

Application of metathesis reaction in the synthesis of sterically congested medium-sized rings. A direct ring closing versus a double bond migration – ring closing process.

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Polish Academy of Sciences

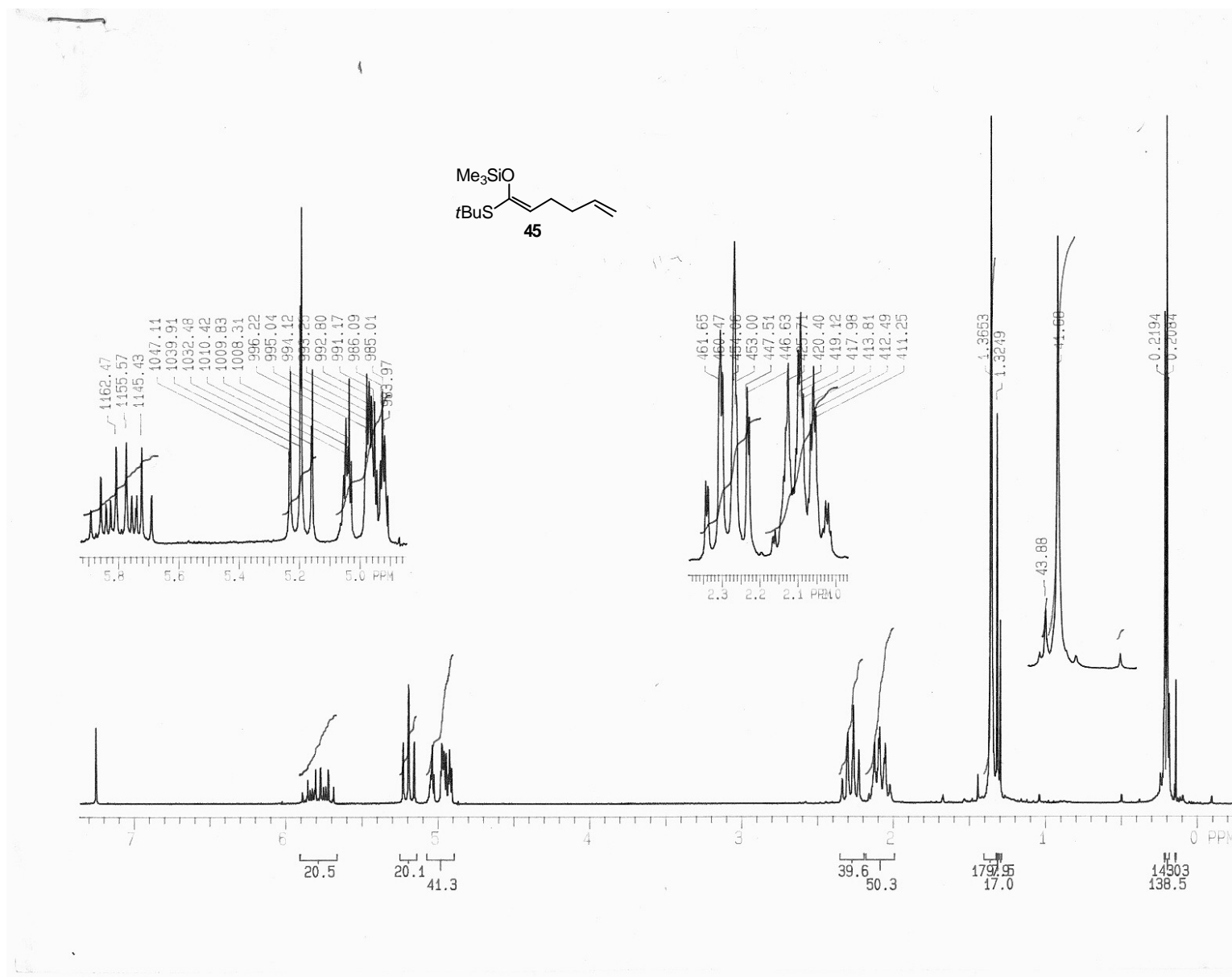
Kasprzaka 44/52, 01-224 Warsaw

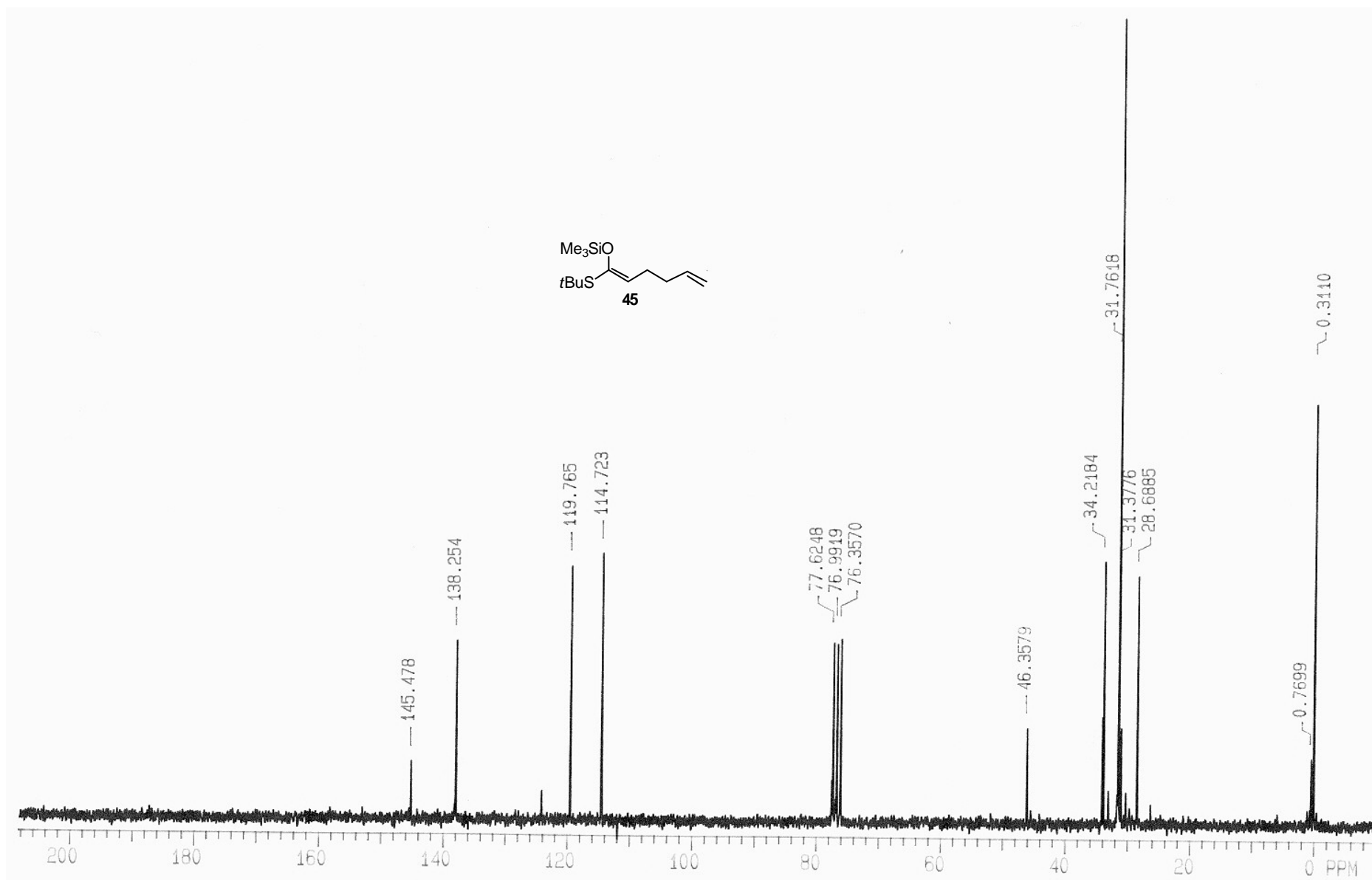
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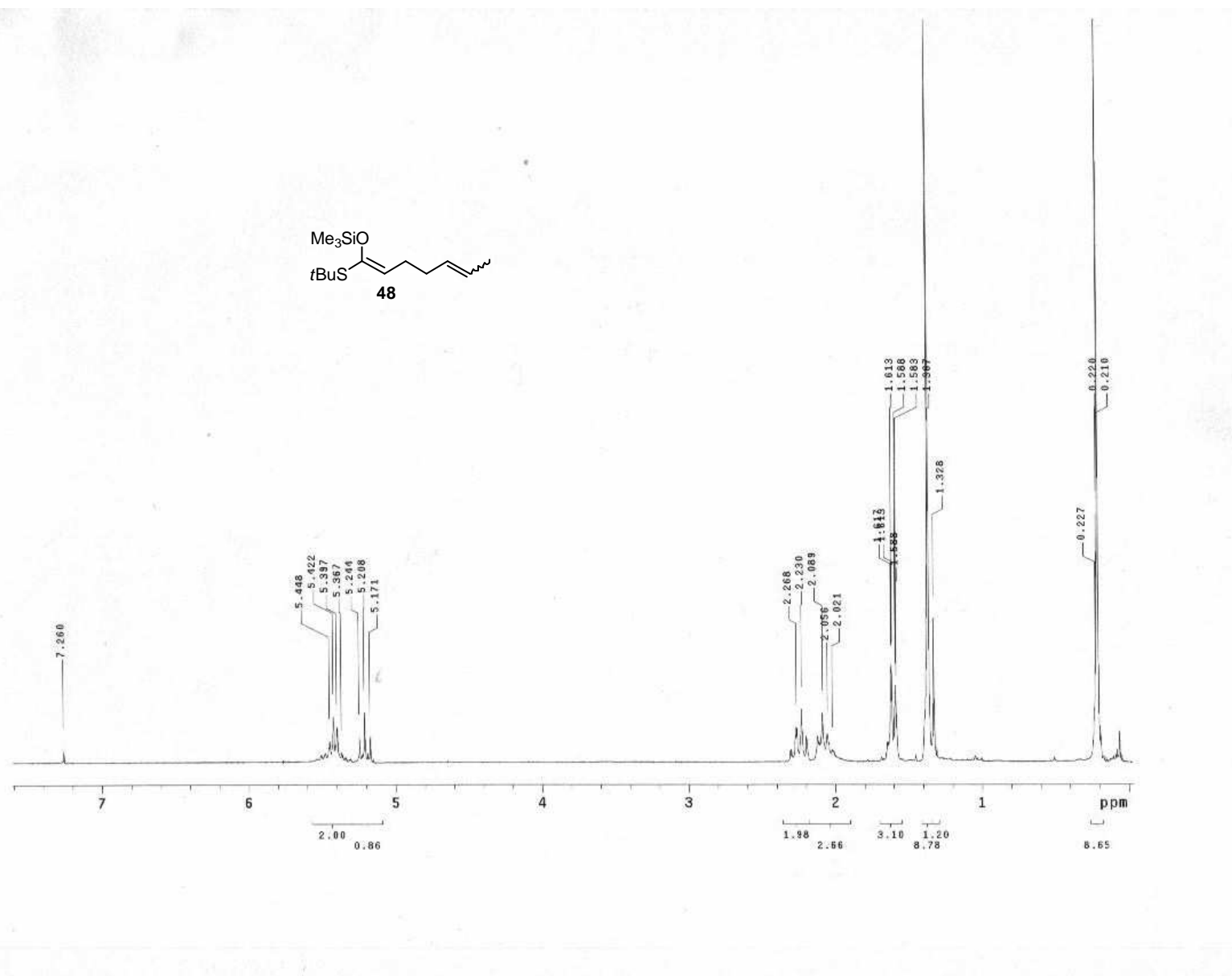
E-mail: jwicha@icho.edu.pl

Supporting Information

Selected ¹H and ¹³C NMR spectra of reported compounds

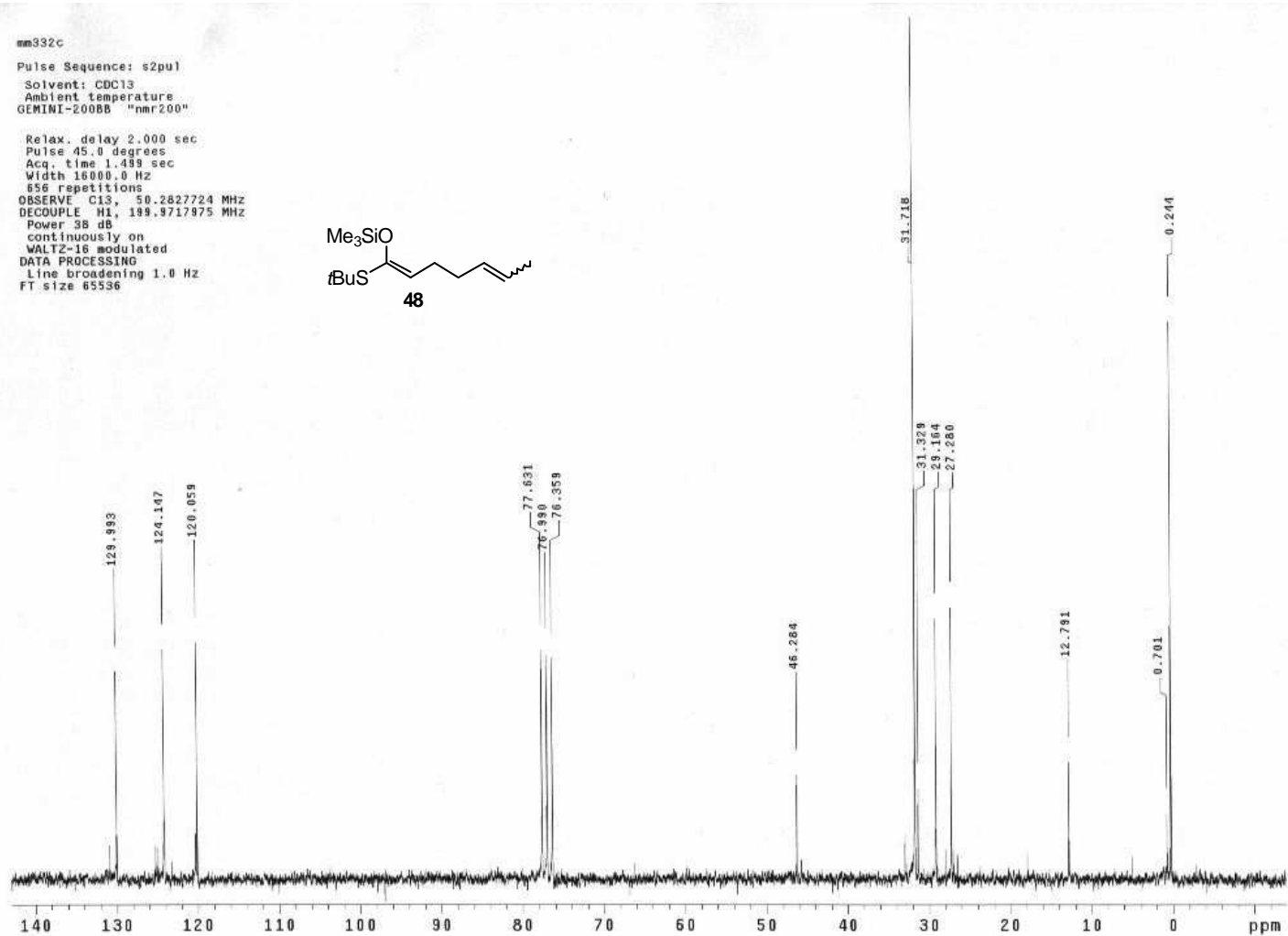
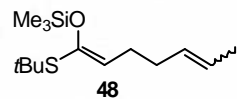






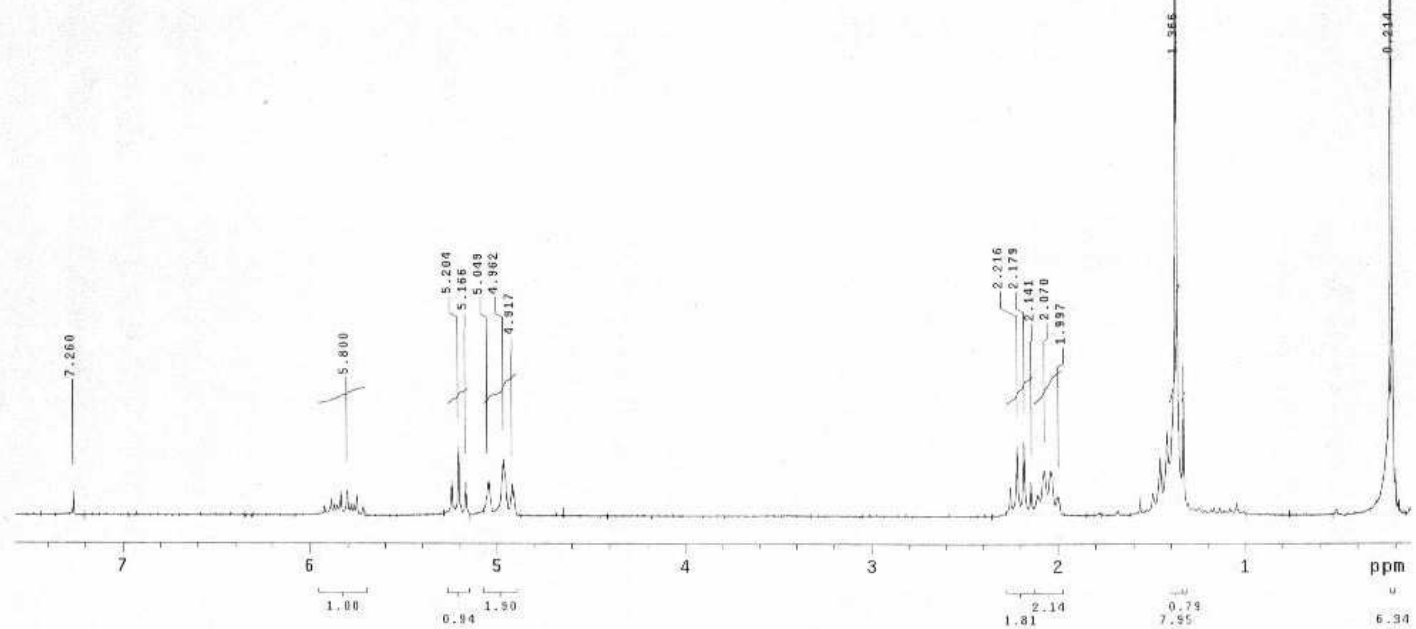
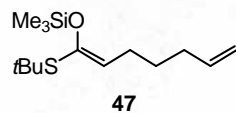
mm332c
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
GEMINI-200BB "nmr200"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.499 sec
Width 16000.0 Hz
656 repetitions
OBSERVE C13, 50.2827724 MHz
DECOUPLE H1, 199.9717975 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536



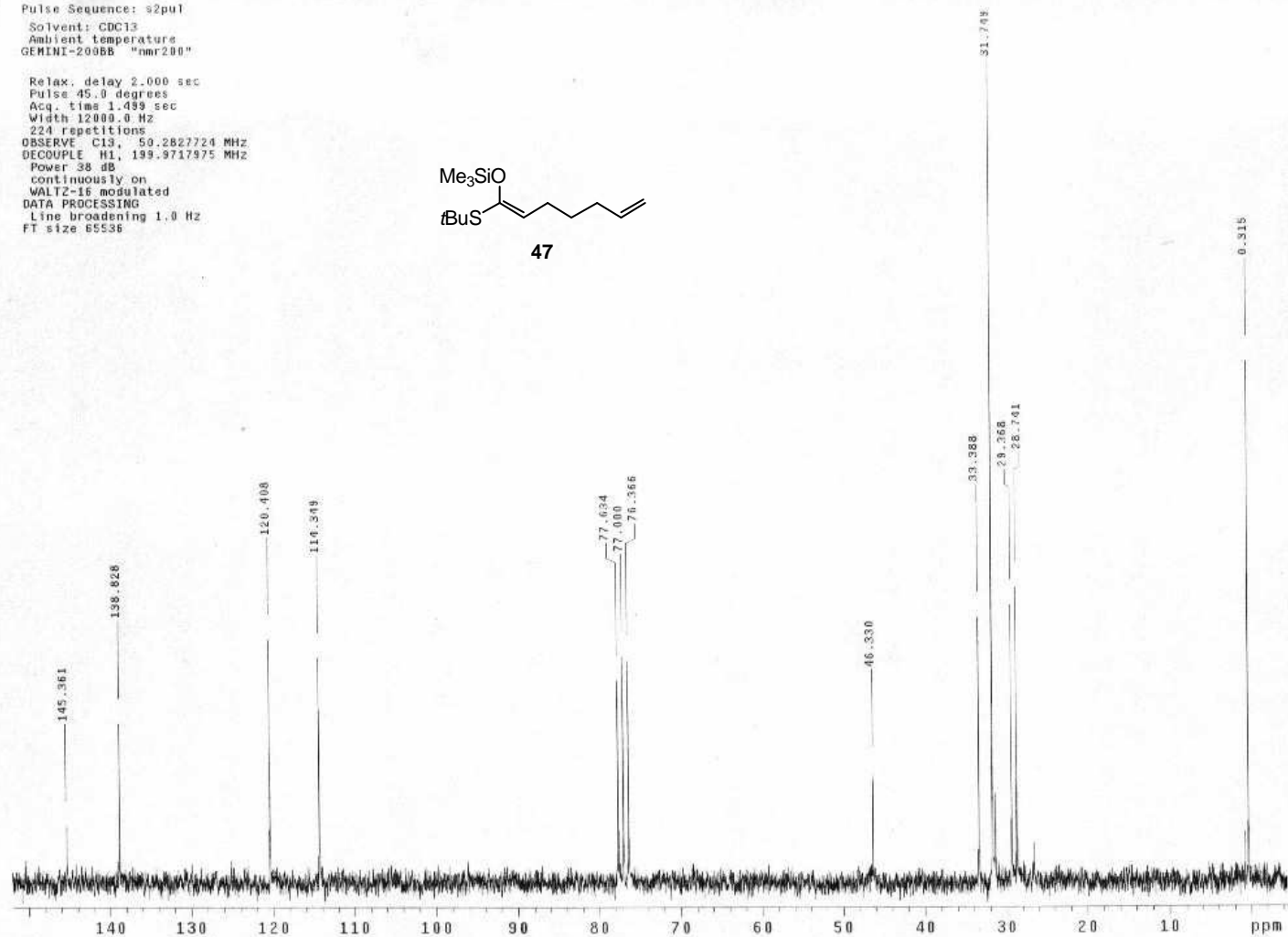
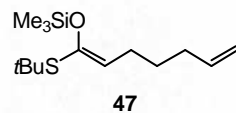
alkoholderabiany
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
CEMIRI-200BB "nmr200"

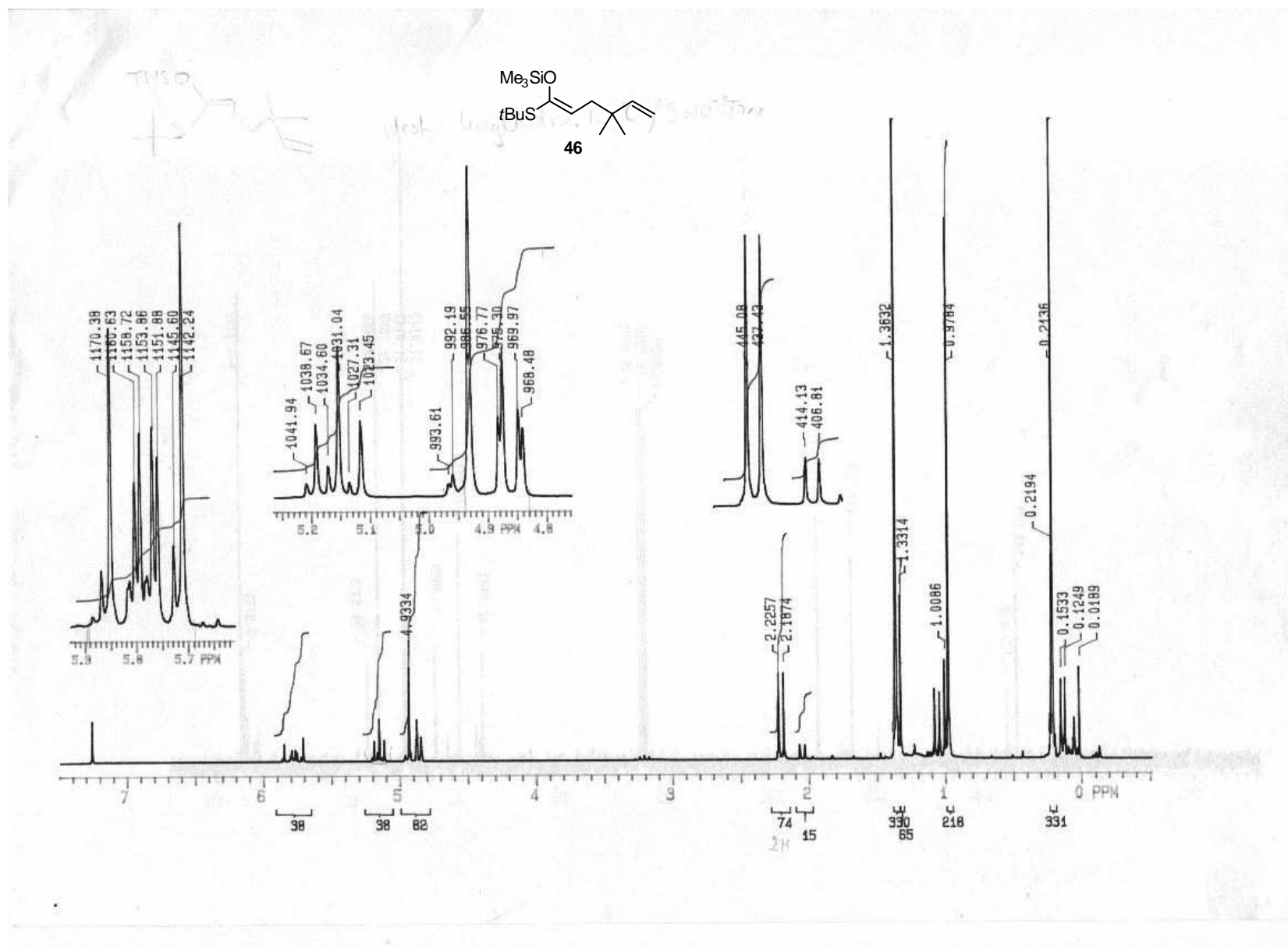
Relax. delay 0.100 sec
Pulse 56.2 degrees
Acq. time 4.009 sec
Width 2531.2 Hz
44 repetitions
OBSERVE H1, 199.9710974 MHz
DATA PROCESSING
FT size 32768

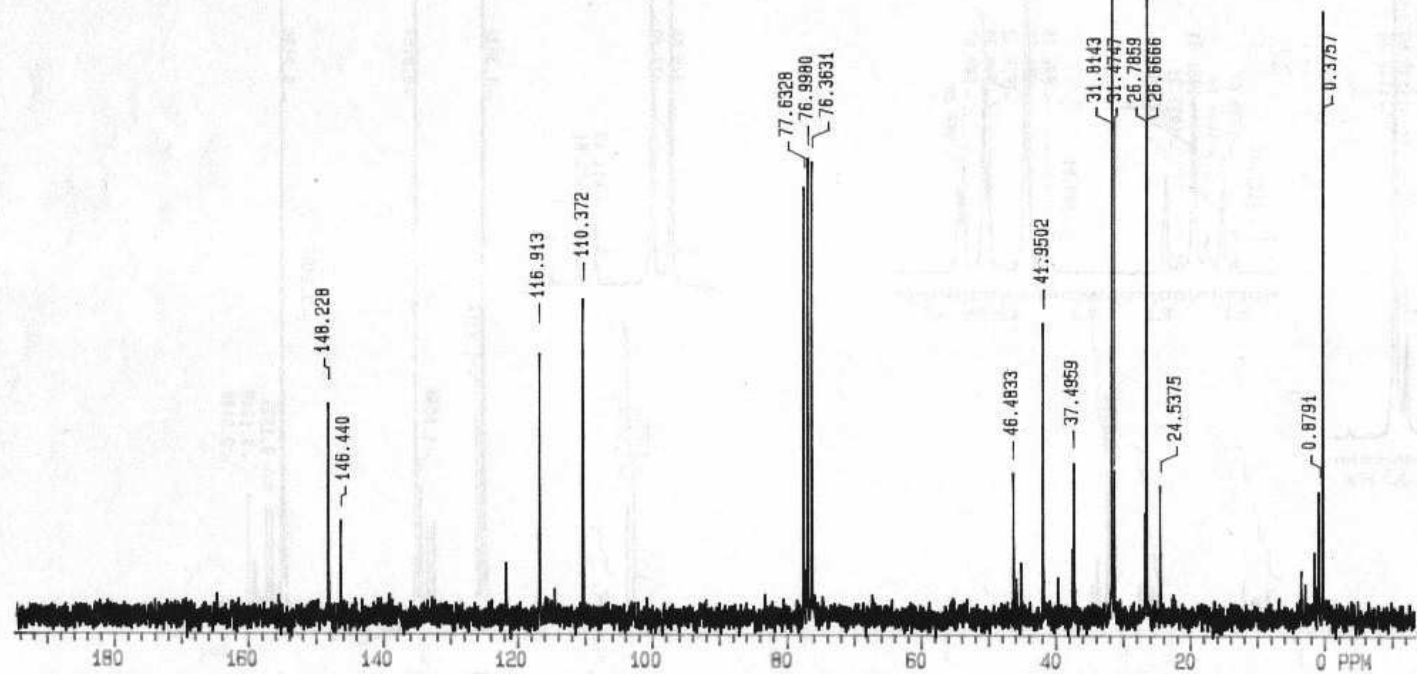
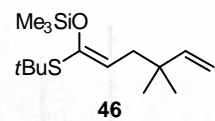


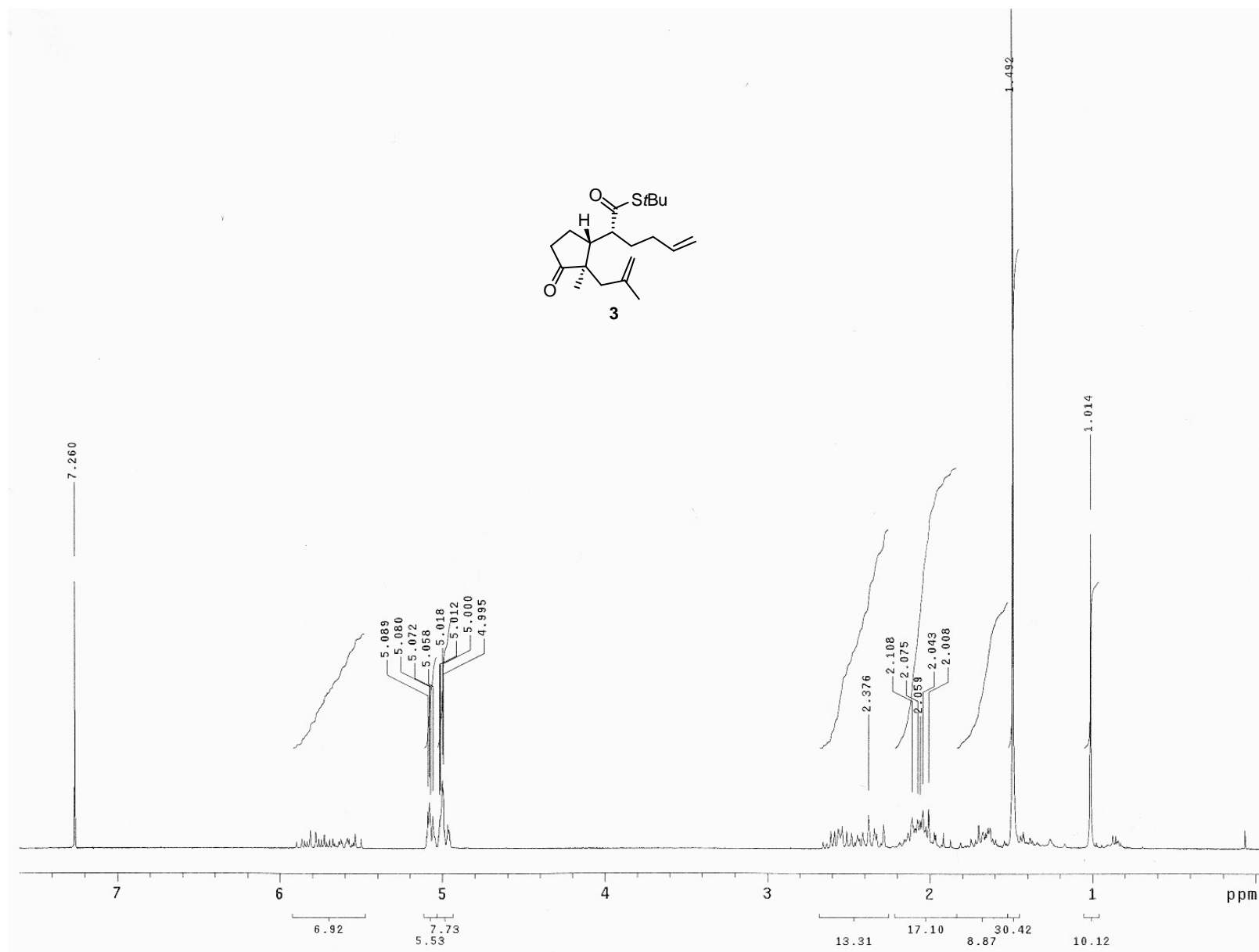
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Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-200BB "nmr200"

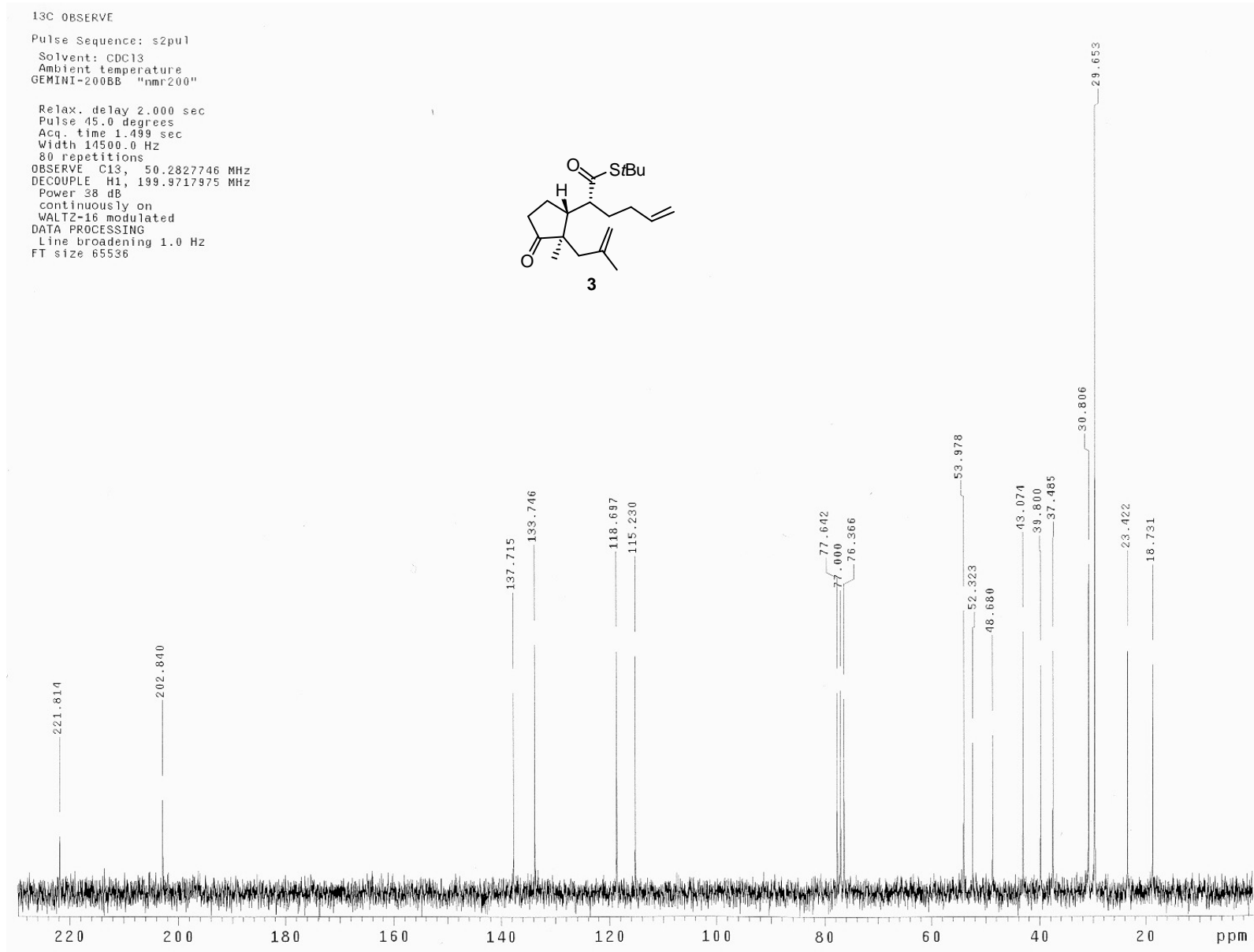
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.499 sec
Width 12000.0 Hz
224 repetitions
OBSERVE C13, 50.2827724 MHz
DECOUPLE H1, 199.9717975 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536





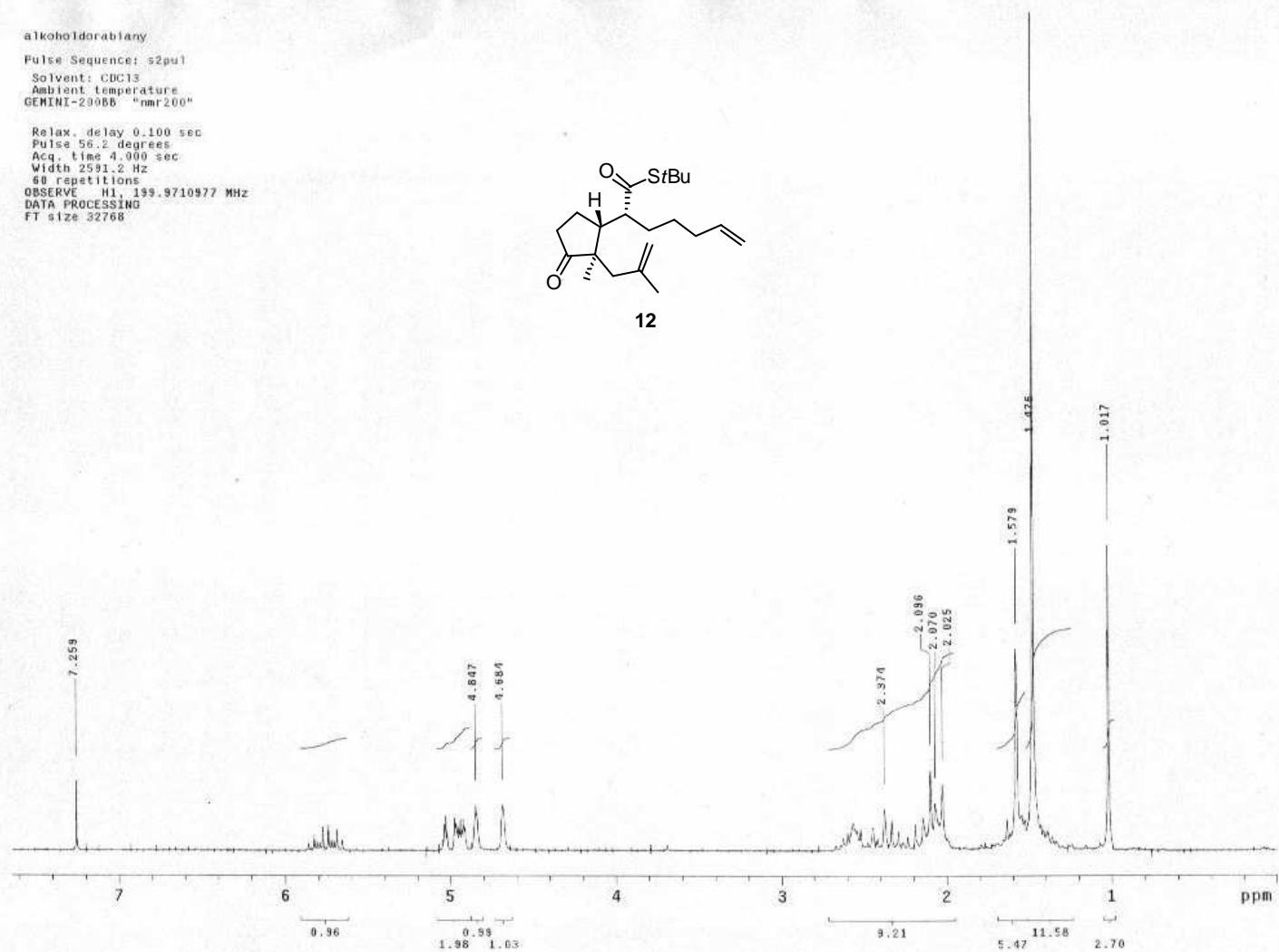
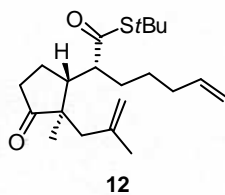






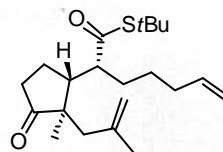
alkoholadorabiany
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
GEMINI-2006B "nmr200"

Relax. delay 0.100 sec
Pulse 56.2 degrees
Acq. time 4.000 sec
Width 2581.2 Hz
60 repetitions
OBSERVE H1, 199.9710977 MHz
DATA PROCESSING
FT size 32768

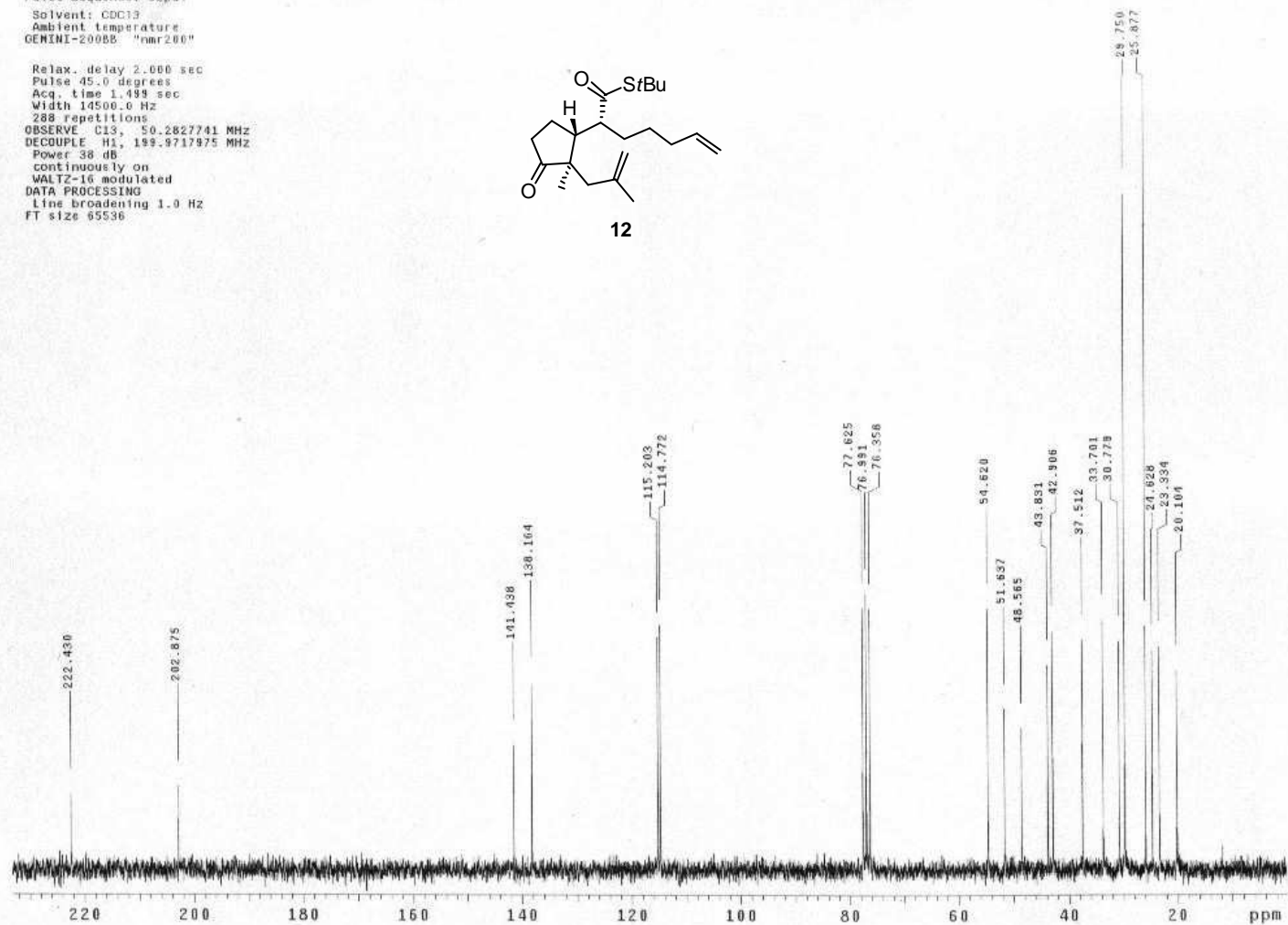


13C OBSERVE
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
GENI-200SB "nmr200"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.489 sec
Width 14500.0 Hz
288 repetitions
OBSERVE C13, 50.2827741 MHz
DECOUPLE H1, 199.9717975 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536

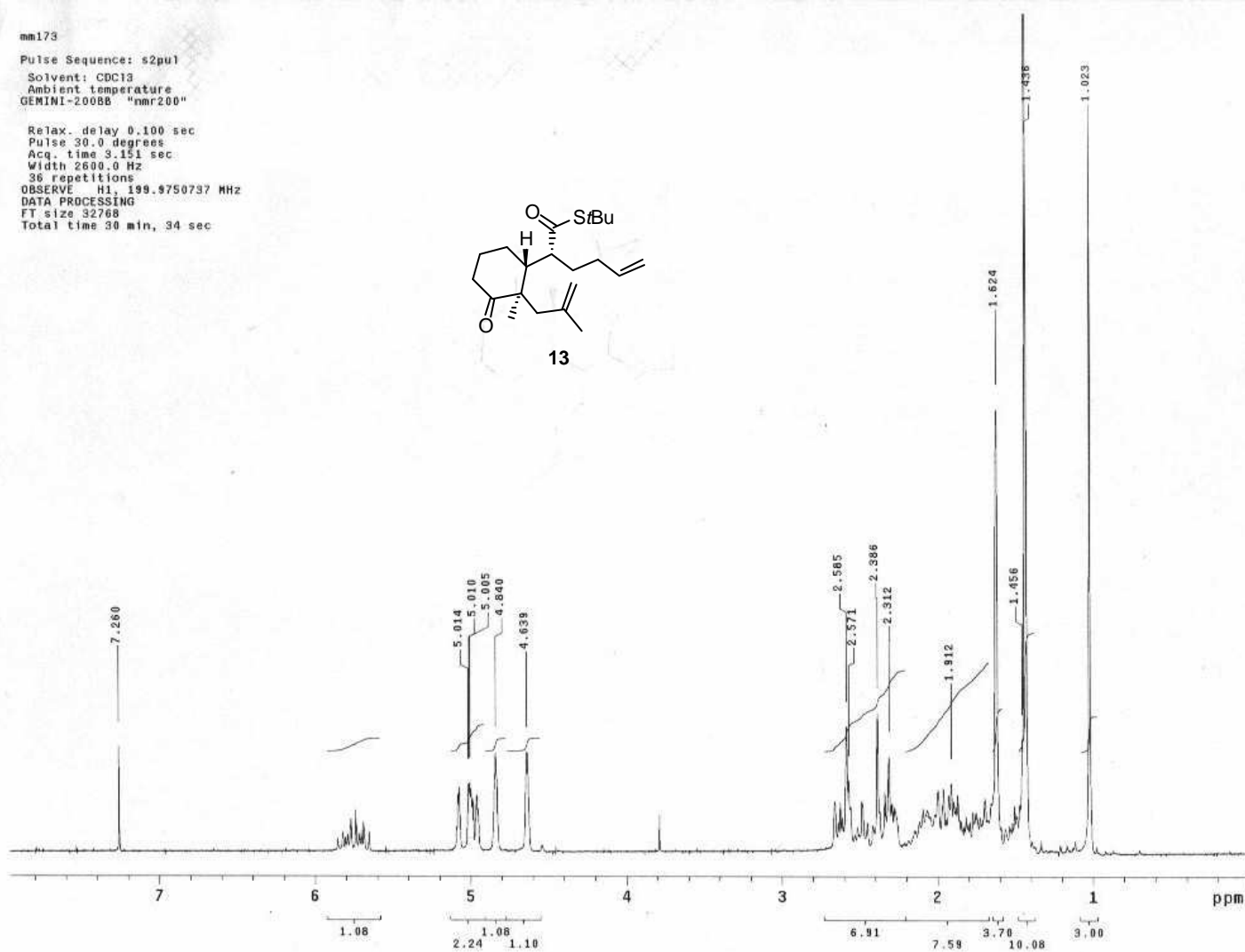
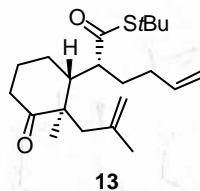


12



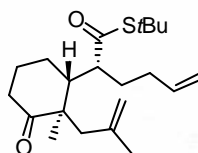
nm173
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
GERINI-2008B "nmr200"

Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 3.151 sec
Width 2600.0 Hz
36 repetitions
OBSERVE H1, 199.9750737 MHz
DATA PROCESSING
FT size 32768
Total time 30 min, 34 sec

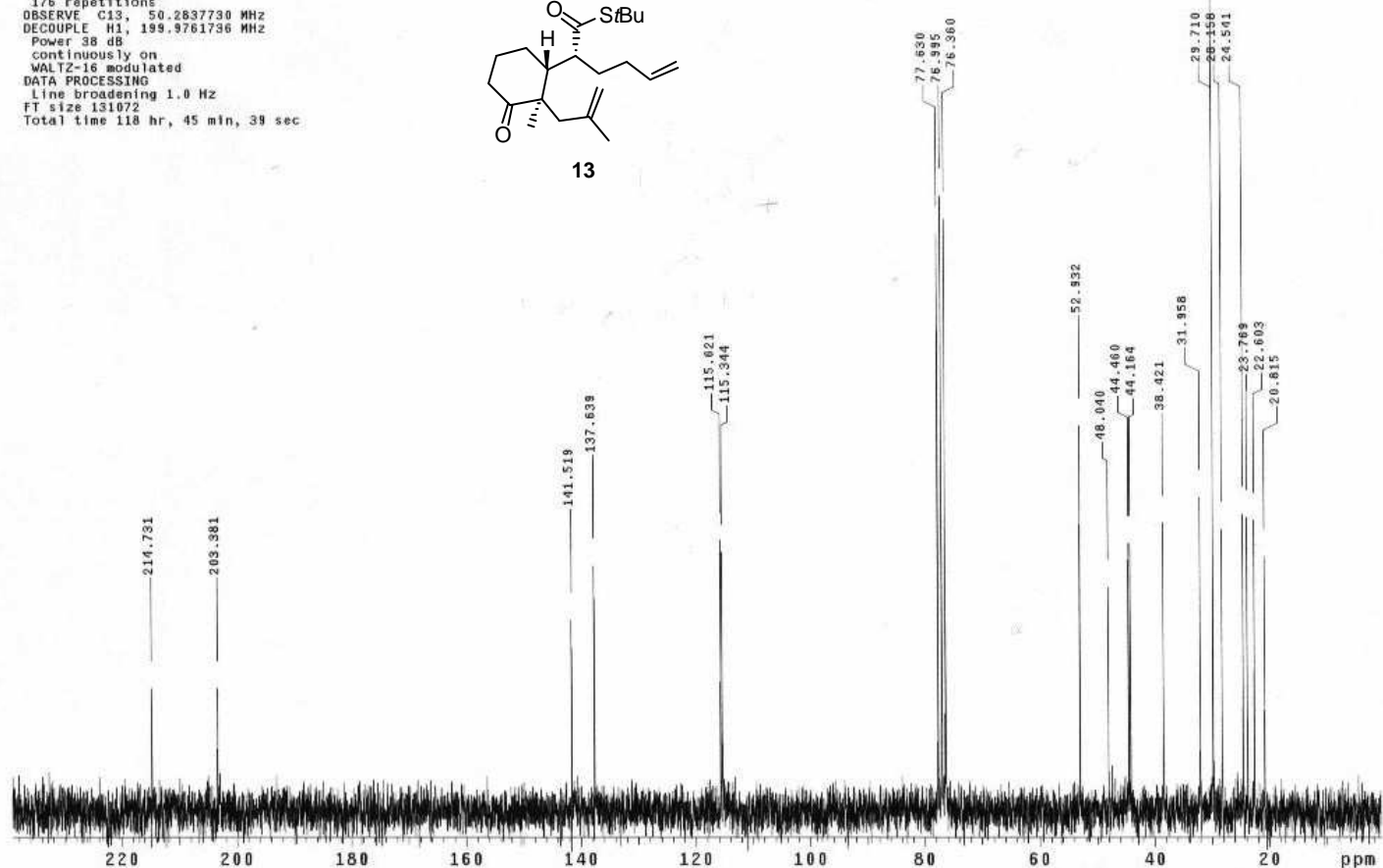


nm173
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-200BB "nmr200"

Relax. delay 2.000 sec
Pulse 48.0 degrees
Acq. time 2.000 sec
Width 15500.0 Hz
176 repetitions
OBSERVE C13, 50.2837730 MHz
DECOUPLE H1, 199.9761736 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 118 hr, 45 min, 39 sec



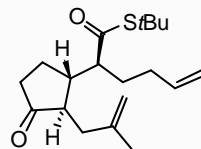
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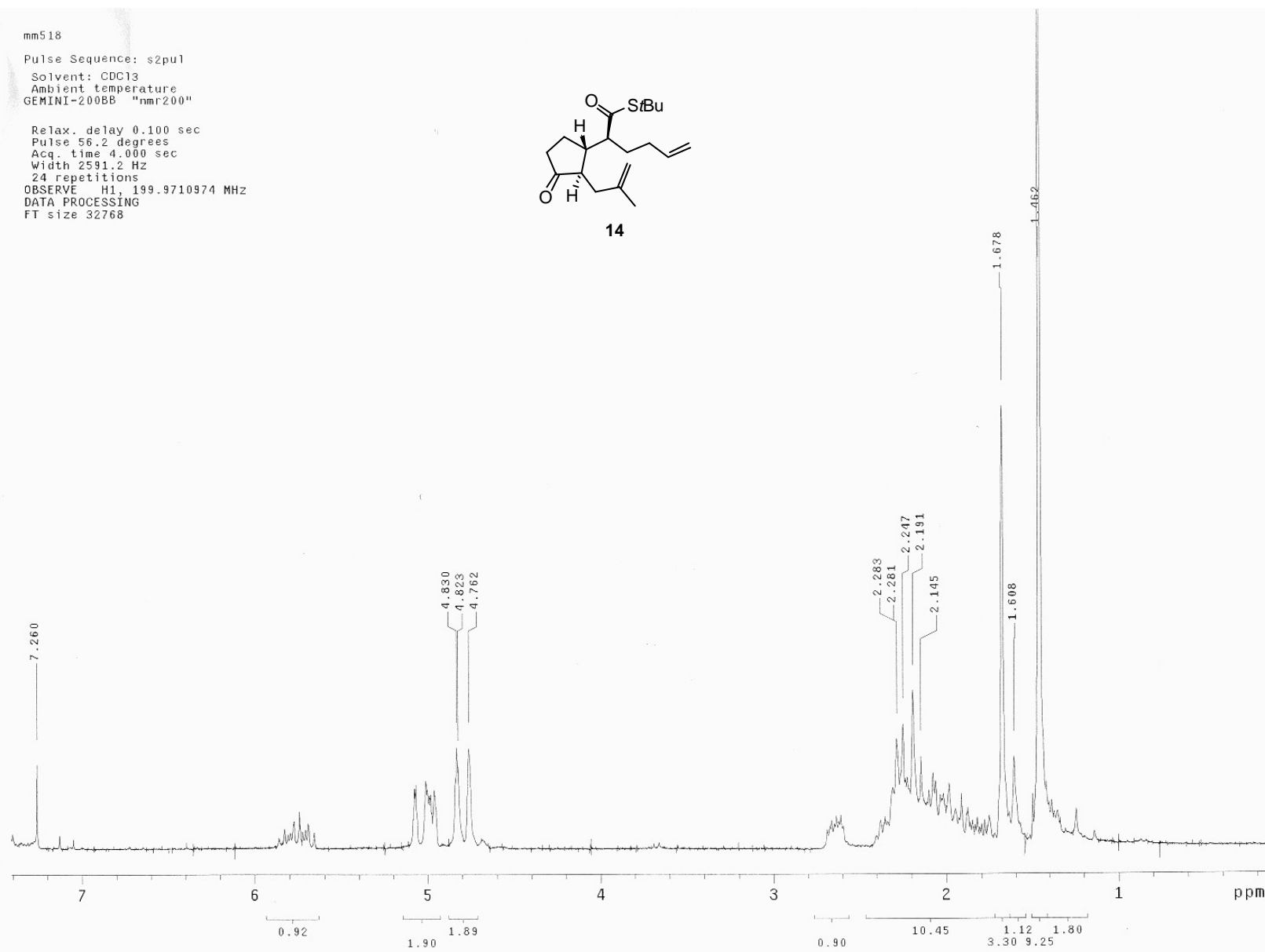
mm518

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
GEMINI-200BB "nmr200"

Relax. delay 0.100 sec
Pulse 56.2 degrees
Acq. time 4.000 sec
Width 2591.2 Hz
24 repetitions
OBSERVE H1, 199.9710974 MHz
DATA PROCESSING
FT size 32768



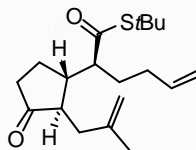
14



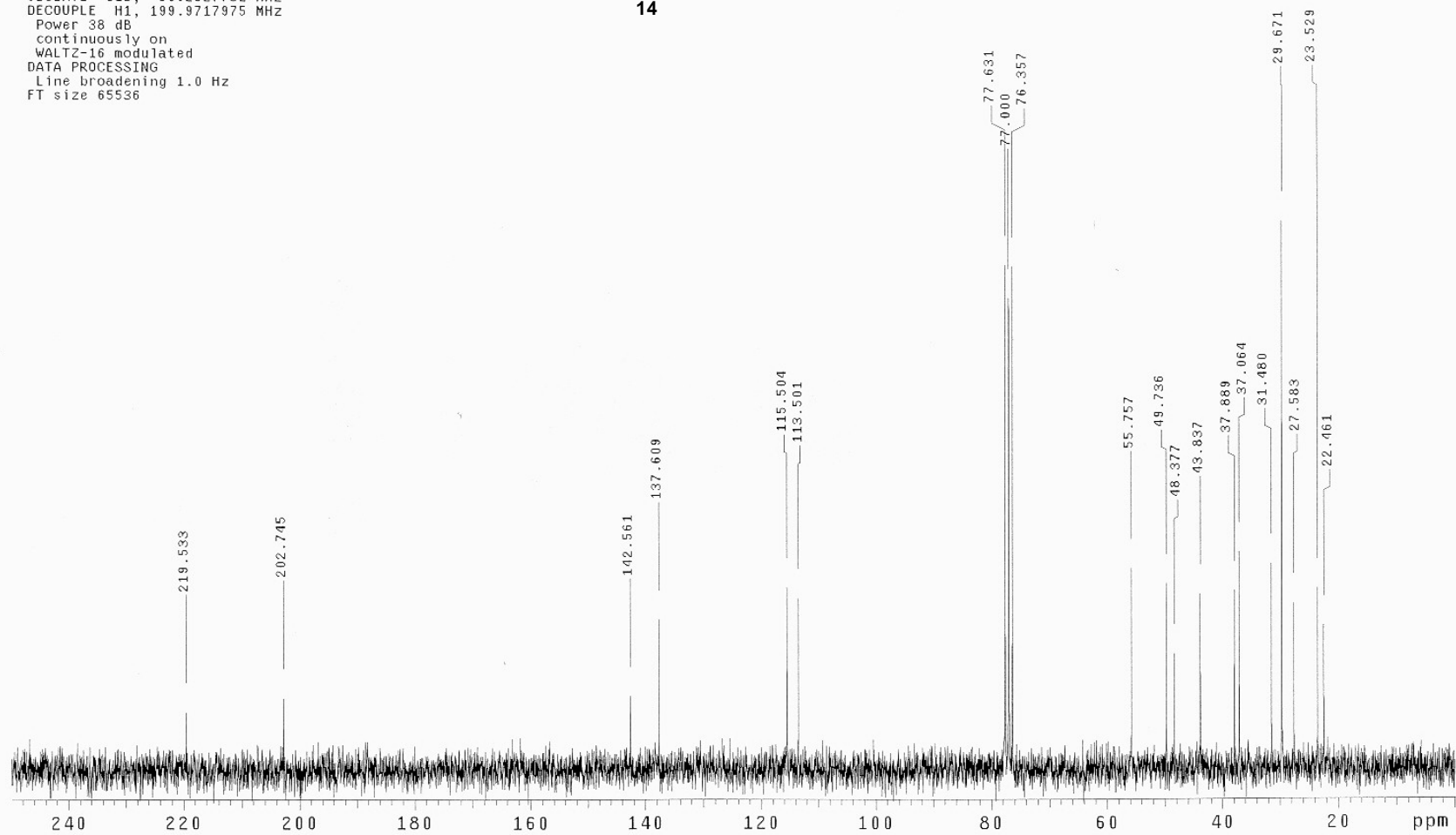
mm491

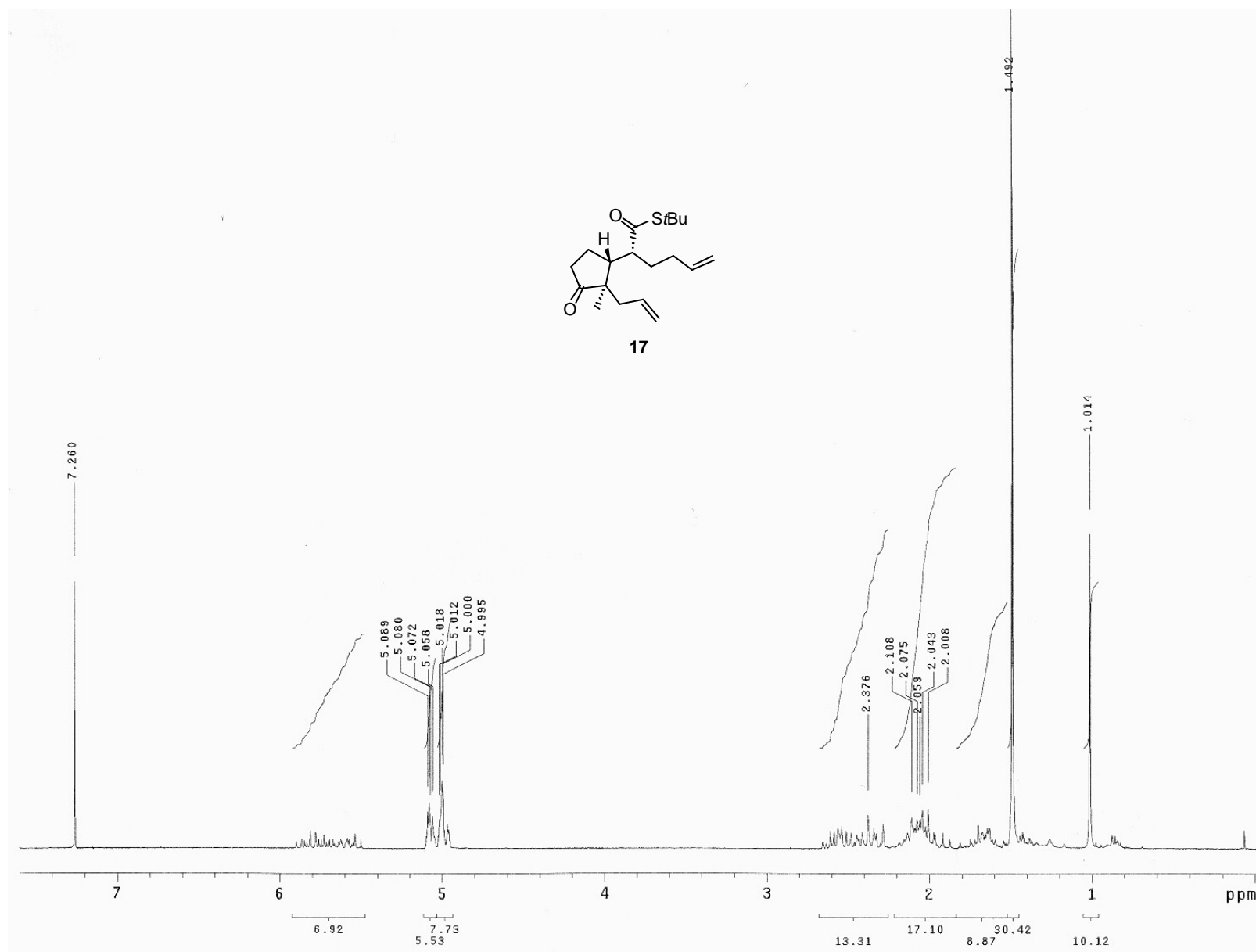
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
GEMINI-200BB "nmr200"

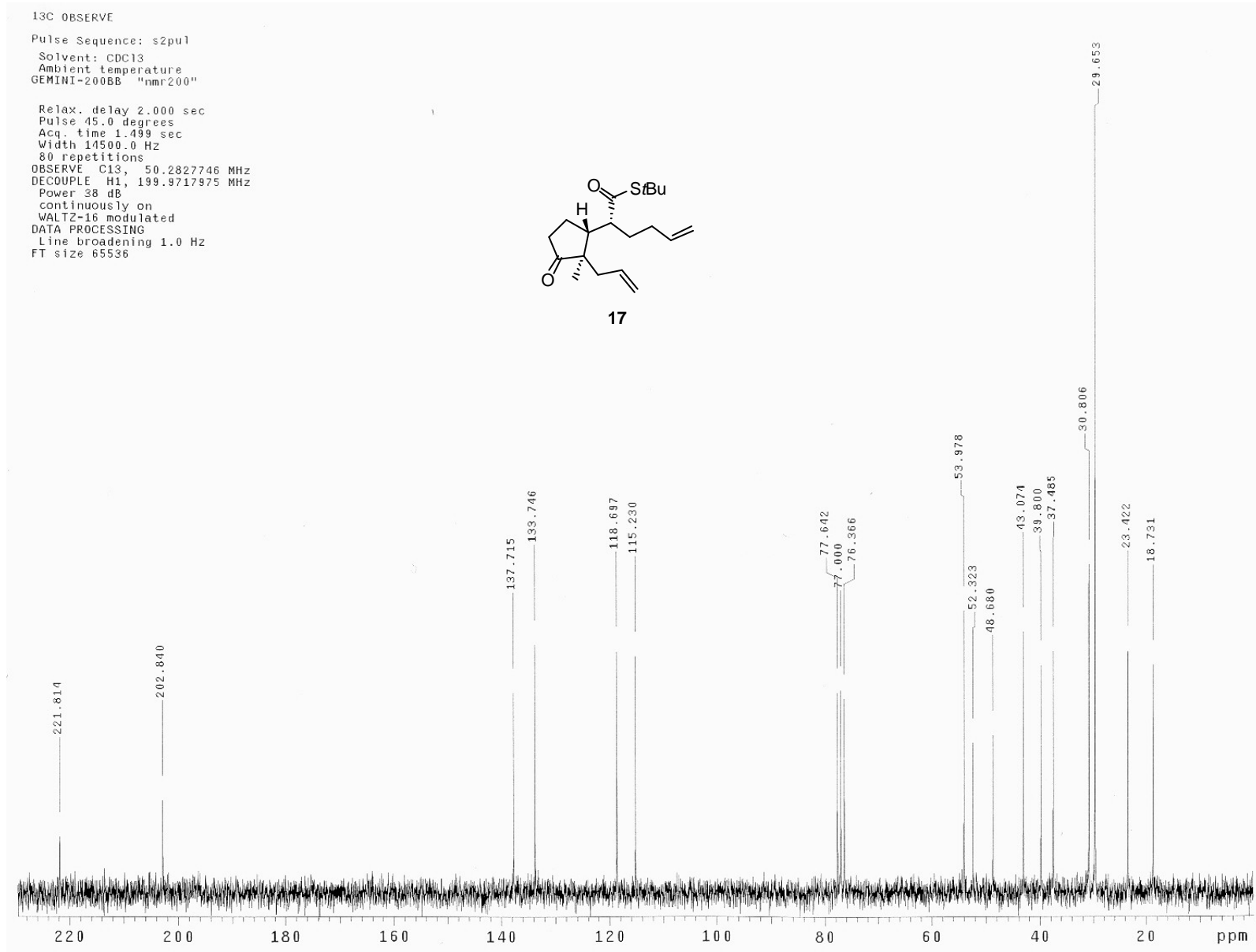
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.499 sec
Width 20000.0 Hz
272 repetitions
OBSERVE C13, 50.2827732 MHz
DECOUPLE H1, 199.9717975 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536

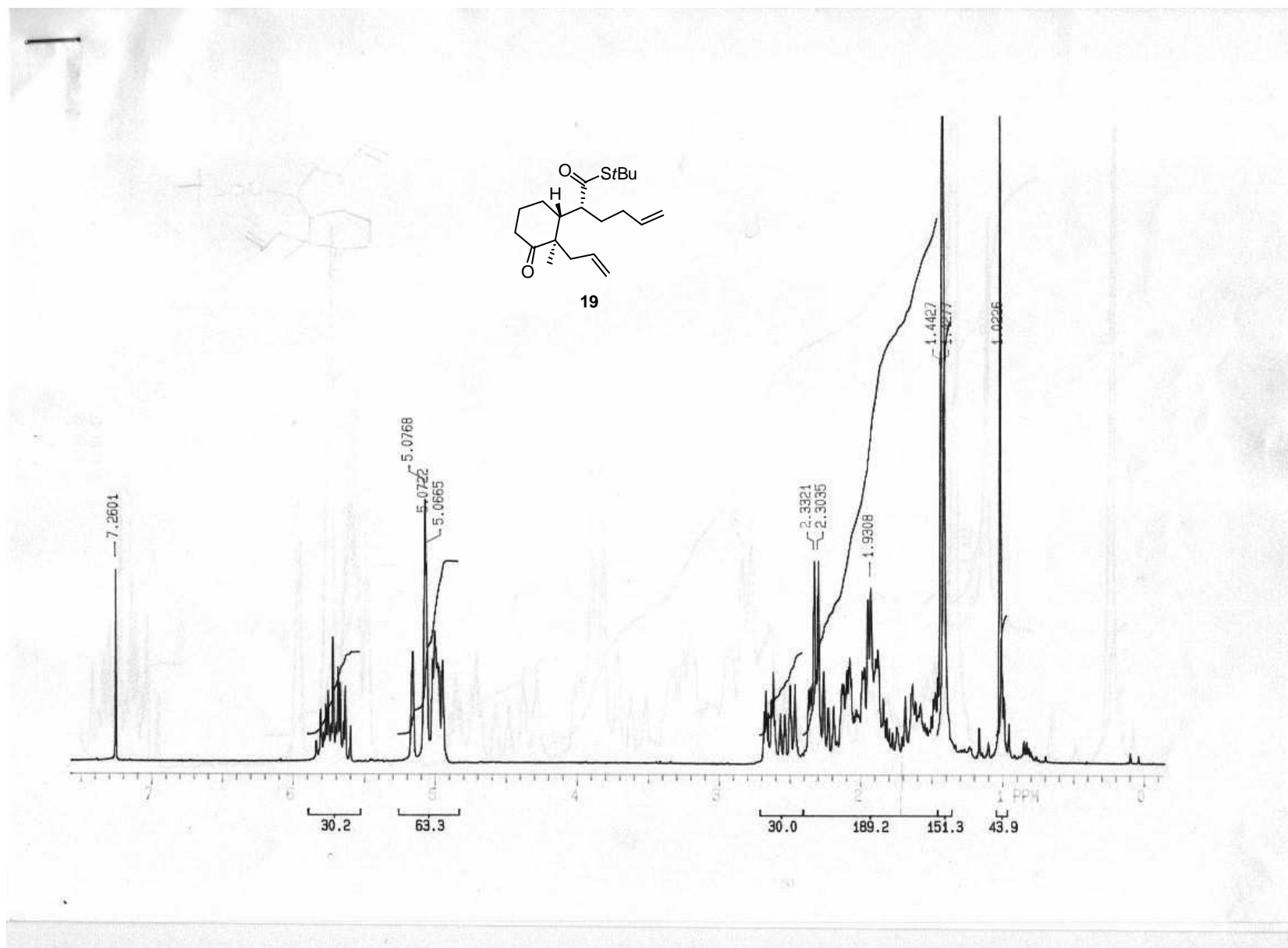


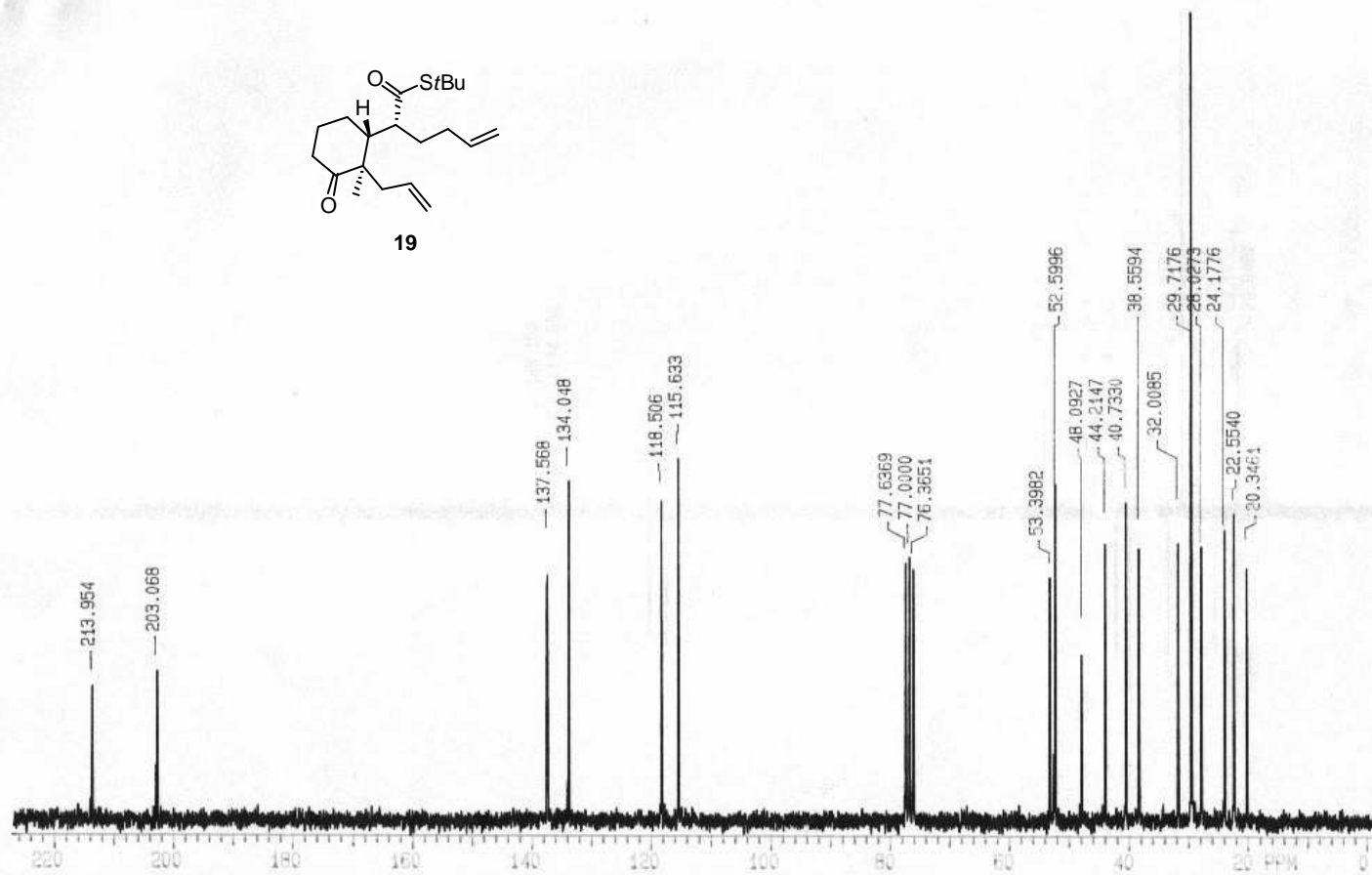
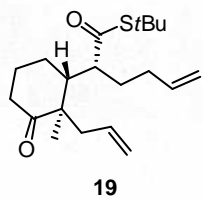
14





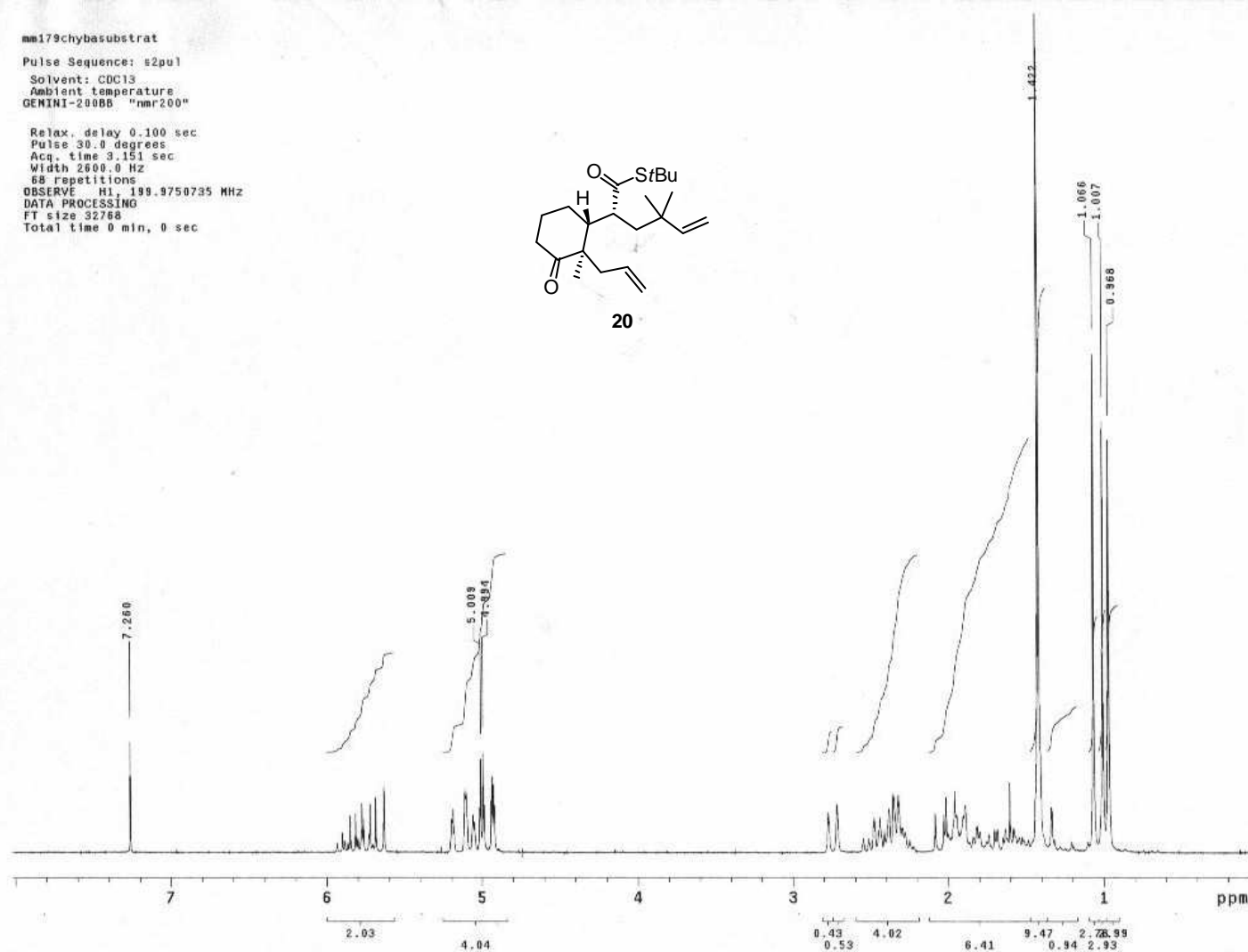
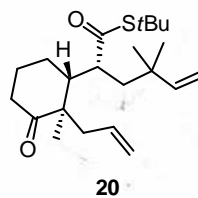






nm179chybasubstrat
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
GENI1-200BB "nmr200"

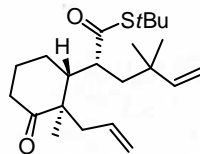
Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 3.151 sec
Width 2600.0 Hz
68 repetitions
OBSERVE H1, 199.9750735 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 0 sec



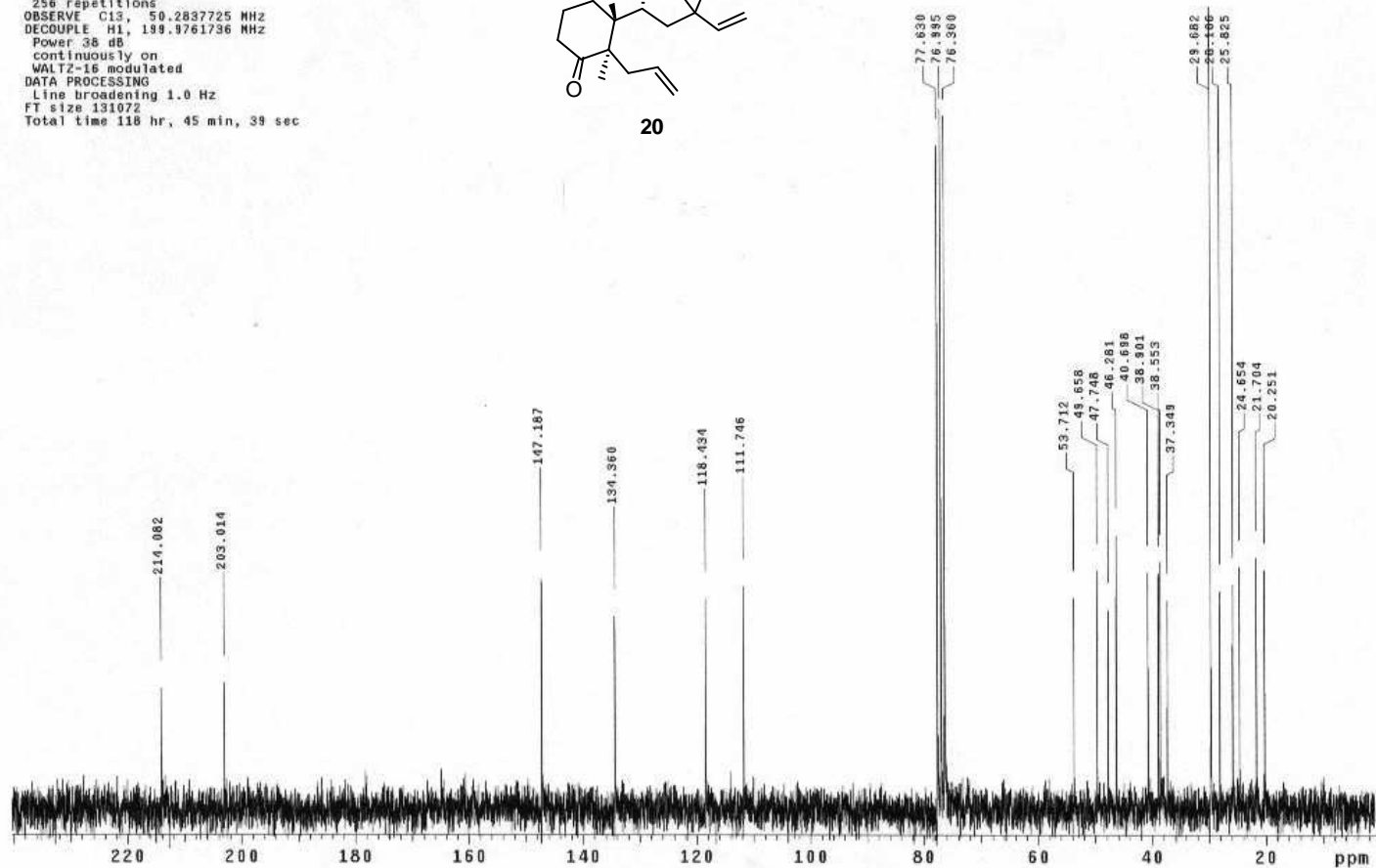
nm173

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GERINI-2008B "nmr200"

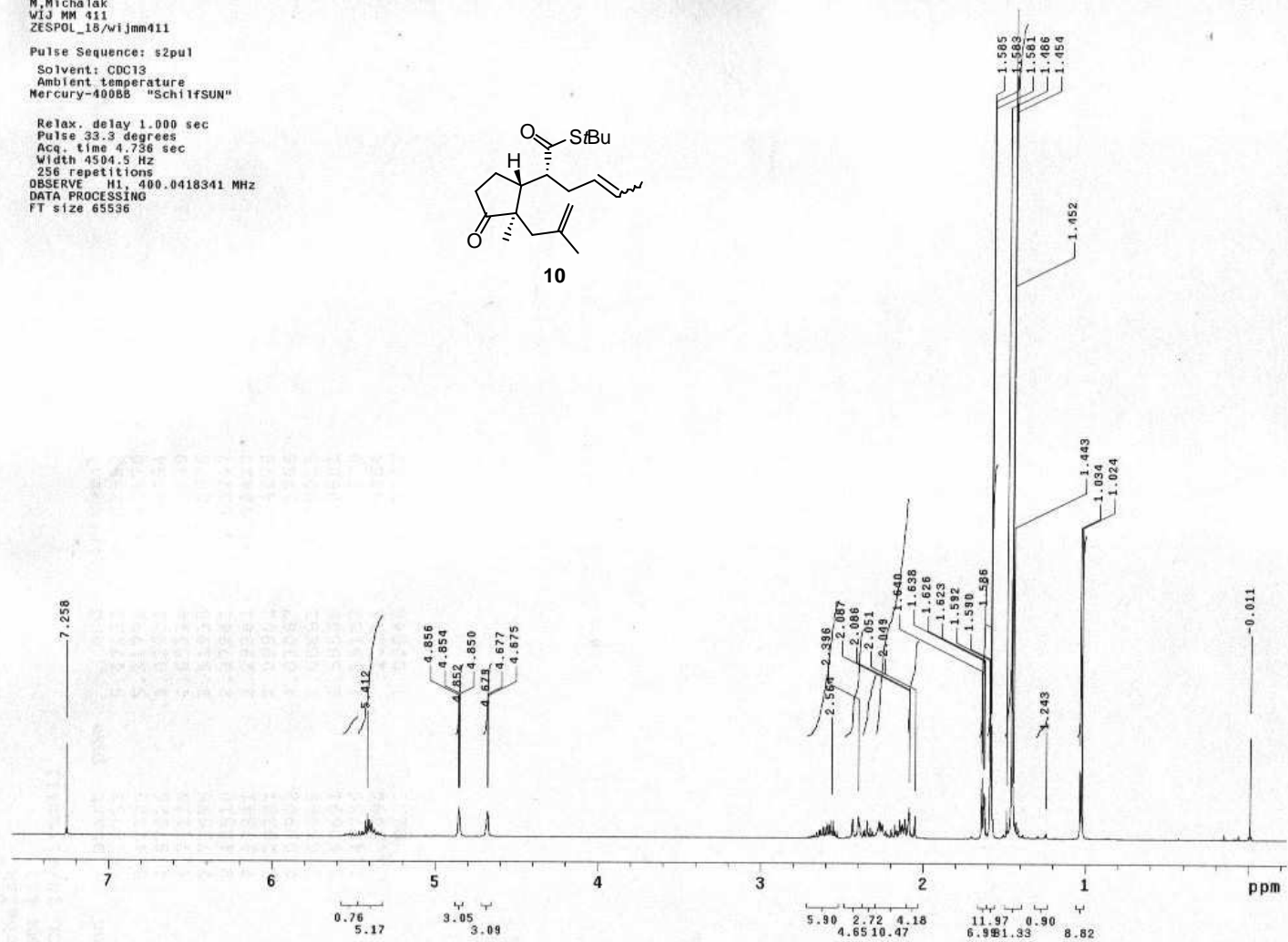
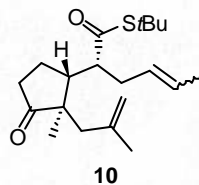
Relax. delay 2.000 sec
Pulse 48.0 degrees
Acq. time 2.000 sec
Width 15500.0 Hz
256 repetitions
OBSERVE C13, 50.2837725 MHz
DECOUPLE H1, 199.9761736 MHz
Power 38 dB
continuously On
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 118 hr, 45 min, 39 sec



20

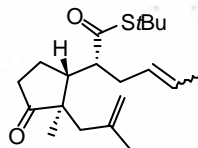


M. Michalak
VIJ MM 411
ZESPOL_18/vijmm411
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "SchiffSUN"
Relax. delay 1.000 sec
Pulse 33.3 degrees
Acq. time 4.736 sec
Width 4504.5 Hz
256 repetitions
OBSERVE H1, 400.0418341 MHz
DATA PROCESSING
FT size 65536

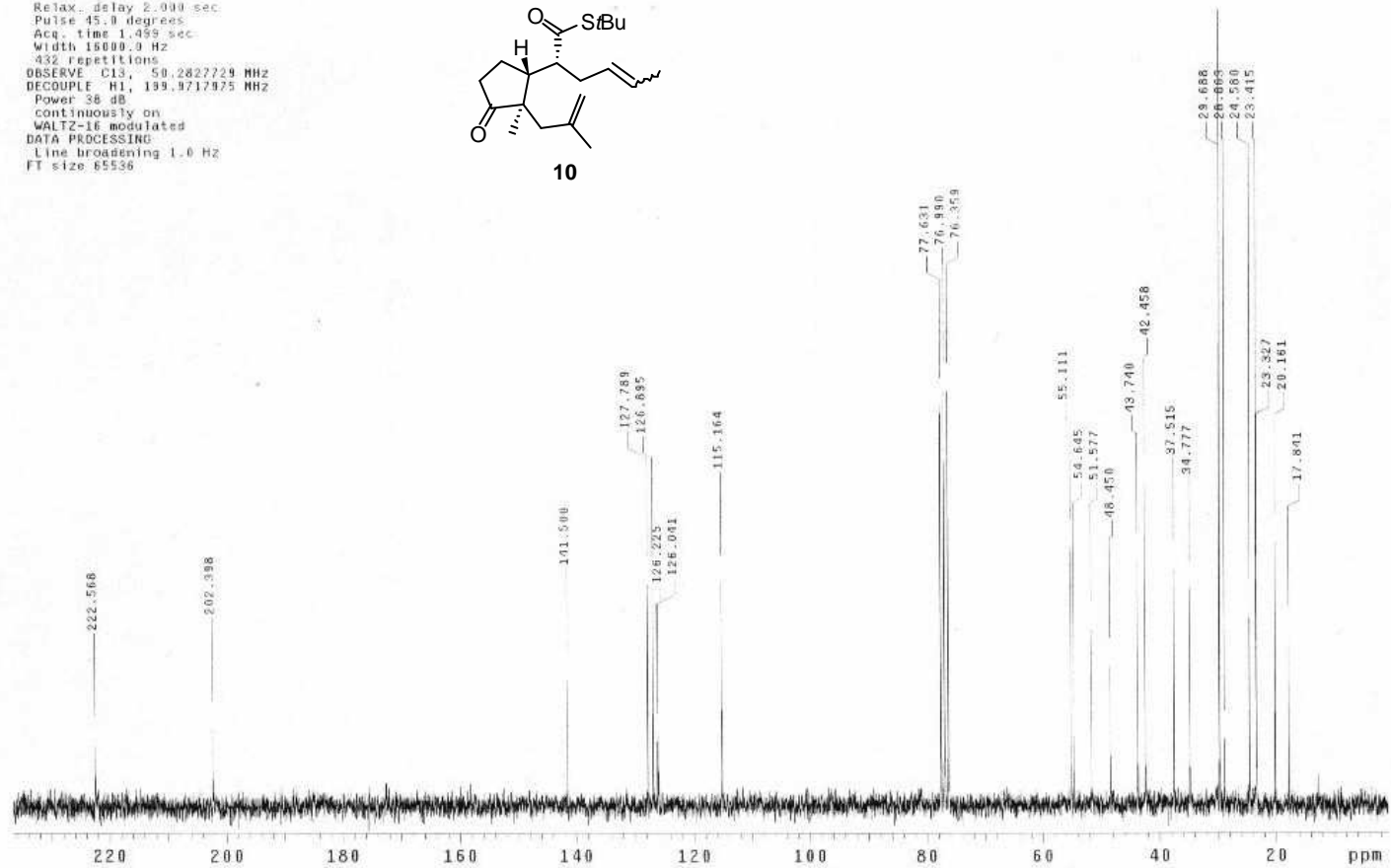


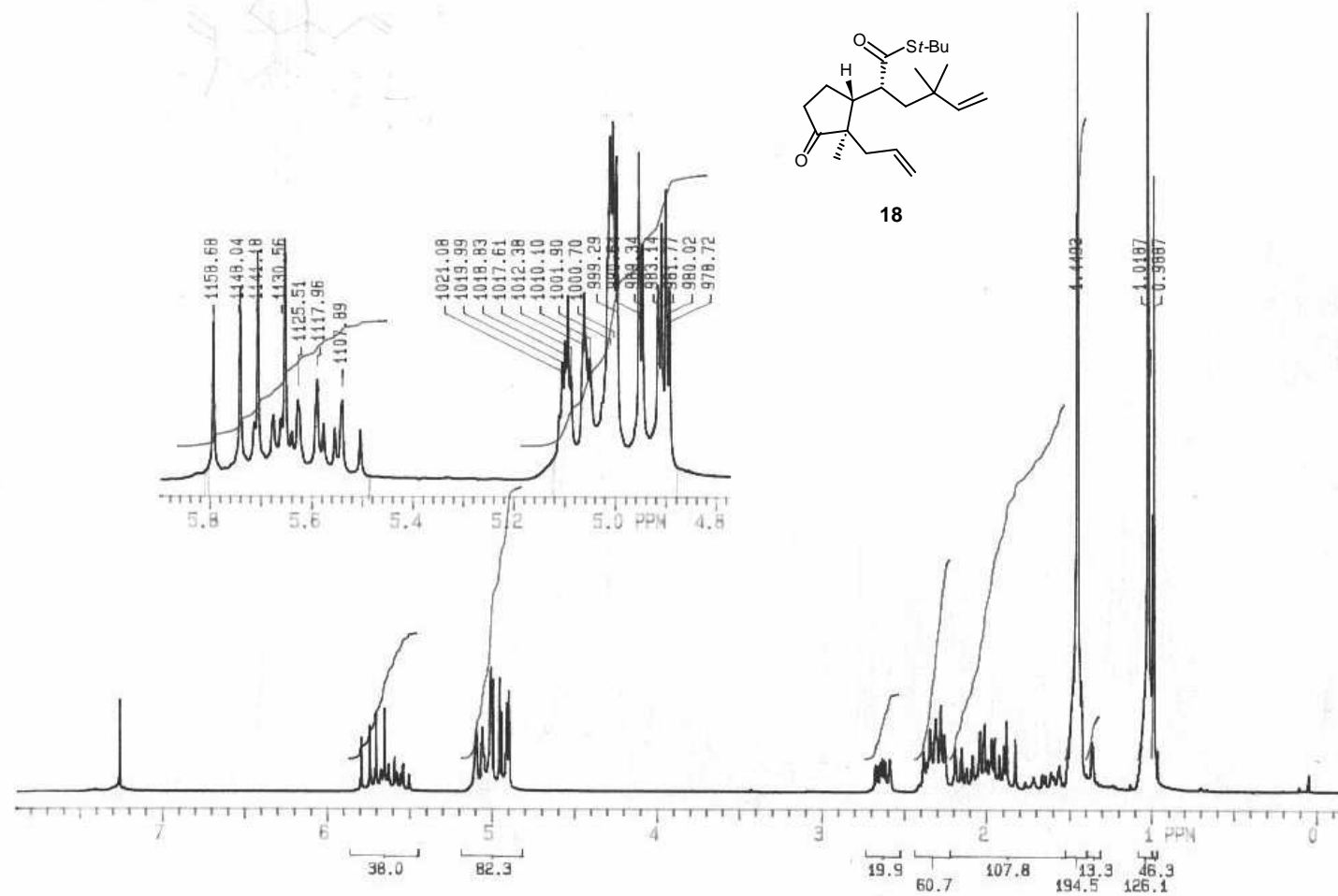
mm378wyzs2yrf-c
Pulse Sequence: szpul
Solvent: CDCl3
Ambient temperature
GENI1-200BB "nmr200"

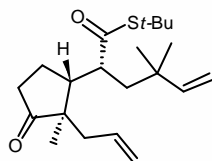
Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.459 sec
Width 16000.0 Hz
432 repetitions
OBSERVE C13, 50.2827729 MHz
DECOUPLE H1, 199.9717975 MHz
Power 38 dB
Continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536



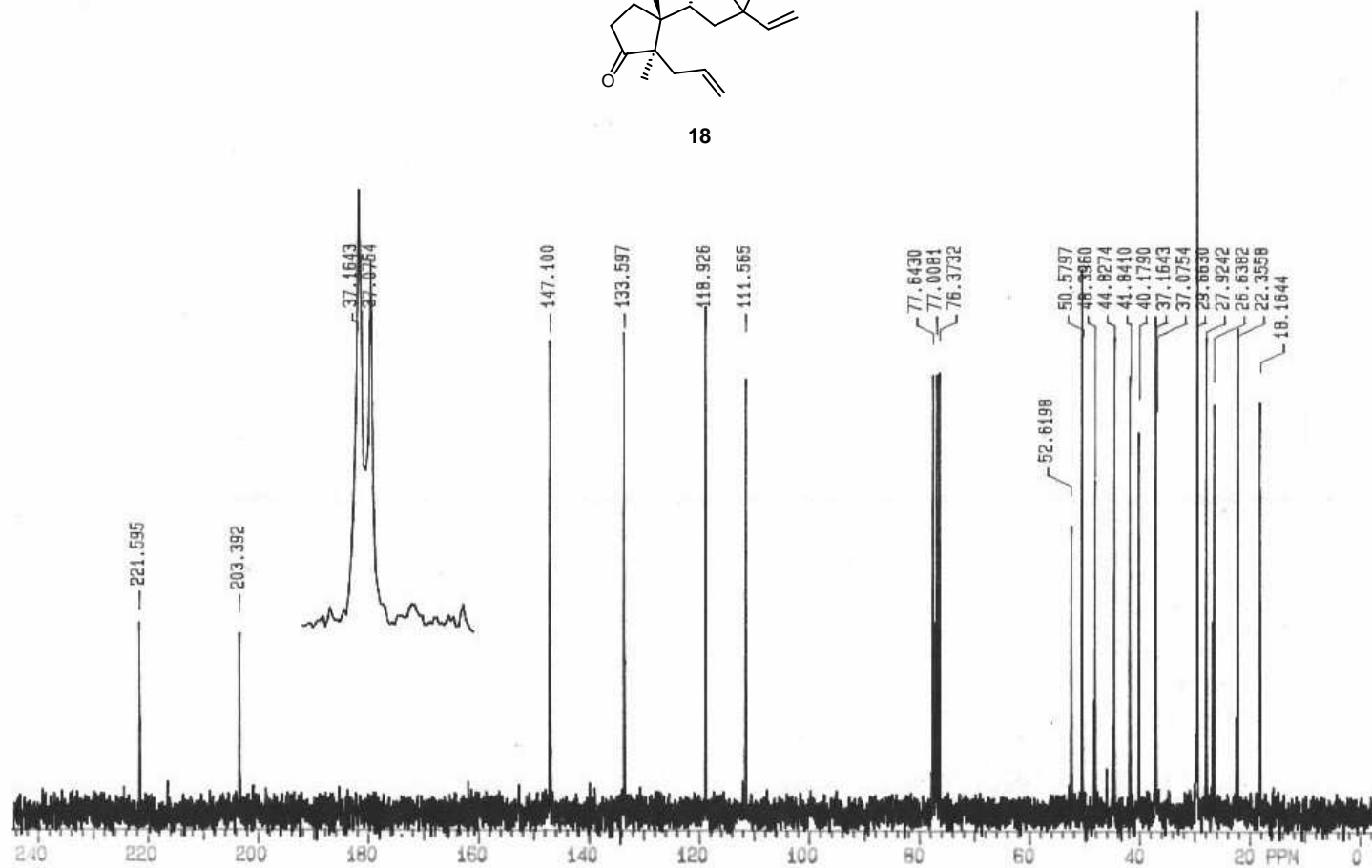
10







18

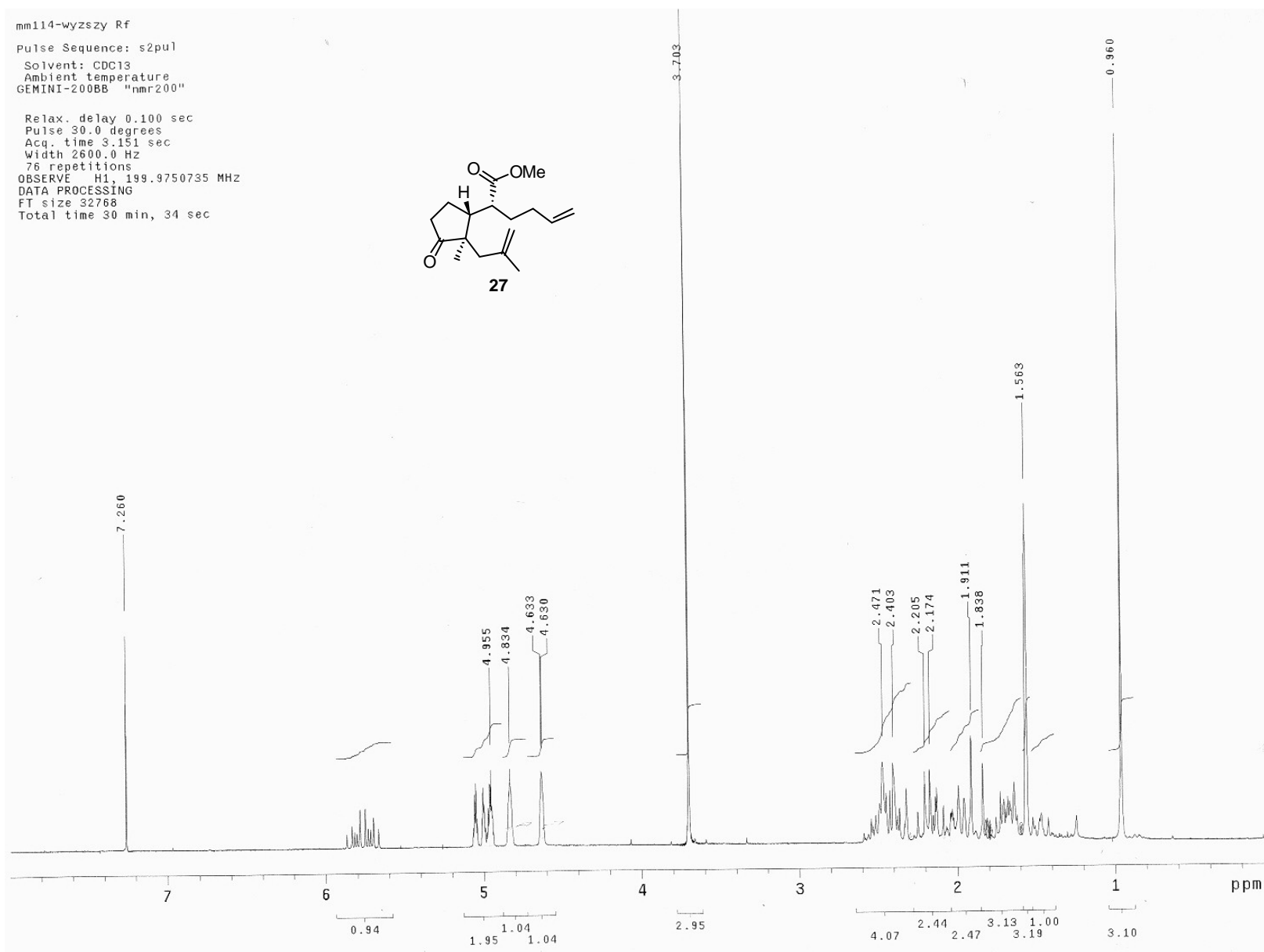
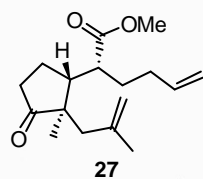


mm114-wyzszy Rf

Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature
GEMINI-200BB "nmr200"

Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 3.151 sec
Width 2600.0 Hz
76 repetitions
OBSERVE H1, 199.9750735 MHz
DATA PROCESSING
FT size 32768
Total time 30 min, 34 sec



mm114-wyzszyRf

Pulse Sequence: s2pu1

Solvent: CDC13

Ambient temperature

GEMINI-200BB "nmr200"

Relax. delay 2.000 sec

Pulse 48.0 degrees

Acq. time 2.000 sec

Width 15500.0 Hz

512 repetitions

OBSERVE C13, 50.2837730 MHz

DECOUPLE H1, 199.9761736 MHz

Power 38 dB

continuously on

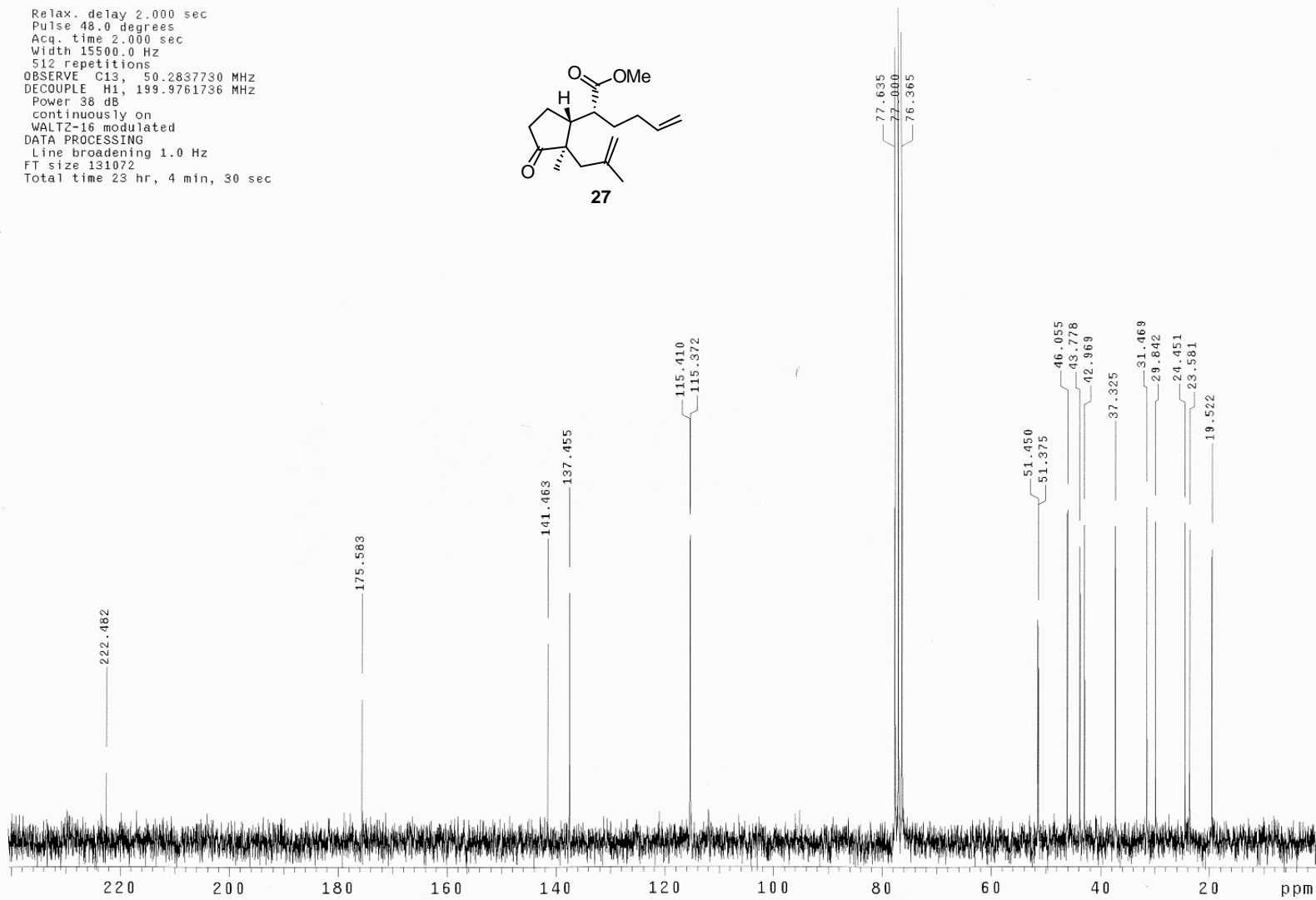
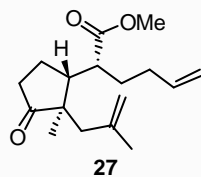
WALTZ-16 modulated

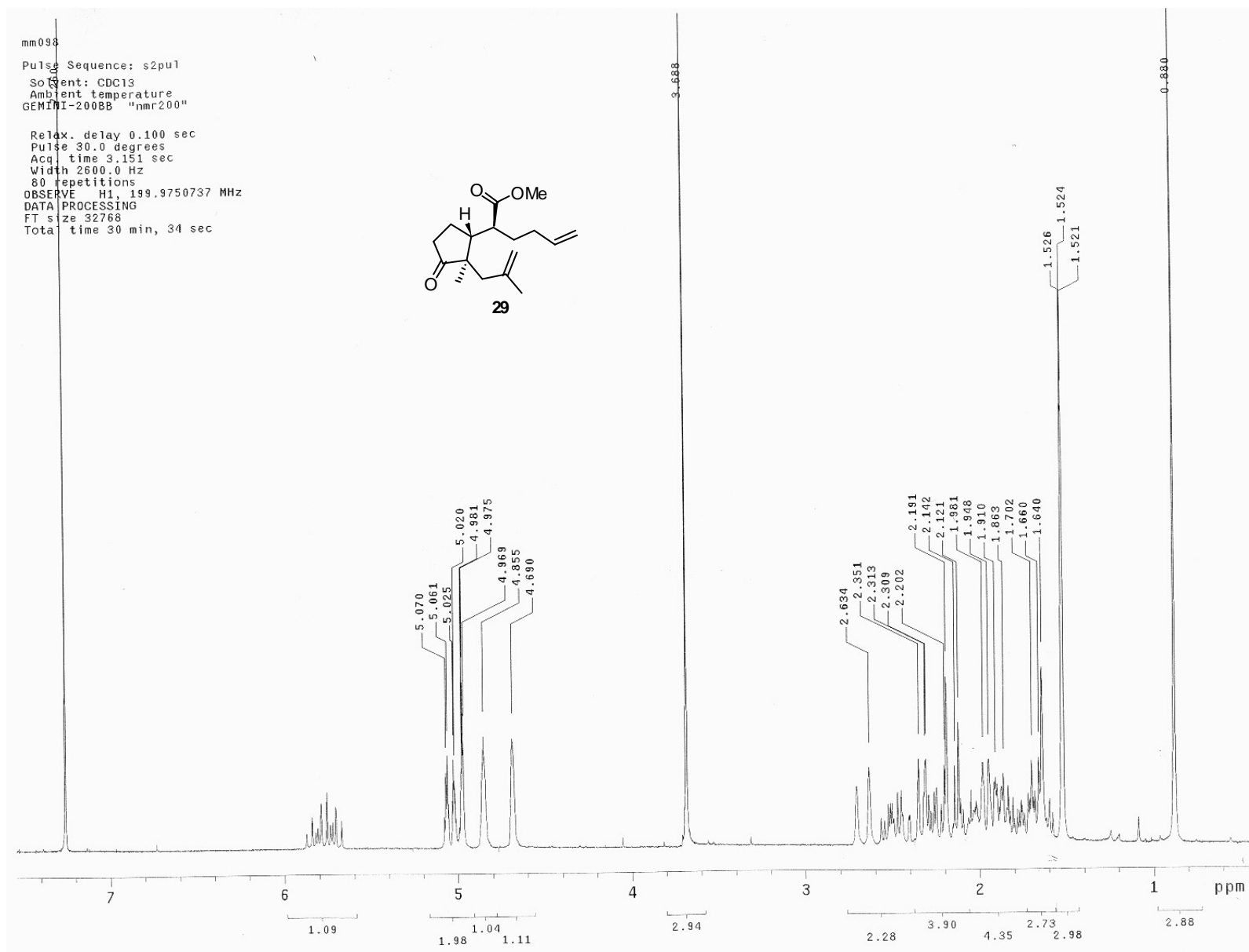
DATA PROCESSING

Line broadening 1.0 Hz

FT size 131072

Total time 23 hr, 4 min, 30 sec





114-nizszyRf

Pulse Sequence: s2pu1

Solvent: CDCl₃

Ambient temperature

GEMINI-200BB "nmr200"

Relax. delay 2.000 sec

Pulse 48.0 degrees

Acq. time 2.000 sec

Width 15500.0 Hz

416 repetitions

OBSERVE C13, 50.2837732 MHz

DECOUPLE H1, 199.9761736 MHz

Power 38 dB

continuously on

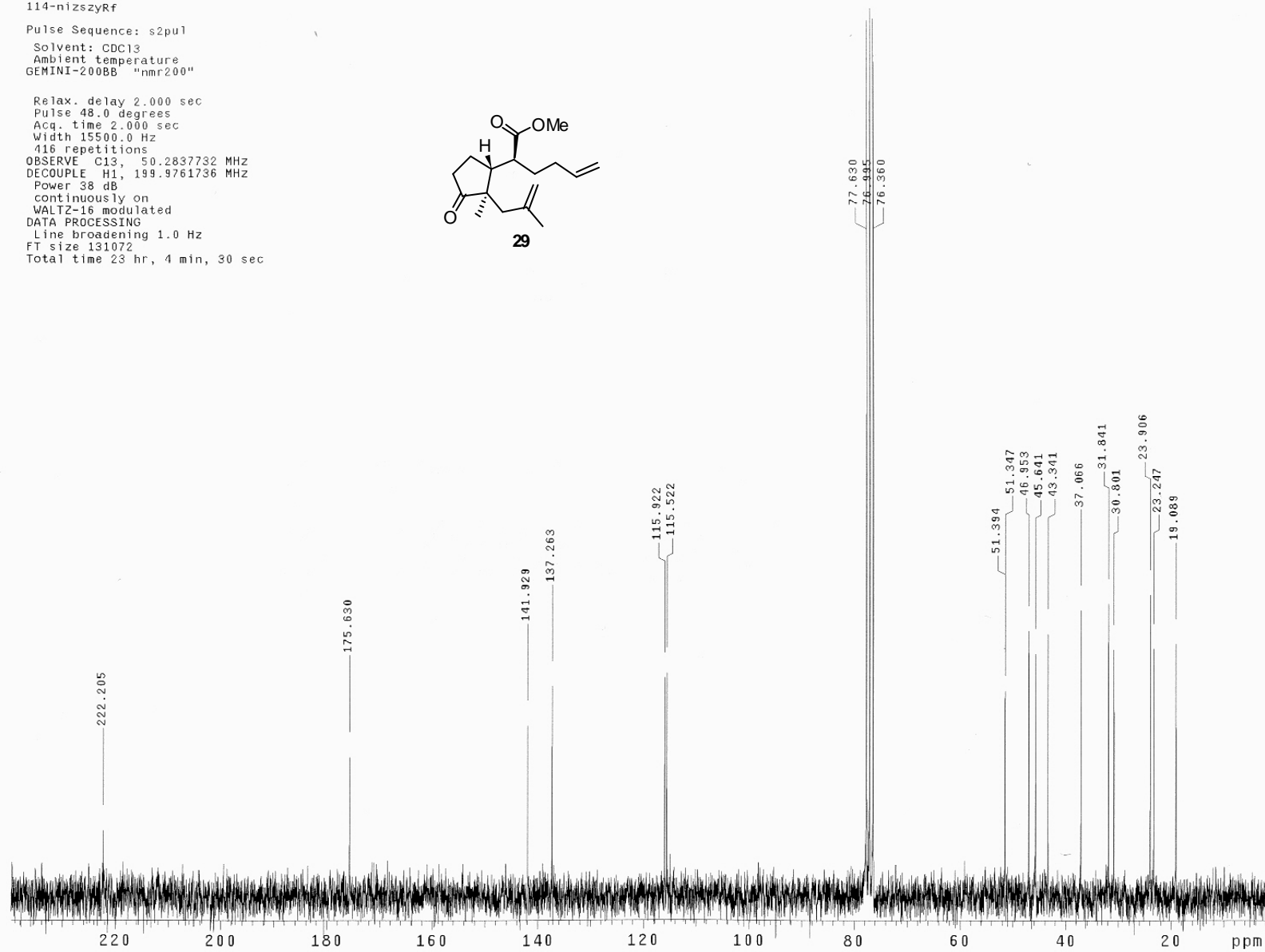
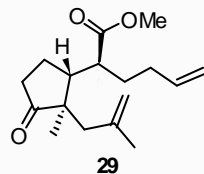
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

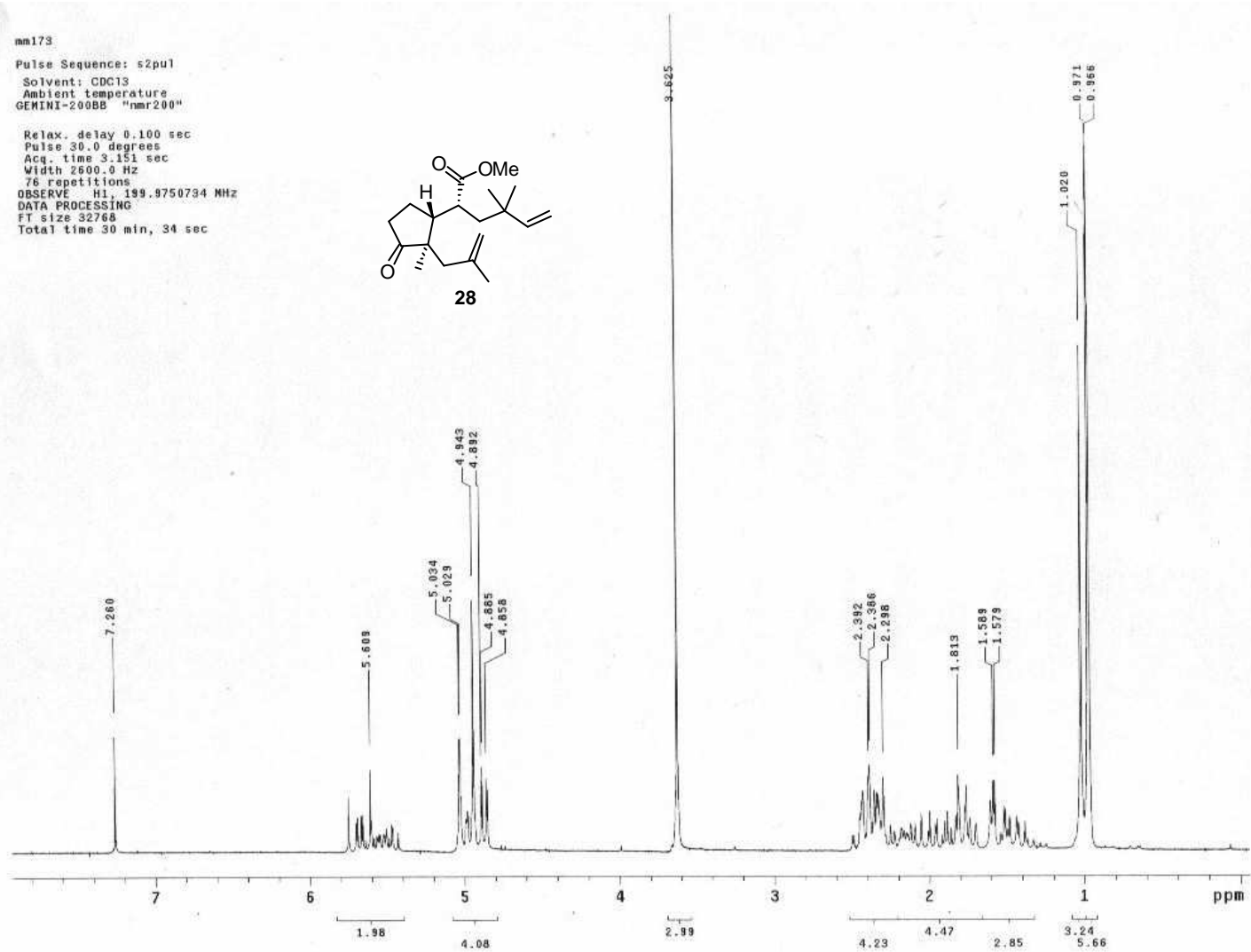
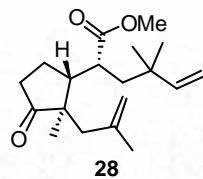
FT size 131072

Total time 23 hr, 4 min, 30 sec



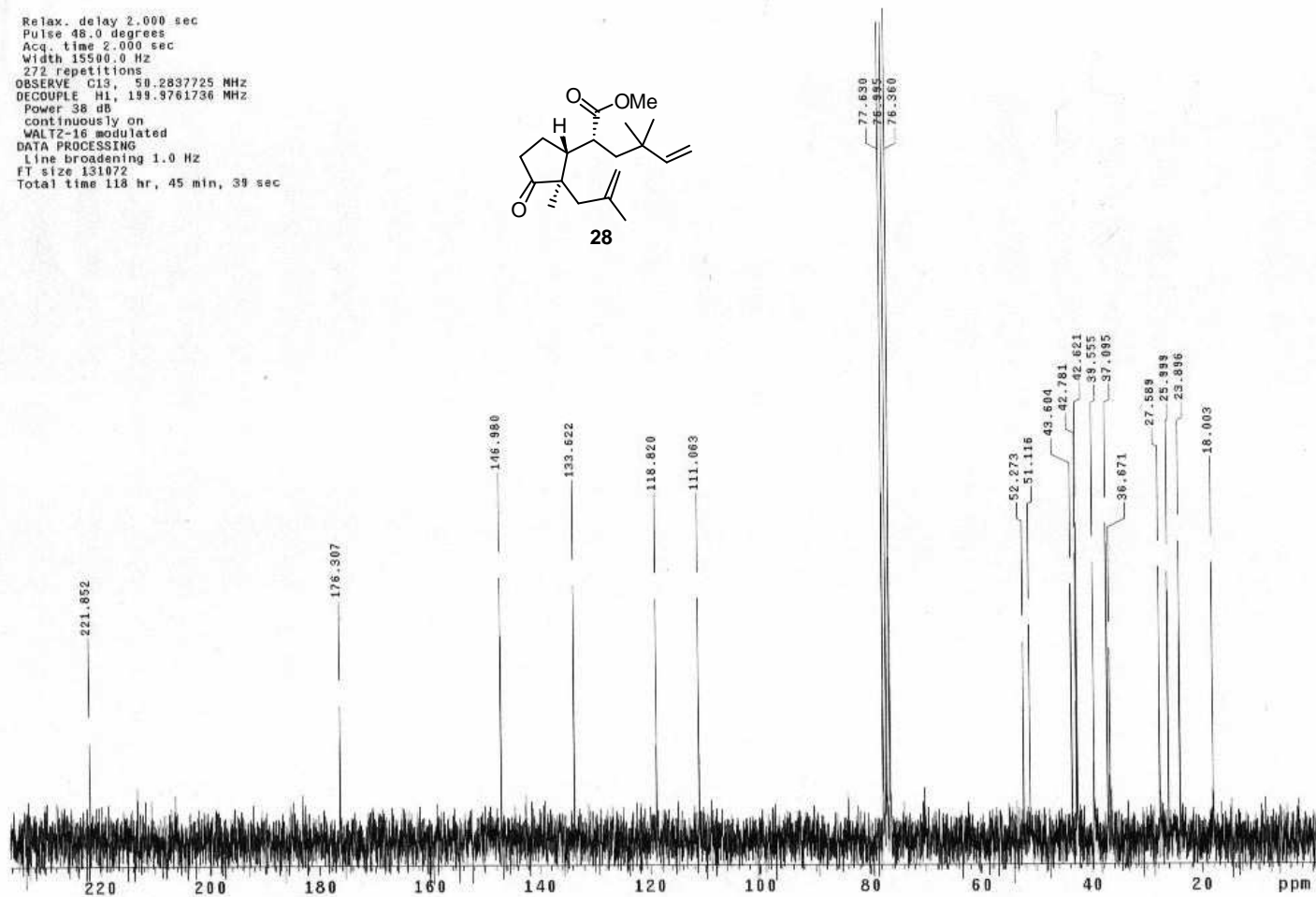
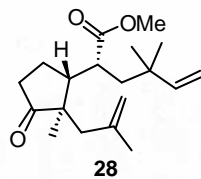
nm173
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
GEMINI-200BB "nmr200"

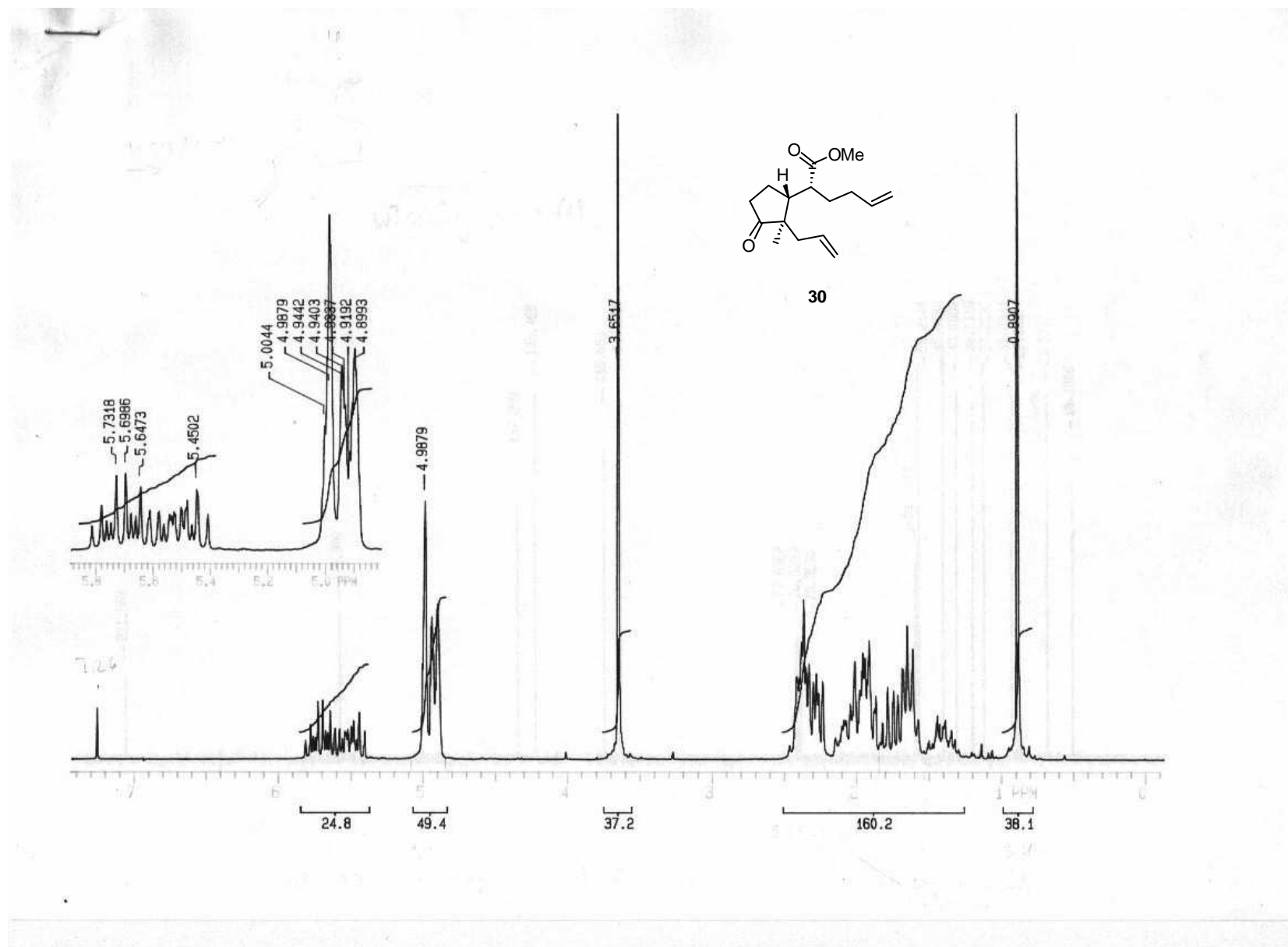
Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 3.151 sec
Width 2600.0 Hz
76 repetitions
OBSERVE H1, 199.9750734 MHz
DATA PROCESSING
FT size 32768
Total time 30 min, 34 sec

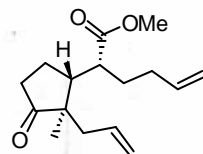


nm173
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
GEMINI-200BB "nmr200"

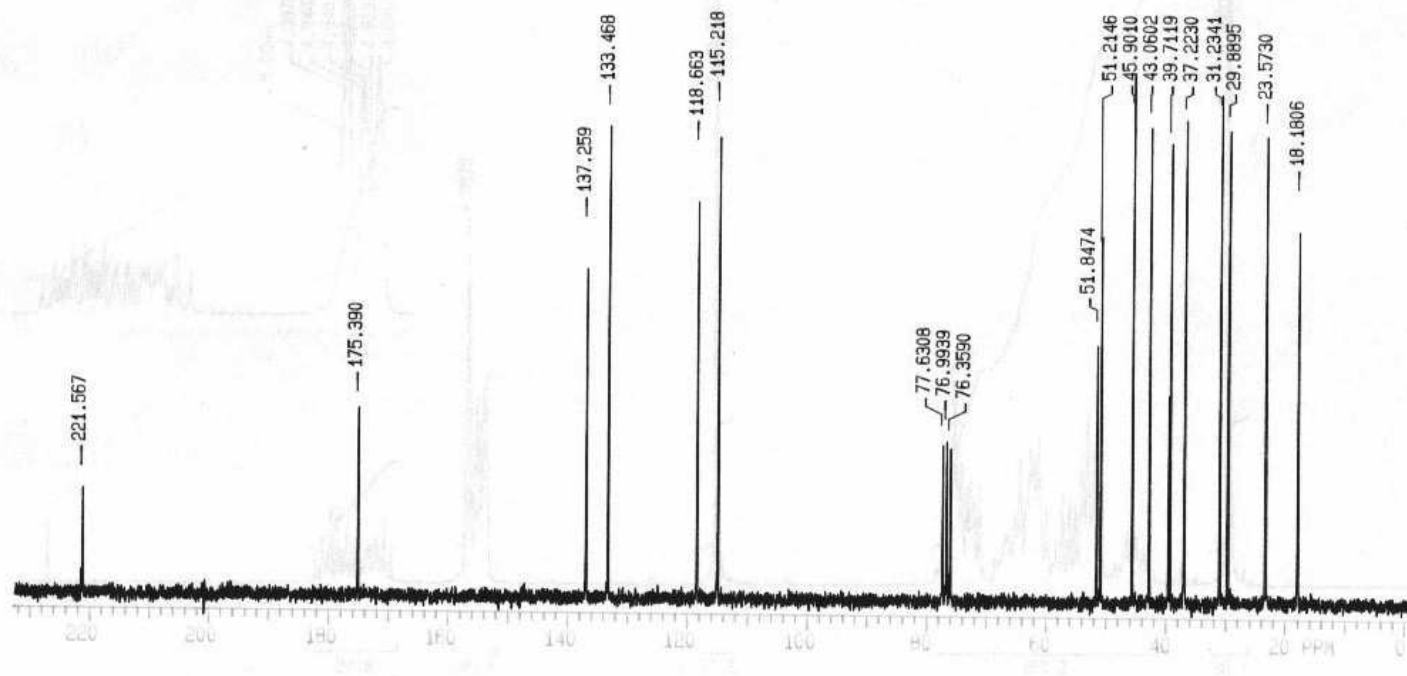
Relax. delay 2.000 sec
Pulse 48.0 degrees
Acq. time 2.000 sec
Width 15500.0 Hz
272 repetitions
OBSERVE C13, 50.2837725 MHz
DECOUPLE H1, 199.9761736 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 118 hr, 45 min, 39 sec

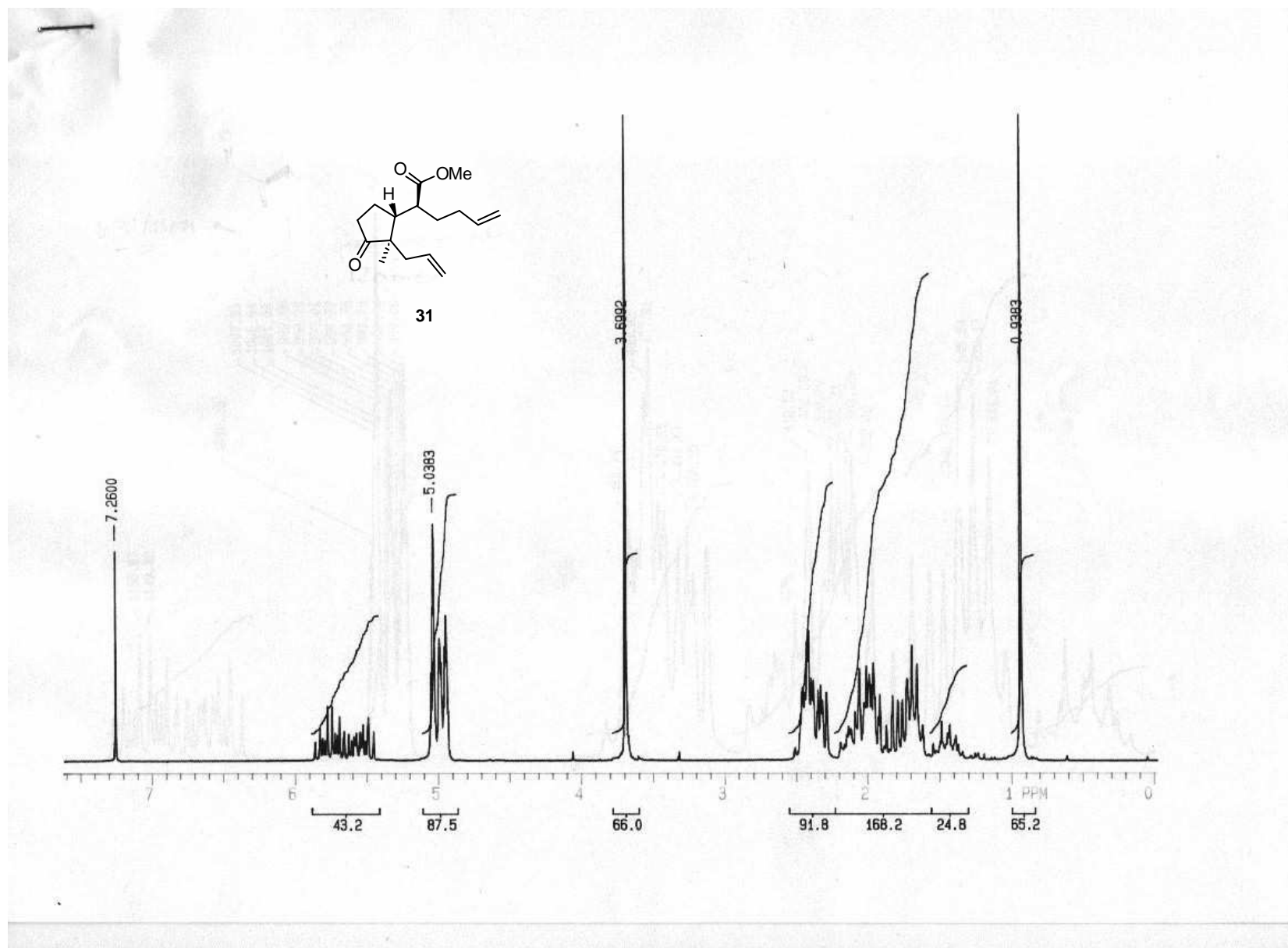


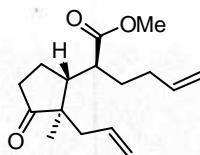




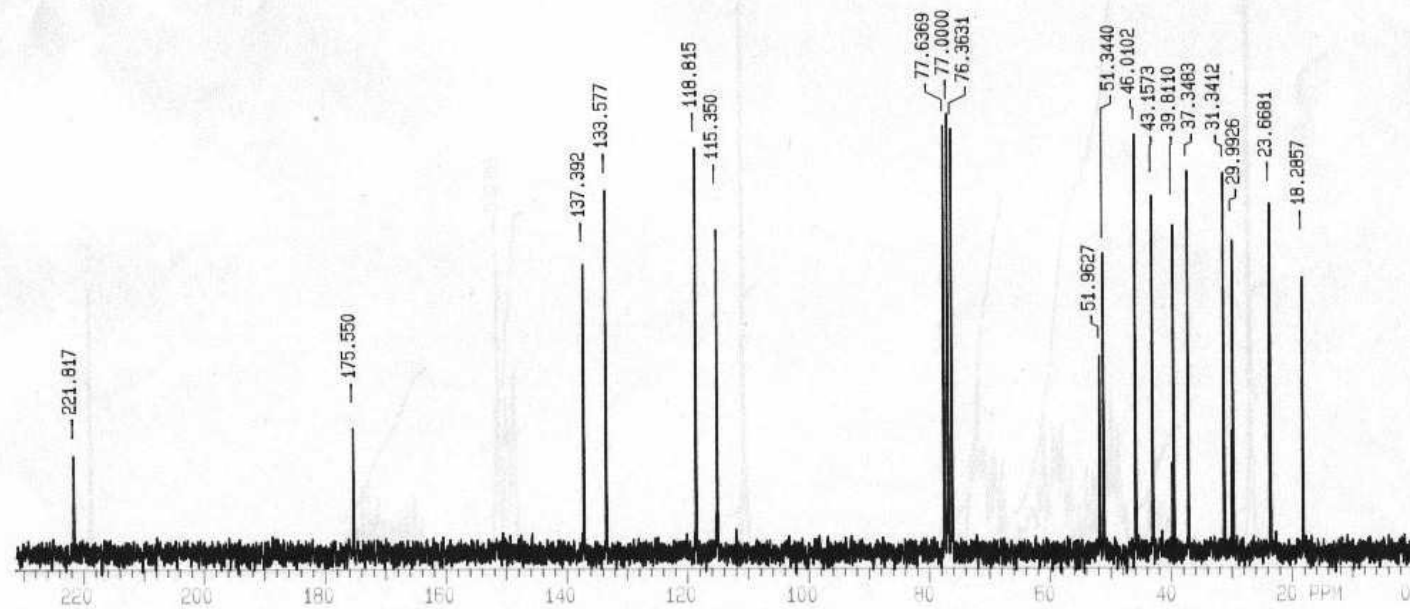
30

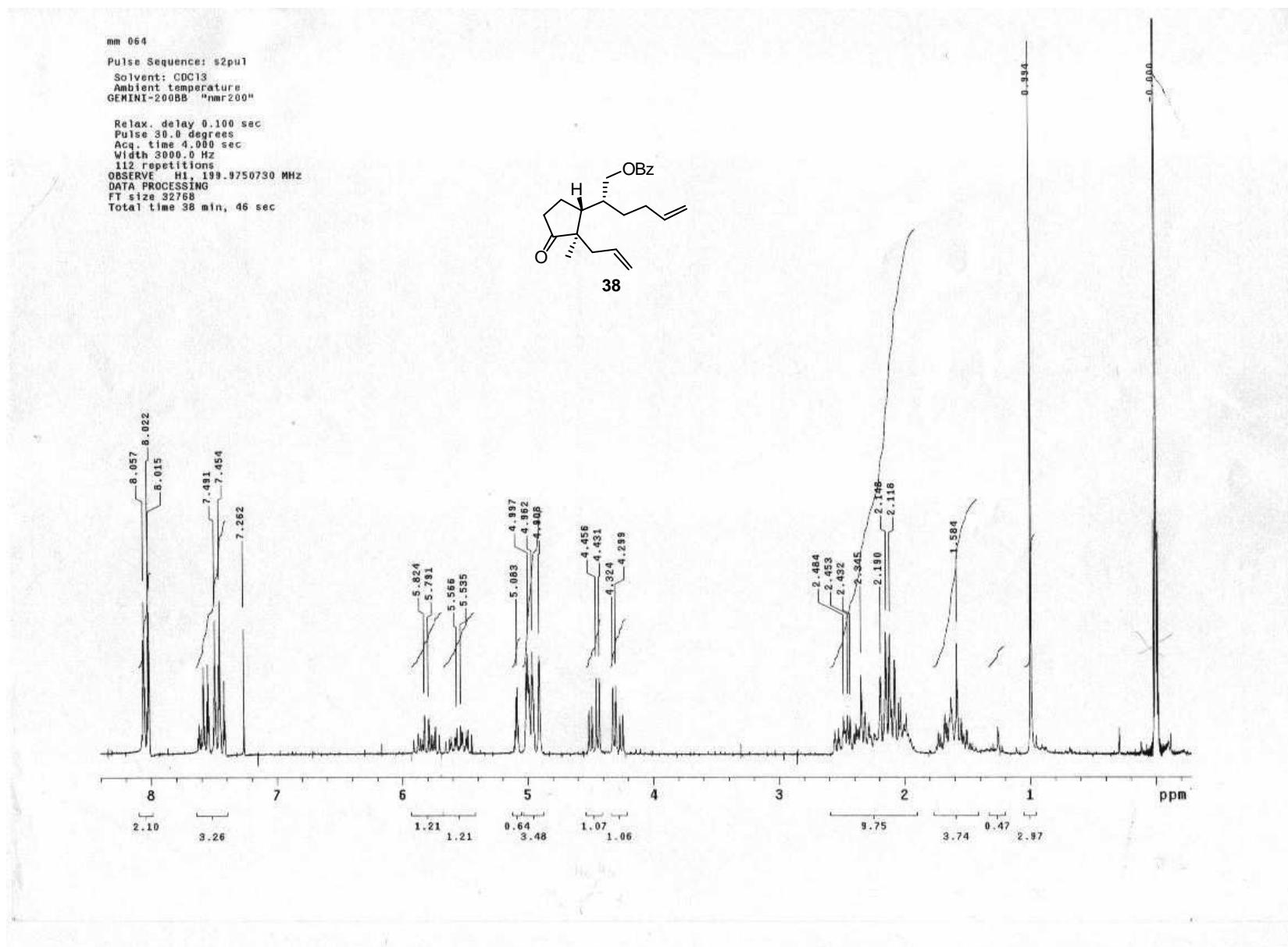


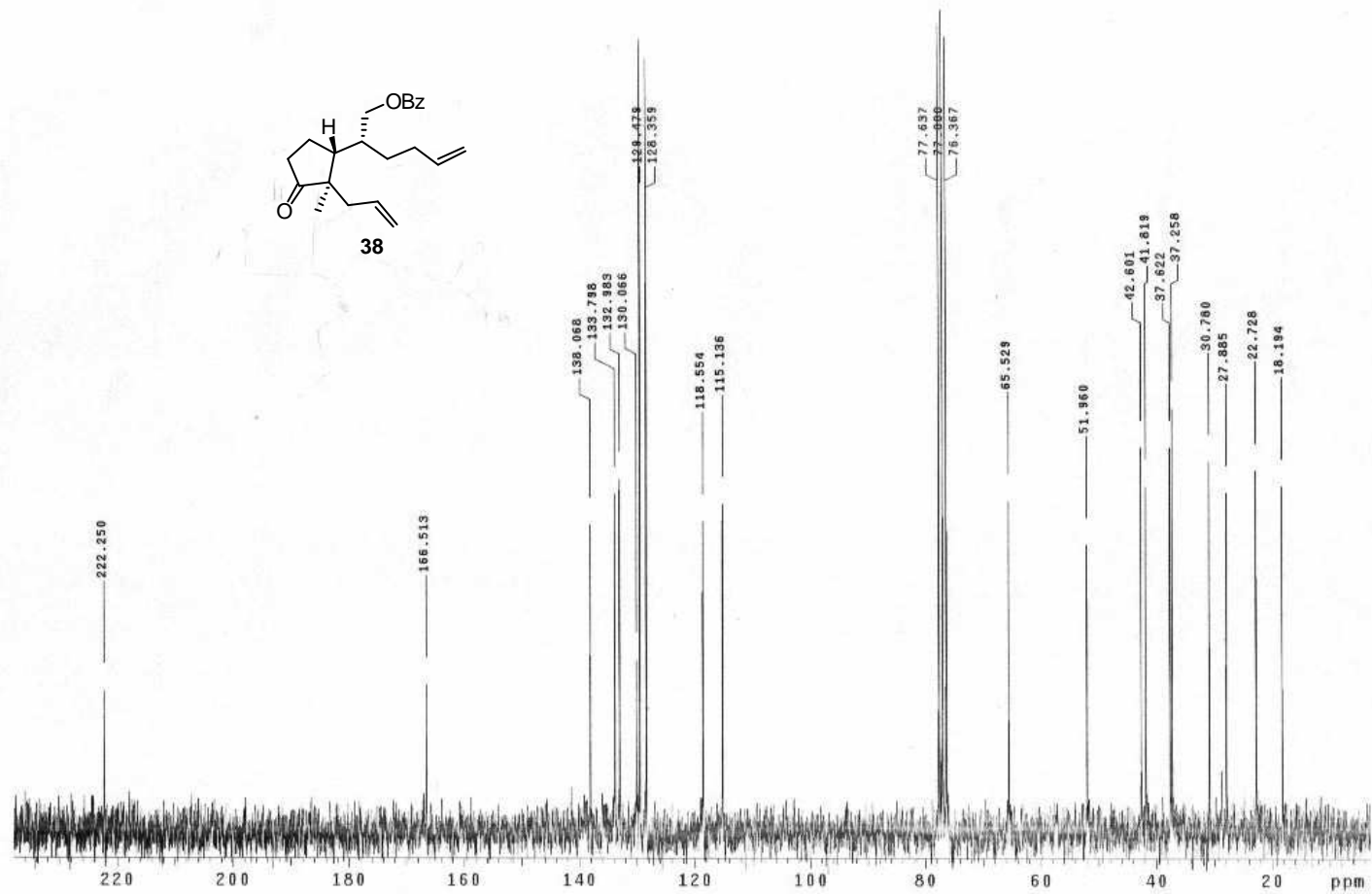
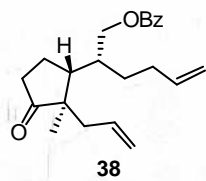




31

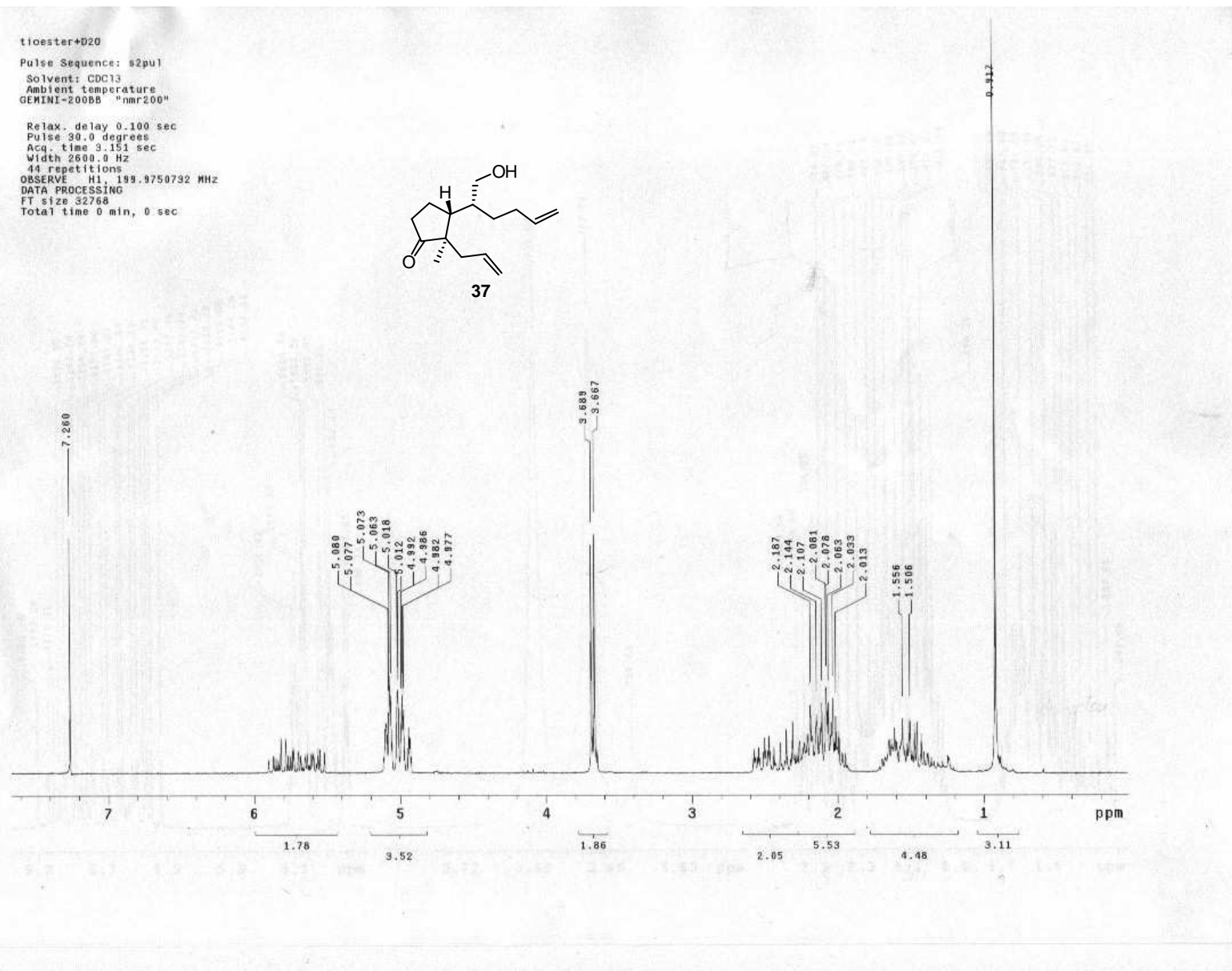
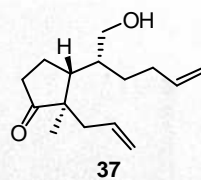


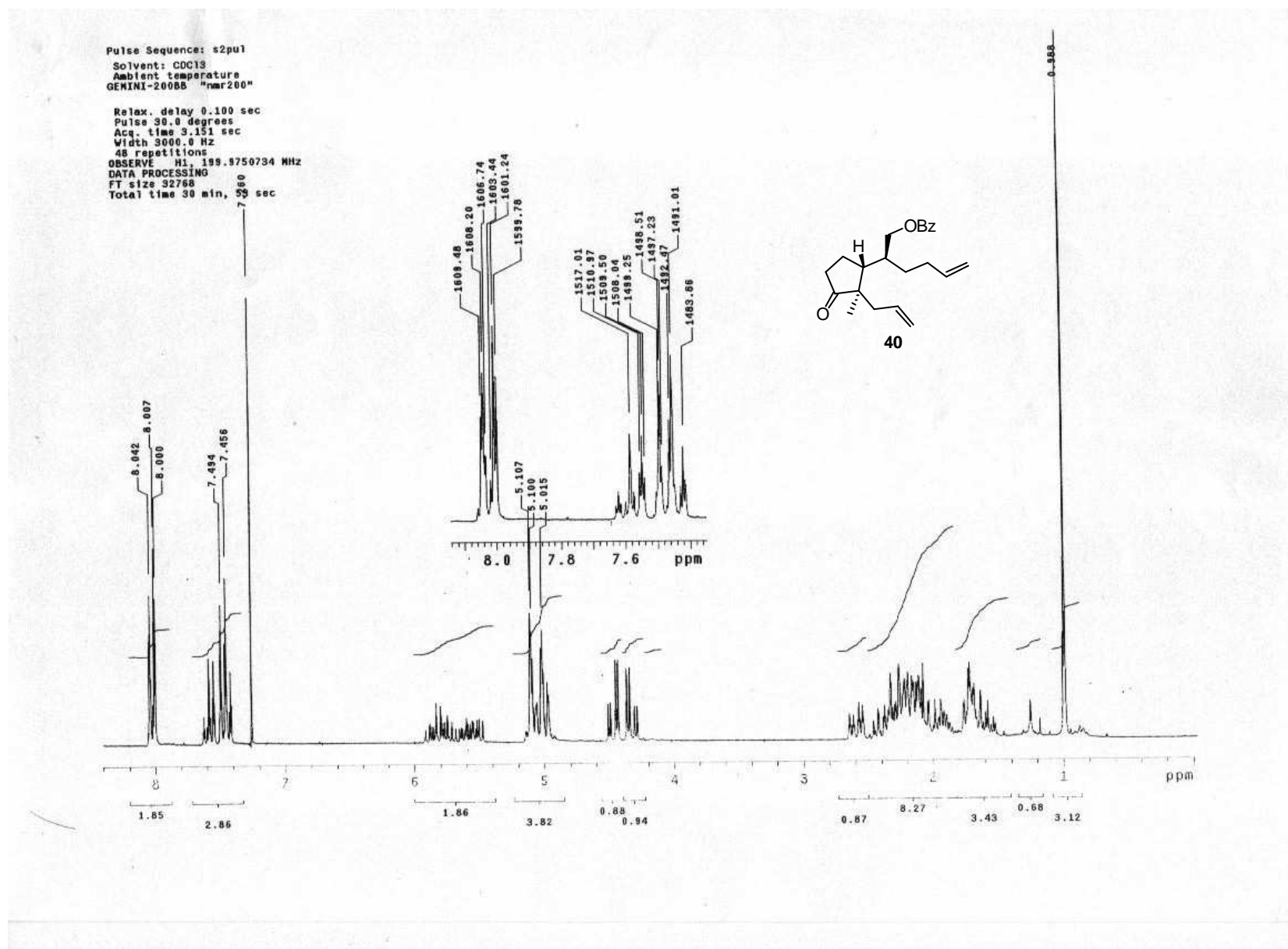




tioester+D2O
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-200BB "nmr200"

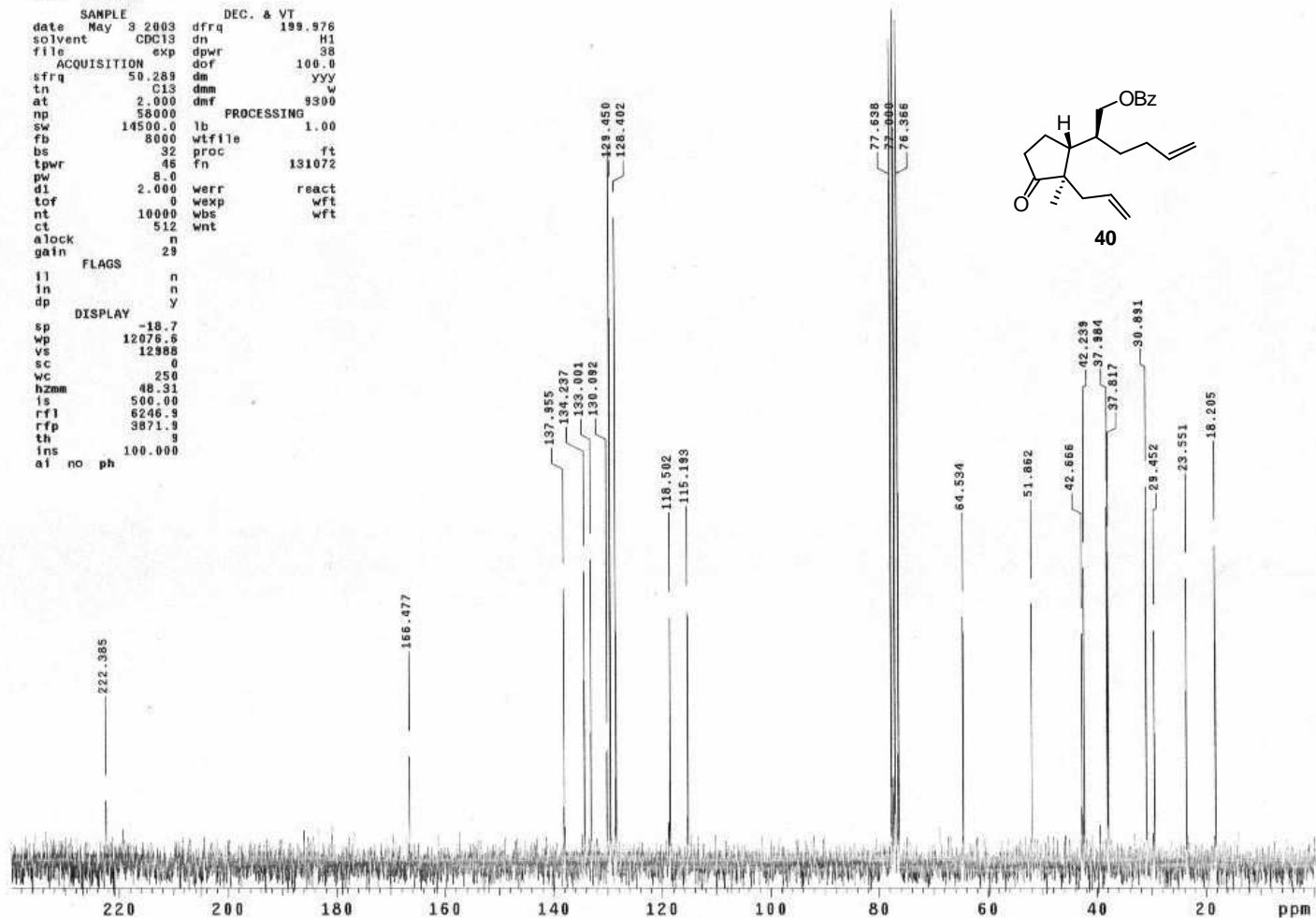
Relax. delay 0.100 sec
Pulse 30.0 degrees
Acq. time 3.151 sec
Width 2600.0 Hz
44 repetitions
OBSERVE H1, 199.9750732 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 0 sec

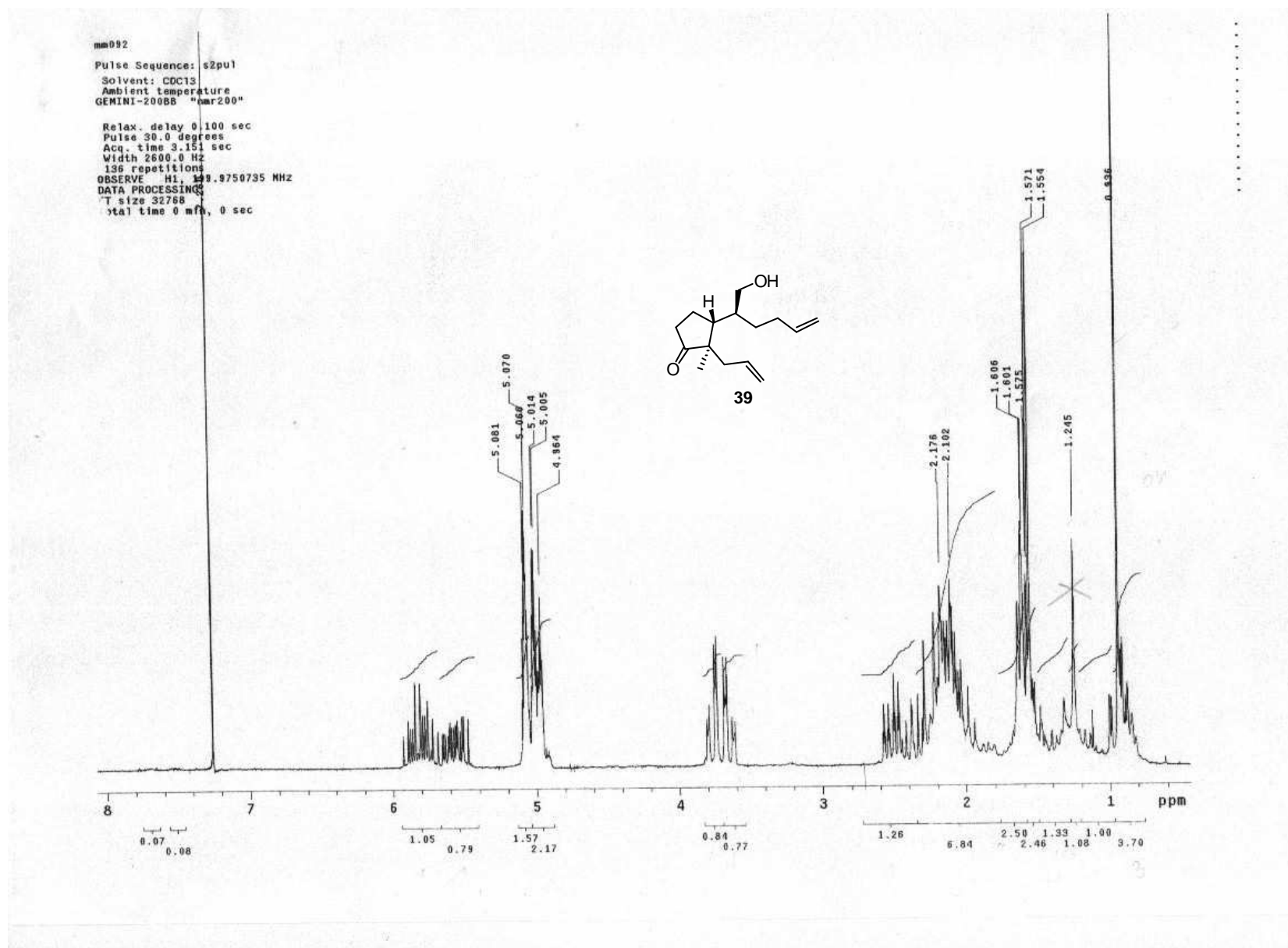


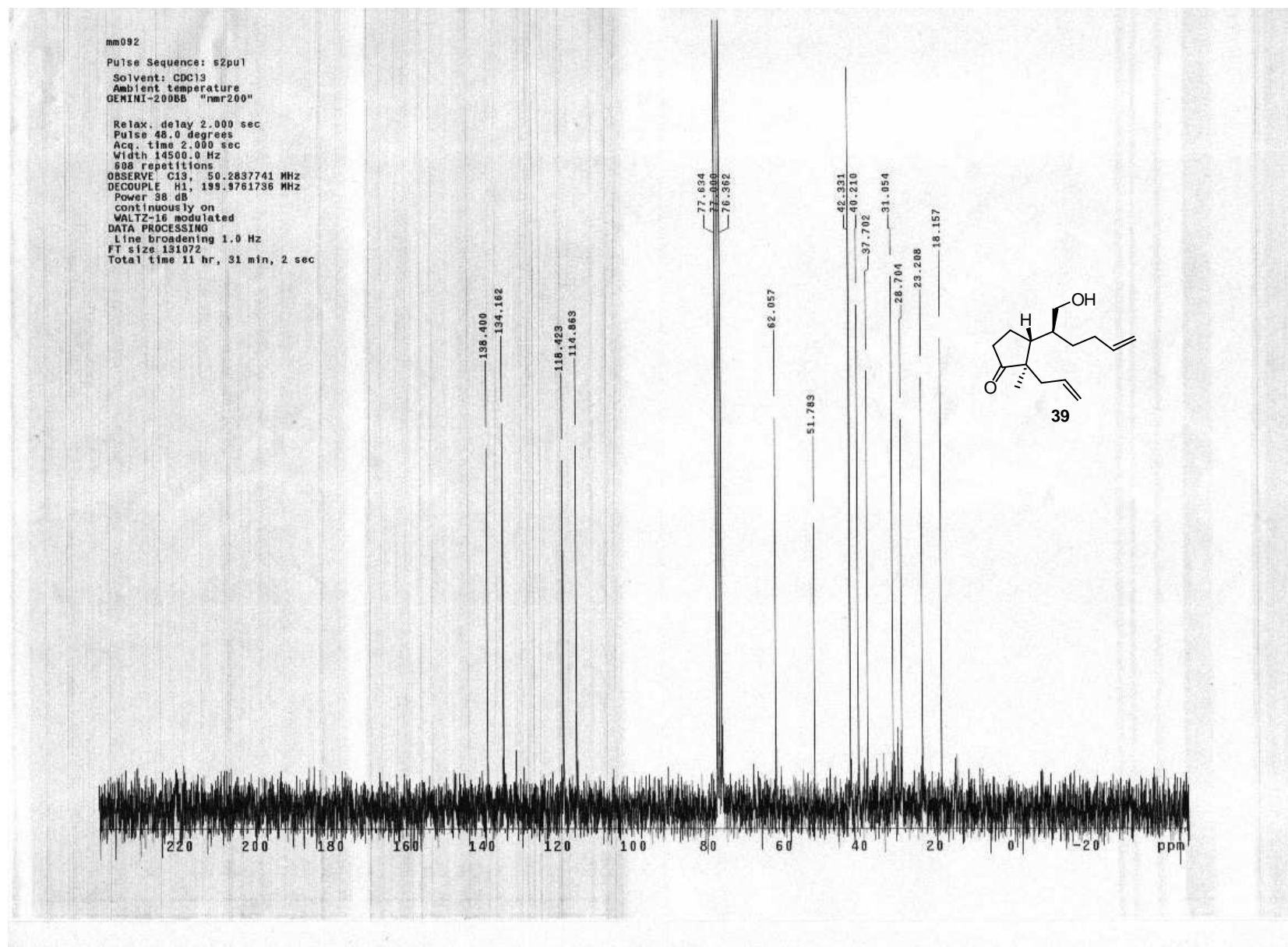


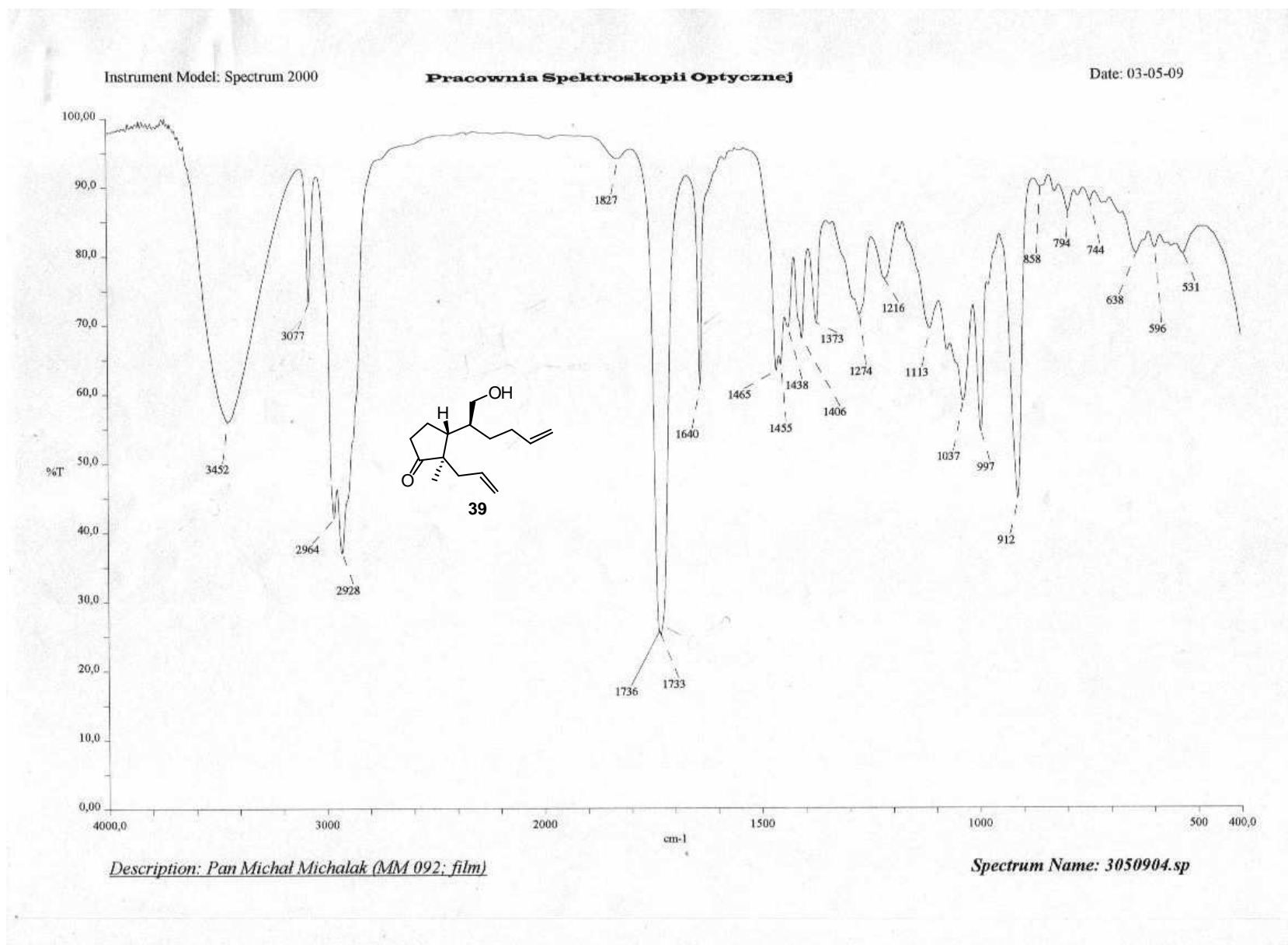
nm090
ppm090
Sequence: s2pu1
exp2 std13c

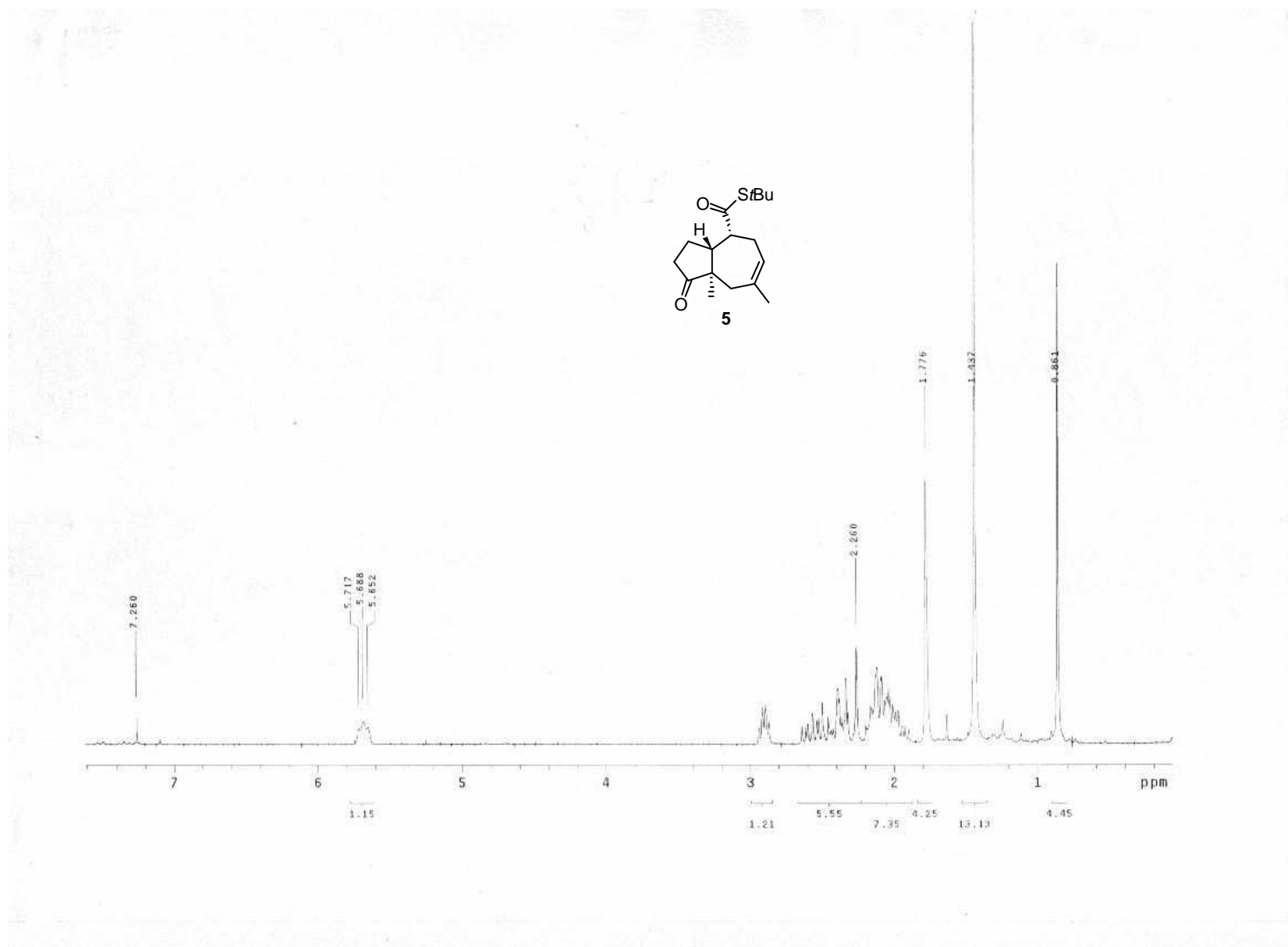
SAMPLE		DEC. & VT	
date	May 3 2003	dfrq	199.976
solvent	CDCl3	dn	H1
file	exp	dpwr	38
ACQUISITION		100.0	
sfrq	50.289	dm	yyy
tn	C13	dmm	w
at	2.000	dmf	9300
np	56000	PROCESSING	
sw	14500.0	lb	1.00
fb	8000	wtfile	
bs	32	proc	ft
tpwr	46	fn	131072
pw	8.0		
d1	2.000	werr	react
tof	0	wexp	wft
nt	10000	wbs	wft
ct	512	wnt	
alock	n		
gain	29		
FLAGS			
ll	n		
ln	n		
dp	y		
DISPLAY			
sp	-18.7		
wp	12076.6		
vs	12988		
sc	0		
wc	250		
hzm	48.31		
is	500.00		
rfl	6246.9		
rff	3871.8		
th	9		
ins	100.000		
ai	no	ph	





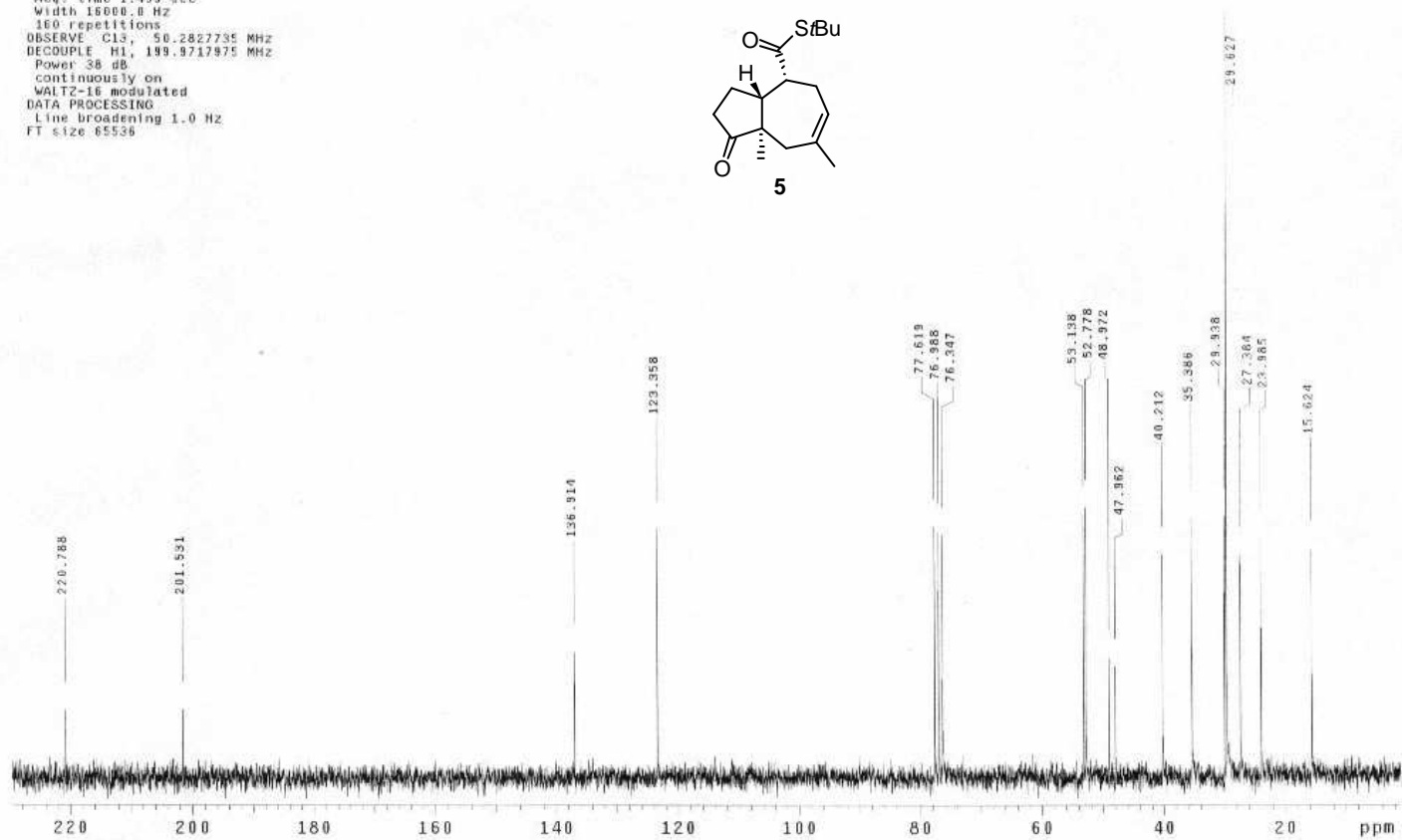
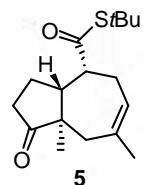






mm370wyzs2yrf-c M1406
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-200EB "nmr200"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.499 sec
Width 16000.0 Hz
160 repetitions
OBSERVE C13, 50.2827735 MHz
DECOUPLE H1, 199.9717975 MHz
Power 38 dB
Continuously On
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536



mm335hwyzsyrf

Pulse Sequence: s2pu1

Solvent: CDC13

Ambient temperature

GEMINI-200BB "nmr200"

Relax. delay 0.100 sec

Pulse 56.2 degrees

Acq. time 4.000 sec

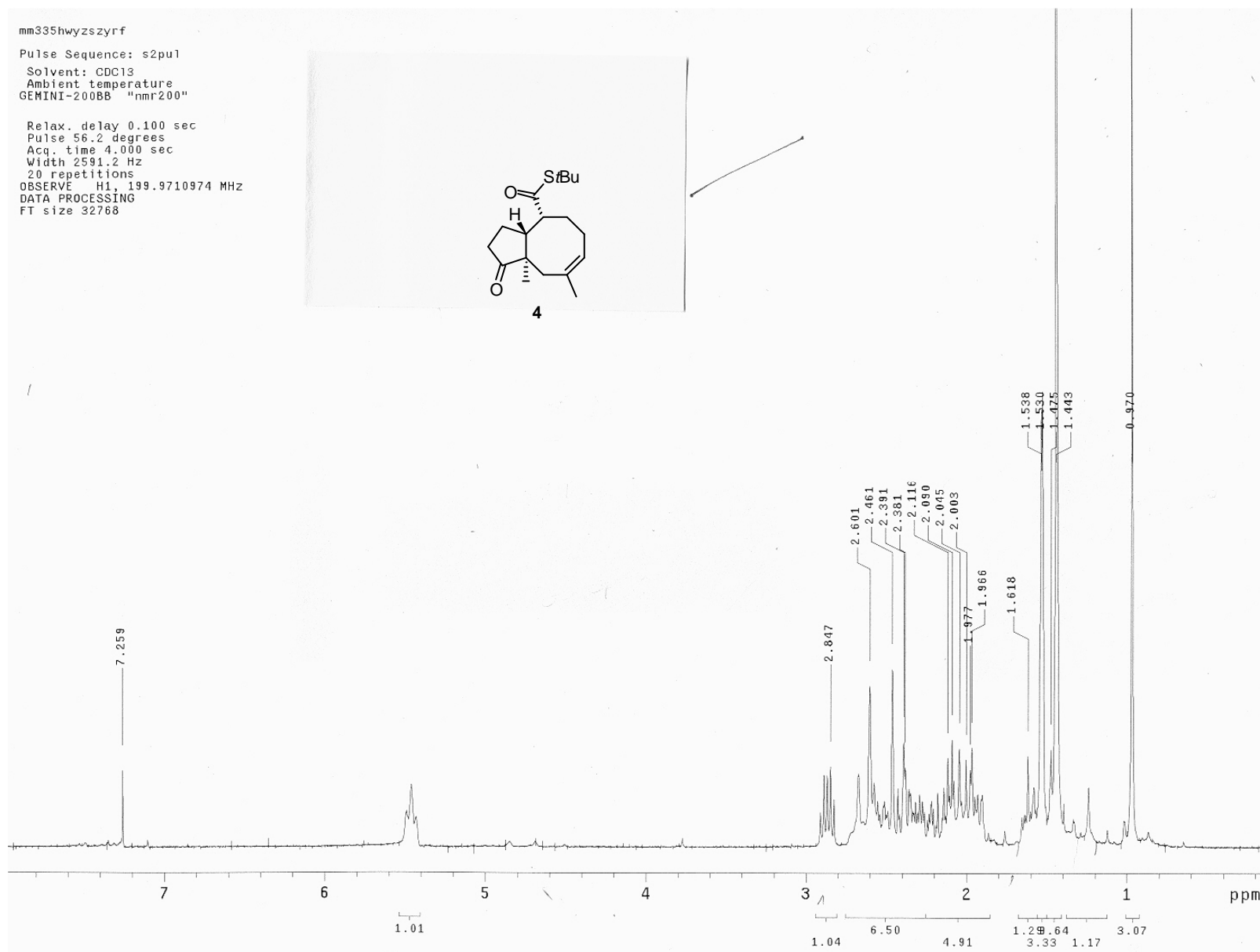
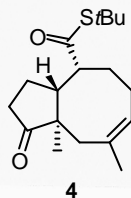
Width 2591.2 Hz

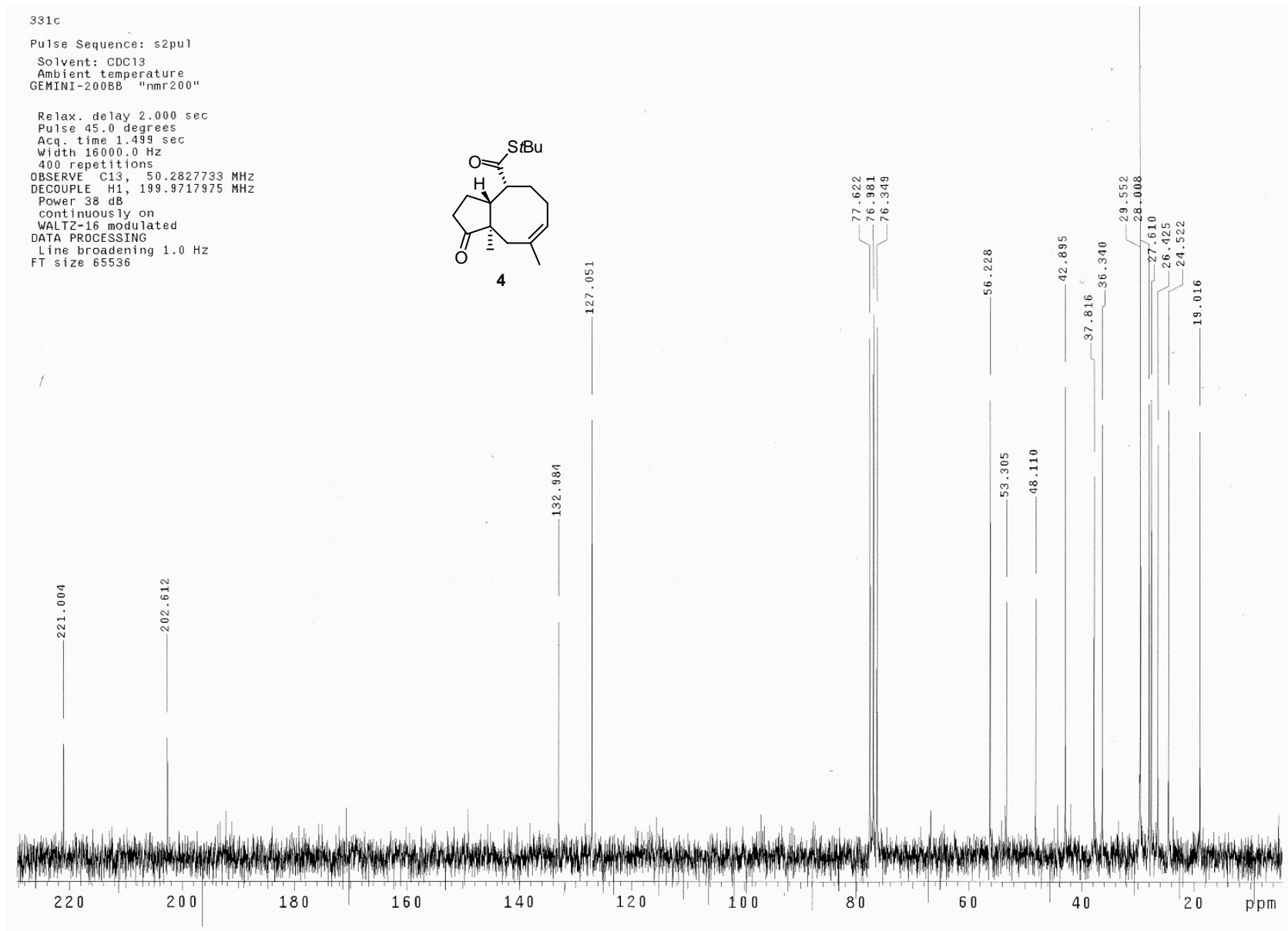
20 repetitions

OBSERVE H1, 199.9710974 MHz

DATA PROCESSING

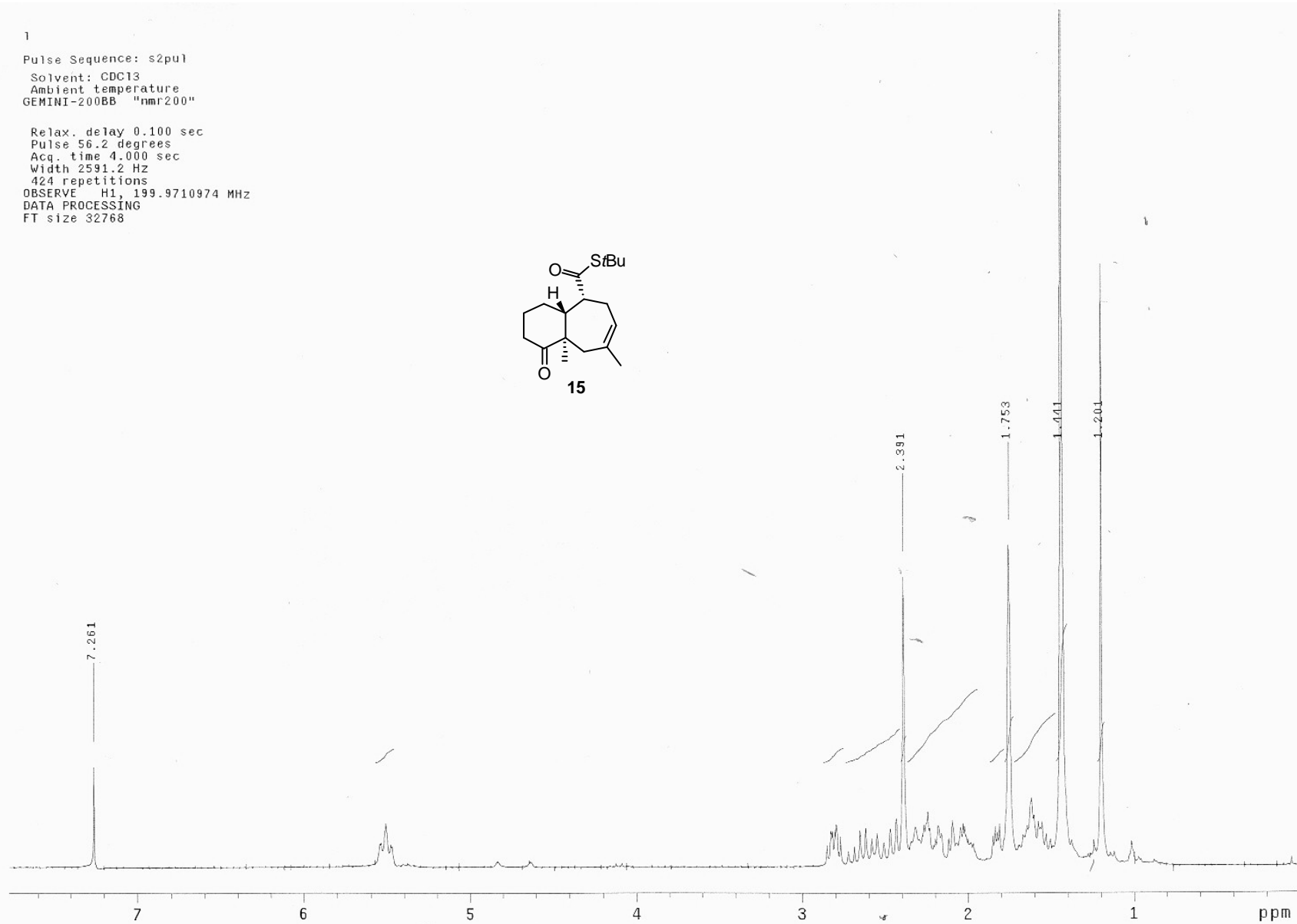
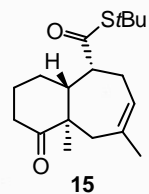
FT size 32768

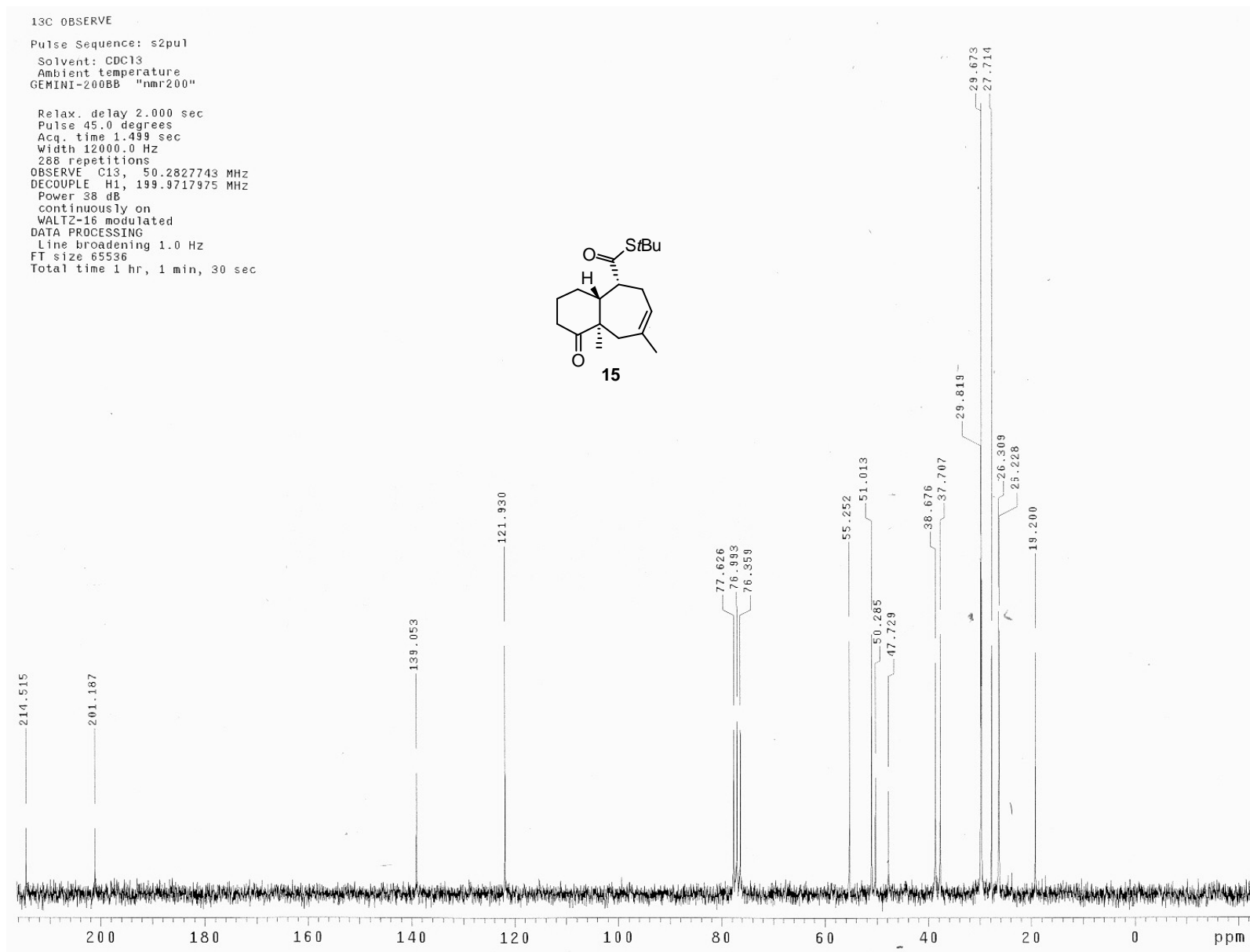


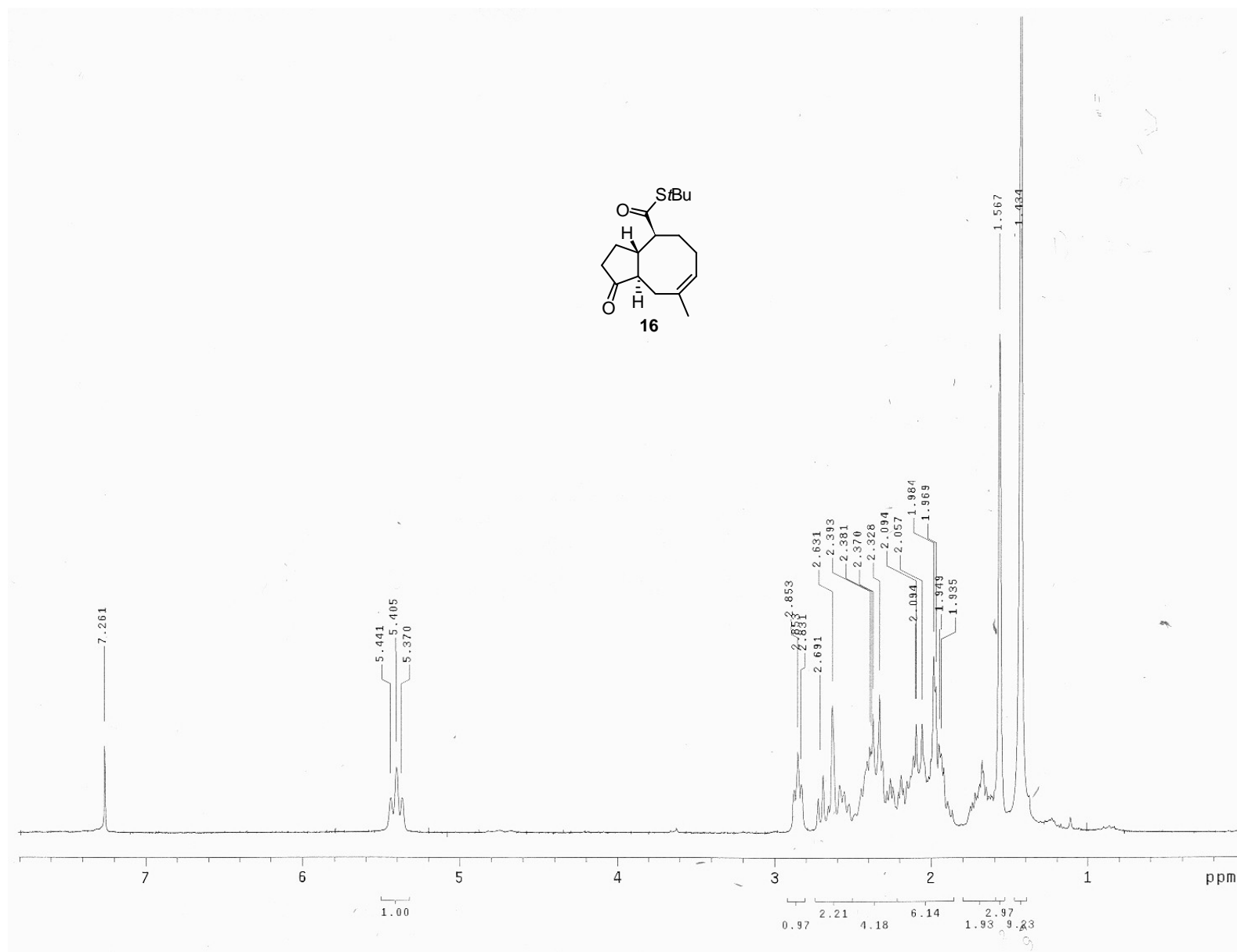


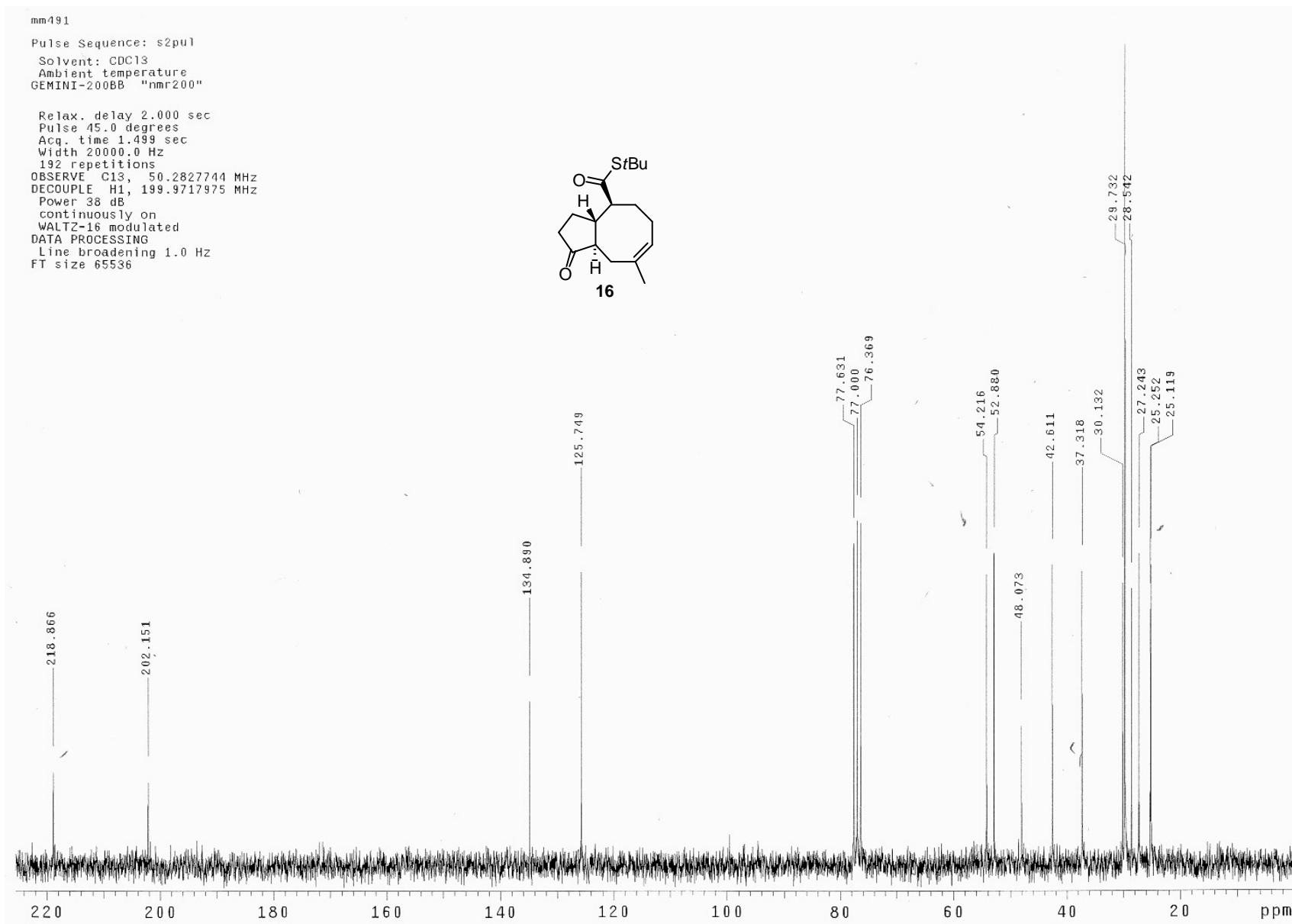
1
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-200BB "nmr200"

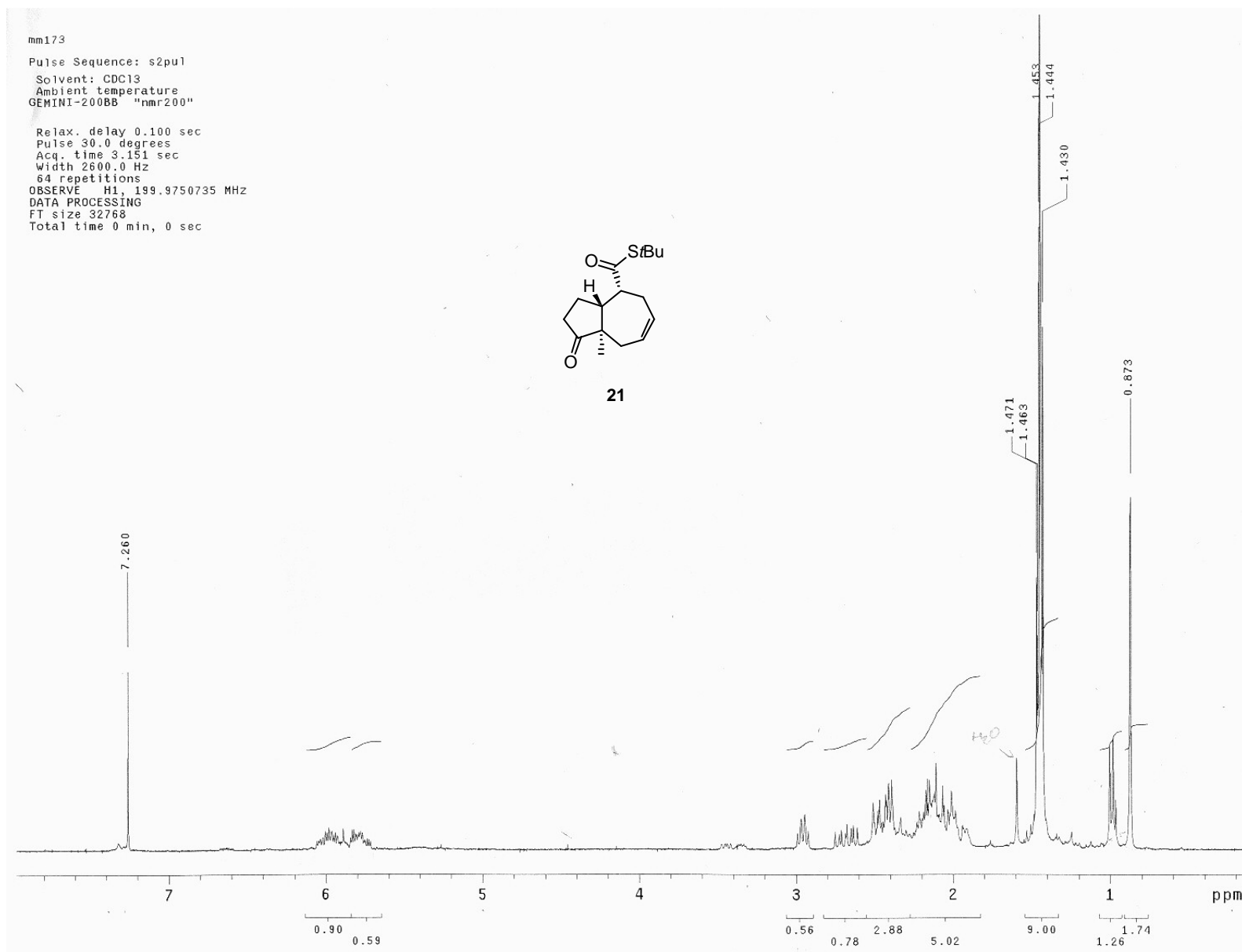
Relax. delay 0.100 sec
Pulse 56.2 degrees
Acq. time 4.000 sec
Width 2591.2 Hz
424 repetitions
OBSERVE H1, 199.9710974 MHz
DATA PROCESSING
FT size 32768

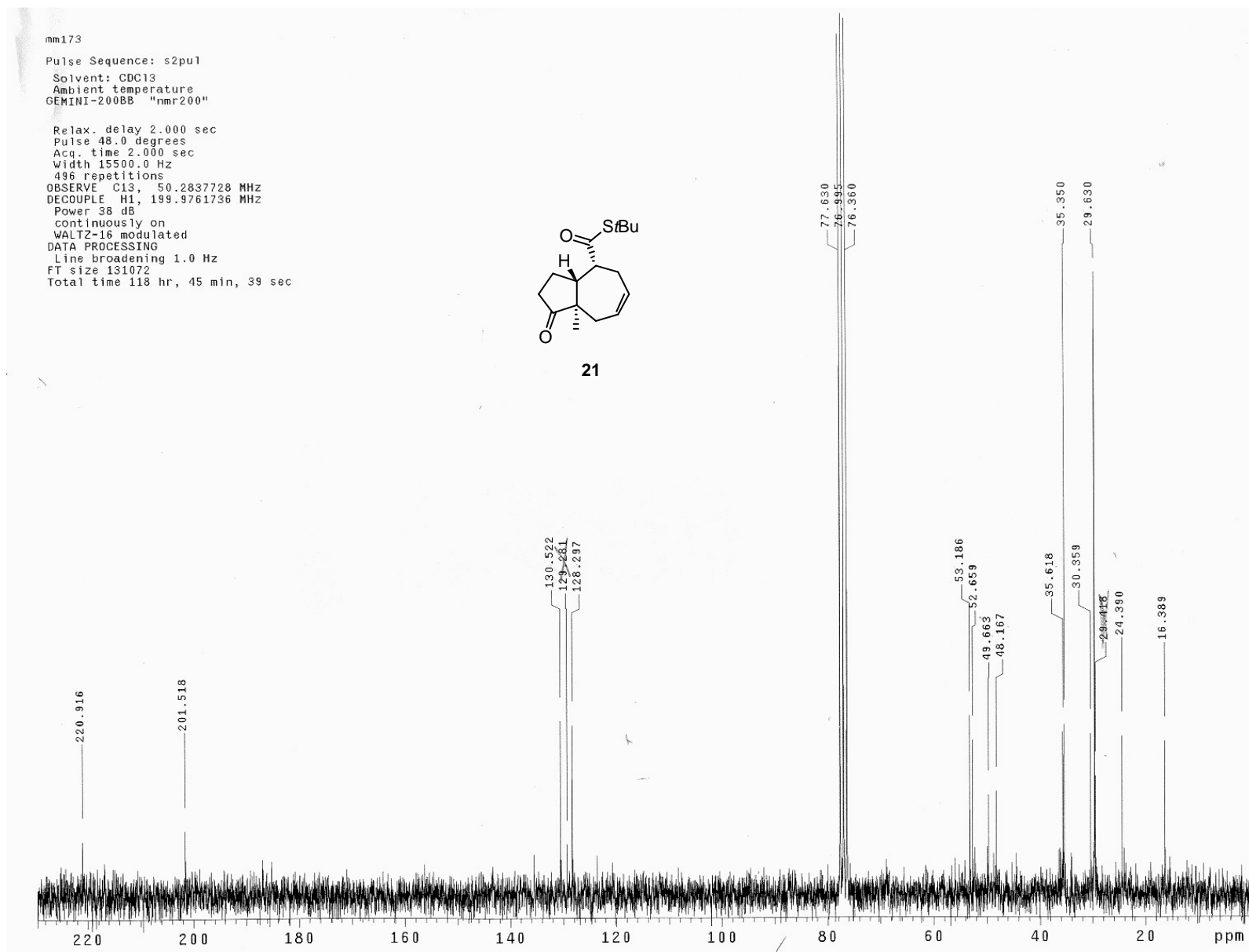


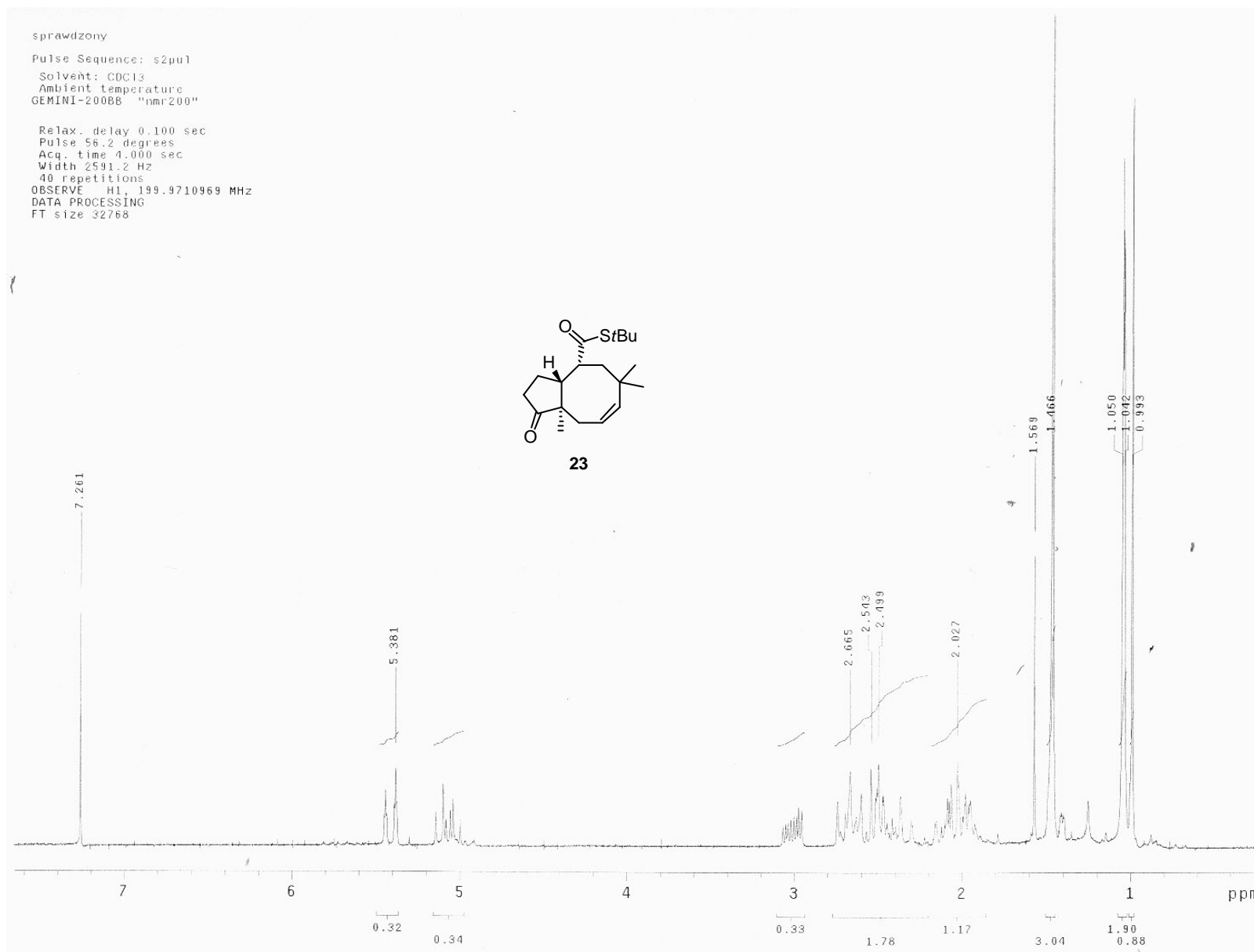


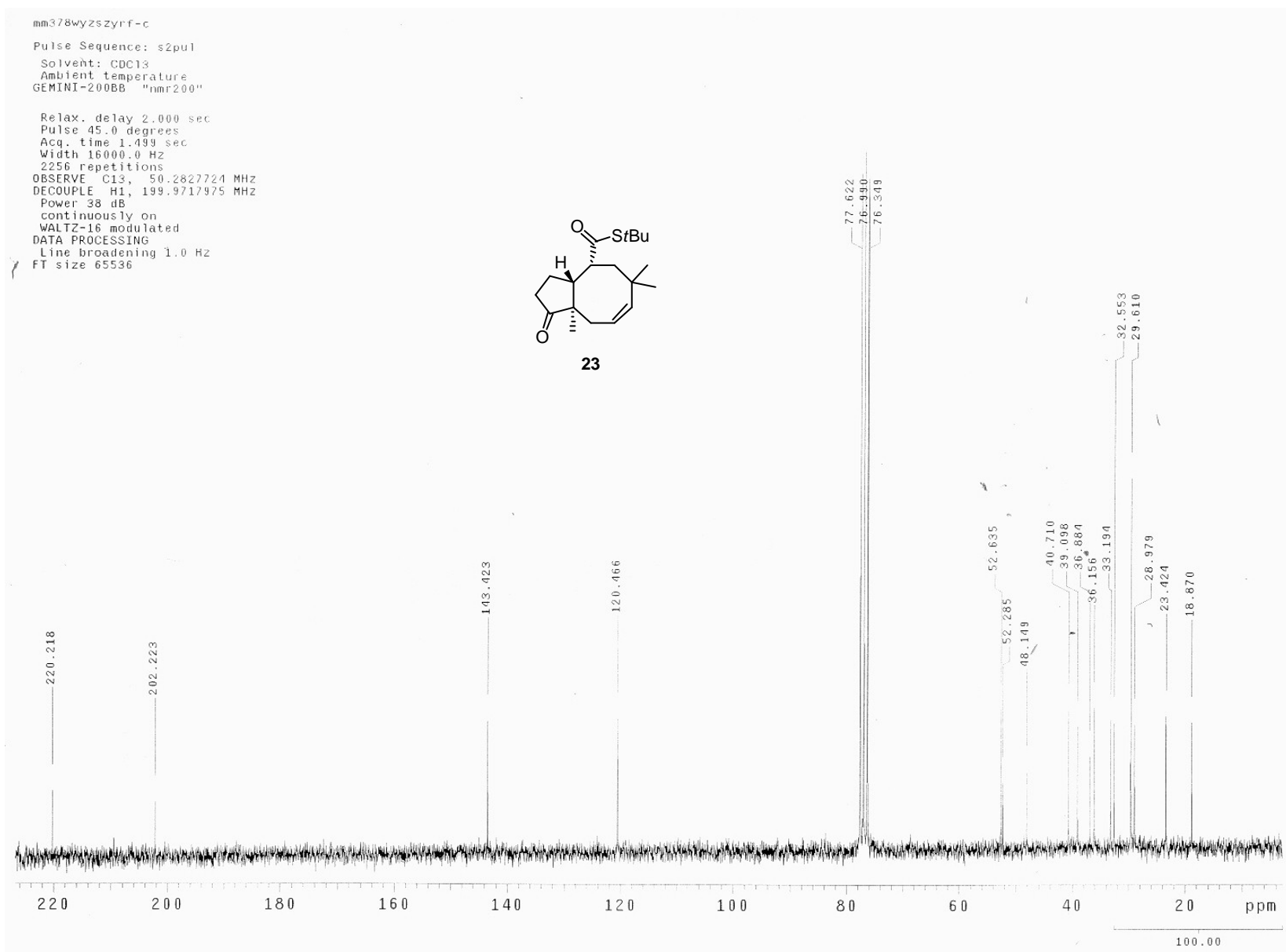


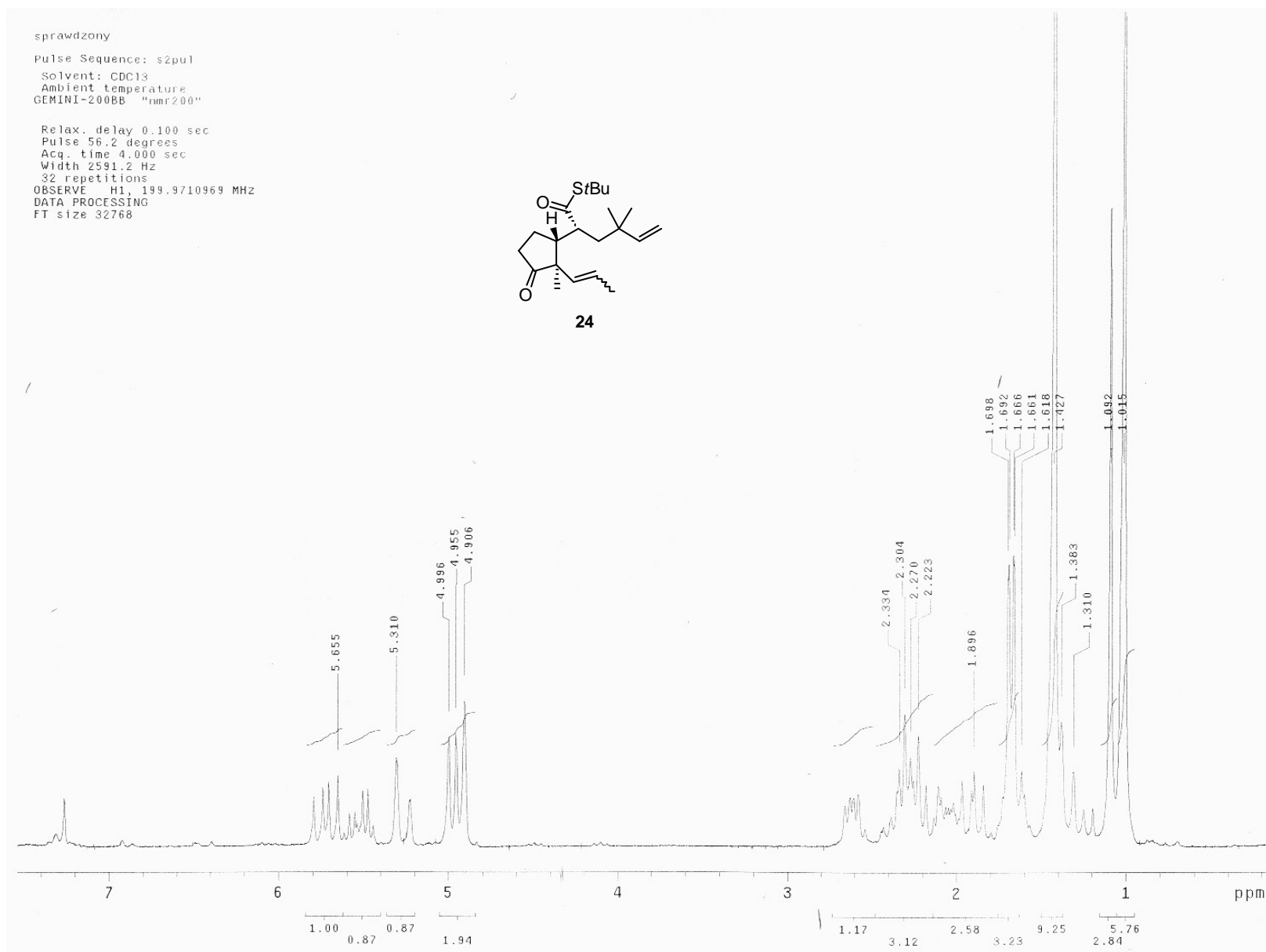


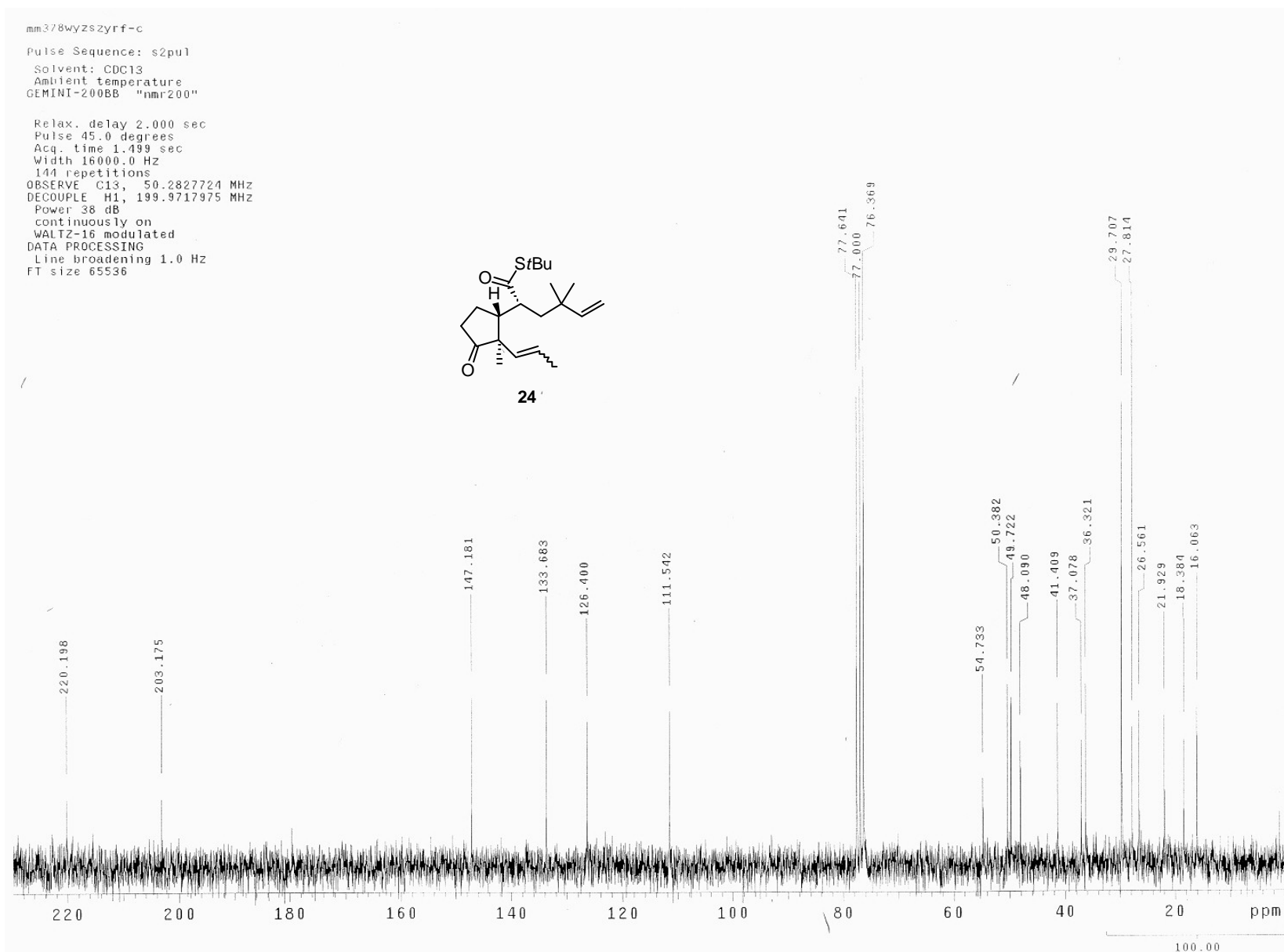


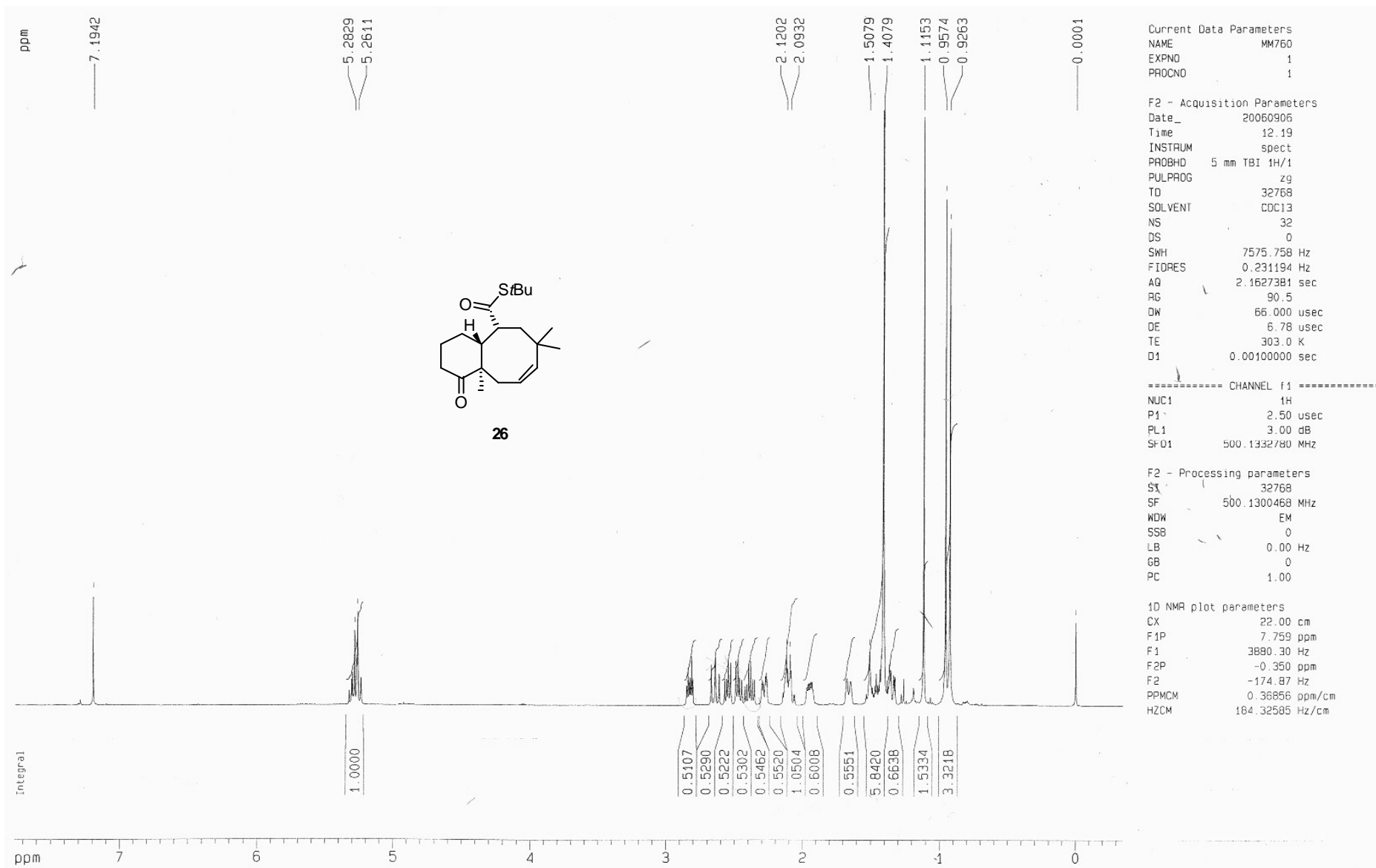


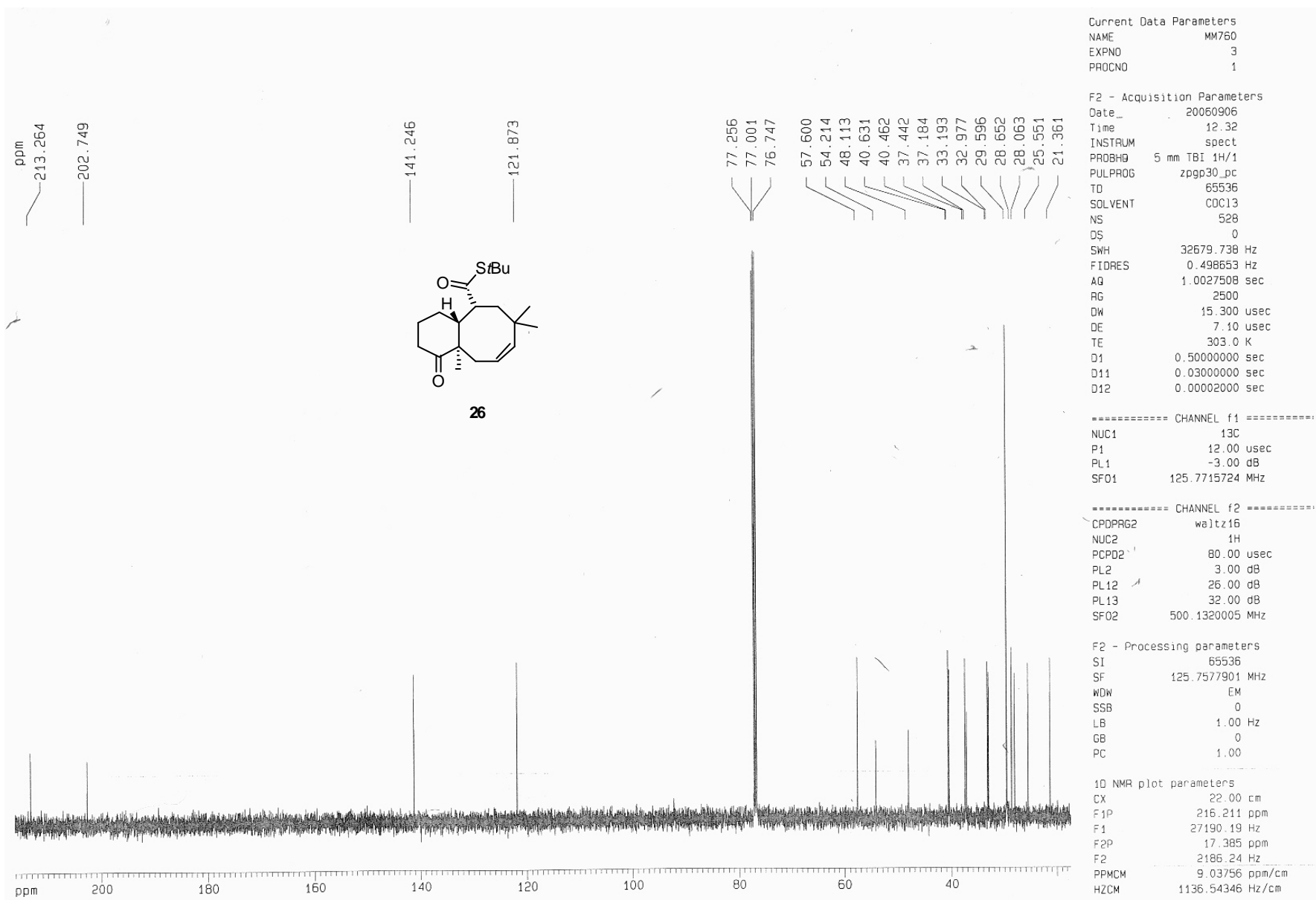


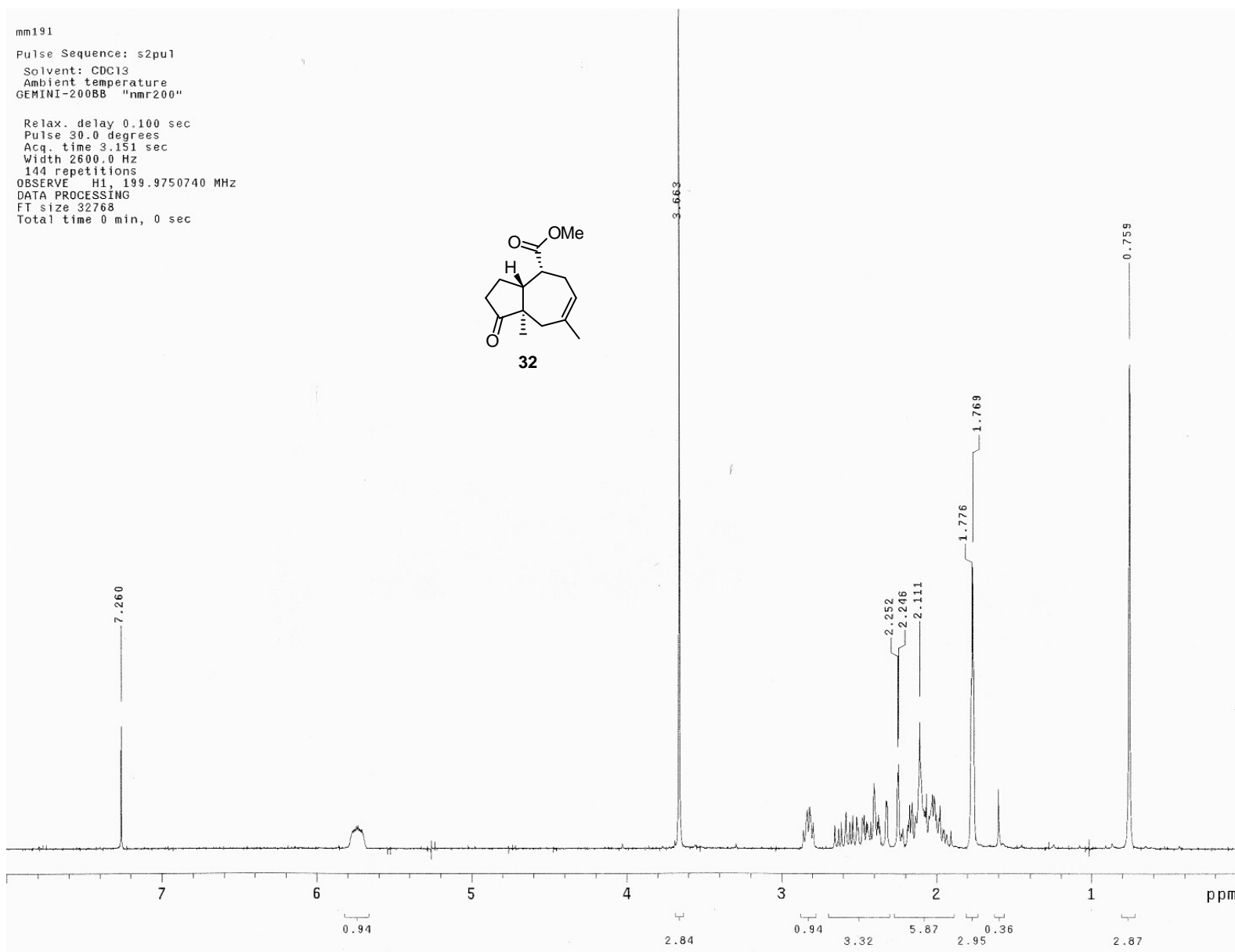


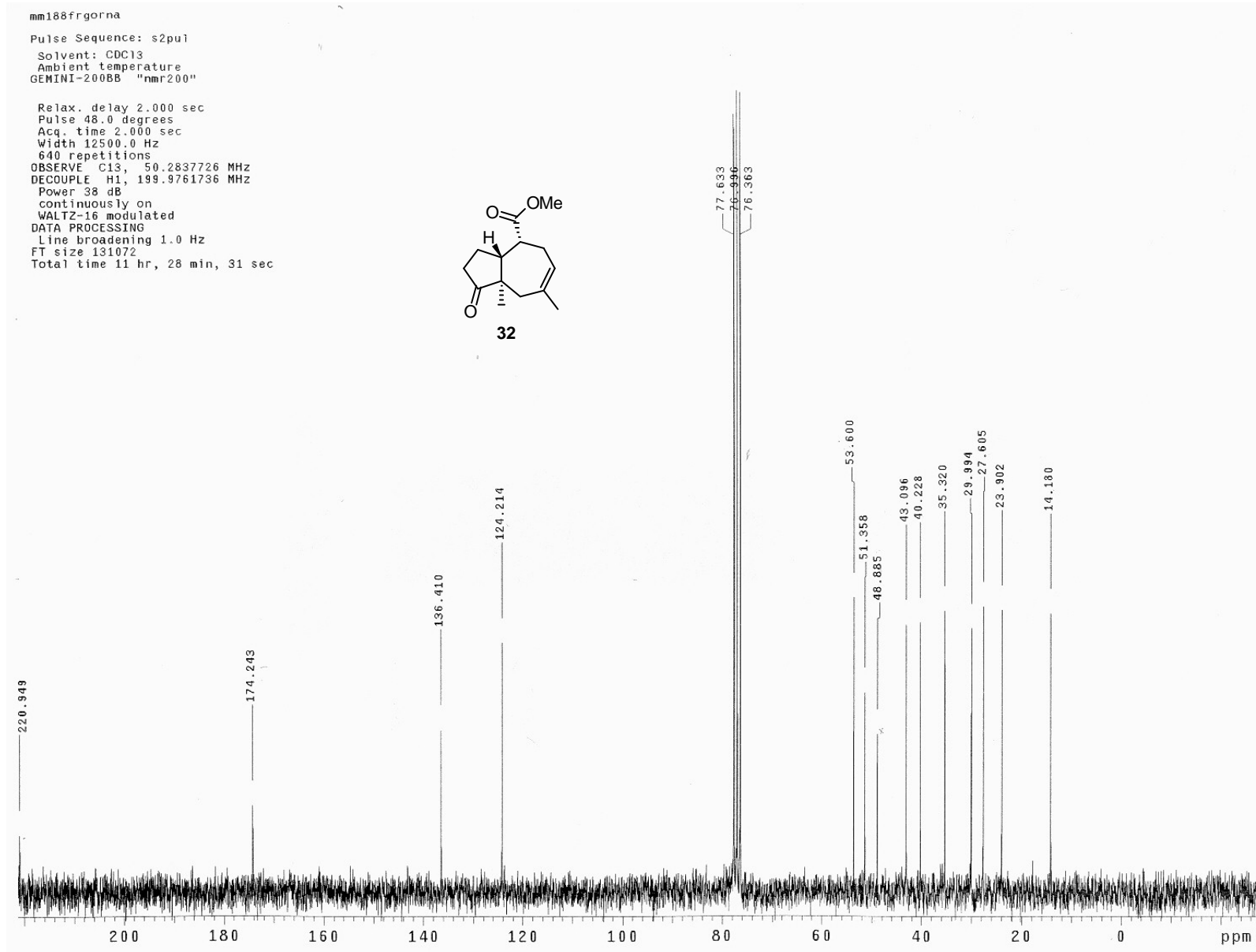


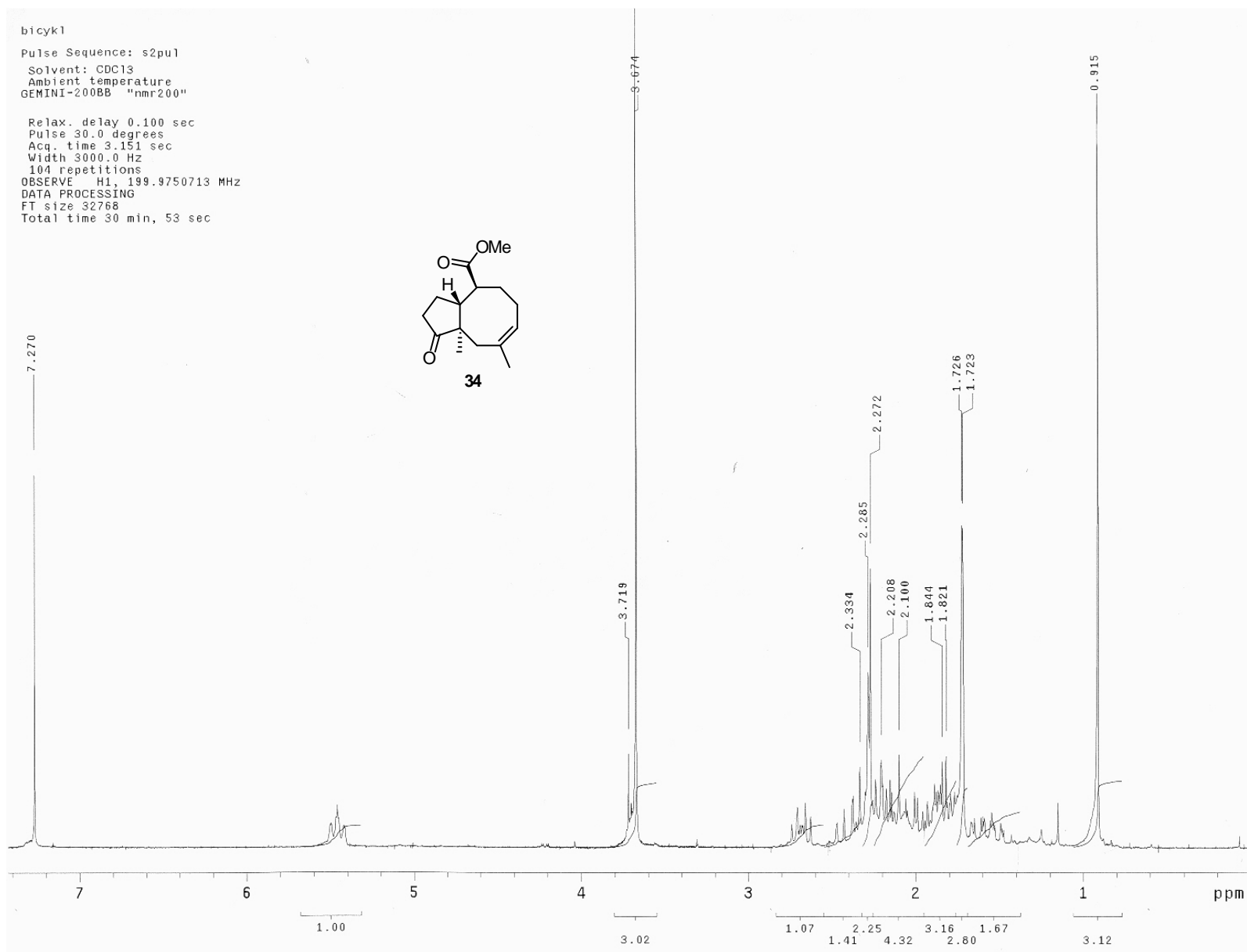


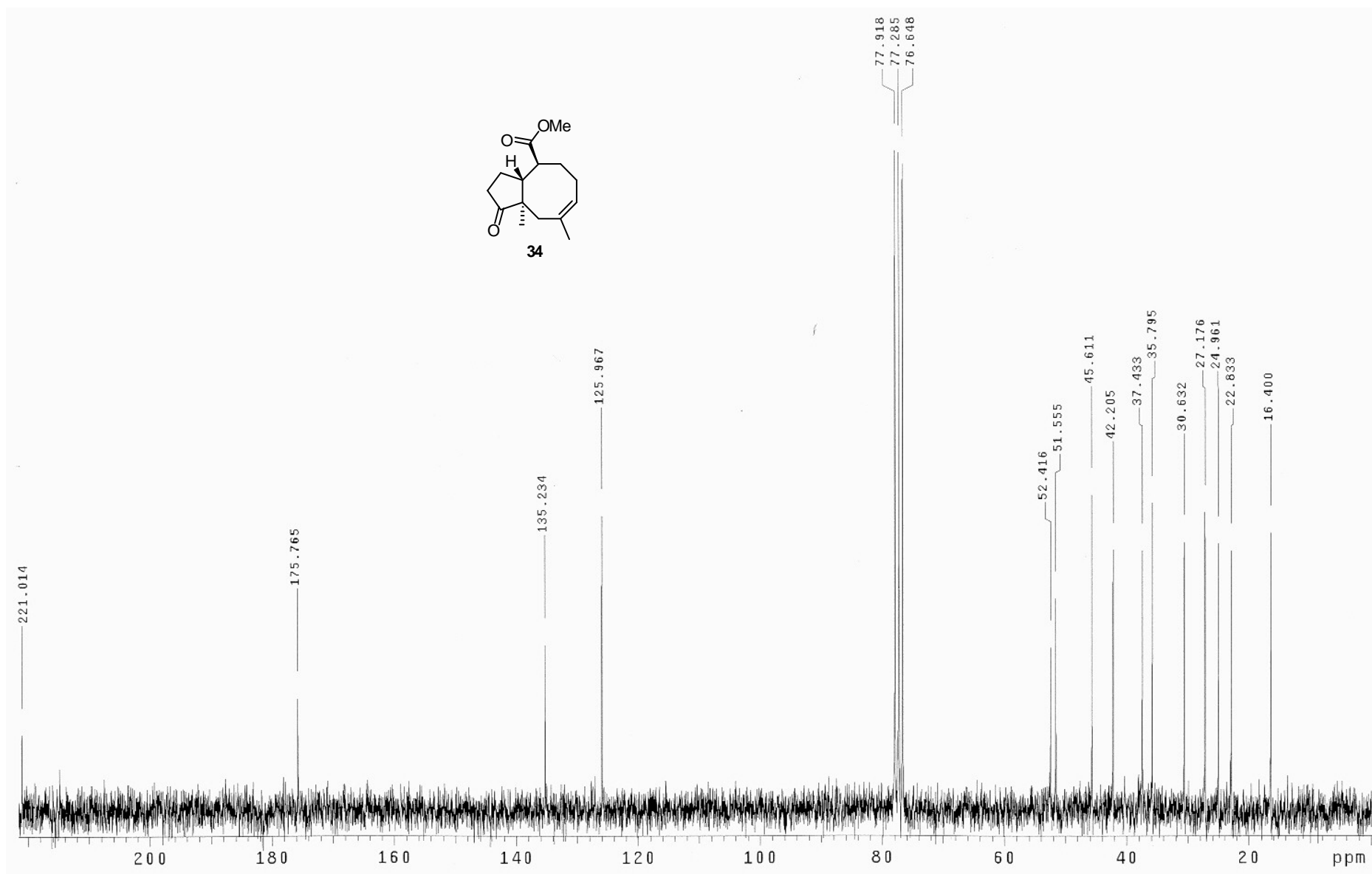


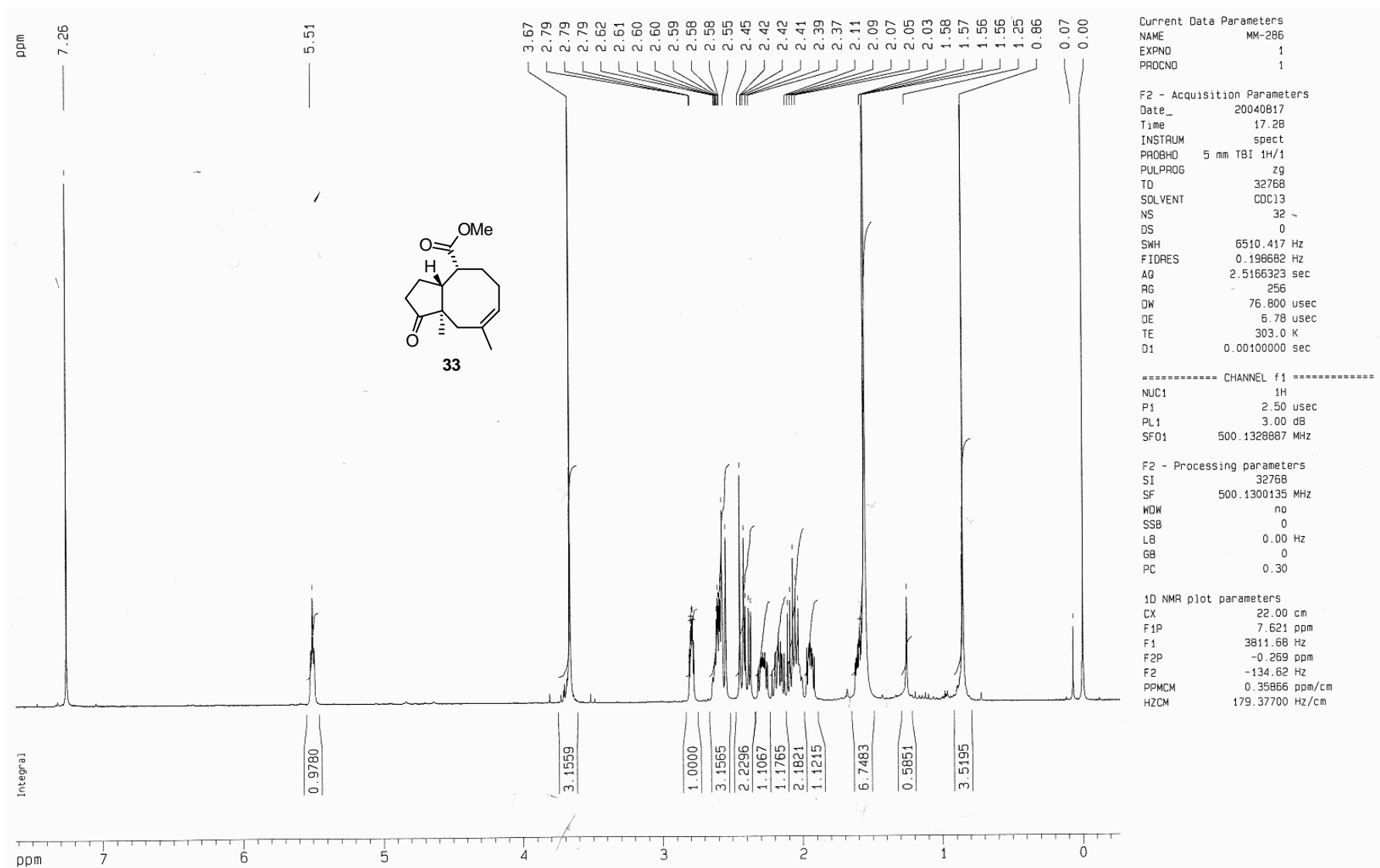












mm286cwyzrf

Pulse Sequence: s2pu1

Solvent: CDCl₃

Ambient temperature

GEMINI-200BB "nmr200"

Relax. delay 2.000 sec

Pulse 45.0 degrees

Acq. time 1.489 sec

Width 14000.0 Hz

1280 repetitions

OBSERVE C13, 50.2827750 MHz

DECOUPLE H1, 199.9717975 MHz

Power 38 dB

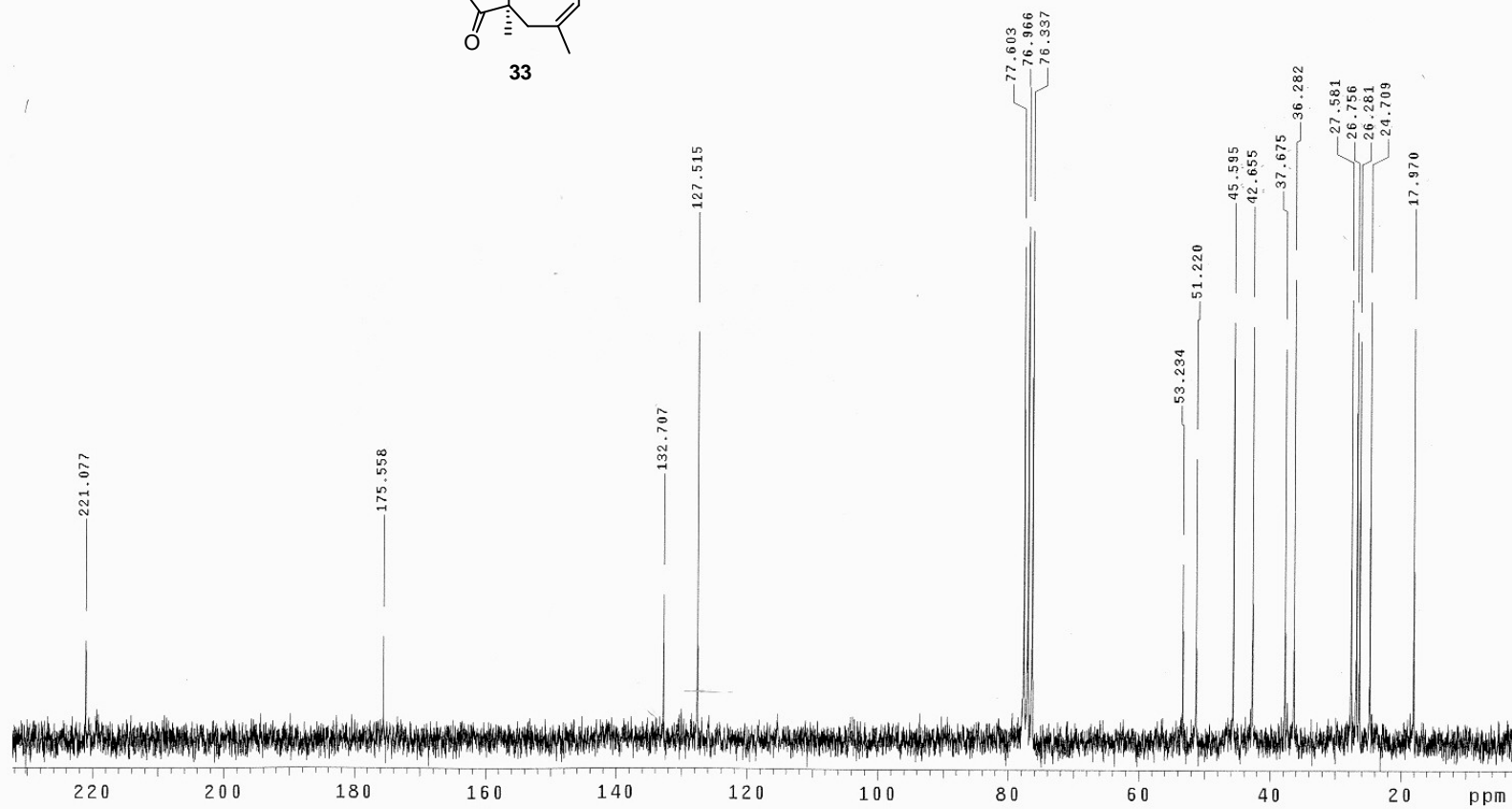
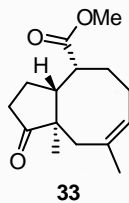
continuously on

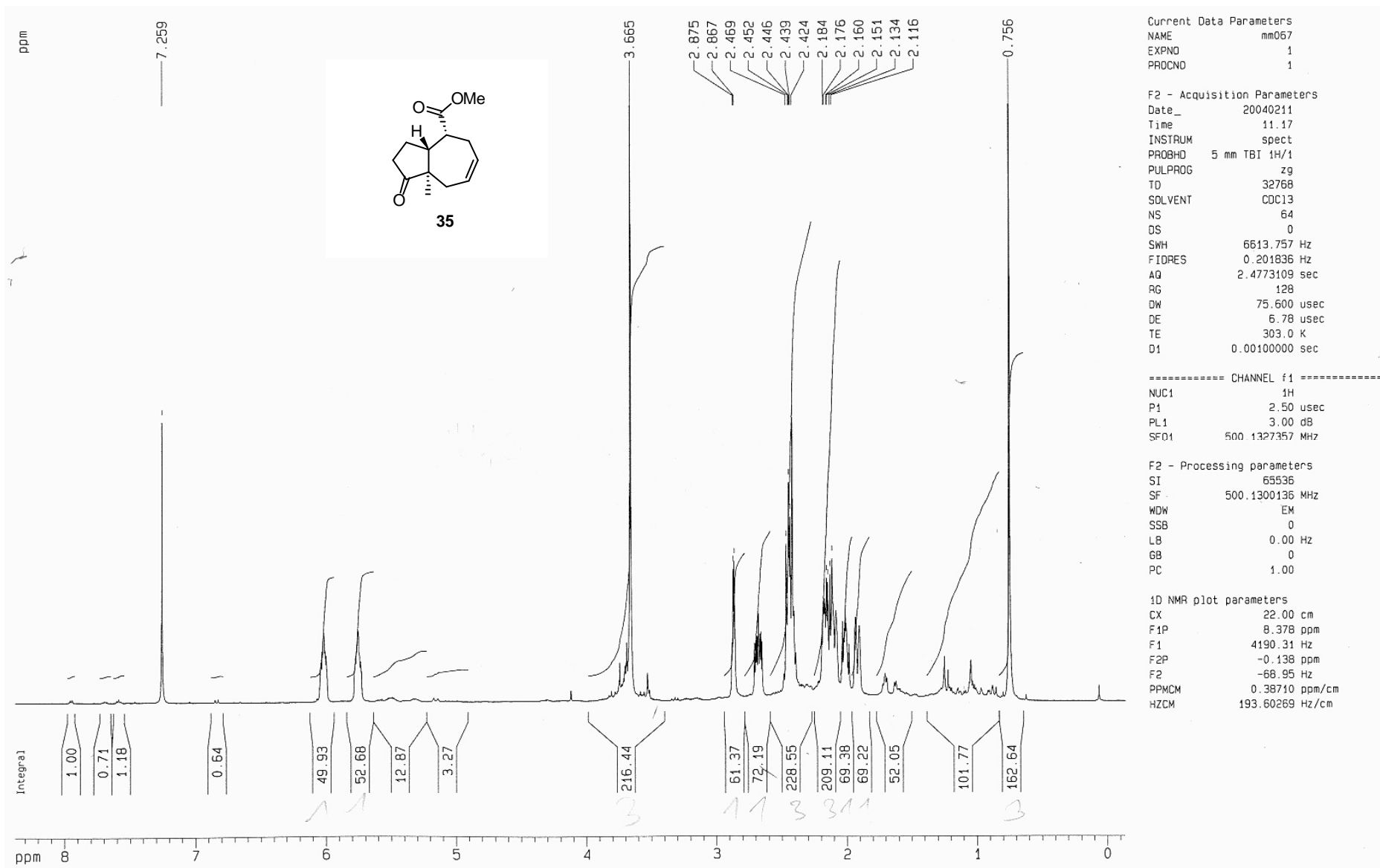
WALTZ-16 modulated

DATA PROCESSING

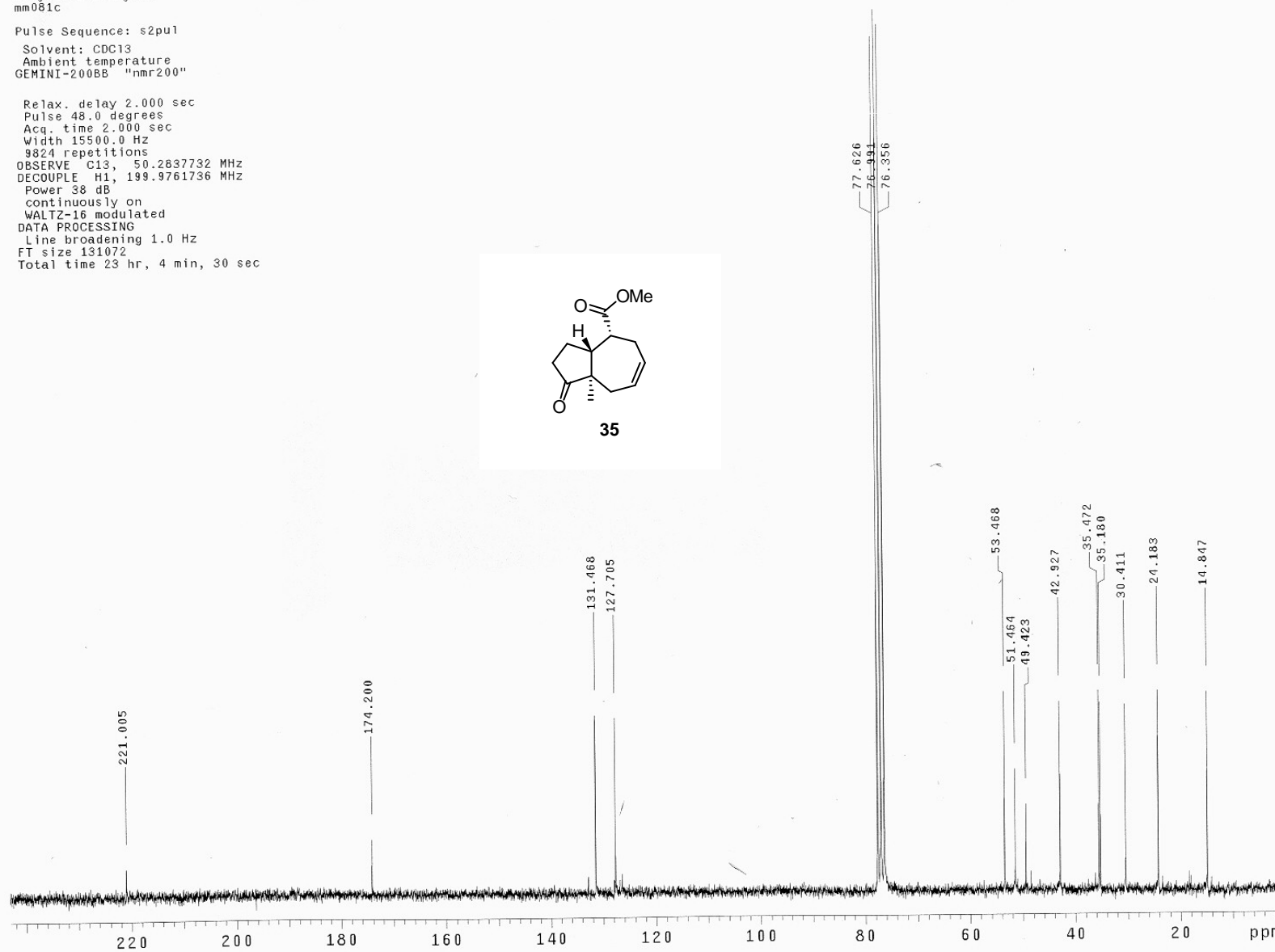
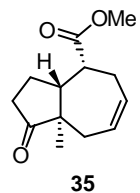
Line broadening 1.0 Hz

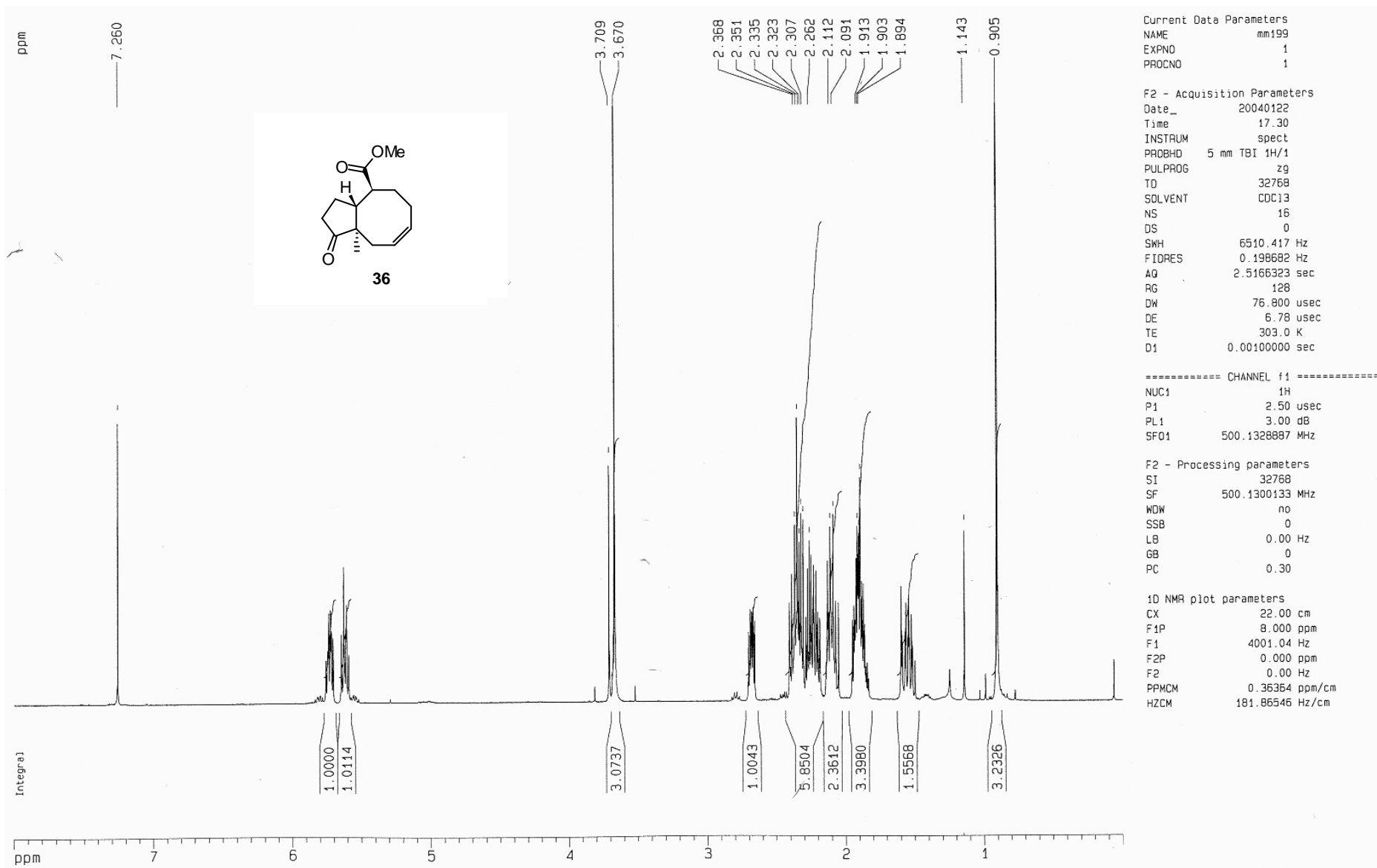
FT size 65536





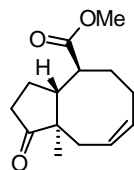
bicyklo z benzylem
mm081c
Pulse Sequence: s2pu1
Solvent: CDC13
Ambient temperature
GEMINI-200BB "nmr200"
Relax. delay 2.000 sec
Pulse 48.0 degrees
Acq. time 2.000 sec
Width 15500.0 Hz
9824 repetitions
OBSERVE C13, 50.2837732 MHz
DECOUPLE H1, 199.9761736 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 23 hr, 4 min, 30 sec



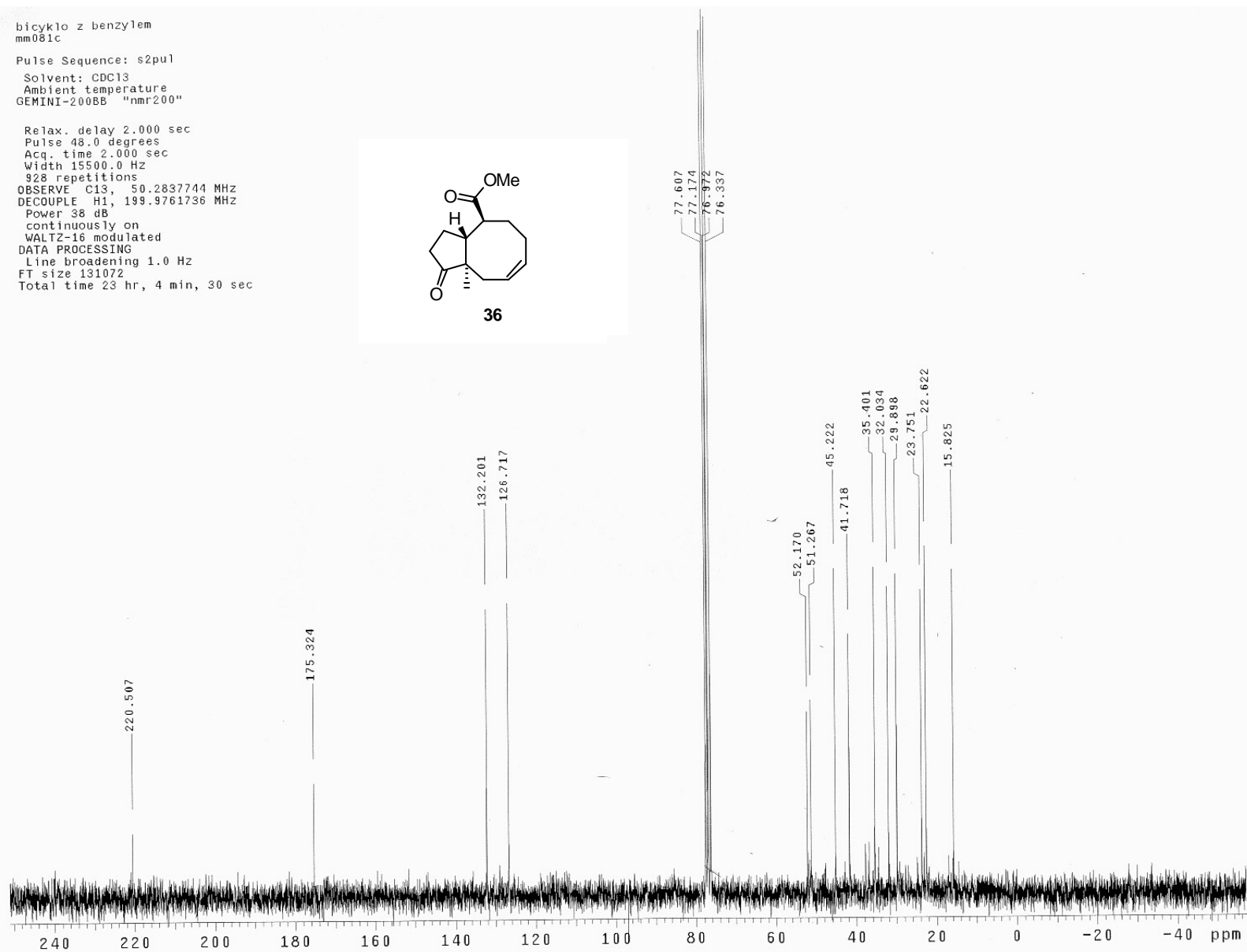


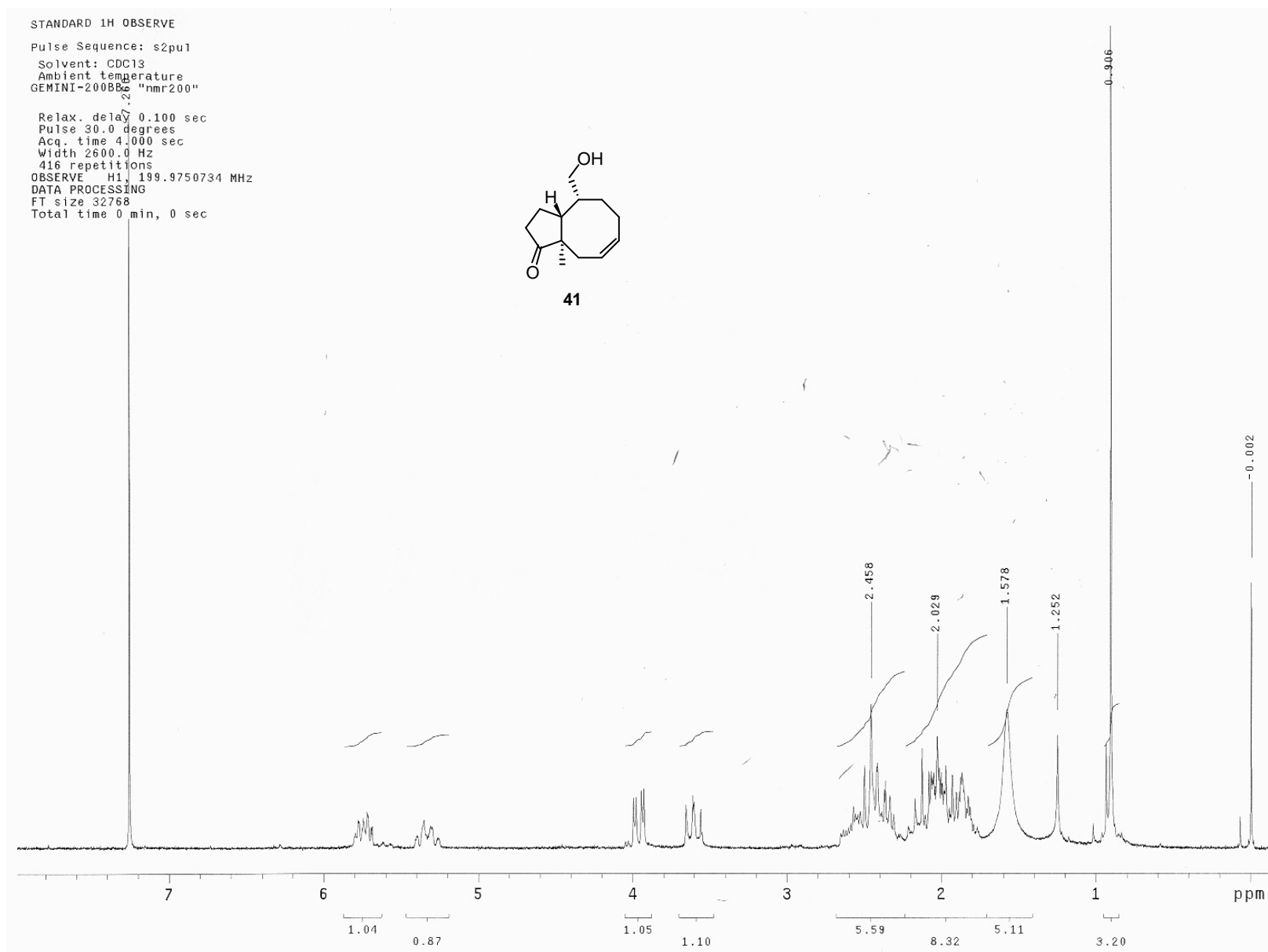
bicyklo z benzylem
mm081c
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
GEMINI-200BB "nmr200"

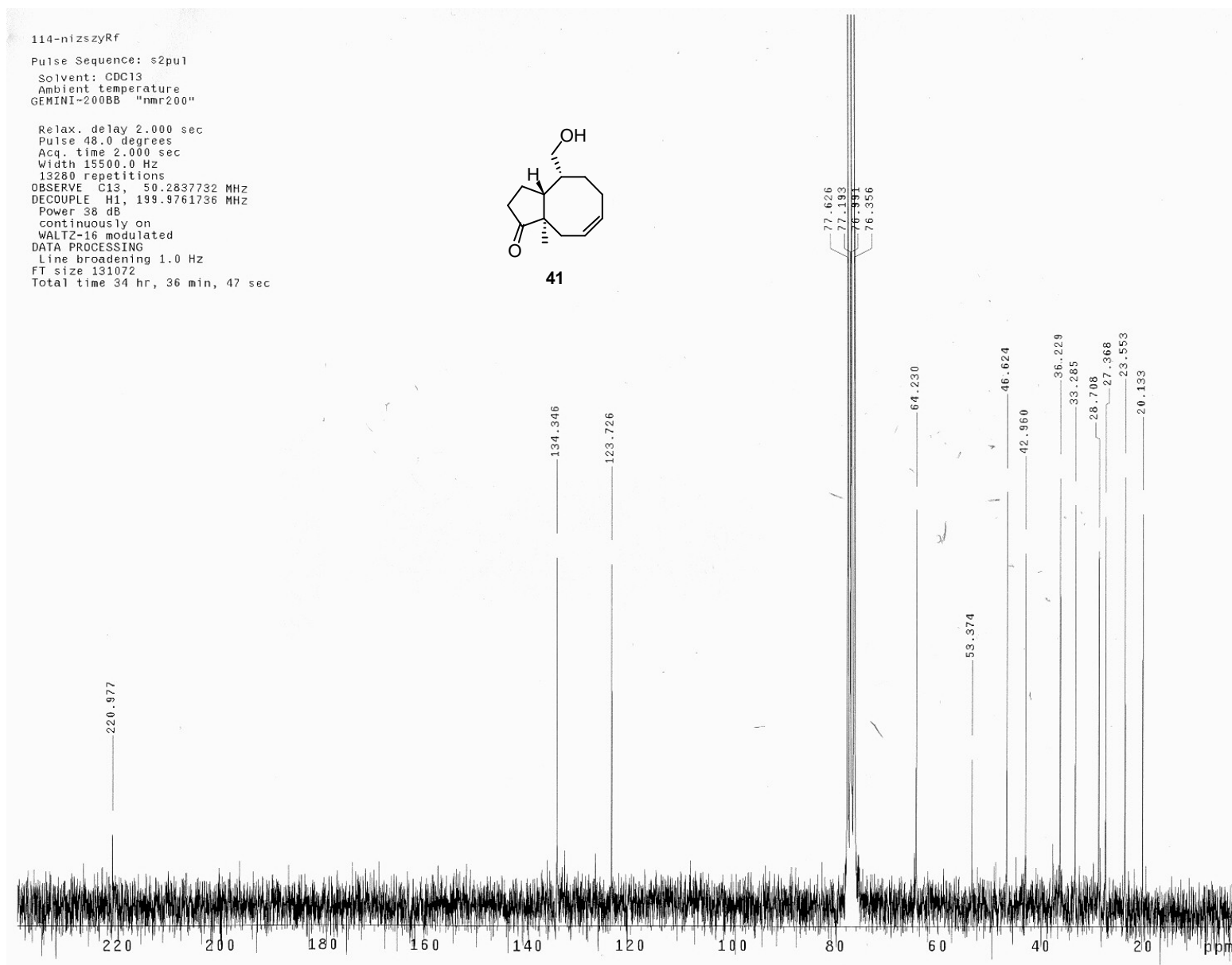
Relax. delay 2.000 sec
Pulse 48.0 degrees
Acq. time 2.000 sec
Width 15500.0 Hz
928 repetitions
OBSERVE C13, 50.2837744 MHz
DECOUPLE H1, 199.9761736 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 23 hr, 4 min, 30 sec

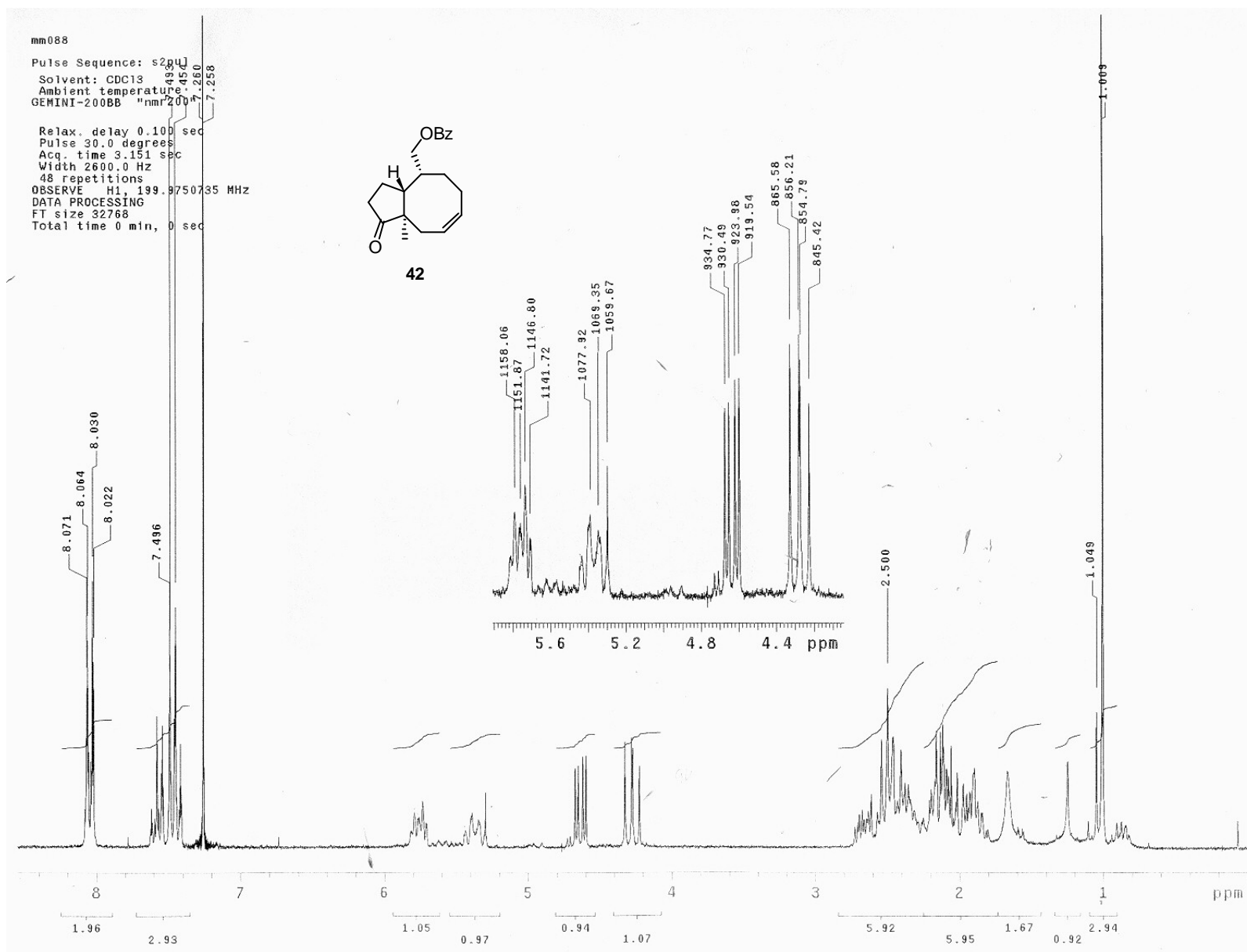


36



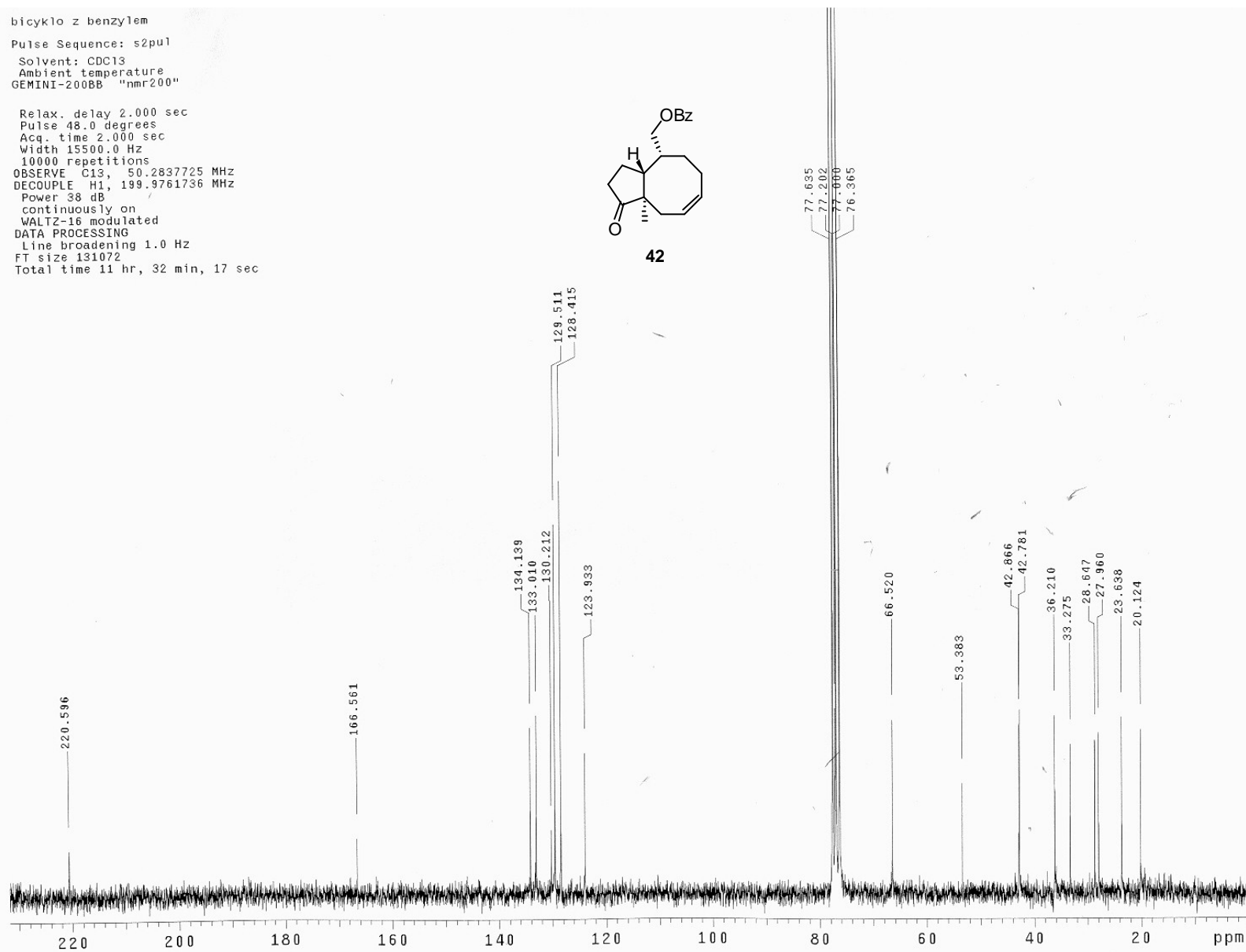
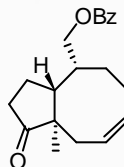


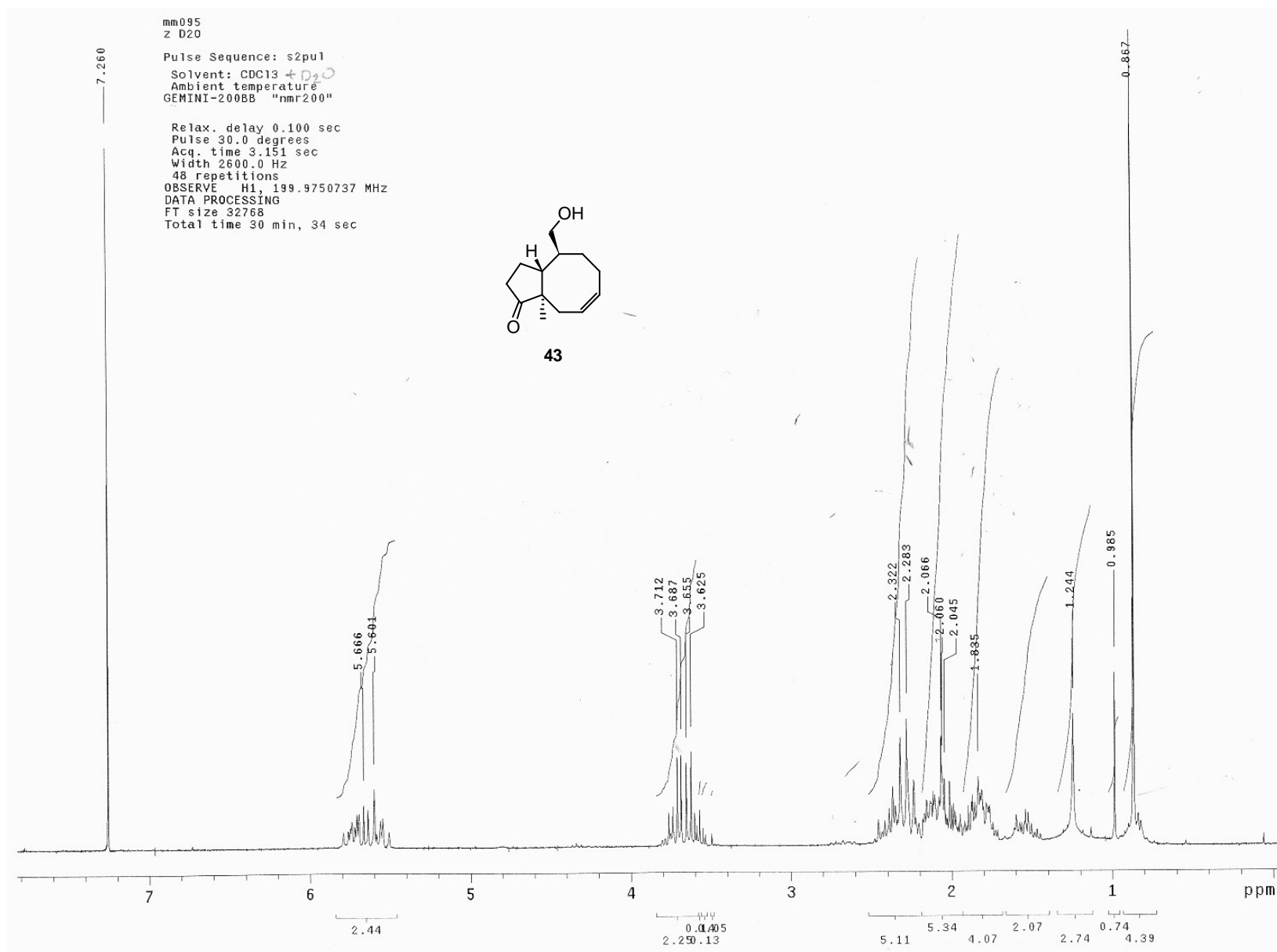




bicyklo z benzylem
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
GEMINI-200BB "nmr200"

Relax. delay 2.000 sec
Pulse 48.0 degrees
Acq. time 2.000 sec
Width 15500.0 Hz
10000 repetitions
OBSERVE C13, 50.2837725 MHz
DECOUPLE H1, 199.9761736 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 11 hr, 32 min, 17 sec

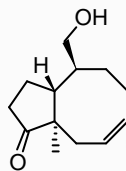




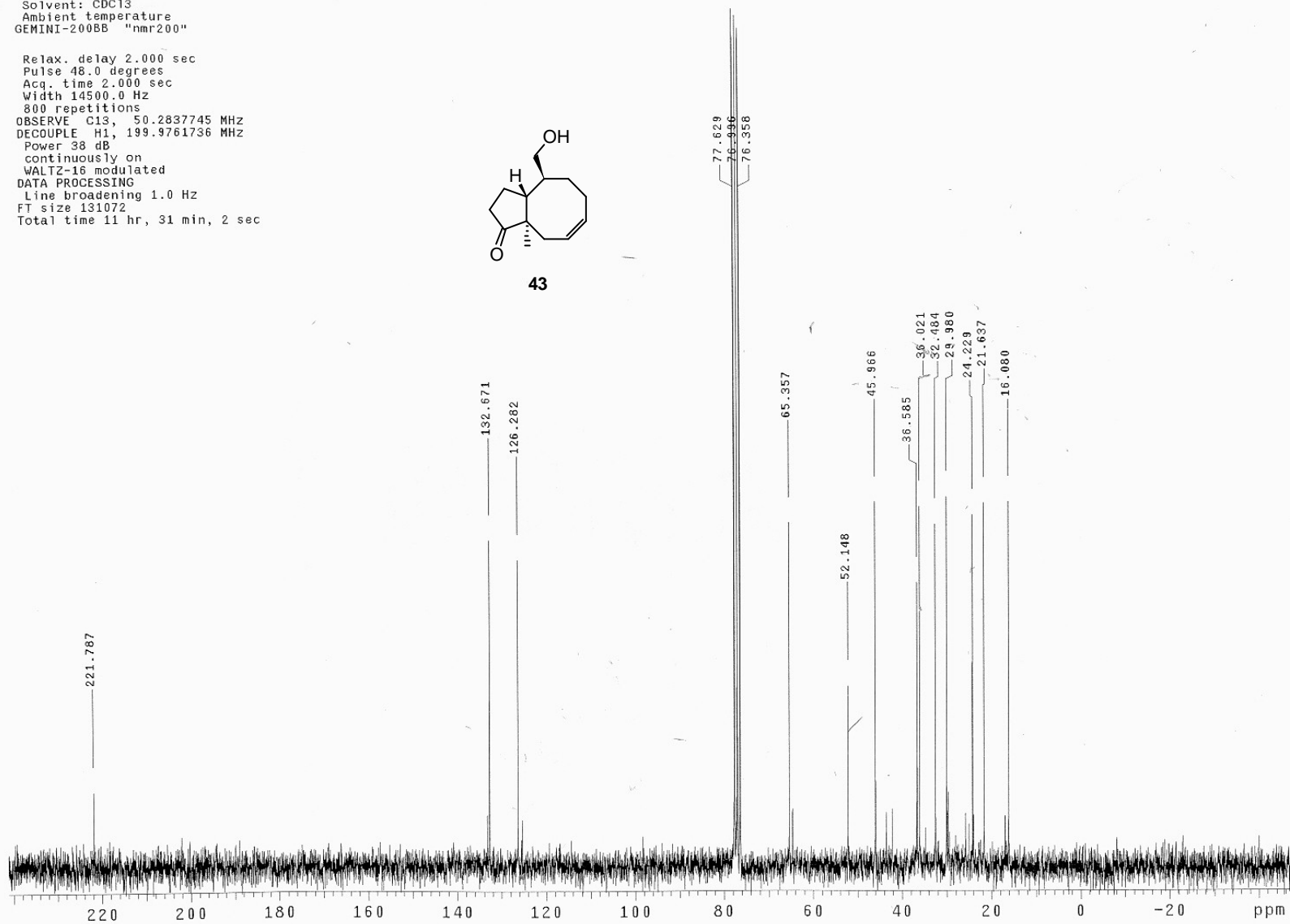
mm094

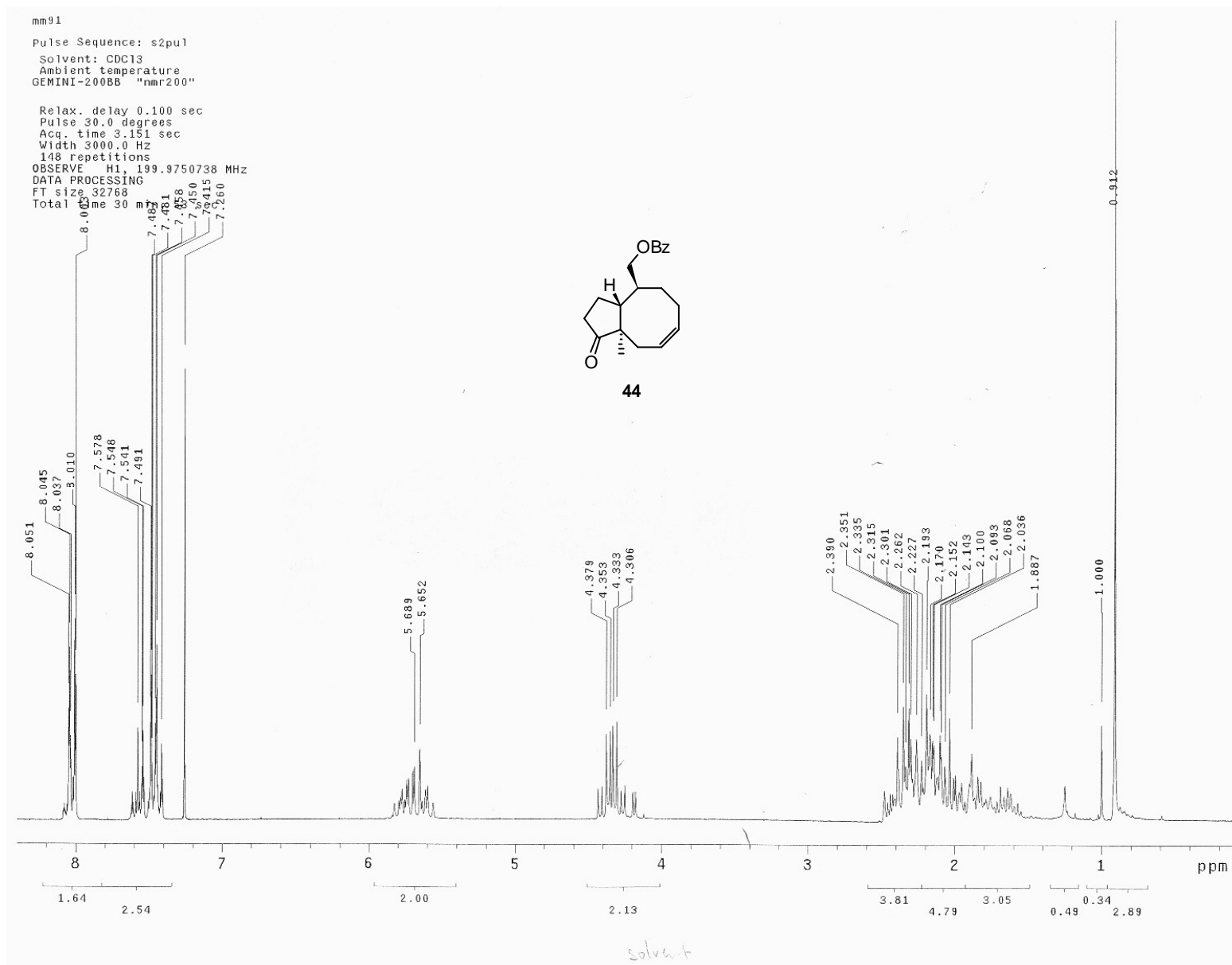
Pulse Sequence: s2pu1
Solvent: CDC13
Ambient temperature
GEMINI-200BB "nmr200"

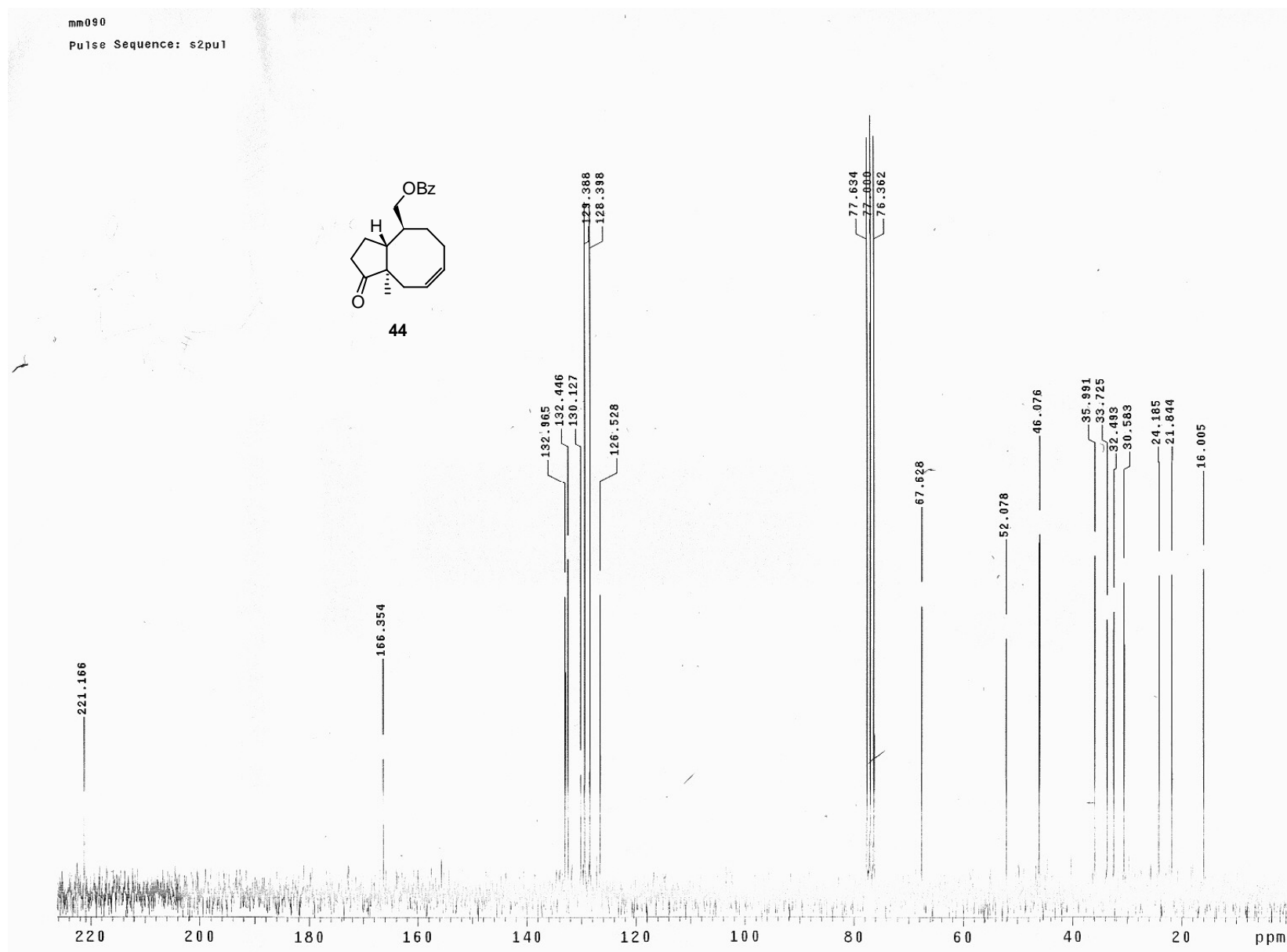
Relax. delay 2.000 sec
Pulse 48.0 degrees
Acq. time 2.000 sec
Width 14500.0 Hz
800 repetitions
OBSERVE C13, 50.2837745 MHz
DECOUPLE H1, 199.9761736 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 11 hr, 31 min, 2 sec



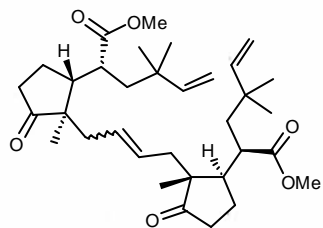
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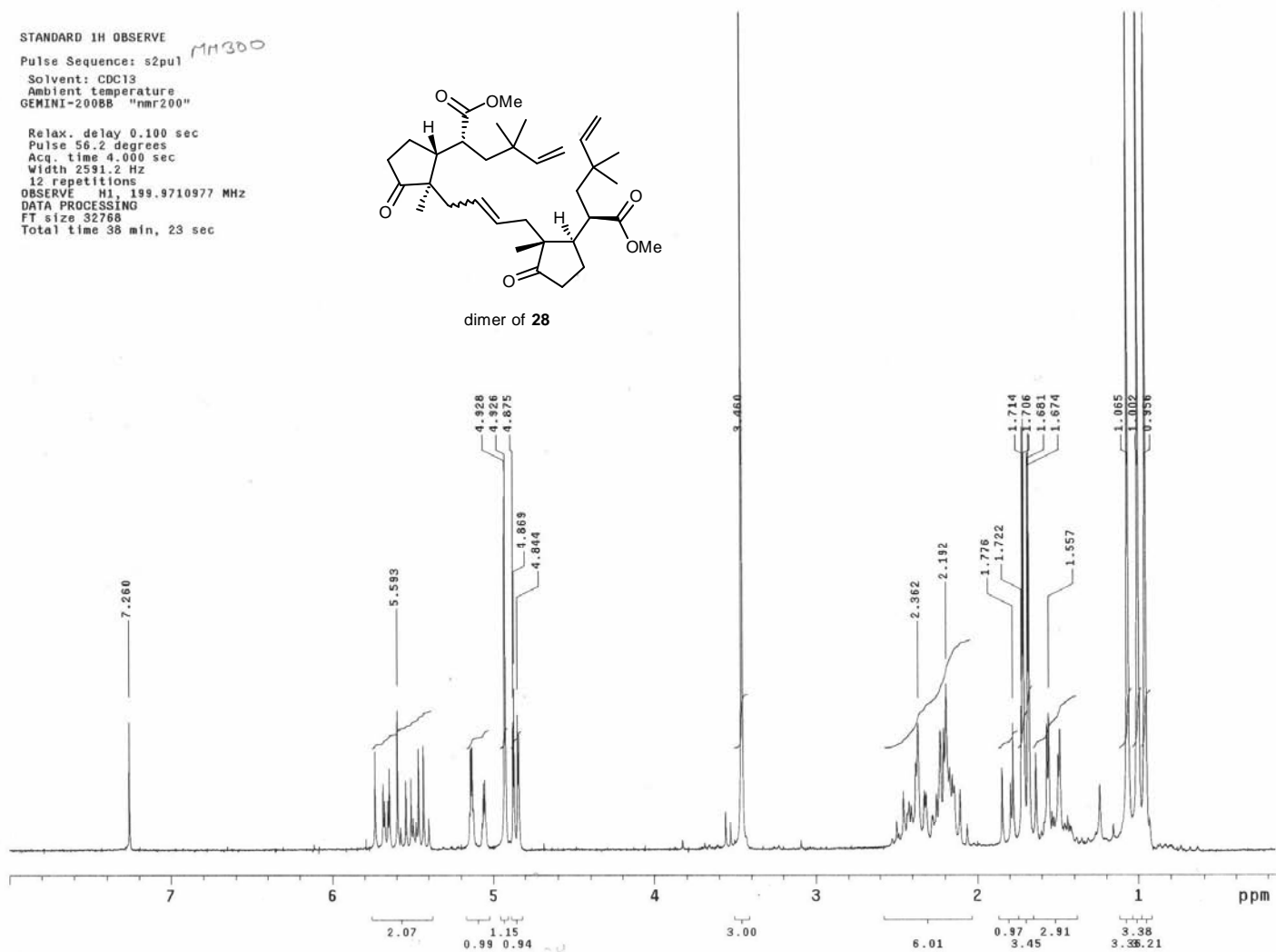




STANDARD 1H OBSERVE
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
GEMINI-2008B "nmr200"
Relax. delay 0.100 sec
Pulse 56.2 degrees
Acq. time 4.000 sec
Width 2591.2 Hz
12 repetitions
OBSERVE H1, 199.9710977 MHz
DATA PROCESSING
FT size 32768
Total time 38 min, 23 sec



dimer of 28

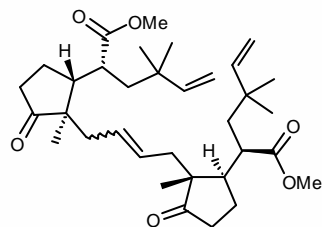


bromekcitronelal

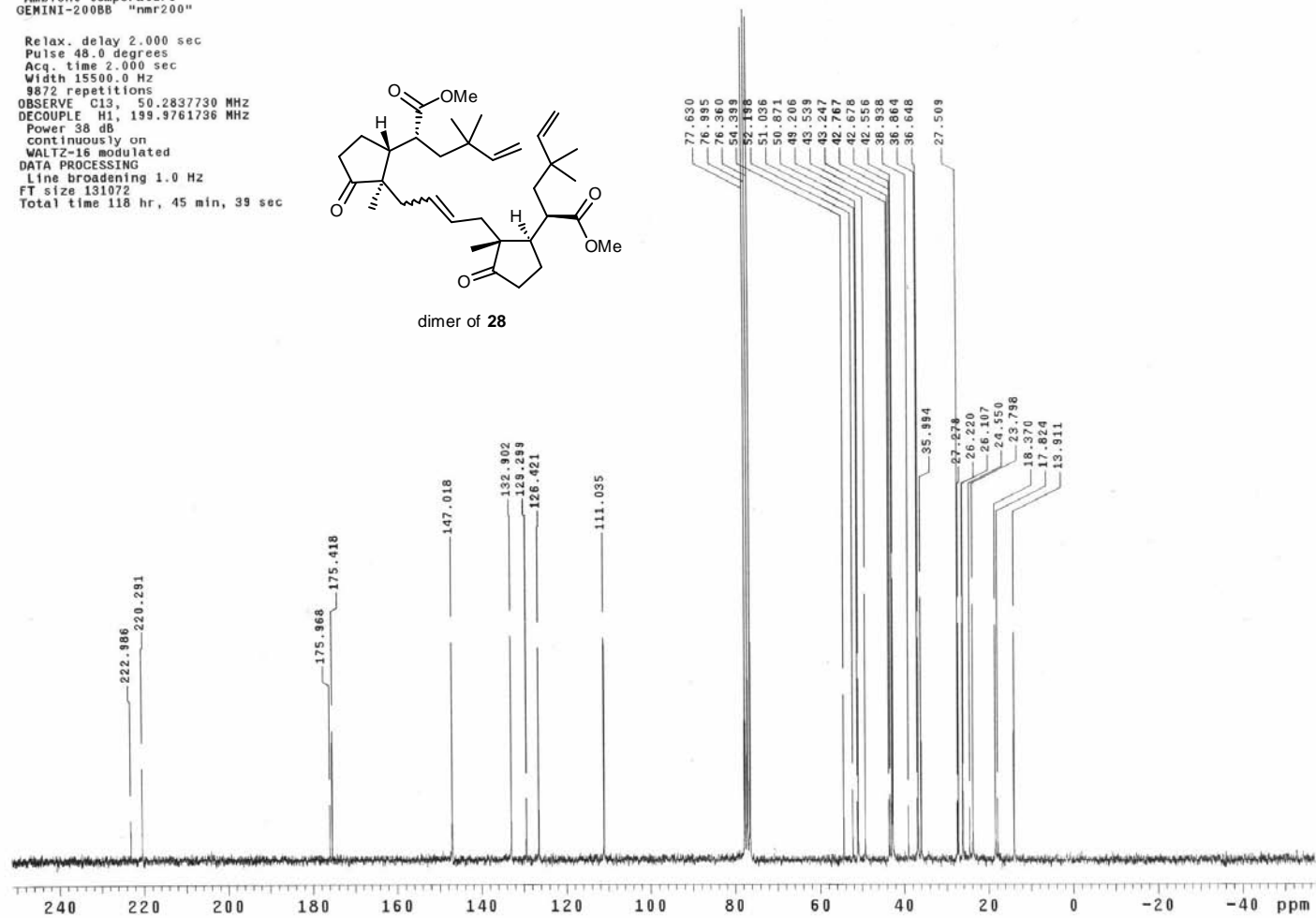
Pulse Sequence: s2pu1

Solvent: CDC13
Ambient temperature
GEMINI-200BB "nmr200"

Relax. delay 2.000 sec
Pulse 48.0 degrees
Acq. time 2.000 sec
Width 15500.0 Hz
9872 repetitions
OBSERVE C13, 50.2837730 MHz
DECOUPLE H1, 199.9761736 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 118 hr, 45 min, 39 sec



dimer of 28



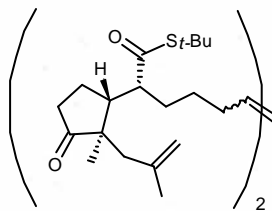
STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

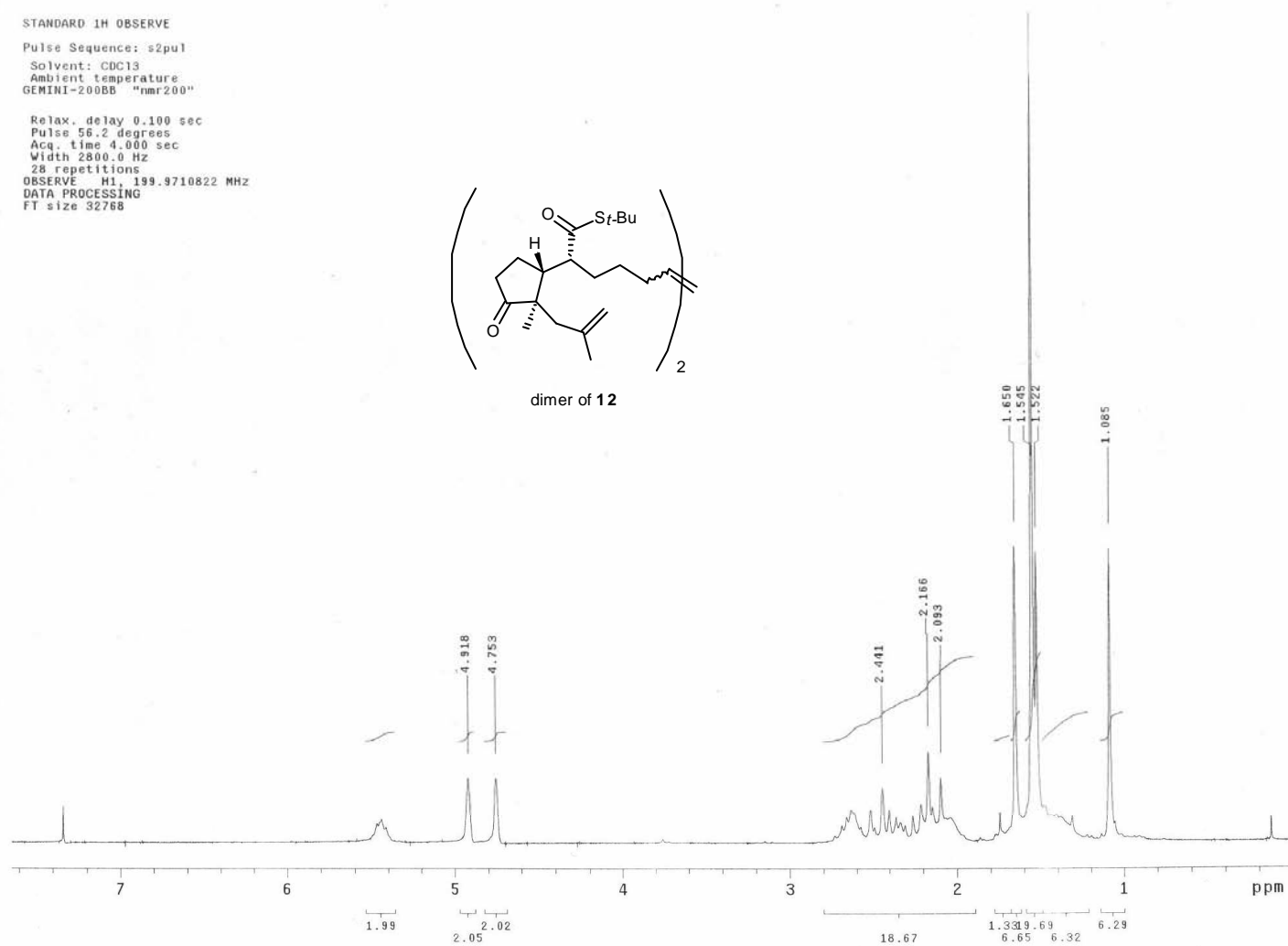
Solvent: CDCl3

Ambient temperature
GEMINI-200BB "nmr200"

Relax. delay 0.100 sec
Pulse 56.2 degrees
Acq. time 4.000 sec
Width 2800.0 Hz
28 repetitions
OBSERVE H1, 199.9710822 MHz
DATA PROCESSING
FT size 32768



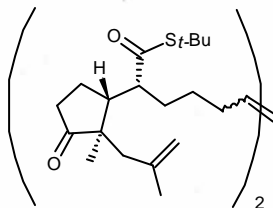
dimer of 12



¹³C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
GEMINI-200BB "nmr200"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.499 sec
Width 14500.0 Hz
320 repetitions
OBSERVE C13, 50.2827746 MHz
DECOUPLE H1, 199.9717975 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536



dimer of 12

