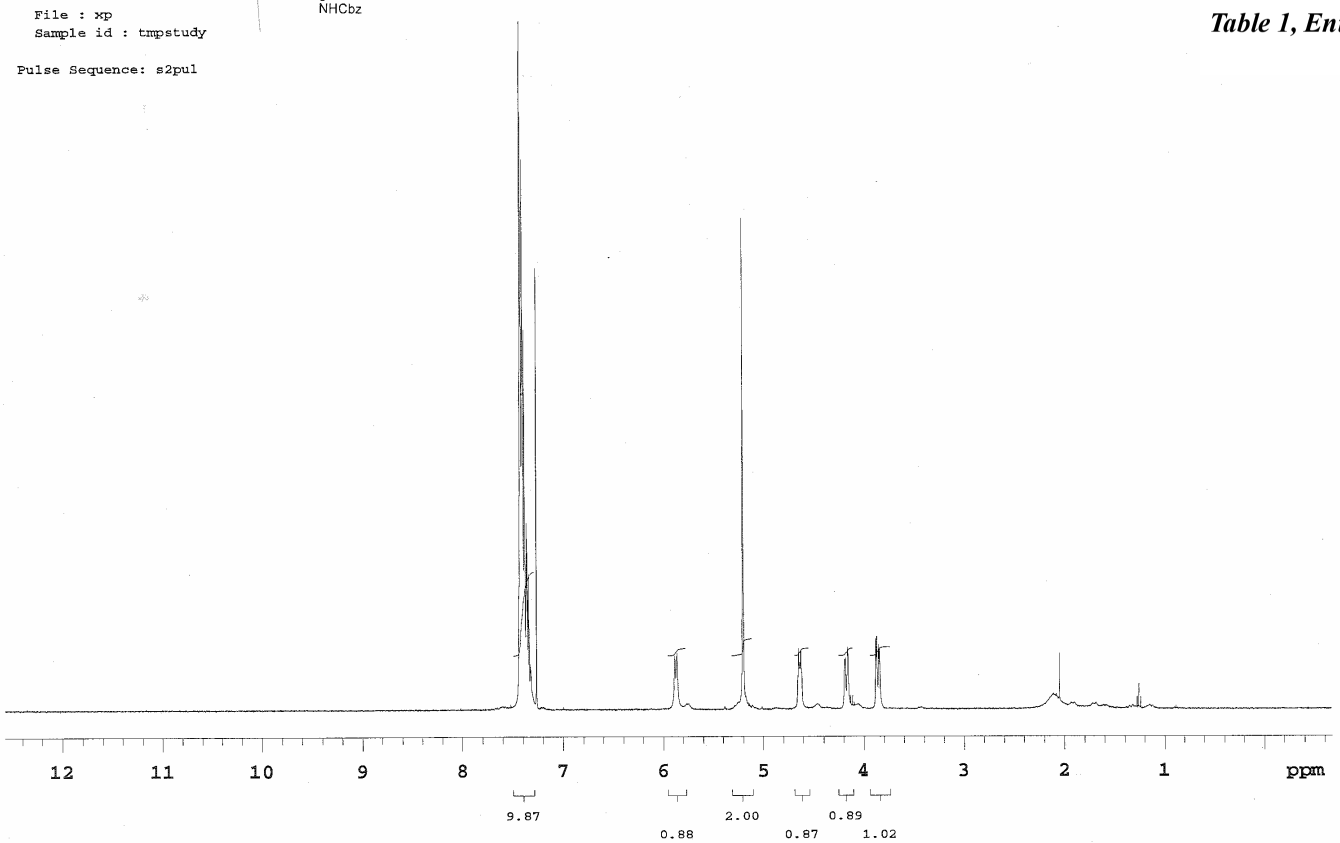


Table 1, Entry 2. R^1=H

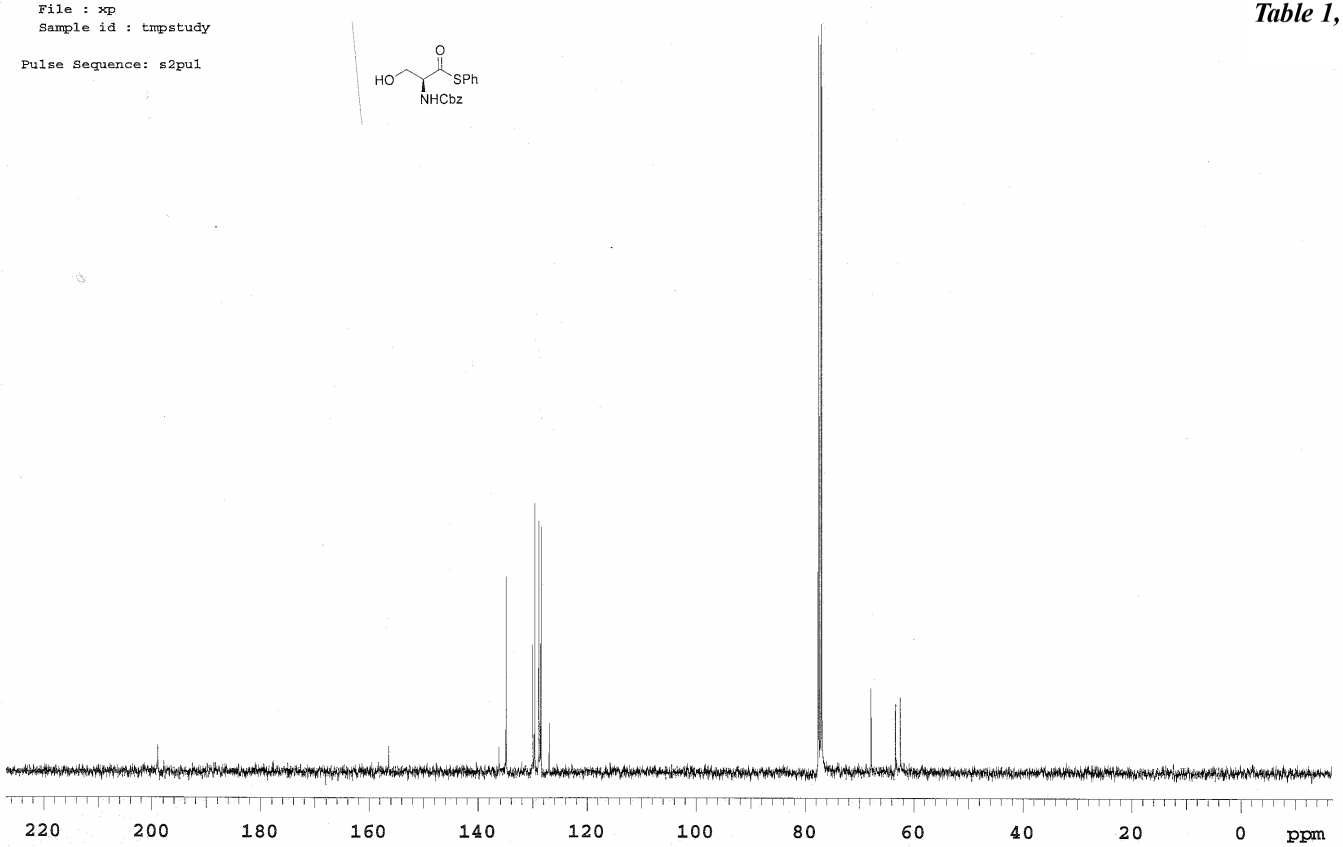


HY-400-ZserSph

File : xp
Sample id : tmpstudy

Pulse Sequence: s2pul

Table 1, Entry 2. R^1=H



HYCbzSerineneC13H27

File : xp
Sample id : tmpstudy

Pulse Sequence: s2pul

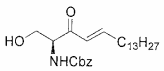


Table 1, Entry 2. R^1=H

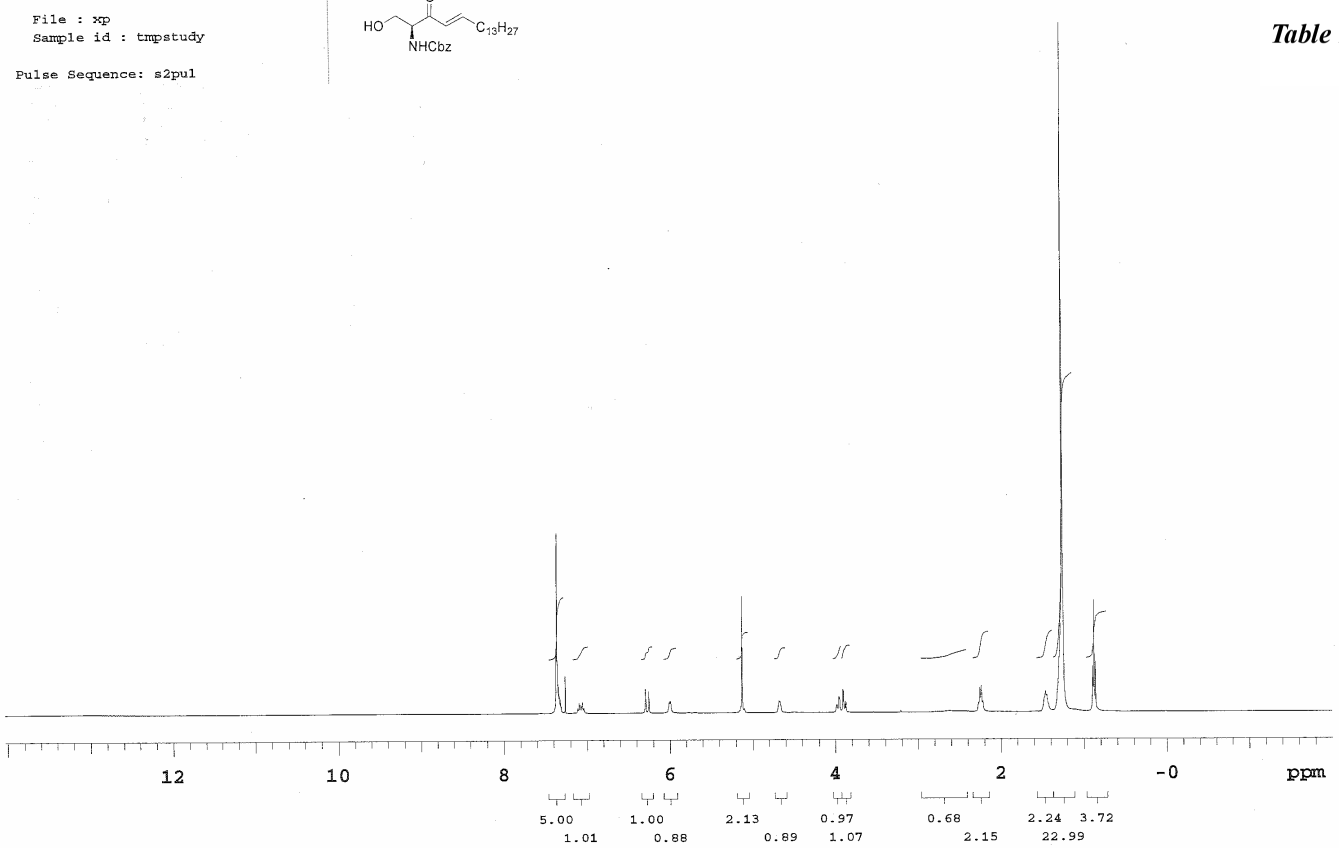
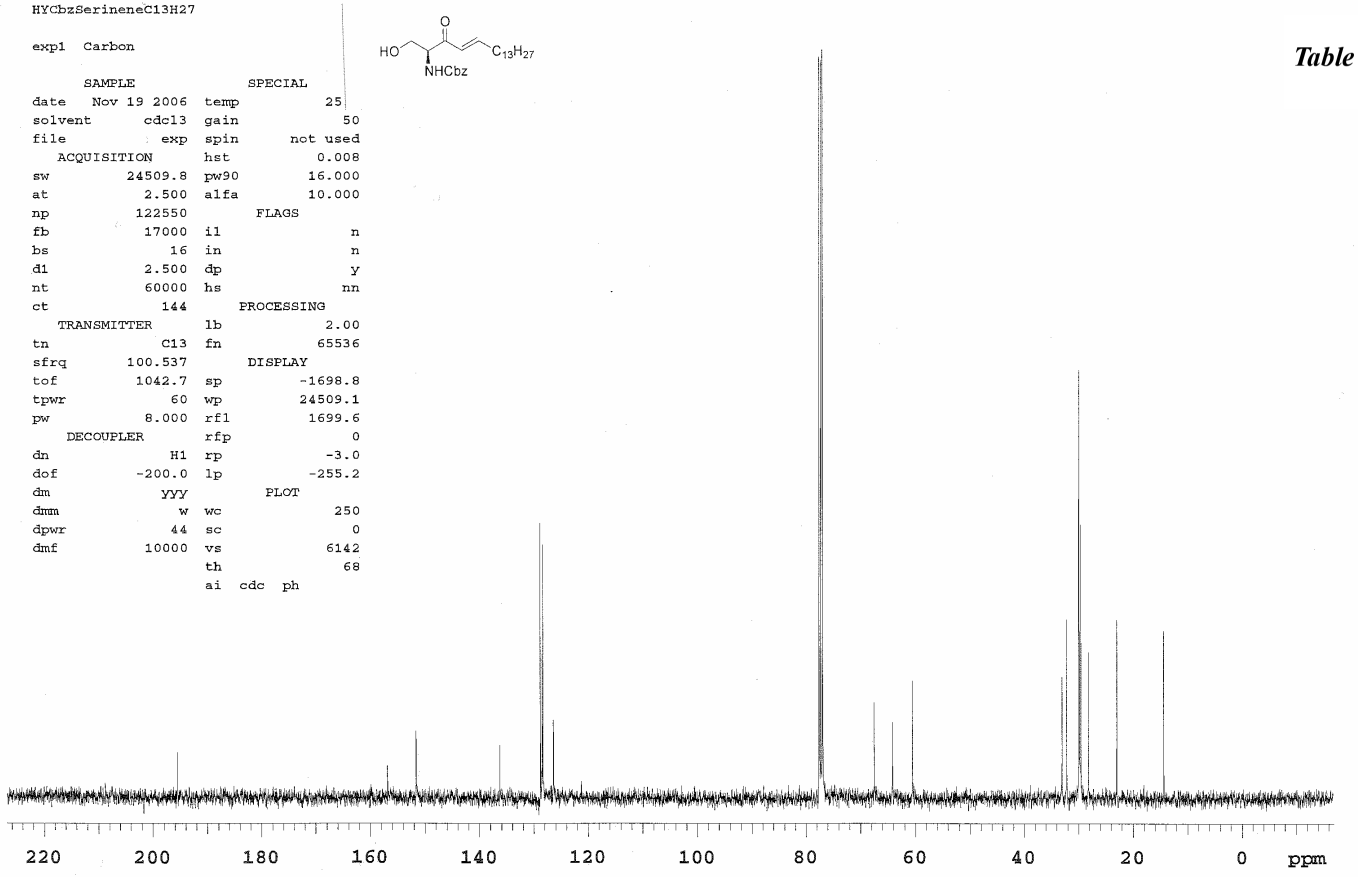


Table 1, Entry 2. R^1=H

HYCbzSerineneC13H27

exp1 Carbon

SAMPLE		SPECIAL	
date	Nov 19 2006	temp	25
solvent	cdcl3	gain	50
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	24509.8	pw90	16.000
at	2.500	alfa	10.000
np	122550	FLAGS	
fb	17000	il	n
bs	16	in	n
d1	2.500	dp	y
nt	60000	hs	nn
ct	144	PROCESSING	
TRANSMITTER		lb	2.00
tn	C13	fn	65536
sfrq	100.537	DISPLAY	
tof	1042.7	sp	-1698.8
tpwr	60	wp	24509.1
pw	8.000	rf1	1699.6
DECOUPLER		rfp	0
dn	H1	rp	-3.0
dof	-200.0	lp	-255.2
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	44	sc	0
dmf	10000	vs	6142
		th	68
		ai cdc ph	



HYTBSNHCbzSerineSPh

File : xp
Sample id : tmpstudy

Pulse Sequence: s2pul

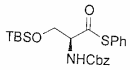
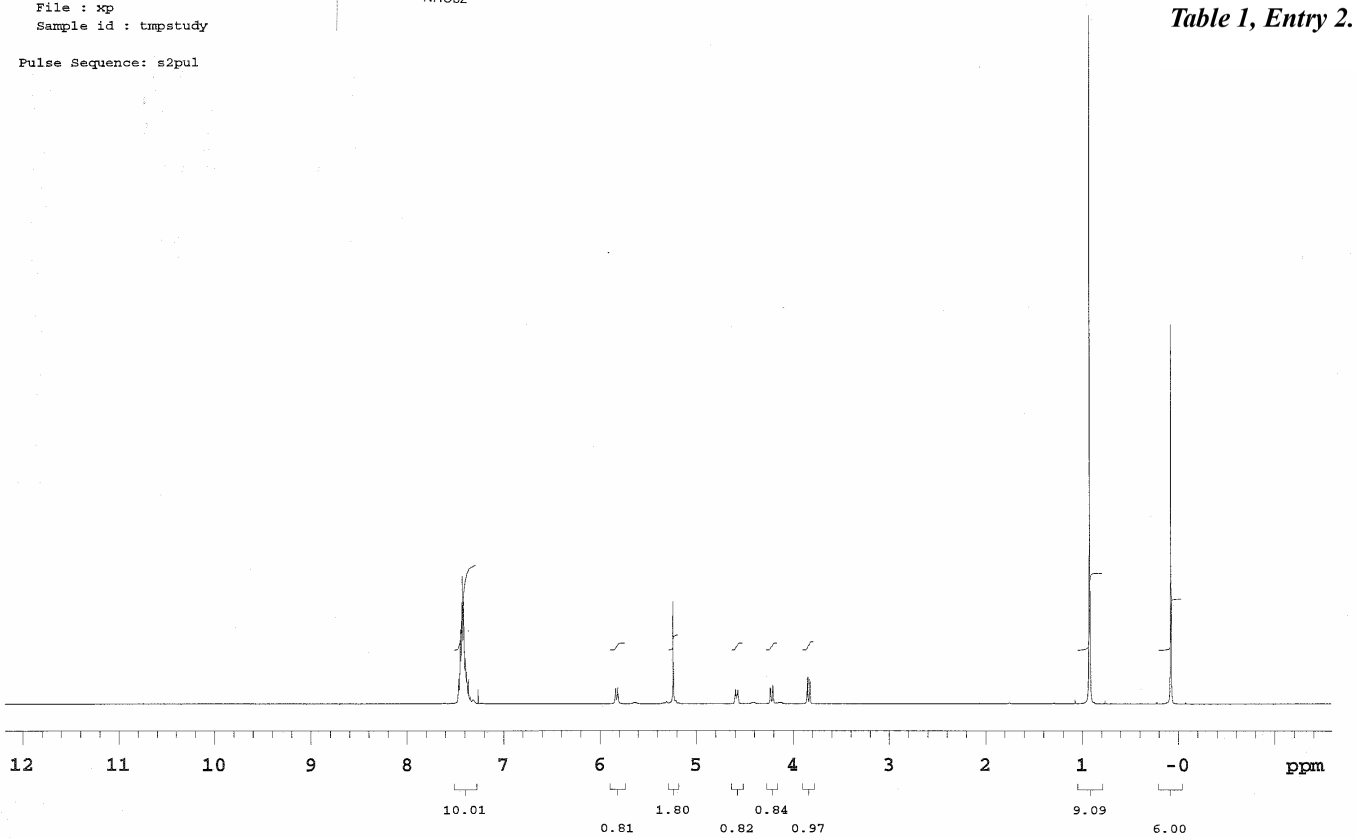


Table 1, Entry 2. R^1=TBDMS



HYTBSNBocSerineSPh

File : xp
Sample id : tmpstudy

Pulse Sequence: s2pul

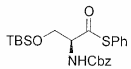
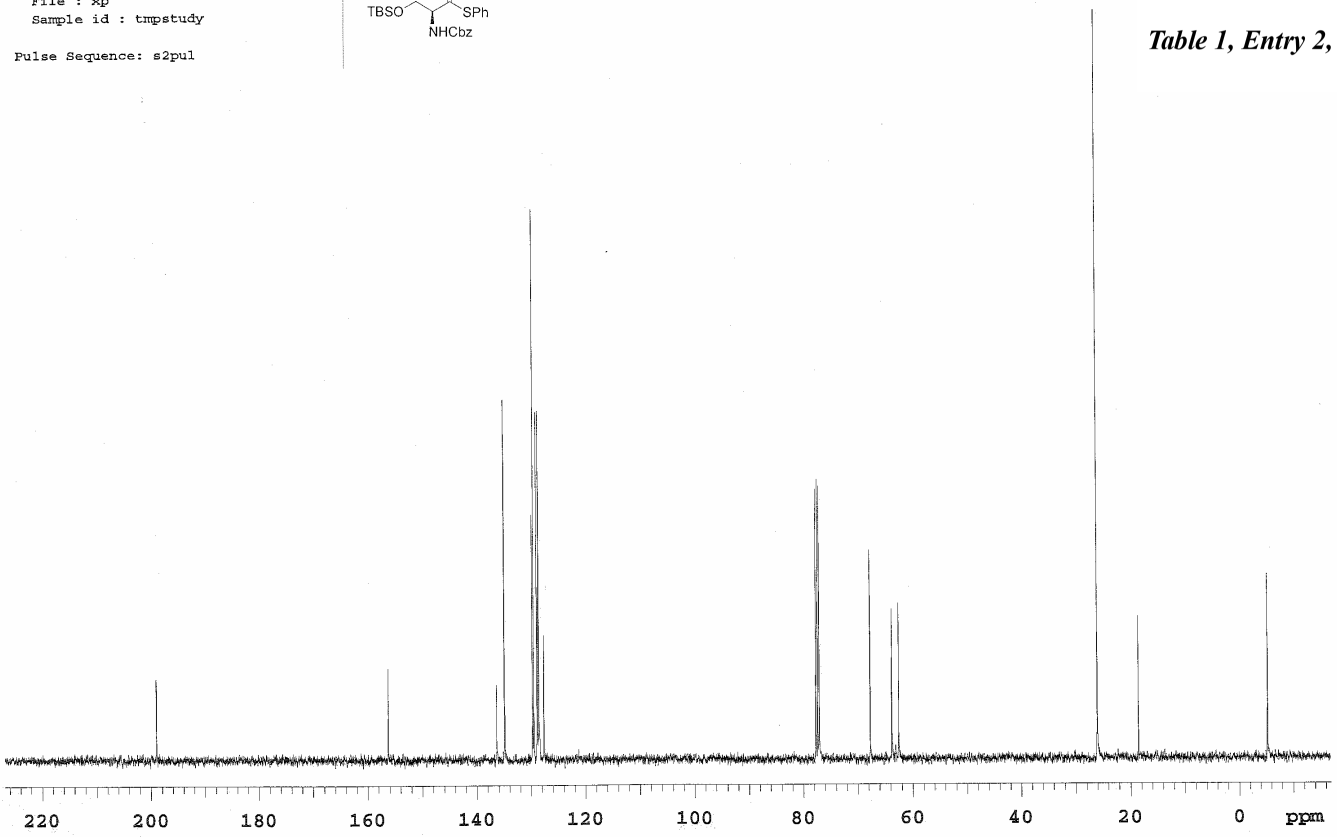


Table 1, Entry 2, R^1=TBDMS



HYTBSCbzSerinenec13h27

Pulse Sequence: s2pul

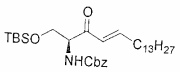
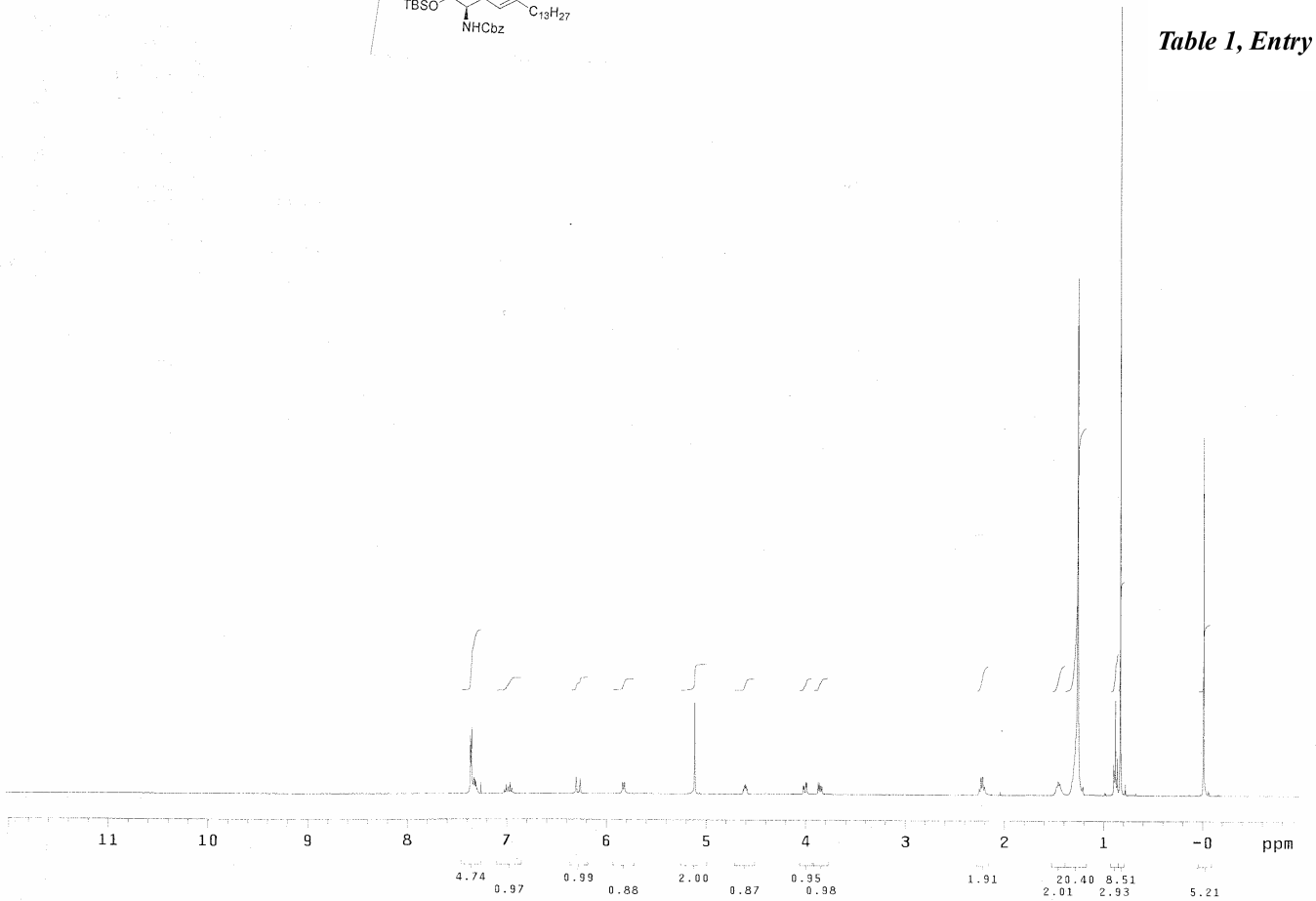


Table 1, Entry 2. R^1=TBDMS



HYTBSCbzSerinenec13h27

Pulse Sequence: s2pul

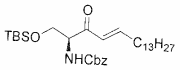


Table 1, Entry 2. R^1=TBDMS

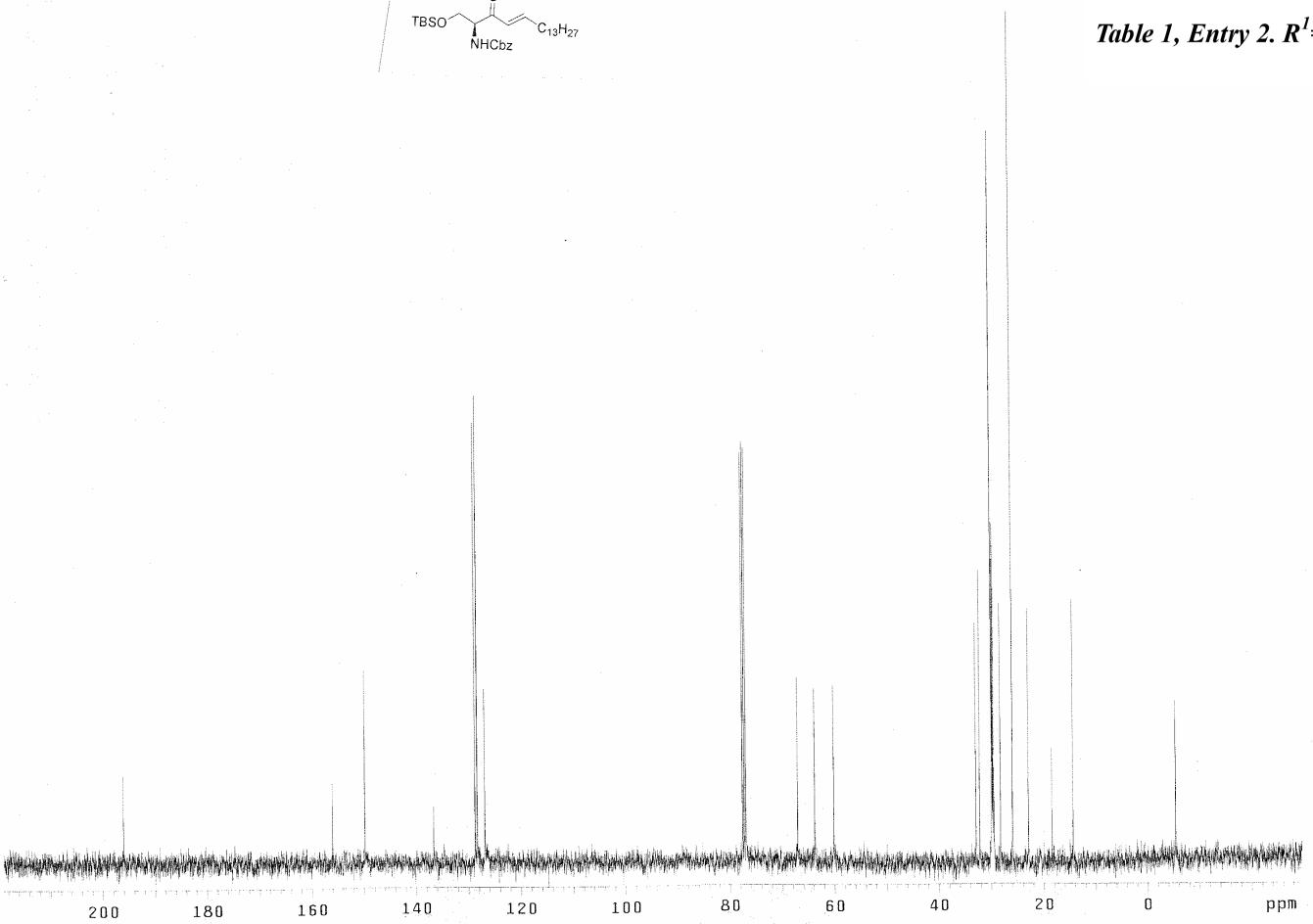
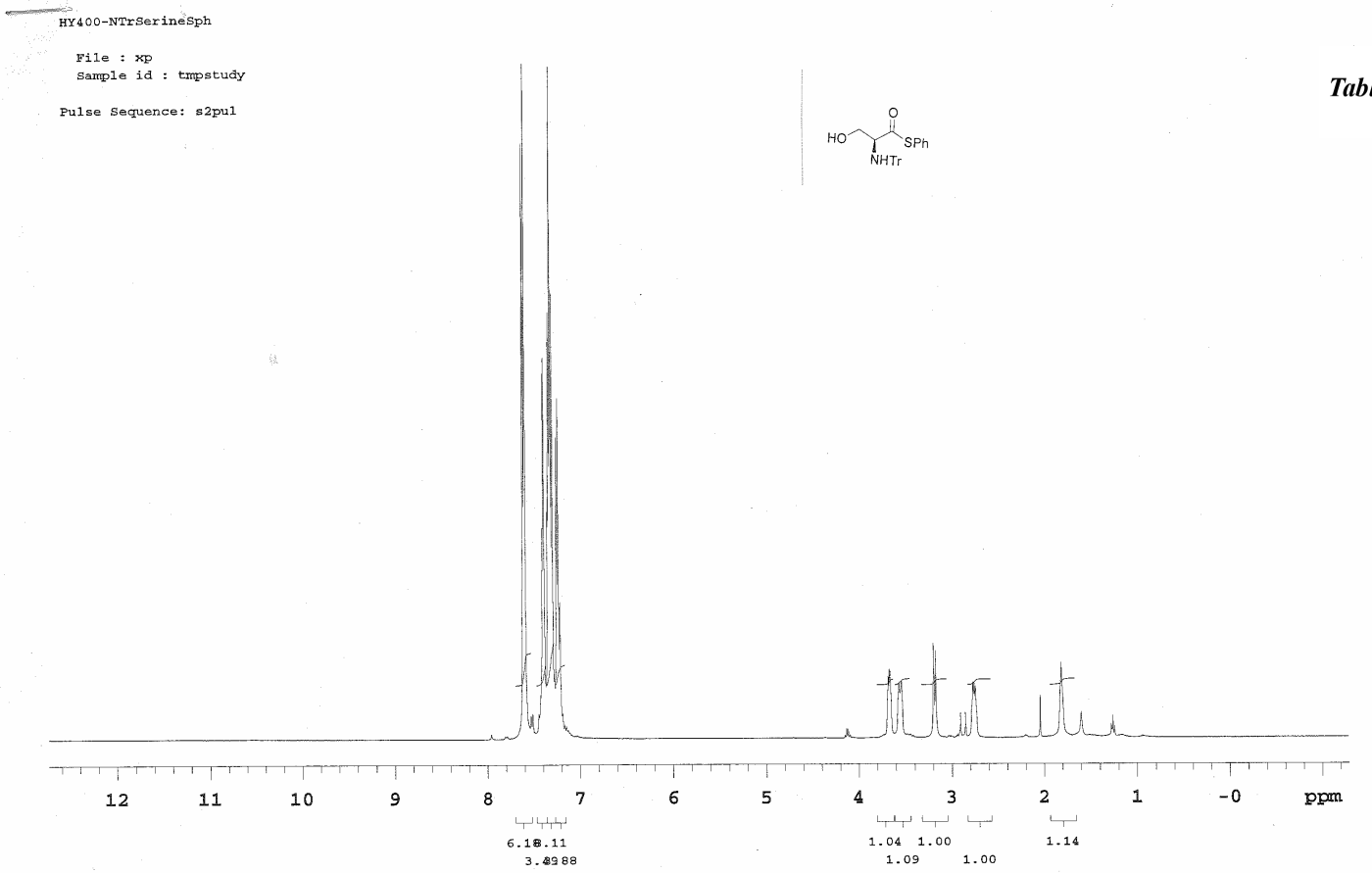


Table 1, Entry 3. R^1=H

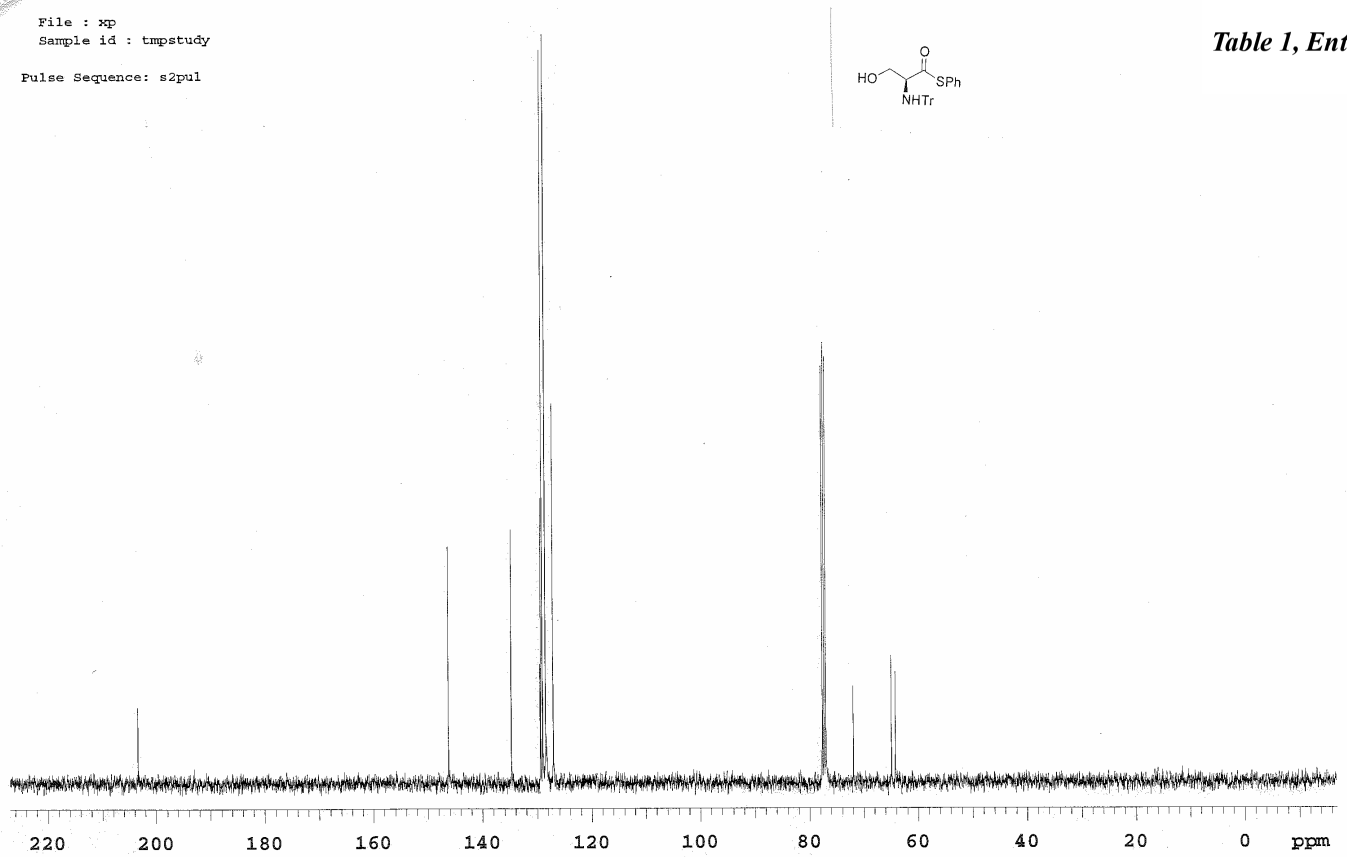


400-NTrSerineSph

File : xp
Sample id : tmpstudy

Pulse Sequence: s2pul

Table 1, Entry 3. R^1=H



STANDARD 1H OBSERVE

exp1 std1h

SAMPLE		DEC. & VT	
date	Nov 20 2006	dfrq	399.940
solvent	cdcl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	-630.7
sfrq	399.941	dm	nnn
tn	H1	dmm	c
at	2.202	dmf	200
np	22912	dseq	
sw	5203.3	dres	1.0
fb	3000	homo	n
bs	16	PROCESSING	
tpwr	58	lb	0.20
pw	8.0	wtfile	
d1	1.000	proc	ft
tof	207.0	fn	not used
nt	1	math	f
ct	1		
alock	n	werr	react
gain	not used	wexp	procplot
FLAGS		wbs	
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-395.0		
wp	5203.3		
vs	151		
sc	0		
wc	250		
hzmm	20.81		
is	176.12		
rfl	395.0		
rfp	0		
th	20		
ins	1.000		
nm cdc ph			

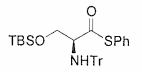


Table 1, Entry 3. R^1=TBDMS

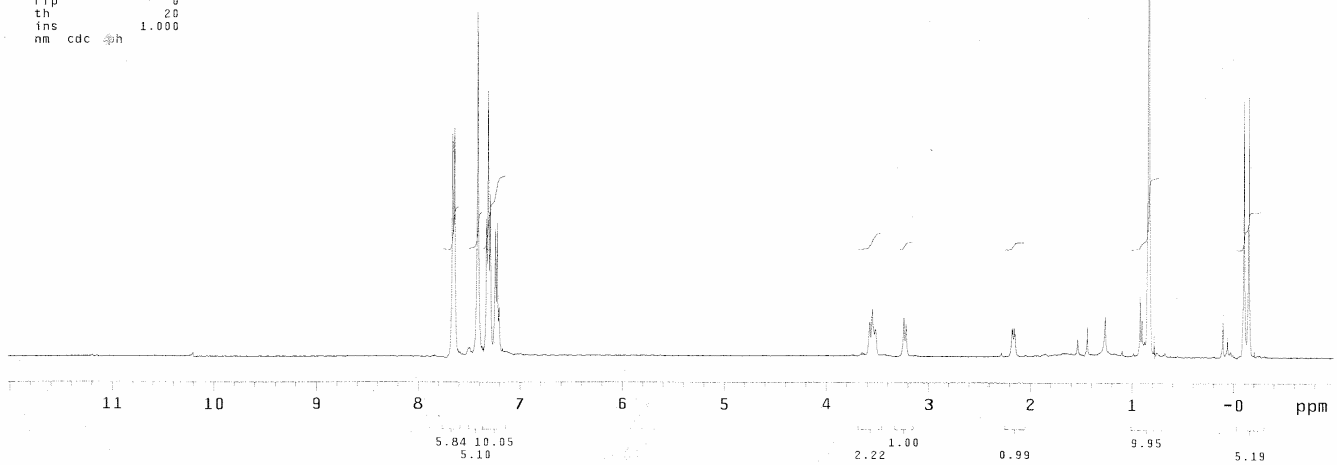
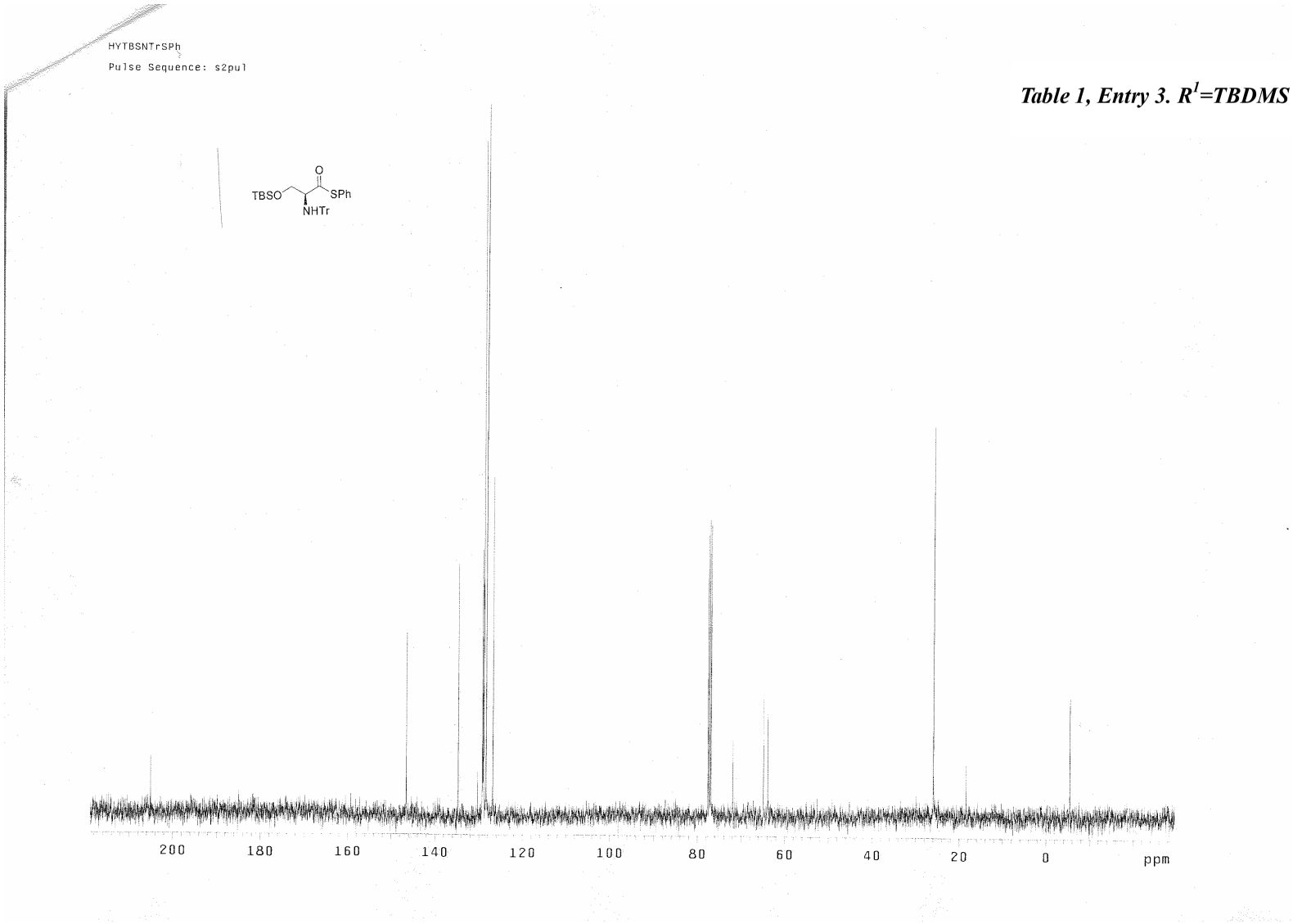


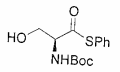
Table 1, Entry 3. R^1=TBDMS



STANDARD 1H OBSERVE

exp2 std1h

SAMPLE		DEC. & VT	
date	Feb 1 2006	dfrq	399.940
solvent	cdcl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	-630.7
sfrq	399.941	dm	nnn
tn	H1	dmm	c
at	2.202	dmf	200
np	22912	dseq	
sw	5203.3	dres	1.0
fb	3000	homo	n
bs	16	PROCESSING	
tpwr	58	lb	0.20
pw	8.0	wtfile	
d1	1.000	proc	ft
tof	207.0	fn	not used
nt	10	math	f
ct	10		
alock	n	werr	react
gain	not used	wexp	procplot
FLAGS		wbs	
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	296.8		
wp	3033.4		
vs	144		
sc	0		
wc	250		
hzmm	12.13		
is	106.92		
rfl	395.0		
rfp	0		
th	20		
ins	1.000		
nm cdc ph			



7

Table 1, Entry 4. R^1=H

Compound 7

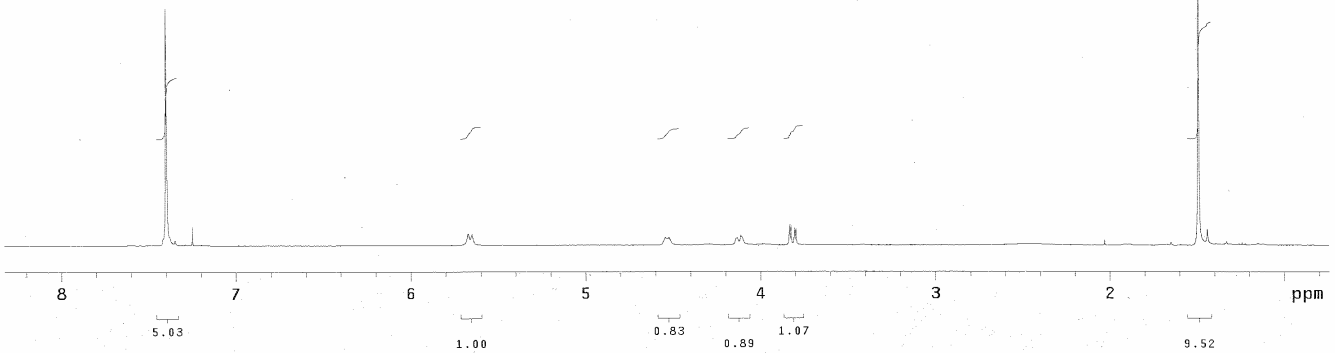


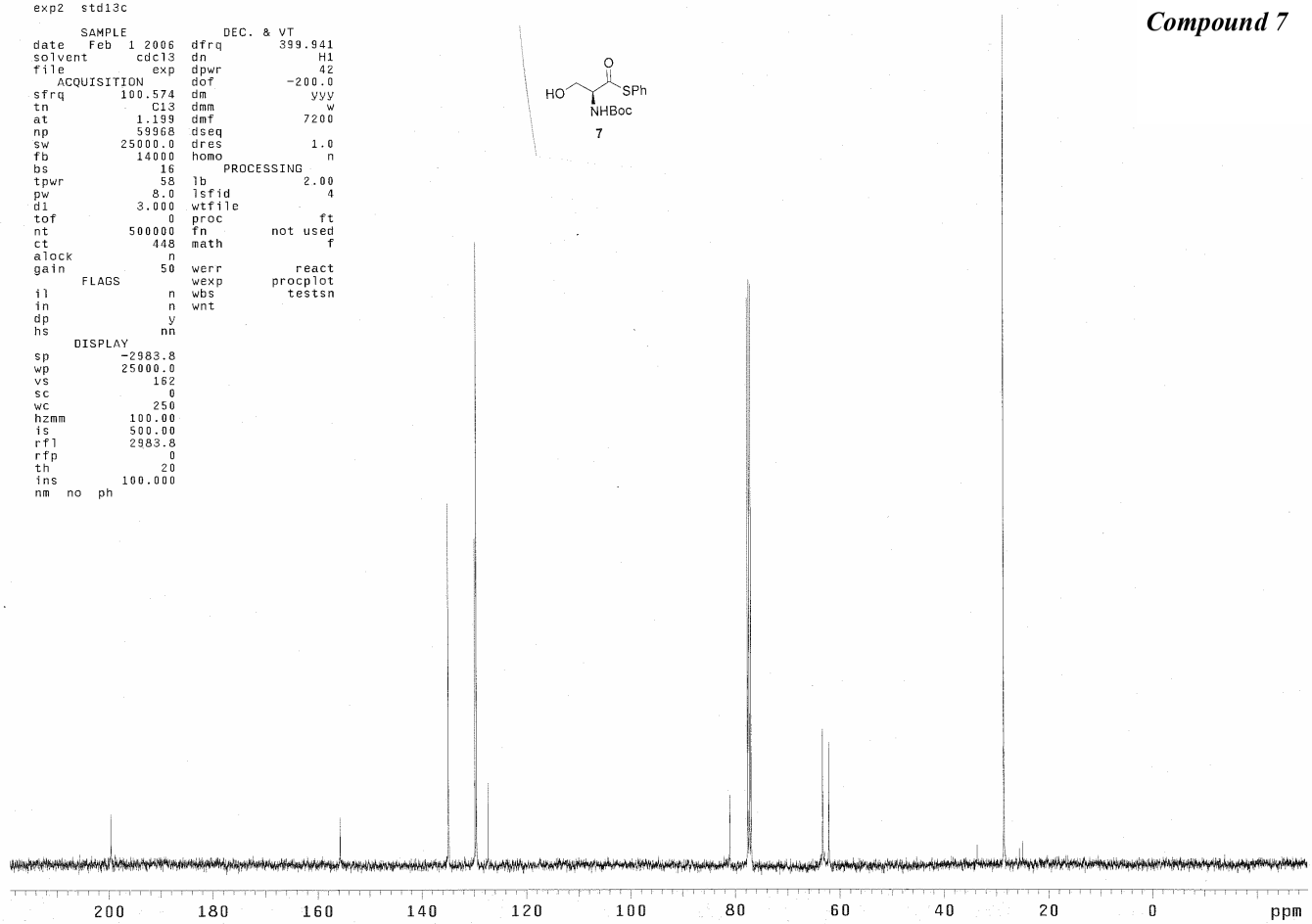
Table 1, Entry 4. R^1=H
Compound 7

13C OBSERVE

exp2 std13c

SAMPLE		DEC. & VT	
date	Feb 1 2006	dfrq	399.941
solvent	cdcl3	dn	H1
file	exp	dpwr	42
ACQUISITION		dof	-200.0
sfrq	100.574	dm	yyy
tn	C13	dmm	w
at	1.199	dmf	7200
np	59968	dseq	
sw	25000.0	dres	1.0
fb	14000	homo	n
bs	16	PROCESSING	
tpwr	58	lb	2.00
pw	8.0	lsfid	4
d1	3.000	wtfile	
tof	0	proc	ft
nt	500000	fn	not used
ct	448	math	f
alock	n		
gain	50	werr	react
FLAGS		wexp	procplot
il	n	wbs	testsn
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-2983.8		
wp	25000.0		
vs	162		
sc	0		
wc	250		
hzmm	100.00		
is	500.00		
rfl	2983.8		
rfp	0		
th	20		
ins	100.000		
nm no ph			

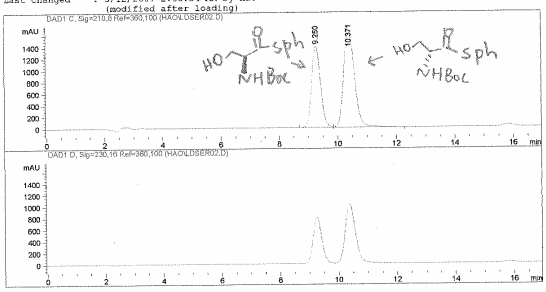
7



Injection Date : 2/25/2006 3:02:20 AM
Sample Name : LDmixture
Acq. Operator : HaoYang

Seq. Line : 2
Location : Vial 2
Inj : 1
Inj Volume : 1 µl

Acq. Method : C:\HPCHEM\1\METHODS\YANGHAO6.M
Last changed : 2/25/2006 2:24:57 AM by HaoYang
Analysis Method : C:\HPCHEM\1\METHODS\YANGHAO9.M
Last changed : 3/12/2007 2:35:54 AM by hao
(modified after loading)



Area Percent Report

Sorted By : Retention Time
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Sig	Type	Area [mAU*s]	Height [mAU]	Area %
1	9.250	1	BV	2.99001e4	1399.27161	40.5982
2	10.371	1	VB	4.37508e4	1692.85803	59.4046

Totals : 7.36488e4 3090.12964

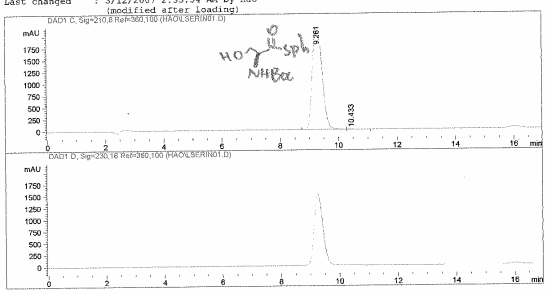
Results obtained with enhanced integrator!

*** End of Report ***

Injection Date : 2/25/2006 2:36:16 AM
Sample Name : Lserinesph
Acq. Operator : HaoYang

Seq. Line : 1
Location : Vial 1
Inj : 1
Inj Volume : 1 µl

Acq. Method : C:\HPCHEM\1\METHODS\YANGHAO6.M
Last changed : 2/25/2006 2:24:57 AM by HaoYang
Analysis Method : C:\HPCHEM\1\METHODS\YANGHAO9.M
Last changed : 3/12/2007 2:35:54 AM by hao
(modified after loading)



Area Percent Report

Sorted By : Retention Time
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Sig	Type	Area [mAU*s]	Height [mAU]	Area %
1	9.261	1	BB	5.04642e4	2086.64185	99.7945
2	10.433	1	BP	105.90656	4.44473	0.2094

Totals : 5.05682e4 2089.08657

Results obtained with enhanced integrator!

*** End of Report ***

Table 1, Entry . R^1=H Compound 7

R564//600MHz//3-17-06

exp1 s2pul

SAMPLE		DEC. & VT	
date	Mar 17 2006	dfrq	599.738
solvent	cdcl3	dn	H1
file	exp	dpwr	0
ACQUISITION		dof	0
sfrq	599.738	dm	nnn
tn	H1	dmm	w
at	1.598	dmf	200
np	24948	dseq	
sw	7804.1	dres	1.0
fb	4000	homo	n
bs	16	PROCESSING	
tpwr	56	lb	0.20
pw	5.0	wtfile	
d1	1.000	proc	ft
tof	285.3	fn	not used
nt	1	math	f
ct	1		
alock	n	werr	react
gain	not used	wexp	procplot
FLAGS		wbs	testsn
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-196.1		
wp	5271.3		
vs	151		
sc	0		
wc	250		
hzmm	21.09		
is	172.93		
rfl	608.1		
rfp	0		
th	8		
ins	1.000		
nm cdc ph			

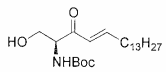
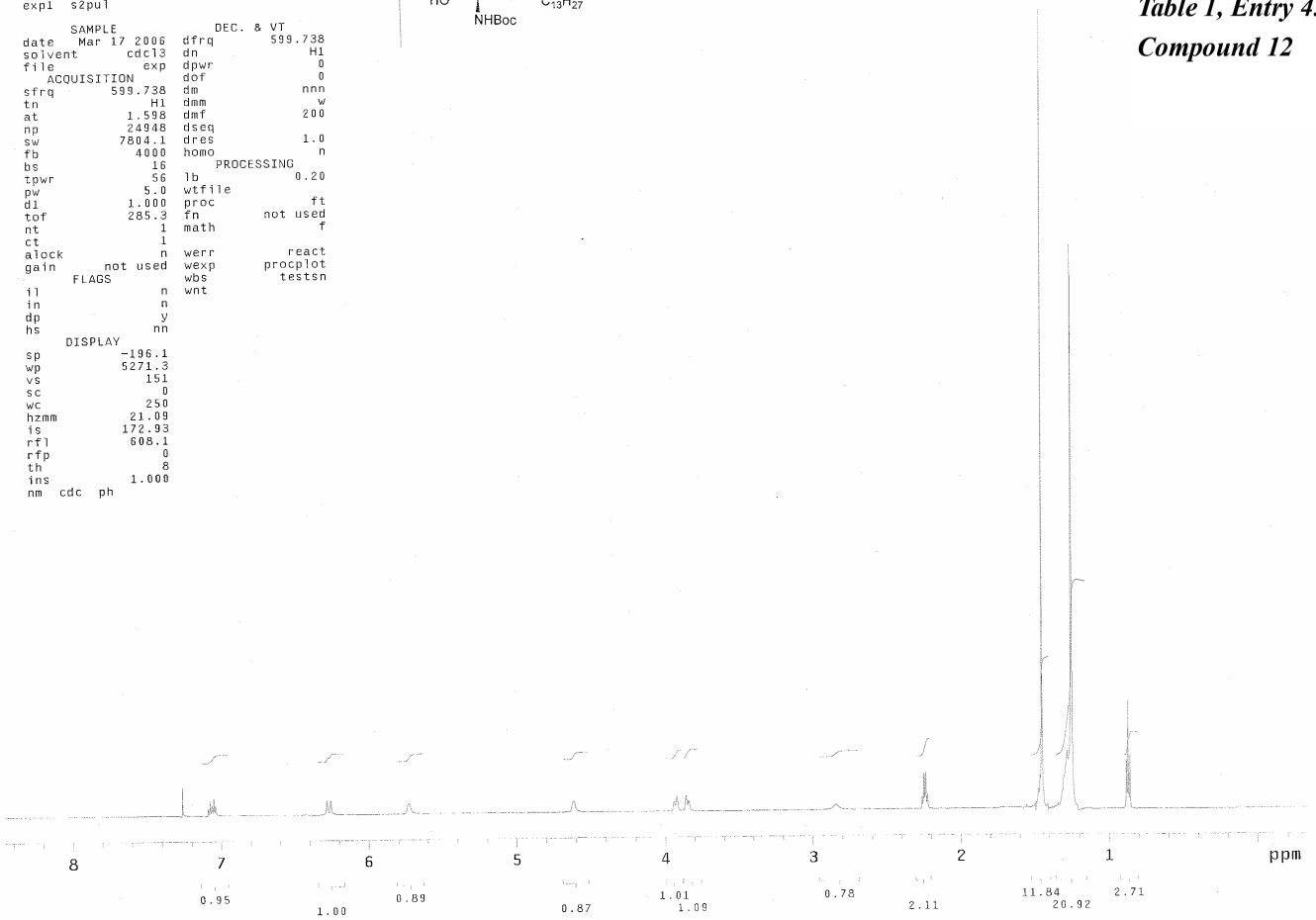


Table 1, Entry 4. R^1=H

Compound 12



13C SENSITIVITY
ASTM SAMPLE

exp1 s2pul

SAMPLE		DEC. & VT	
date	Mar 17 2006	dn	H1
solvent	cdcl3	dof	-400.0
file	exp	dm	yyy
ACQUISITION		dmm	w
sfrq	150.818	dmf	8000
tn	C13	dpwr	38
at	0.443	PROCESSING	
np	30108	lb	2.00
sw	33984.7	fn	not used
fb	19000	math	f
bs	16		
pw	5.0	werr	react
pw	5.0	wexp	procplot
tpwr	56	wbs	testsn
d1	2.000	wnt	
tof	1400.8	DISPLAY	
nt	2e+06	sp	-2096.2
ct	576	wp	33984.7
alock	n	vs	1303
gain	not used	sc	0
FLAGS		wc	250
il	n	hzmm	135.94
in	n	is	500.00
dp	y	rfl	2096.2
hs	nn	rfp	0
		th	23
		ins	1.000
		ai ph	

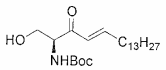


Table 1, Entry . R^1=H
Compound 12

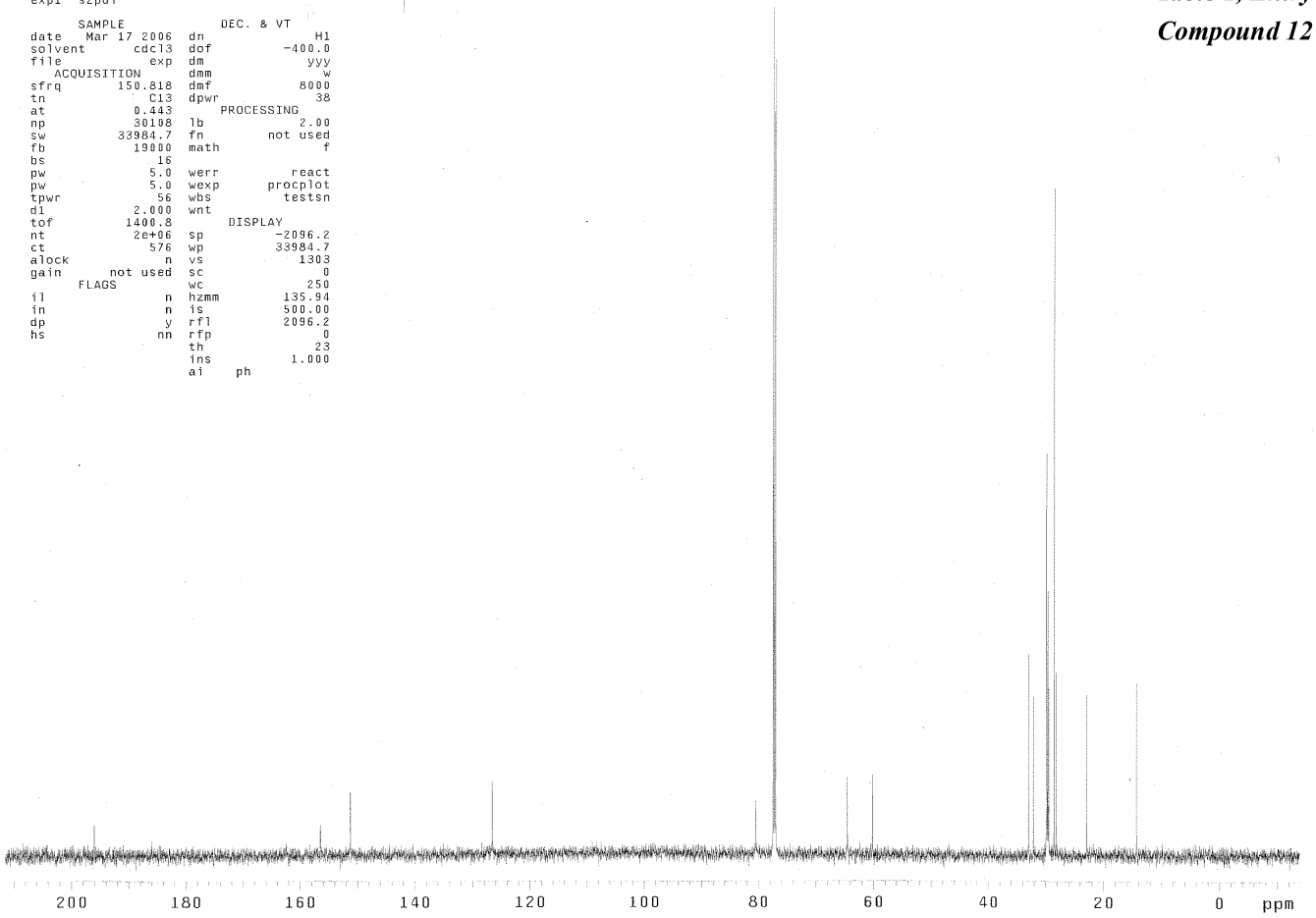


Table 1, Entry 4. R^1=TBDMS

Compound 8

STANDARD PROTON PARAMETERS

exp1 s2pul

SAMPLE		DEC. & VT	
date	Feb 27 2006	dfrq	599.608
solvent	cdcl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	599.607	dm	nnn
tn	H1	dmm	c
at	2.002	dmf	200
np	32832	dseq	
sw	8201.8	dres	1.0
fb	4600	homo	n
bs	16	PROCESSING	
tpwr	56	lb	0.20
pw	3.0	wtfile	
d1	1.000	proc	ft
tof	-657.6	fn	not used
nt	50	math	f
ct	50		
alock	y	werr	react
gain	6	wexp	procplot
FLAGS		wbs	
il	n	wnt	wft
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-313.6		
wp	5329.0		
vs	151		
sc	35		
wc	215		
hzmm	24.75		
is	272.32		
rfl	603.9		
rfp	0		
th	3		
ins	1.000		
nm cdc ph			

8

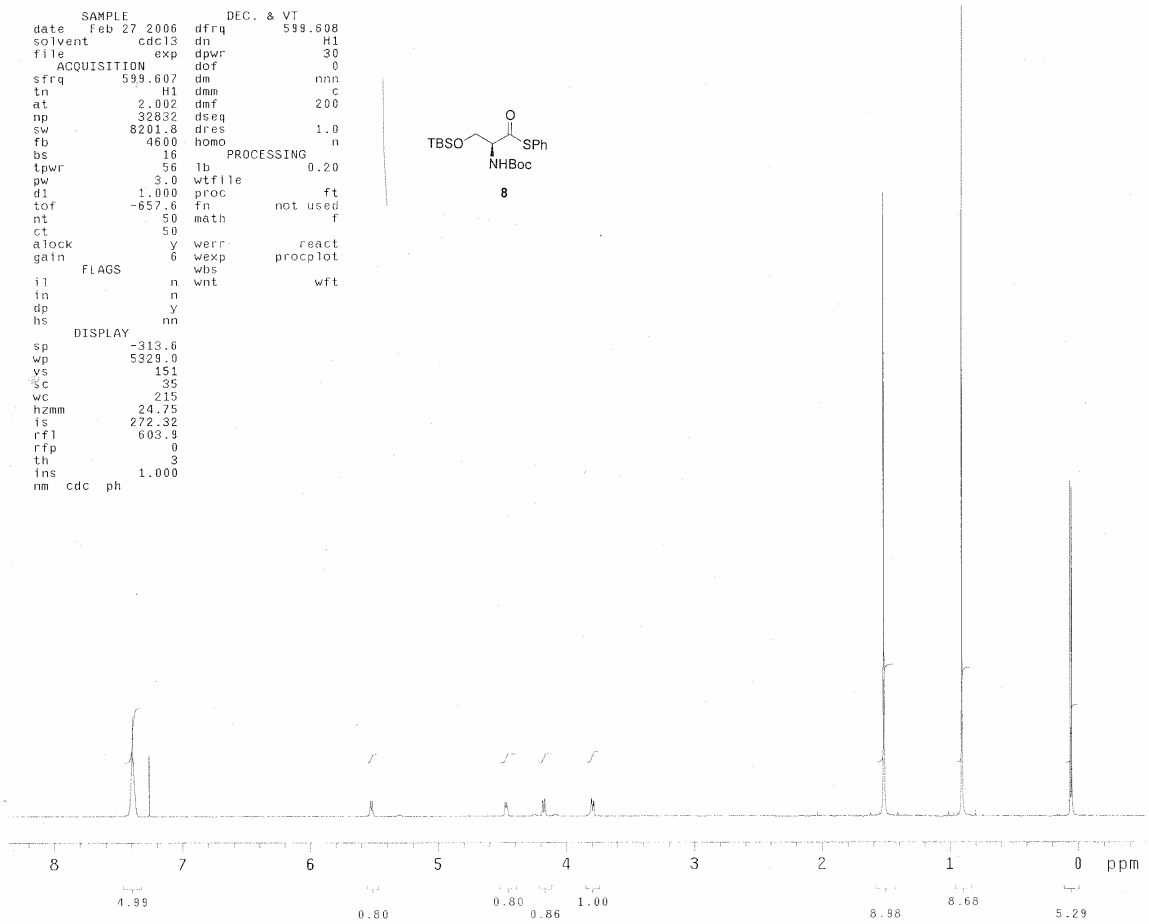


Table 1, Entry 4. R^1=TBDMS

Compound 8

STANDARD CARBON PARAMETERS

exp1 s2pul

SAMPLE		DEC. & VT	
date	Feb 27 2006	dfrq	599.606
solvent	cdcl3	dn	H1
file	exp	dpwr	35
ACQUISITION		dof	-1500.0
sfrq	150.785	dm	yyy
tn	C13	dmm	w
at	1.299	dmf	8000
np	90944	dseq	
sw	34995.6	dres	1.0
fb	19200	homo	n
bs	32	PROCESSING	
tpwr	60	lb	3.00
pw	10.0	wtfile	
d1	2.000	proc	ft
tof	902.2	fn	not used
nt	200000	math	f
ct	96		
alock	y	werr	react
gain	40	wexp	procplot
FLAGS		wbs	testsn
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-2826.3		
wp	34995.6		
vs	2167		
sc	0		
wc	250		
hzmm	139.98		
is	500.00		
rfl	14470.3		
rfp	11644.0		
th	2		
ins	100.000		
ai cdc ph			

8

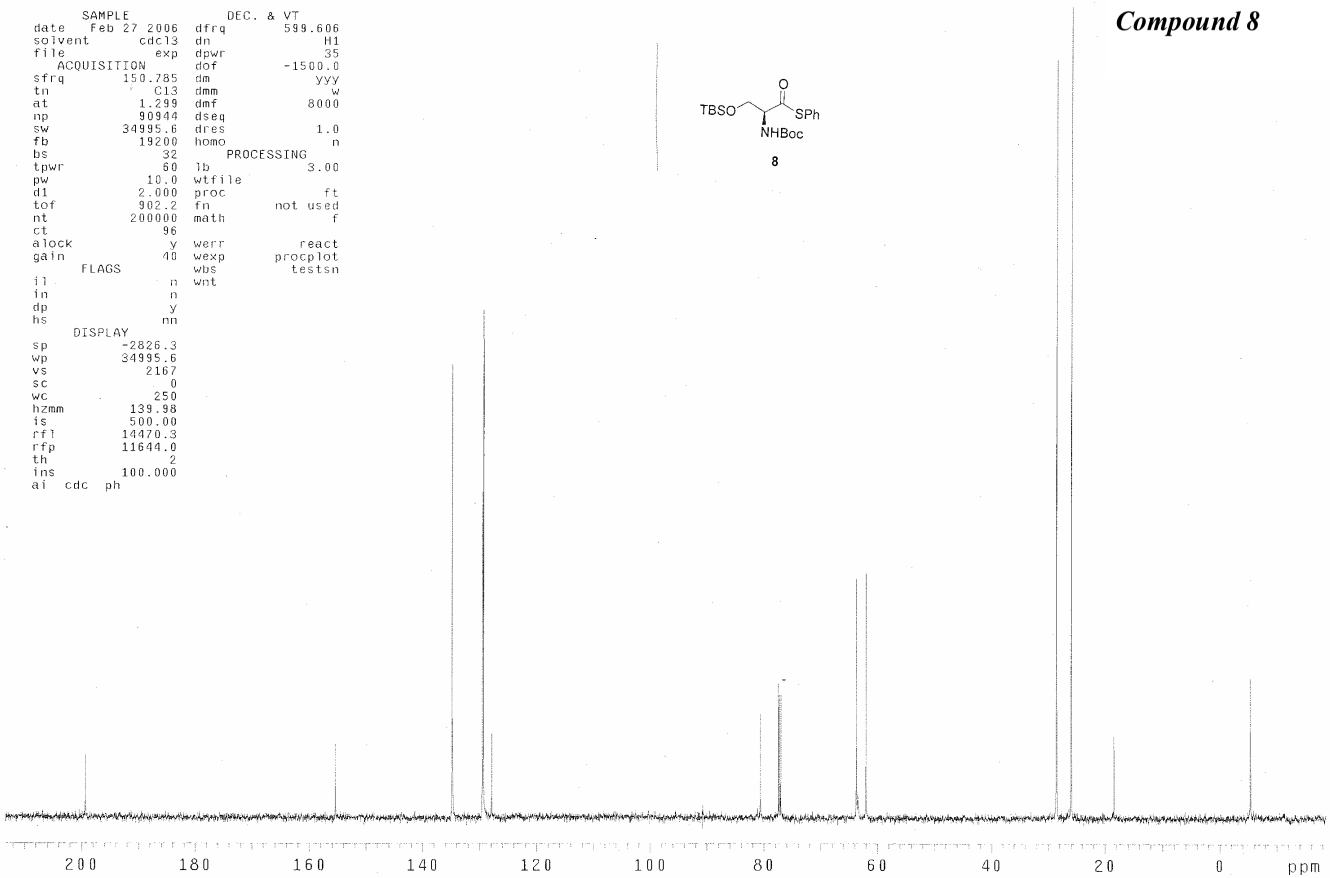
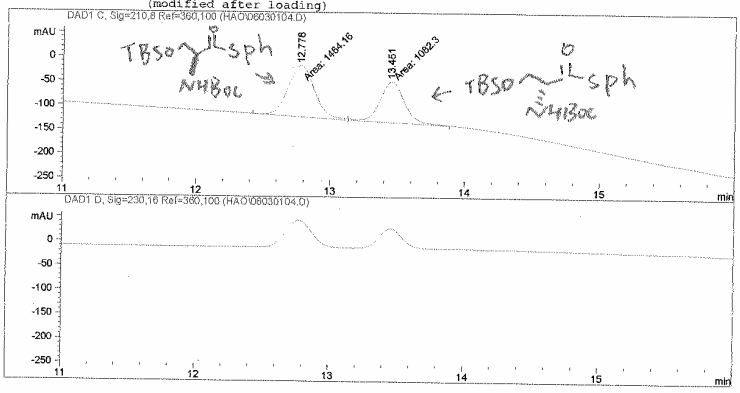


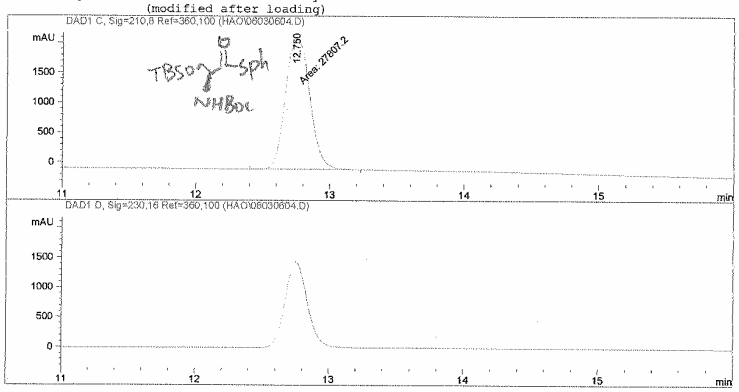
Table 1, Entry 4. R^1=TBDMS Compound 8

```
Injection Date  : 3/2/2006 3:03:20 AM                Seq. Line :   1
Sample Name     : 060301-04                          Location : Vial 15
Acq. Operator   : HaoYang                                Inj :   1
                                                   Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\RUEDIGER.M
Last changed    : 2/24/2006 6:22:29 AM by Harry
Analysis Method : C:\HPCHEM\1\METHODS\YANGHAO9.M
Last changed    : 3/12/2007 2:58:09 AM by hao
                  (modified after loading)
```




```
                          Area Percent Report

Sorted By              :      Retention Time
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Sig Type    Area       Height      Area
  #   [min]            [mAU*s]     [mAU]        %
----|-------|---|----|----------|----------|--------|
   1  12.778  1  MM   1464.15771  105.55557  57.5431
   2  13.451  1  MM   1082.29785   84.85347  42.5355

Totals :                2544.45557  188.40904

 Results obtained with enhanced integrator!

                      *** End of Report ***
```

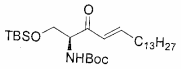
```
Injection Date  : 3/7/2006 2:57:32 AM               Seq. Line :   1
Sample Name     : 060306-04                          Location : Vial 23
Acq. Operator   : hao                                    Inj :   1
                                                   Inj Volume : 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\RUEDIGER.M
Last changed    : 2/24/2006 6:22:29 AM by Harry
Analysis Method : C:\HPCHEM\1\METHODS\YANGHAO9.M
Last changed    : 3/12/2007 3:00:14 AM by hao
                  (modified after loading)
```




```
                          Area Percent Report

Sorted By              :      Retention Time
Multiplier             :      1.0000
Dilution               :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Sig Type    Area       Height      Area
  #   [min]            [mAU*s]     [mAU]        %
----|-------|---|----|----------|----------|--------|
   1  12.750  1  MM   2.78072e4  2173.24683 100.0072

Totals :                2.78052e4  2171.24683

 Results obtained with enhanced integrator!

                      *** End of Report ***
```

Table 1, Entry 4. R^1=TBDMS

Compound 9



9

```
Standard Proton Parameters

exp1  s2pul

      SAMPLE                DEC. & VT
date   Jun 24 2005  dfrq        599.738
solvent       cdcl3  dn               H1
file            exp  dpwr              0
   ACQUISITION       dof              0
sfrq         599.738  dm             nnn
tn                H1  dmm              w
at             1.598  dmf            200
np             24948  dseq
sw            7804.1  dres           1.0
fb              4000  homo             n
bs                16      PROCESSING
tpwr              56  lb            0.20
pw               5.0  wtfile
d1             1.000  proc             ft
tof            285.3  fn        not used
nt                 5  math             f
ct                 5
alock              n  werr         react
gain       not used   wexp      procplot
      FLAGS           wbs        testsn
il                 n  wnt
in                 n
dp                 y
hs                nn
      DISPLAY
sp            -280.9
wp            4981.2
vs               151
sc                 0
wc               250
hzmm           19.92
is            109.64
rfl           4963.1
rfp           4354.1
th                19
ins            1.000
nm  cdc  ph
```

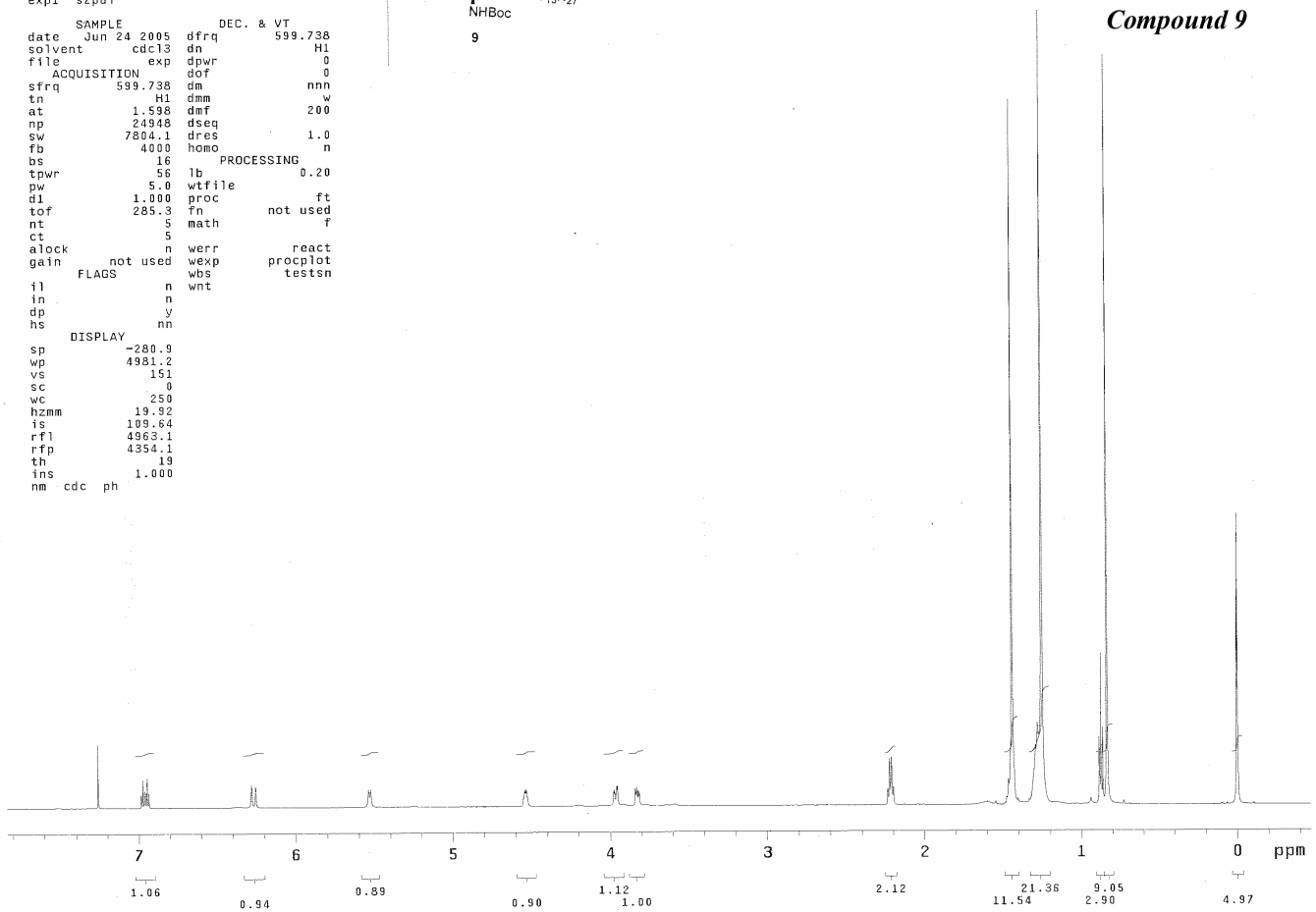
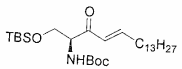


Table 1, Entry 4. R^1=TBDMS

Compound 9



9

```
13C OBSERVE

exp1  std13c

      SAMPLE                DEC. & VT
date   Jul 18 2005  dfrq        399.941
solvent      cdcl3  dn              H1
file           exp  dpwr            42
   ACQUISITION      dof         -200.0
sfrq       100.574  dm             yyy
tn             C13  dmm              w
at           1.199  dmf           7200
np           59968  dseq
sw         25000.0  dres           1.0
fb           14000  homo             n
bs              16      PROCESSING
tpwr            58  lb            2.00
pw             8.0  lsfid            4
d1           3.000  wtfile
tof              0  proc             ft
nt           10000  fn        not used
ct             224  math             f
alock            n
gain            50  werr         react
      FLAGS         wexp      procplot
il               n  wbs        testsn
in               n  wnt
dp               y
hs              nn
     DISPLAY
sp         -1291.5
wp         22006.1
vs             162
sc               0
wc             250
hzmm         88.02
is          500.00
rfl         2983.8
rfp              0
th              20
ins         100.000
nm  no  ph
```

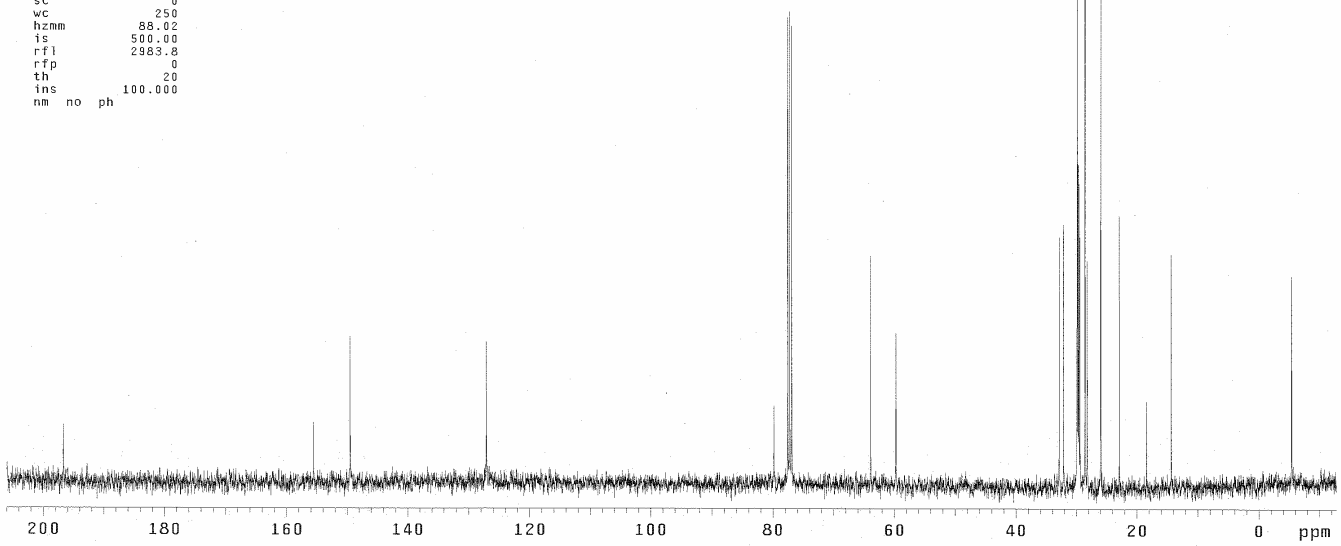
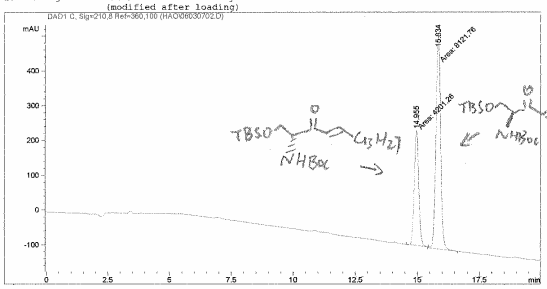


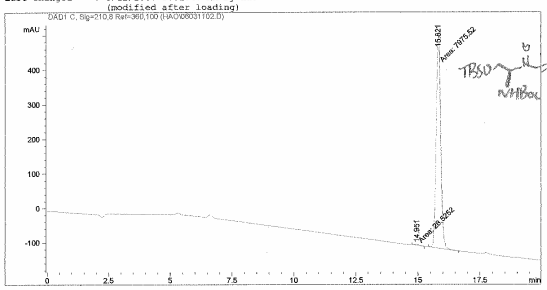
Table 1, Entry 4. R^1=TBDMS Compound 9

```
Injection Date  : 3/7/2006 11:25:42 PM          Seq. Line :  1
Sample Name     : 06030702                       Location : Vial 2
Acq. Operator   : Hao                                Inj :  1
                                                Inj Volume : 1 µl

Acq. Method     : C:\HPCHEM\1\METHODS\YANGHAO7.M
Last changed    : 3/2/2006 11:08:47 PM by hao
Analysis Method : C:\HPCHEM\1\METHODS\YANGHAO9.M
Last changed    : 3/12/2007 3:01:47 AM by hao
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Retention Time
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Sig Type     Area       Height      Area
  #   [min]            [mAU*s]     [mAU]        %
----|-------|---|----|----------|----------|--------|
   1  14.955  1  MM   4201.26416  330.11292  34.0928
   2  15.834  1  MM   8121.76172  614.05029  65.9072

Totals :                1.23230e4   944.16321

 Results obtained with enhanced integrator!
==================================================================
                        *** End of Report ***
```

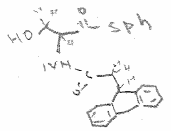
```
Injection Date  : 3/12/2006 9:15:17 AM          Seq. Line :  1
Sample Name     : 06031102                       Location : Vial 2
Acq. Operator   : Hao                                Inj :  1
                                                Inj Volume : 1 µl

Acq. Method     : C:\HPCHEM\1\METHODS\YANGHAO7.M
Last changed    : 3/2/2006 11:08:47 PM by hao
Analysis Method : C:\HPCHEM\1\METHODS\YANGHAO9.M
Last changed    : 3/12/2007 3:01:47 AM by hao
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Retention Time
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Sig Type     Area       Height      Area
  #   [min]            [mAU*s]     [mAU]        %
----|-------|---|----|----------|----------|--------|
   1  14.951  1  MM     28.52520    2.84855   0.3564
   2  15.821  1  MM   7975.51563  616.33783  99.6436

Totals :                8004.04082  619.18638

 Results obtained with enhanced integrator!
==================================================================
                        *** End of Report ***
```

hy400-fmocsersph

Pulse Sequence: s2pul



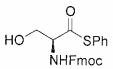
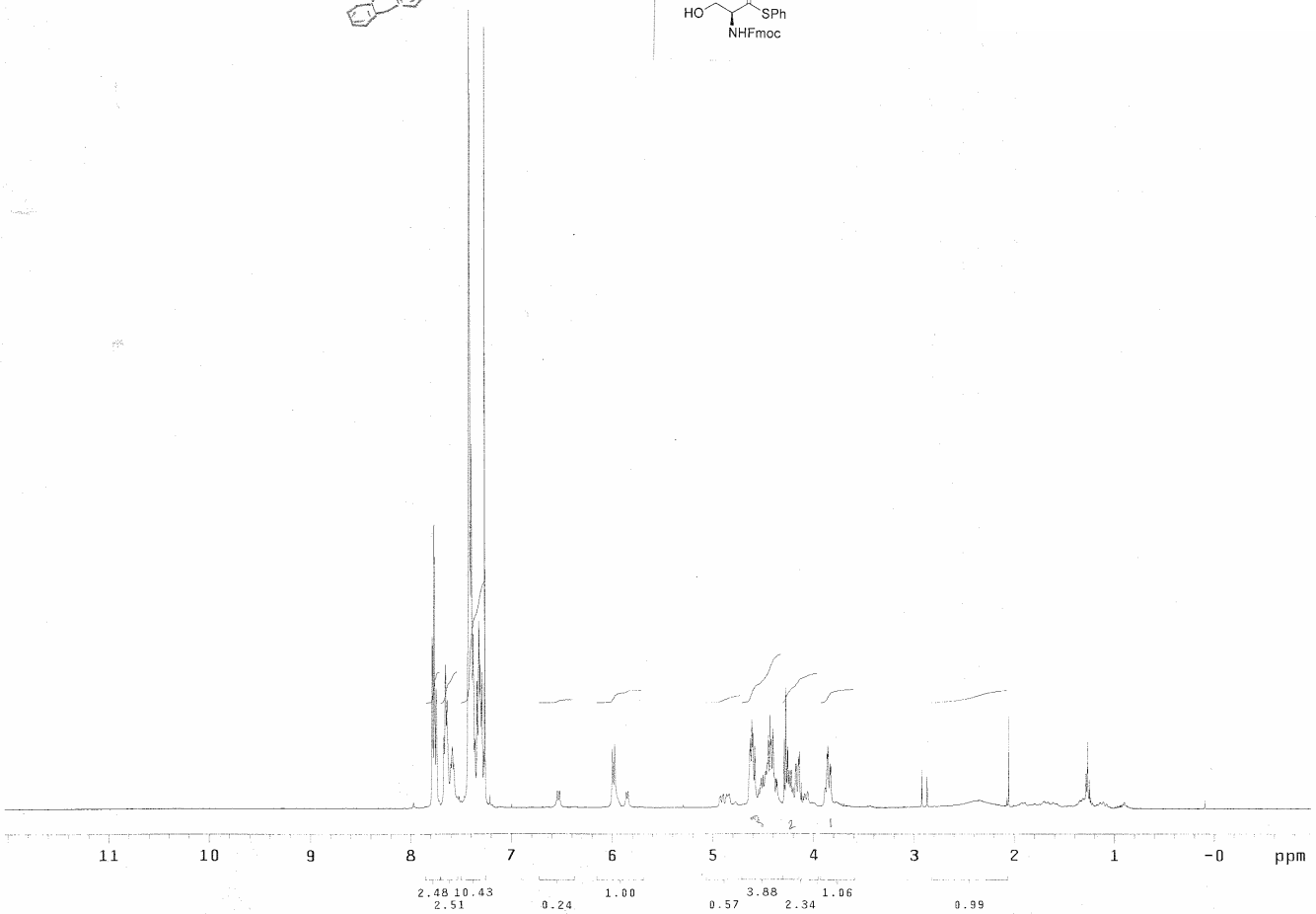


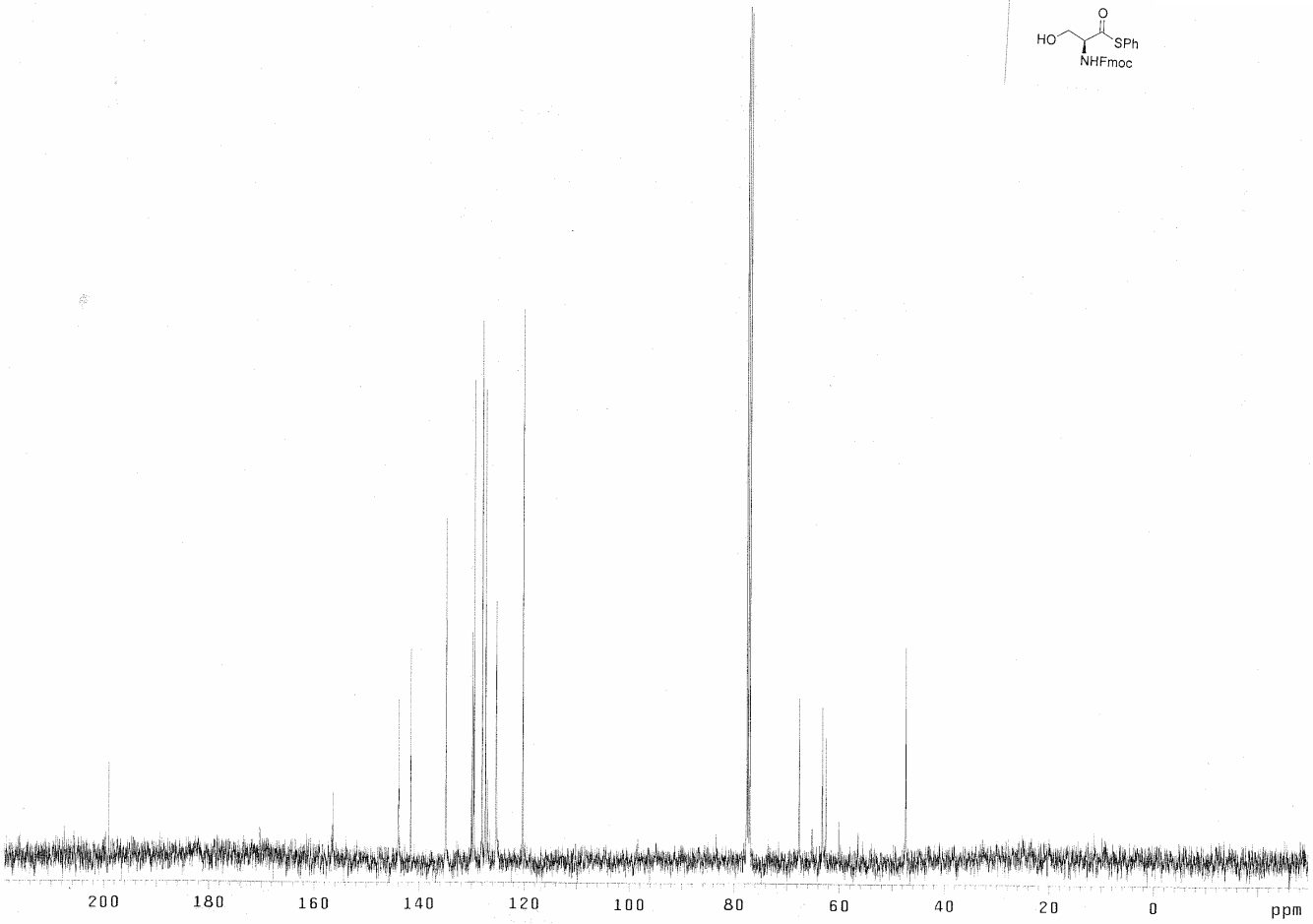
Table 1, Entry 5. R^1=H



13C OBSERVE

Pulse Sequence: s2pul

Table 1, Entry 5. R^1=H



HYFmocSerinenec13h27

Pulse Sequence: s2pul

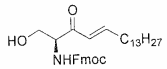
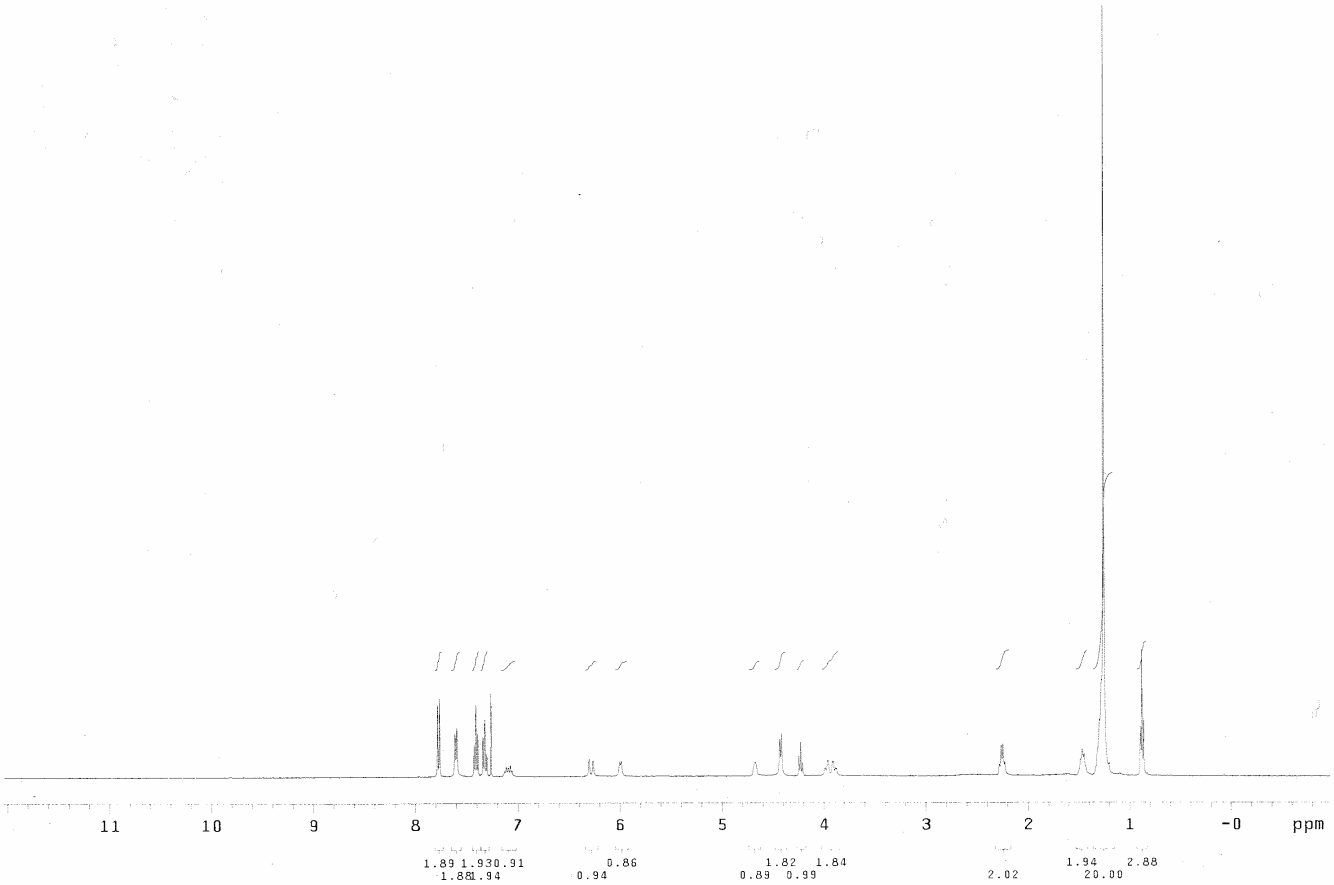


Table 1, Entry 5. R^1=H



13C OBSERVE

exp1 std13c

SAMPLE		DEC. & VT	
date	Nov 18 2006	dfrq	399.941
solvent	cdcl3	dn	H1
file	exp	dpwr	42
ACQUISITION		dof	-200.0
sfrq	100.574	dm	yyy
tn	C13	dmm	w
at	1.199	dmf	7200
np	59968	dseq	
sw	25000.0	dres	1.0
fb	14000	homo	n
bs	16	PROCESSING	
tpwr	58	lb	2.00
pw	8.0	lsfid	4
d1	3.000	wtfile	
tof	0	proc	ft
nt	20000	fn	not used
ct	400	math	f
alock	n		
gain	50	werr	react
FLAGS		wexp	procplot
il	n	wbs	testsn
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-2983.8		
wp	25000.0		
vs	162		
sc	0		
wc	250		
hzmm	100.00		
is	500.00		
rfl	2983.8		
rfp	0		
th	20		
ins	100.000		
nm no ph			

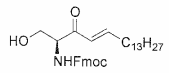
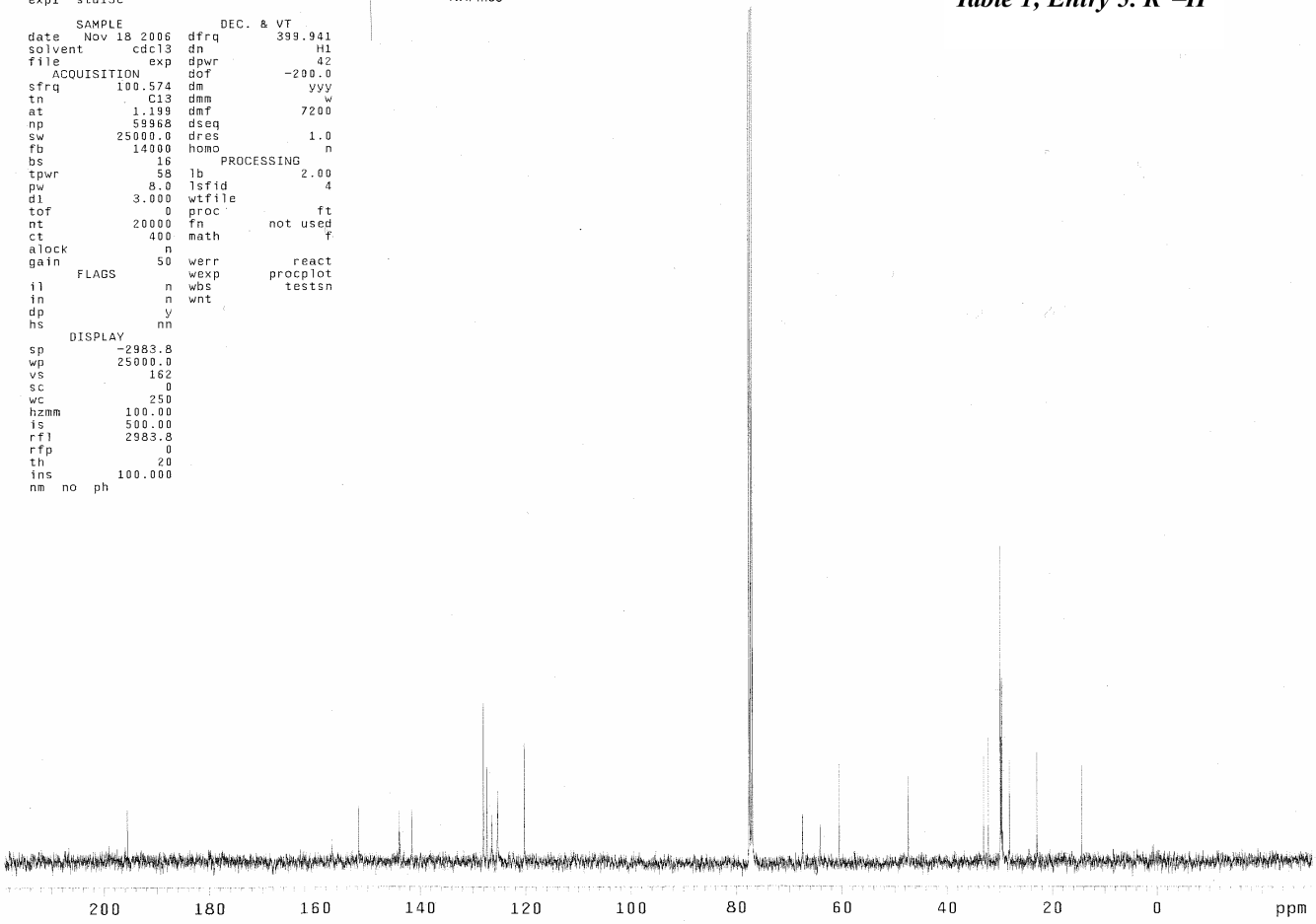


Table 1, Entry 5. R^1=H



OTBSNFmocSerineSph

File : xp
Sample id : tmpstudy

Pulse Sequence: s2pul1

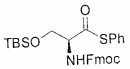
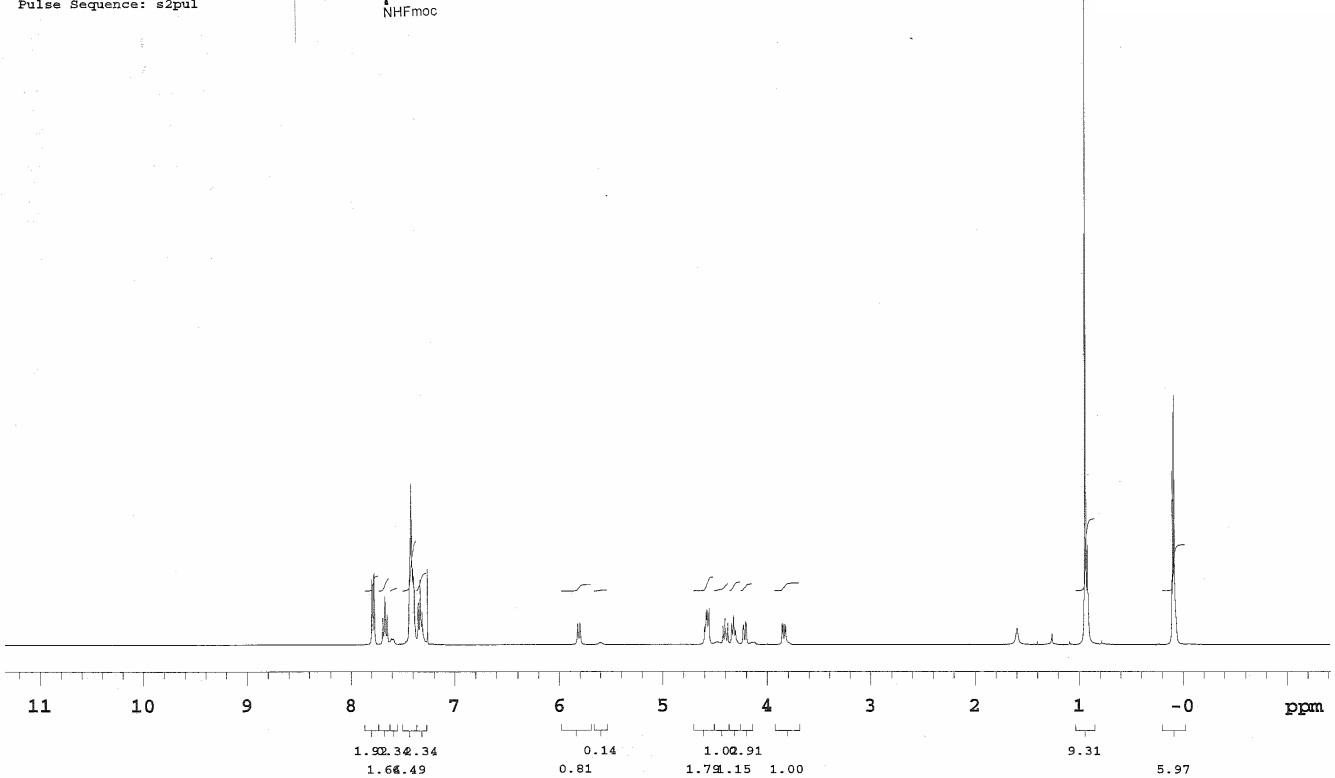


Table 1, Entry 5. R^1=TBDMS

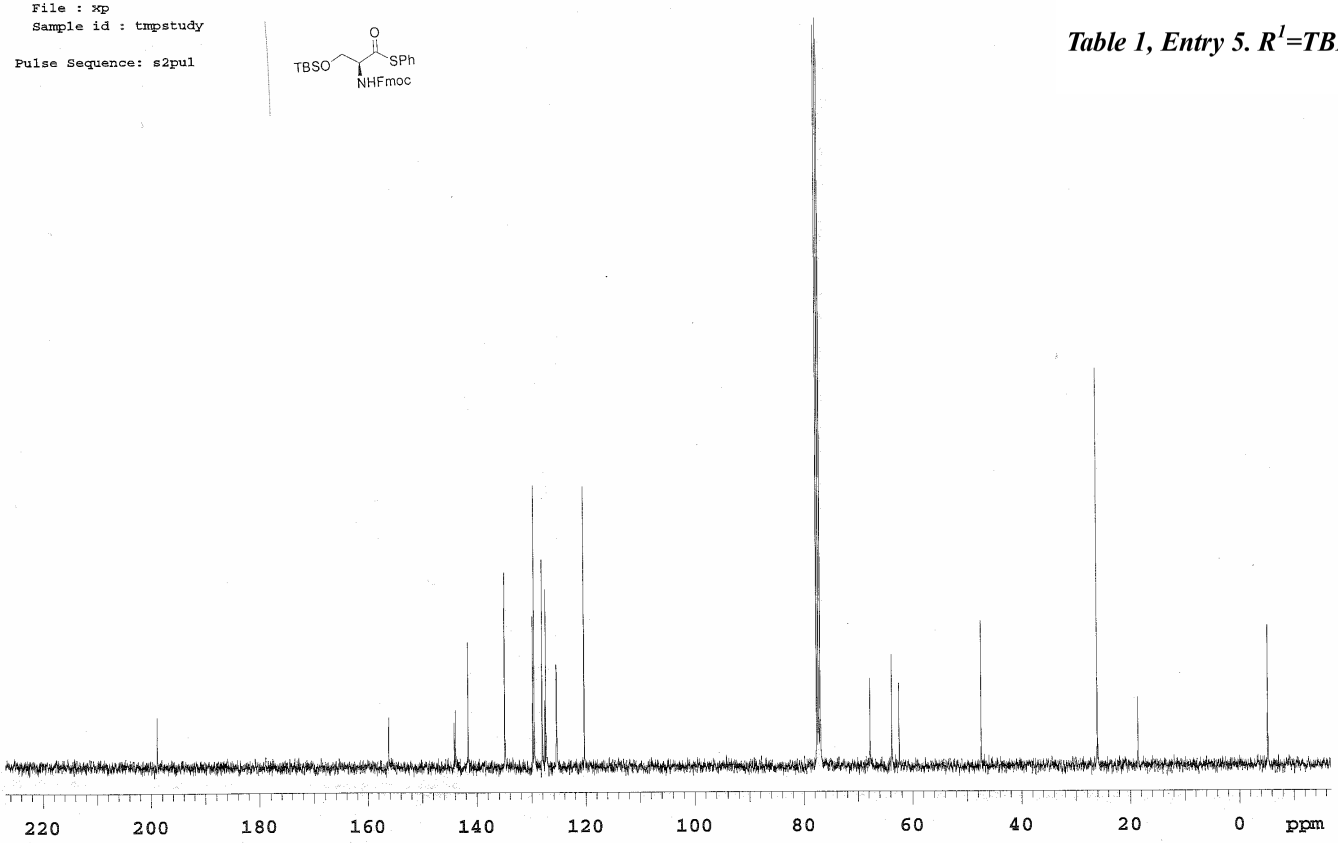


OTBSNFmocSerineSph

File : xp
Sample id : tmpstudy

Pulse Sequence: s2pul

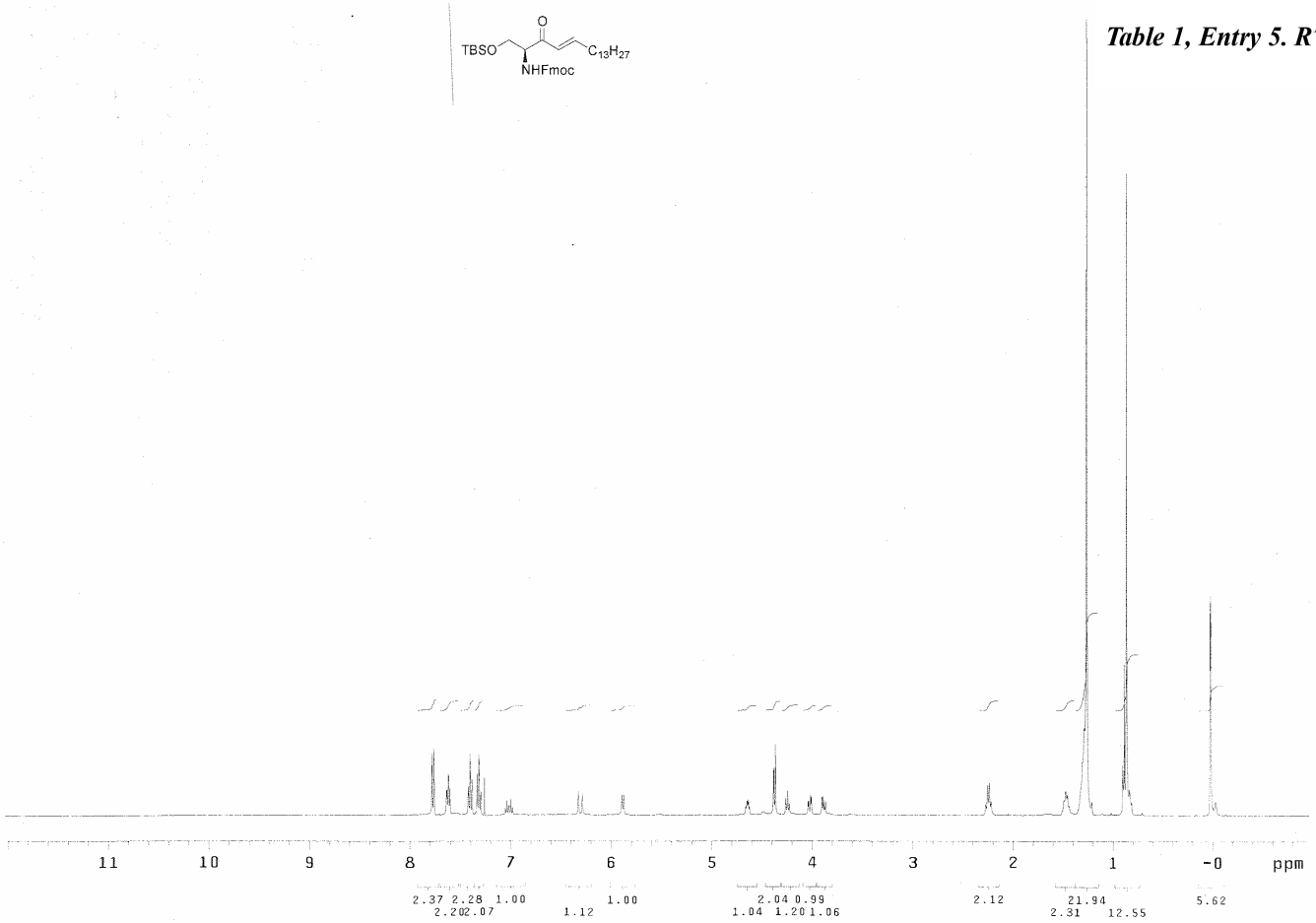
Table 1, Entry 5. R^1=TBDMS



HYTBSFmocSer,inenec13h27

Pulse Sequence: s2pul

Table 1, Entry 5. R^1=TBDMS

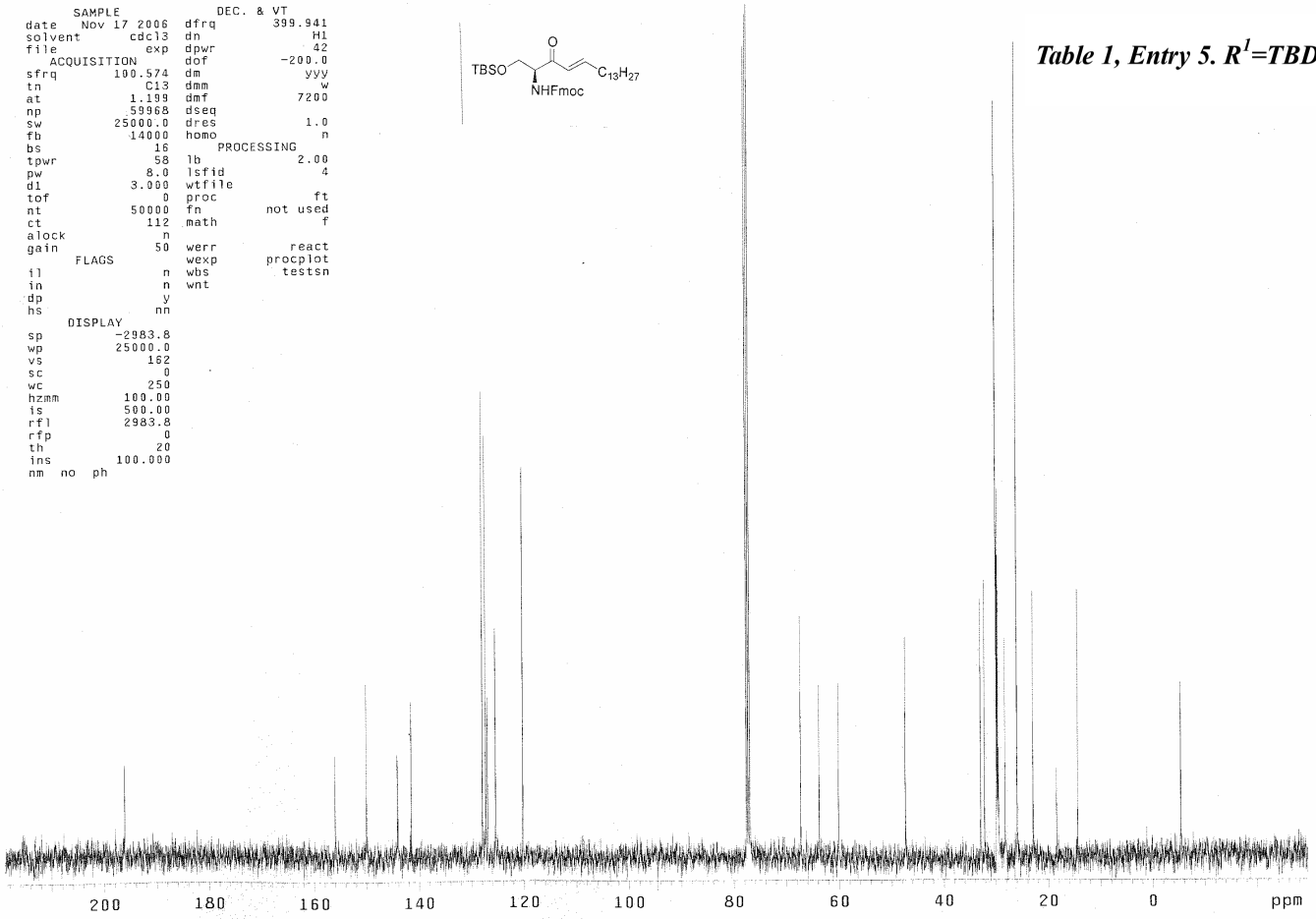


HYTBSFmocSerinenec13h37

expl std13c

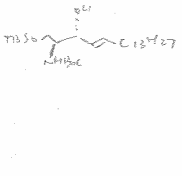
SAMPLE		DEC. & VT	
date	Nov 17 2006	dfrq	399.941
solvent	cdcl3	dn	H1
file	exp	dpwr	42
ACQUISITION		dof	-200.0
sfrq	100.574	dm	yyy
tn	C13	dmm	w
at	1.199	dmf	7200
np	59968	dseq	
sw	25000.0	dres	1.0
fb	14000	homo	n
bs	16	PROCESSING	
tpwr	58	lb	2.00
pw	8.0	lsfid	4
d1	3.000	wtfile	
tof	0	proc	ft
nt	50000	fn	not used
ct	112	math	f
alock	n		
gain	50	werr	react
FLAGS		wexp	procplot
il	n	wbs	testsn
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-2983.8		
wp	25000.0		
vs	162		
sc	0		
wc	250		
hzmm	100.00		
is	500.00		
rfl	2983.8		
rfp	0		
th	20		
ins	100.000		
nm no ph			

Table 1, Entry 5. R^1=TBDMS



Scheme 2, Compound 10

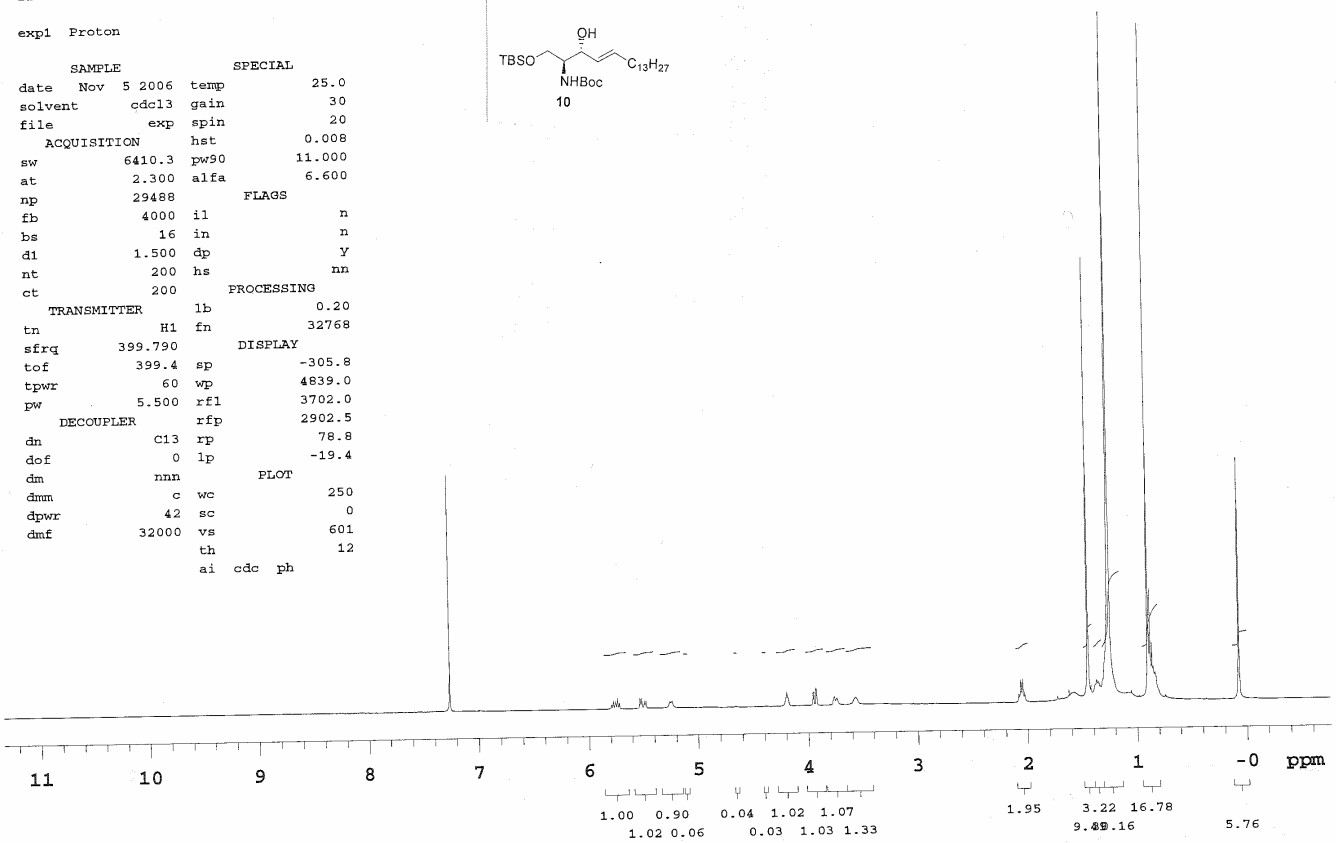
Selectivity 3:1



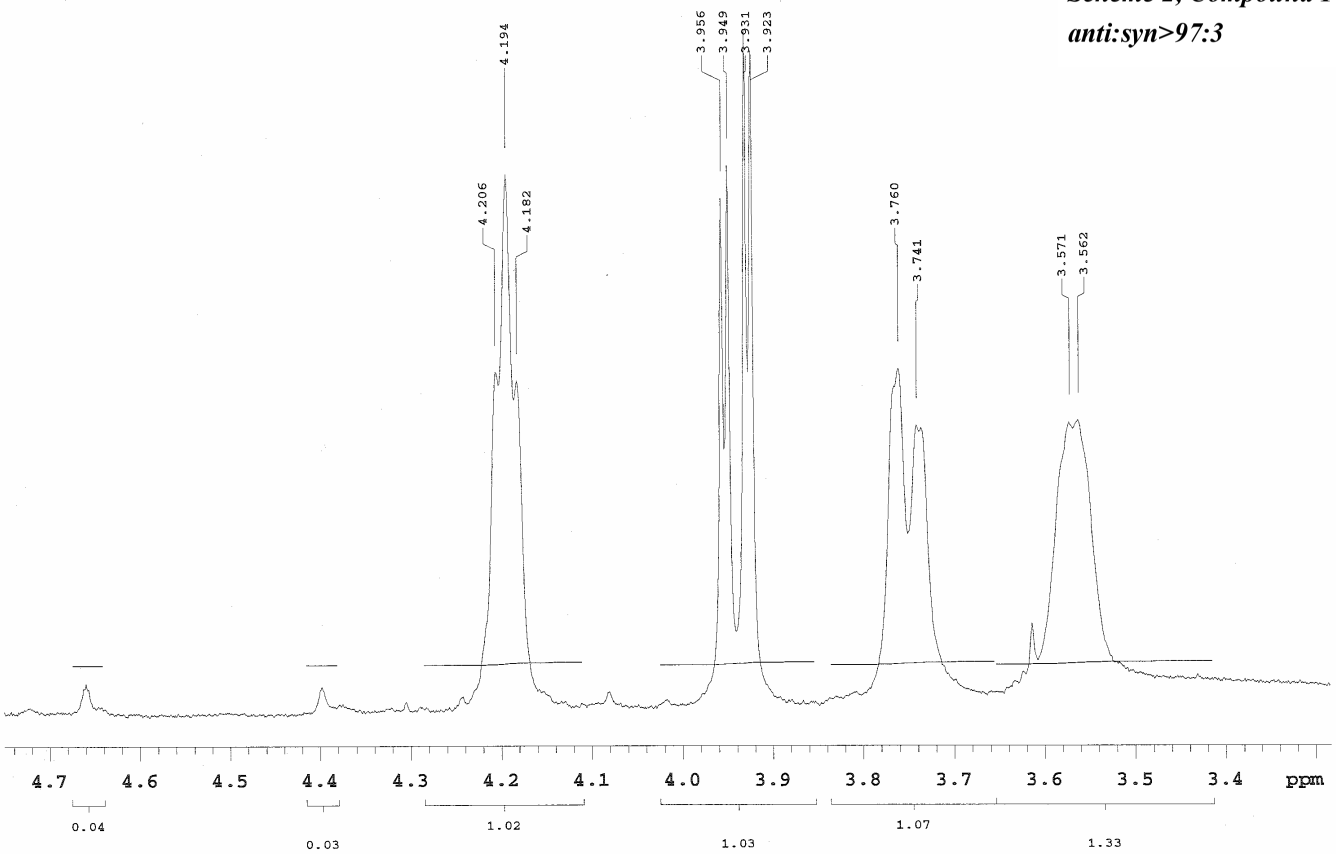
110506HY

exp1 Proton

SAMPLE		**SPECIAL**	
date	Nov 5 2006	temp	25.0
solvent	cdcl3	gain	30
file	exp	spin	20
ACQUISITION		hst	0.008
sw	6410.3	pw90	11.000
at	2.300	alfa	6.600
np	29488	**FLAGS**	
fb	4000	il	n
bs	16	in	n
d1	1.500	dp	y
nt	200	hs	nn
ct	200	**PROCESSING**	
TRANSMITTER		lb	0.20
tn	H1	fn	32768
sfrq	399.790	**DISPLAY**	
tof	399.4	sp	-305.8
tpwr	60	wp	4839.0
pw	5.500	rf1	3702.0
DECOUPLER		rfp	2902.5
dn	C13	rp	78.8
dof	0	lp	-19.4
dm	nnn	**PLOT**	
dmm	c	wc	250
dpwr	42	sc	0
dmf	32000	vs	601
		th	12
		ai cdc ph	



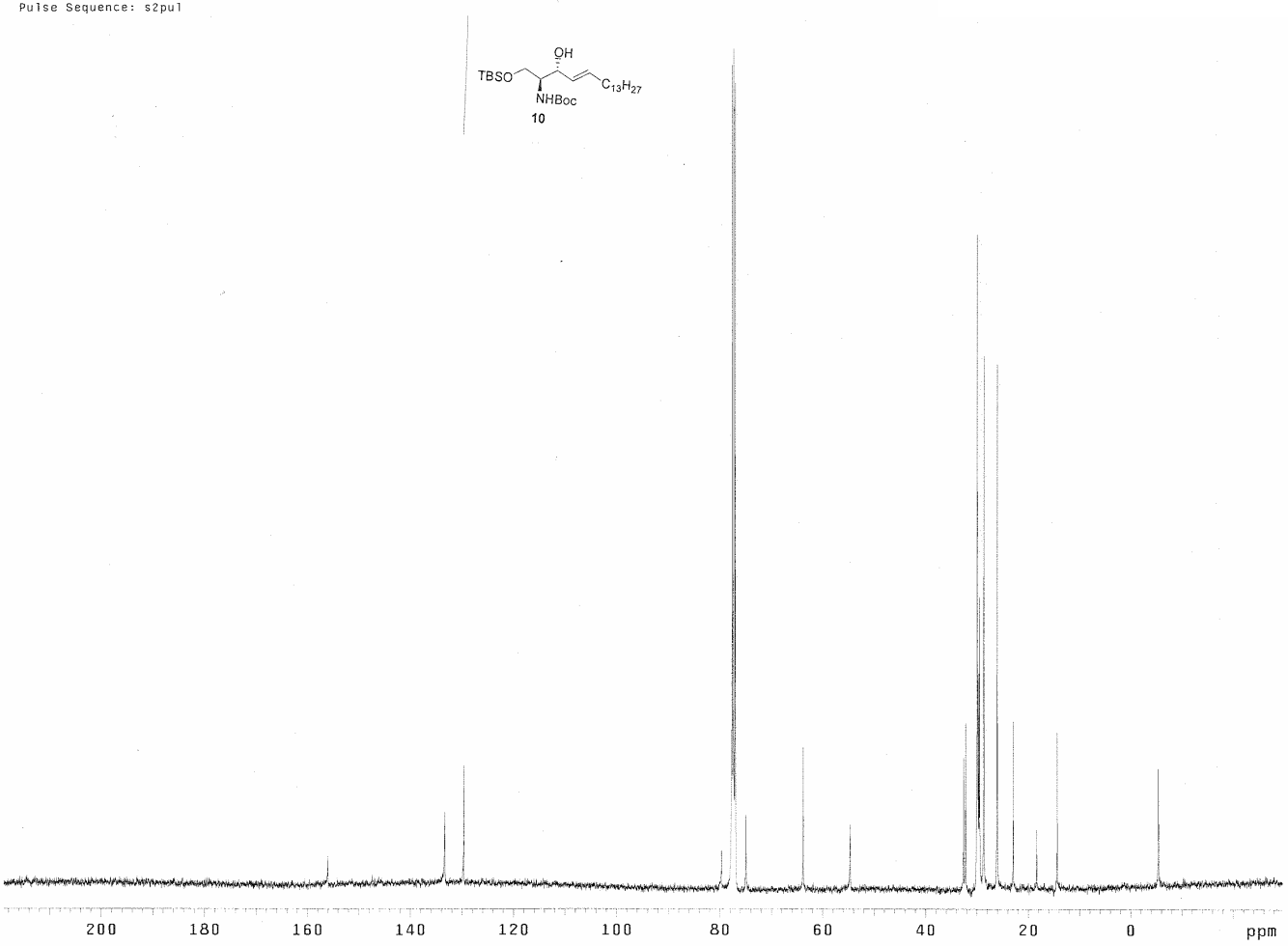
Scheme 2, Compound 10
anti:syn>97:3



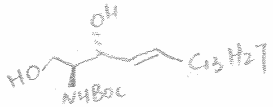
Scheme 2, Compound 10

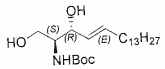
tbsbocsphingsoine400

Pulse Sequence: s2pul



Scheme 2, Compound 11

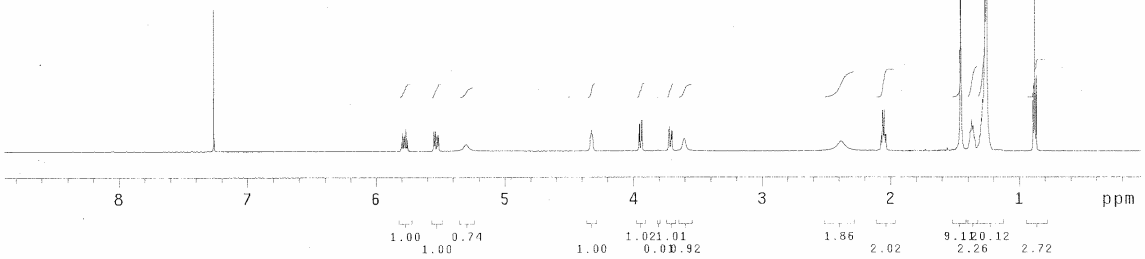




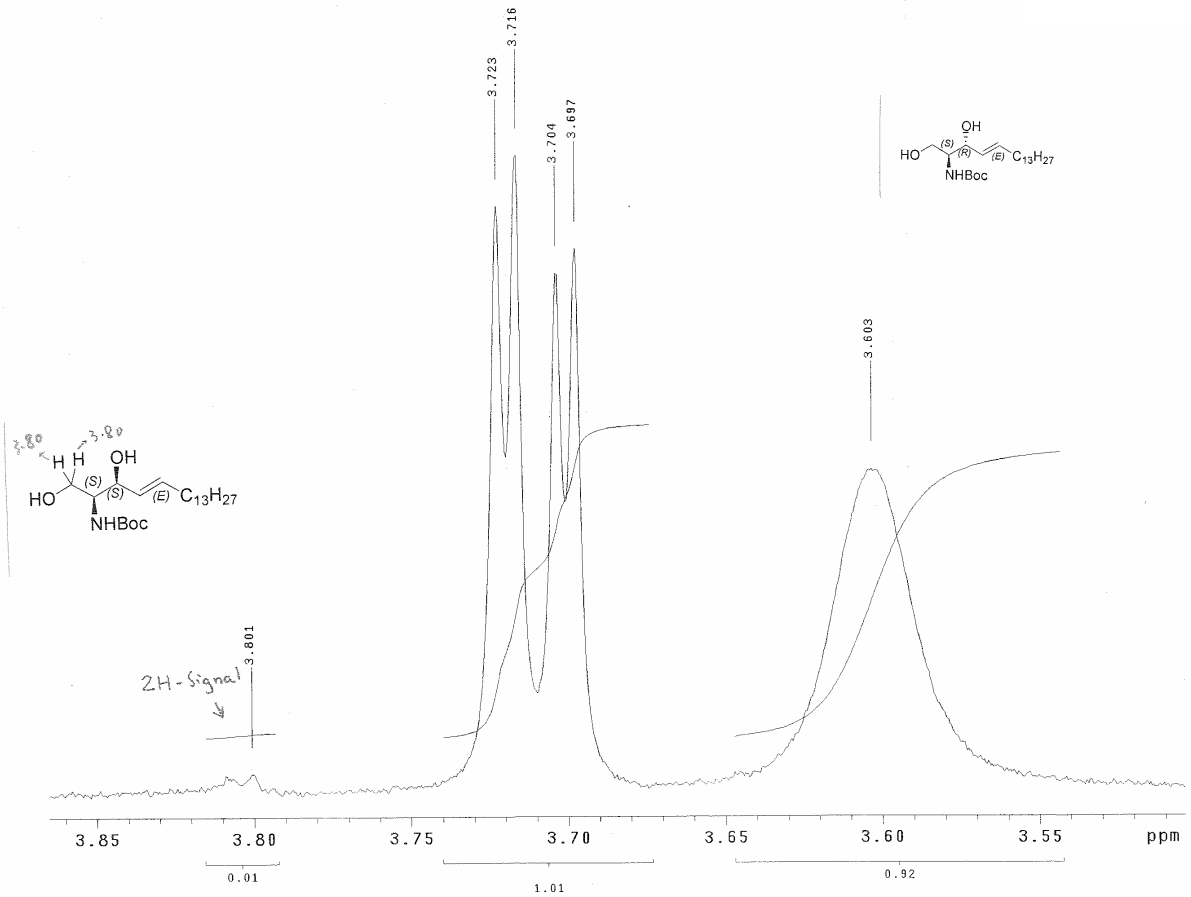
STANDARD PROTON PARAMETERS

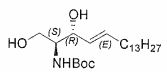
exp1 s2pul

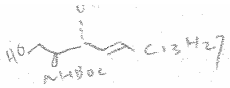
SAMPLE		DEC. & VT	
date	Mar 24 2006	dfrq	599.608
solvent	cdcl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	599.607	dm	nnn
tn	H1	dmm	c
at	2.002	dmf	200
np	32832	dseq	
sw	8201.8	dres	1.0
fb	4600	homo	n
bs	16	PROCESSING	
tpwr	56	lb	0.20
pw	3.0	wtfile	
d1	1.000	proc	ft
tof	-657.6	fn	not used
nt	50	math	f
ct	50		
alock	y	werr	react
gain	6	wexp	procplot
FLAGS		wbs	
il	n	wnt	wft
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	32.1		
wp	5297.7		
vs	151		
sc	35		
wc	215		
hzmm	24.61		
is	6408.67		
rfl	4956.6		
rfp	4353.1		
th	0		
ins	1.000		
nm cdc ph			



Scheme 2, Compound 11
d.r. > 200:1

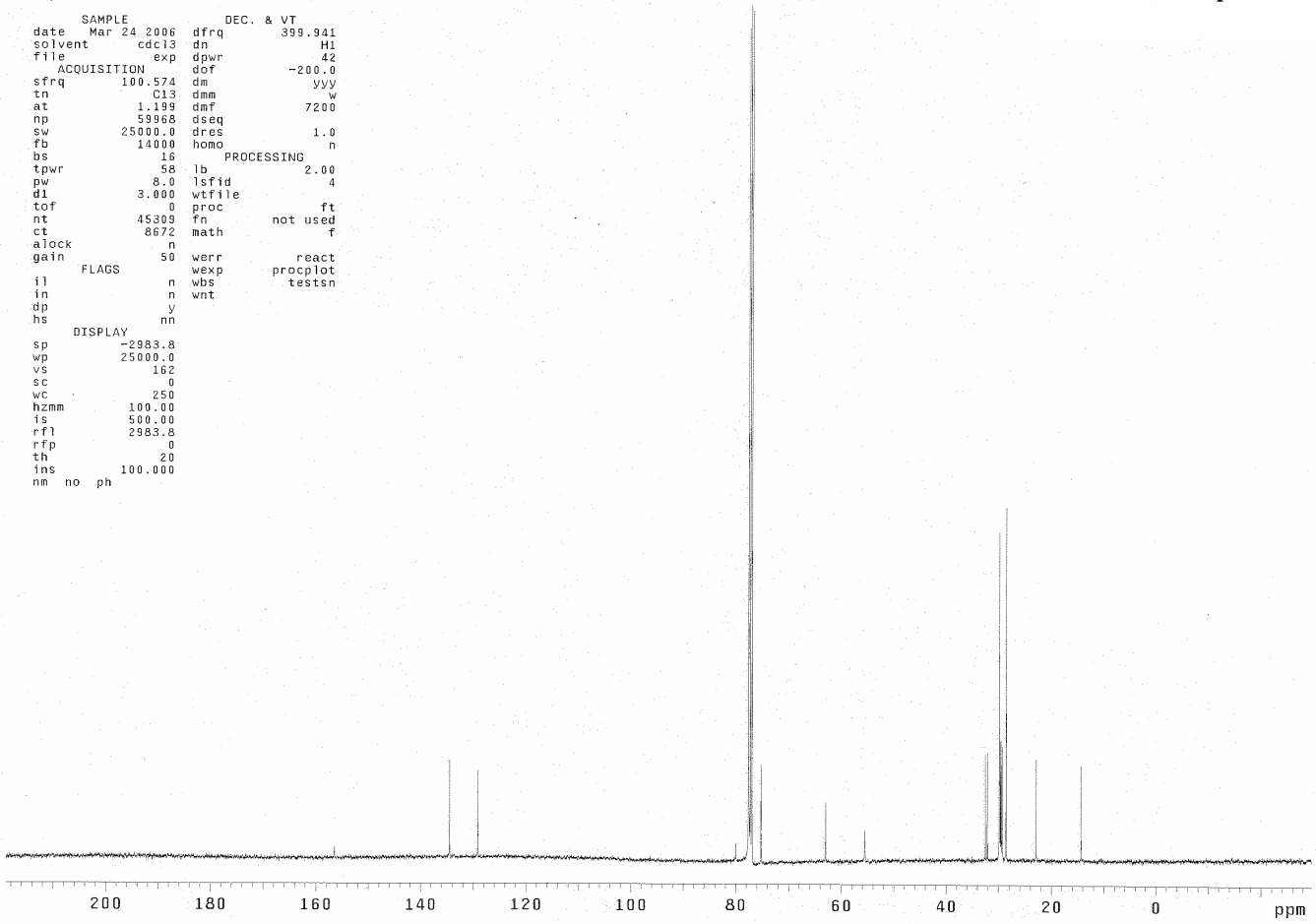






Scheme 2, Compound 11

```
13C OBSERVE

exp1  std13c

      SAMPLE                DEC. & VT
date   Mar 24 2006  dfrq        399.941
solvent       cdcl3  dn             H1
file            exp  dpwr            42
   ACQUISITION       dof         -200.0
sfrq        100.574  dm             yyy
tn              C13  dmm              w
at            1.199  dmf           7200
np            59968  dseq
sw          25000.0  dres           1.0
fb            14000  homo             n
bs               16     PROCESSING
tpwr             58  lb           2.00
pw              8.0  lsfid           4
d1            3.000  wtfile
tof               0  proc            ft
nt            45309  fn        not used
ct             8672  math             f
alock             n
gain             50  werr         react
      FLAGS          wexp      procplot
il                n  wbs         testsn
in                n  wnt
dp                y
hs               nn
     DISPLAY
sp          -2983.8
wp          25000.0
vs              162
sc                0
wc              250
hzmm         100.00
is           500.00
rfl          2983.8
rfp               0
th               20
ins         100.000
nm  no  ph
```

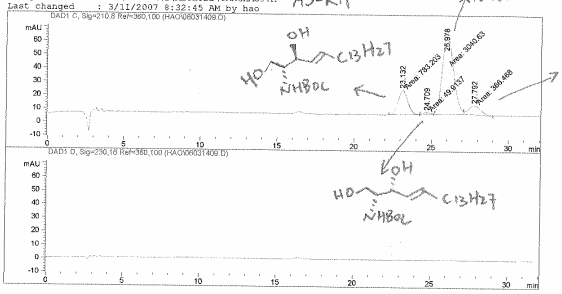


Injection Date : 3/15/2006 2:33:10 AM
Sample Name : 06031409
Acq. Operator : HAO

Seq. Line : 1
Location : Vial 3
Inj : 1
Inj Volume : 1 µl

Acq. Method : C:\HPCHEM\1\METHODS\YANGHAO9.M
Last changed : 3/15/2006 2:25:51 AM by HAO
Analysis Method : C:\HPCHEM\1\METHODS\YANGHAO9.M
Last changed : 3/11/2007 8:32:45 AM by hao

AS-RH



Area Percent Report

Sorted By : Retention Time
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Sig	Type	Area [mAU*s]	Height [mAU]	Area %
1	23.132	1	MM	783.20319	17.85378	18.4796
2	24.709	1	MM	49.91369	1.36458	1.1777
3	25.978	1	MM	3040.63037	59.94418	71.7432
4	27.792	1	MM	366.46802	6.66012	8.6468

Totals : 4238.21527 83.82267

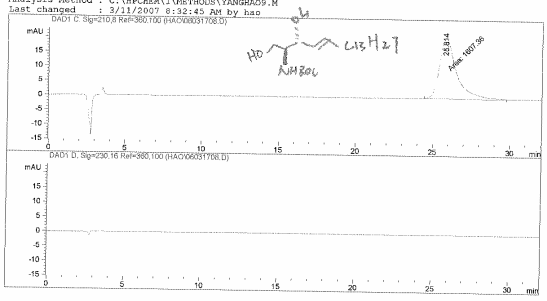
Results obtained with enhanced integrator!

*** End of Report ***

Injection Date : 3/17/2006 12:02:34 PM
Sample Name : nbocsphingosine
Acq. Operator : Hao

Seq. Line : 1
Location : Vial 42
Inj : 1
Inj Volume : 1 µl

Acq. Method : C:\HPCHEM\1\METHODS\YANGHAO9.M
Last changed : 3/17/2006 11:51:20 AM by Hao
Analysis Method : C:\HPCHEM\1\METHODS\YANGHAO9.M
Last changed : 3/11/2007 8:32:45 AM by hao



Area Percent Report

Sorted By : Retention Time
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Sig	Type	Area [mAU*s]	Height [mAU]	Area %
1	25.814	1	MM	1607.36011	20.71342	100.1246

Totals : 1605.36011 18.71342

Results obtained with enhanced integrator!

*** End of Report ***

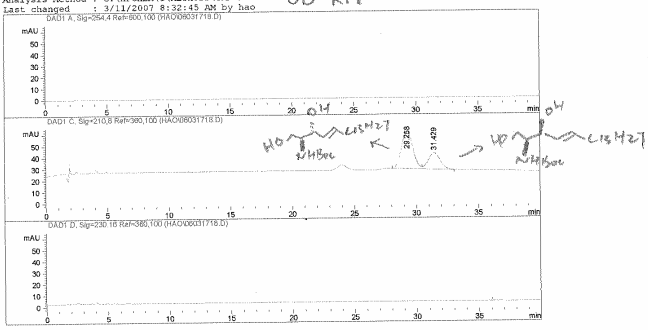
Scheme 2, Compound 11 enantiopurity

Injection Date : 3/9/2007 4:35:20 AM
Sample Name : nbocsphingosine
Acq. Operator : Hao

Seq. Line : 1
Location : Vial 43
Inj : 1
Inj Volume : 1 µl

Acq. Method : C:\HPCHEM\1\METHODS\YANGHAO9.M
Last changed : 3/9/2007 4:31:02 AM by Hao
Analysis Method : C:\HPCHEM\1\METHODS\YANGHAO9.M
Last changed : 3/11/2007 8:32:45 AM by hao

OD-RH



Area Percent Report

Sorted By : Retention Time
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 2: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Sig	Type	Area [mAU*s]	Height [mAU]	Area %
1	29.258	2	BV	2117.04785	33.79112	69.1005
2	31.429	2	VB	950.67285	14.37019	31.0300

Totals : 3063.72070 44.16131

Results obtained with enhanced integrator!

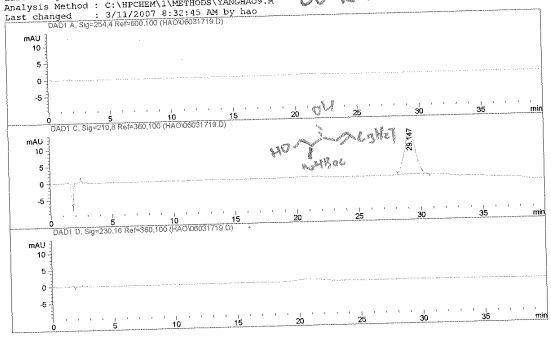
*** End of Report ***

Injection Date : 3/9/2007 5:21:08 AM
Sample Name : nbocsphingosine
Acq. Operator : Hao

Seq. Line : 1
Location : Vial 42
Inj : 1
Inj Volume : 1 µl

Acq. Method : C:\HPCHEM\1\METHODS\YANGHAO9.M
Last changed : 3/9/2007 4:31:02 AM by Hao
Analysis Method : C:\HPCHEM\1\METHODS\YANGHAO9.M
Last changed : 3/11/2007 8:32:45 AM by hao

OD-RH



Area Percent Report

Sorted By : Retention Time
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 2: DAD1 C, Sig=210,8 Ref=360,100

Peak #	RetTime [min]	Sig	Type	Area [mAU*s]	Height [mAU]	Area %
1	29.147	2	BB	793.96118	12.67131	100.5064

Totals : 789.96118 8.67131

Results obtained with enhanced integrator!

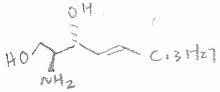
*** End of Report ***

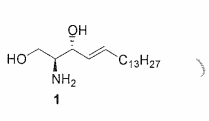
Table 1, Entry 1. R^1=H diastereomer purity

STANDARD 1H OBSERVE

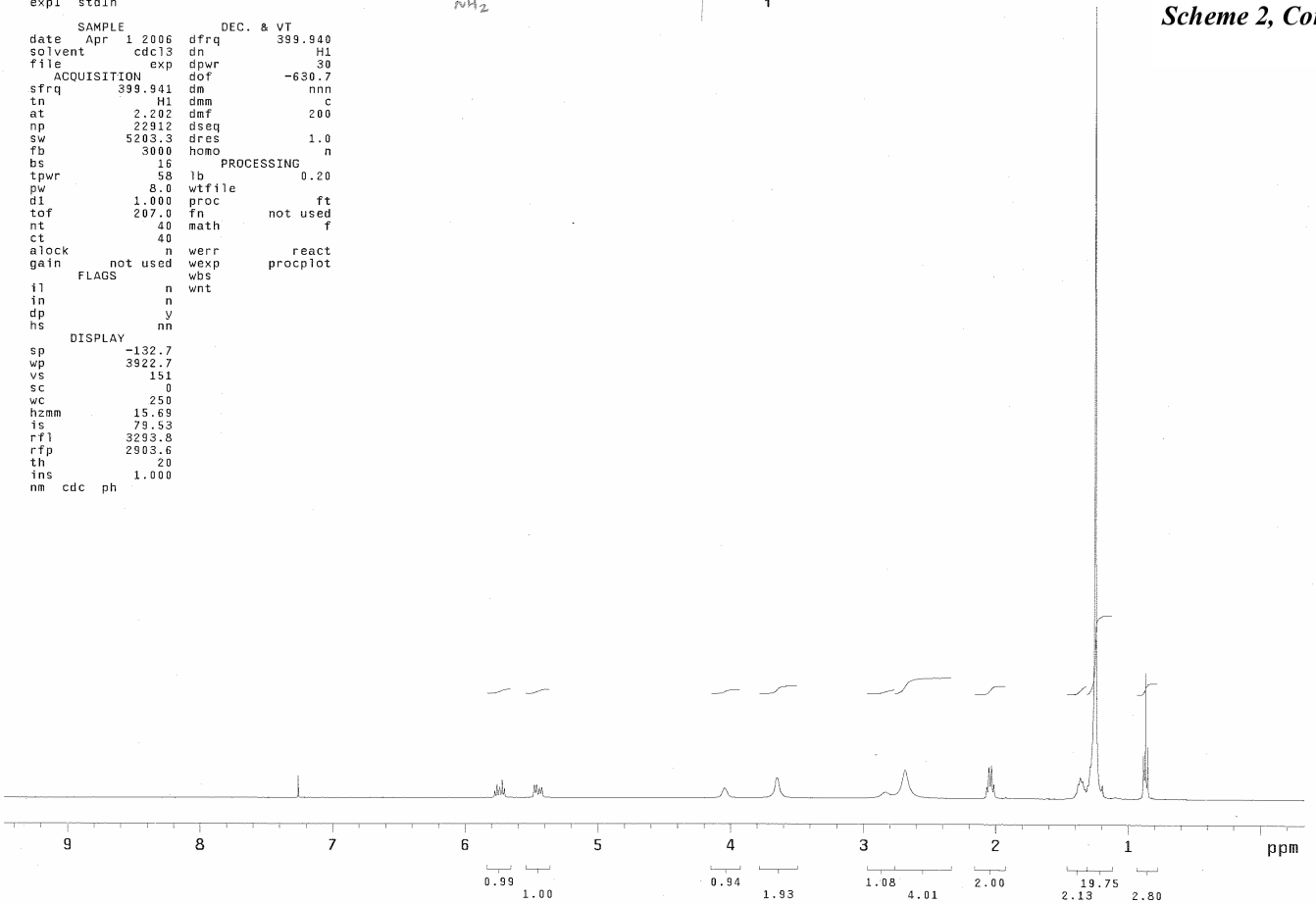
exp1 std1h

SAMPLE		DEC. & VT	
date	Apr 1 2006	dfrq	399.940
solvent	cdcl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	-630.7
sfrq	399.941	dm	nnn
tn	H1	dmm	c
at	2.202	dmf	200
np	22912	dseq	
sw	5203.3	dres	1.0
fb	3000	homo	n
bs	16	PROCESSING	
tpwr	58	lb	0.20
pw	8.0	wtfile	
d1	1.000	proc	ft
tof	207.0	fn	not used
nt	40	math	f
ct	40		
alock	n	werr	react
gain	not used	wexp	procplot
FLAGS		wbs	
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-132.7		
wp	3922.7		
vs	151		
sc	0		
wc	250		
hzmm	15.69		
is	79.53		
rfl	3293.8		
rfp	2903.6		
th	20		
ins	1.000		
nm cdc ph			





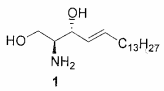
Scheme 2, Compound 1



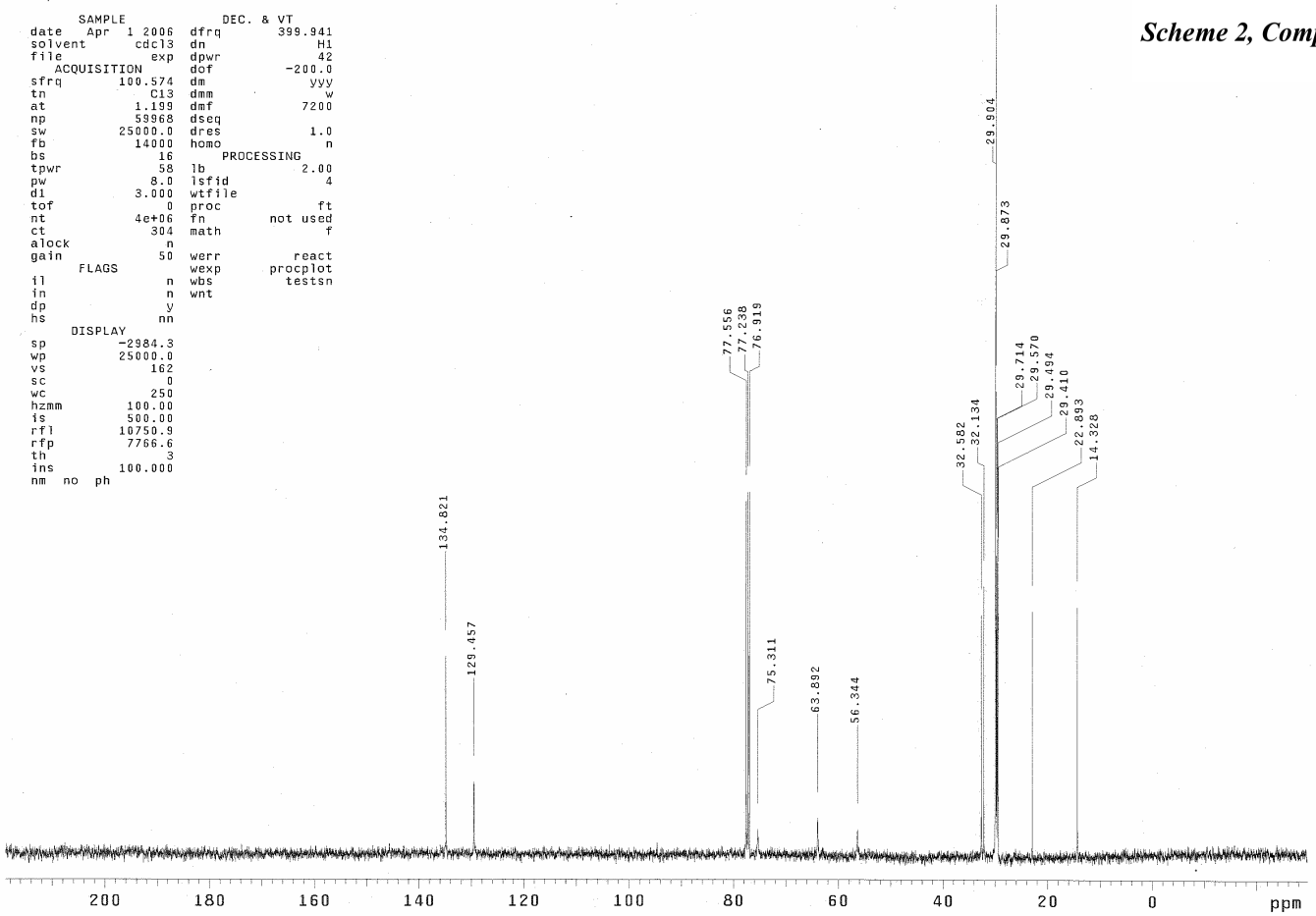
13C OBSERVE

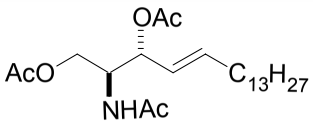
exp1 std13c

SAMPLE		DEC. & VT	
date	Apr 1 2006	dfrq	399.941
solvent	cdcl3	dn	H1
file	exp	dpwr	42
ACQUISITION		dof	-200.0
sfrq	100.574	dm	yyy
tn	C13	dmm	w
at	1.199	dmf	7200
np	59968	dseq	
sw	25000.0	dres	1.0
fb	14000	homo	n
bs	16	PROCESSING	
tpwr	58	lb	2.00
pw	8.0	lsfid	4
d1	3.000	wtfile	
tof	0	proc	ft
nt	4e+06	fn	not used
ct	304	math	f
alock	n		
gain	50	werr	react
FLAGS		wexp	procplot
il	n	wbs	testsn
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-2984.3		
wp	25000.0		
vs	162		
sc	0		
wc	250		
hzmm	100.00		
is	500.00		
rfl	10750.9		
rfp	7766.6		
th	3		
ins	100.000		
nm no ph			



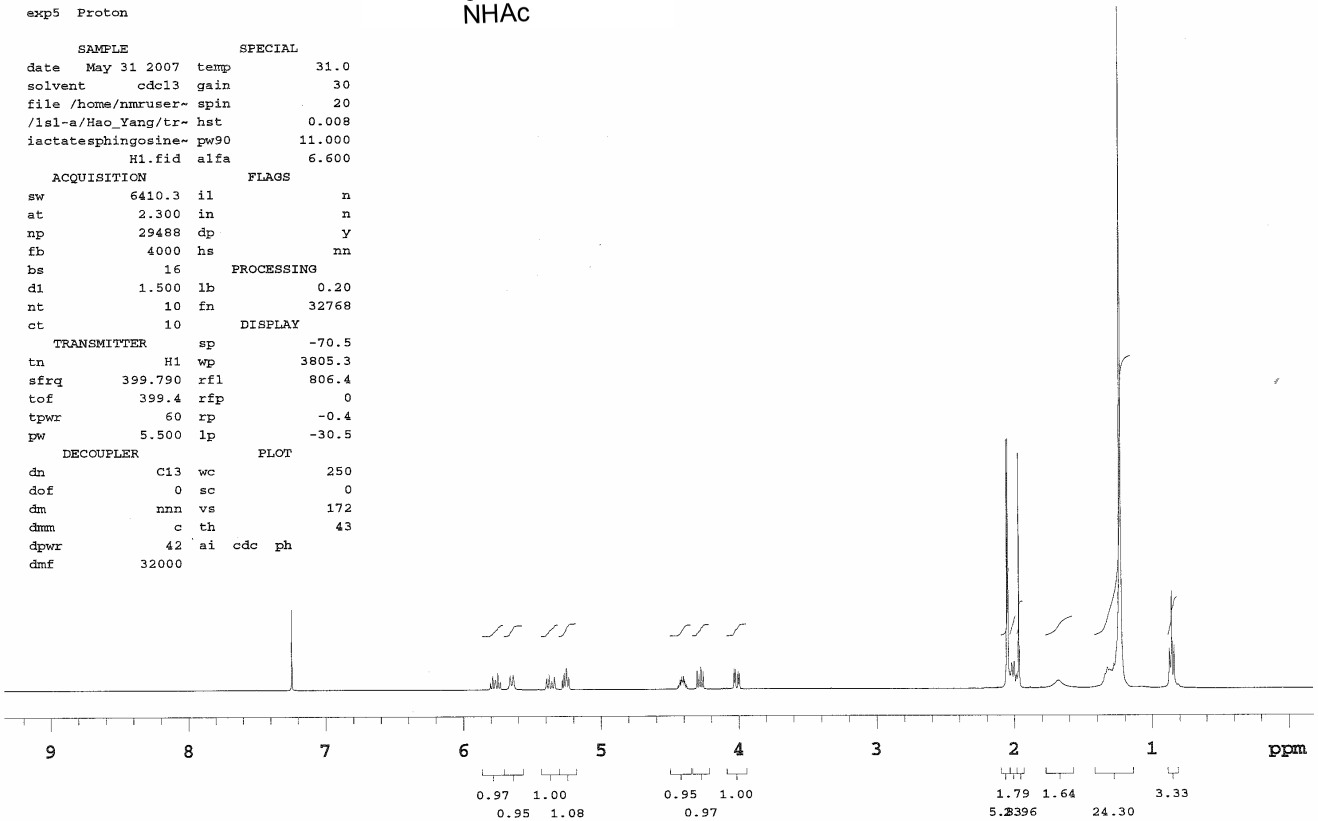
Scheme 2, Compound 1

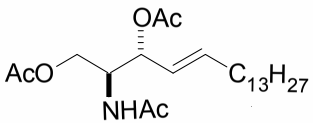




exp5 Proton

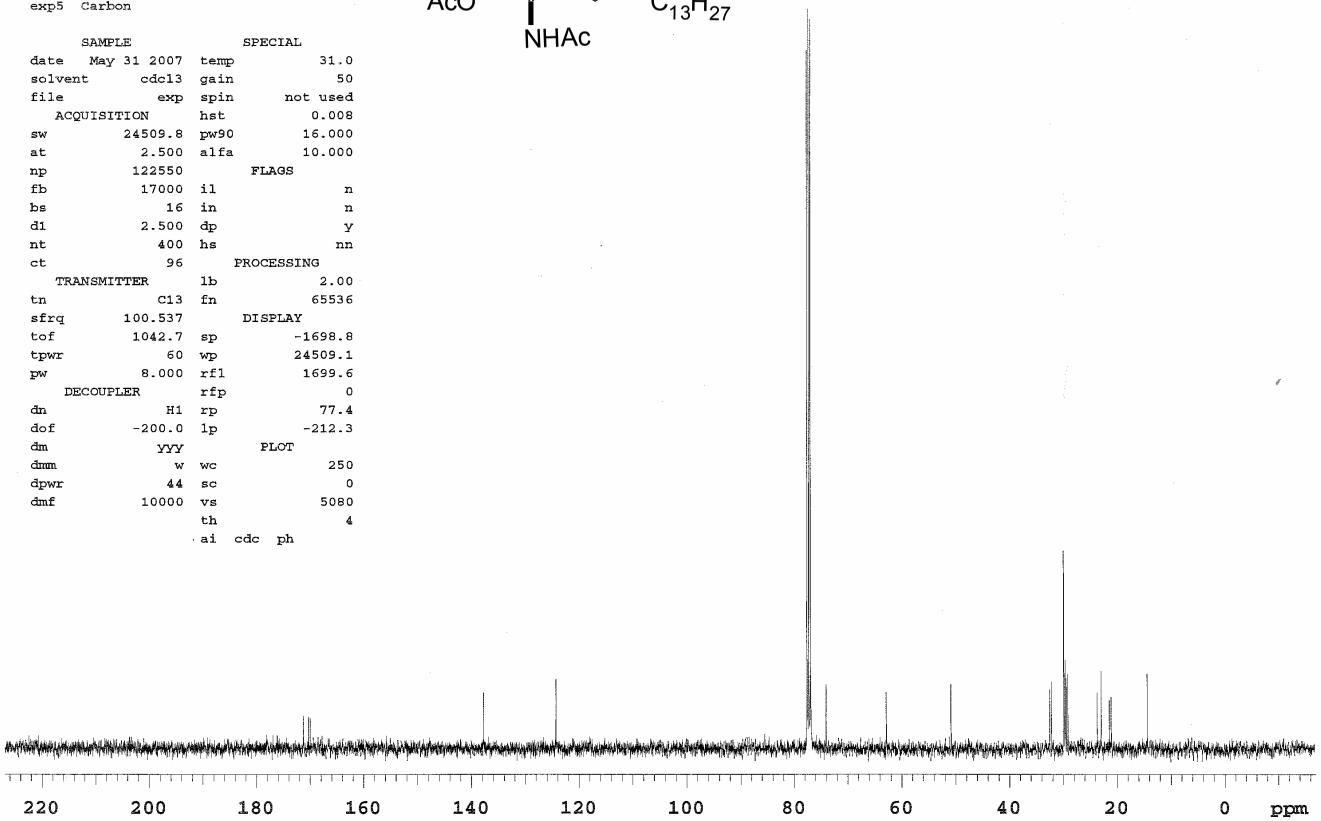
SAMPLE		SPECIAL	
date	May 31 2007	temp	31.0
solvent	cdcl3	gain	30
file	/home/nmruser~	spin	20
	/1sl-a/Hao_Yang/tr~	hst	0.008
	iactatesphingosine~	pw90	11.000
	H1.fid	alfa	6.600
ACQUISITION		FLAGS	
sw	6410.3	il	n
at	2.300	in	n
np	29488	dp	y
fb	4000	hs	nn
bs	16	PROCESSING	
d1	1.500	lb	0.20
nt	10	fn	32768
ct	10	DISPLAY	
TRANSMITTER		sp	-70.5
tn	H1	wp	3805.3
sfrq	399.790	rfl	806.4
tof	399.4	rfp	0
tpwr	60	rp	-0.4
pw	5.500	lp	-30.5
DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	sc	0
dm	nnn	vs	172
dmm	c	th	43
dpwr	42	ai cdc ph	
dmf	32000		





exp5 Carbon

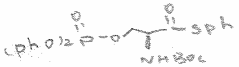
SAMPLE		SPECIAL	
date	May 31 2007	temp	31.0
solvent	cdcl3	gain	50
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	24509.8	pw90	16.000
at	2.500	alfa	10.000
np	122550	FLAGS	
fb	17000	il	n
bs	16	in	n
d1	2.500	dp	y
nt	400	hs	nn
ct	96	PROCESSING	
TRANSMITTER		lb	2.00
tn	C13	fn	65536
sfrq	100.537	DISPLAY	
tof	1042.7	sp	-1698.8
tpwr	60	wp	24509.1
pw	8.000	rf1	1699.6
DECOUPLER		rfp	0
dn	H1	rp	77.4
dof	-200.0	lp	-212.3
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	44	sc	0
dmf	10000	vs	5080
		th	4
		ai cdc ph	

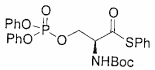


HYPhphosphateNbocSerineSph

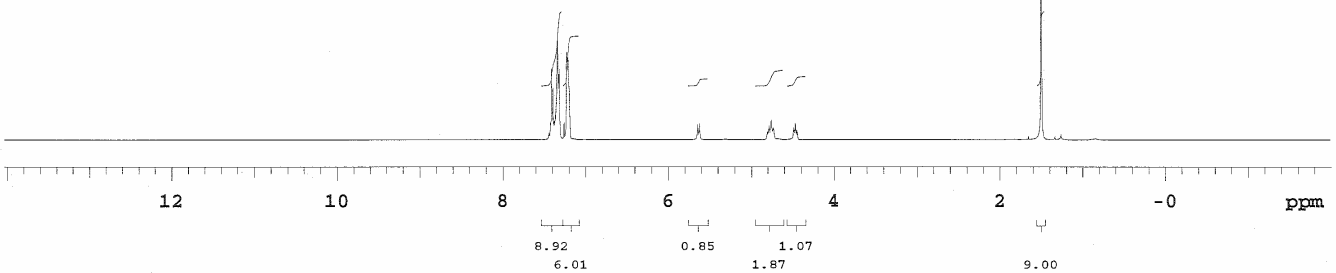
File : xp
Sample id : tmpstudy

Pulse Sequence: s2pul





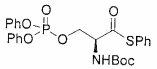
Scheme 3, Compound 13



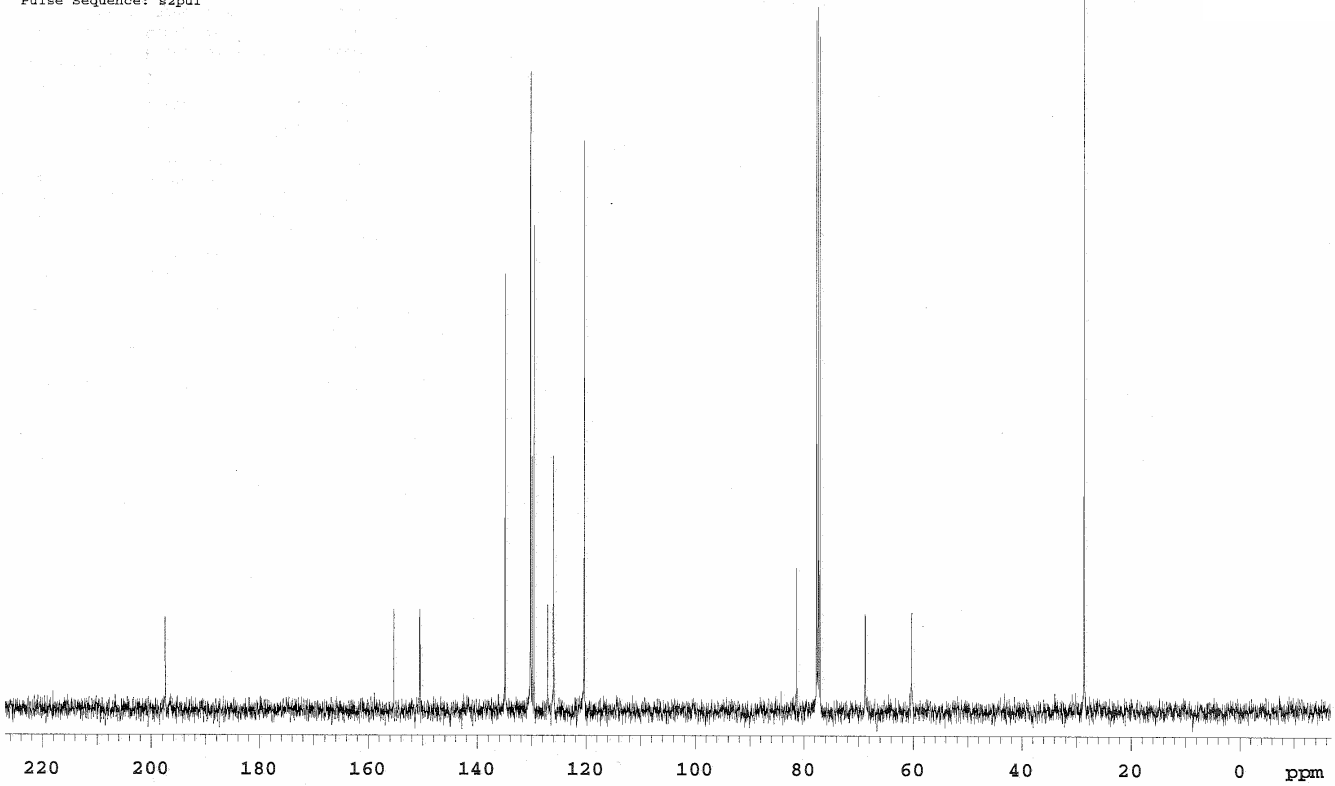
HYPhphosphateNbocSerineSph

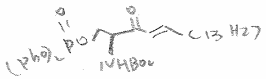
File : xp
Sample id : tmpstudy

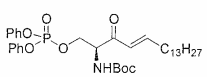
Pulse Sequence: s2pul



Scheme 3, Compound 13

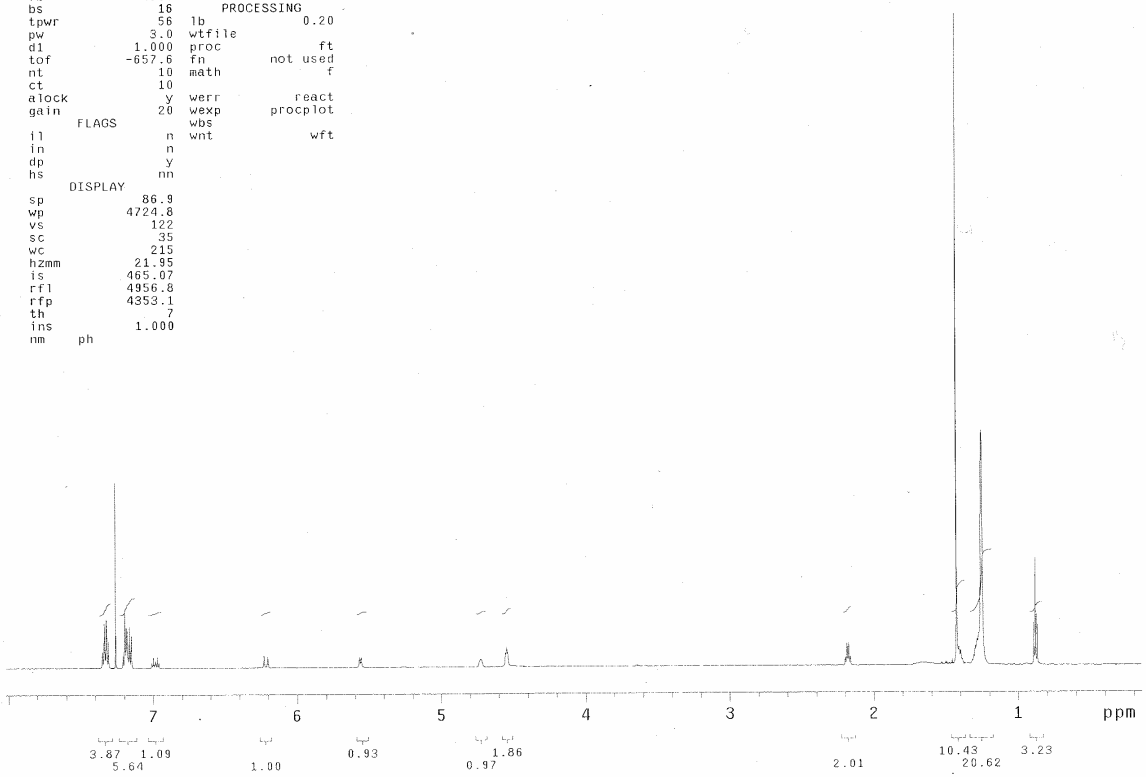






Scheme 3, Compound 14

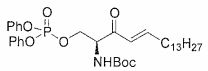
```
STANDARD PROTON PARAMETERS

exp1  s2pul

      SAMPLE                DEC. & VT
date    Jan 17 2006  dfrq        599.608
solvent       cdcl3  dn              H1
file            exp  dpwr            30
   ACQUISITION       dof              0
sfrq        599.607  dm             nnn
tn               H1  dmm              c
at            2.002  dmf            200
np            32832  dseq
sw           8201.8  dres           1.0
fb             4600  homo             n
bs               16      PROCESSING
tpwr             56  lb            0.20
pw              3.0  wtfile
d1            1.000  proc            ft
tof          -657.6  fn        not used
nt               10  math             f
ct               10
alock             y  werr         react
gain             20  wexp      procplot
      FLAGS          wbs
il                n  wnt            wft
in                n
dp                y
hs               nn
      DISPLAY
sp             86.9
wp           4724.8
vs              122
sc               35
wc              215
hzmm          21.95
is           465.07
rfl          4956.8
rfp          4353.1
th                7
ins           1.000
nm    ph
```



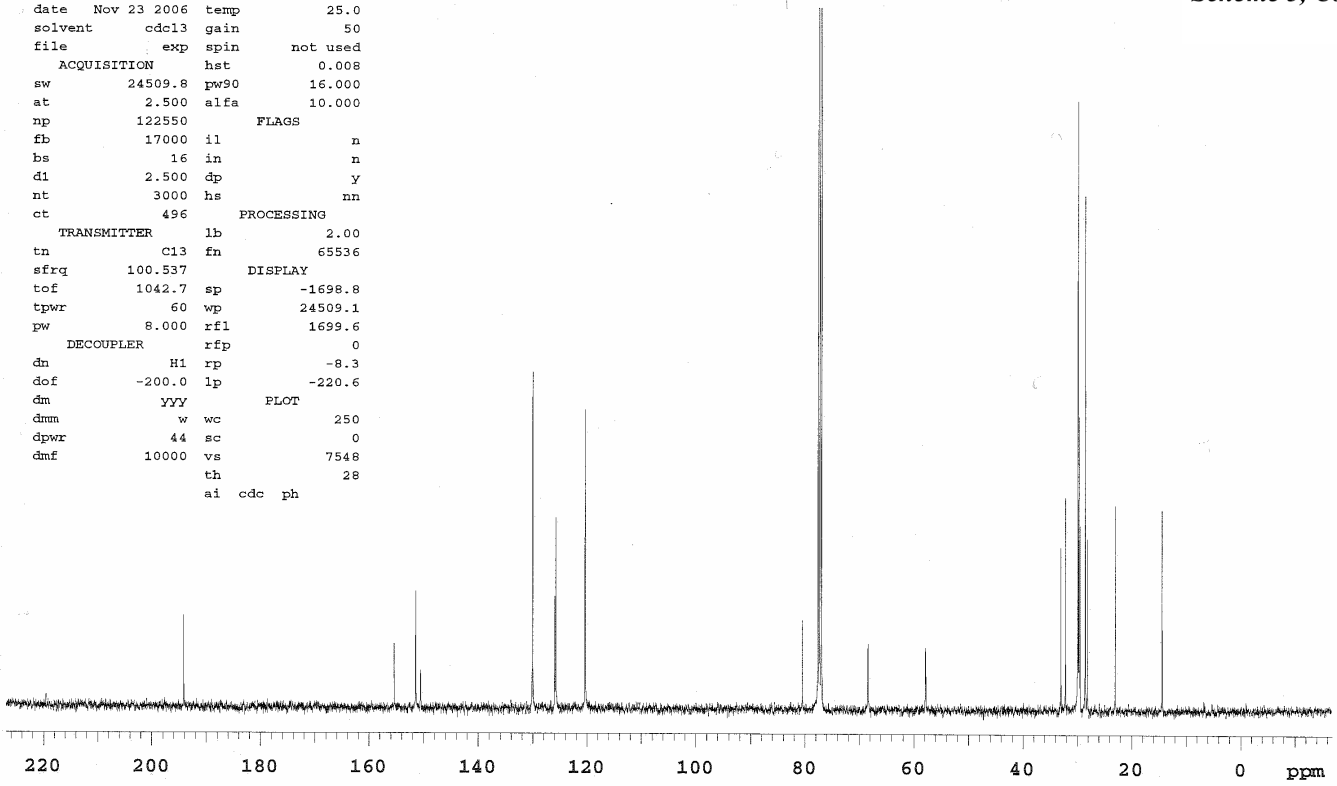
pbocserinene13

exp1 Carbon

SAMPLE		SPECIAL	
date	Nov 23 2006	temp	25.0
solvent	cdcl3	gain	50
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	24509.8	pw90	16.000
at	2.500	alfa	10.000
np	122550	FLAGS	
fb	17000	il	n
bs	16	in	n
d1	2.500	dp	y
nt	3000	hs	nn
ct	496	PROCESSING	
TRANSMITTER		lb	2.00
tn	C13	fn	65536
sfrq	100.537	DISPLAY	
tof	1042.7	sp	-1698.8
tpwr	60	wp	24509.1
pw	8.000	rfl	1699.6
DECOUPLER		rfp	0
dn	H1	rp	-8.3
dof	-200.0	lp	-220.6
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	44	sc	0
dmf	10000	vs	7548
		th	28
		ai cdc ph	

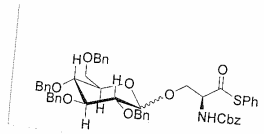


Scheme 3, Compound 14

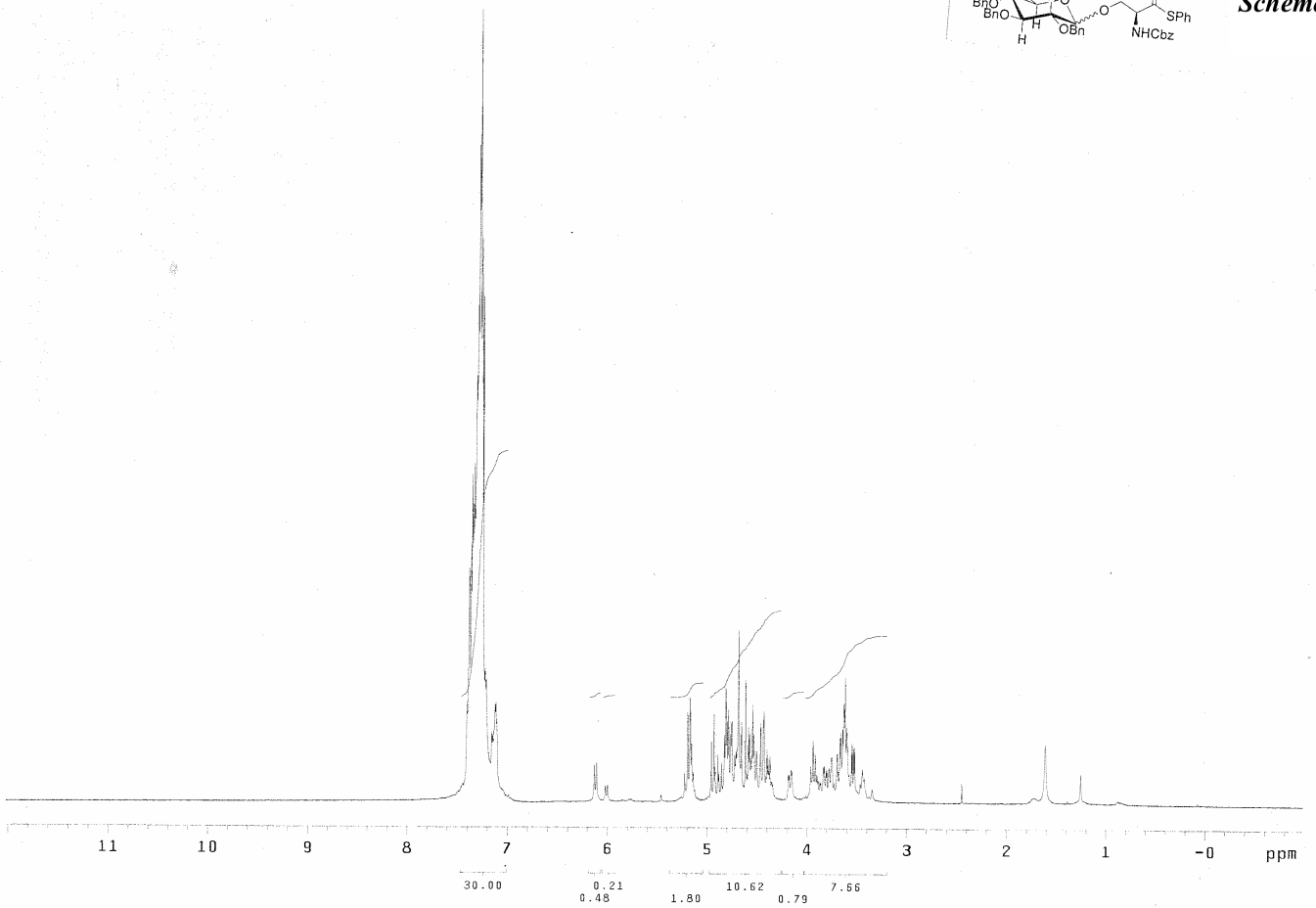


HYglycoNcbzserinesph

Pulse Sequence: s2pul

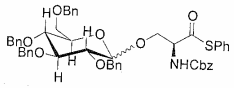


Scheme 3, Compound 15

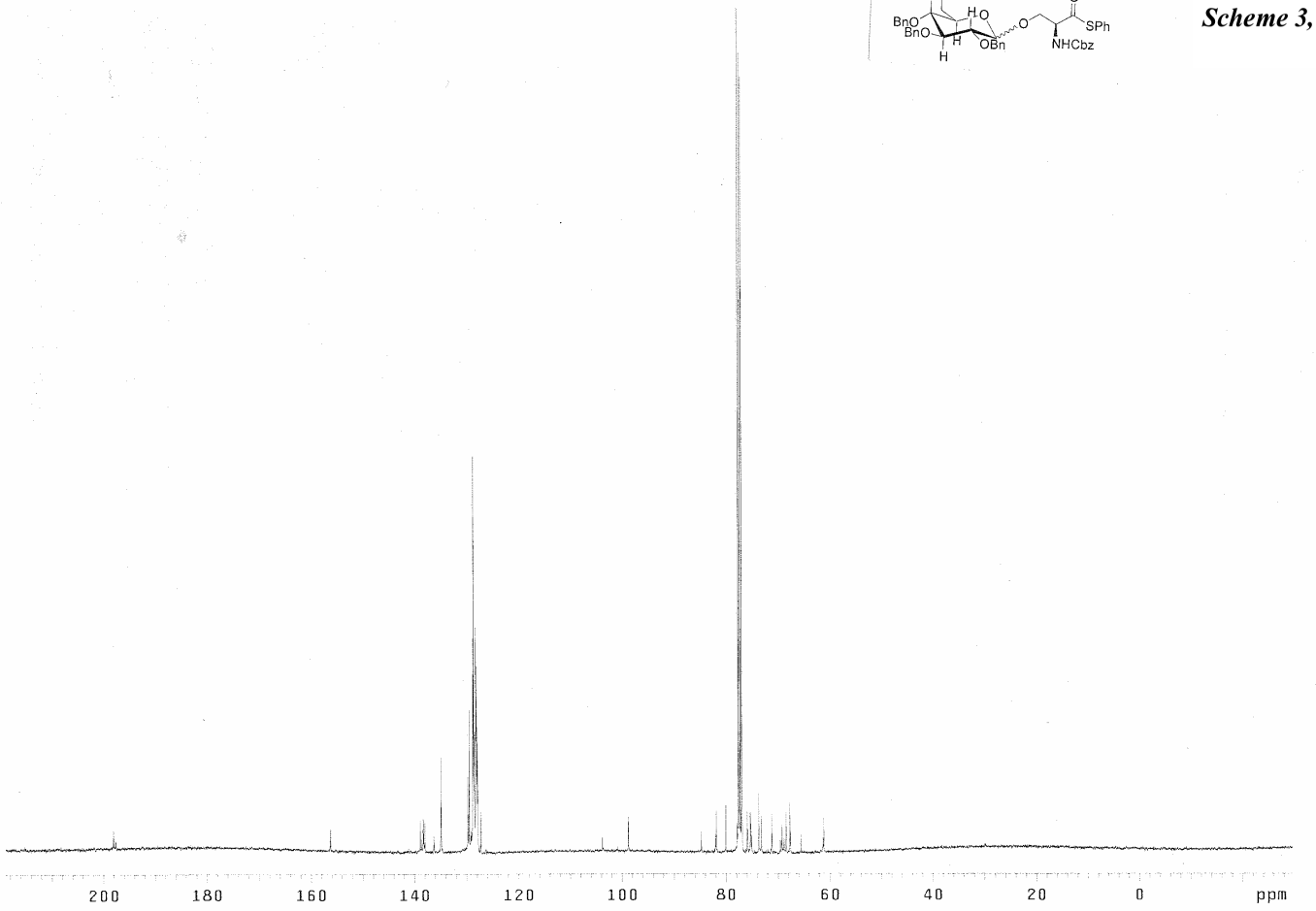


glycocbzserineSPh

Pulse Sequence: s2pul



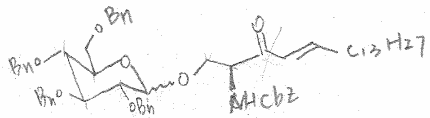
Scheme 3, Compound 15

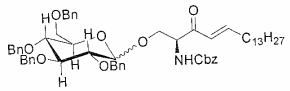


R467//400MHz//11-21-05

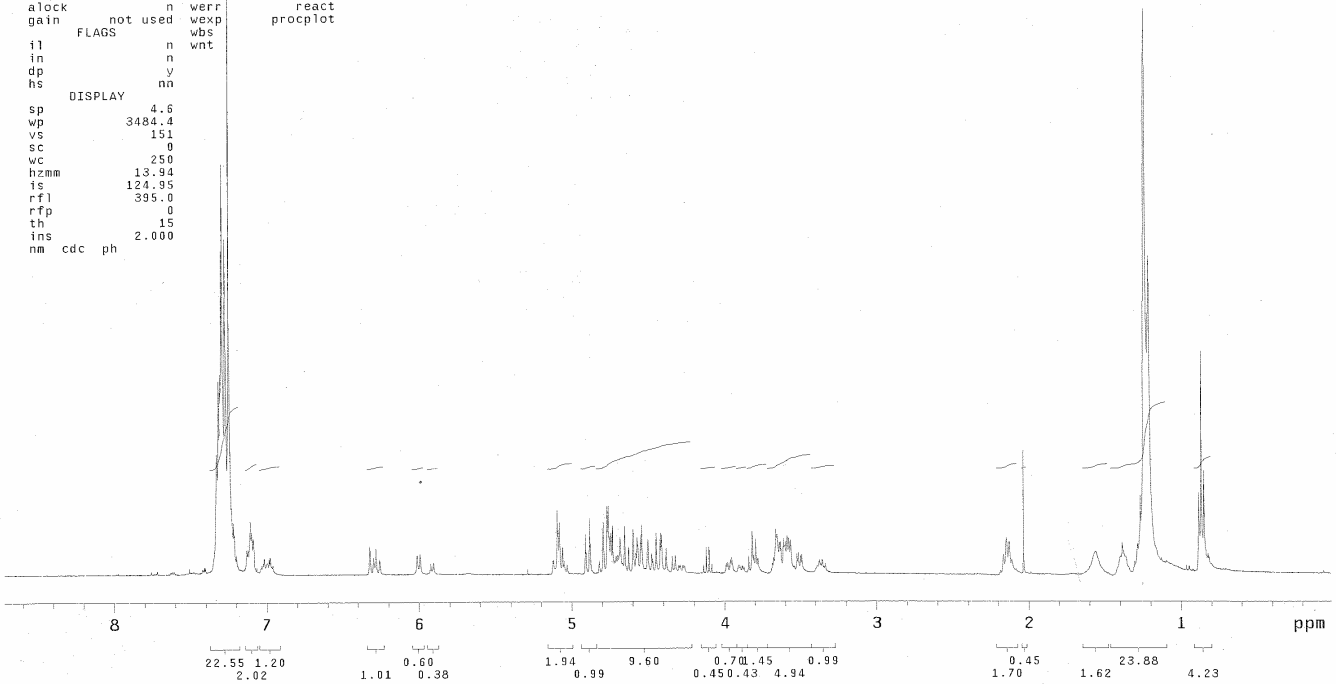
exp1 std1h

SAMPLE		DEC. & VT	
date	Nov 22 2005	dfrq	399.940
solvent	cdcl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	-630.7
sfrq	399.941	dm	nnn
tn	H1	dmm	c
at	2.202	dmf	200
np	22912	dseq	
sw	5203.3	dres	1.0
fb	3000	homo	n
bs	16	PROCESSING	
tpwr	58	lb	0.20
pw	8.0	wtfile	
d1	1.000	proc	ft
tof	207.0	fn	not used
nt	200	math	f
ct	200		
alock	n	werr	react
gain	not used	wexp	procplot
FLAGS		wbs	
il	n	wnt	
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	4.6		
wp	3484.4		
vs	151		
sc	0		
wc	250		
hzmm	13.94		
is	124.95		
rfl	395.0		
rfp	0		
th	15		
ins	2.000		
nm cdc ph			



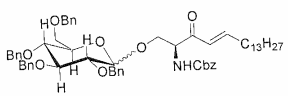


Scheme 3, Compound 16

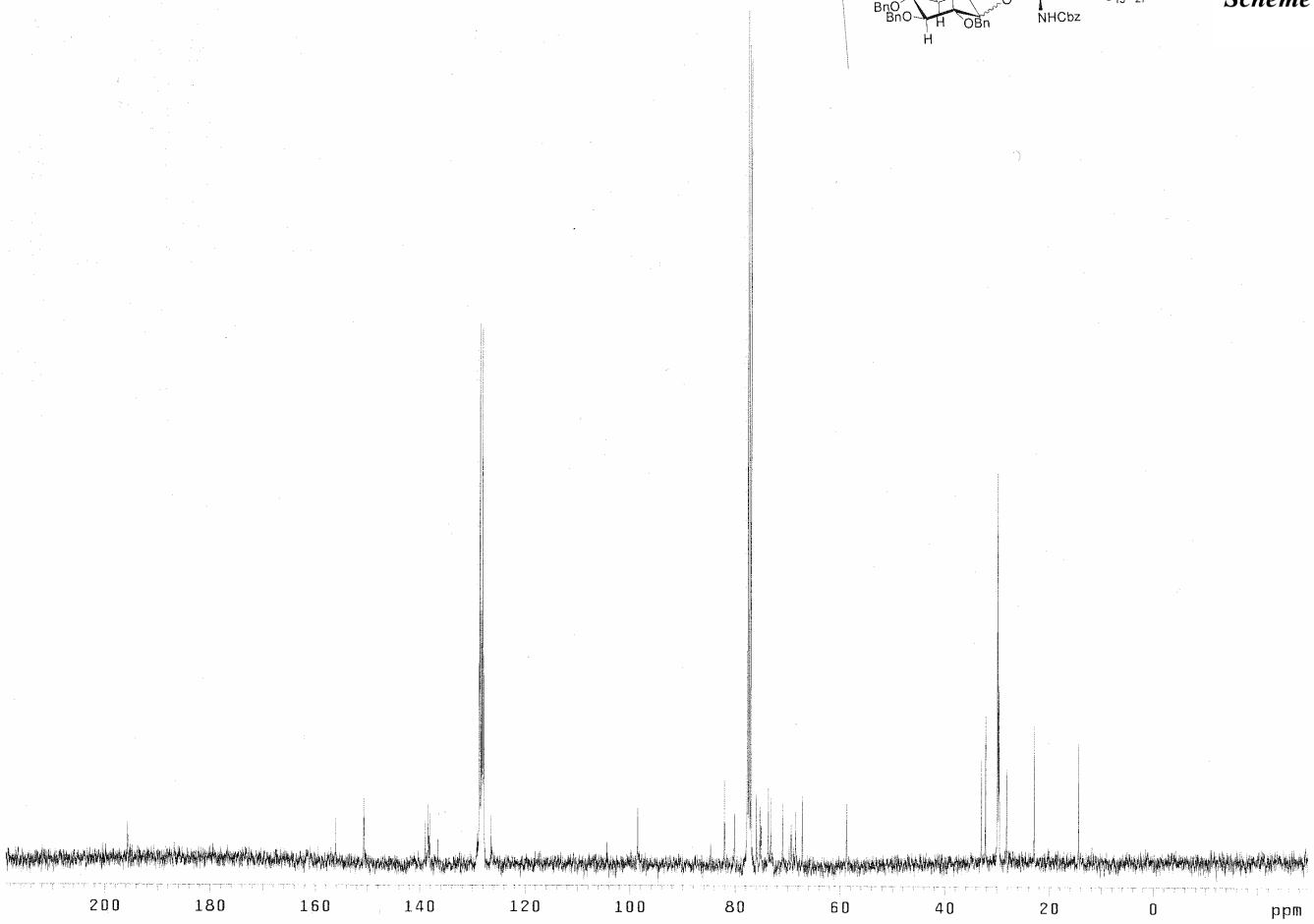


gcbzserinene13

Pulse Sequence: s2pul



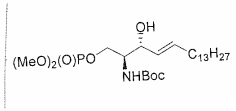
Scheme 3, Compound 16



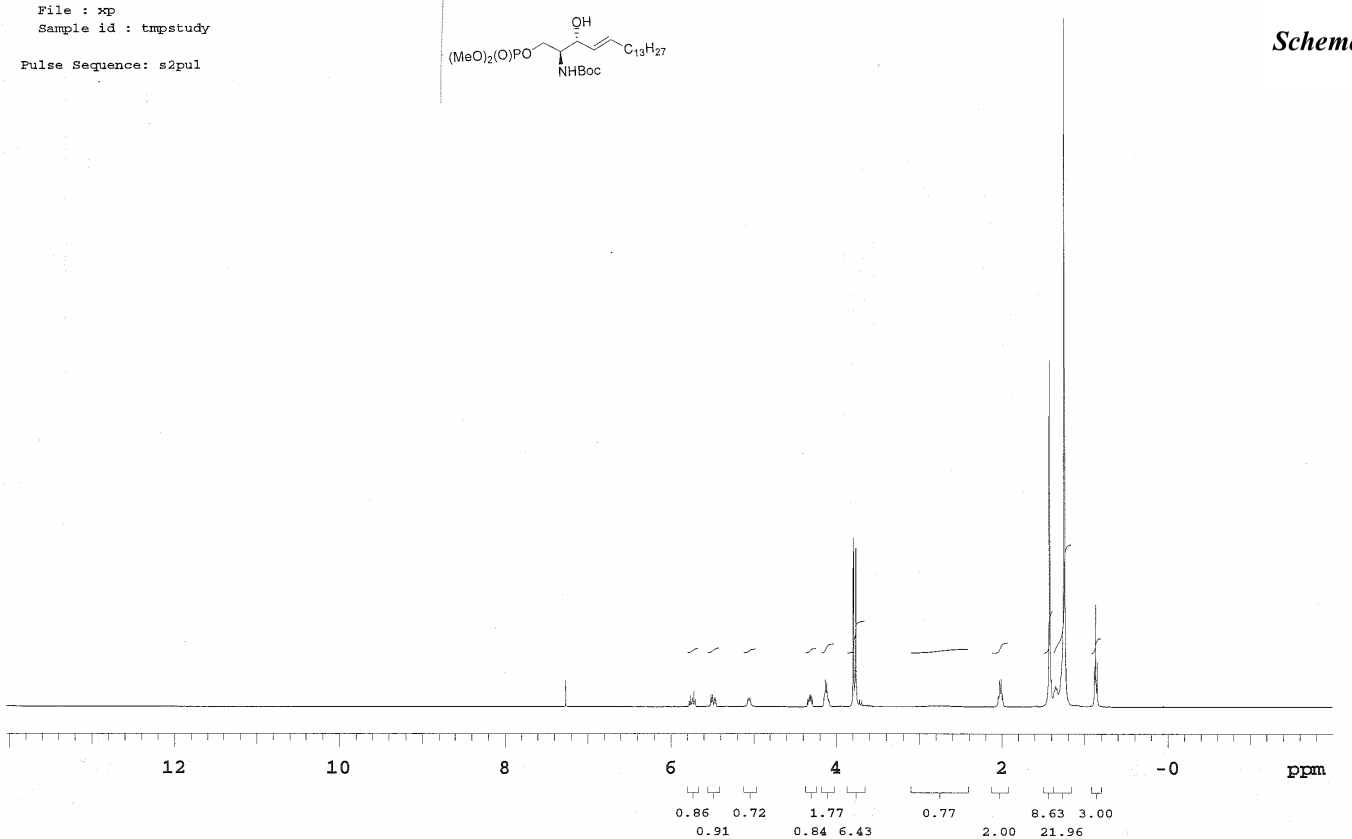
HY-400-NBocDimethylphosphateS1P

File : xp
Sample id : tmpstudy

Pulse Sequence: s2pul



Scheme 4, Compound 17

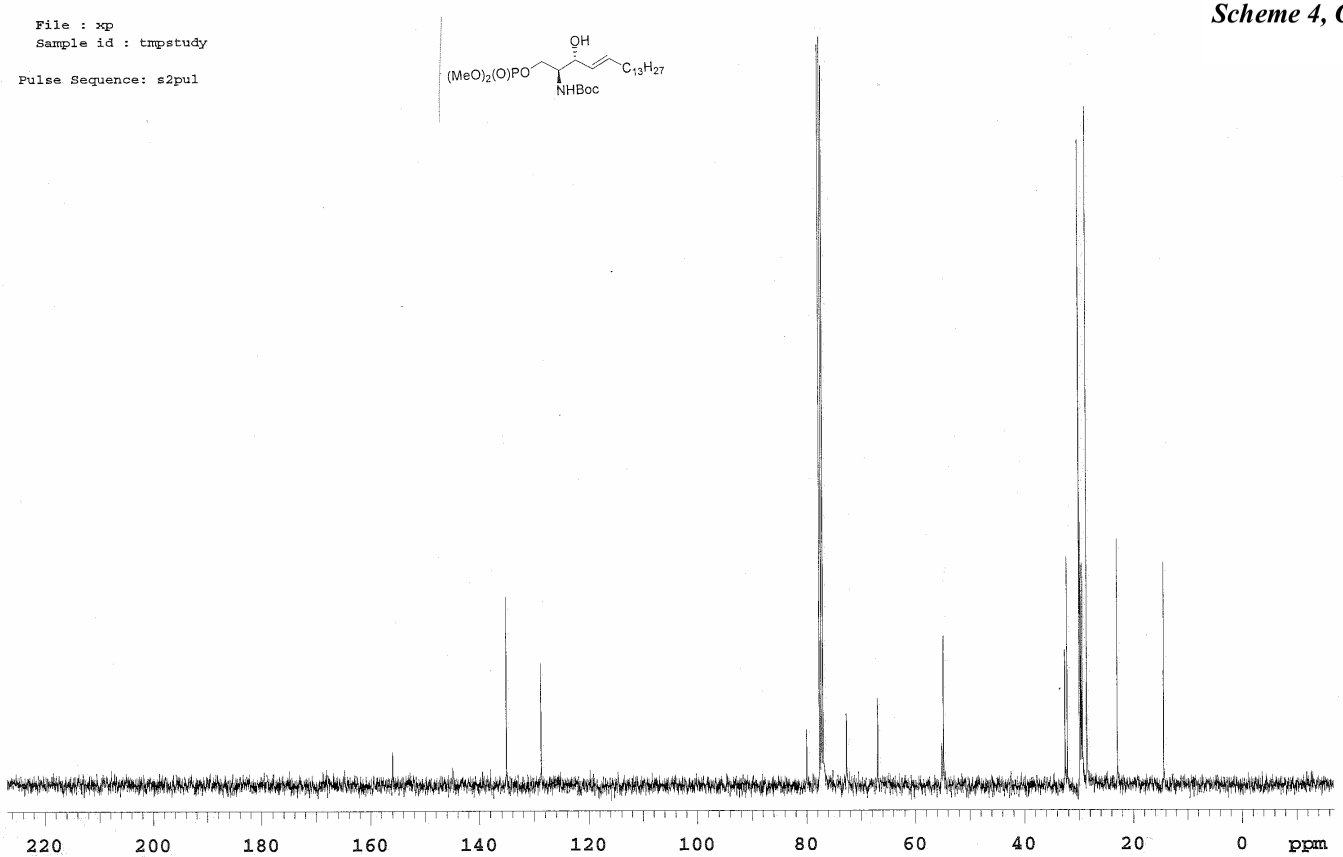


HY-400-NBocDimethylphosphateS1P

File : xp
Sample id : tmpstudy

Pulse Sequence: s2pul

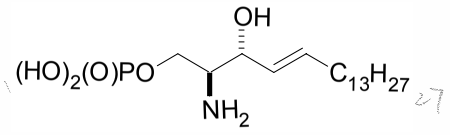
Scheme 4, Compound 17



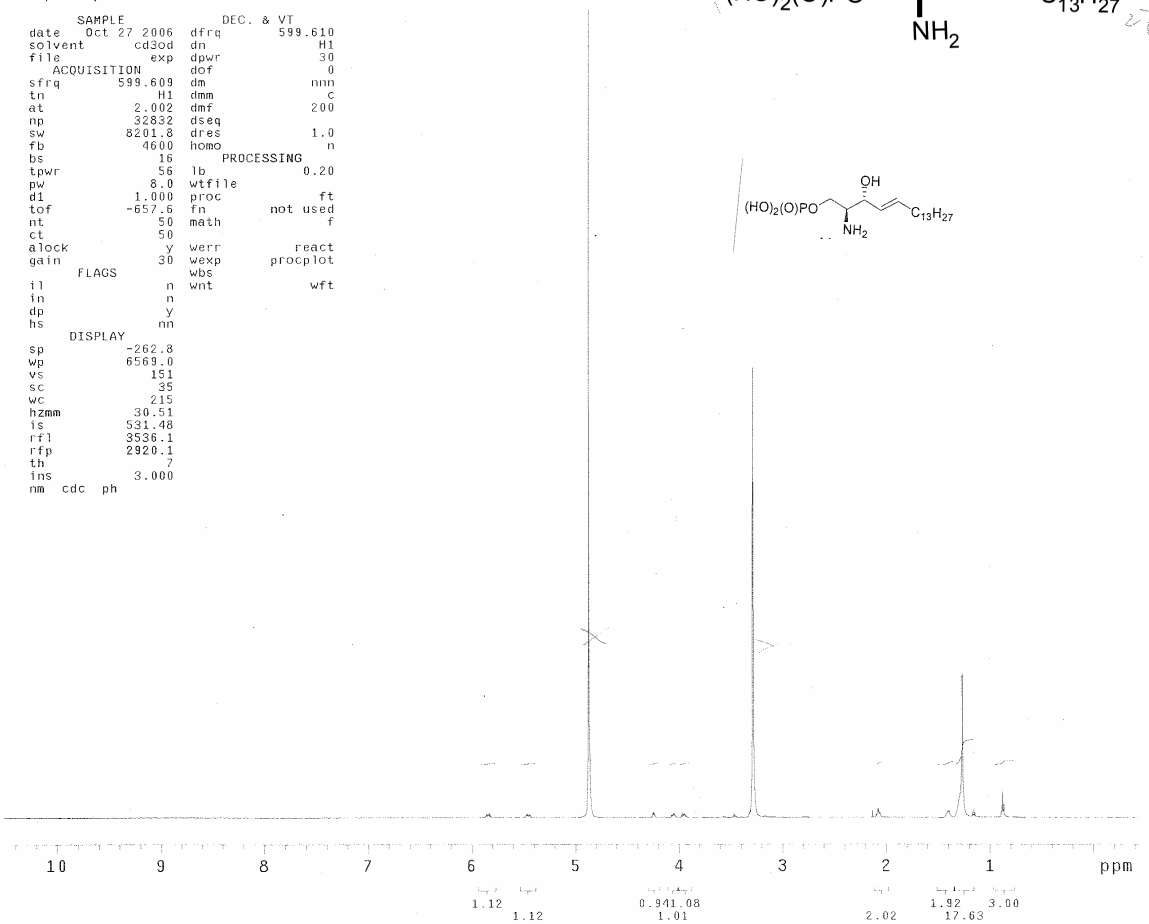
STANDARD PROTON PARAMETERS

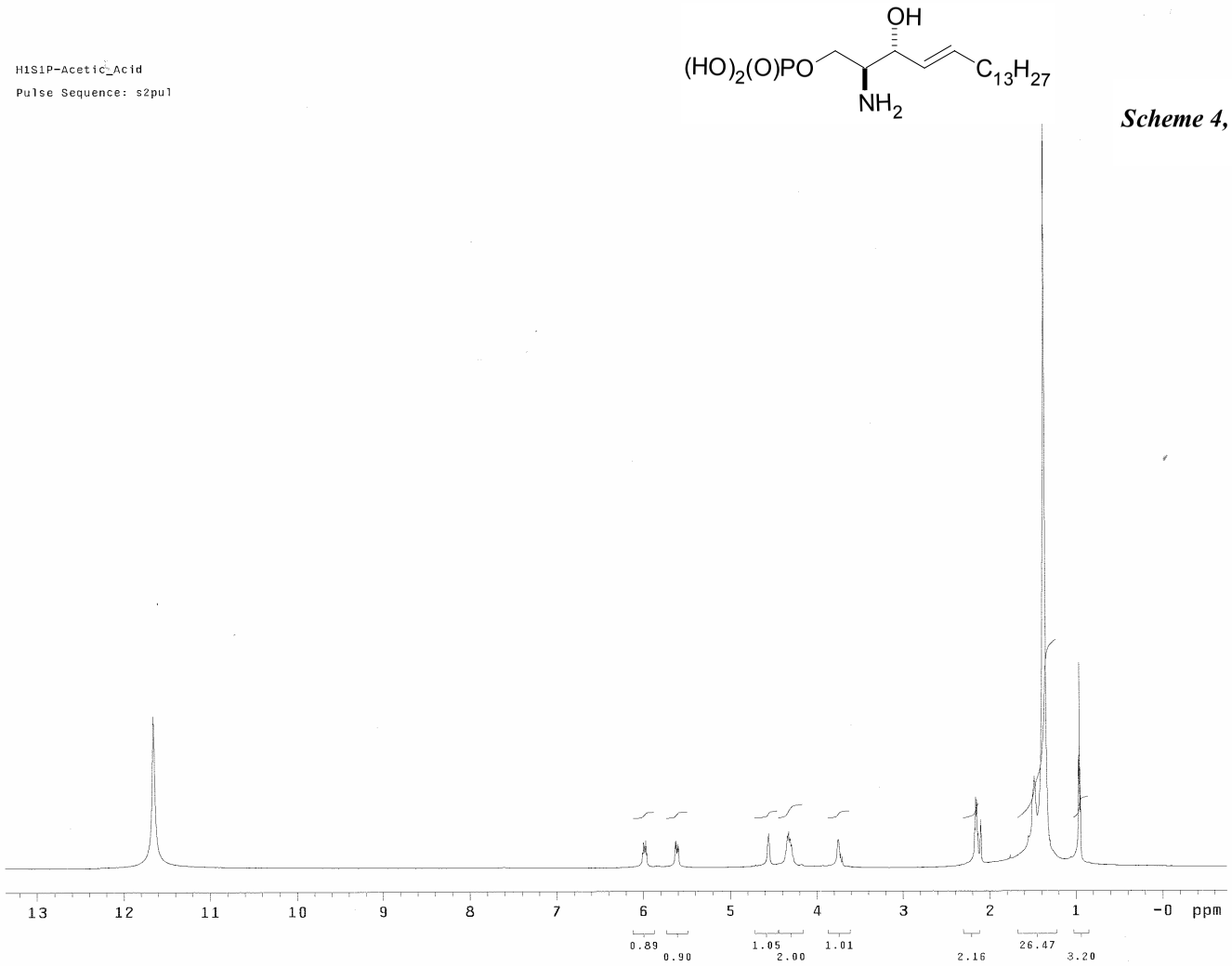
exp1 s2pul

SAMPLE		DEC. & VT	
date	Oct 27 2006	dfrq	599.610
solvent	cd3od	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	599.609	dm	nnn
tn	H1	dmm	c
at	2.002	dmf	200
np	32832	dseq	
sw	8201.8	dres	1.0
fb	4600	homo	n
bs	16	PROCESSING	
tpwr	56	lb	0.20
pw	8.0	wtfile	
d1	1.000	proc	ft
tof	-657.6	fn	not used
nt	50	math	f
ct	50		
alock	y	werr	react
gain	30	wexp	procplot
FLAGS		wbs	
il	n	wnt	wft
in	n		
dp	y		
hs	nn		
DISPLAY			
sp	-262.8		
wp	6569.0		
vs	151		
sc	35		
wc	215		
hzmm	30.51		
is	531.48		
rfl	3536.1		
rfp	2920.1		
th	7		
ins	3.000		
nm cdc ph			

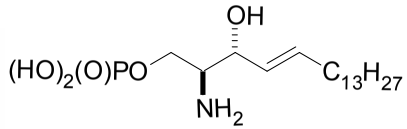


Scheme 4, Compound 18



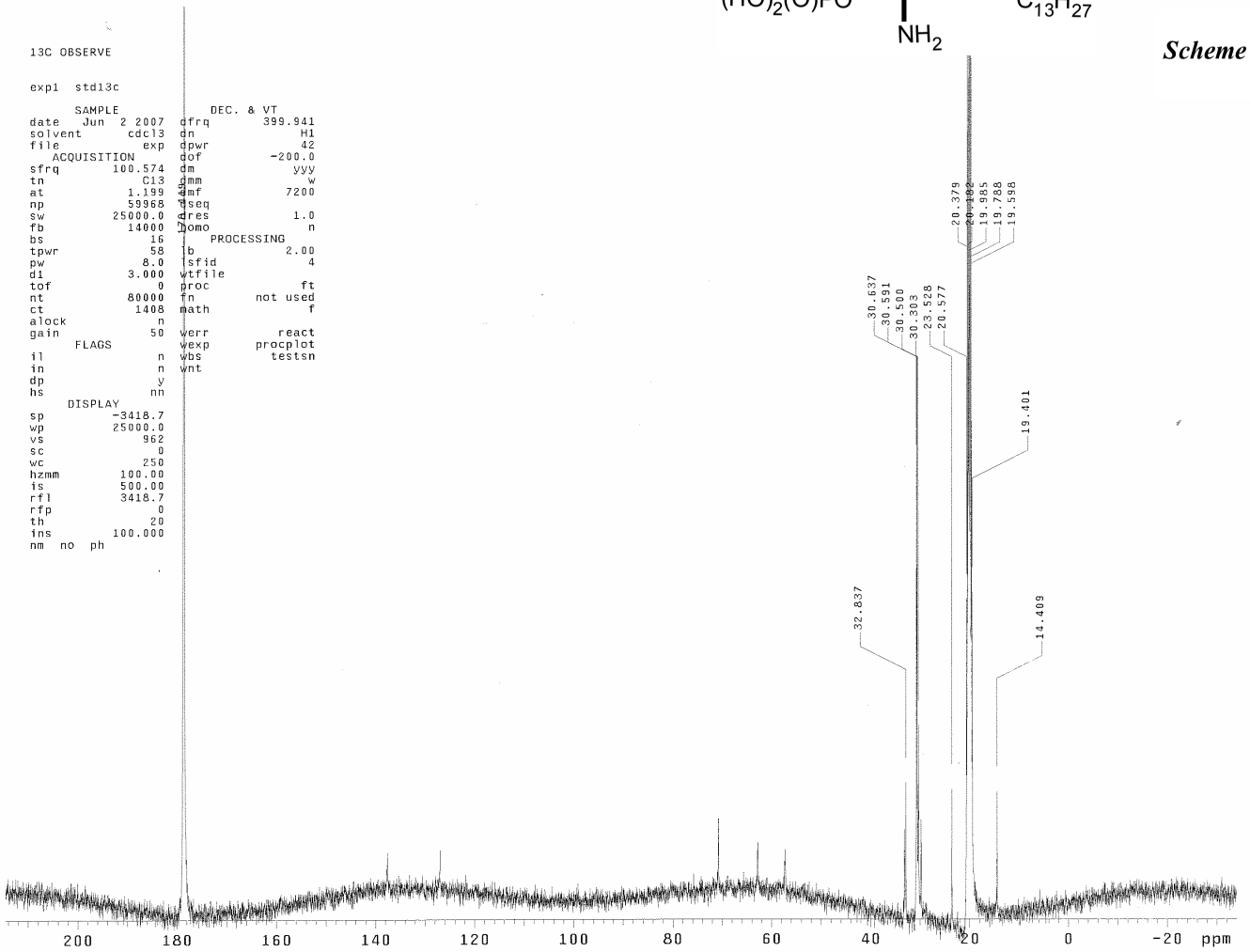


Scheme 4, Compound 18



Scheme 4, Compound 18

13C OBSERVE

exp1 std13c

SAMPLE		DEC. & VT	
date	Jun 2 2007	dfrq	399.941
solvent	cdcl3	dn	H1
file	exp	dpwr	42
ACQUISITION		dof	-200.0
sfrq	100.574	dm	yyy
tn	C13	dmm	w
at	1.199	dmf	7200
np	59968	dseq	
sw	25000.0	dres	1.0
fb	14000	homo	n
bs	16	PROCESSING	
tpwr	58	lb	2.00
pw	8.0	lsfid	4
d1	3.000	wtfile	
tof	0	proc	ft
nt	80000	fn	not used
ct	1408	math	f
alock	n		
gain	50	werr	react
FLAGS		wexp	procplot
il	n	wbs	testsn
in	n	wnt	
dp	y		
hs	nn		
DISPLAY			
sp	-3418.7		
wp	25000.0		
vs	962		
sc	0		
wc	250		
hzmm	100.00		
is	500.00		
rfl	3418.7		
rfp	0		
th	20		
ins	100.000		
nm no ph