Different *C*-glycosidation products of glucal with alkynyl or propargyl silanes under acidic conditions

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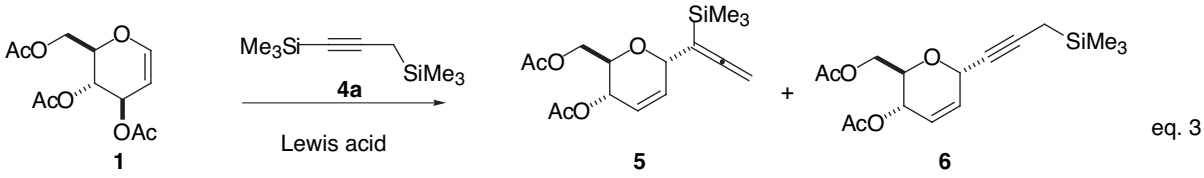
Supporting Information

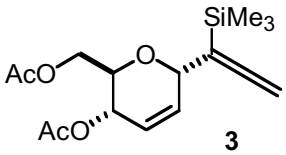
Experimental Section



eq. 3

entry	Lewis acid	solvent	time	(%)yield of **5+6**	**5/6**
1	$BF_3{\cdot}OEt_2$	CH_2Cl_2	1.0 h	74	1 : 2.5
2	$BF_3{\cdot}OEt_2$	CH_2Cl_2	1.5 h	87	1 : 2.5
3	$SnCl_4$	CH_2Cl_2	15 min	83	1 : 2
4	$SnCl_4$	CH_3CN	15 min	60	4 : 1

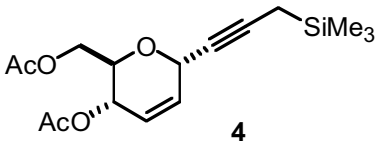
To a solution of **1** (0.05 g, 0.18 mmol) and 1,3-bis(trimethylsilyl)propyne **2** (0.08 g, 0.37 mmol) in 35 ml. CH_2Cl_2 was added dropwise the solution of $SnCl_4$ (0.02 ml, 0.18 mmol) in 5 ml CH_2Cl_2 at –20°C under argon atmosphere. The mixture was stirred for 15 min. and then poured into the mixture of saturated sodium hydrogen carbonate and saturated potassium sodium tartrate (1vol./1 vol.). After stirring at 0°C for 30 min, the organic layer was separated and the water layer was extracted with CH_2Cl_2. The combined organic layer was washed with water, brine, dried over anhydrous sodium sulfate and concentrated in vacuo. The residue was purified by silica gel column chromatrography (10% EtOAc/hexane) to afford a mixture of compound **3** and **4**.



3

[RNS-59-2, 49-2-2]

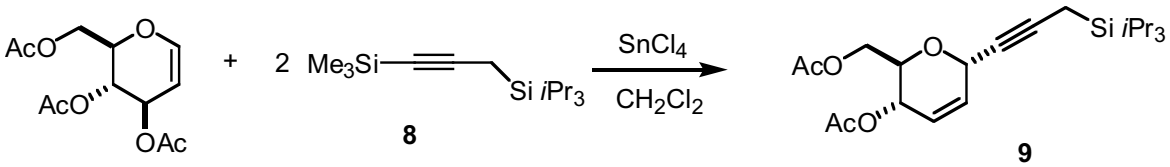
Compound 3. $[\alpha]_D^{25}$ 62.8° (c = 0.06, $CHCl_3$). IR (KBr) : 2955, 2926, 2854, 1933, 1744, 1370, 1233, 1047, 844 cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) : δ 0.11 (9H, s, $SiMe_3$), 2.05 (3H, s, *Ac*), 2.06 (3H, s, *Ac*), 3.74 (1H, ddd, *J* = 9.0, 6.0, 2.5 Hz, H-5), 4.12 (1H, dd, *J* = 12.0, 6 Hz, H-6a), 4.22 (1H, dd, *J* = 12.0, 2.5 Hz, H-6b), 4.46 (1H, dd, *J* = 12.0, 2.5 Hz, H-3'a), 4.50 (1H, dd, *J* = 12.0, 2.5 Hz, H-3'b), 4.85 (1H, m, H-1), 5.29 (1H, ddd, *J* = 9.0, 4.0, 2.0 Hz, H-4), 5.71 (1H, dt, *J* = 10.0, 2.0 Hz, H-2), 5.93 (1H, dt, *J* = 10.0, 2.0 Hz, H-3). ^{13}C-NMR (100 MHz, $CDCl_3$): δ -0.64, 20.8, 63.5, 65.5, 70.9, 74.4, 75.4, 97.0, 124.5, 133.1, 170.3, 170.9, 208.9. Anal. Calcd. For $C_{16}H_{24}O_5Si$: C, 59.23; H, 7.46 Found: C, 59.39; H, 7.50.



4

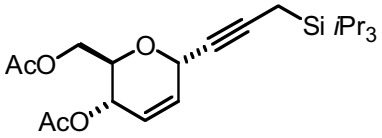
[RA-20-3 = RNS-21]

Compound 4. $[\alpha]_D^{26}$ –64.0° (c = 0.84, $CHCl_3$). IR (KBr) : 2956, 2891, 2211, 1743, 1370, 1235, 1051, 853, 699 cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) : δ 0.09 (9H, s, $SiMe_3$), 1.49 (2H, d, *J* = 2.0 Hz, H-3'), 2.06 (3H, s, *Ac*), 2.07 (3H, s, *Ac*), 4.09 (1H, dt, *J* = 9.0, 4.0 Hz, H-5), 4.18 (2H, d, *J* = 4.0 Hz, H-6), 4.92 (1H, brs, H-1), 5.24 (1H, ddd, *J* = 9.0, 4.0, 2.0 Hz, H-4), 5.68 (1H, dt, *J* = 10.0, 2.0 Hz, H-2), 5.84 (1H, ddd, *J* = 10.0, 4.0, 2.0 Hz, H-3). ^{13}C-NMR (125 MHz, $CDCl_3$): δ -2.1, 7.2, 20.8, 20.9, 63.3, 64.5, 65.0, 69.5, 74.6, 85.9, 124.4, 130.4, 170.3, 170.9. Anal. Calcd. For $C_{16}H_{24}O_5Si$: C, 59.23; H, 7.46 Found: C, 59.38; H, 7.16.



1 **8** **9**

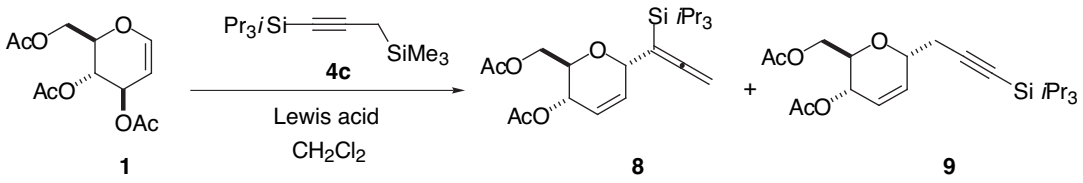
To a solution of **1** (0.05 g, 0.18 mmol) and 1-trimethylsilyl-3-triisopropylsilylpropyne **8** (0.06 g, 0.22 mmol) in 35 ml. CH_2Cl_2 was added dropwise the solution of $SnCl_4$ (0.02 ml, 0.18 mmol) in 5 ml CH_2Cl_2 at –25°C under argon atmosphere. The mixture was stirred for 15 min. and then poured into the mixture of saturated sodium hydrogen carbonate and saturated potassium sodium tartrate (1vol./1 vol.). After stirring at 0°C for 30 min, the organic layer was separated and the water layer was extracted with CH_2Cl_2. The combined organic layer was washed with water, brine, dried over anhydrous sodium sulfate and concentrated in vacuo. The residue was purified by silica gel column chromatrography (10% EtOAc/hexane) to afford the product **9**.



9

[RNS-28]

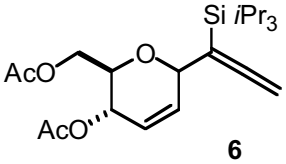
Compound 9. $[\alpha]_D^{26}$ –53.0° (c = 0.59, $CHCl_3$). IR (KBr) : 2943, 2867, 2212, 1747, 1465, 1369, 1240, 1054, 883 cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) : δ 1.06, 1.07 (18H, s, 3xSiCH(CH_3)$_2$), 1.12 (3H, m, 3xSiCHMe$_2$), 1.56 (2H, d, *J* = 2.5 Hz, H-3'), 2.06 (3H, s, *Ac*), 2.07 (3H, s, *Ac*), 4.08 (1H, ddd, *J* = 9.0, 5.0, 2.5 Hz, H-5), 4.14 (1H, dd, *J* = 12.0, 2.5 Hz, H-6a), 4.23 (1H, dd, *J* = 12.0, 5.0 Hz, H-6b), 4.90 (1H, brs, H-1), 5.26 (1H, ddd, *J* = 9.0, 4.0, 2.0 Hz, H-4), 5.68 (1H, dt, *J* = 10.0, 2.0 Hz, H-2), 5.83 (1H, ddd, *J* = 10.0, 4.0, 2.0 Hz, H-3). ^{13}C-NMR (75 MHz, $CDCl_3$): δ -0.9, 10.9, 18.4, 20.7, 20.9, 63.2, 64.5, 64.9, 69.4, 74.4, 86.7, 124.3, 130.4, 170.5, 171.1. Anal. Calcd. For $C_{22}H_{36}O_5Si$: C, 64.67; H, 8.88 Found: C, 64.67; H; 8.91



1 **8** **9**

entry	equiv. of **4**	Lewis acid	solvent	time	(%) yield of **8+9**	**8/9**
1	2	$SnCl_4$	CH_2Cl_2	15 min	74	1 : 2.5
2	1.1	$TiCl_4$	CH_2Cl_2	15 min	87	1 : 2.5
3	1.1	$SnCl_4$	CH_2Cl_2	45 min	69	1 : 5
4	1.1	$SnCl_4$	CH_3CN	1 h	58	1 : 9
5	1.1	$SnCl_4$	CH_3CN	15 min	60	1 : 9
6	0.5	$SnCl_4$	CH_3CN	1.5 h	30	1 : 80

To a solution of **1** (0.05 g, 0.18 mmol) and 1-triisopropylsilyl-3-trimethylsilylpropyne **5** (0.10 g, 0.37 mmol) in 35 ml. CH_2Cl_2 was added dropwise the solution of $SnCl_4$ (0.02 ml, 0.18 mmol) in 5 ml CH_2Cl_2 at –20ºC under argon atmosphere. The mixture was stirred for 15 min. and then poured into the mixture of saturated sodium hydrogen carbonate and saturated potassium sodium tartrate (1vol./1 vol.). After stirring at 0ºC for 30 min, the organic layer was separated and the water layer was extracted with CH_2Cl_2. The combined organic layer was washed with water, brine, dried over anhydrous sodium sulfate and concentrated in vacuo. The residue was purified by silica gel column chromatrography (10% EtOAc/hexane) to afford a mixture of compound **6** and **7**.



6

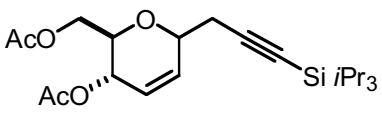
[RNS-52-1-2]

Compound 6. $[\alpha]_D^{27}$ –22.9º (c = 0.24, $CHCl_3$). IR (KBr) : 2943, 2866, 1922, 1747, 1237, 1049, 804 cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) : δ 1.06, 1.07, 1.08, 1.09 (18H, s, 3xSiCH(C*H*$_3$)$_2$), 1.25 (3H, m, 3xSiCHMe$_2$), 2.04 (3H, s, *Ac*), 2.06 (3H, s, *Ac*), 3.96 (1H, ddd, *J* = 8.5, 5.5, 2.5 Hz, H-5), 4.10 (1H, dd, *J* = 12.0, 2.5 Hz, H-6a), 4.15 (1H, dd, *J* = 12.0, 5.5 Hz, H-6b), 4.50 (2H, m, H-3'), 4.77 (1H, m, H-1), 5.20 (1H, ddd, *J* = 10.0, 3.0, 1.5 Hz, H-4), 5.79 (1H, dt, *J* = 10.5, 1.5 Hz, H-2), 5.85 (1H, ddd, *J* = 10.5, 3.0, 1.5 Hz, H-3).

^{13}C-NMR (75 MHz, $CDCl_3$): δ 11.9, 12.1, 18.5, 18.6, 20.8, 21.1, 29.7, 63.4, 65.4, 67.8, 70.6,

71.2, 74.5, 90.1, 124.0, 131.6, 170.4, 171.0, 213.4.

Anal. Calcd. For $C_{22}H_{36}O_5Si$: C, 64.67; H, 8.88 Found: C, 64.67; H, 8.93

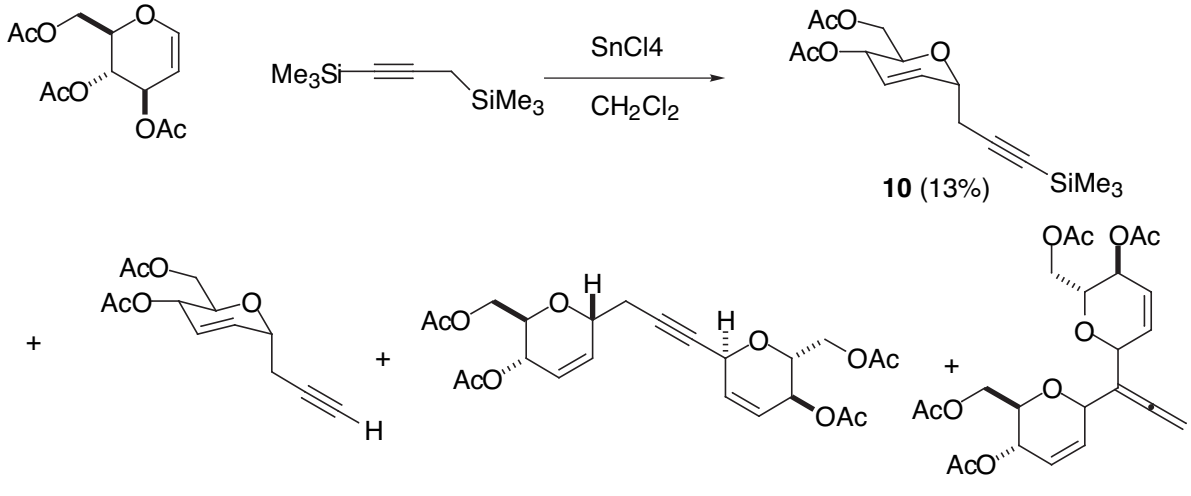


7

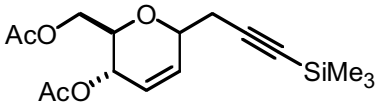
[RNS-57-P]

Compound 7. $[\alpha]_D^{27}$ –15.0° (c = 1.45, $CHCl_3$). IR (KBr) : 2942, 2865, 2175, 1747, 1464, 1369, 1233, 1022, 883, 678 cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) : δ 1.02, 1.03 (18H, s, 3xSiCH(CH_3)$_2$), 1.03 (3H, m, 3xSiCHMe$_2$), 2.05 (3H, s, *Ac*), 2.07 (3H, s, *Ac*), 2.57 (1H, dd, *J* = 16.5, 8.0 Hz, H-1'a), 2.70 (1H, dd, *J* = 16.5, 5.0 Hz, H-1'b), 4.07 (1H, td, *J* = 6.0, 3.5 Hz, H-5), 4.14 (1H, dd, *J* = 12.0, 3.5 Hz, H-6a), 4.21 (1H, dd, *J* = 12.0, 6.0 Hz, H-6b), 4.36 (1H, m, H-1), 5.12 (1H, ddd, *J* = 6.0, 4.5, 2.5 Hz, H-4), 5.85 (1H, dt, *J* = 10.5, 2.5 Hz, H-2), 6.10 (1H, ddd, *J* = 10.5, 2.5, 2.0 Hz, H-3). ^{13}C-NMR (75 MHz, $CDCl_3$): δ 11.2, 18.6, 20.8, 21.0, 62.7, 64.8, 70.5, 83.7, 103.8, 124.6, 131.7, 170.4, 170.8

Anal. Calcd. For $C_{22}H_{36}O_5Si$: C, 64.67; H, 8.88 Found: C, 64.68; H, 8.94



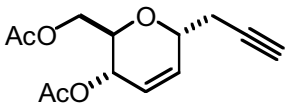
To a solution of **1** (0.10 g, 0.37 mmol) and 1,3-bis(trimethylsilyl)propyne **2** (0.04 g, 0.19 mmol) in 35 ml. CH_2Cl_2 was added dropwise the solution of $SnCl_4$ (0.04 ml, 0.37 mmol) in 5 ml CH_2Cl_2 at –20°C under argon atmosphere. The mixture was stirred for 40 min. and then poured into the mixture of saturated sodium hydrogen carbonate and saturated potassium sodium tartrate (1vol./1 vol.). After stirring at 0°C for 30 min, the organic layer was separated and the water layer was extracted with CH_2Cl_2. The combined organic layer was washed with water, brine, dried over anhydrous sodium sulfate and concentrated in vacuo. The residue was purified by silica gel column chromatrography (30-50% EtO_2/hexane) to afford a mixture of compound **10**, **11**, **12** and **13**.



10

[RNS-55-1, RNS-59-1]

Compound 10. IR (KBr) : 2957, 1743, 1606, 1370, 1236, 843 cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) : δ 0.14 (9H, s, $SiMe_3$), 2.06 (3H, s, *Ac*), 2.07 (3H, s, *Ac*), 2.61 (1H, dd, *J* = 14.0, 7.0 Hz, H-1'a), 2.75 (1H, dd, *J* = 16.5, 7.5 Hz, H-1'b), 3.95 (1H, m, H-5), 4.12 (1H, dd, *J* = 12.0, 4.0 Hz, H-6a), 4.28 (1H, dd, *J* = 12.0, 6.5 Hz, H-6b), 4.61 (1H, m, H-1), 5.10 (1H, m, H-4), 5.85 (1H, ddd, *J* = 10.0, 3.5, 2.0 Hz, H-2), 5.96 (1H, ddd, *J* = 10.0, 2.0, 1.5 Hz, H-3).

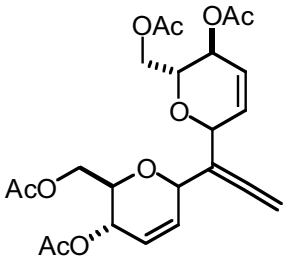


11

[RNS-49-3, RNS-55-2]

Compound 11. $[\alpha]_D^{25}$ +29.4° (c = 0.62, $CHCl_3$). IR (KBr) : 3284, 2923, 1740, 1370, 1234, 1046 cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) : δ 2.04 (1H, t, *J* = 2.5 Hz, H-3'), 2.06 (3H, s, *Ac*), 2.07 (3H, s, *Ac*), 2.47 (1H, ddd, *J* = 16.5, 7.5, 2.5 Hz, H-1'a), 2.58 (1H, ddd, *J* = 16.5, 6.5, 2.5 Hz, H-1'b), 3.98

(1H, ddd, *J* = 9.0, 6.5, 3.5 Hz, H-5), 4.14 (1H, dd, *J* = 12.0, 3.5 Hz, H-6a), 4.24 (1H, dd, *J* = 12.0, 6.5 Hz, H-6b), 4.38 (1H, m, H-1), 5.12 (1H, m, H-4), 5.86 (1H, ddd, *J* = 10.0, 3.0, 2.0 Hz, H-2), 6.07 (1H, ddd, *J* = 10.0, 2.5, 1.5 Hz, H-3). ^{13}C-NMR (75 MHz, $CDCl_3$): δ 20.8, 21.1, 23.9, 62.6, 64.7, 69.9, 70.4, 70.7, 79.8, 124.6, 131.6, 170.4, 170.8. Anal. Calcd. For $C_{13}H_{16}O_5$: C, 61.90; H, 6.39 Found: C, 61.93; H, 6.36

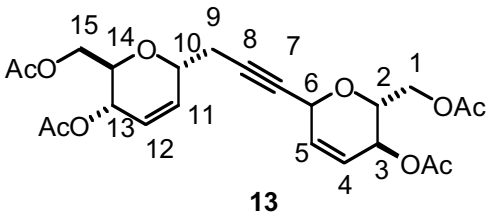


12

[RNS-55-3]

Compound 12. $[\alpha]_D^{27}$ –145.0° (c = 1.1, $CHCl_3$). IR (KBr) : 2952, 1953, 1740, 1371, 1235, 1049 cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) : δ 2.06 (3H, s, *Ac*), 2.07 (3H, s, *Ac*), 3.80 (1H, ddd, *J* = 9.0, 5.0, 4.0 Hz, H-5), 4.14 (2H, m, H-6), 4.95 (1H, m, H-1), 5.24 (1H, ddd, *J* = 9.0, 3.0, 2.0 Hz, H-4), 5.79 (1H, dt, *J* = 10.5, 2.0 Hz, H-2), 5.88 (1H, ddd, *J* = 10.5, 3.0, 2.0 Hz, H-3). ^{13}C-NMR (75 MHz, $CDCl_3$): δ 20.7, 20.9, 63.3, 65.5, 68.5, 70.3, 78.8, 101.1, 125.6, 130.1, 170.3, 170.7, 208.6.

Anal. Calcd. For $C_{23}H_{28}O_{10}$: C, 59.48; H, 6.08 Found: C, 59.49; H, 6.16



13

[RNS-55-4]

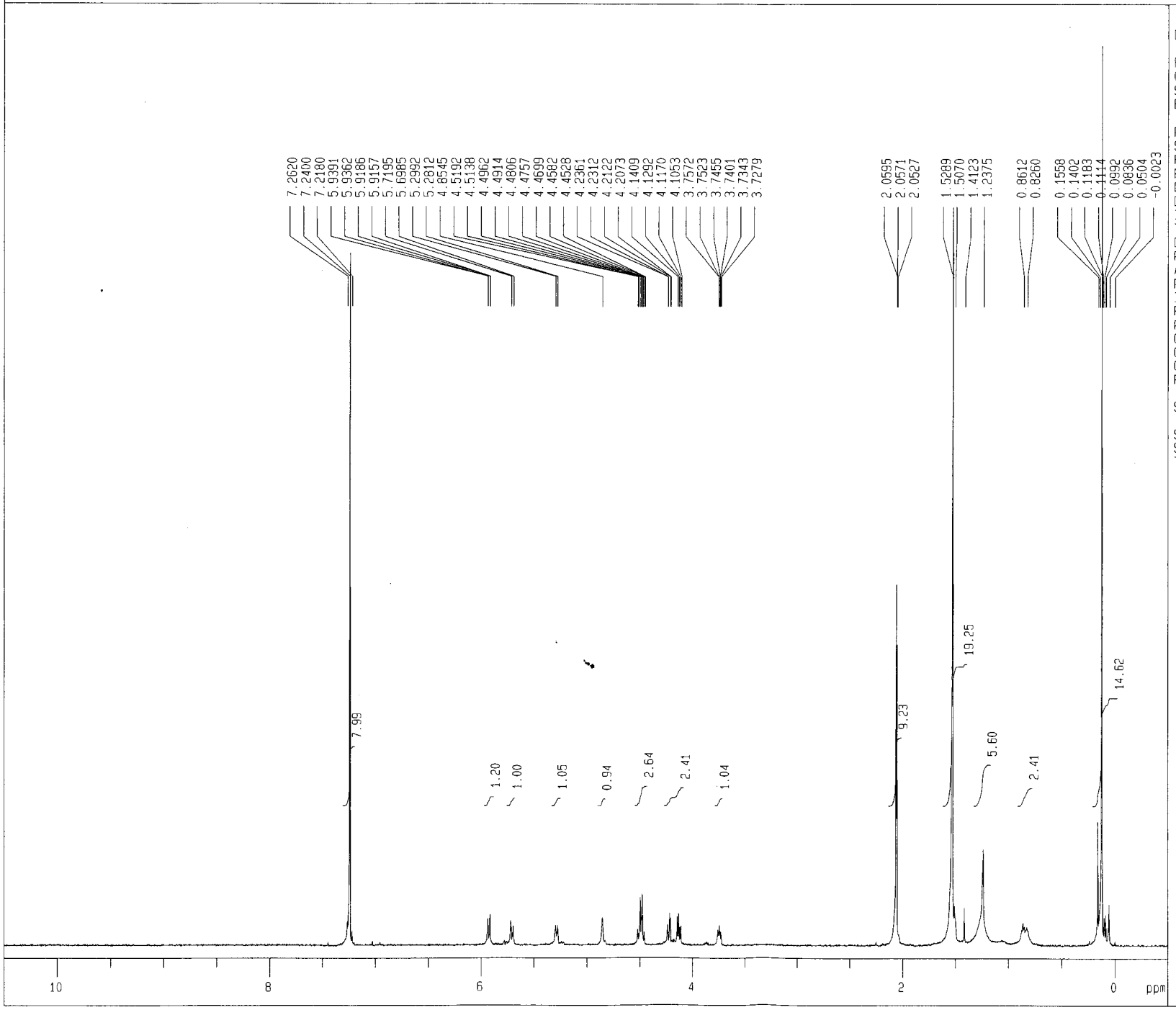
Compound 13. $[\alpha]_D^{27}$ +20.2° (c = 0.35, $CHCl_3$). IR (KBr) : 2922, 1740, 1372, 1234, 1049 cm^{-1}. ^{1}H-NMR (500 MHz, $CDCl_3$) : δ 2.05 (3H, s, *Ac*), 2.06 (3H, s, *Ac*), 2.07 (3H, s, *Ac*), 2.08 (3H, s, *Ac*), 2.52 (1H, ddd, *J* = 17.0, 8.0, 2.0 Hz, H-9a), 2.63 (1H, ddd, *J* = 17.0, 6.0, 2.0 Hz, H-9b), 3.95

(1H, m, H-14), 4.05 (1H, ddd, *J* = 9.0, 4.5, 3.0 Hz, H-2), 4.13 (1H, dd, *J* = 12.0, 3.5 Hz, H-15a), 4.14 (1H, m, H-1a), 4.22 (1H, m, H-1b), 4.24 (1H, dd, *J* = 12.0, 6.0 Hz, H-15b), 4.36 (1H, ddt, *J* = 8.0, 6.0, 2.0 Hz, H-10),

4.96 (1H, m, H-6), 5.12 (1H, m, H-13), 5.27 (1H, ddd, *J* = 9.0, 4.0, 2.0 Hz, H-3), 5.74 (1H, dt, *J* = 10.0, 2.0 Hz, H-4), 5.85 (1H, ddd, *J* = 10.0, 3.5, 2.0 Hz, H-5), 5.86 (1H, ddd, *J* = 10.0, 5.0, 3.0 Hz, H-5), 6.06 (1H, ddd, *J* = 10.0, 2.5, 1.5 Hz, H-3).

Anal. Calcd. For $C_{23}H_{28}O_{10}$: C, 59.48; H, 6.08 Found: C, 59.48; H, 6.27

RNS-59-2-1



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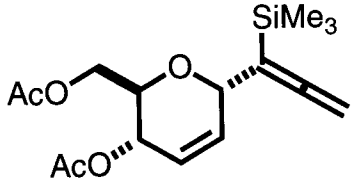

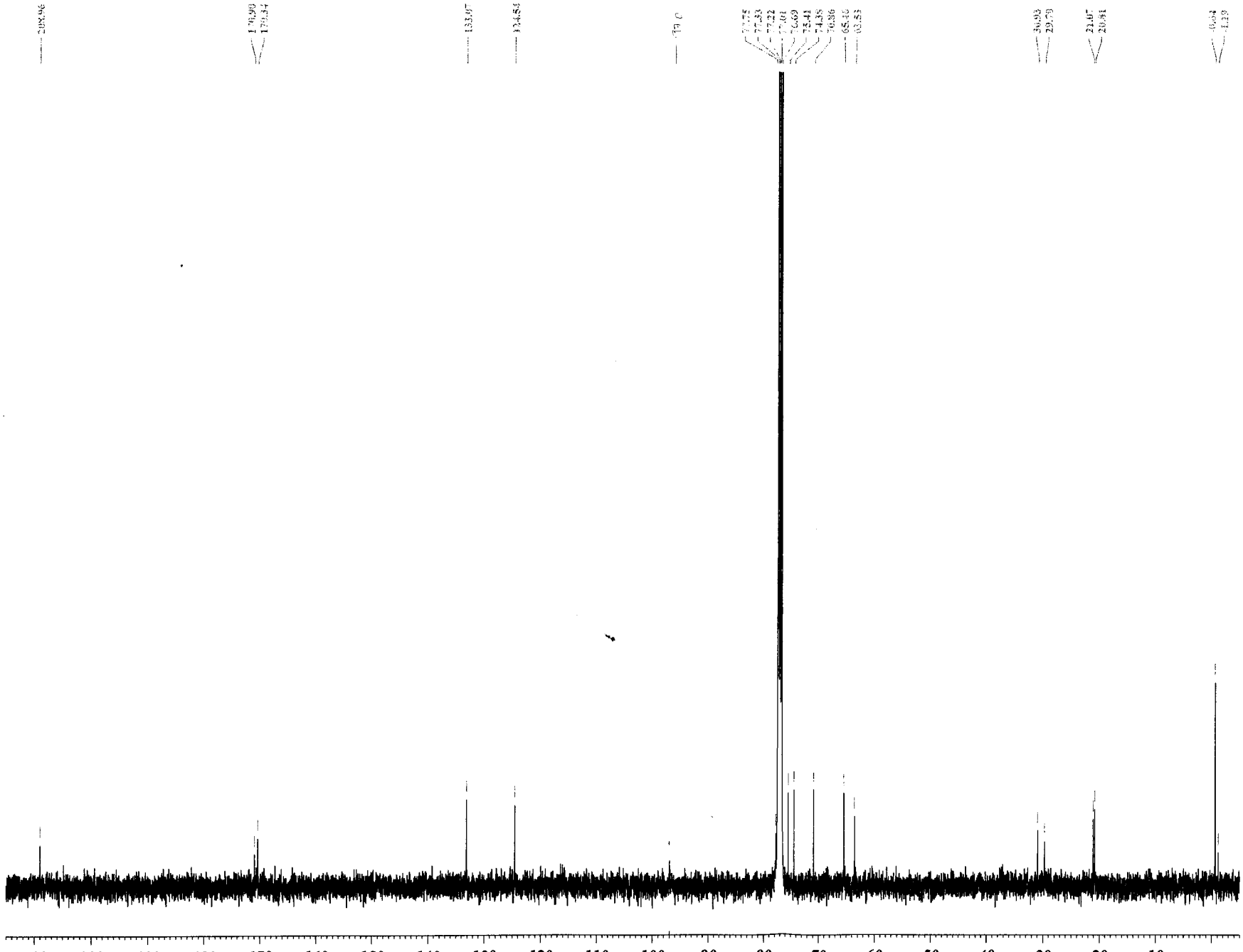
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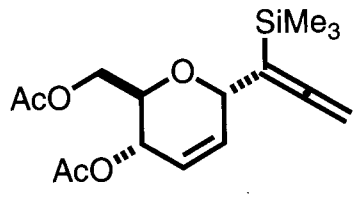
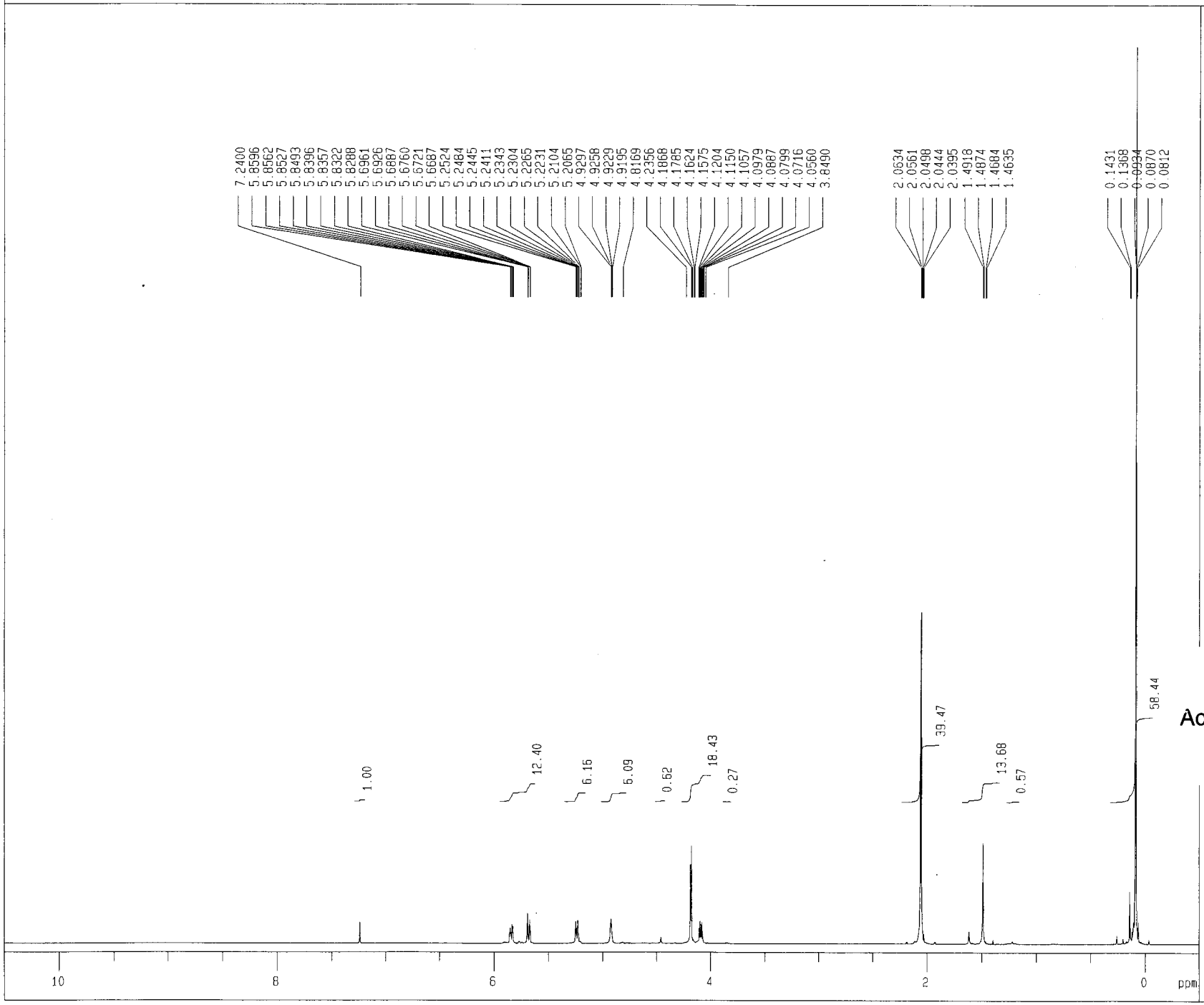
14.39
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

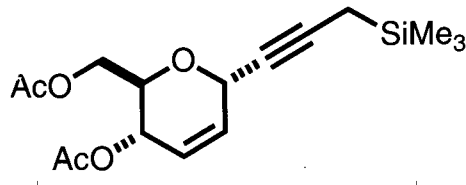

SiMe3
O
AcO
AcO

RA-20-3

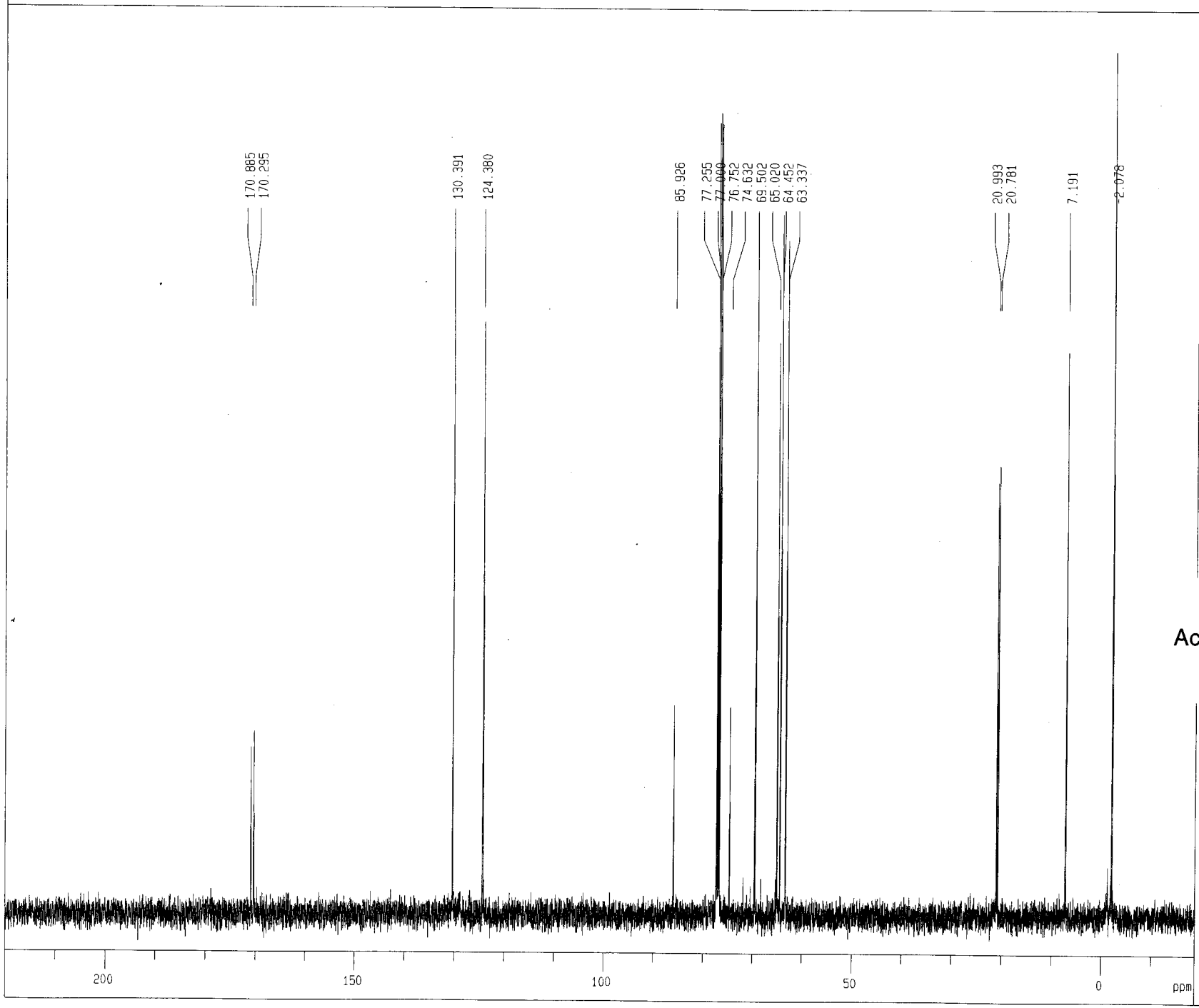


Date : Wed Apr 9 02:47:47 2003

FileName	:	.LoadingFID.nmdata
Comment	:	RA-20-3
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	8000.0 Hz
FILTR	:	4000 Hz
DELAY	:	50.0 usec
DEADT	:	72.3 usec
INTVL	:	125.0 usec
TIMES	:	8 times
DUMMY	:	1 times
PD	:	4.9520 sec
ACQTM	:	4096.0000 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	5.50 usec
OBNUC	:	1H
OBFRQ	:	500.00 MHz
OBSET	:	162410.00 Hz
RGAIN	:	16
SCANS	:	8 times
SLVNT	:	CDCL3
SPINNING	:	12 Hz
TEMP	:	26.8 C

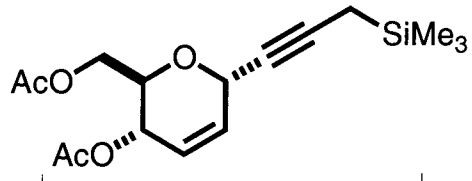


RA-20-3

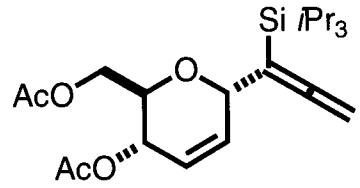


Date : Wed Apr 9 02:56:32 2003

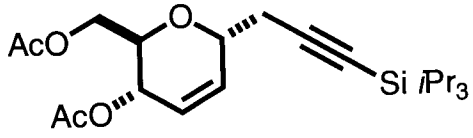
FileName	:	.LoadingFID.nmdata
Comment	:	RA-20-3
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	30030.0 Hz
FILTR	:	15000 Hz
DELAY	:	13.3 usec
OEADT	:	17.3 usec
INTVL	:	33.3 usec
TIMES	:	128 times
DUMMY	:	1 times
PD	:	1.9088 sec
ACQTM	:	1091.1743 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.92 Hz
PW1	:	5.50 usec
OBNUC	:	13C
OBFRQ	:	125.65 MHz
OBSET	:	127958.00 Hz
RGAIN	:	32
IRNUC	:	1H
IRFRQ	:	500.00 MHz
IRSET	:	162410.00 Hz
IRRPW	:	50.0 usec
IRRNS	:	0
SCANS	:	128 times
SLVNT	:	CDCL3
SPINNING	:	12 Hz
TEMP	:	28.5 C



```
expl  std1h

      SAMPLE
date      Jan  3 03
solvent       CDCl3
file             exp
   ACQUISITION
sfrq        299.967
tn               H1
at              3.748
np             33728
sw            4499.5
fb              2250
bs                16
pw               6.0
d1             0.252
tof            149.3
nt                16
ct                16
alock              n
gain       not used
      FLAGS
il                 n
in                 n
dp                 y
     DISPLAY
sp             -60.0
wp           3119.7
vs             2835
sc                 0
wc               400
hzmm            7.80
is          15082.99
rfl            599.1
rfp                0
th                 2
ins            1.000
ai  cdc  ph

      DEC. & VT
dfrq         299.957
dn                H1
dof          -10000.0
dm                nnn
dmm                c
dmf              200
     PROCESSING
wtfile
proc              ft
fn           not used

werr           react
wexp autolist('ent-
erJ','h1_process_J-
                135')
wbs
wnt
```

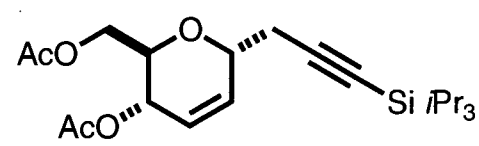


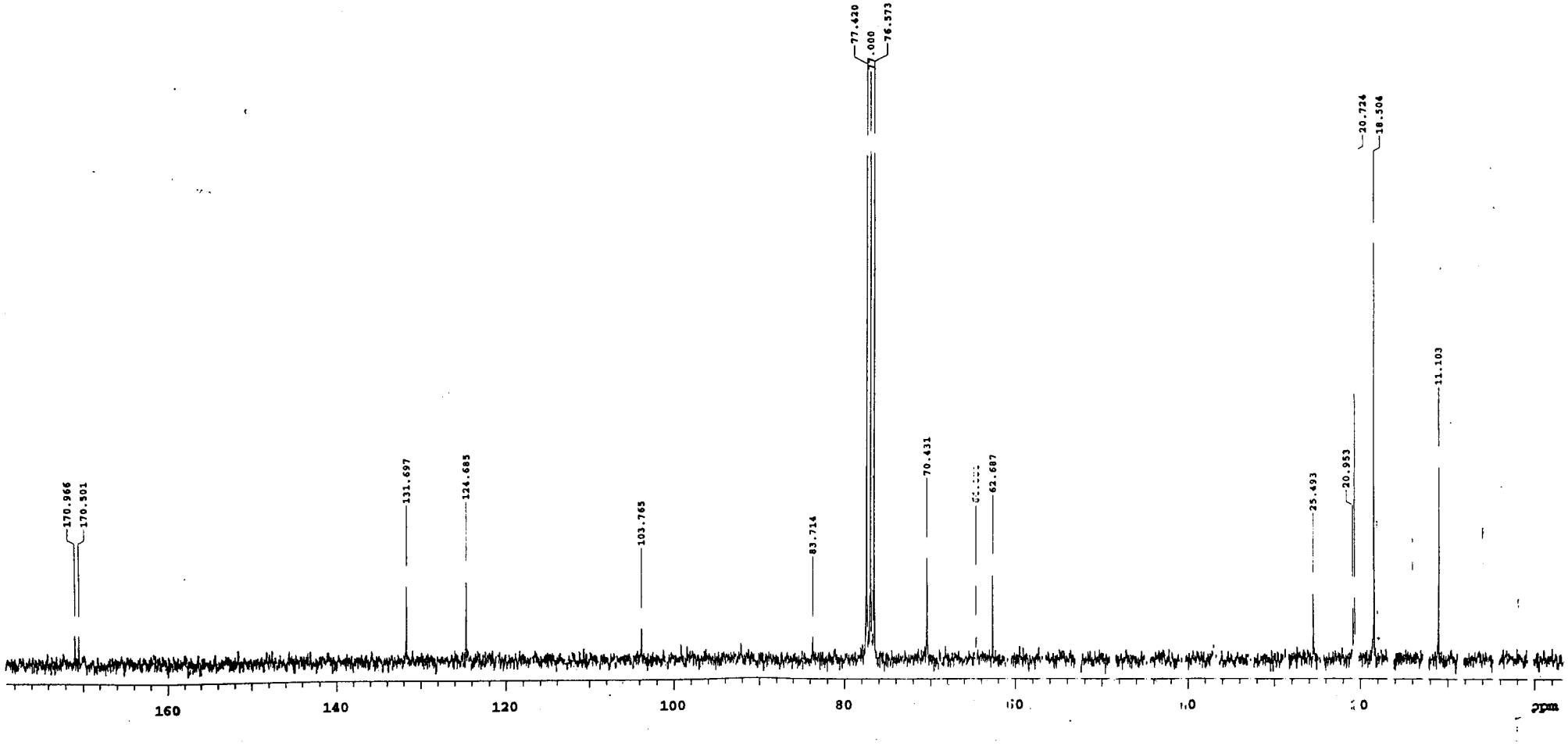
```
expl  std1h

      SAMPLE           DEC. & VT
date      Jan 17 03  dfrq       299.957
solvent       CDCl3  dn             H1
file            exp  dof       -10000.0
   ACQUISITION       dm              nnn
sfrq       299.967  dmm              c
tn              H1  dmf            200
at           3.748      PROCESSING
np          93728  wtfile
sw          4499.5  proc            ft
fb            2250  fn         not used
bs              16
pw             6.0  werr          react
d1           0.252  wexp autolist('ent-
tof          149.3  erJ','h1_process_J-
nt              16                136')
ct              16  wbs
alock            n  wnt
gain       not used
      FLAGS
il               n
in               n
dp               y
     DISPLAY
sp           -60.0
wp          3119.7
vs             858
sc               0
wc             400
hzcm          7.80
is         13956.52
rfl          598.3
rfp              0
th               3
ins          1.000
ai  cdc  ph
```



Solvent: CDCl3
Ambient temperature
GEMINI-300 "varian"

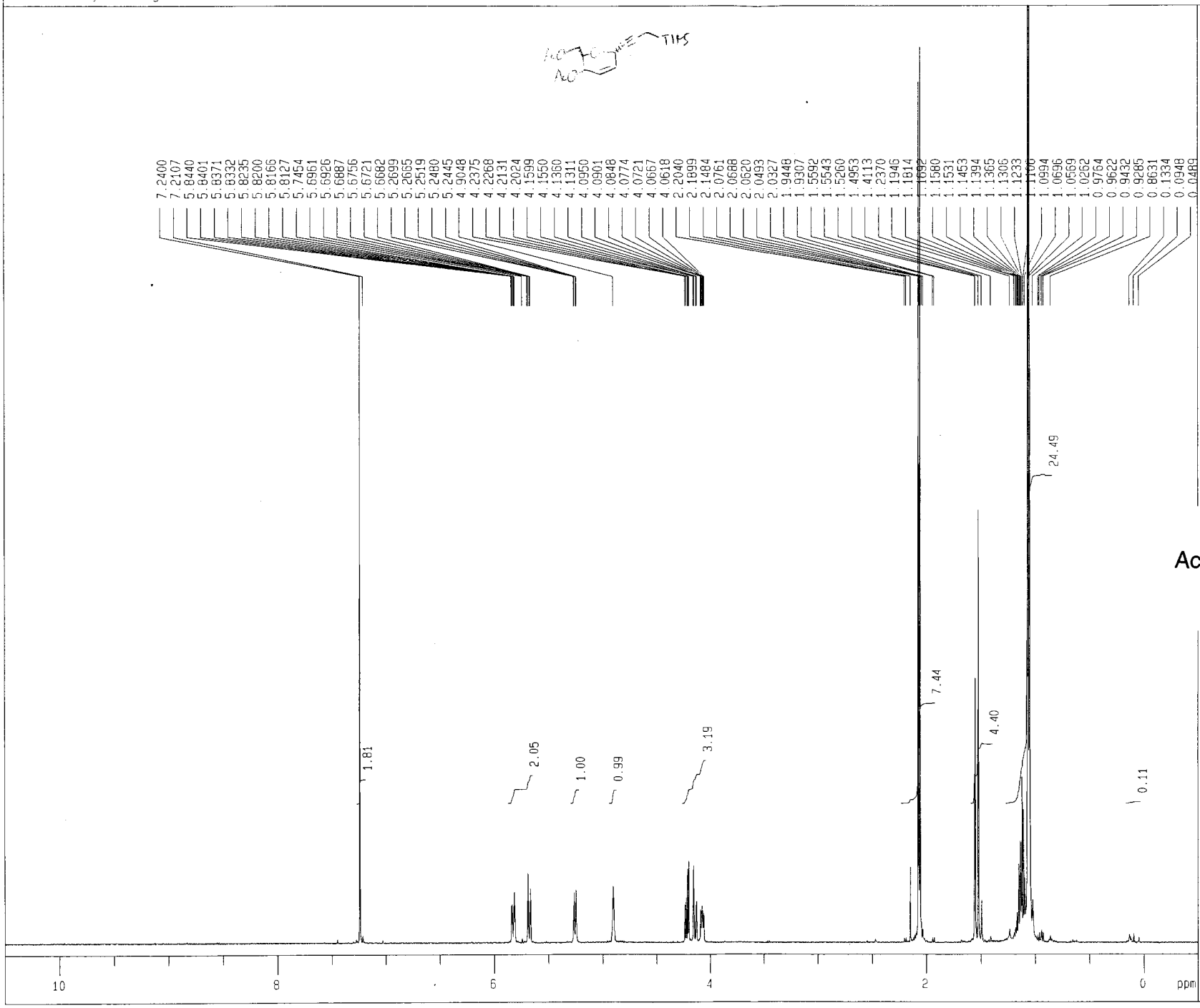
PULSE SEQUENCE
Pulse 35.0 degrees
Acq. time 1.697 sec
Width 18858.5 Hz
384 repetitions
OBSERVE C13, 75.4264040 MHz
DECOUPLE H1, 299.9672195 MHz
Power 30 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 10 minutes





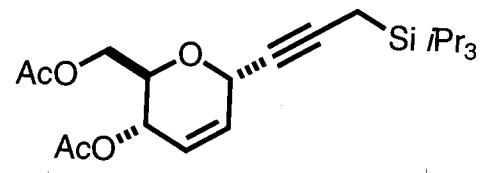
PNS-28

TIPSacetylene-glucal



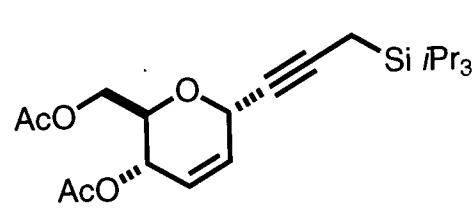
Date : Mon Apr 7 03:28:36 2003

FileName	LoadingFID.nmdata
Comment	TIPSacetylene-glucal
SliceHistory	
EXMODE	non
POINT	32768 points
SAMPO	32768 points
FREQU	8000.0 Hz
FILTR	4000 Hz
DELAY	50.0 usec
DEADT	72.3 usec
INTVL	125.0 usec
TIMES	8 times
DUMMY	1 times
PD	4.9520 sec
ACQTM	4096.0000 msec
PREDL	0.01000 msec
INIWT	1000.0000 msec
RESOL	0.24 Hz
PW1	5.50 usec
OBNUC	1H
OBFRQ	500.00 MHz
OBSET	162410.00 Hz
RGAIN	22
SCANS	8 times
SLVNT	CDCL3
SPINNING	13 Hz
TEMP	27.9 C

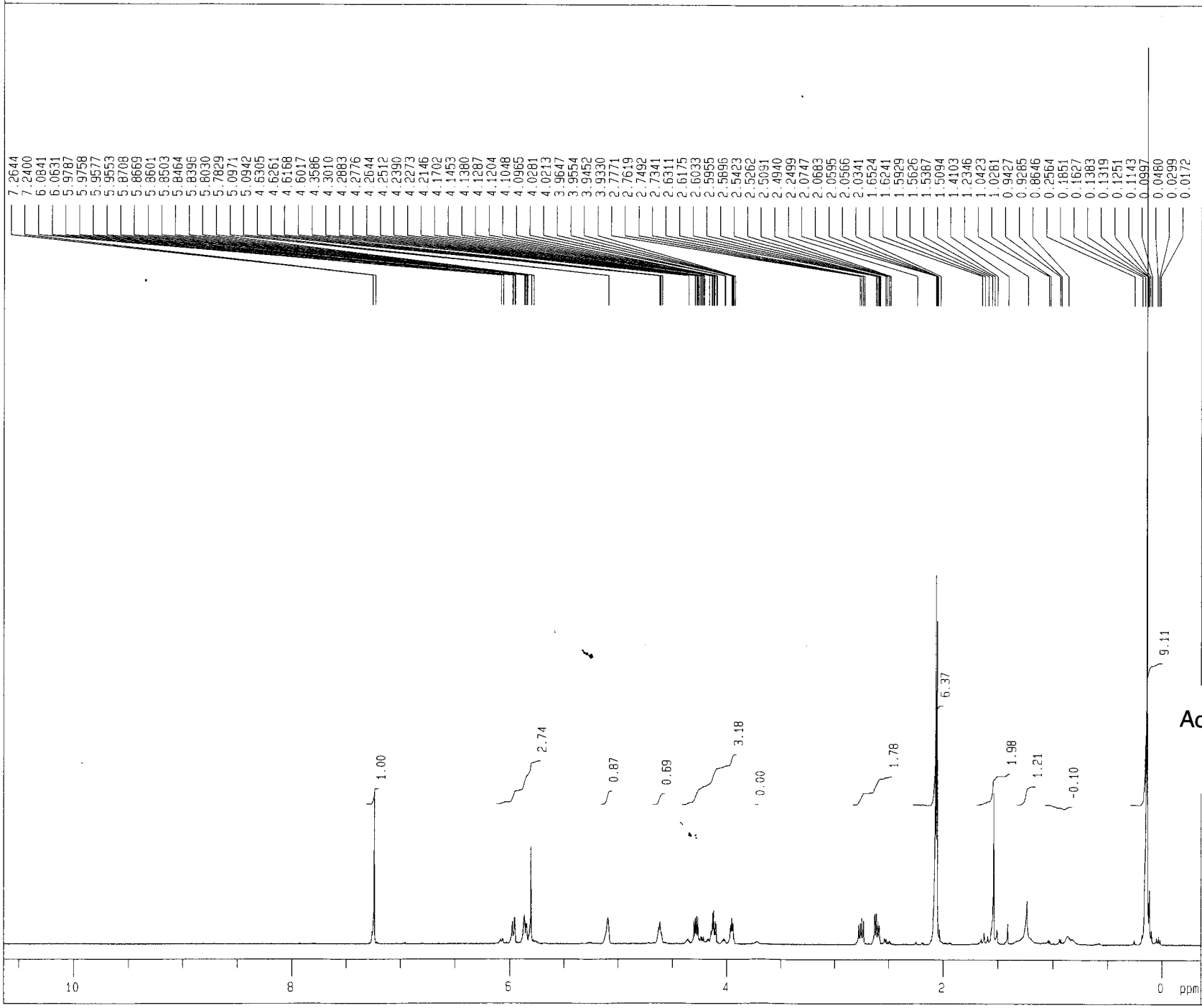


olvent: CDCl3
mbient temperature
MINI-300 "varian"

LSE SEQUENCE
ulse 35.0 degrees
cq. time 1.697 sec
idth 18858.5 Hz
36 repetitions
SERVE C13, 75.4264040 MHz
COUPLE H1, 299.9672195 MHz
ower 30 dB
ontinuously on
ALTZ-16 modulated
TA PROCESSING
ine broadening 1.0 Hz
' size 65536
tal time 9 minutes

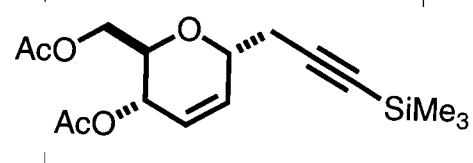


RNS-55-1



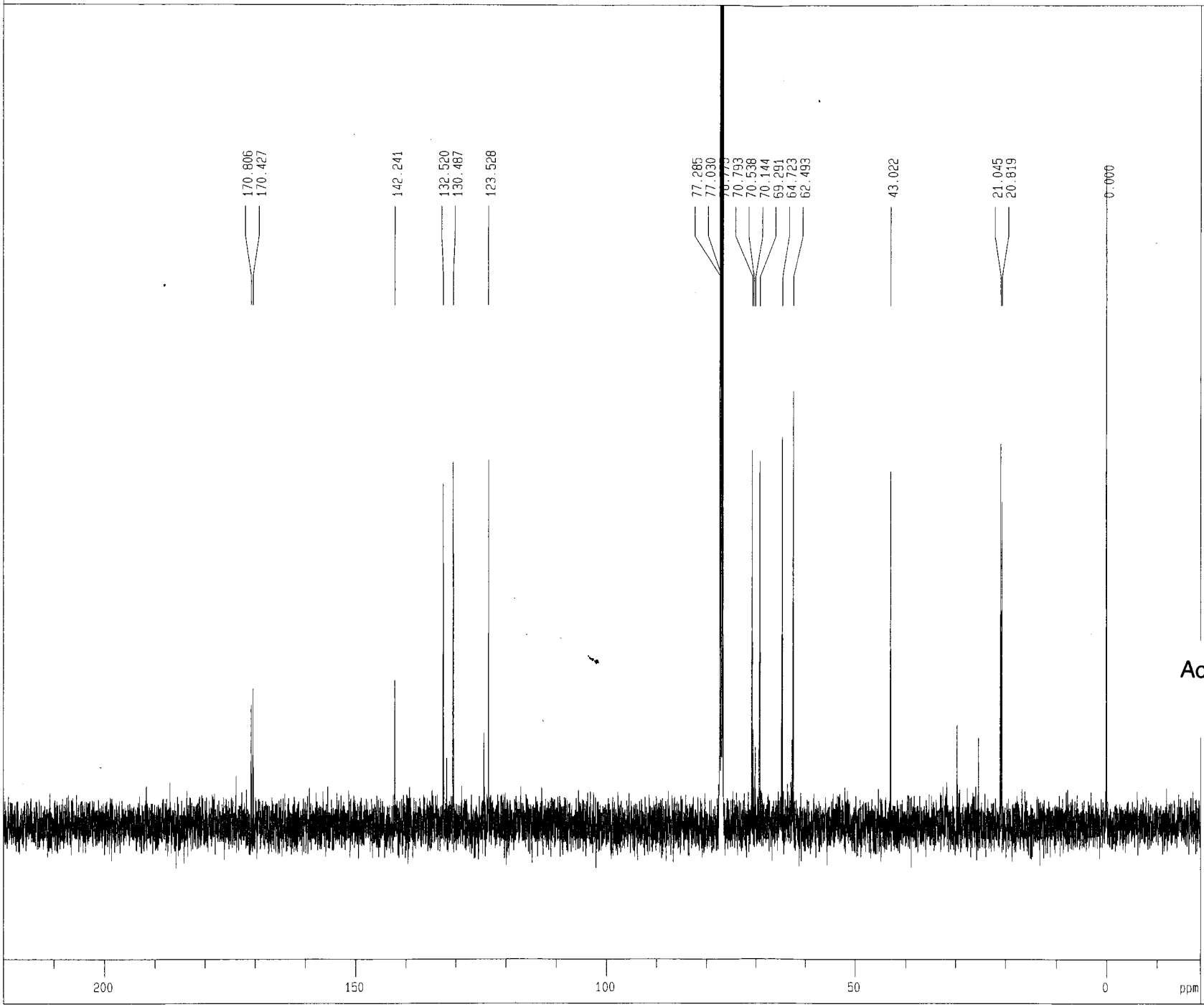
Date : Thu May 22 01:48:49 2003

FileName	:	.LoadingFID.nmdata
Comment	:	RNS-55-1
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	8000.0 Hz
FILTR	:	4000 Hz
DELAY	:	50.0 usec
DEADT	:	72.3 usec
INTVL	:	125.0 usec
TIMES	:	8 times
DUMMY	:	1 times
PD	:	4.9520 sec
ACQTM	:	4096.0000 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	5.50 usec
OBNUC	:	1H
OBFRQ	:	500.00 MHz
OBSET	:	162410.00 Hz
RGAIN	:	22
SCANS	:	8 times
SLVNT	:	CDCL3
SPINNING	:	10 Hz
TEMP	:	27.3 C



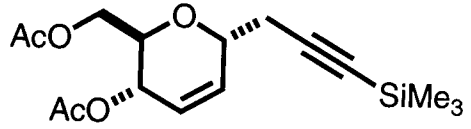
RNS-95-1

1H S/N

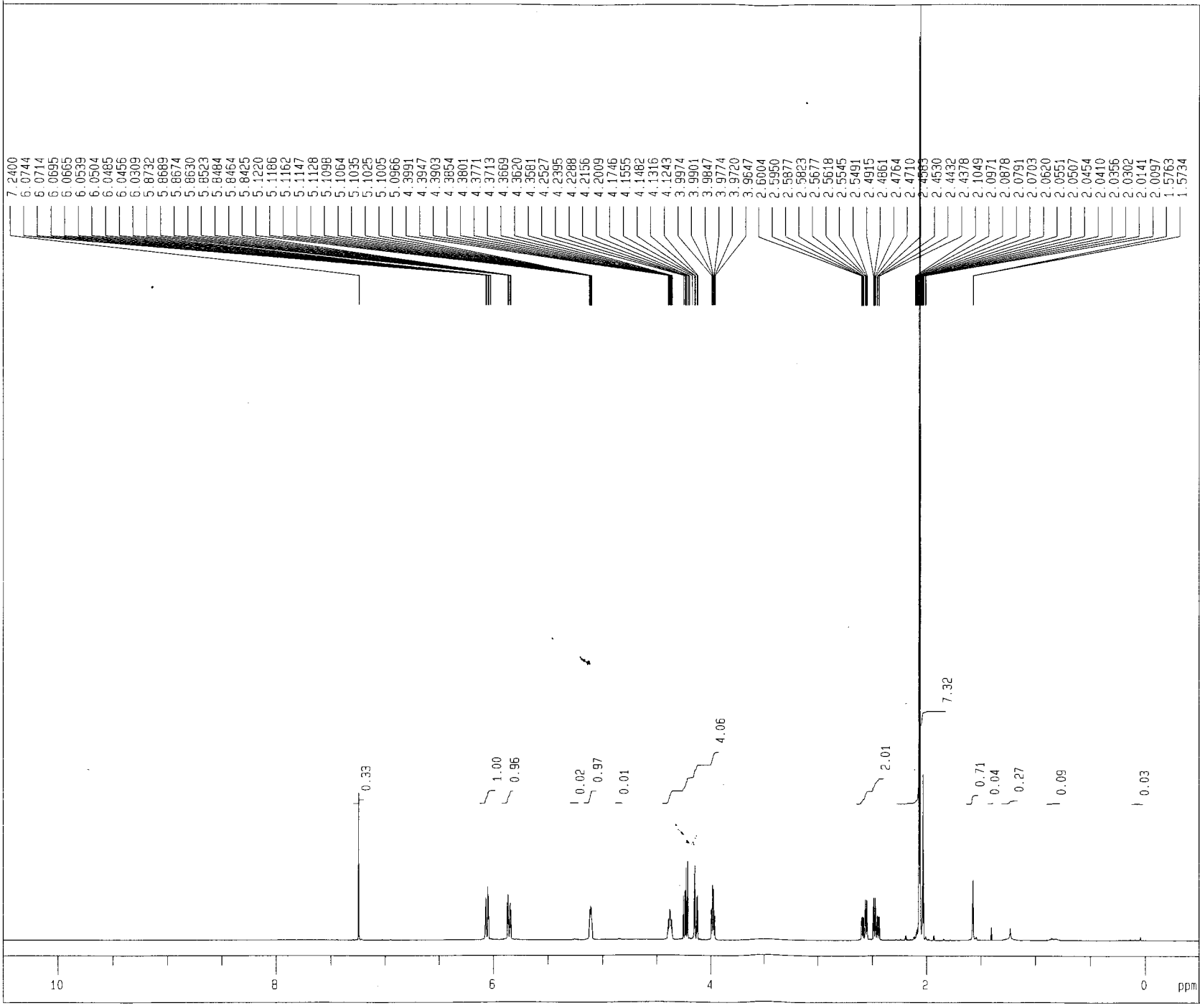


Date : Fri May 23 03:23:15 2003

FileName	:	LoadingFID.nmdata
Comment	:	1H S/N
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	30030.0 Hz
FILTR	:	15000 Hz
DELAY	:	13.3 usec
DEADT	:	17.3 usec
INTVL	:	33.3 usec
TIMES	:	768 times
DUMMY	:	1 times
PD	:	1.9088 sec
ACQTM	:	1091.1743 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.92 Hz
PW1	:	5.50 usec
OBNUC	:	13C
OBFRQ	:	125.65 MHz
OBSET	:	127958.00 Hz
RGAIN	:	31
IRNUC	:	1H
IRFRQ	:	500.00 MHz
IRSET	:	162410.00 Hz
IRRPW	:	50.0 usec
IRRNS	:	0
SCANS	:	768 times
SLVNT	:	CDCL3
SPINNING	:	13 Hz
TEMP	:	29.7 C

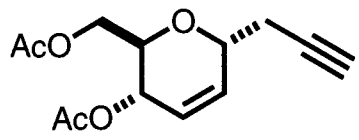


RNS-49-3

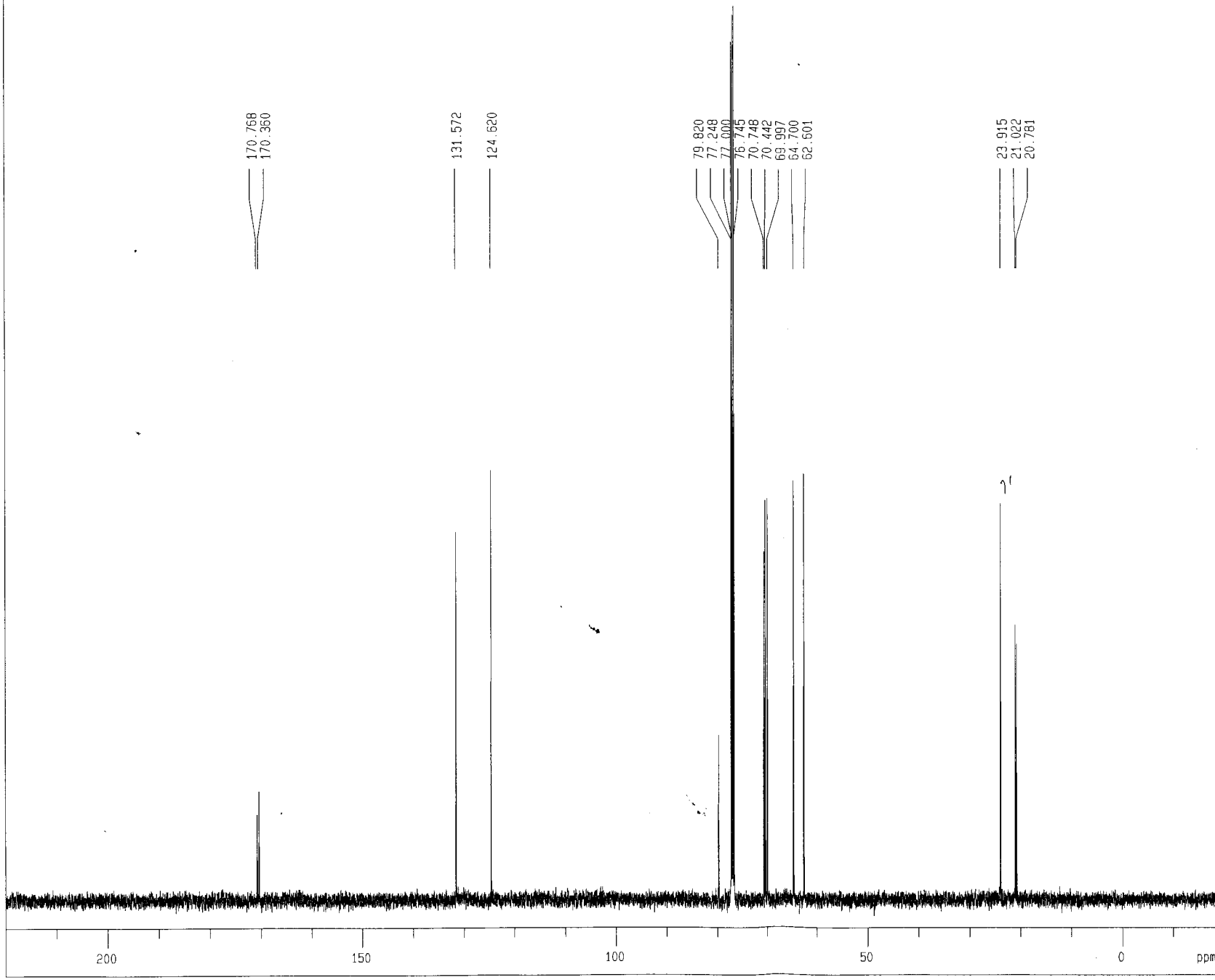


Date : Wed Apr 30 07:17:40 2003

FileName	:	LoadingFID.nmdata
Comment	:	RNS-49-3
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	8000.0 Hz
FILTR	:	4000 Hz
DELAY	:	50.0 usec
DEADT	:	72.3 usec
INTVL	:	125.0 usec
TIMES	:	8 times
DUMMY	:	1 times
PD	:	4.9520 sec
ACQTM	:	4096.0000 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	5.50 usec
OBNUC	:	1H
OBFRQ	:	500.00 MHz
OBSET	:	162410.00 Hz
RGAIN	:	20
SCANS	:	8 times
SLVNT	:	CDCL3
SPINNING	:	12 Hz
TEMP	:	465.0 C

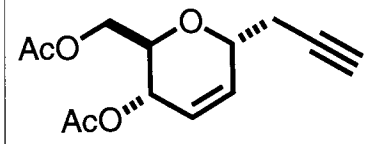


RNS-49-3

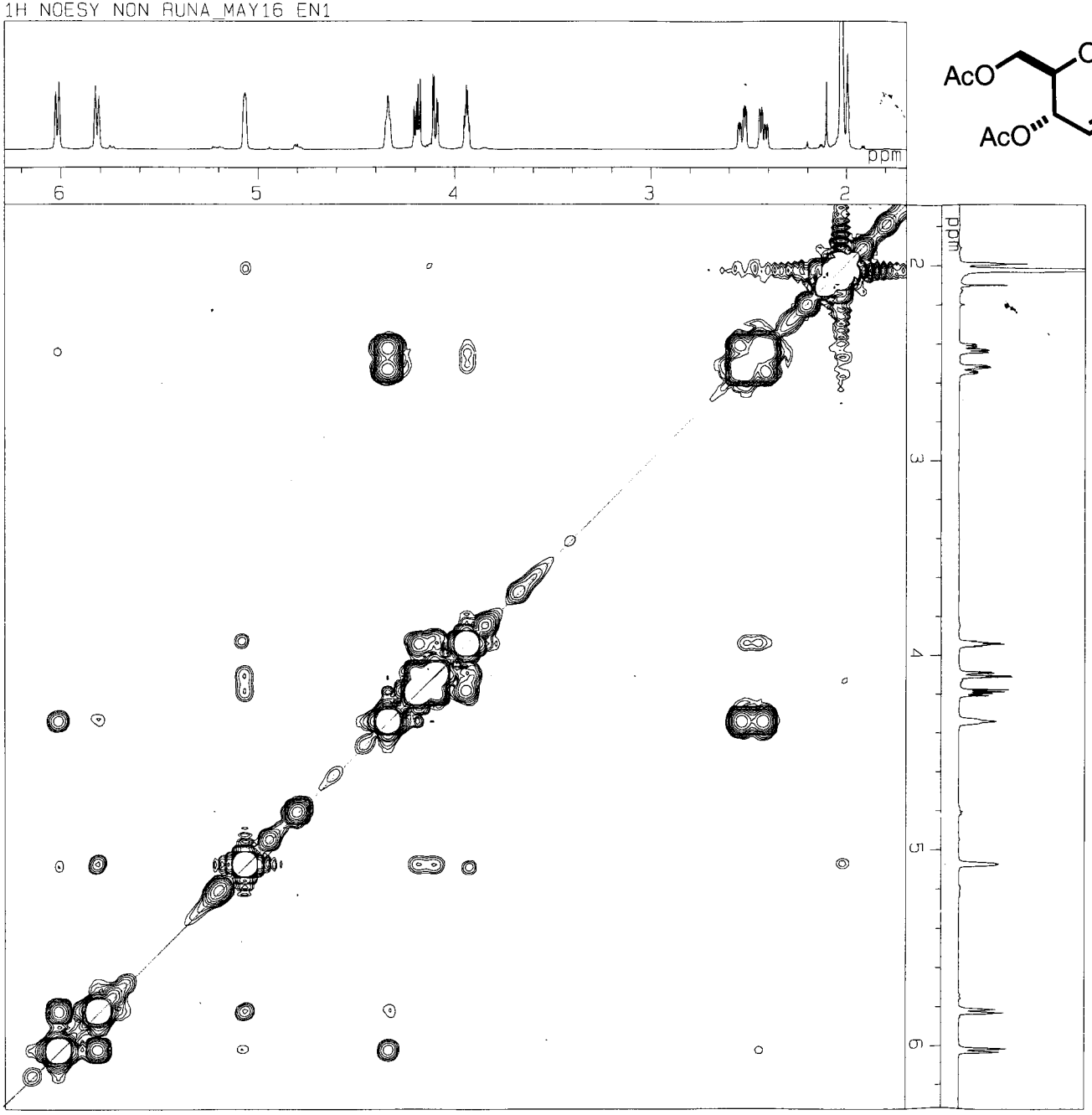


Date : Wed Apr 30 07:50:38 2003

FileName	:	.LoadingFID.nmdata
Comment	:	RNS-49-3
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	30030.0 Hz
FILTR	:	15000 Hz
DELAY	:	13.3 usec
DEADT	:	17.3 usec
INTVL	:	33.3 usec
TIMES	:	512 times
DUMMY	:	1 times
PD	:	1.9088 sec
ACQTM	:	1091.1743 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.92 Hz
PW1	:	5.50 usec
OBNUC	:	13C
OBFRQ	:	125.65 MHz
OBSET	:	127958.00 Hz
OBAIN	:	31
IRNUC	:	1H
IRFRQ	:	500.00 MHz
IRSET	:	162410.00 Hz
IRPPW	:	50.0 usec
IRPNS	:	0
SCANS	:	512 times
SLVNT	:	CDCL3
SPINNING	:	12 Hz
TEMP	:	465.0 C



1H NOESY NON RUNA_MAY16 EN1



16-MAY-2003 15:08:27.37

DFILE : $ALPHA2
SFILE : RUNA_MAY162NOESY_E1
HR2FILE: RUNA_MAY161NON_E1
HR1FILE: RUNA_MAY161NON_E1

COMNT : 1H NOESY NON RUNA_MAY16 EN1
EXMOD : NOESY
IRMOD : NON
POINT : 512
FREQU : 5189.41 Hz
SCANS : 16
DUMMY : 4
ACQTM : 0.0493 sec
PD : 2.4507 sec
RGAIN : 13

CLFRQ : 5189.41 Hz
CLPNT : 512
TOSCN : 256
CINWT : 10.00 usec
CINTV : 192.70 usec

PW1 : 12.90 usec
PW2 : 20.00 usec
PI1 : 500.0000 msec
PI2 : 1.0000 msec

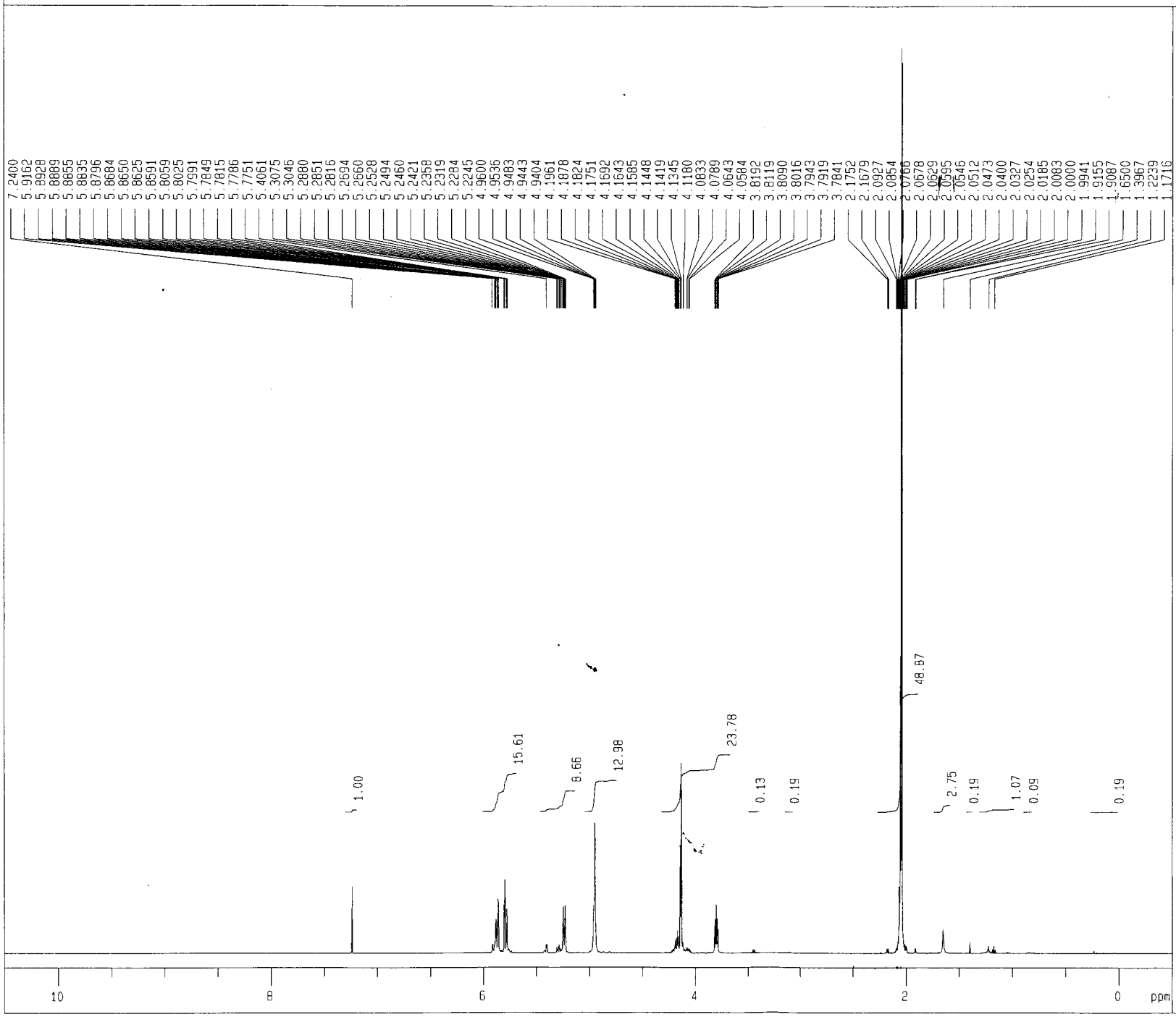
OBNUC : 1H
OBFRQ : 600.05 MHz
OBSET : 124505.37 Hz

IRNUC : 1H
IRFRQ : 600.05 MHz
IRSET : 125300.00 Hz
IRATN : 511
IRRPW : 50.0 usec
IRBP1 : 30
IRBP2 : 6
IRRNS : 0

ADBIT : 16
CTEMP : 22.3 c
CSPED : 12 Hz
SLVNT : CDCL3

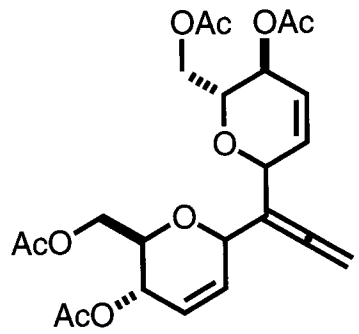
RESOL : 10.14 Hz
CLRSO : 10.14 Hz
TLINE : 10
THTOP : 2.0000
THBTM : 0.0500
operator

RNS-55-3-1

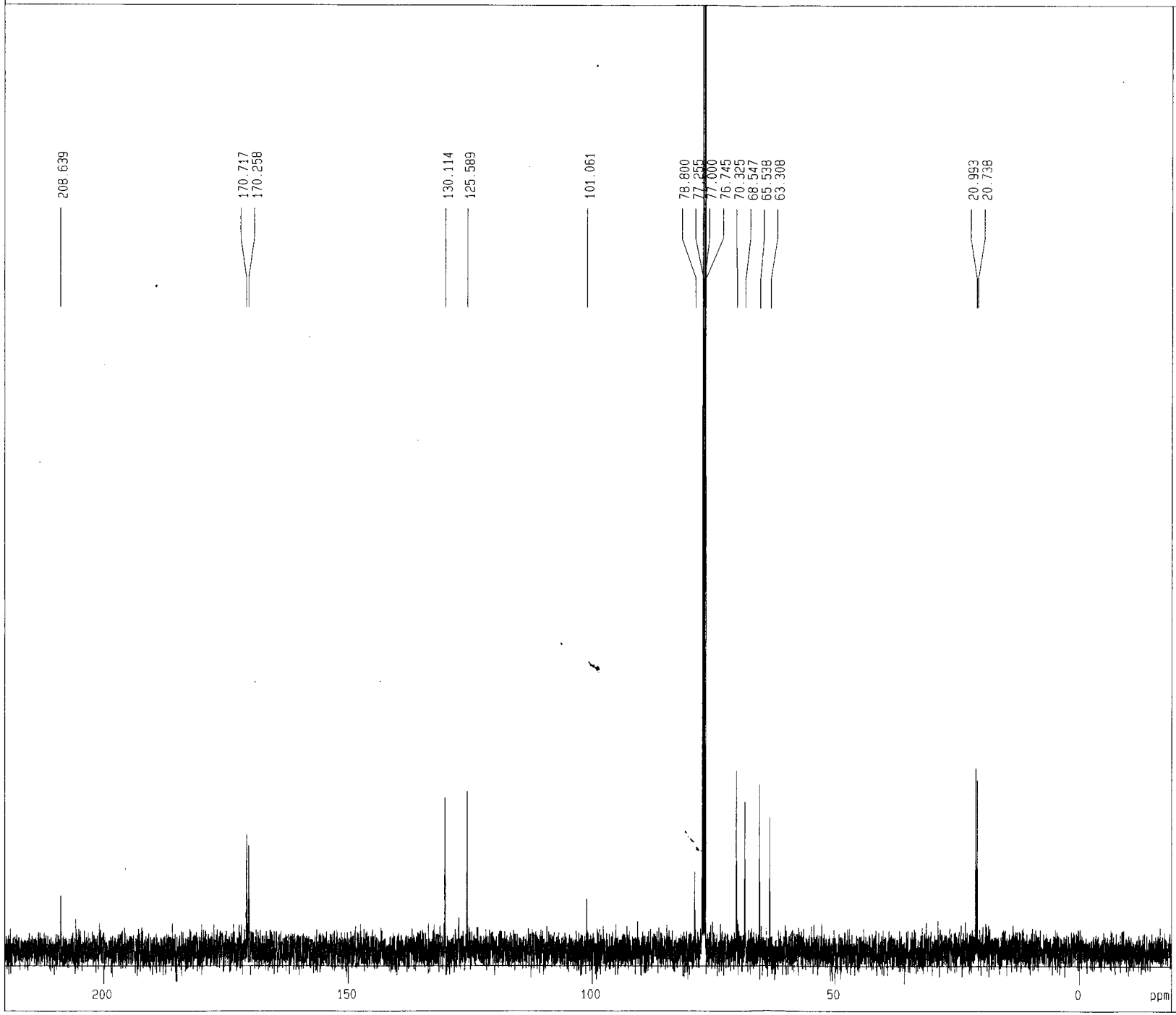


Date : Mon May 26 07:19:52 2003

FileName	:	.LoadingFID.nmdata
Comment	:	RNS-55-3-1
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	8000.0 Hz
FILTR	:	4000 Hz
DELAY	:	50.0 usec
DEADT	:	72.3 usec
INTVL	:	125.0 usec
TIMES	:	8 times
DUMMY	:	1 times
PD	:	4.9520 sec
ACQTM	:	4096.0000 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	5.50 usec
OBNUC	:	1H
OBFRQ	:	500.00 MHz
OBSET	:	162410.00 Hz
RGAIN	:	17
SCANS	:	8 times
SLVNT	:	CDCL3
SPINNING	:	11 Hz
TEMP	:	27.5 C

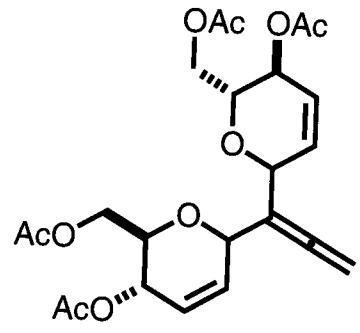


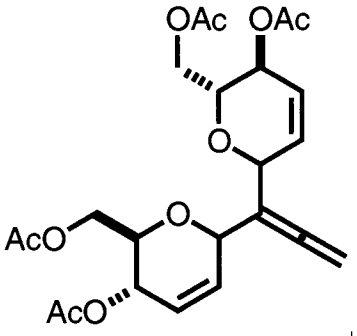
RNS-55-3-1



Date : Mon May 26 08:42:19 2003

FileName	:	.LoadingFID.nmdata
Comment	:	RNS-55-3-1
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	30030.0 Hz
FILTR	:	15000 Hz
DELAY	:	13.3 usec
DEADT	:	17.3 usec
INTVL	:	33.3 usec
TIMES	:	1024 times
DUMMY	:	1 times
PD	:	1.9088 sec
ACQTM	:	1091.1743 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.92 Hz
PW1	:	5.50 usec
OBNUC	:	13C
OBFRQ	:	125.65 MHz
OBSET	:	127958.00 Hz
RGAIN	:	33
IRNUC	:	1H
IRFRQ	:	500.00 MHz
IRSET	:	162410.00 Hz
IRRPW	:	50.0 usec
IRRNS	:	0
SCANS	:	1024 times
SLVNT	:	CDCL3
SPINNING	:	12 Hz
TEMP	:	29.8 C





Current Data Parameters
NAME 1aaa01
EXPNO 1
PROCNO 1

F1 - Acquisition Parameters
Date_ 20110401
Time 17:52
INSTRUM spect
PROBHD 5 mm QNP H1/X
PULPROG Excou2
TD 32768
SOLVENT CDCl3
NS 16
DS 4
SWH 23980.814 Hz
FIDRES 5.855660 Hz
AQ 0.6854516 sec
RG 16384
DW 20.850 usec
DE 6.00 usec
TE 300.0 K
D0 [redacted]
D1 2.00000000 sec
D2 0.00344828 sec
D3 0.00229885 sec
D11 0.03000000 sec
D12 0.00002000 sec
DW0 0.00012230 sec
MCREST 0.00000000 sec
MCWRK 0.03000000 sec

CHANNEL f1
NUC1 13C
P1 9.00 usec
P2 18.00 usec
PL1 -3.00 dB
SFO1 100.6228298 MHz

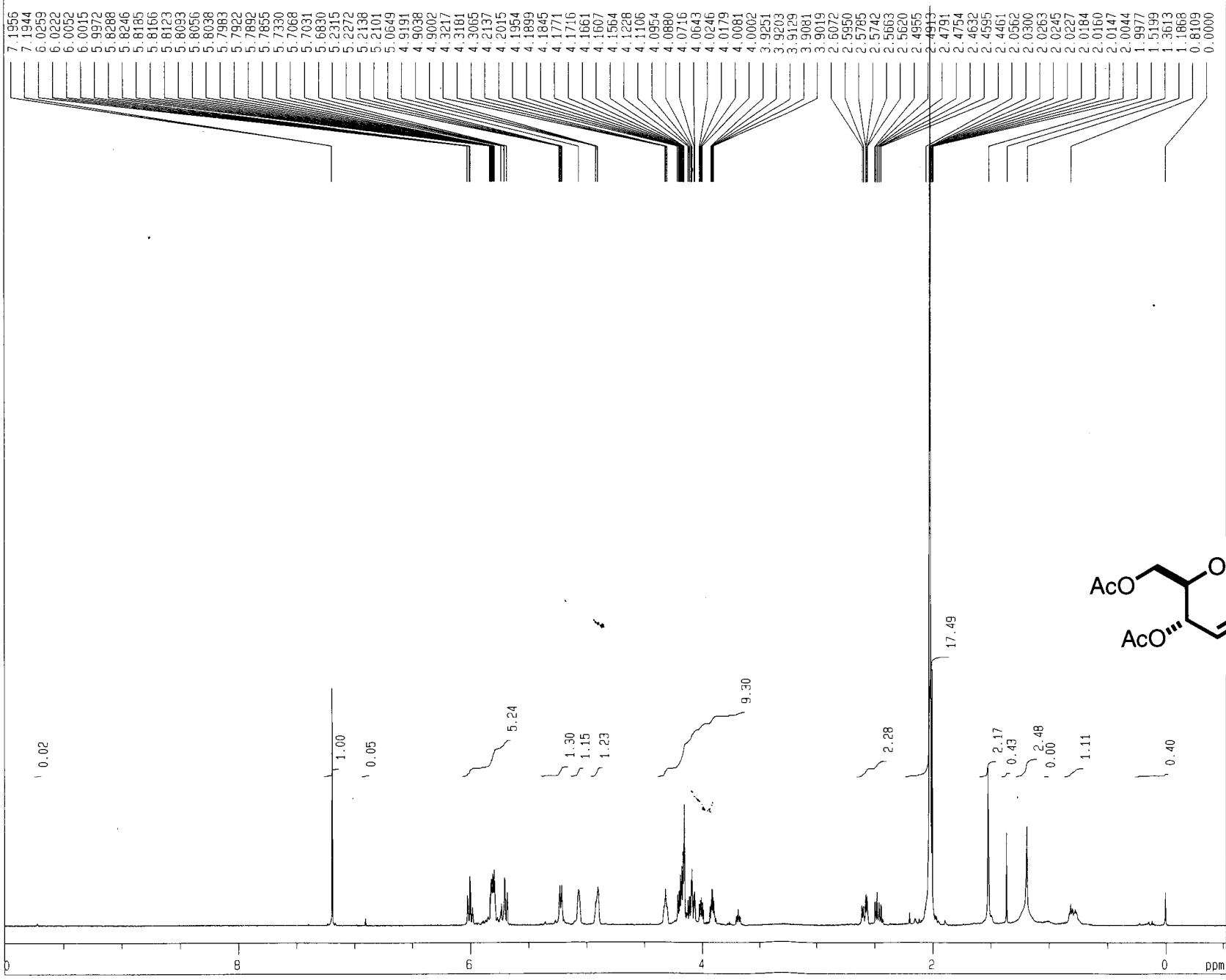
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 14.50 dB
SFO2 400.1318300 MHz

F1 - Acquisition parameters
NDM 2
TD 128
SFO1 400.1318 MHz
FIDRES 31.309953 Hz
SW 10.17 6 ppm
INAVOBL QF

F2 - Processing parameters
SI 2048
SF 400.0127600 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.40

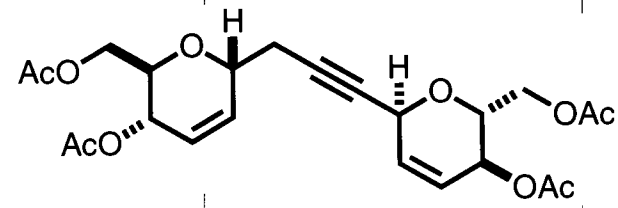
F1 - Processing parameters
SI 1024
MC2 QF
SF 400.1300000 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

non RNS-55-4

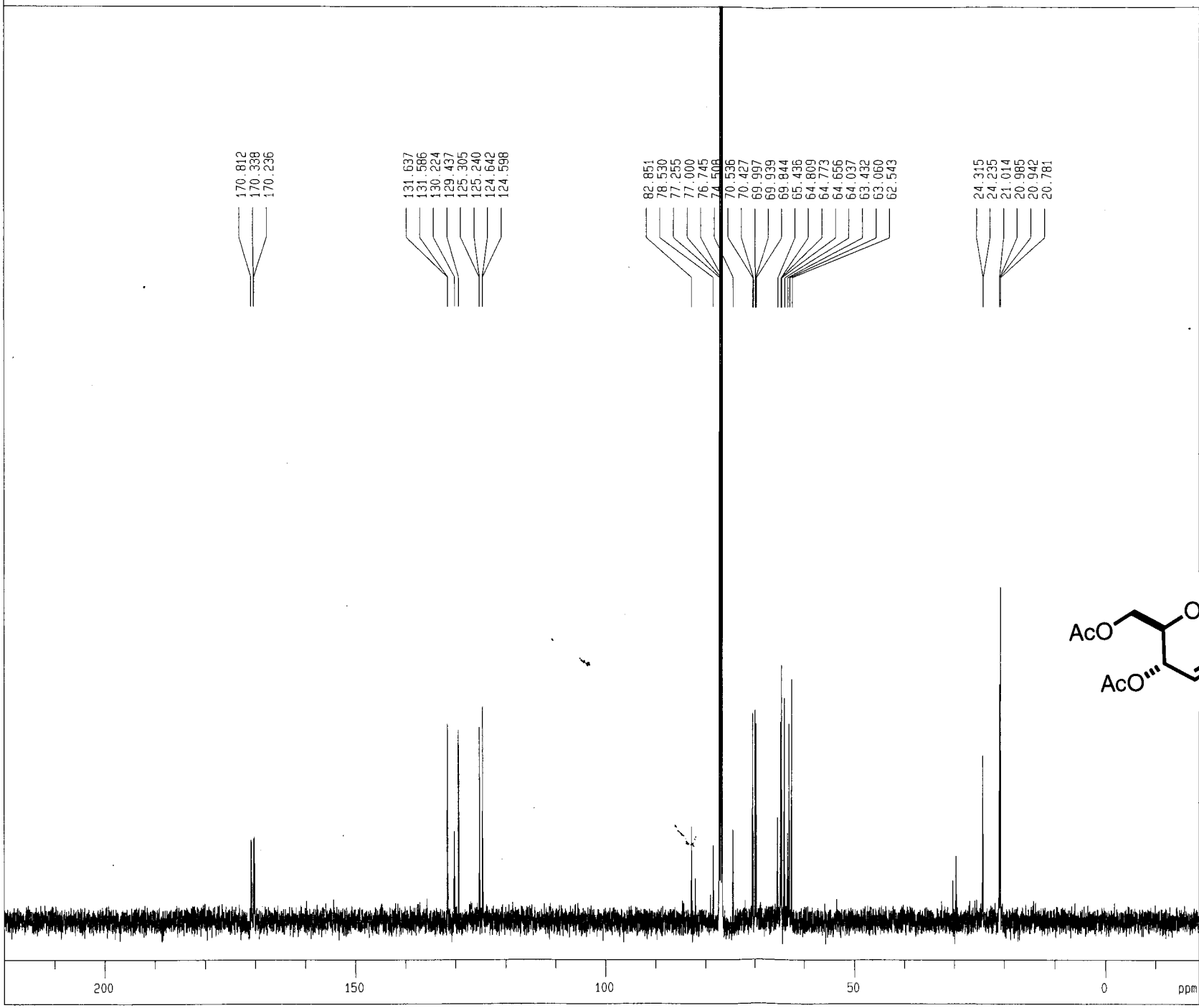


Date : Thu May 22 06:49:07 2003

FileName	:	auto_1H.nmdata
Comment	:	non
EXMODE	:	non
POINT	:	32768 points
SAMPD	:	32768 points
FREQU	:	10000.0 Hz
FILTR	:	5000 Hz
DELAY	:	40.0 usec
DEADT	:	57.1 usec
INTVL	:	100.0 usec
TIMES	:	8 times
DUMMY	:	0 times
PD	:	3.7232 sec
ACQTM	:	3276.7998 msec
PREDL	:	10.00000 msec
INIWT	:	0.5000 msec
RESOL	:	0.31 Hz
PW1	:	5.70 usec
OBNUC	:	1H
OBFRQ	:	500.00 MHz
OBSET	:	162160.00 Hz
RGAIN	:	21
SCANS	:	8 times
SLVNT	:	CDCL3
SPINNING	:	12 Hz
TEMP	:	28.3 C

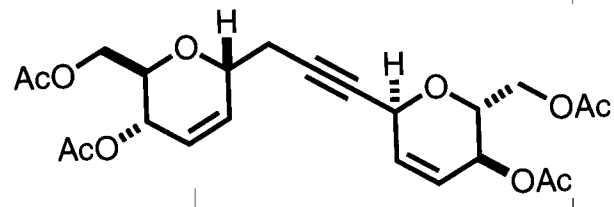


RNS-55-4

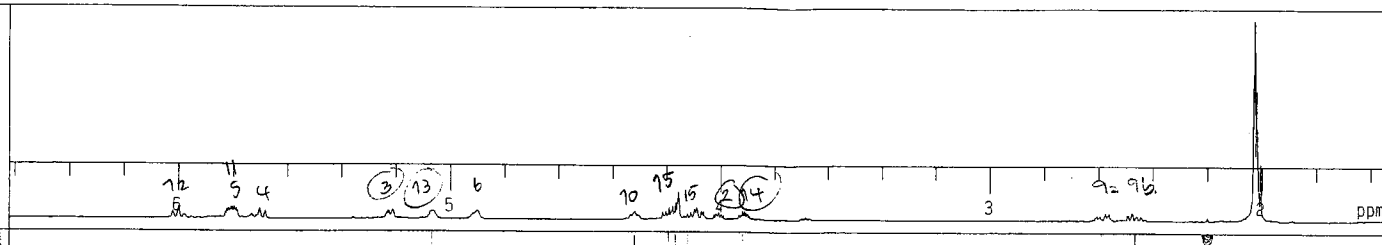


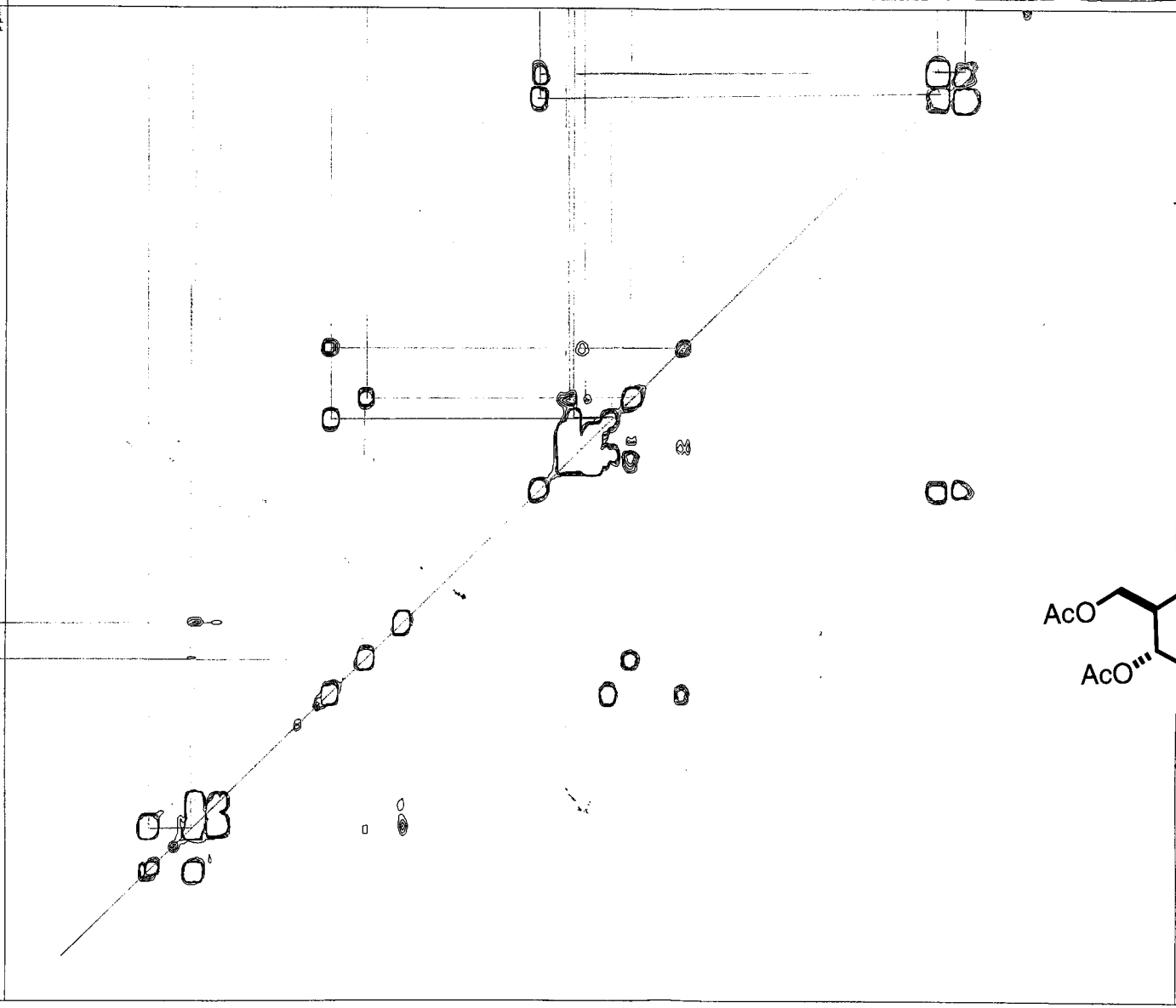
Date : Fri May 23 02:37:46 2003

FileName	:	.LoadingFID.nmdata
Comment	:	RNS-55-4
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	30030.0 Hz
FILTR	:	15000 Hz
DELAY	:	13.3 usec
DEADT	:	17.3 usec
INTVL	:	33.3 usec
TIMES	:	1024 times
DUMMY	:	1 times
PD	:	1.9088 sec
ACQTM	:	1091.1743 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.92 Hz
PW1	:	5.50 usec
OBNUC	:	13C
OBFRQ	:	125.65 MHz
OBSET	:	127958.00 Hz
RGAIN	:	31
IRNUC	:	1H
IRFRQ	:	500.00 MHz
IRSET	:	162410.00 Hz
IRRPW	:	50.0 usec
IRRNS	:	0
SCANS	:	1024 times
SLVNT	:	CDCL3
SPINNING	:	11 Hz
TEMP	:	29.6 C



cosy





Date : Thu May 22 08:06:16 2003

FileName	:	auto_HH2D.nmdata
Comment	:	cosy
SliceHistory	:	
EXMODE	:	cosy
POINT	:	512 points
SAMPO	:	512 points
FREQU	:	3963.5 Hz
FILTR	:	2000 Hz
DELAY	:	100.0 usec
DEADT	:	144.3 usec
INTVL	:	252.3 usec
TIMES	:	16 times
DUMMY	:	4 times
PD	:	0.9488 sec
ACQTM	:	129.1776 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	7.74 Hz
PW1	:	11.40 usec
OBNUC	:	1H
OBFRQ	:	500.00 MHz
OBSET	:	161754.12 Hz
RGAIN	:	19
CLFRQ	:	3963.5 Hz
CLPNT	:	256 points
TODAT	:	256 points
CINWT	:	5.00 usec
CINTV	:	252.3 usec
PW2	:	11.40 usec
IRNUC	:	1H
IRFRQ	:	500.00 MHz
IRSET	:	161754.12 Hz
IRATN	:	511
SCANS	:	16 times
SLVNT	:	CDCL3
SPINNING	:	14 Hz
TEMP	:	27.7 C