A Dinuclear Zinc Catalyzed Asymmetric Alkynylation of Unsaturated Aldehydes

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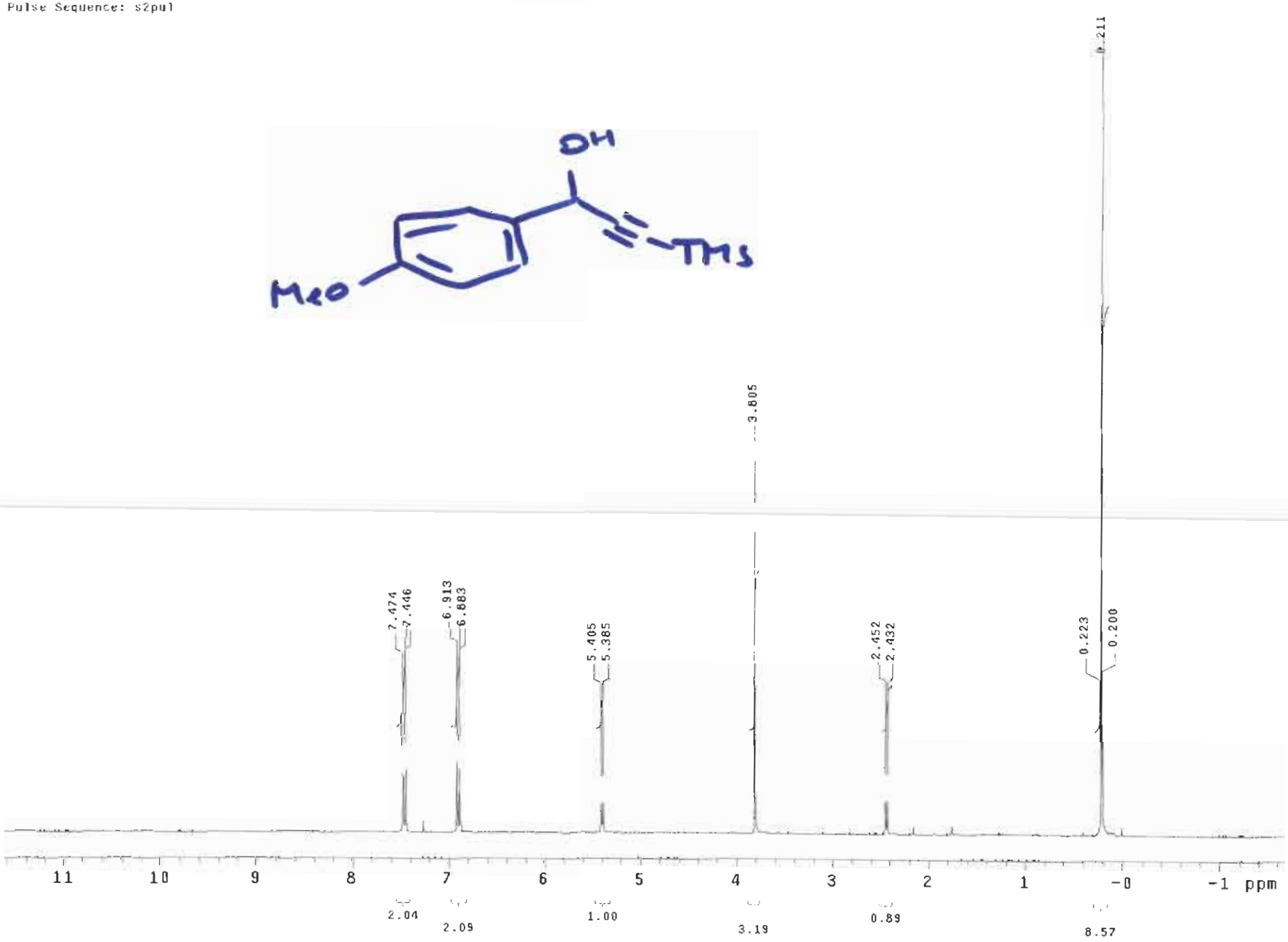
Supporting Information

Part B: NMR Spectra

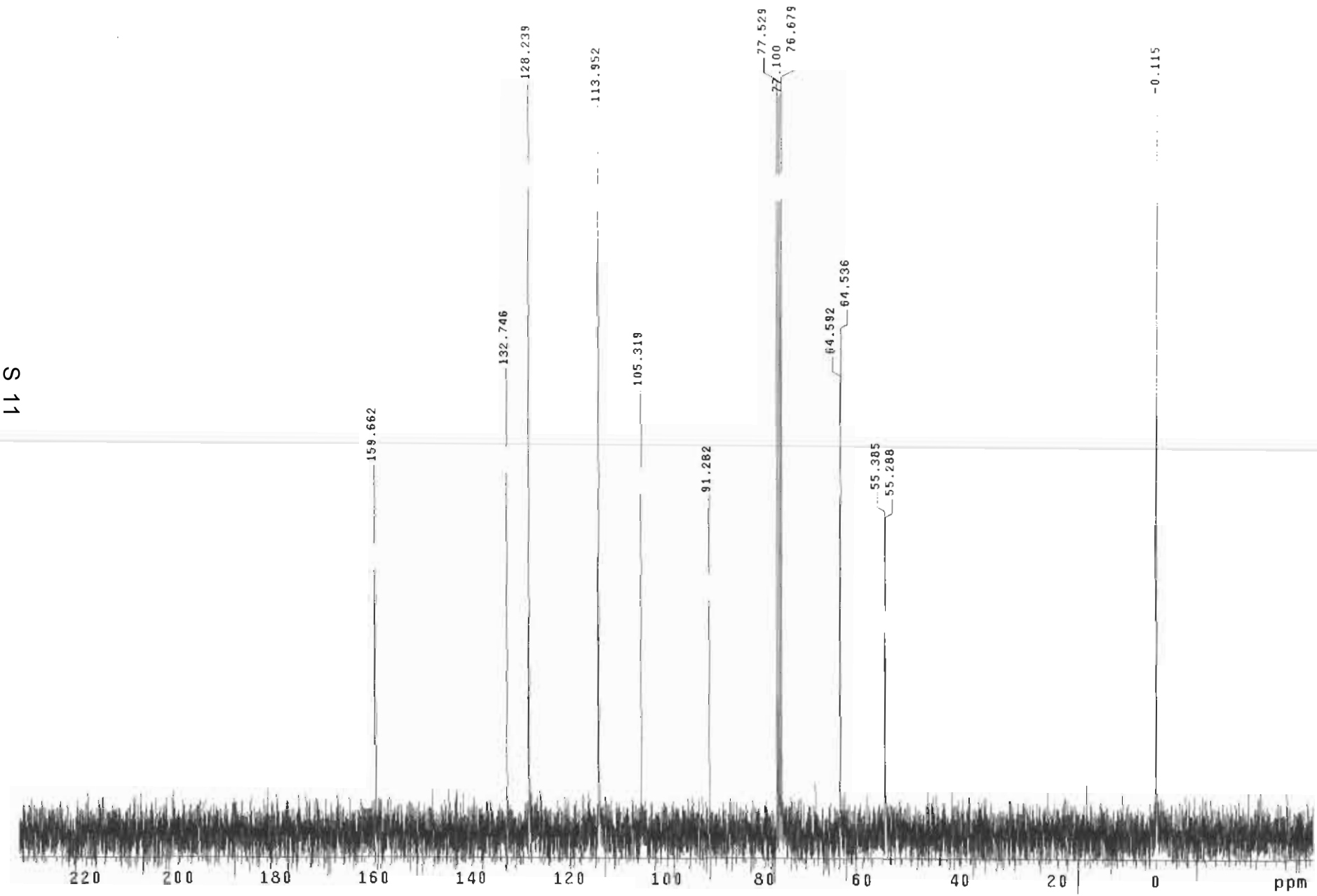
a3-177

Pulse Sequence: s2pul



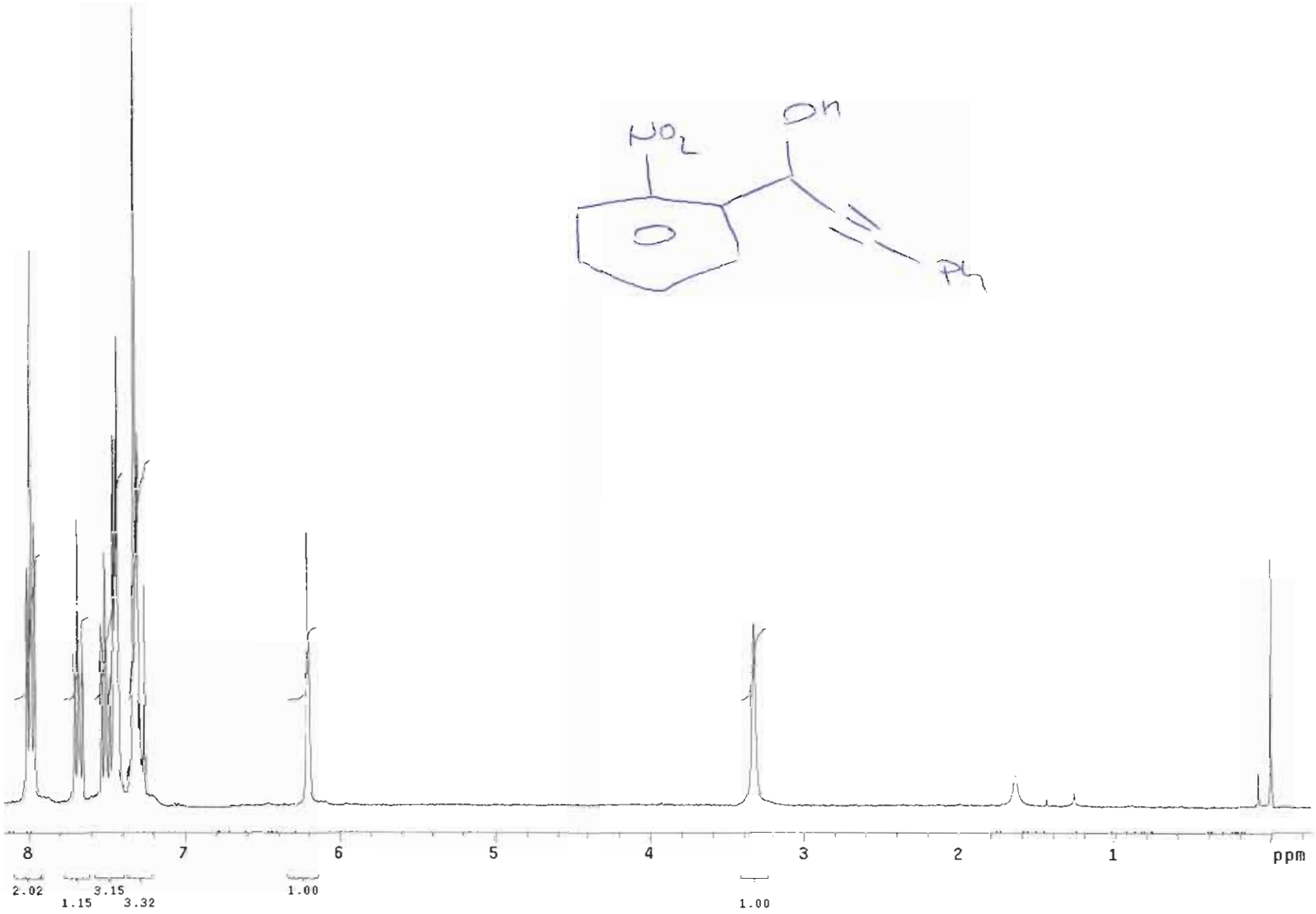
a3-177

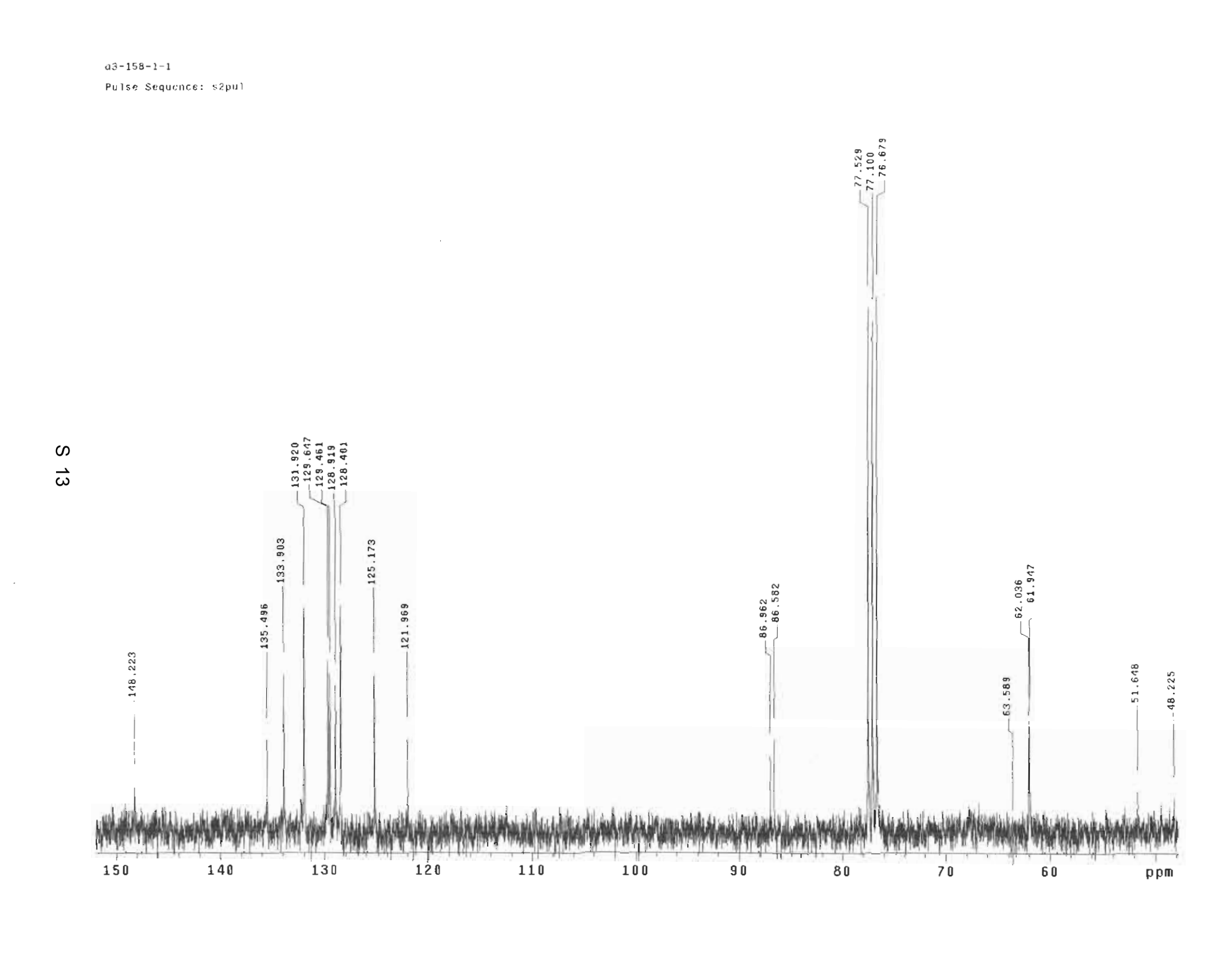
Pulse Sequence: s2pul



a3-158-1-1

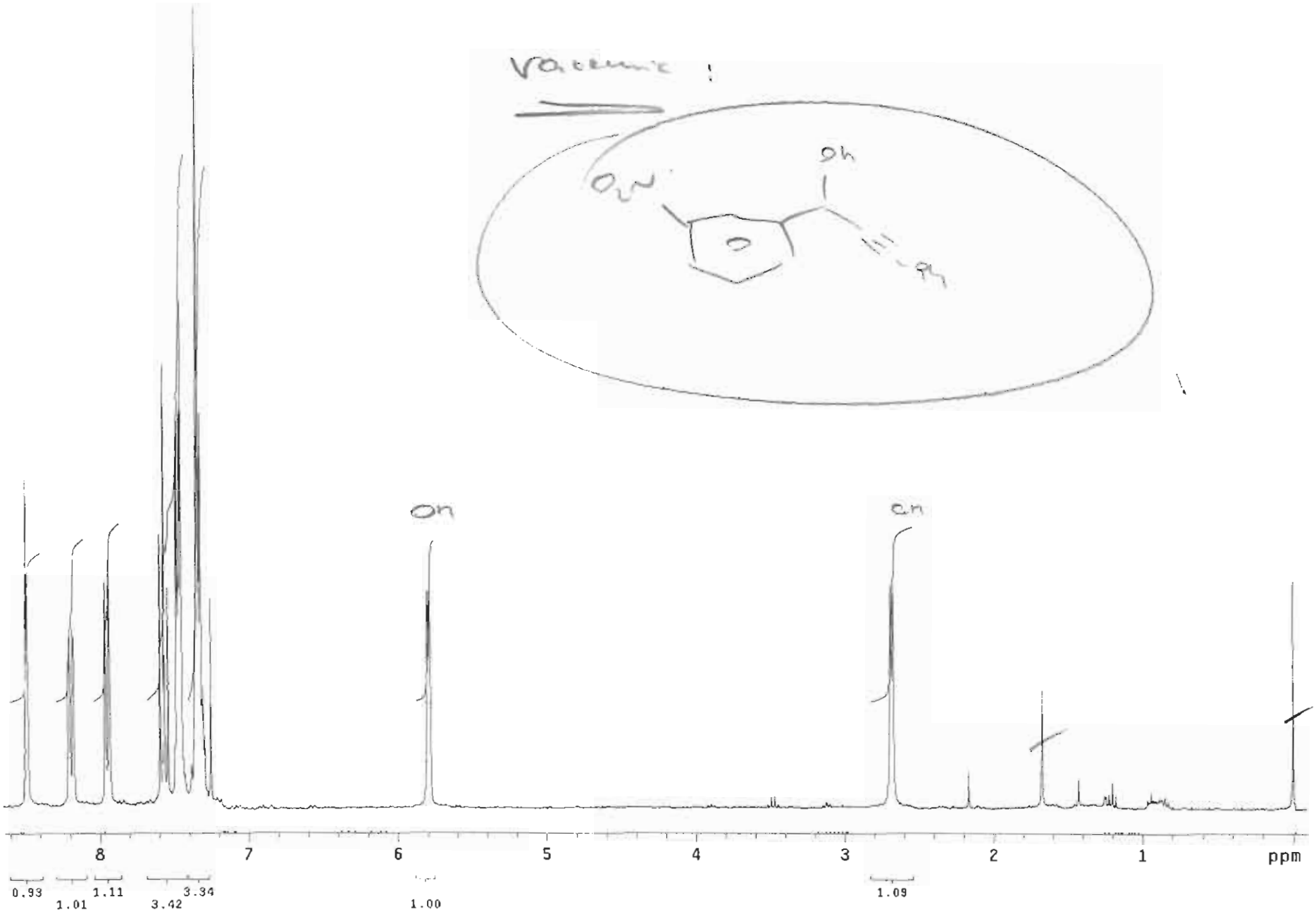
Pulse Sequence: s2pul

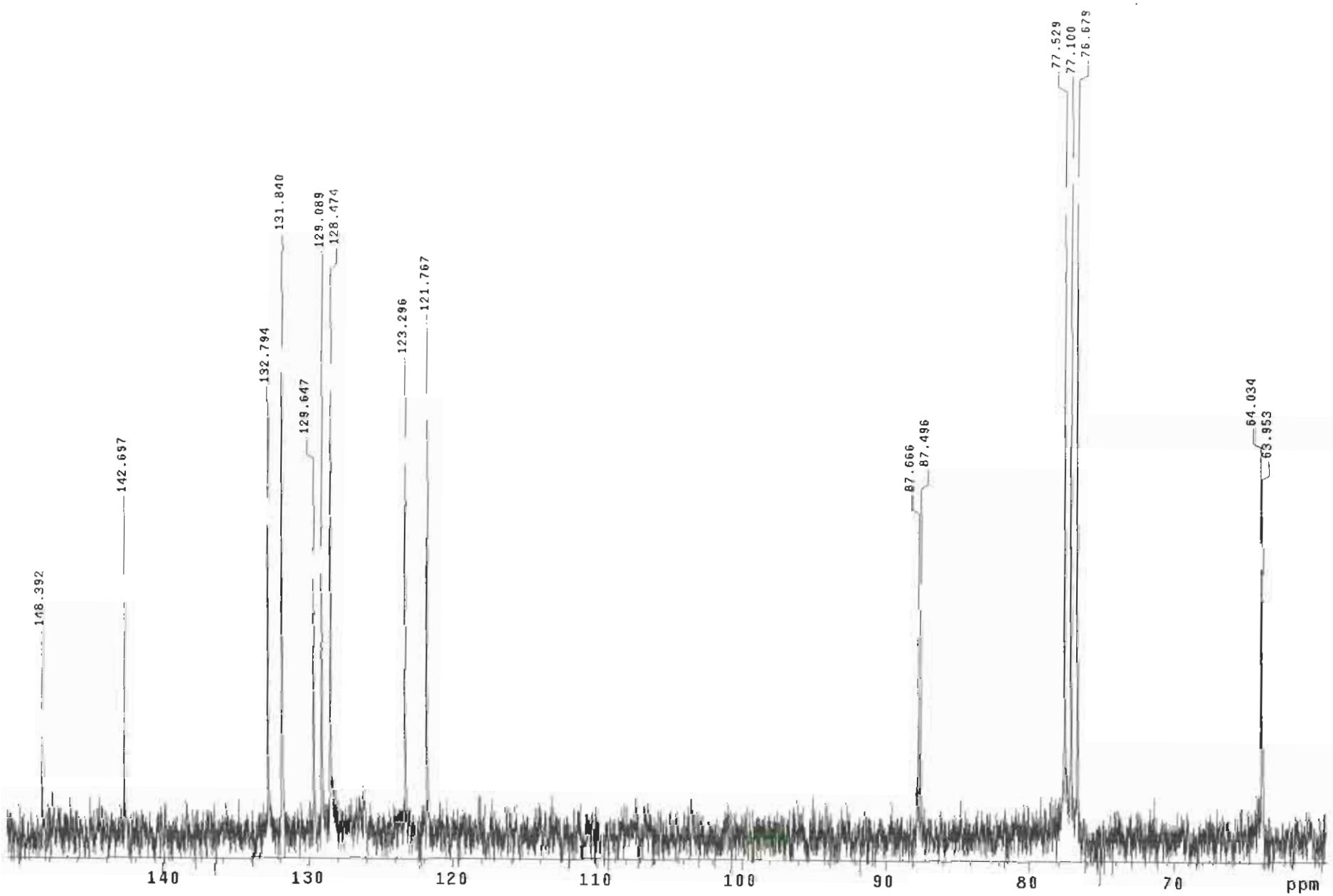



a3-158-1-1
Pulse Sequence: s2pul
148.223
135.496
133.903
131.920
129.647
129.461
128.919
128.401
125.173
121.969
86.962
86.582
77.529
77.100
76.679
63.589
62.036
61.947
51.648
48.225
150
140
130
120
110
100
90
80
70
60
ppm

a3-161

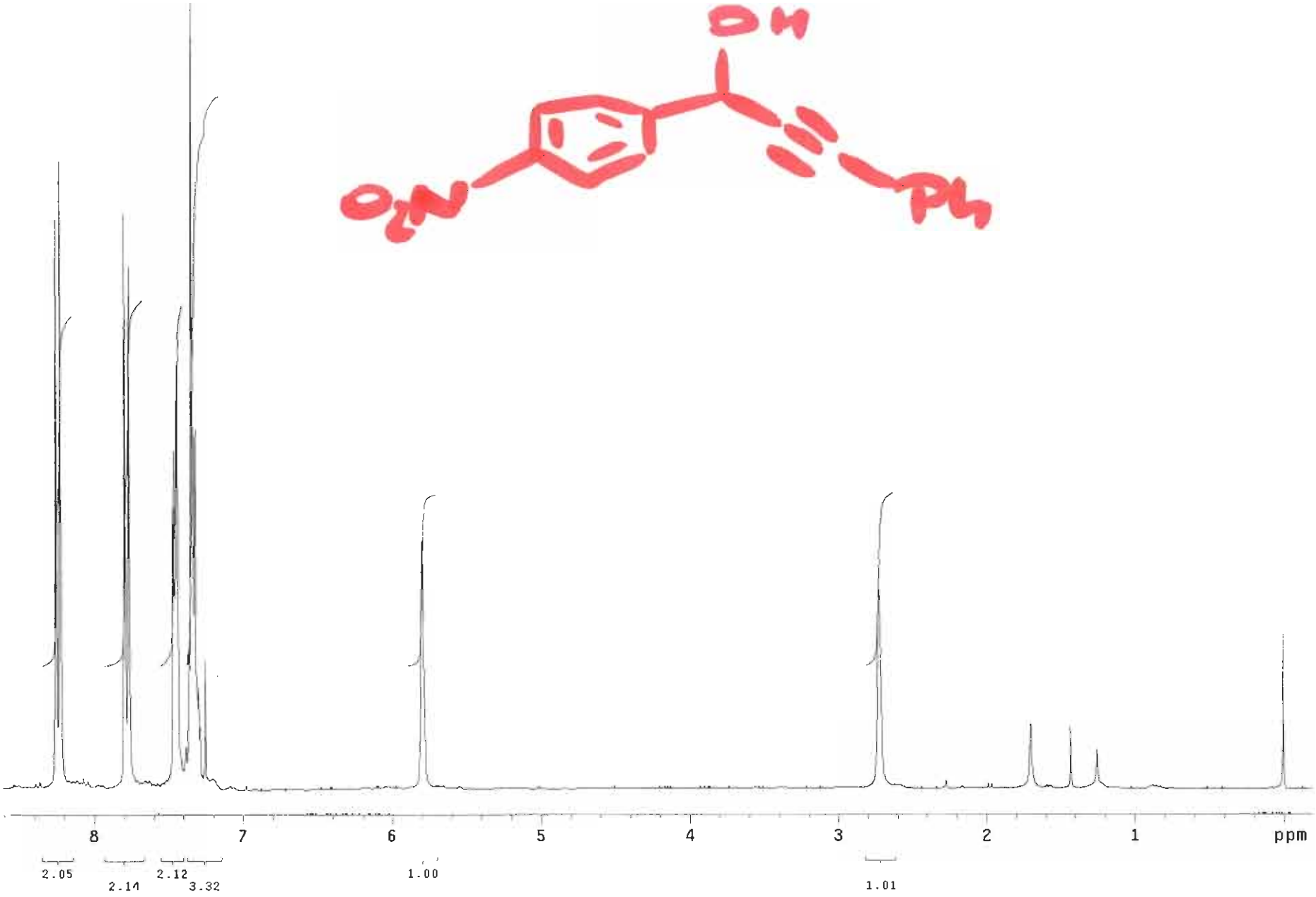
Pulse Sequence: s2pul

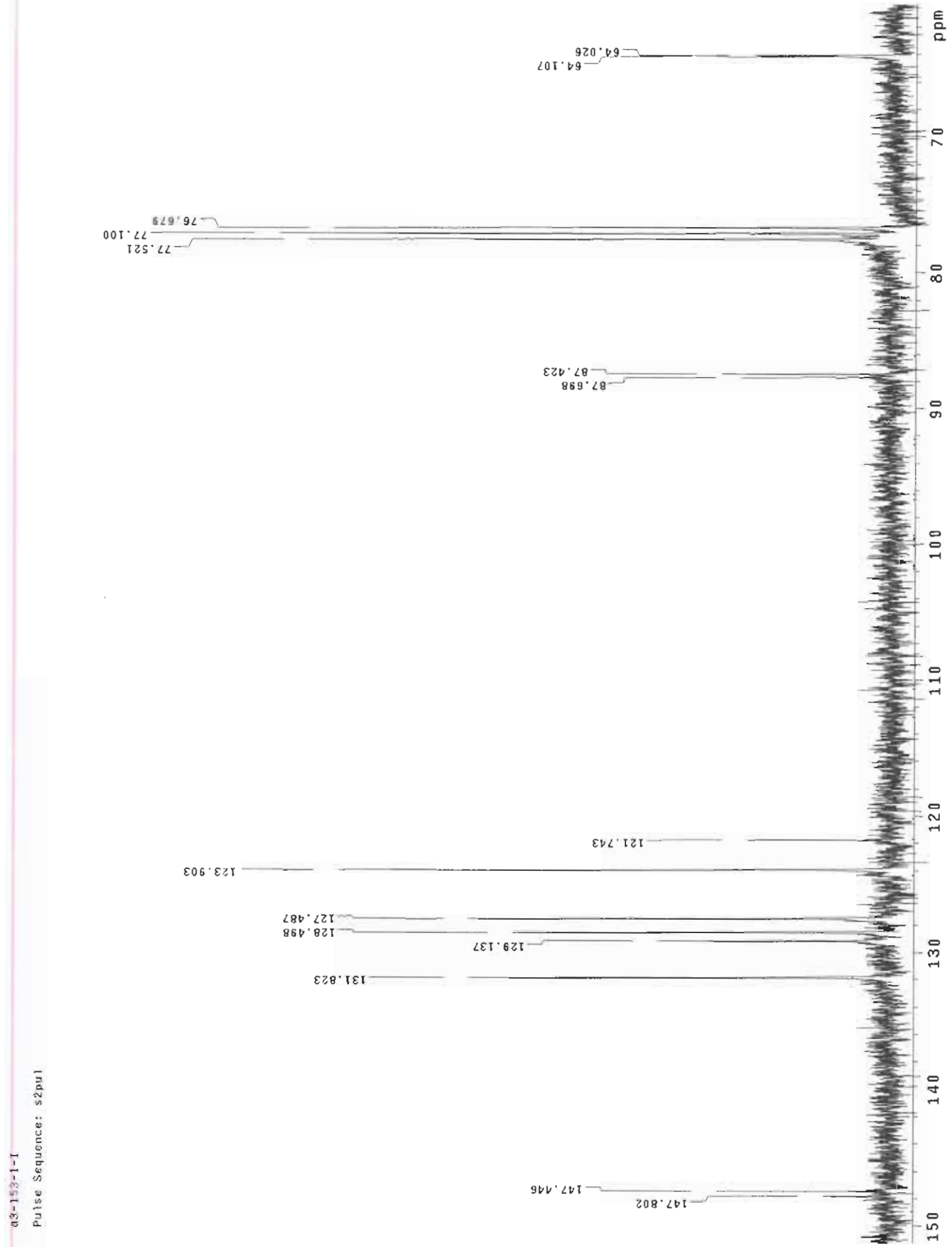



d3-158-2
Pulse Sequence: s2pul
148.392
142.697
132.794
131.840
129.647
129.089
128.474
123.296
121.767
87.666
87.496
77.529
77.100
76.679
64.034
63.953
140
130
120
110
100
90
80
70
ppm

a3-153-1-I

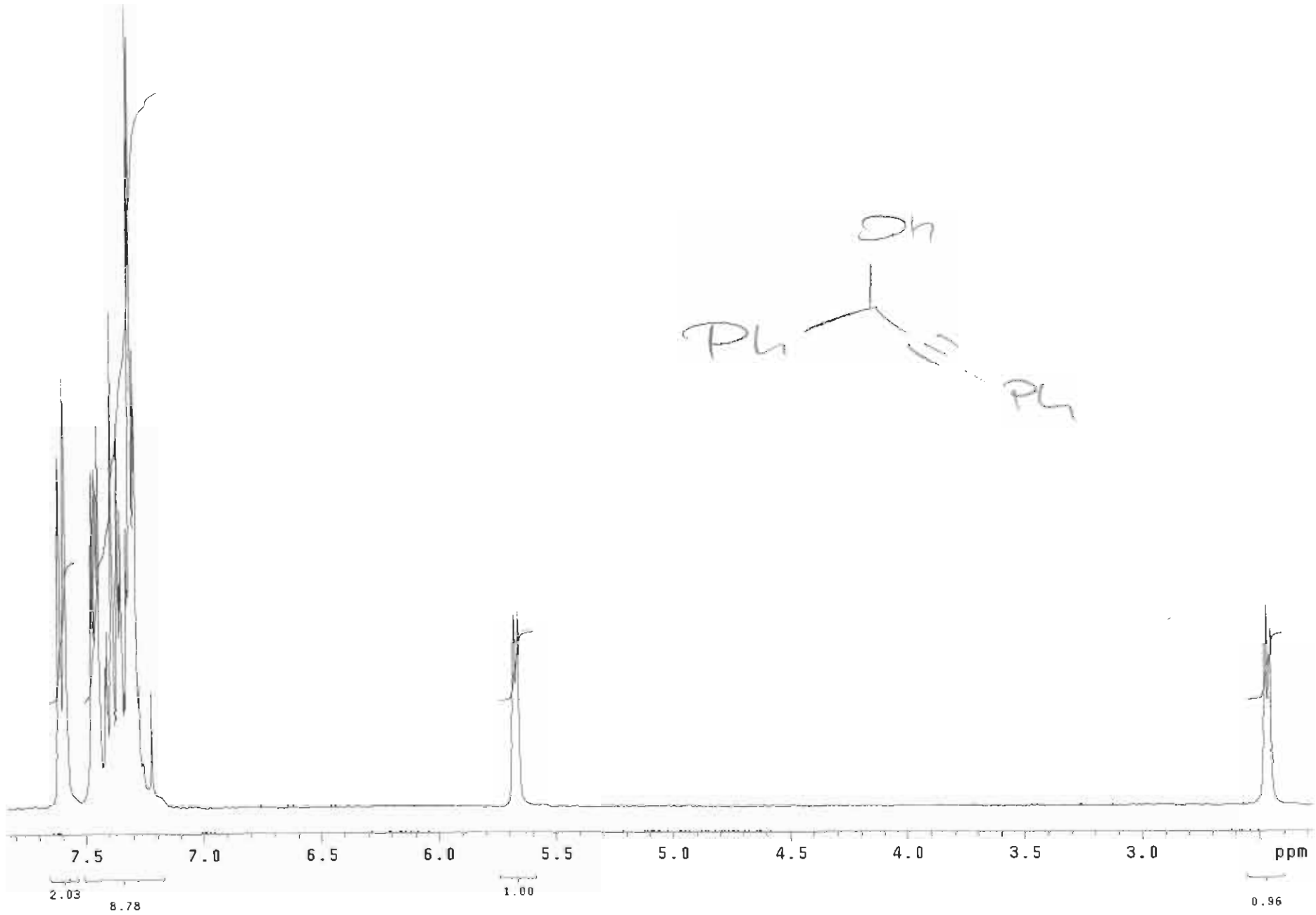
Pulse Sequence: s2pul

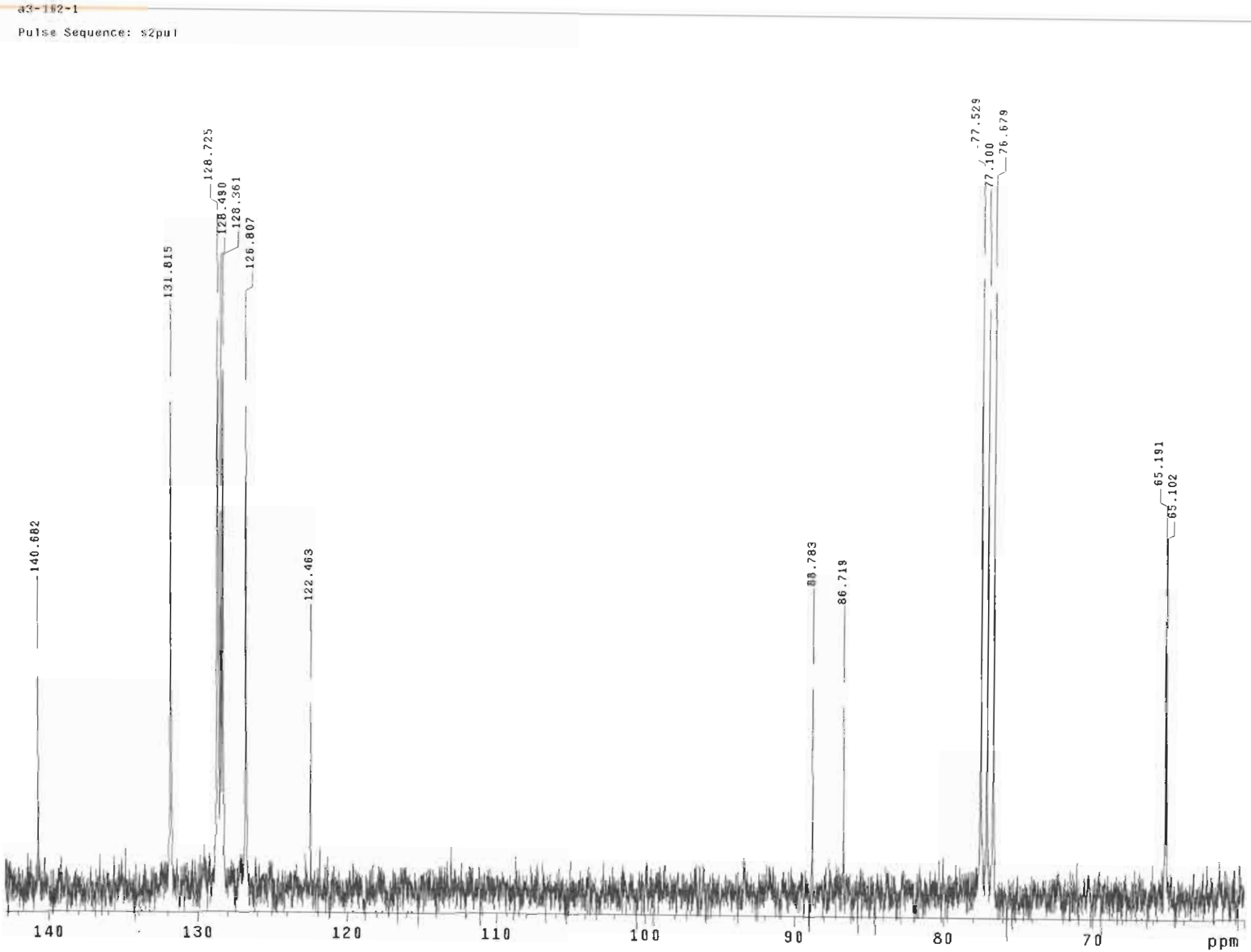



ppm
70
80
90
100
110
120
130
140
150
64.026
64.107
76.679
77.100
77.521
87.423
87.698
121.743
123.903
127.487
128.498
129.137
131.823
147.446
147.802
a3-153-1-1
Pulse Sequence: s2pul

a3-162-1

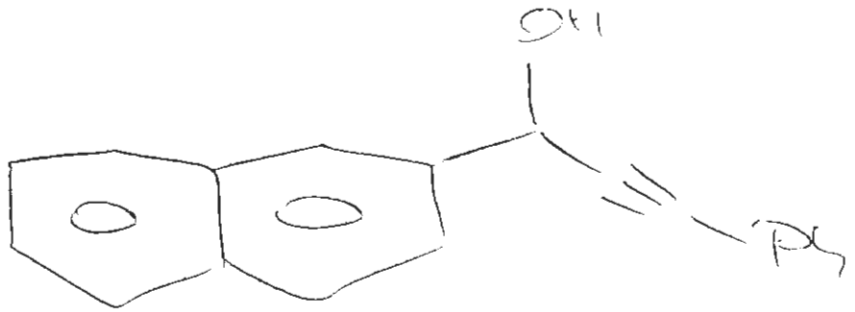
Pulse Sequence: s2pul

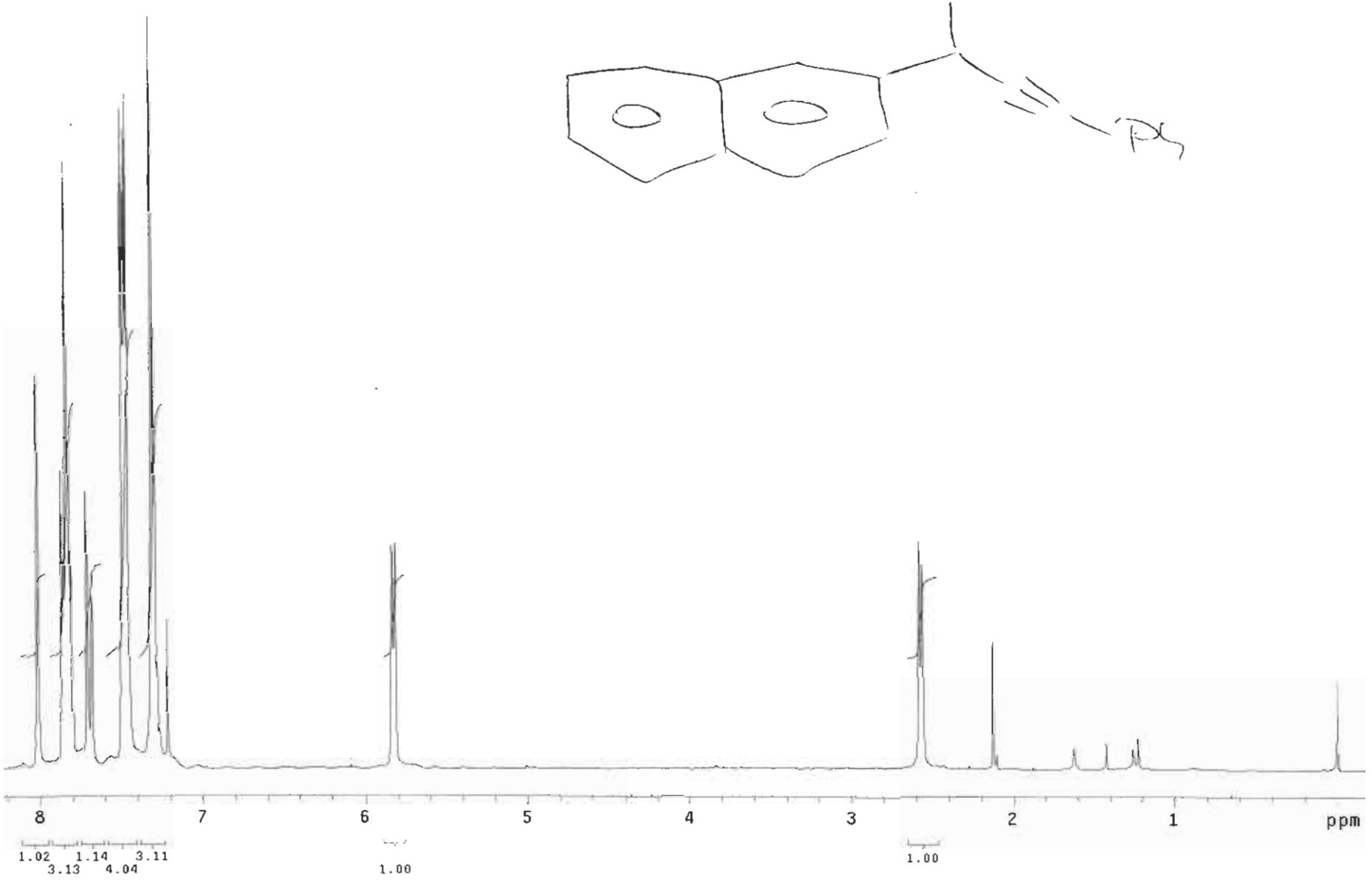



a3-182-1
Pulse Sequence: s2pul
140.682
131.815
128.725
128.490
128.361
126.807
122.463
88.783
86.719
77.529
77.100
76.679
65.191
65.102
140
130
120
110
100
90
80
70
ppm

a3-174-1

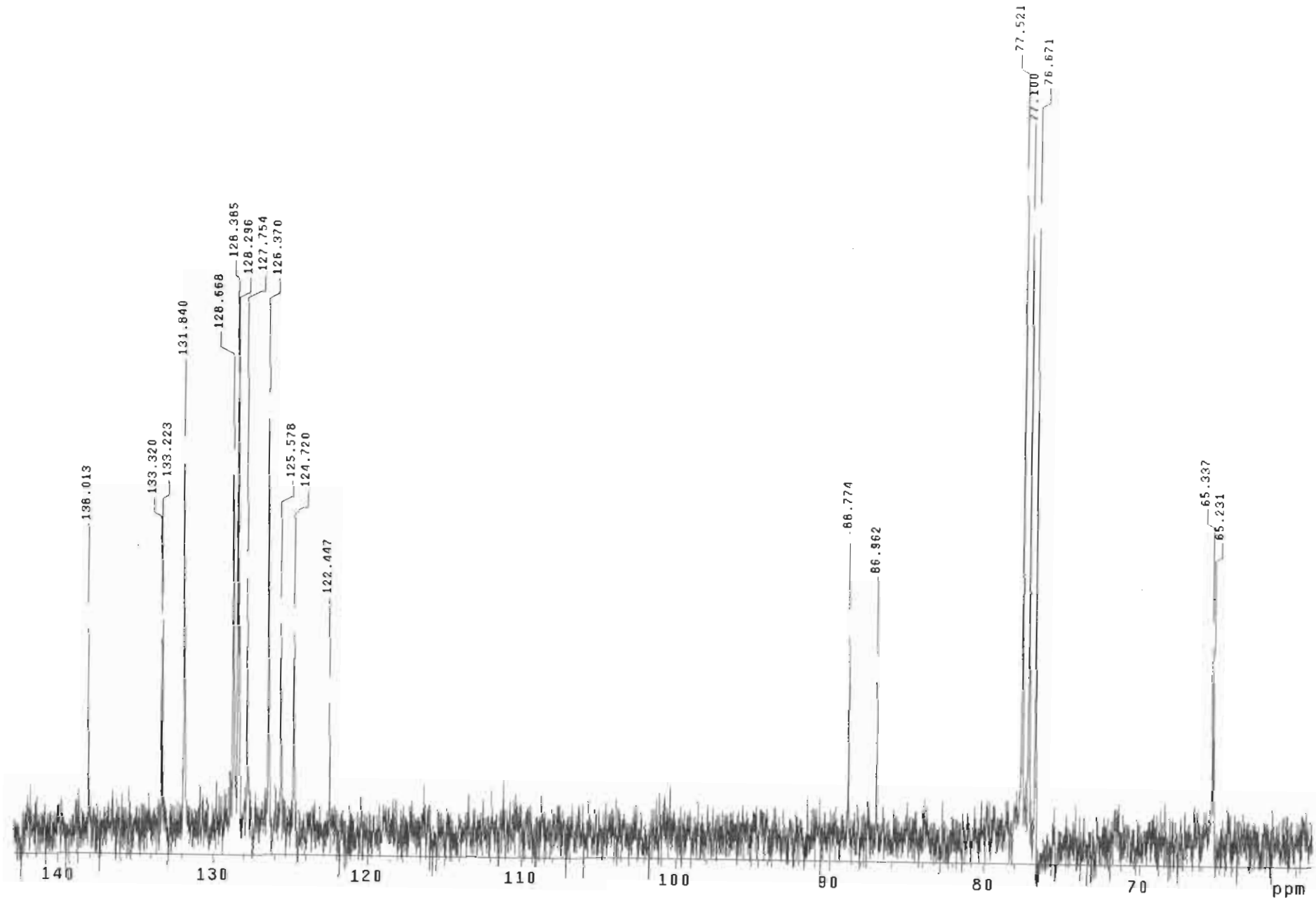
Pulse Sequence: s2pul





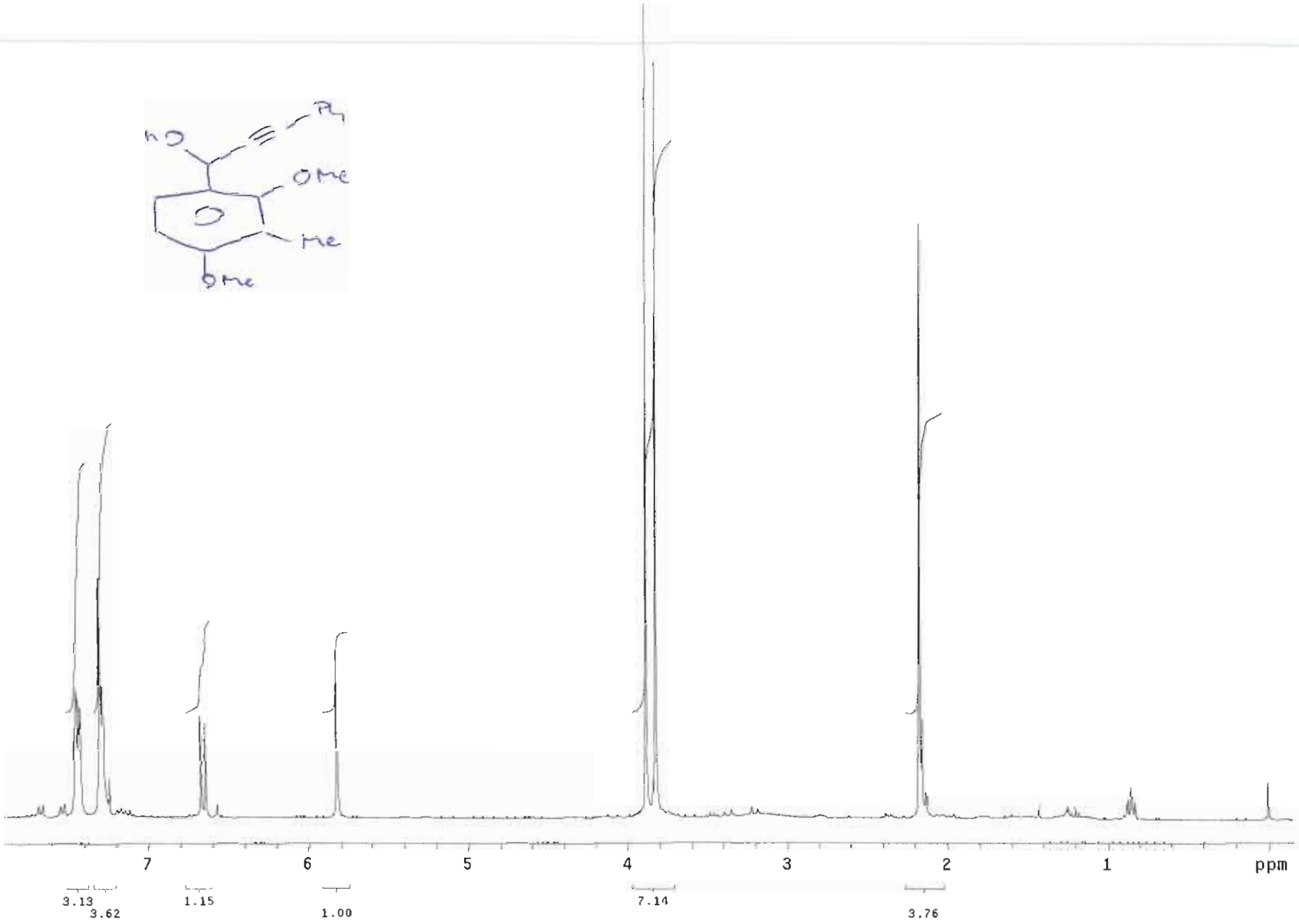
a3-174-1

Pulse Sequence: s2pul



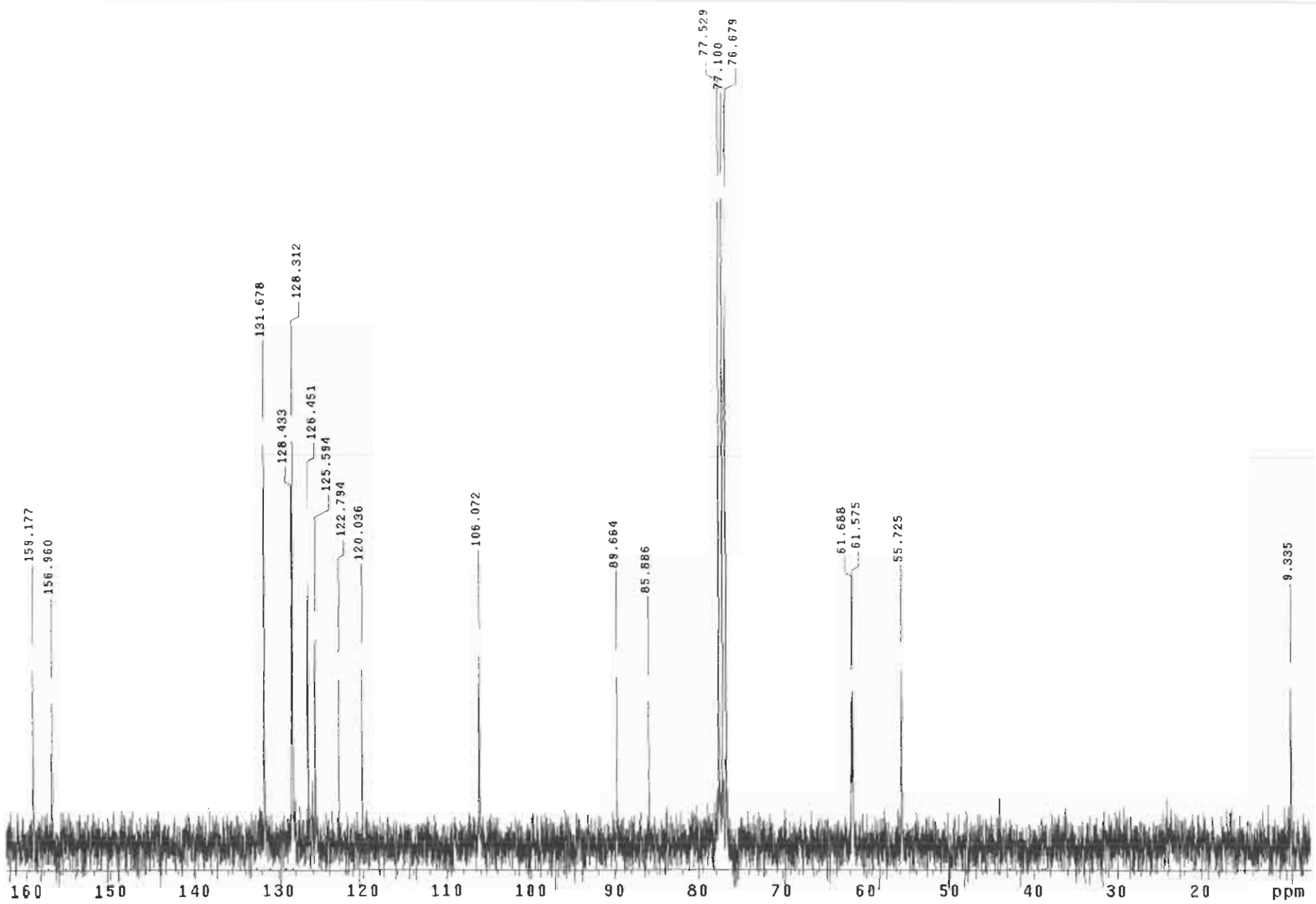
a3-157-2

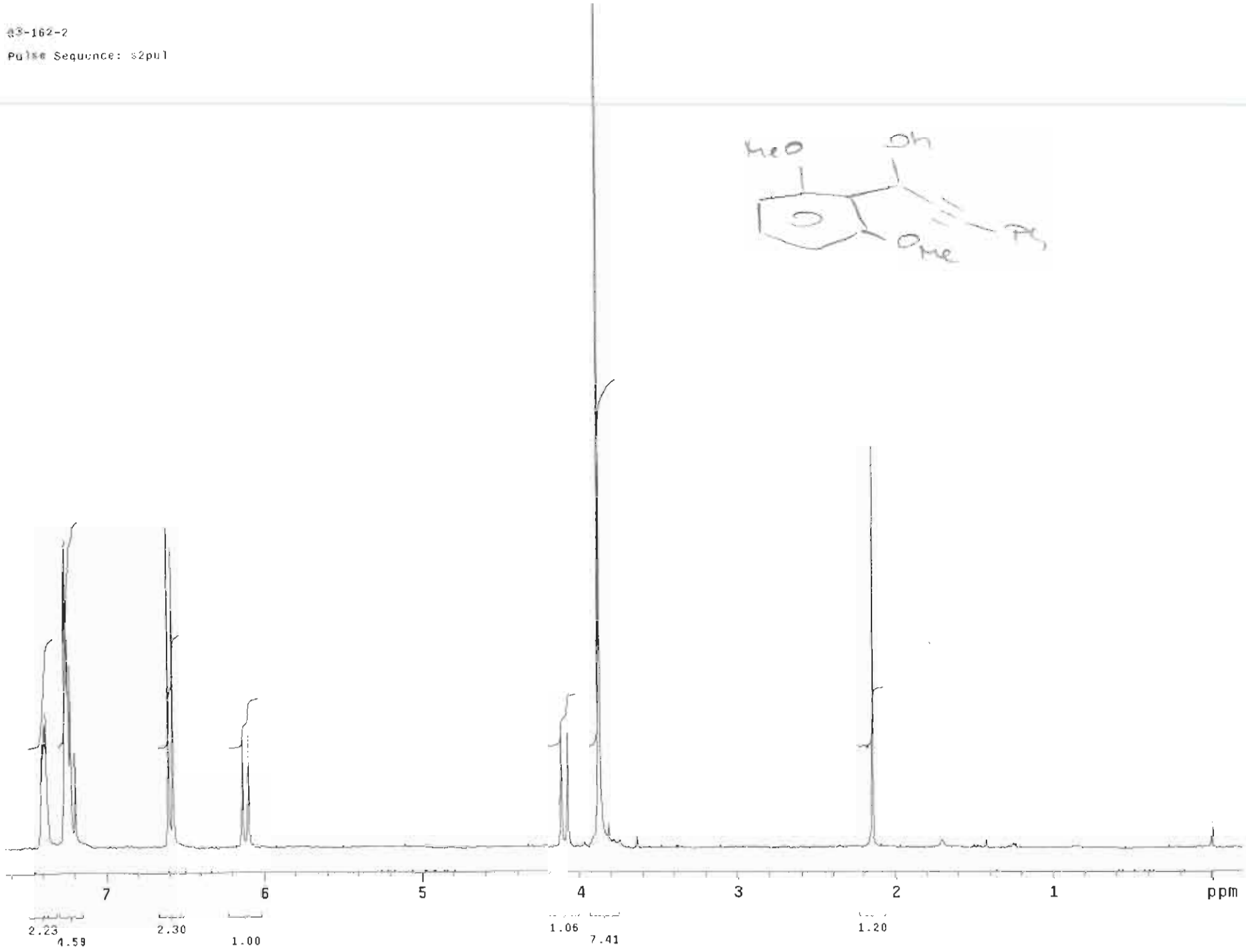
Pulse Sequence: s2pul



a3-157-2

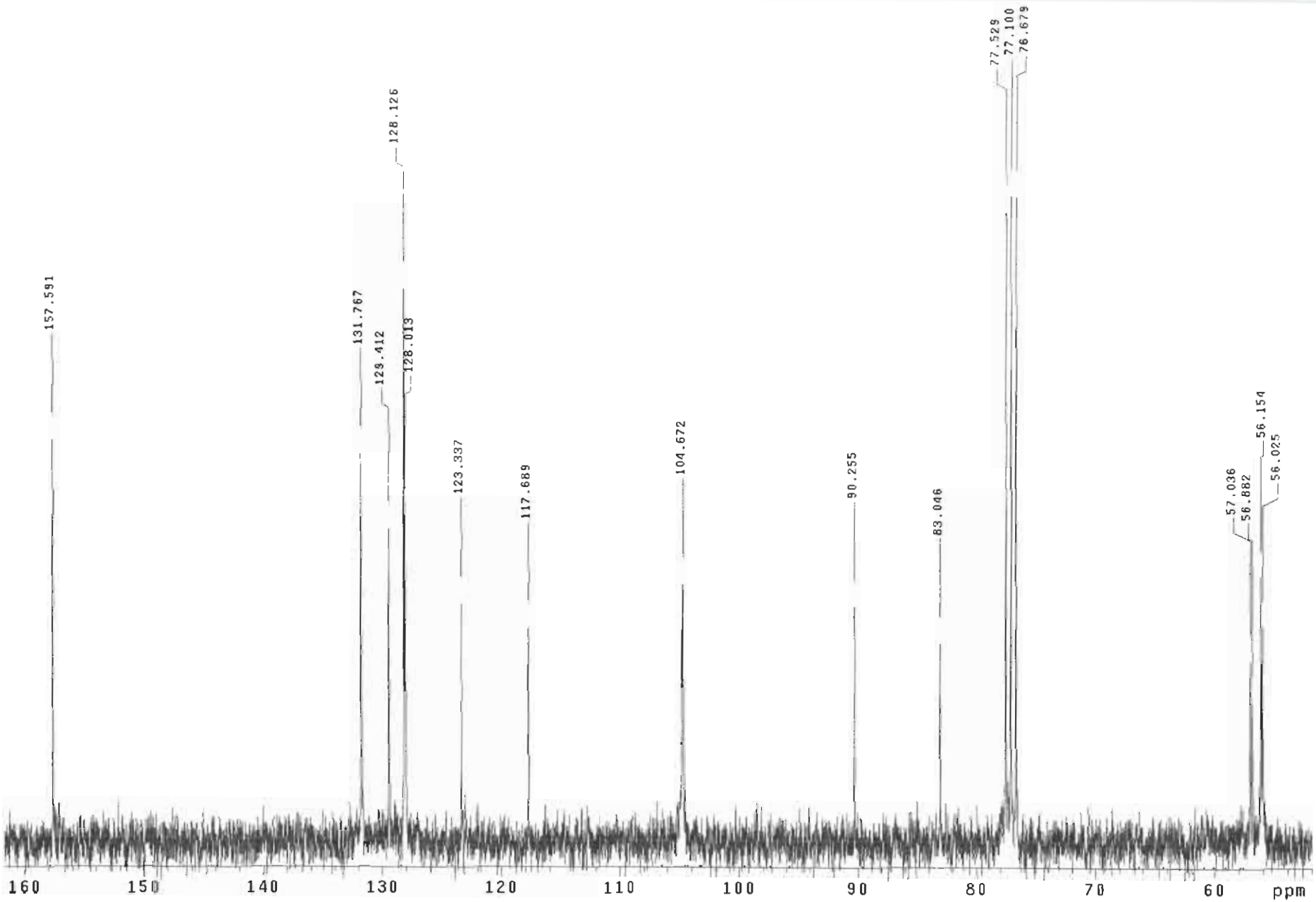
Pulse Sequence: s2pul




03-162-2
Pulse Sequence: s2pul
MeO
OH
OMe
7
6
5
4
3
2
1
ppm
2.23
4.59
2.30
1.00
1.06
7.41
1.20

a3-162-2

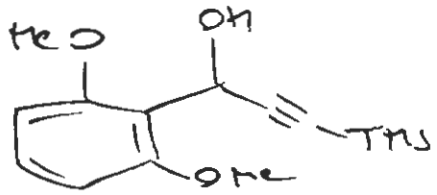
Pulse Sequence: s2pul

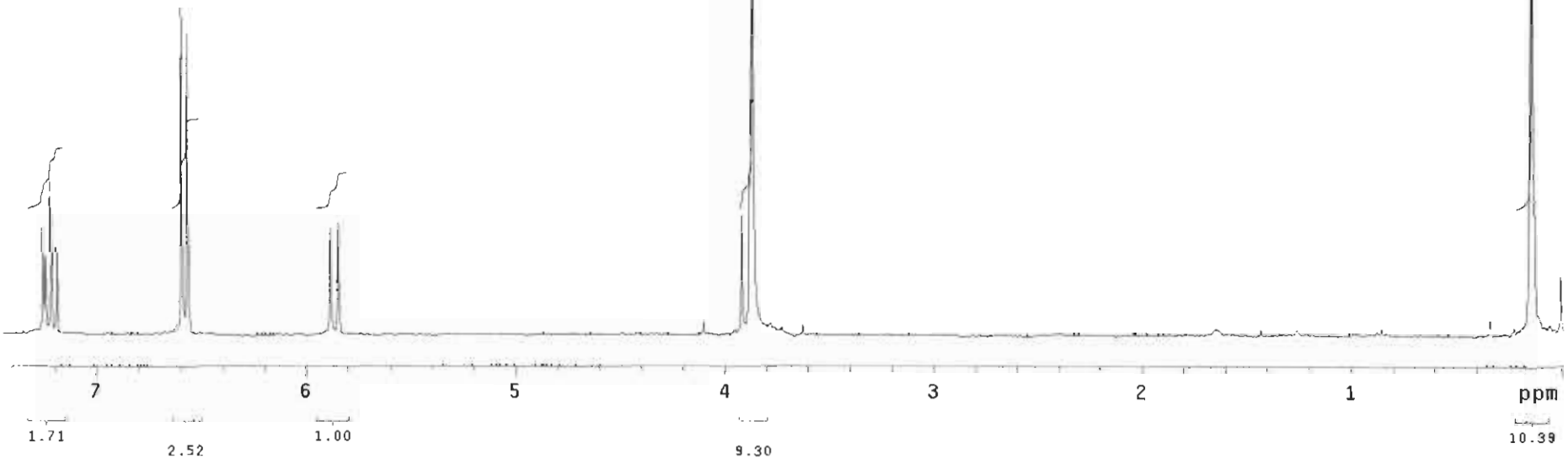


a3-181-

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300 "gem300"

Relax. delay 1.000 sec
Pulse 51.4 degrees
Acq. time 2.000 sec
Width 4000.0 Hz
12 repetitions
OBSERVE H1, 300.0354248 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 0 min, 0 sec

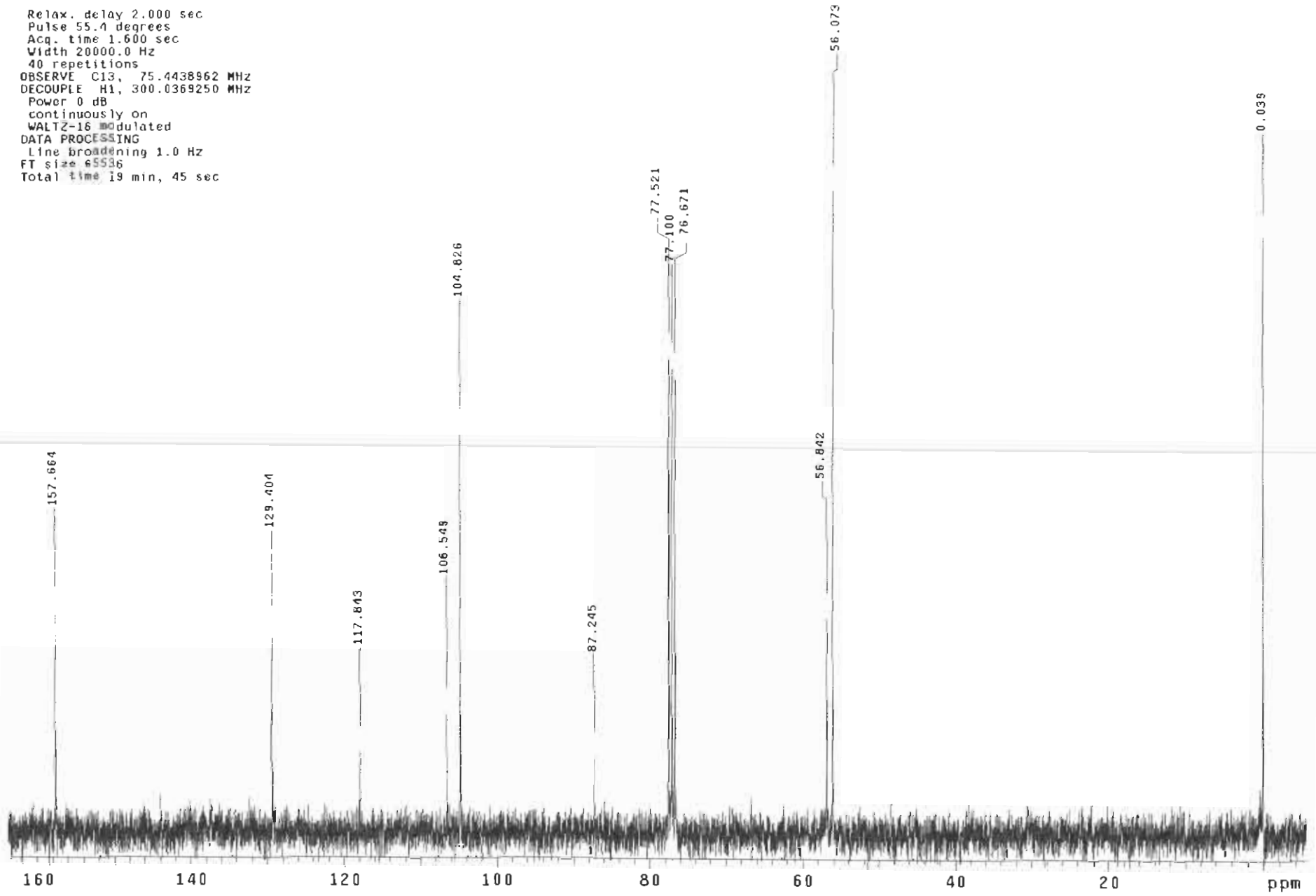




a3-181

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300 "gem300"

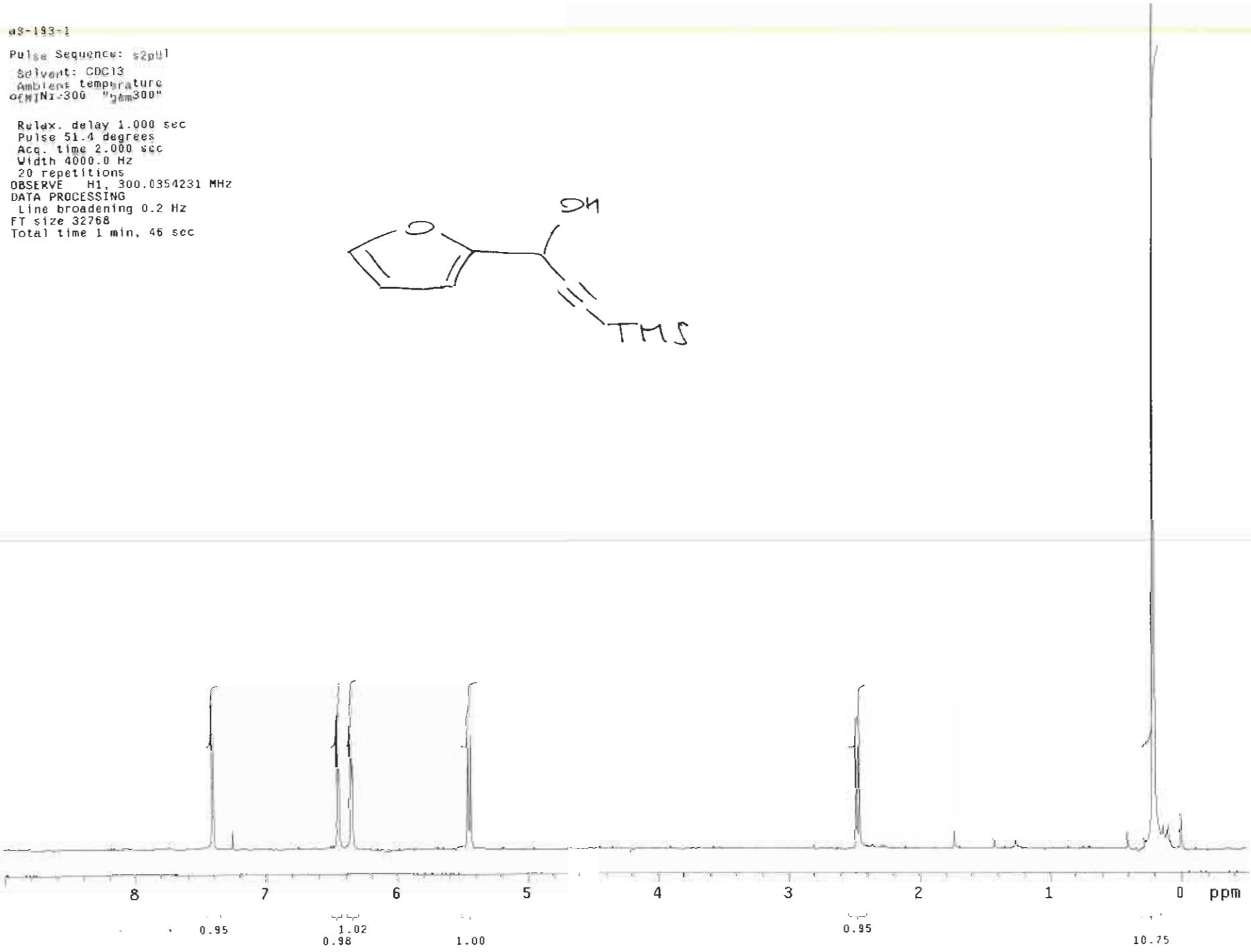
Relax. delay 2.000 sec
Pulse 55.4 degrees
Acq. time 1.600 sec
Width 20000.0 Hz
40 repetitions
OBSERVE C13, 75.4438962 MHz
DECOUPLE H1, 300.0369250 MHz
Power 0 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 19 min, 45 sec



a3-193-1

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300 "gem300"

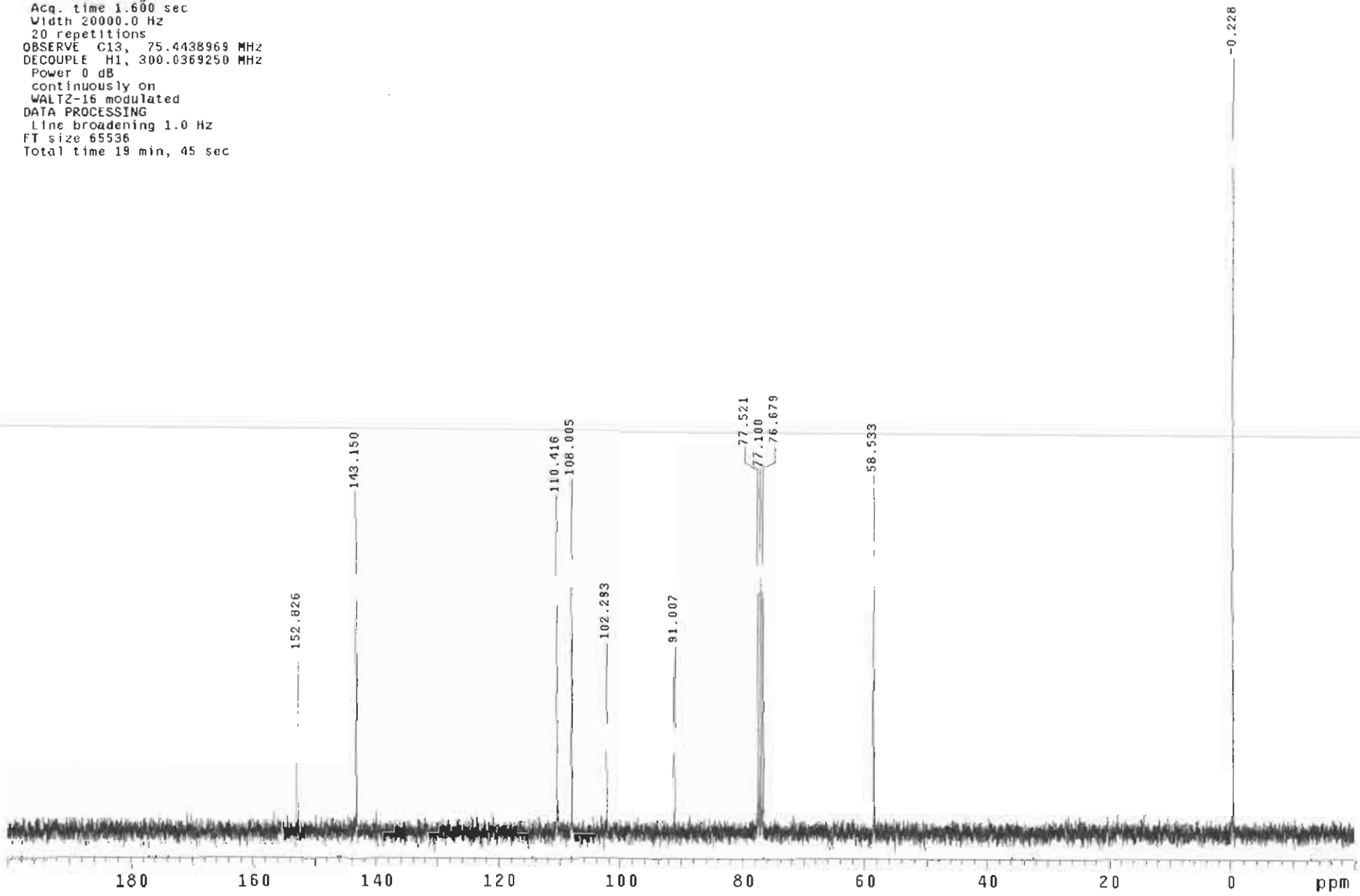
Relax. delay 1.000 sec
Pulse 51.4 degrees
Acq. time 2.000 sec
Width 4000.0 Hz
20 repetitions
OBSERVE H1, 300.0354231 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 1 min, 46 sec



a3-193-1

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300 "gem300"

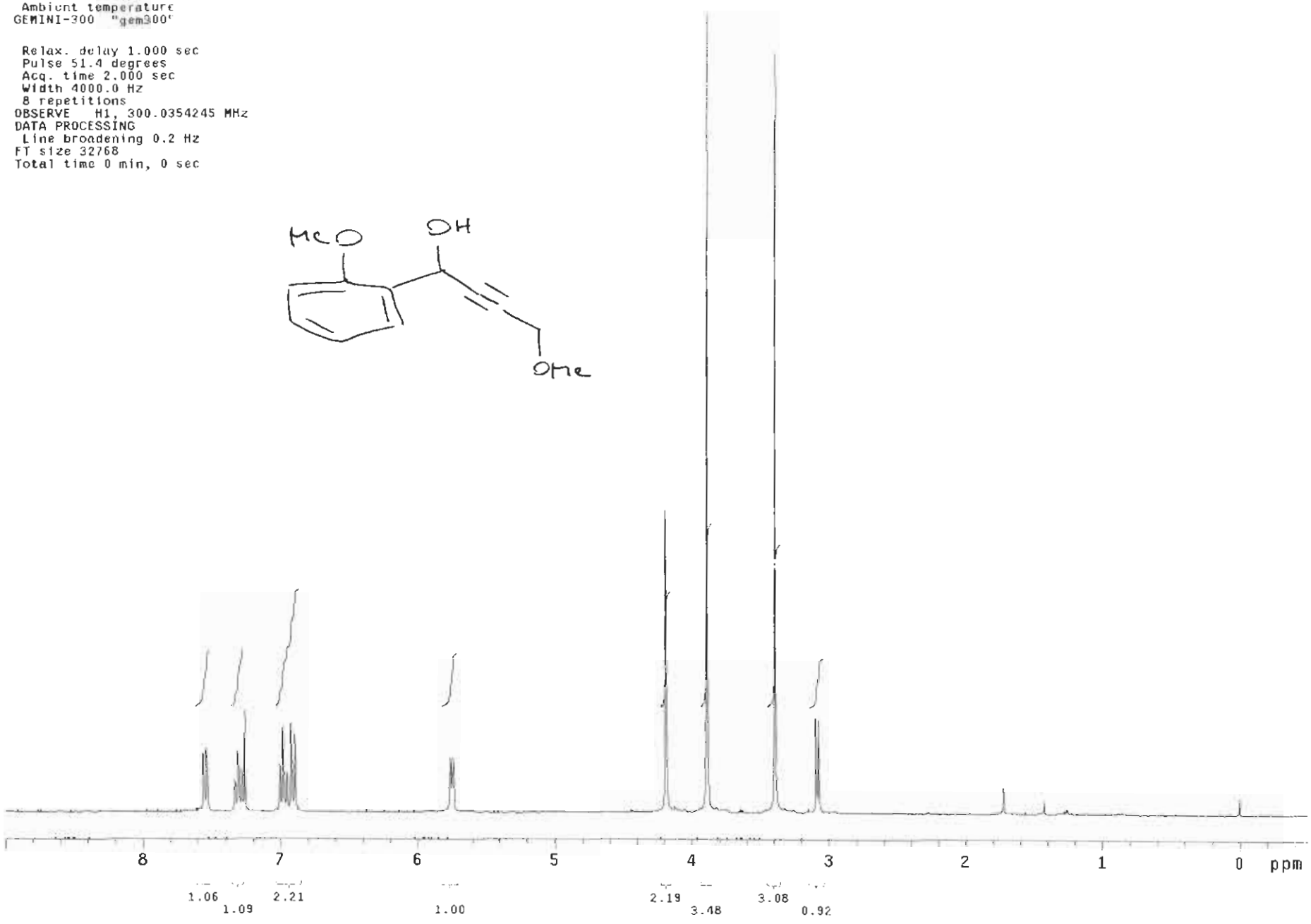
Relax. delay 2.000 sec
Pulse 55.4 degrees
Acq. time 1.600 sec
Width 20000.0 Hz
20 repetitions
OBSERVE C13, 75.4438969 MHz
DECOUPLE H1, 300.0369250 MHz
Power 0 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 19 min, 45 sec



a3-188-1

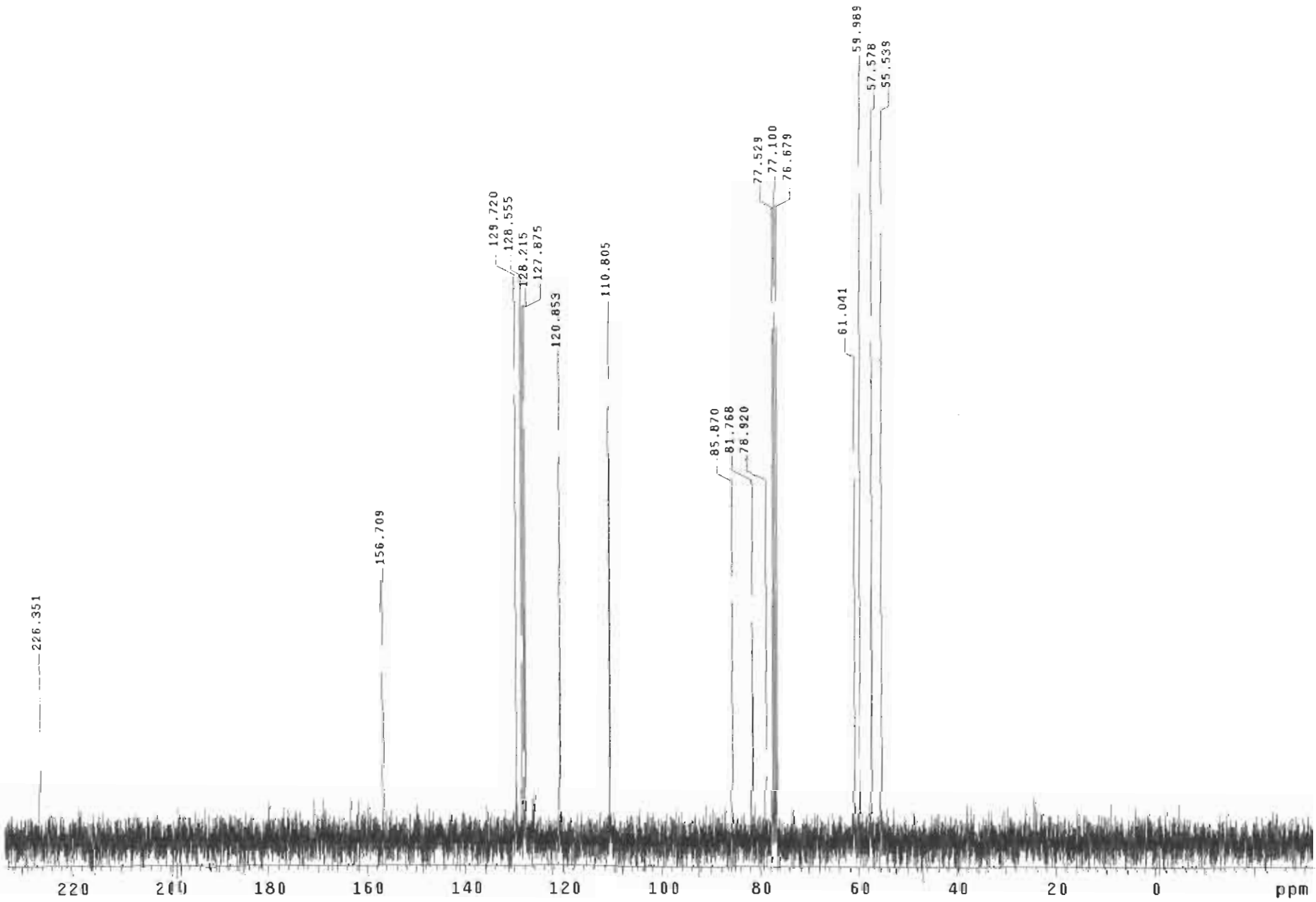
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300 "gem300"

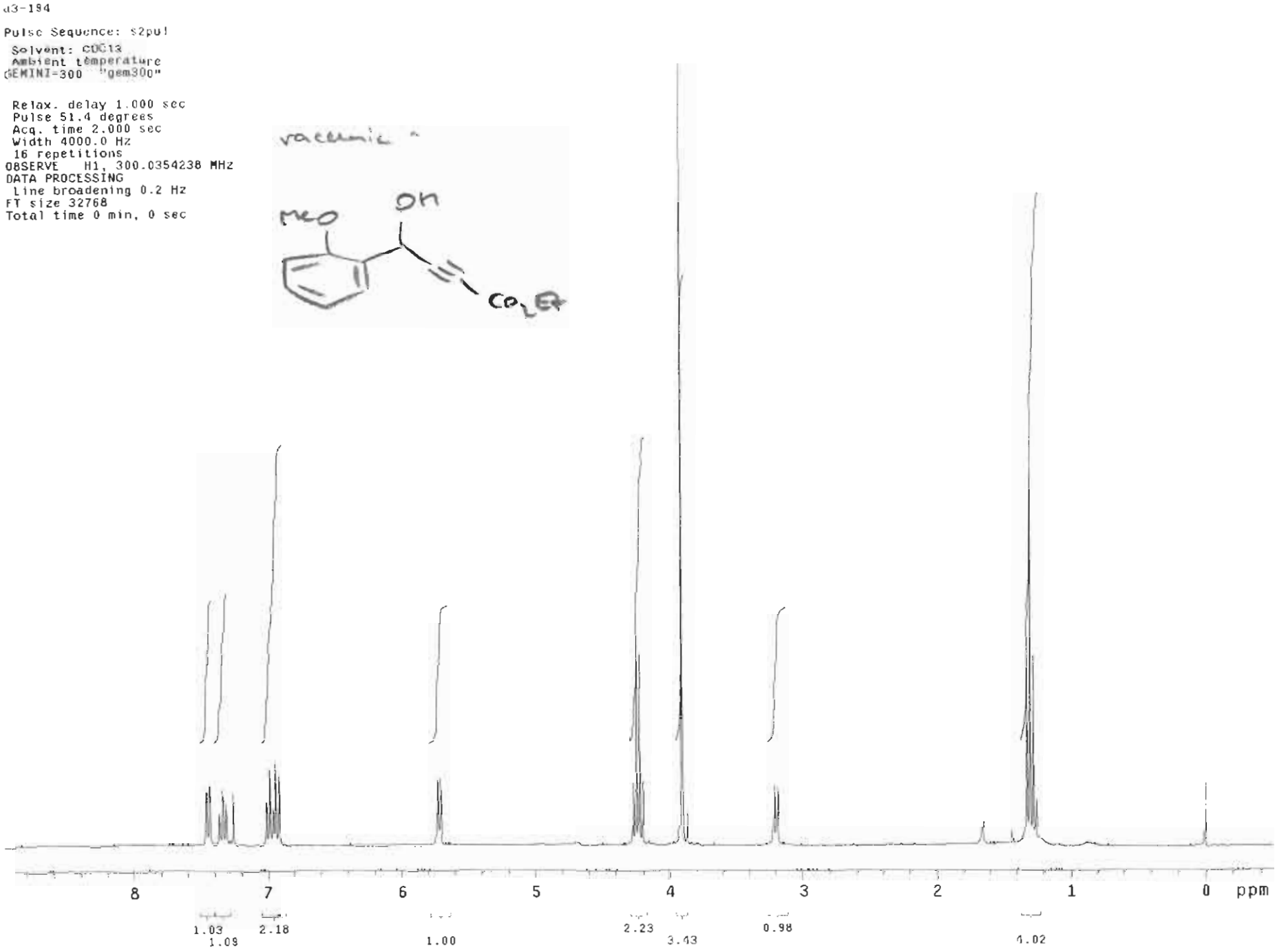
Relax. delay 1.000 sec
Pulse 51.4 degrees
Acq. time 2.000 sec
Width 4000.0 Hz
8 repetitions
OBSERVE H1, 300.0354245 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 0 min, 0 sec



a3-187-1

Pulse Sequence: s2pul

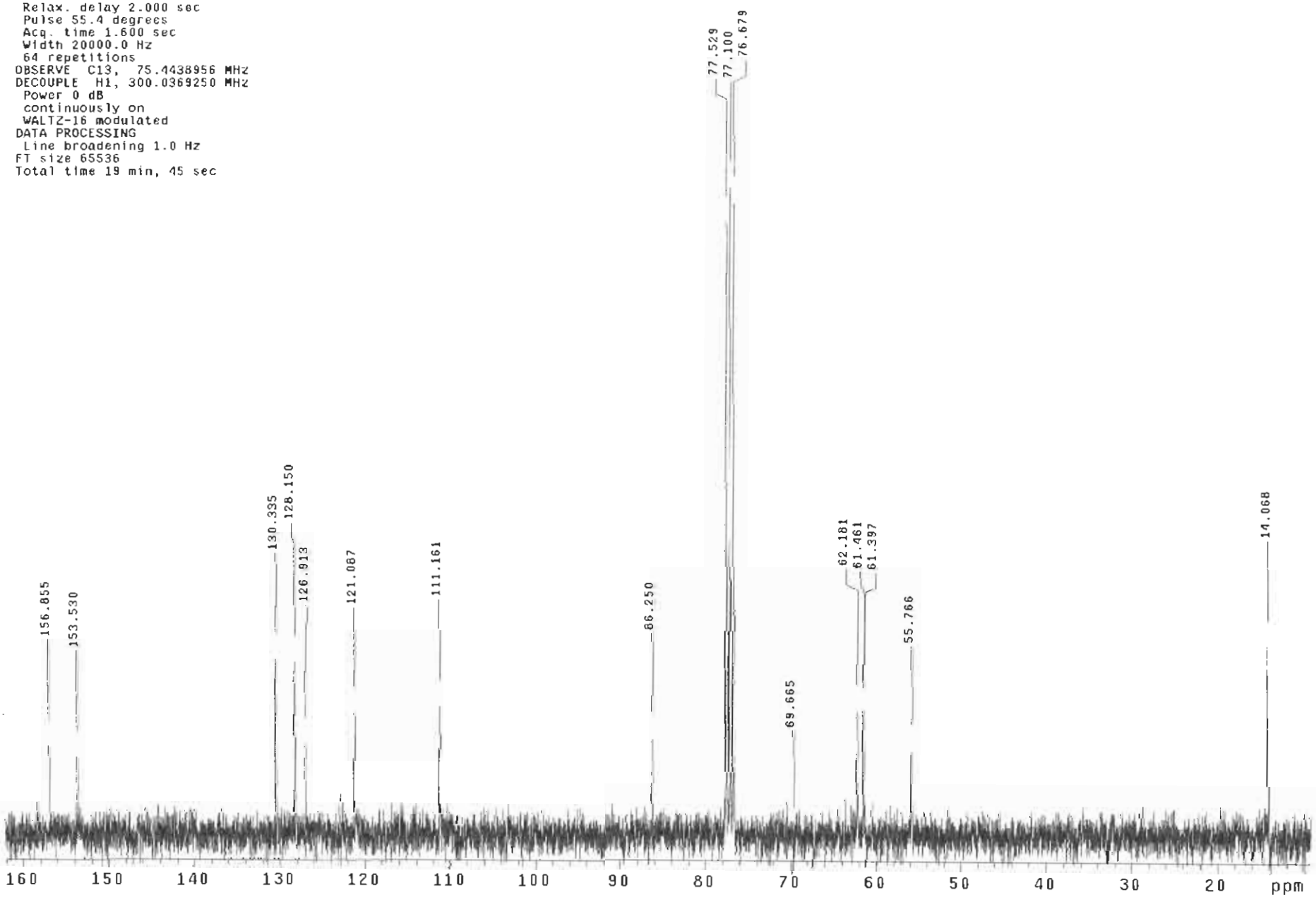



a3-194
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300 "gem300"
Relax. delay 1.000 sec
Pulse 51.4 degrees
Acq. time 2.000 sec
Width 4000.0 Hz
16 repetitions
OBSERVE H1, 300.0354238 MHz
DATA PROCESSING
Line broadening 0.2 Hz
FT size 32768
Total time 0 min, 0 sec
racemic
MeO
OH
CO2Et
8
7
6
5
4
3
2
1
0
ppm
1.03
1.09
2.18
1.00
2.23
3.43
0.98
4.02

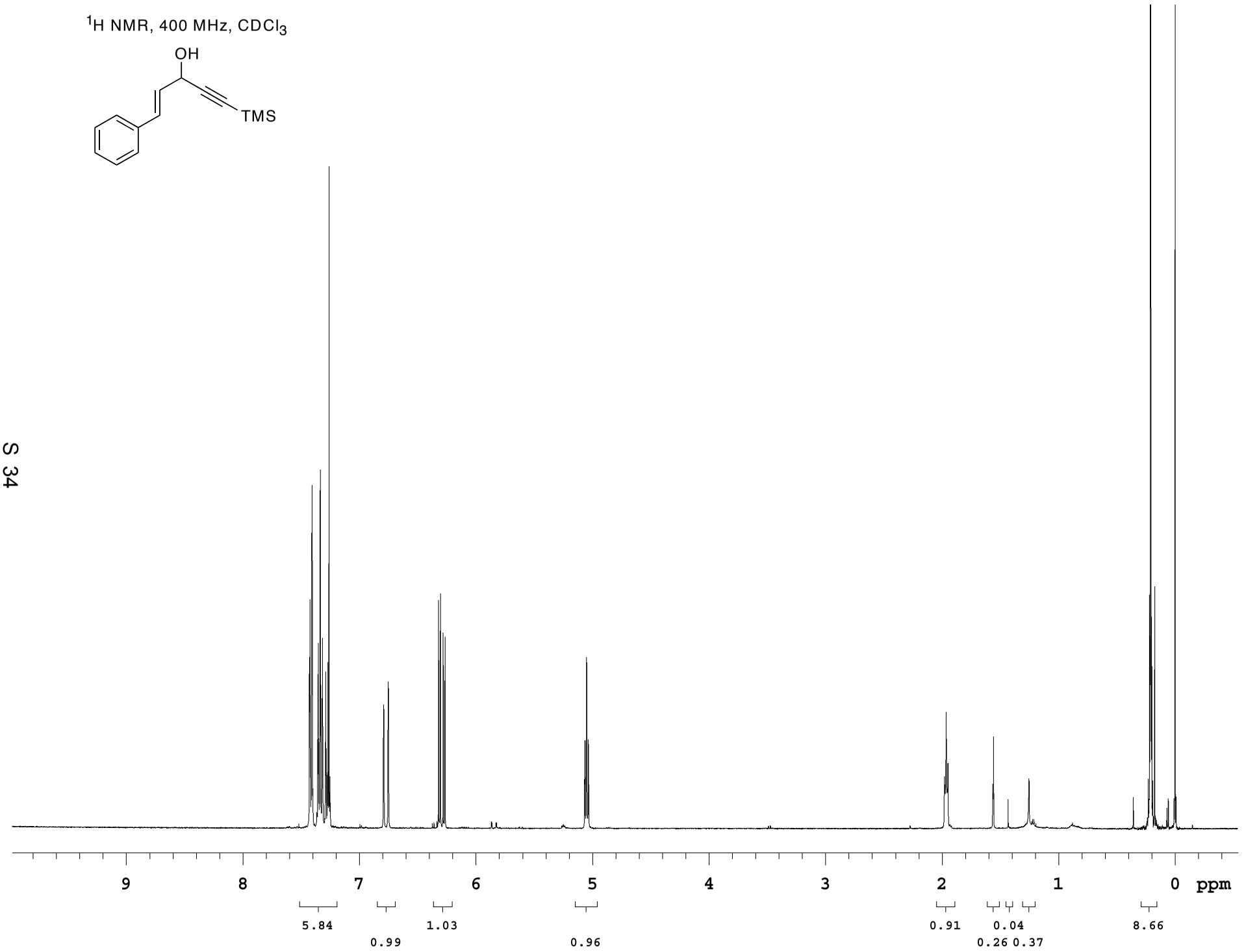
a3-194

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-300 "gem300"

Relax. delay 2.000 sec
Pulse 55.4 degrees
Acq. time 1.600 sec
Width 20000.0 Hz
64 repetitions
OBSERVE C13, 75.4438956 MHz
DECOUPLE H1, 300.0369250 MHz
Power 0 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 19 min, 45 sec

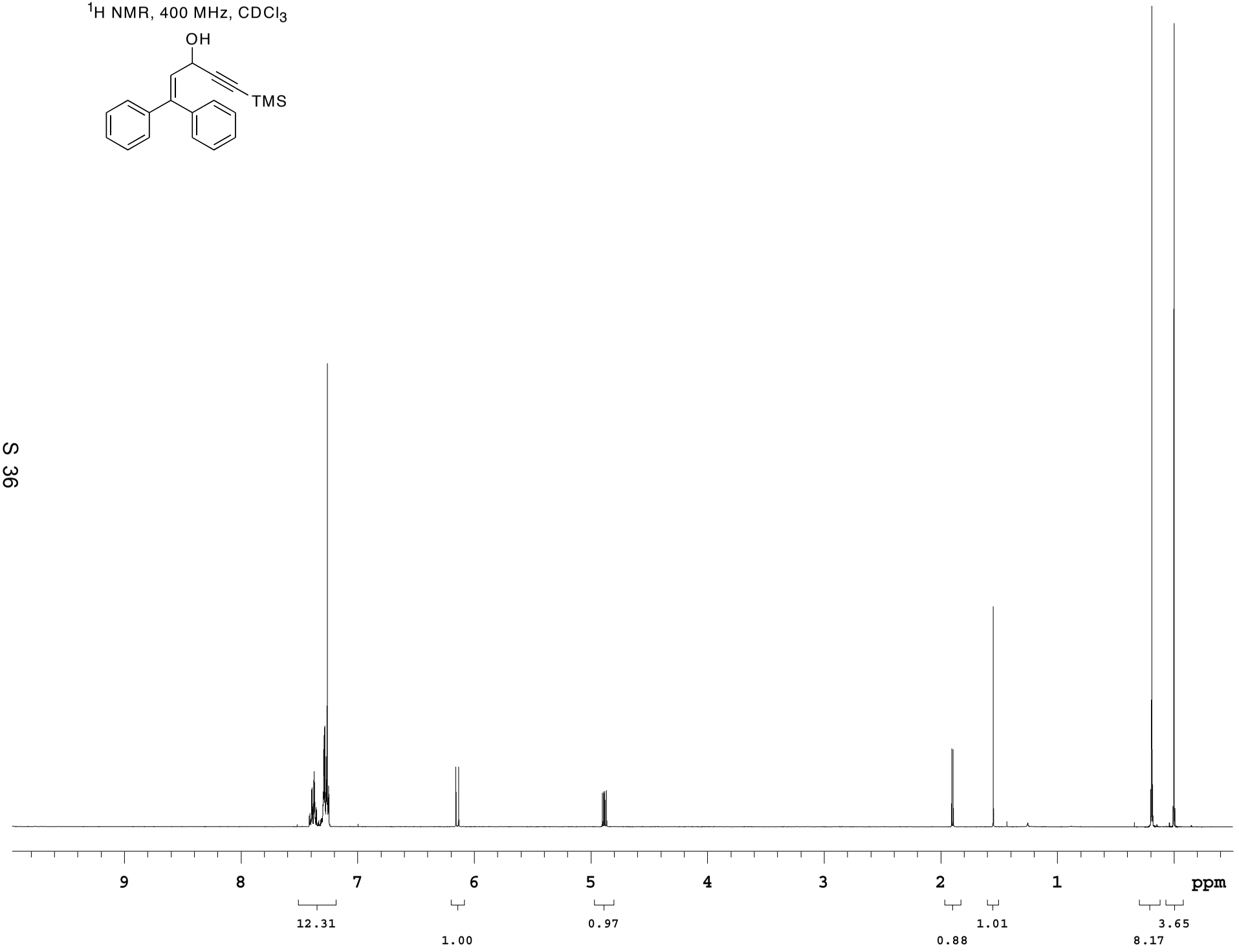


^{1}H NMR, 400 MHz, $CDCl_3$



^{13}C NMR. 101 MHz, $CDCl_3$
OH
TMS
Ph
166.741
136.036
132.093
128.597
128.119
127.800
126.799
104.103
91.393
77.318
77.000
76.682
63.388
-0.175
ppm
200
180
160
140
120
100
80
60
40
20
0

^{1}H NMR, 400 MHz, $CDCl_3$

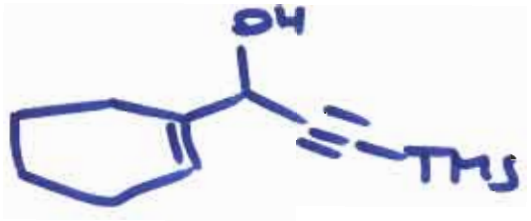


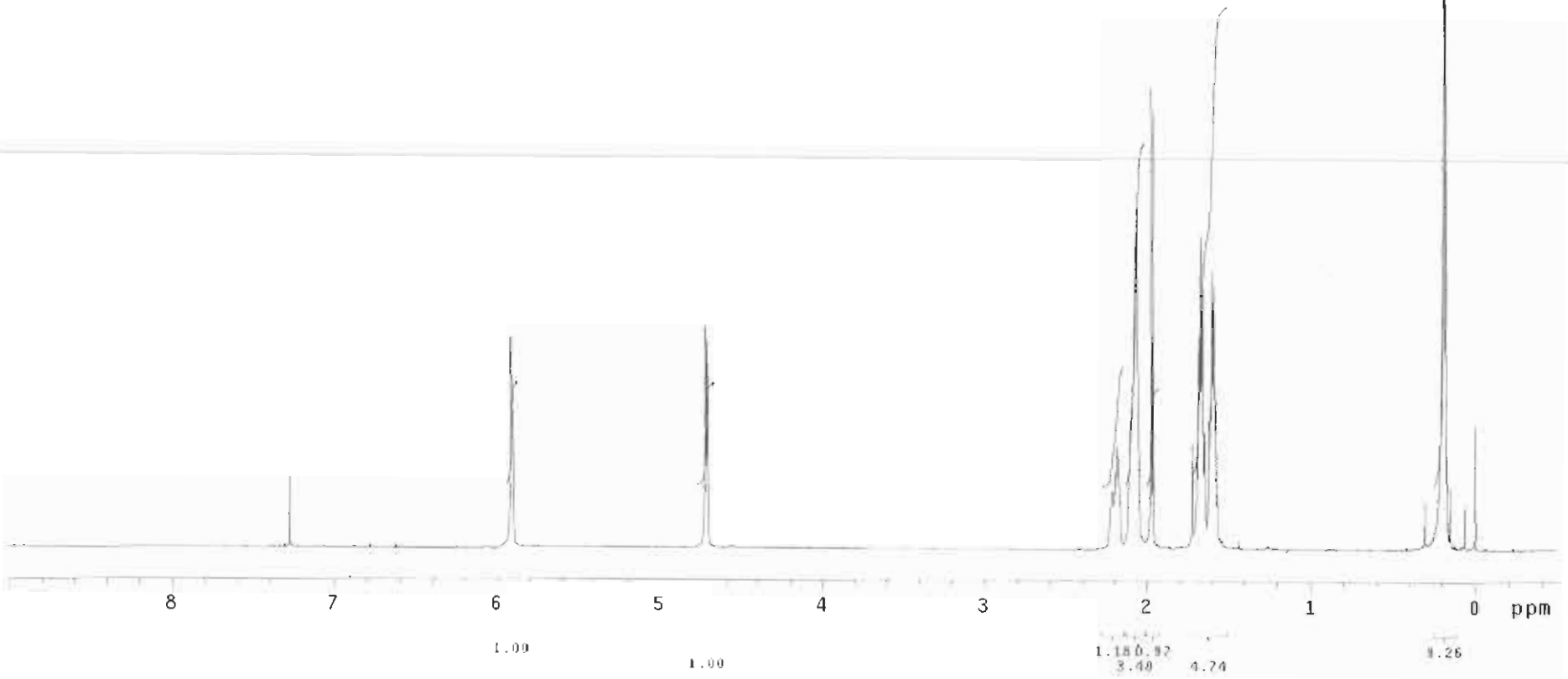
^{13}C NMR. 101 MHz, $CDCl_3$
OH
TMS
Ph
Ph
-0.167
60.575
76.682
77.000
77.318
90.506
105.316
127.072
127.838
127.944
128.180
128.202
128.240
129.916
138.417
141.276
144.476
ppm
0
20
40
60
80
100
120
140
160
180
200

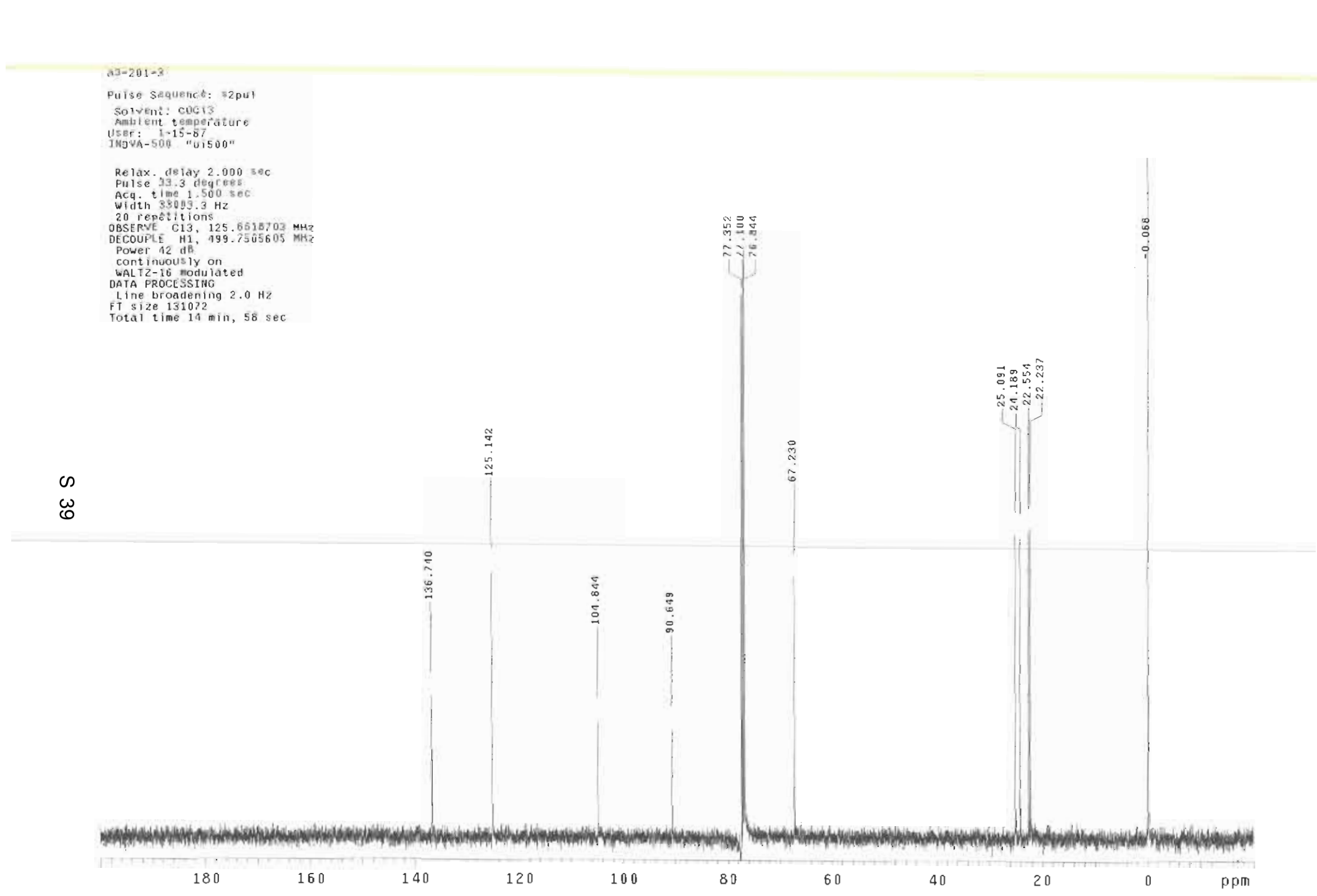
a3-201-3

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
INOVA-500 "u1500"

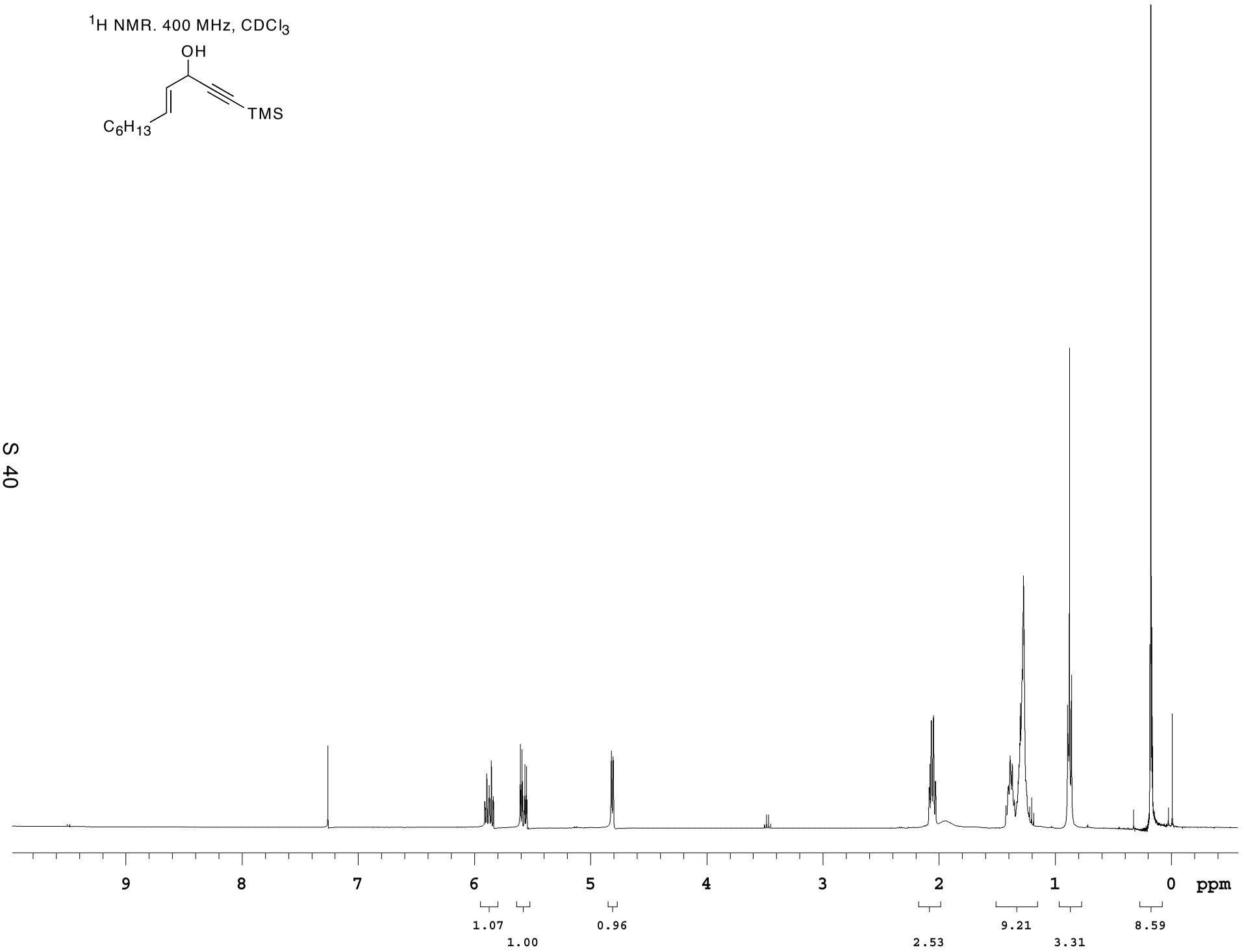
Relax. delay 1.000 sec
Pulse 60.0 degrees
Acq. time 4.000 sec
Width 8000.0 Hz
28 repetitions
OBSERVE H1, 499.7485672 MHz
DATA PROCESSING
FT size 131072
Total time 5 min, 20 sec



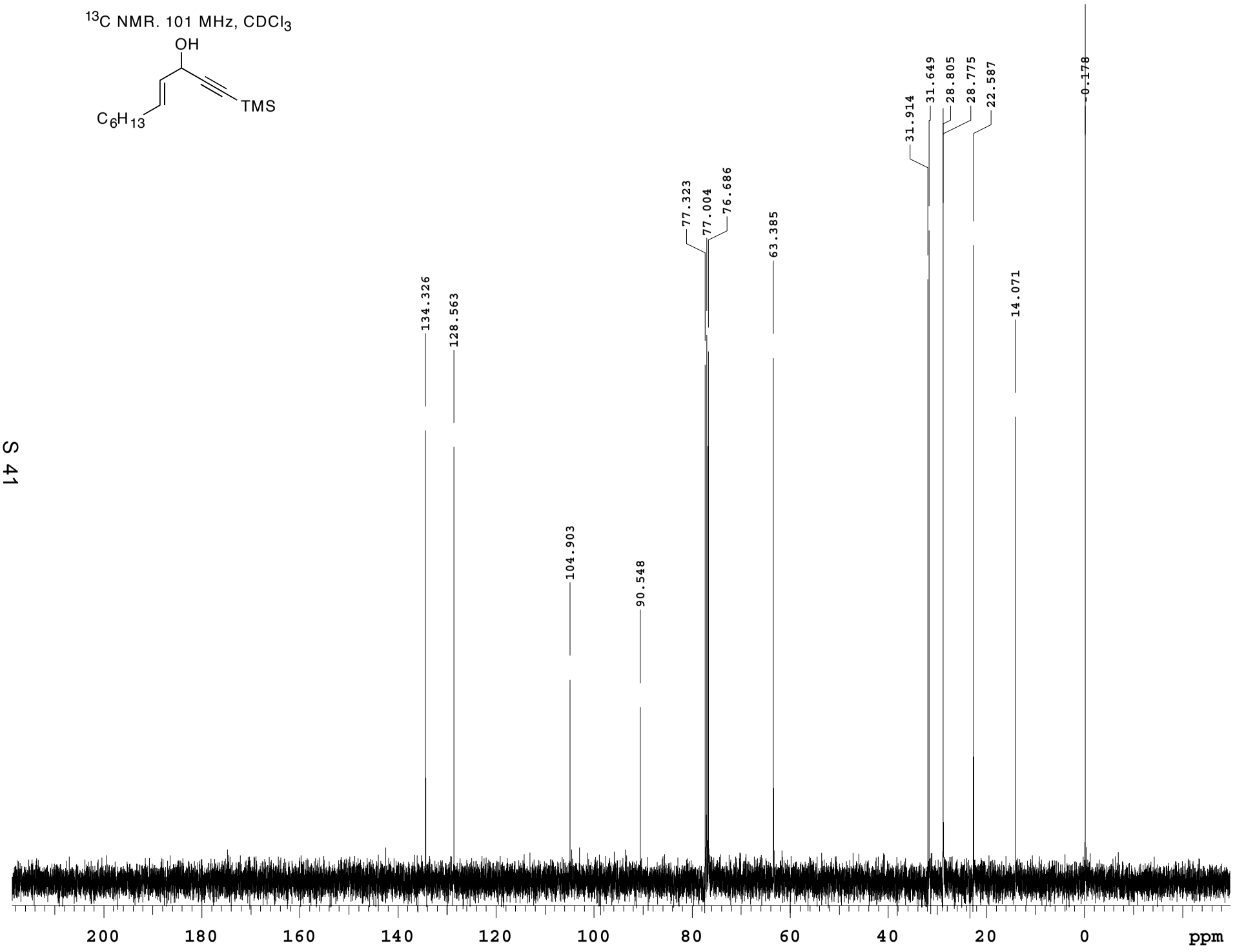



a3-201-3
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-15-87
INOVA-500 "oi500"
Relax. delay 2.000 sec
Pulse 33.3 degrees
Acq. time 1.500 sec
Width 33093.3 Hz
20 repetitions
OBSERVE C13, 125.6618703 MHz
DECOUPLE H1, 499.7505605 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 131072
Total time 14 min, 58 sec
136.740
125.142
104.844
90.649
77.352
77.100
76.844
67.230
25.091
24.189
22.554
22.237
-0.068
180
160
140
120
100
80
60
40
20
0
ppm

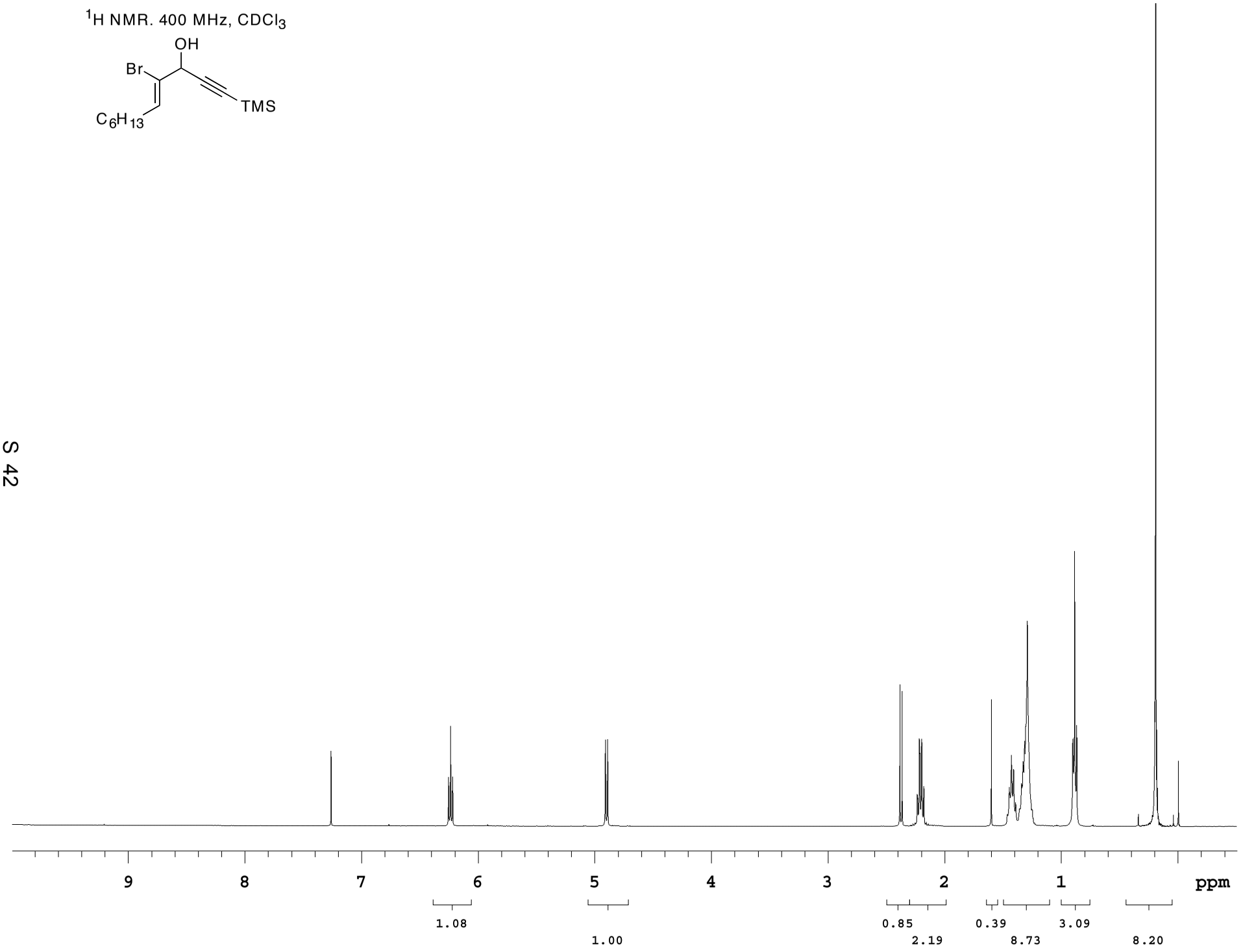
^{1}H NMR. 400 MHz, $CDCl_3$

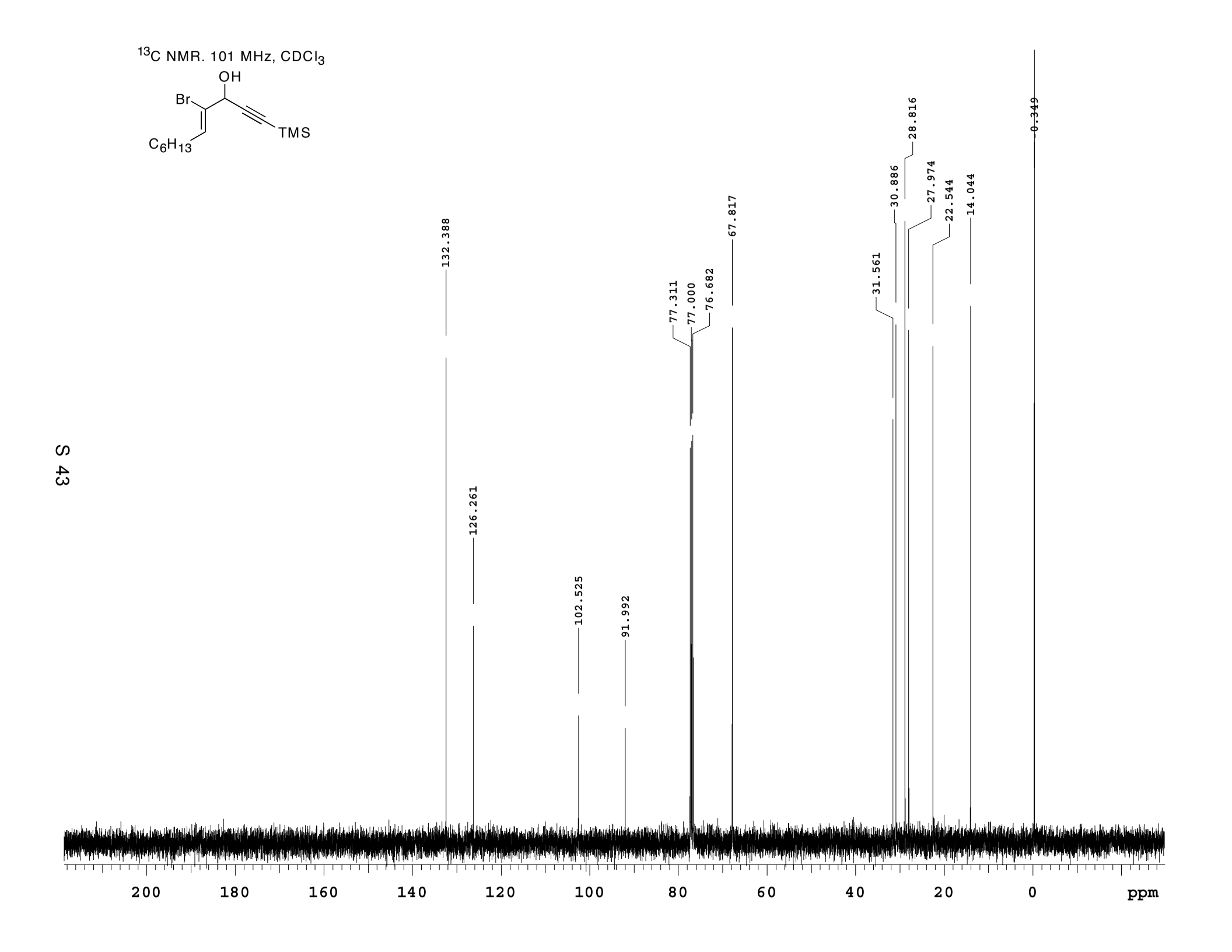


^{13}C NMR. 101 MHz, $CDCl_3$



^{1}H NMR. 400 MHz, $CDCl_3$



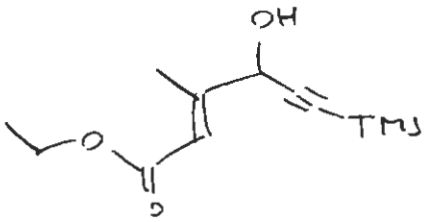

^{13}C NMR. 101 MHz, $CDCl_3$
OH
Br
TMS
C_6H_{13}
132.388
126.261
102.525
91.992
77.311
77.000
76.682
67.817
31.561
30.886
28.816
27.974
22.544
14.044
0.349
200
180
160
140
120
100
80
60
40
20
0
ppm

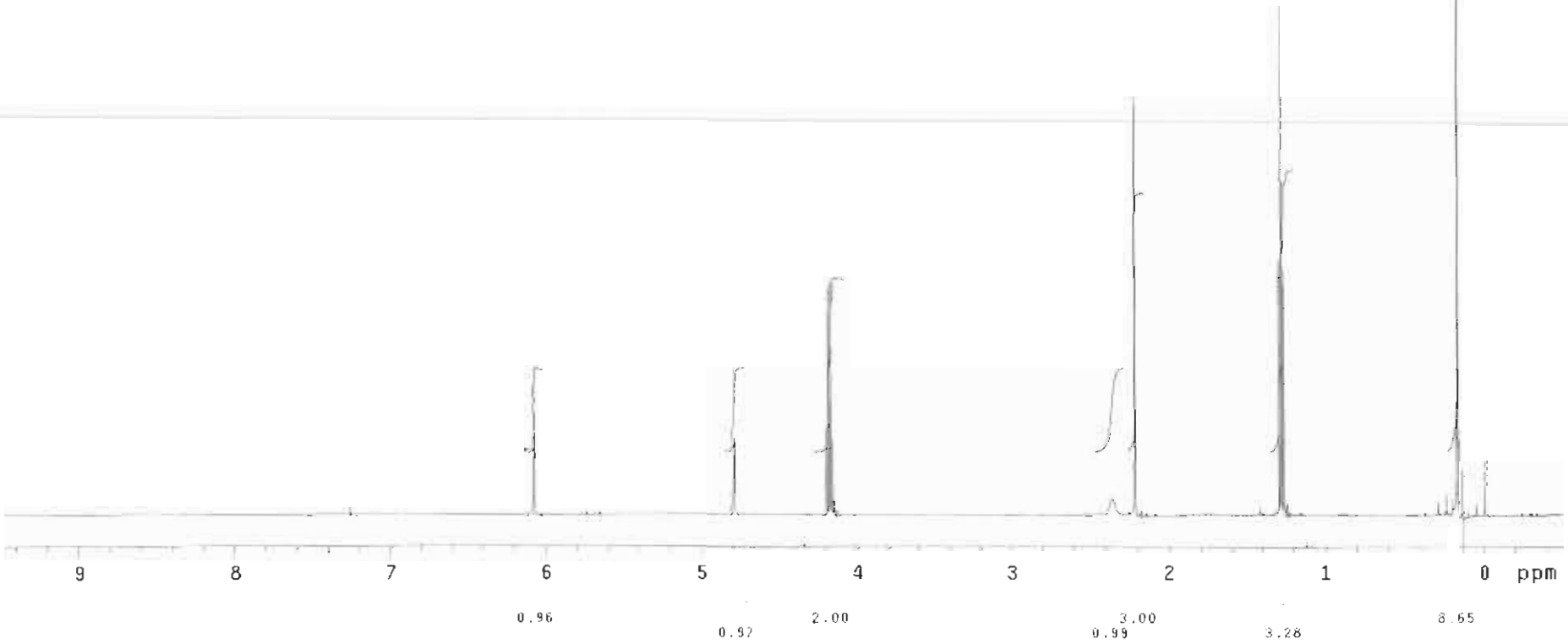
a3-196-2-1

Pulse Sequence: s2pul

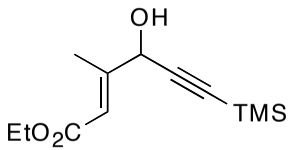
Solvent: CDCl3
Ambient temperature
VNMRS-500 "ui500"

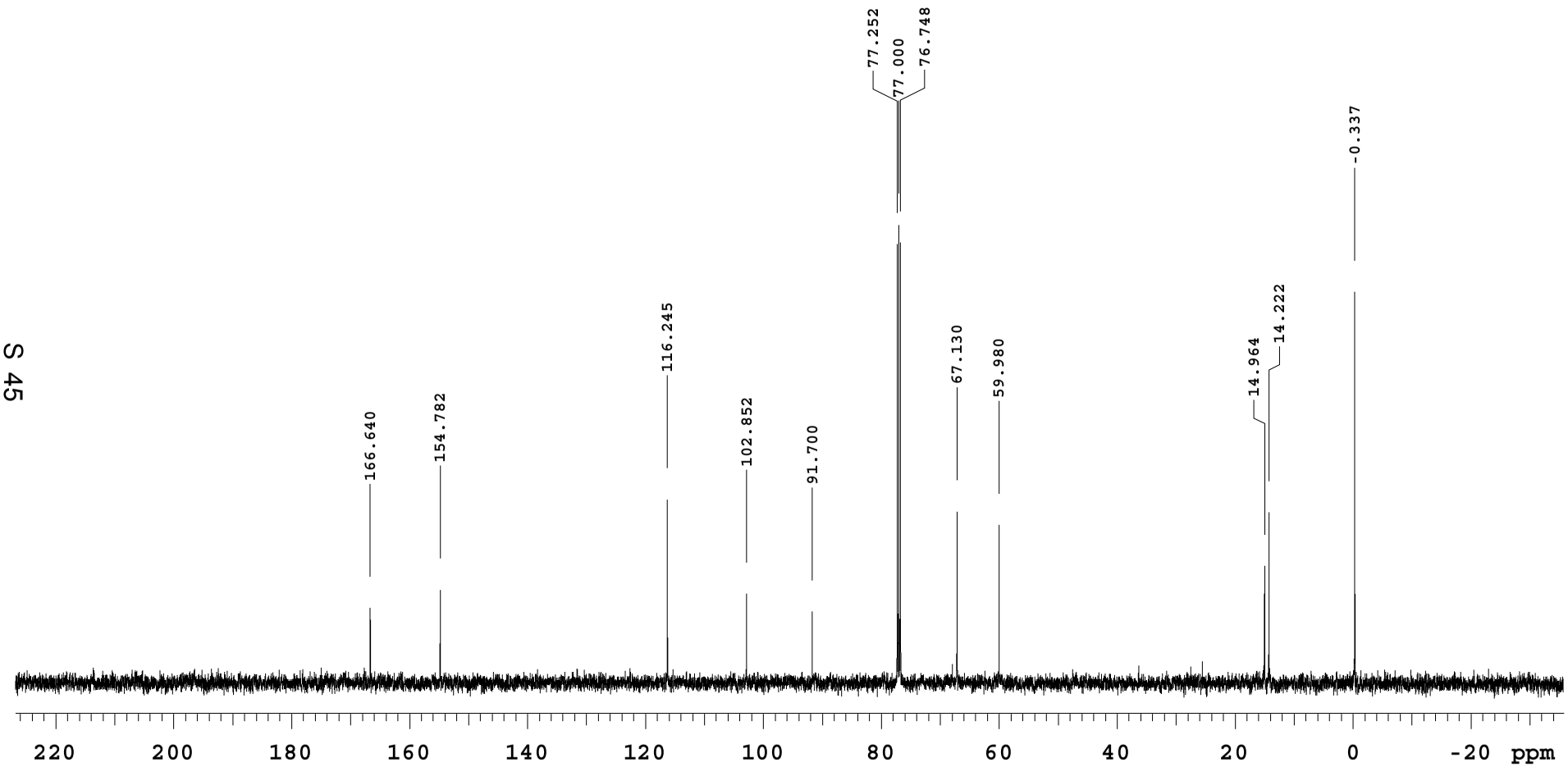
Relax. delay 1.000 sec
Pulse 60.0 degrees
Acq. time 4.000 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7485737 MHz
DATA PROCESSING
FT size 131072
Total time 2 min, 40 sec



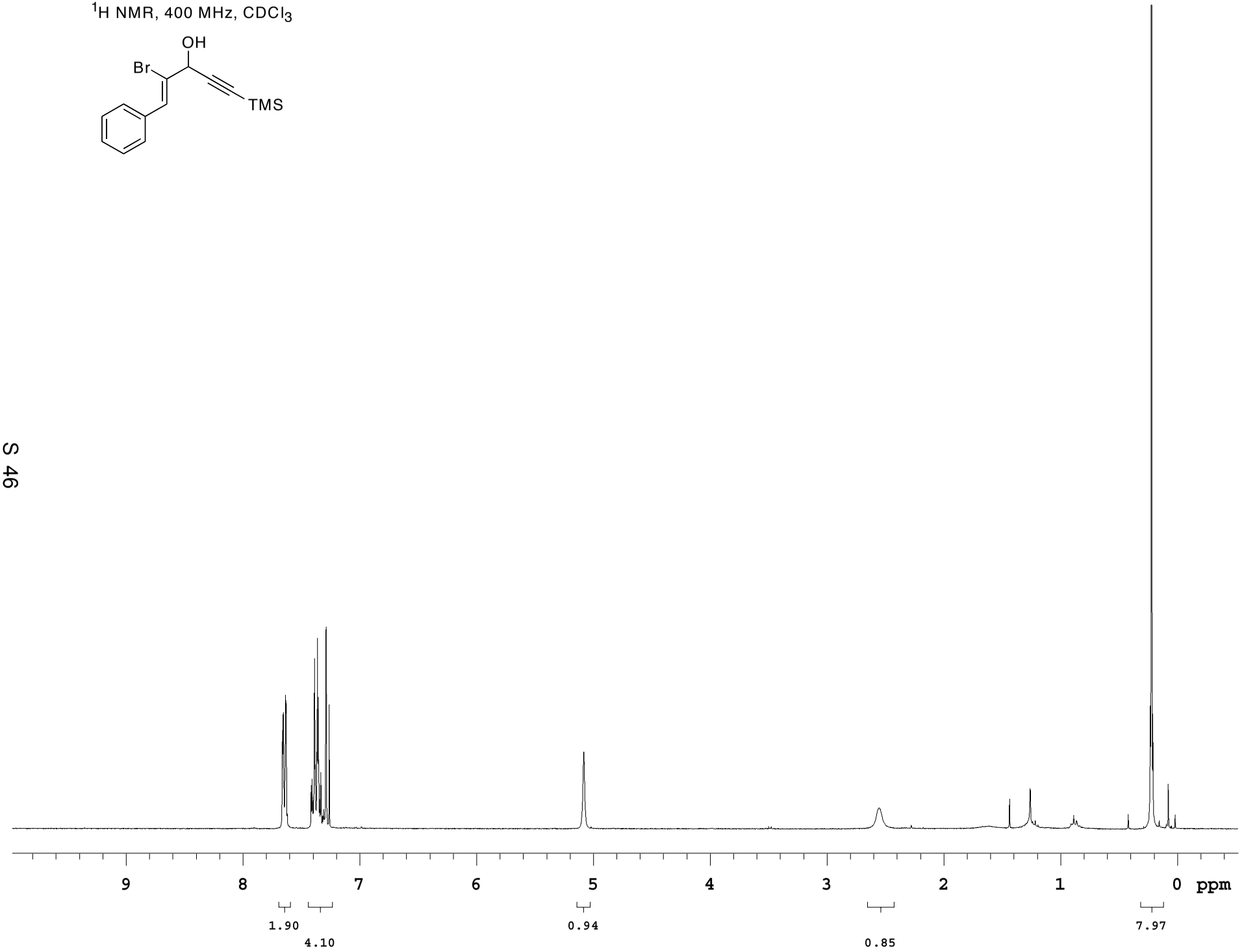


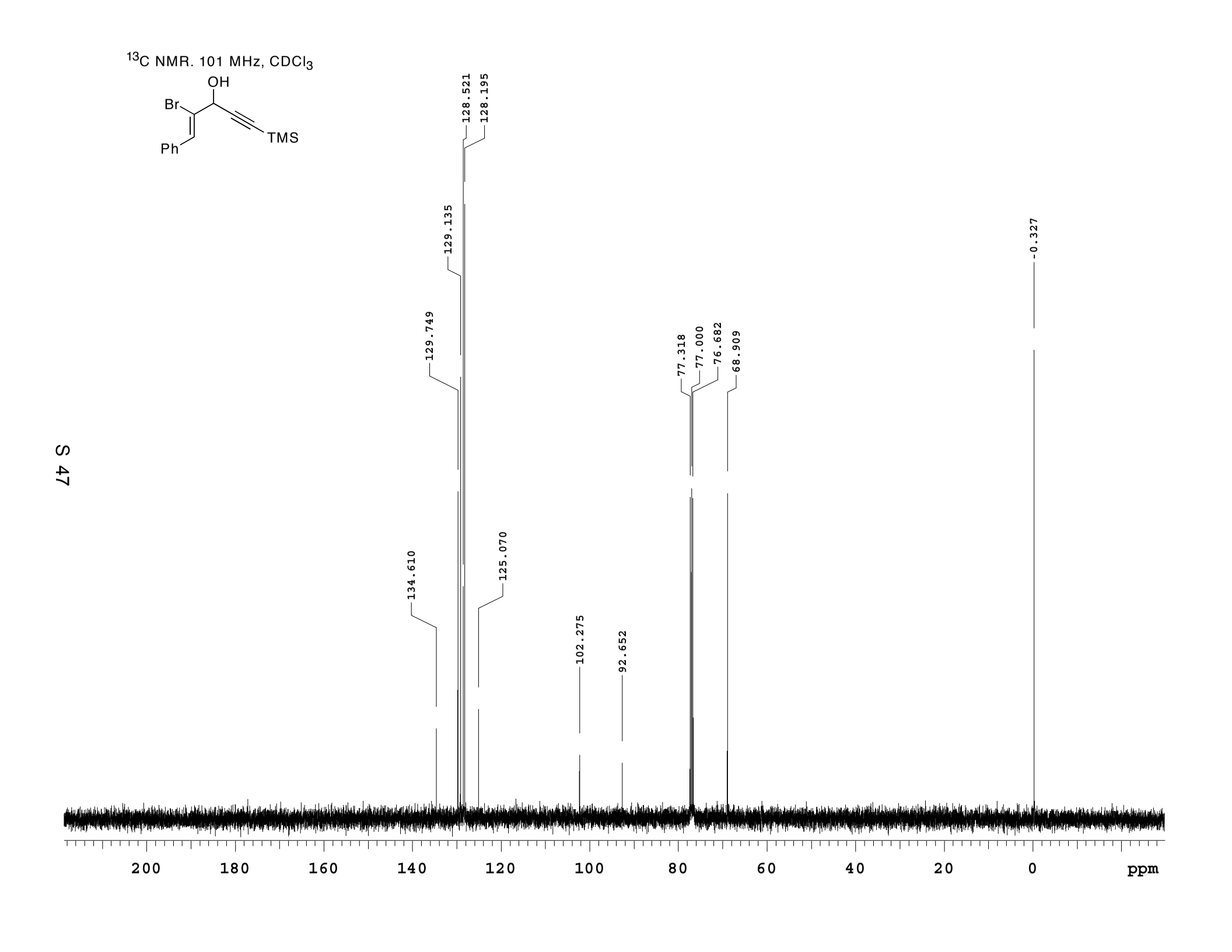
^{13}C NMR. 101 MHz, $CDCl_3$





^{1}H NMR, 400 MHz, $CDCl_3$




^{13}C NMR. 101 MHz, $CDCl_3$
OH
Br
Ph
TMS
128.521
128.195
129.135
129.749
77.318
77.000
76.682
68.909
-0.327
134.610
125.070
102.275
92.652
200
180
160
140
120
100
80
60
40
20
0
ppm

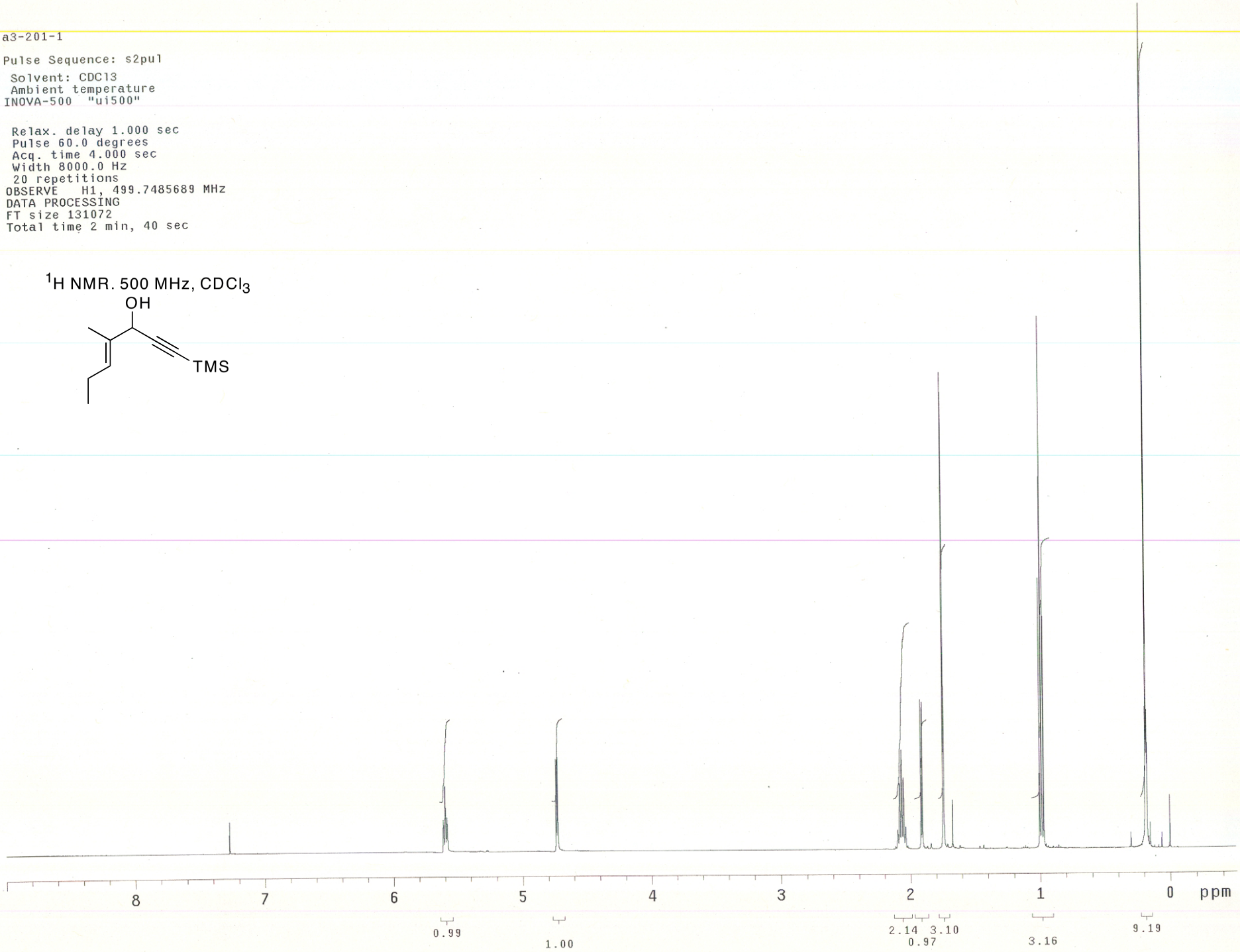
a3-201-1

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
INOVA-500 "ui500"

Relax. delay 1.000 sec
Pulse 60.0 degrees
Acq. time 4.000 sec
Width 8000.0 Hz
20 repetitions
OBSERVE H1, 499.7485689 MHz
DATA PROCESSING
FT size 131072
Total time 2 min, 40 sec

^{1}H NMR. 500 MHz, $CDCl_3$

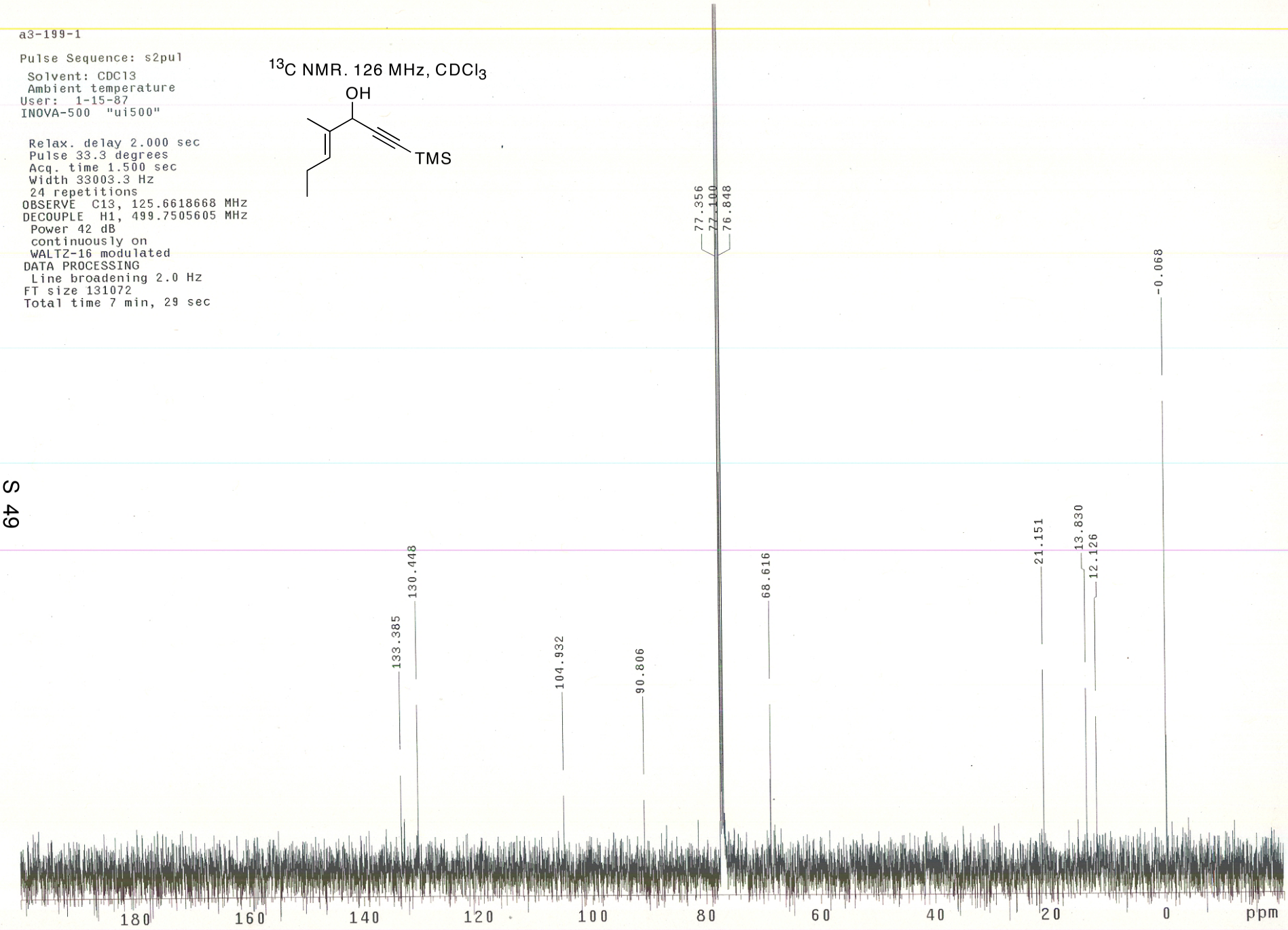


a3-199-1

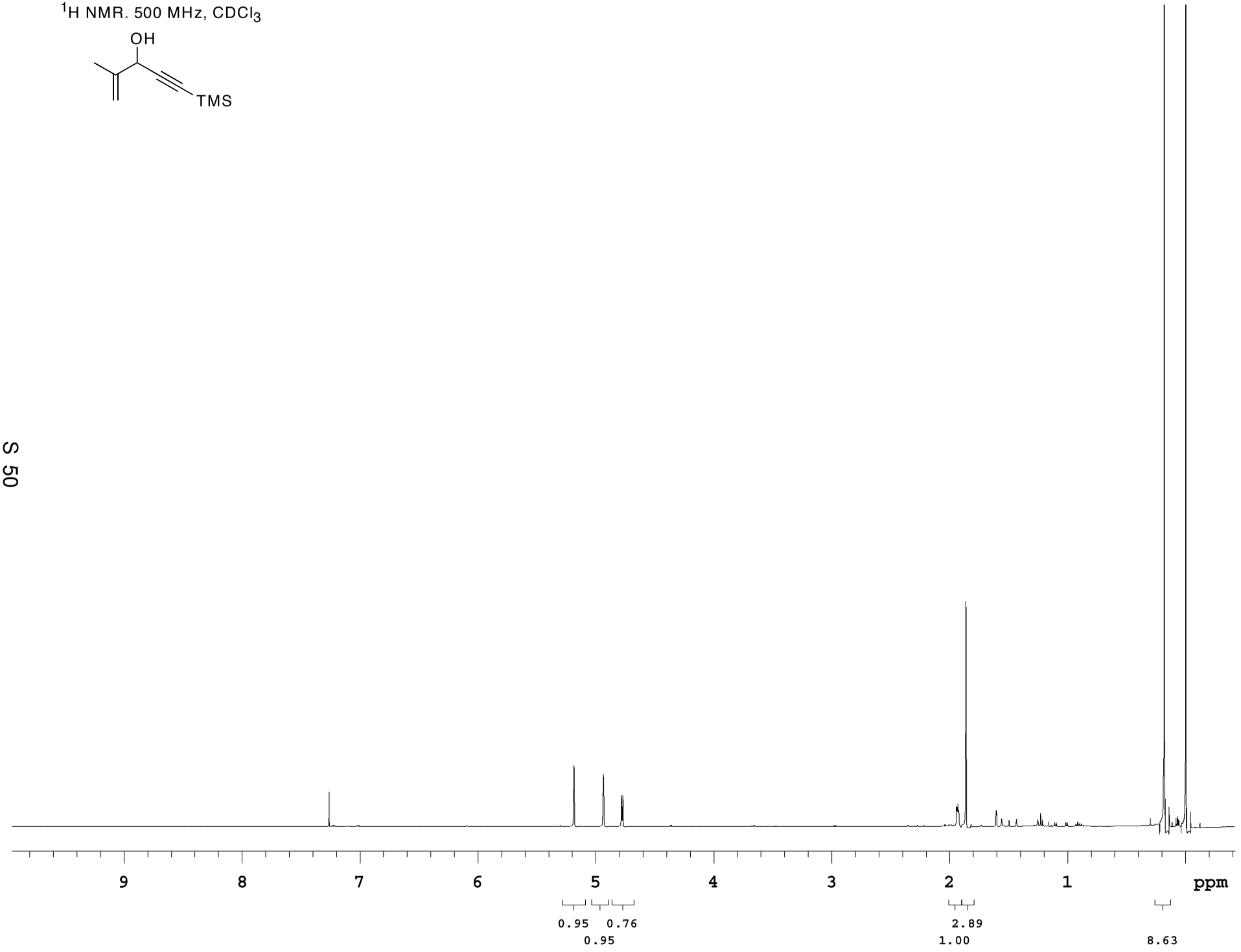
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-15-87
INOVA-500 "ui500"

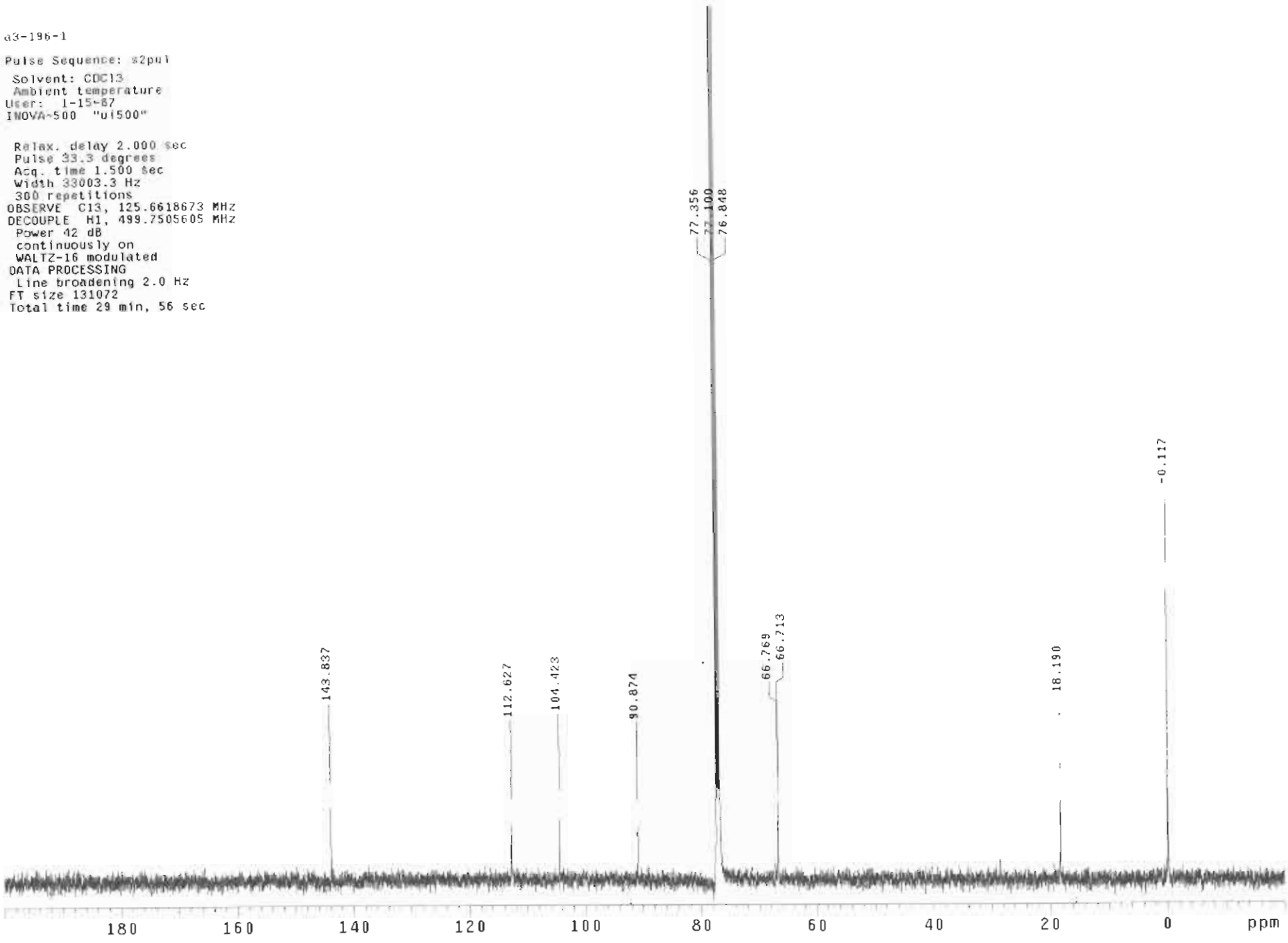
Relax. delay 2.000 sec
Pulse 33.3 degrees
Acq. time 1.500 sec
Width 33003.3 Hz
24 repetitions
OBSERVE C13, 125.6618668 MHz
DECOUPLE H1, 499.7505605 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 131072
Total time 7 min, 29 sec

^{13}C NMR. 126 MHz, $CDCl_3$

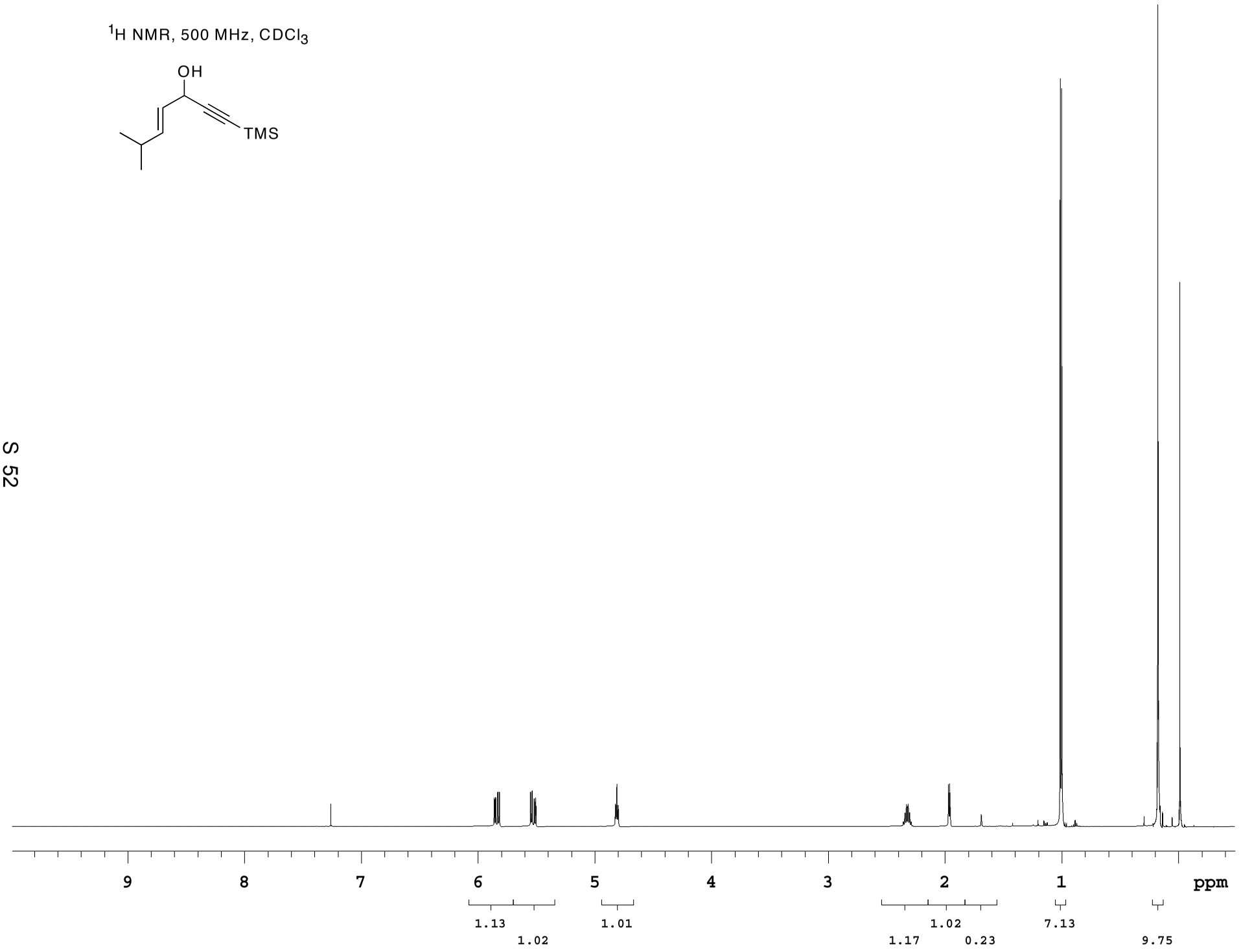


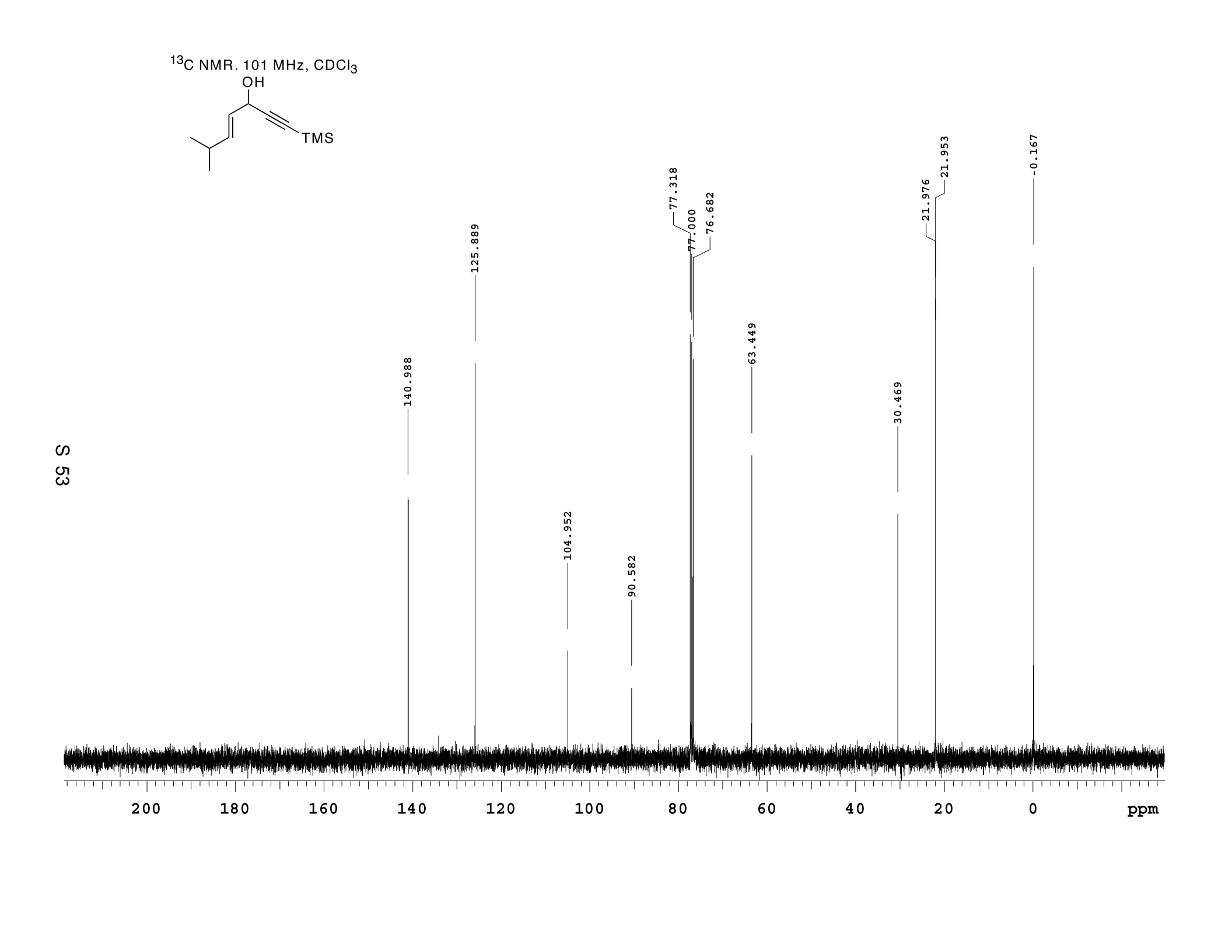
^{1}H NMR. 500 MHz, $CDCl_3$



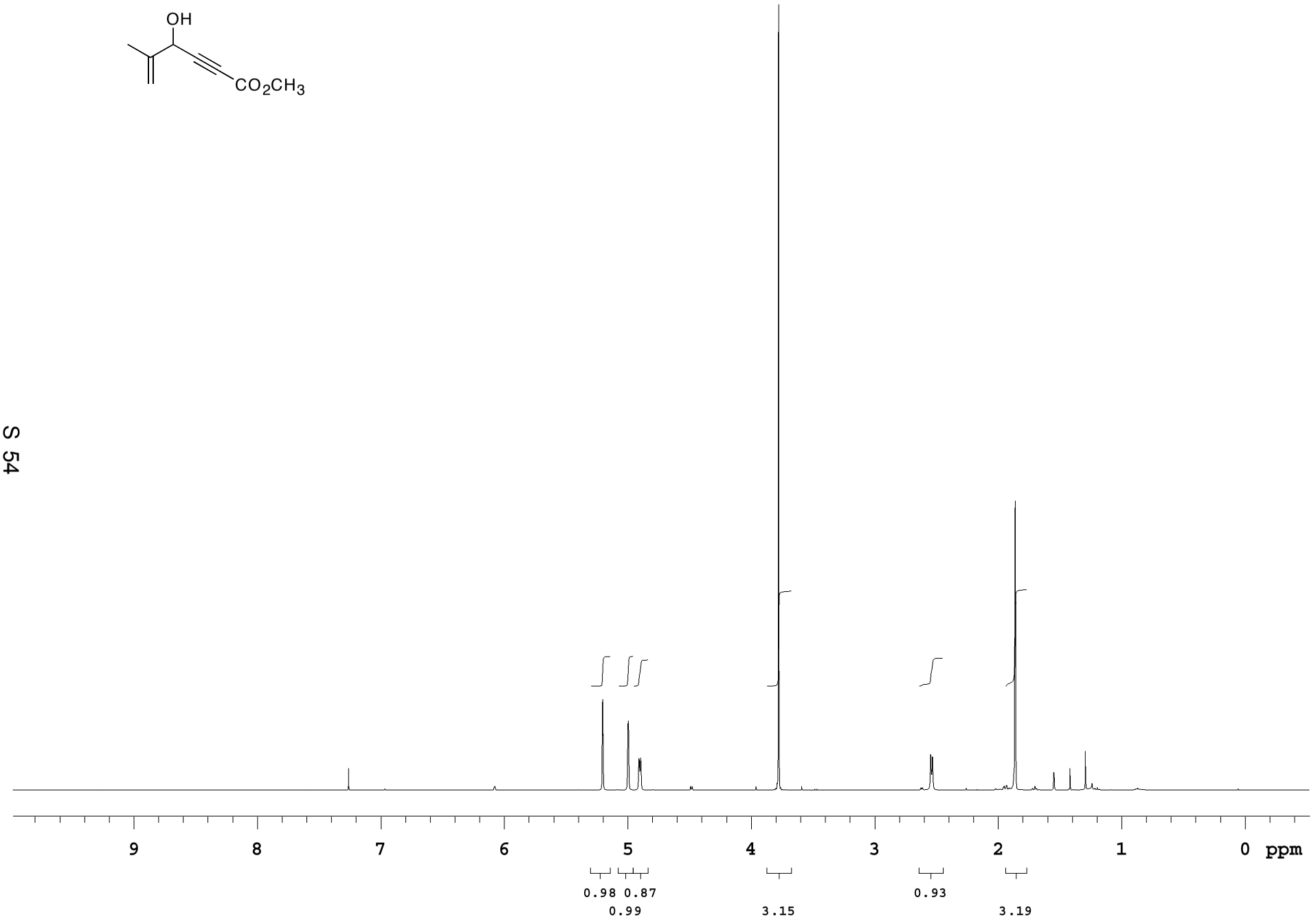

a3-196-1
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-15-87
INOVA-500 "ui500"
Relax. delay 2.000 sec
Pulse 33.3 degrees
Acq. time 1.500 sec
Width 33003.3 Hz
300 repetitions
OBSERVE C13, 125.6618673 MHz
DECOUPLE H1, 499.7505605 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 131072
Total time 29 min, 56 sec
77.356
77.100
76.848
143.837
112.627
104.423
90.874
66.769
66.713
18.190
-0.117
180
160
140
120
100
80
60
40
20
0
ppm

^{1}H NMR, 500 MHz, $CDCl_3$

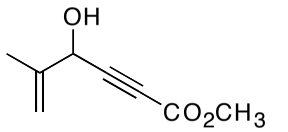


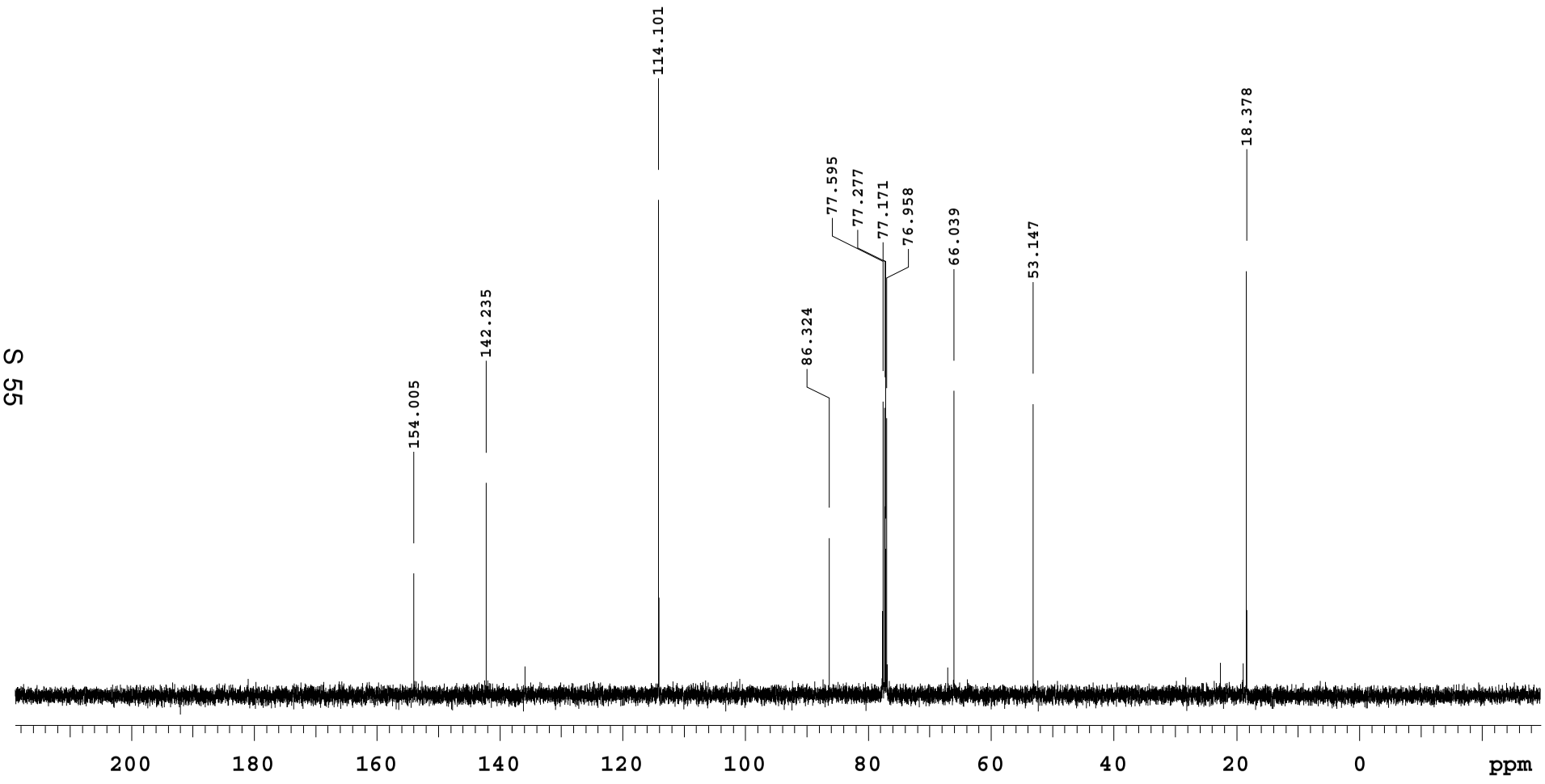

^{13}C NMR. 101 MHz, $CDCl_3$
OH
TMS
140.988
125.889
104.952
90.582
77.318
77.000
76.682
63.449
30.469
21.976
21.953
-0.167
200
180
160
140
120
100
80
60
40
20
0
ppm

^{1}H NMR, 400 MHz, $CDCl_3$

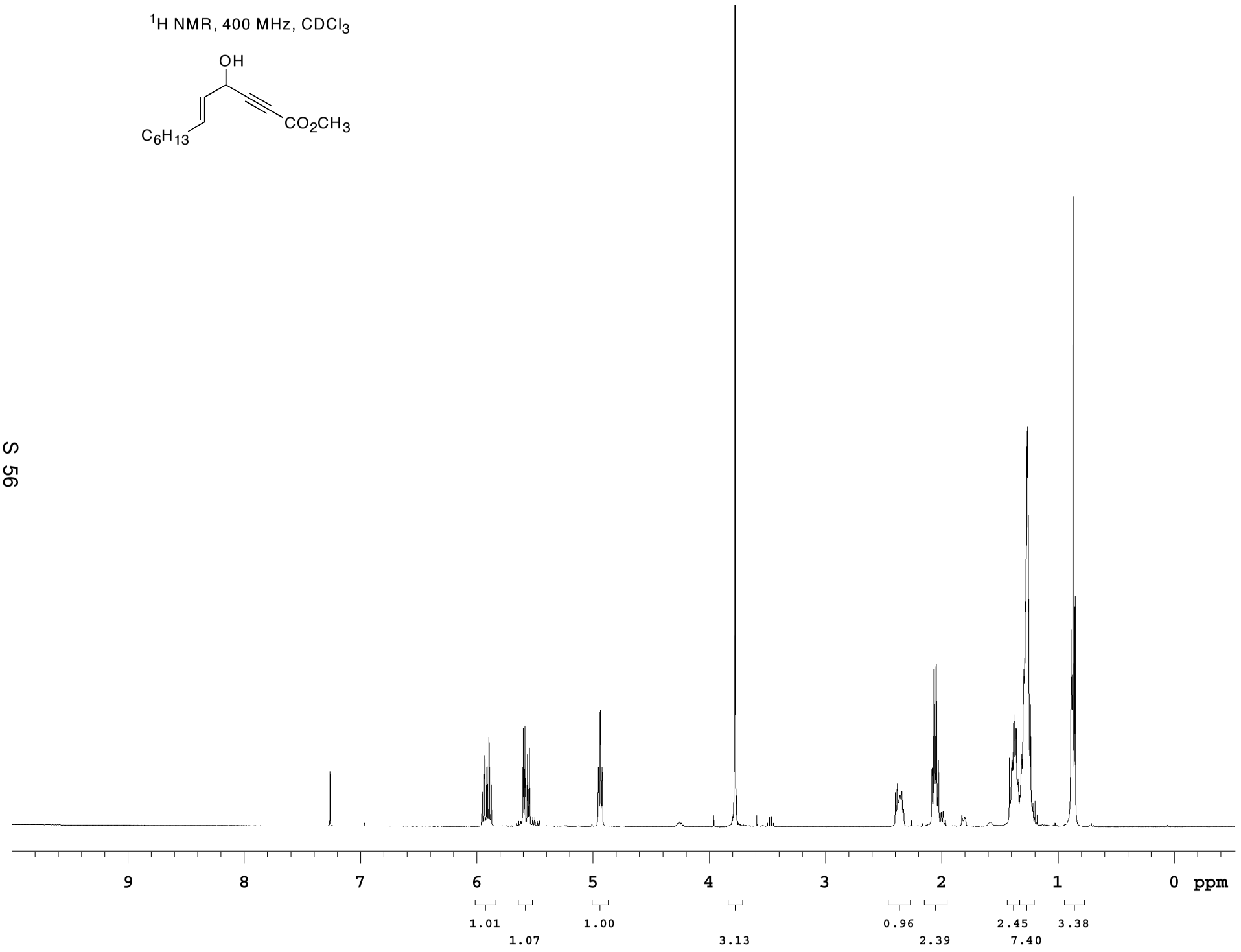


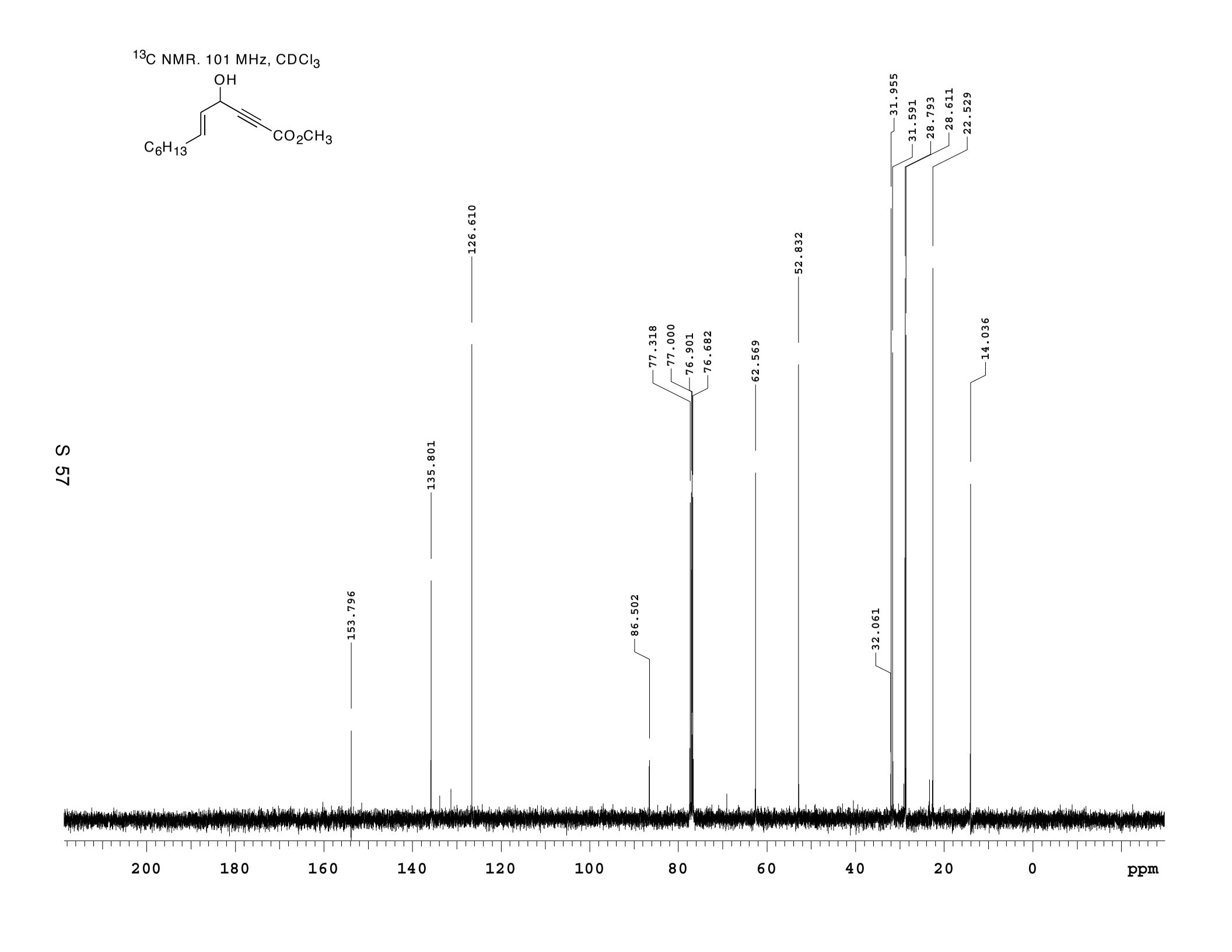
^{13}C NMR. 101 MHz, $CDCl_3$



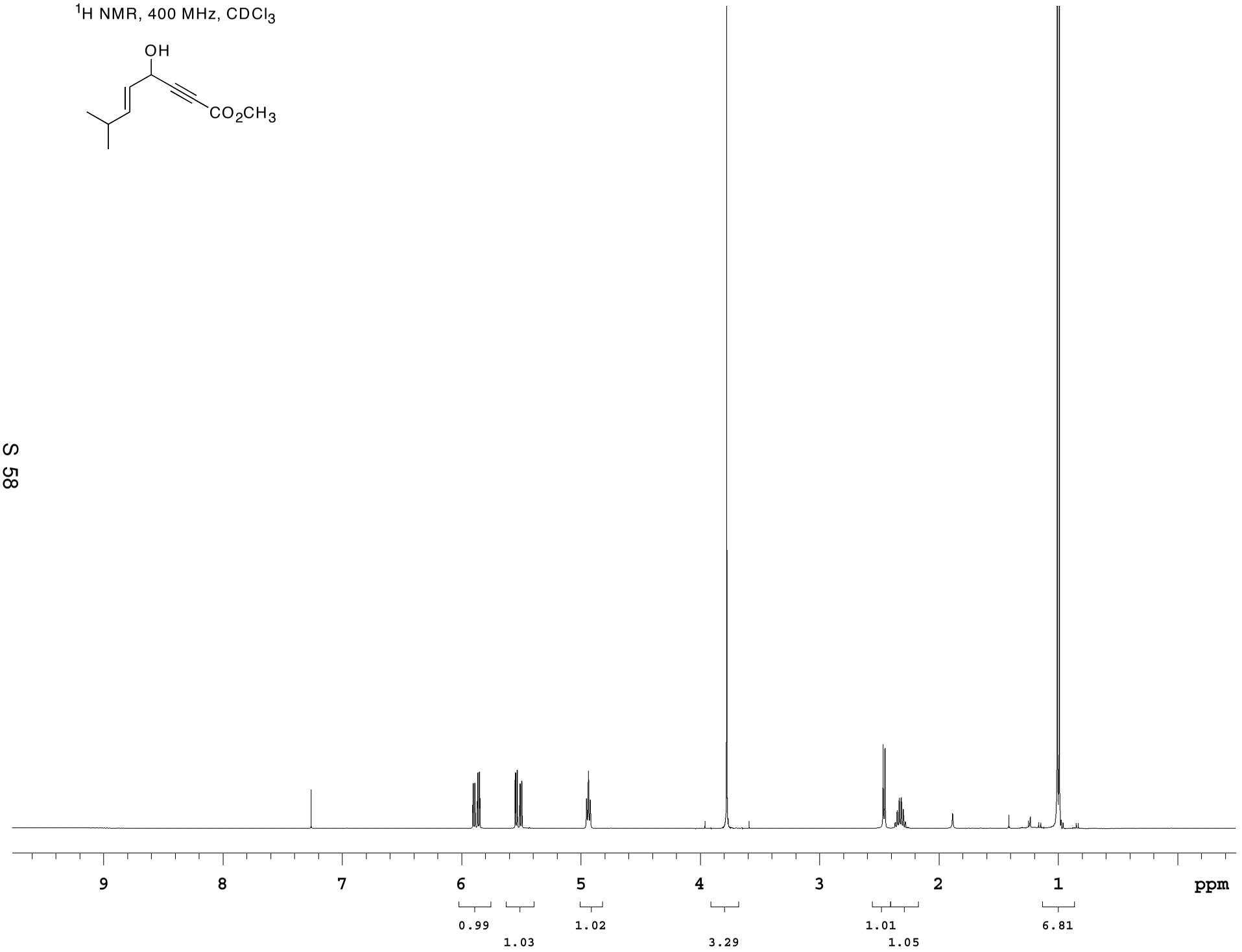


^{1}H NMR, 400 MHz, $CDCl_3$



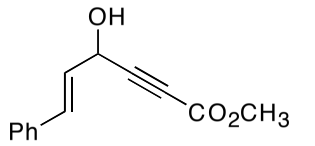

^{13}C NMR. 101 MHz, $CDCl_3$
OH
C_6H_{13}
CO_2CH_3
153.796
135.801
126.610
86.502
77.318
77.000
76.901
76.682
62.569
52.832
32.061
31.955
31.591
28.793
28.611
22.529
14.036
200
180
160
140
120
100
80
60
40
20
0
ppm

^{1}H NMR, 400 MHz, $CDCl_3$

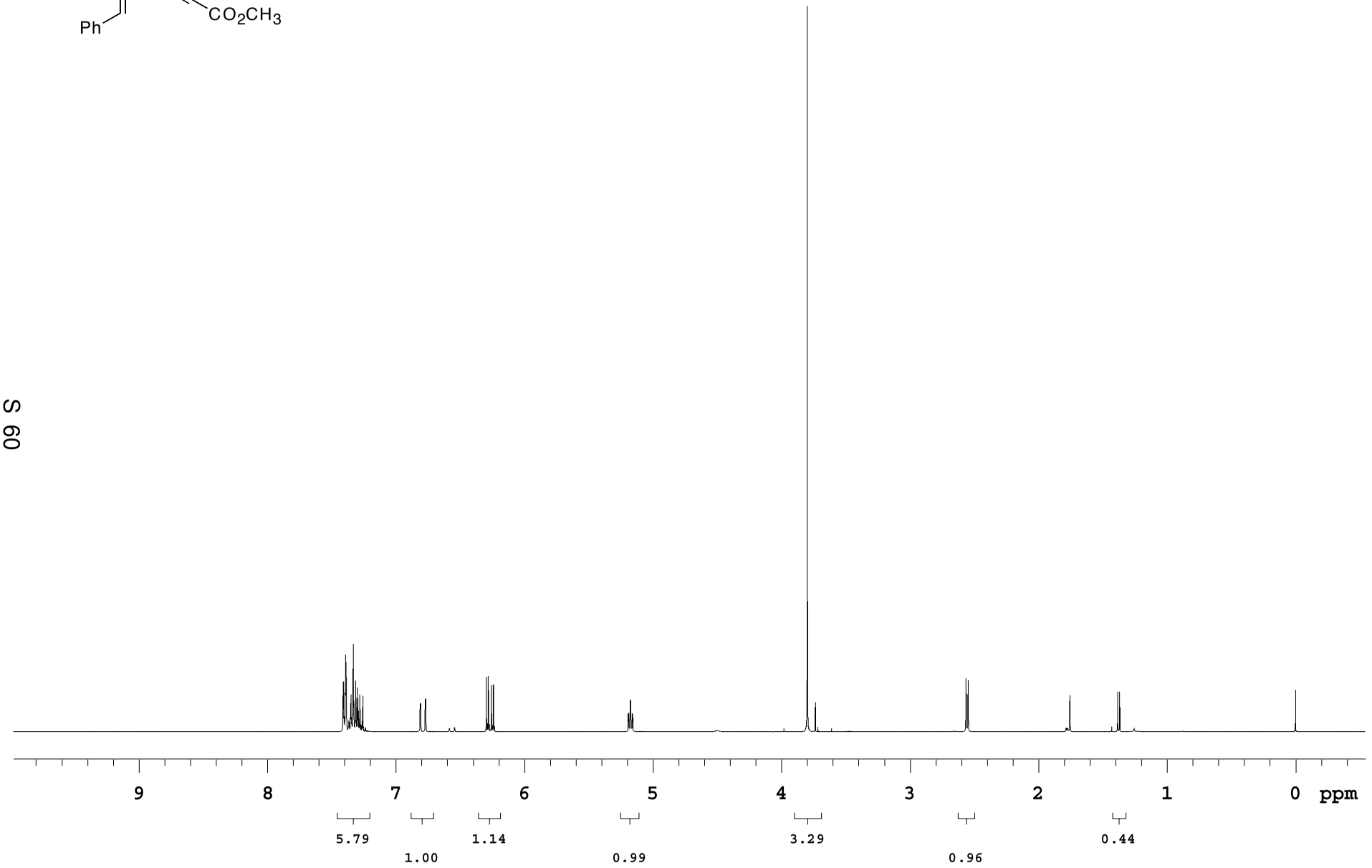


^{13}C NMR. 101 MHz, $CDCl_3$
OH
CO_2CH_3
153.841
142.254
123.978
86.608
77.318
77.000
76.886
76.682
62.592
52.847
30.545
21.786
ppm
200
180
160
140
120
100
80
60
40
20
0

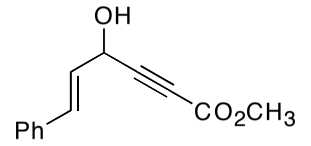
^{1}H NMR. 400 MHz, $CDCl_3$

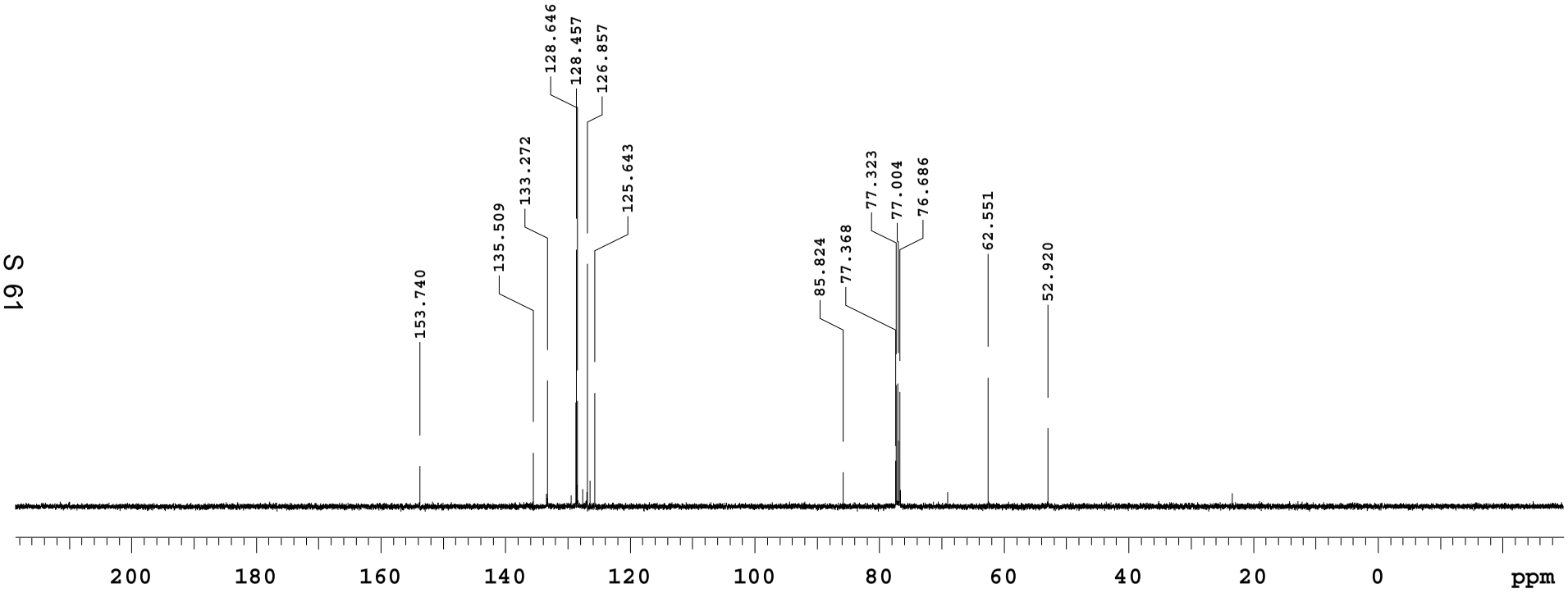


S 60

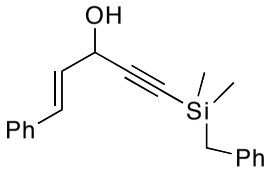


^{13}C NMR. 101 MHz, $CDCl_3$

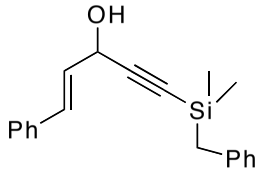


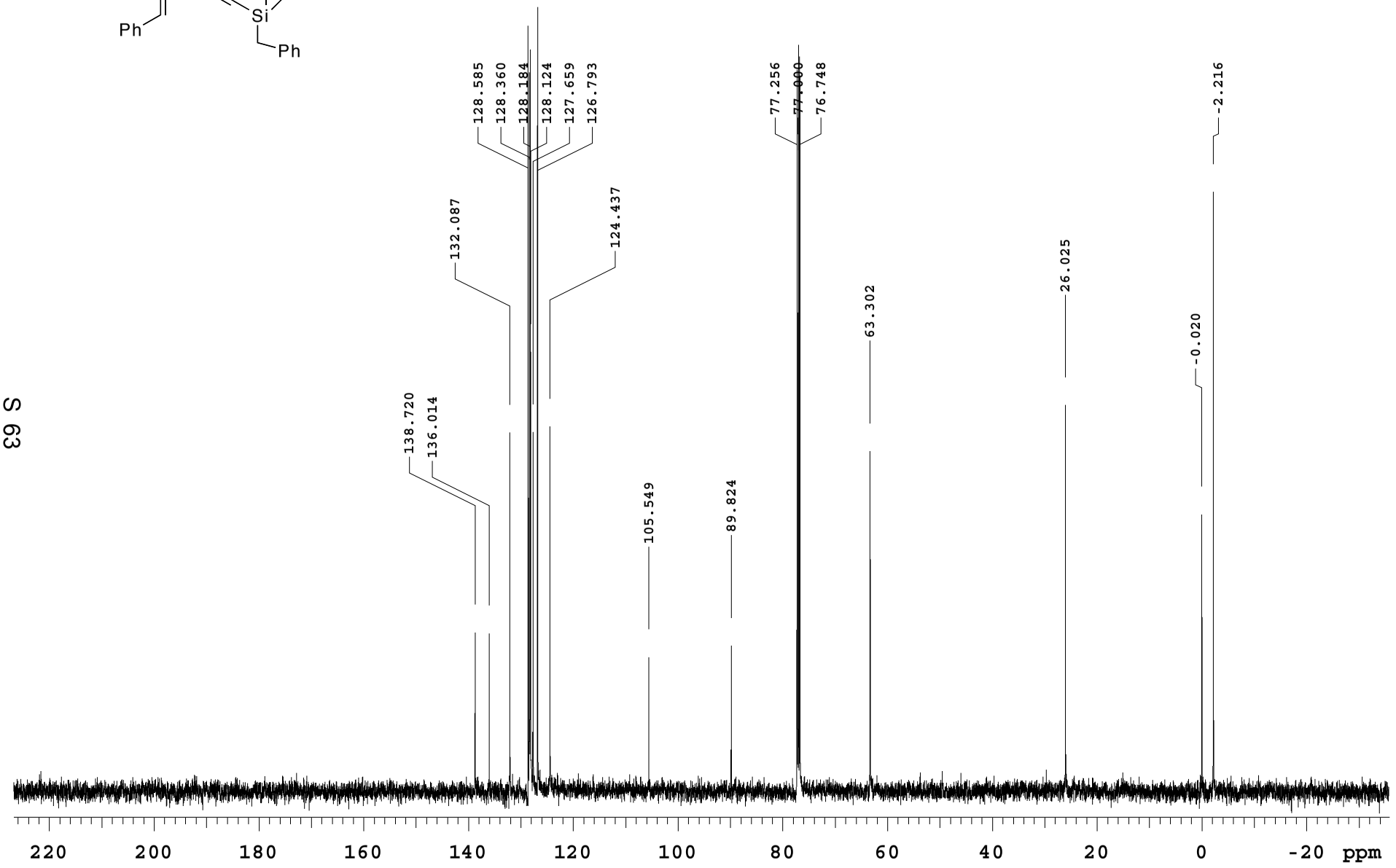


^{1}H NMR, 400 MHz, $CDCl_3$

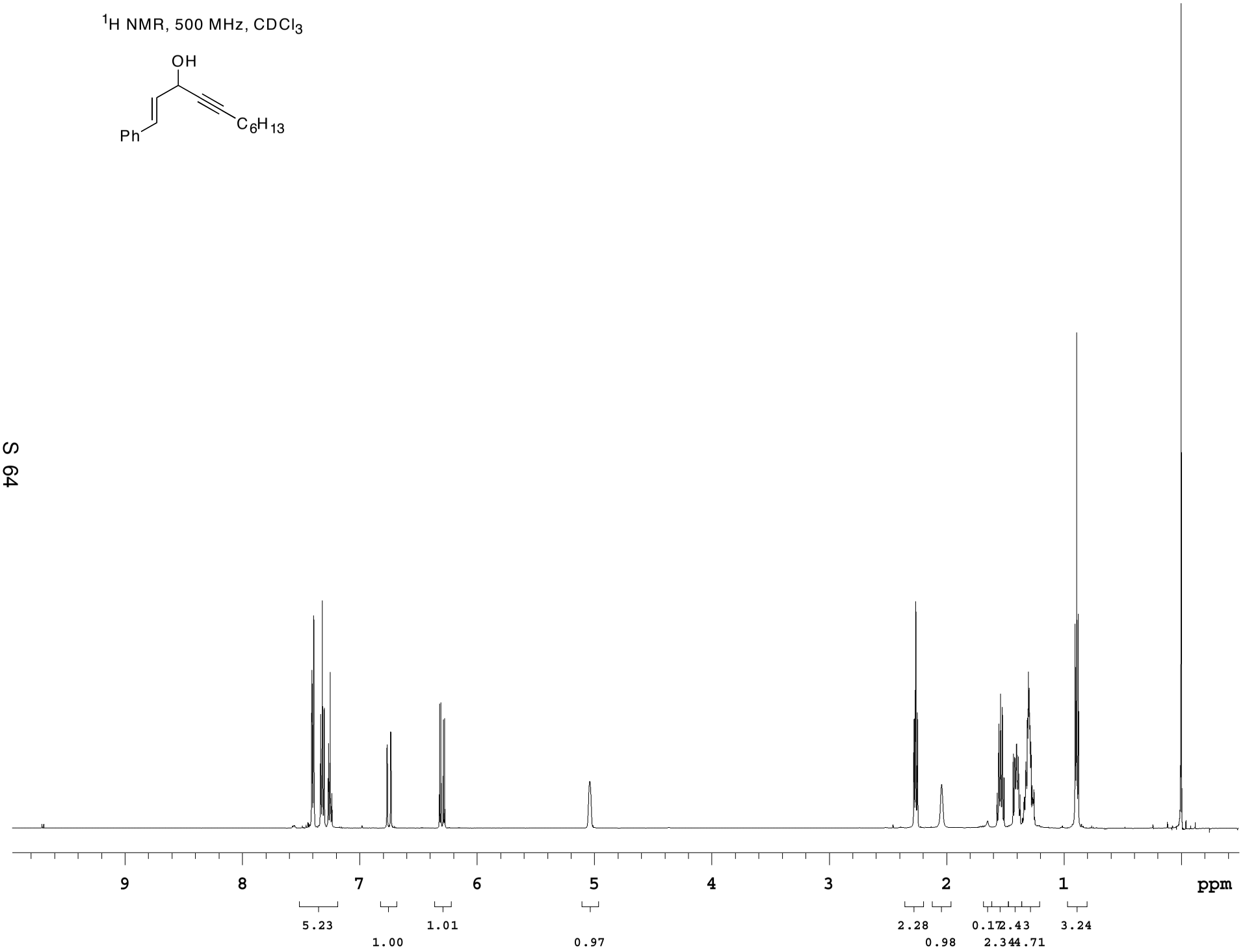


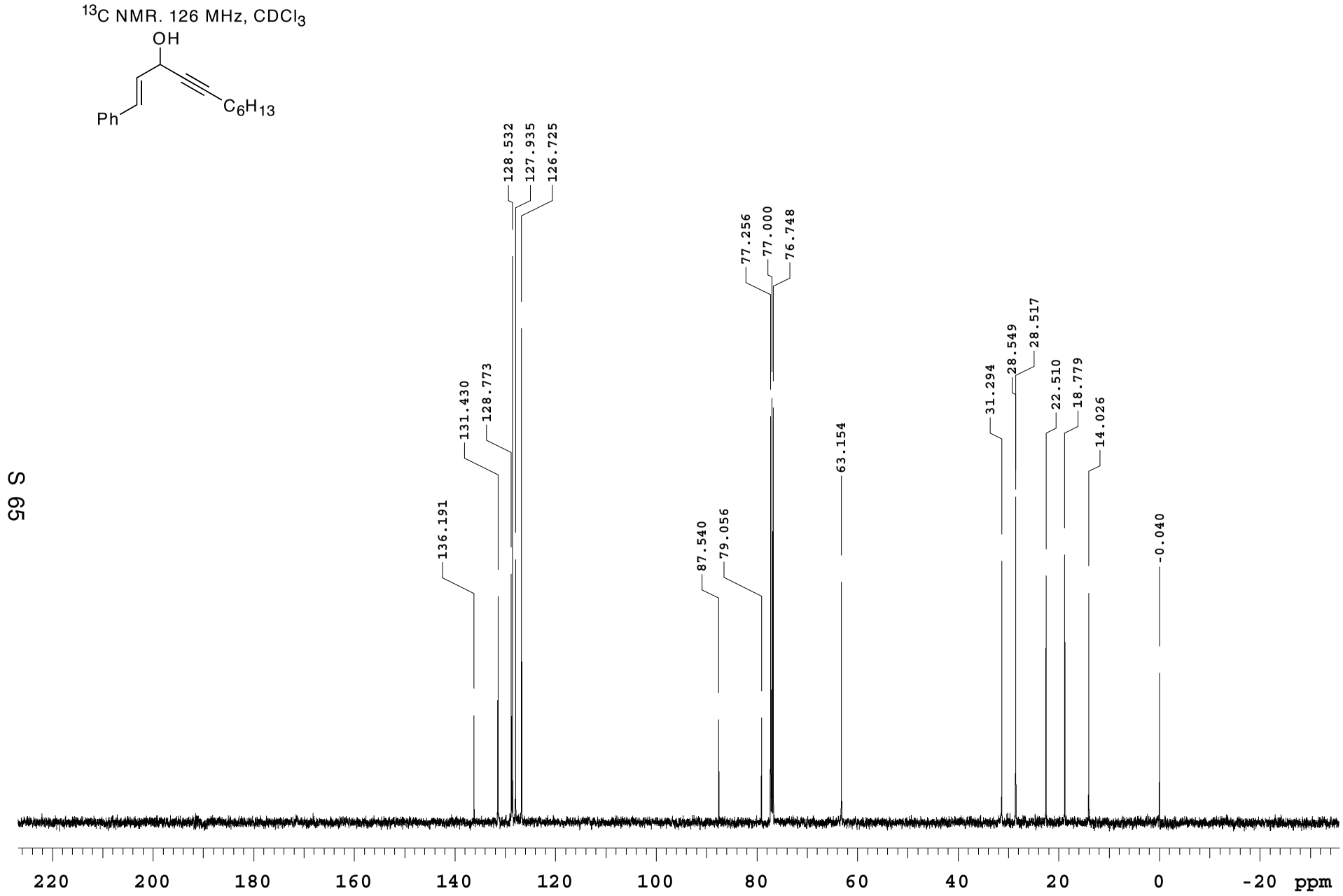
^{13}C NMR. 126 MHz, $CDCl_3$





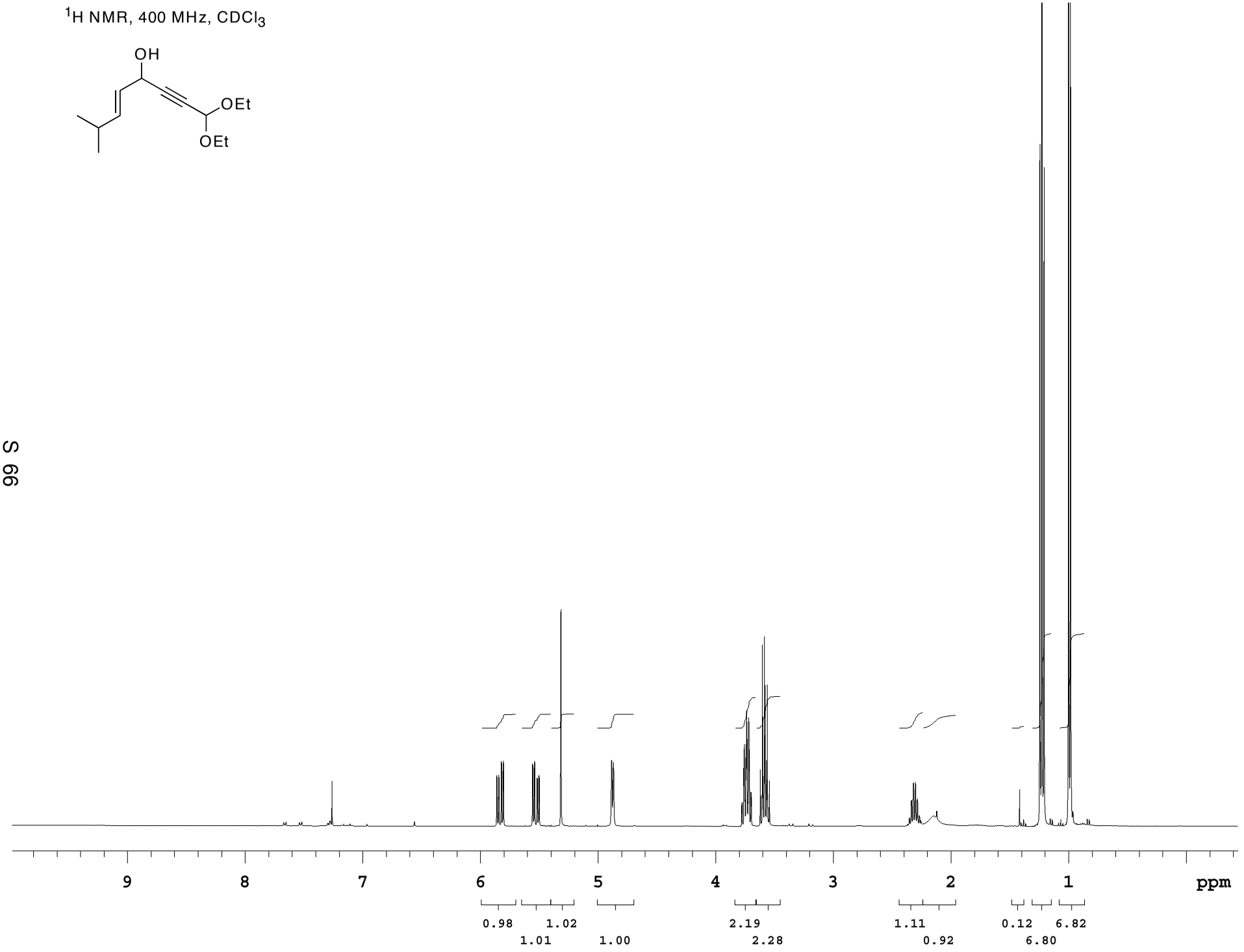
^{1}H NMR, 500 MHz, $CDCl_3$



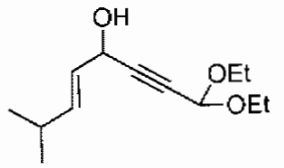

^{13}C NMR. 126 MHz, $CDCl_3$
OH
Ph
C_6H_{13}
128.532
127.935
126.725
77.256
77.000
76.748
131.430
128.773
136.191
87.540
79.056
63.154
31.294
28.549
28.517
22.510
18.779
14.026
-0.040
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm

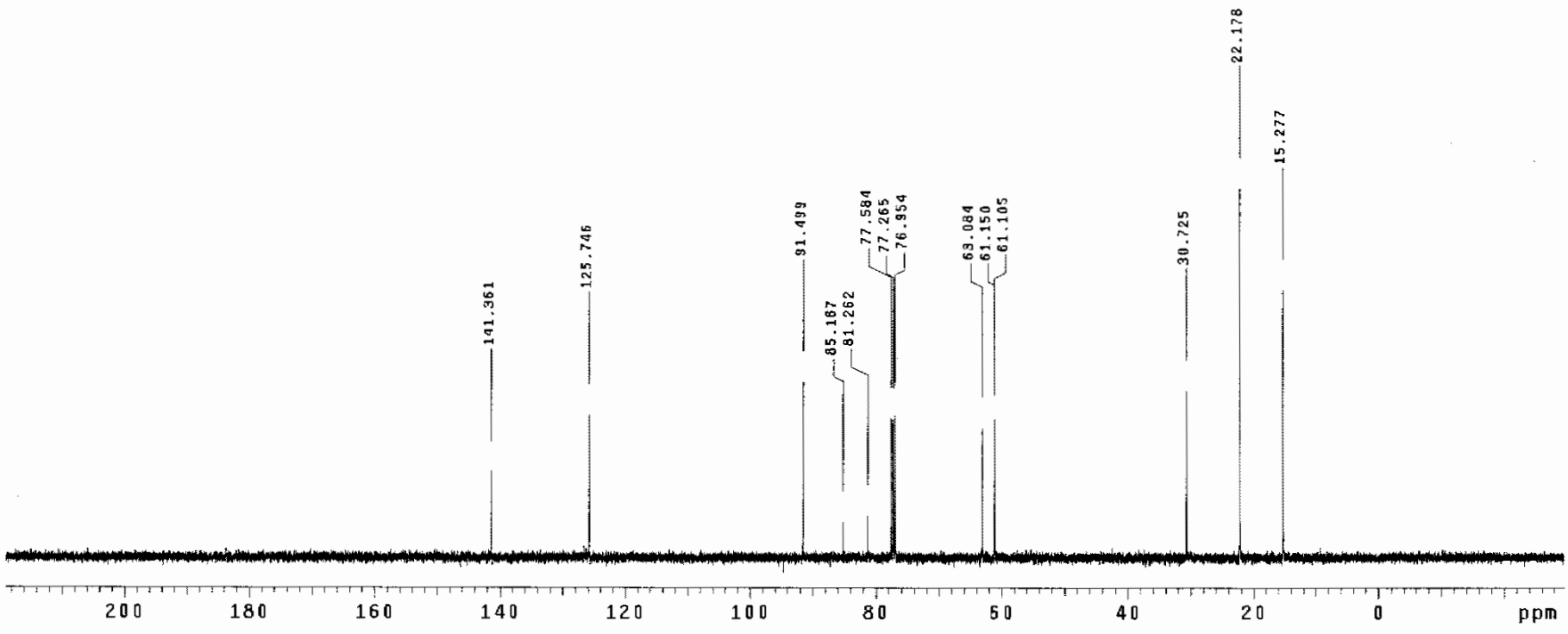
^{1}H NMR, 400 MHz, $CDCl_3$

S 66

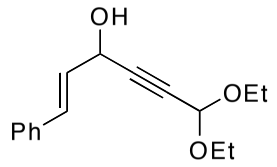


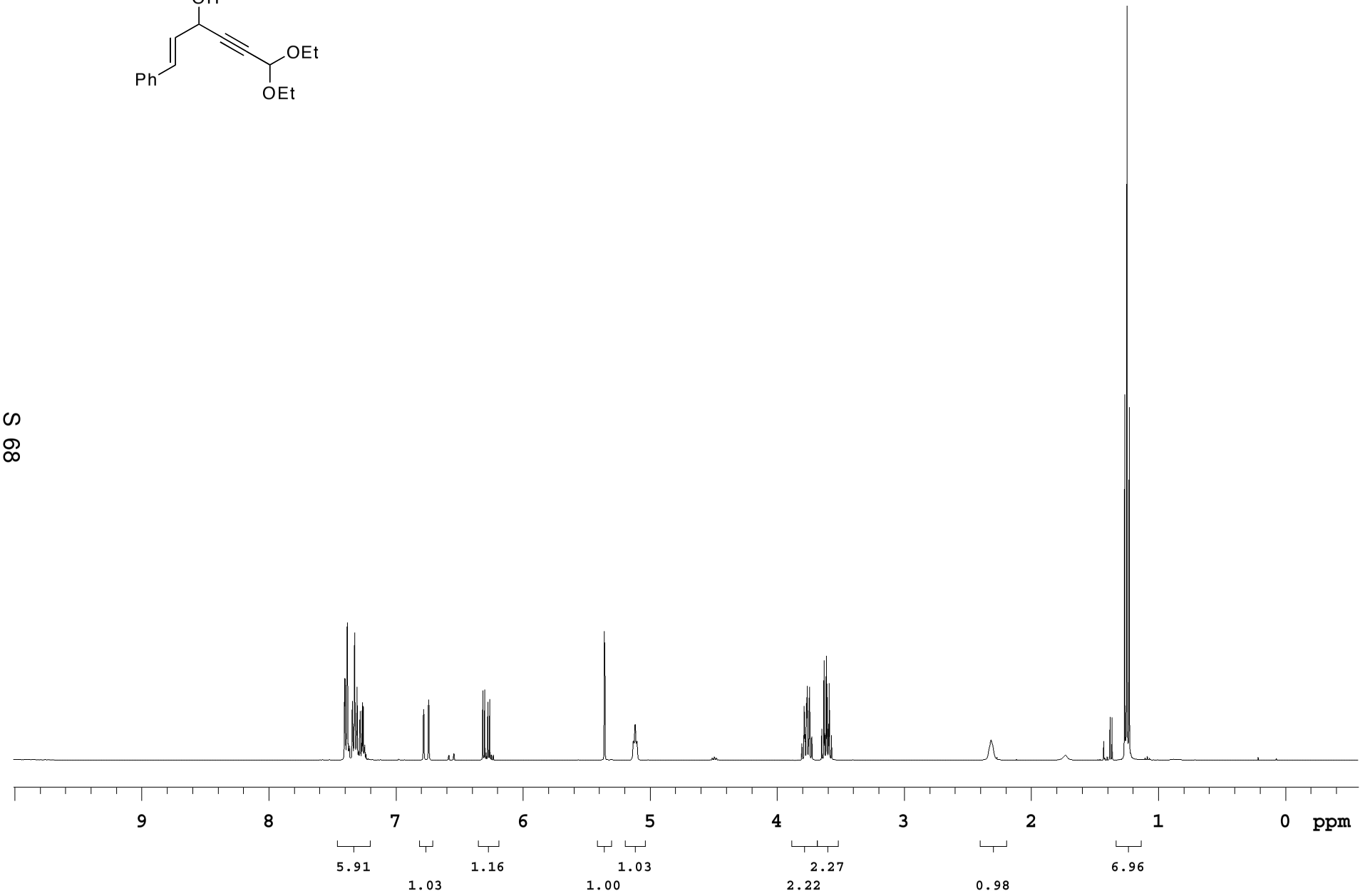
^{13}C NMR. 101 MHz, $CDCl_3$





^{1}H NMR, 400 MHz, $CDCl_3$





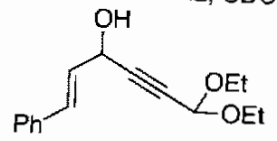
AHWII-98-13C

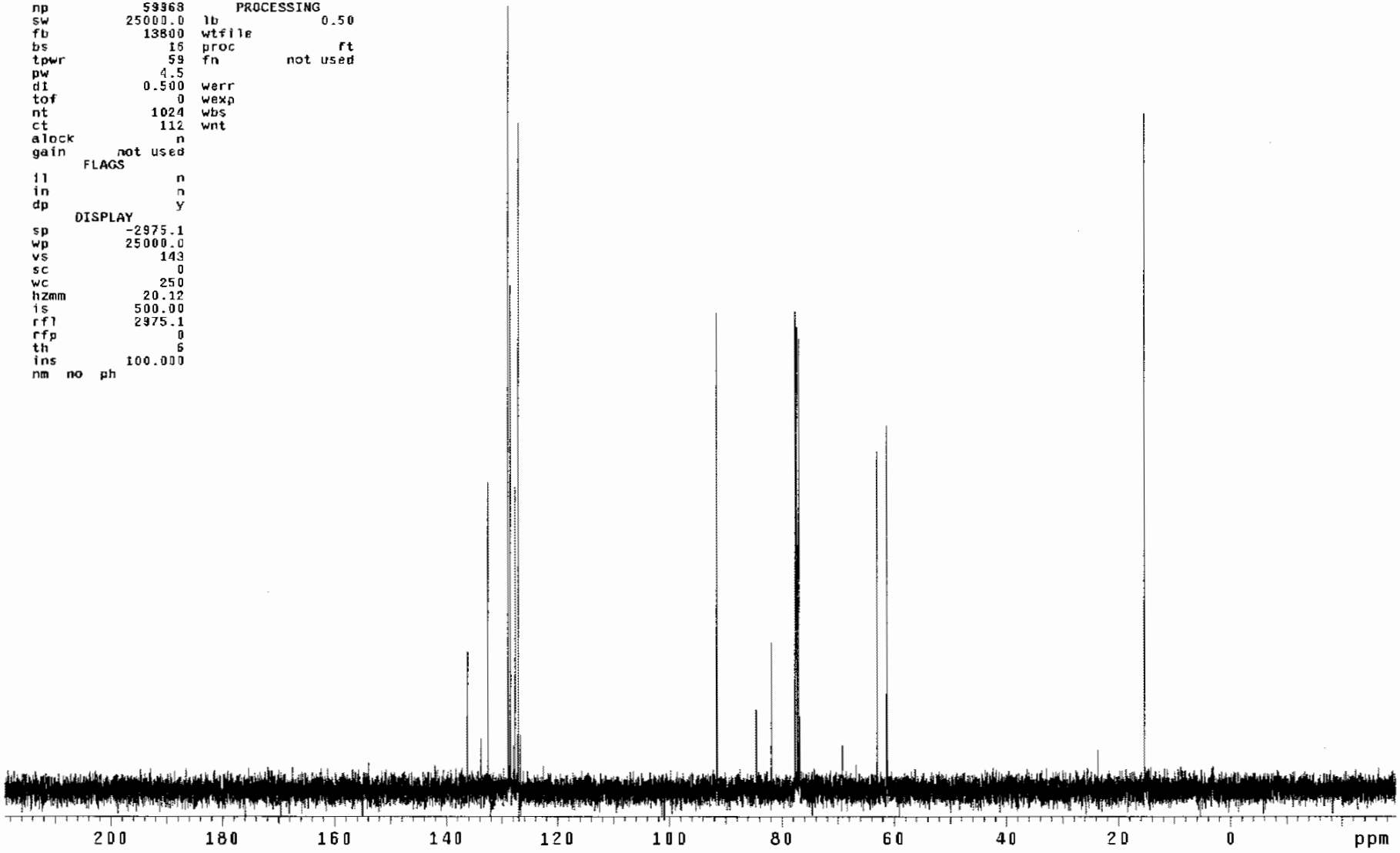
exp4 std13c

SAMPLE		DEC. & VT	
date	Apr 7 2005	dfrq	400.114
solvent	CDCl3	dn	H1
file	exp	dpwr	43
ACQUISITION		dof	0
sfrq	100.618	dm	yyy
tn	C13	dmm	w
at	1.199	dmf	8900
np	59368	PROCESSING	
sw	25000.0	lb	0.50
fb	13800	wtfile	
bs	16	proc	ft
tpwr	59	fn	not used
pw	4.5		
dl	0.500	werr	
tof	0	wexp	
nt	1024	wbs	
ct	112	wnt	
alock	n		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-2975.1		
wp	25000.0		
vs	143		
sc	0		
wc	250		
hzmm	20.12		
is	500.00		
rfl	2975.1		
rfp	0		
th	6		
ins	100.000		

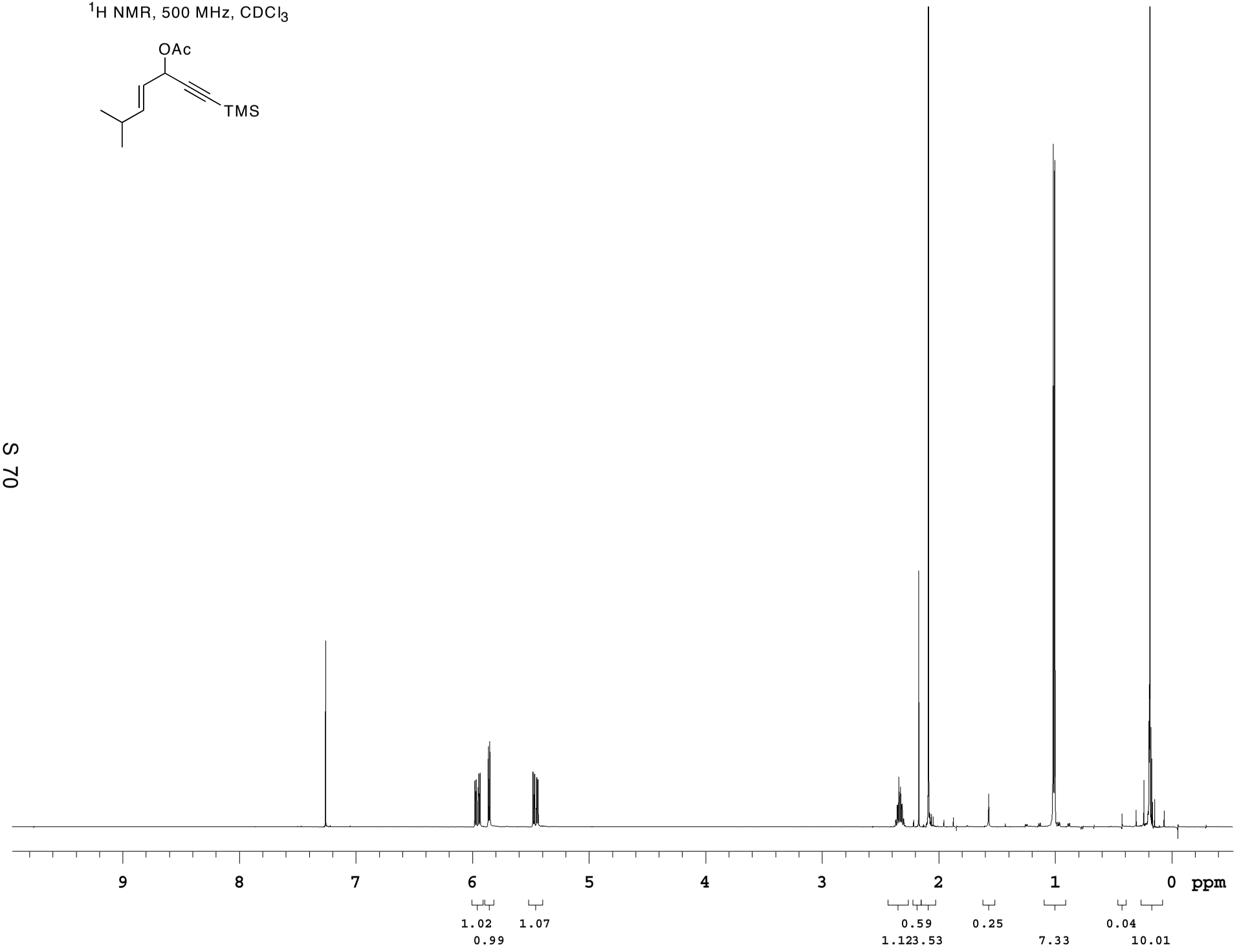
nm no ph

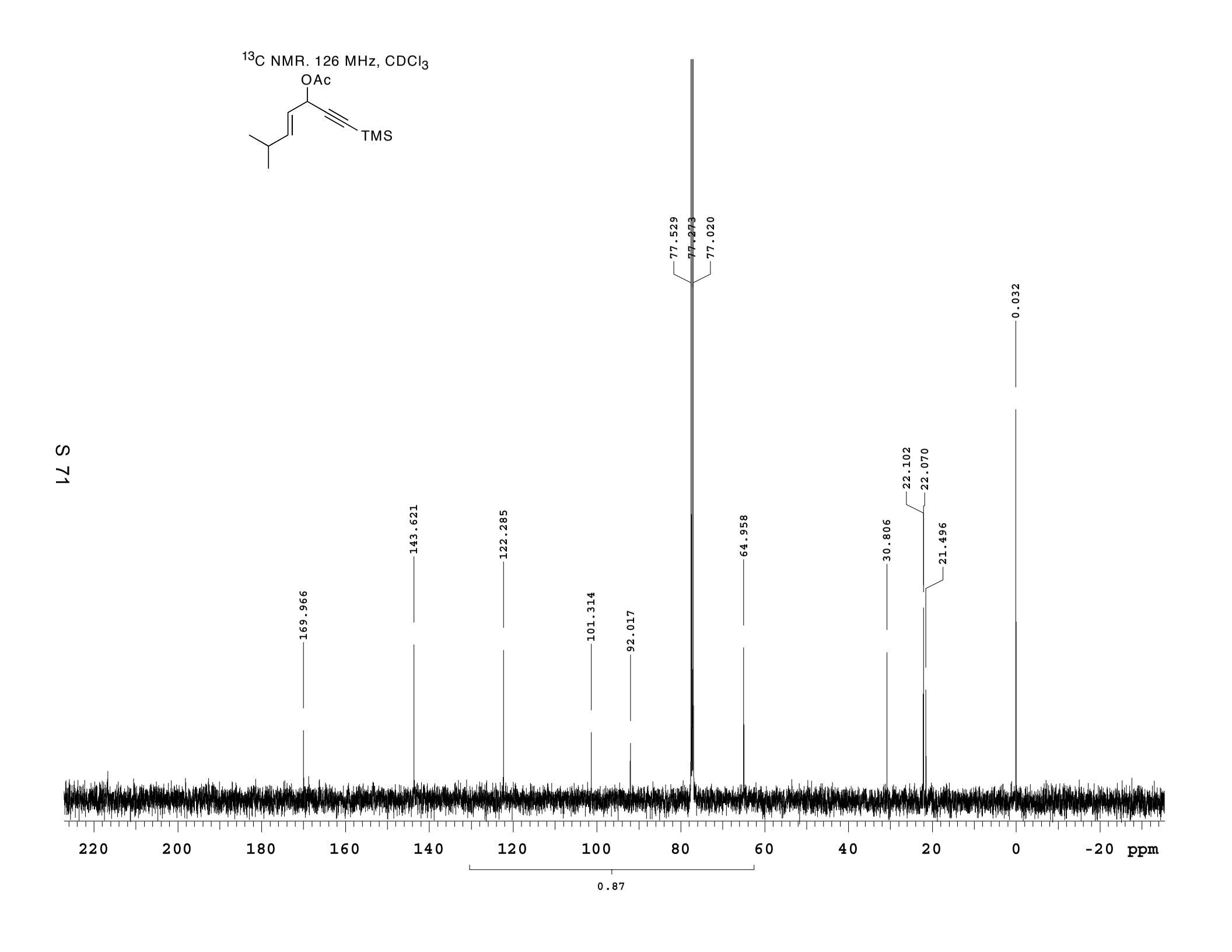
^{13}C NMR. 101 MHz, $CDCl_3$



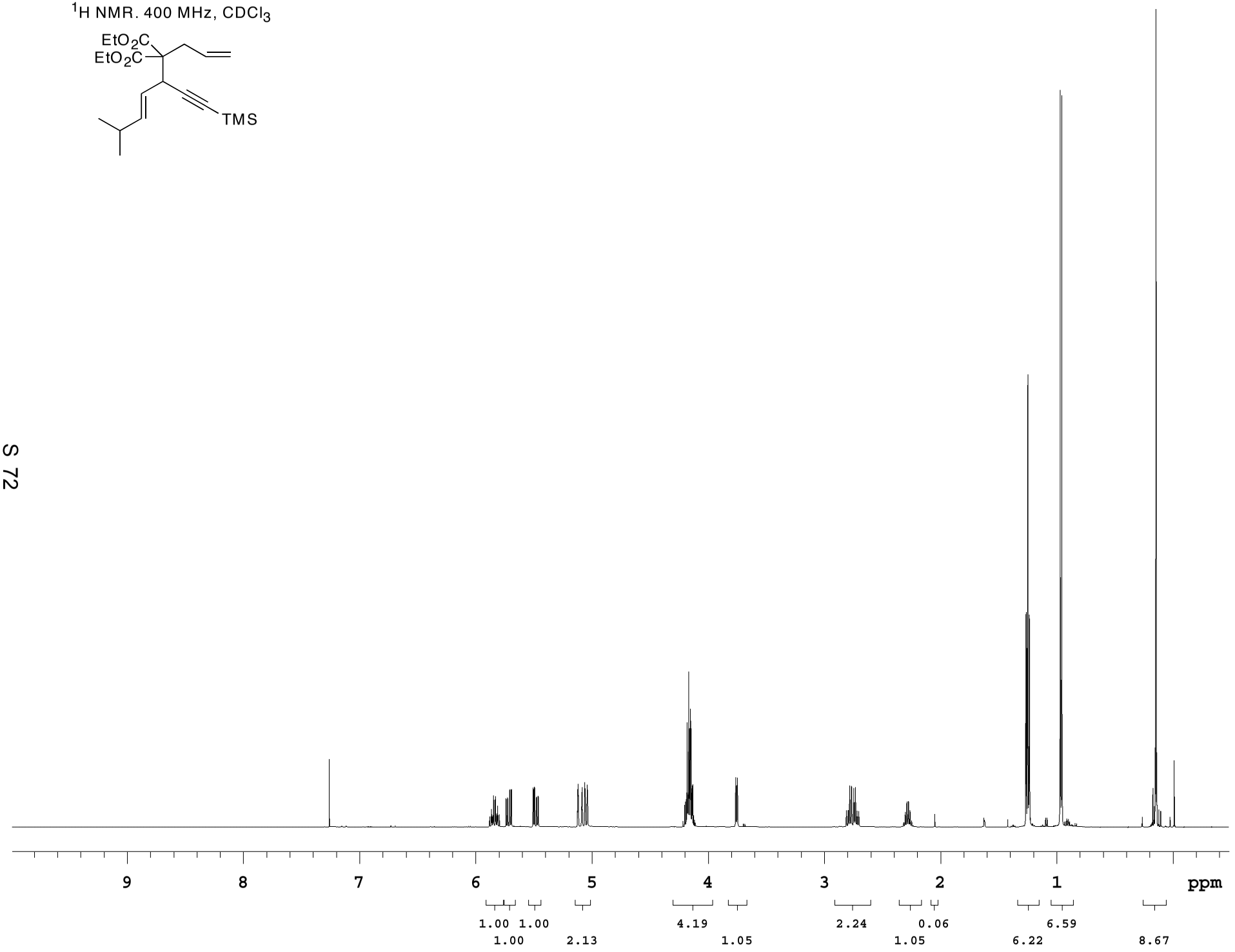


^{1}H NMR, 500 MHz, $CDCl_3$



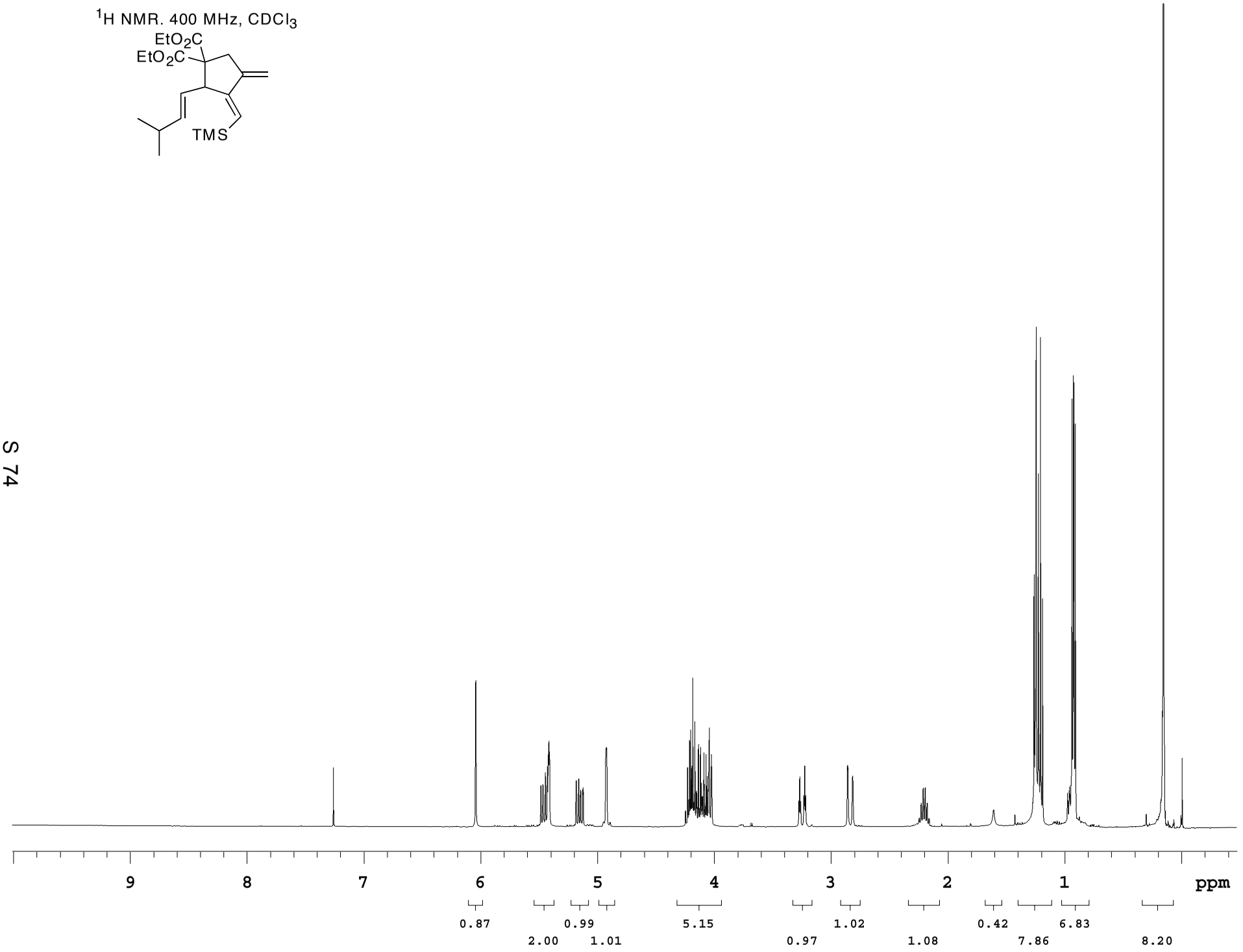

^{13}C NMR. 126 MHz, $CDCl_3$
OAc
TMS
77.529
77.273
77.020
0.032
22.102
22.070
21.496
30.806
64.958
143.621
122.285
101.314
92.017
169.966
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm
0.87

^{1}H NMR. 400 MHz, $CDCl_3$



^{13}C NMR. 101 MHz, CDCl$_3$
EtO$_2$C
EtO$_2$C
TMS
169.444
169.118
141.409
133.318
122.125
118.621
103.826
89.797
77.323
77.004
76.686
61.322
61.208
61.064
39.960
37.988
30.883
22.230
14.093
-0.042
ppm
200
180
160
140
120
100
80
60
40
20
0

^{1}H NMR. 400 MHz, $CDCl_3$



^{13}C NMR. 101 MHz, $CDCl_3$
EtO_2C
EtO_2C
TMS
170.949
169.114
155.305
145.098
140.904
124.433
120.665
105.854
77.318
77.000
76.682
62.584
61.416
61.189
52.339
37.370
31.159
22.234
22.203
14.104
13.998
-0.122
ppm
0
20
40
60
80
100
120
140
160
180
200