SUPPORTING INFORMATION

FOR

Ethynylbenzenoid metabolites of *Antrodia camphorata*: synthesis and inhibition of TNF expression

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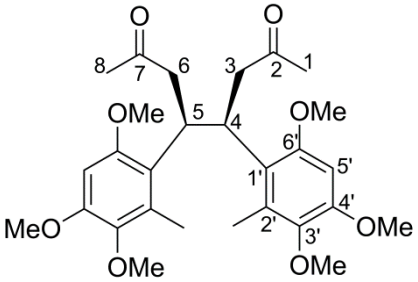
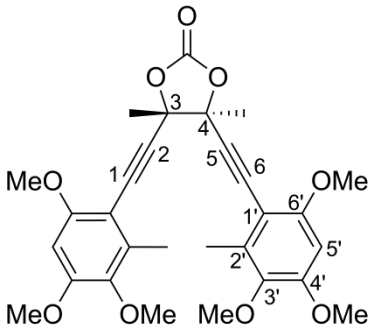
CONTENTS

I. 1) Crystallographic data and structural refinement for 50



Empirical formula	$C_{28}H_{38}O_8$
Formula weight	502.58
Temperature	100(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	a = 9.5690(2) Å
	b = 8.0724(2) Å
	c = 17.2904(3) Å
	β = 96.872(2)°
Volume	1326.00(5) $Å^3$
Z	2
Density (calculated)	1.259 Mg/m^3
Absorption coefficient	0.091 mm^{-1}
Crystal size	0.30 x 0.24 x 0.23 mm^3
θ range for data collection	3.92 to 34.85°.
Index ranges	-15<=h<=15, -12<=k<=12, -27<=l<=27
Reflections collected	30818
Independent reflections	5538 [R(int) = 0.0437]
Completeness to θ = 33.00°	99.8 %
Absorption correction	Semi-empirical from equivalents
Max./min. transmission	1.00/0.93
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5538 / 0 / 168
Goodness-of-fit on F^2	0.985
Final R indices [$I>2\sigma(I)$]	$R1$ = 0.0510, $wR2$ = 0.1371
R indices (all data)	$R1$ = 0.0854, $wR2$ = 0.1514
Largest diff. peak and hole	0.553 and -0.238 $e.Å^{-3}$

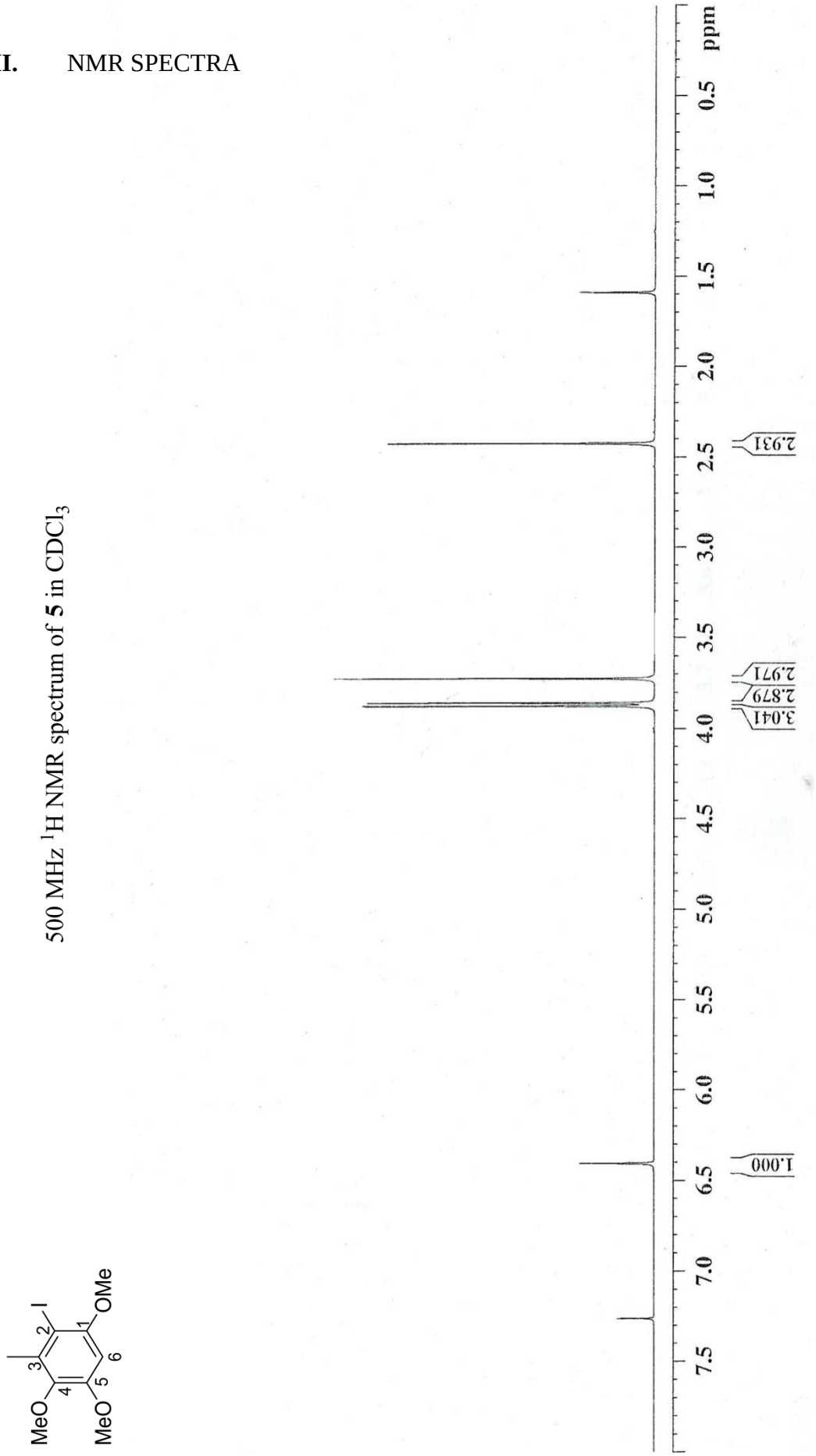
2) Crystallographic data and structural refinement for 51

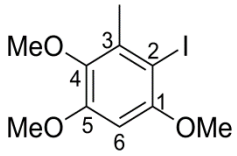


Empirical formula	$C_{29}H_{32}O_9$
Formula weight	524.55
Temperature	100(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	$P2_1$
Unit cell dimensions	a = 7.3517(19) Å
	b = 18.103(5) Å
	c = 10.012(3) Å
	β = 96.47(2)°
Volume	1323.9(6) $Å^3$
Z	2
Density (calculated)	1.316 Mg/m^3
Absorption coefficient	0.098 mm^{-1}
Crystal size	0.31 x 0.16 x 0.09 mm^3
θ range for data collection	2.79 to 25.00°.
Index ranges	-4<=h<=8, -21<=k<=21, -11<=l<=9
Reflections collected	7957
Independent reflections	2406 [R(int) = 0.0820]
Completeness to θ = 25.00°	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.00/0.93
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2406 / 235 / 357
Goodness-of-fit on F^2	1.412
Final R indices [I>2σ(I)]	$R1$ = 0.1458, $wR2$ = 0.3562
R indices (all data)	$R1$ = 0.1602, $wR2$ = 0.3683
Largest diff. peak and hole	1.305 and -0.504 e.$Å^{-3}$

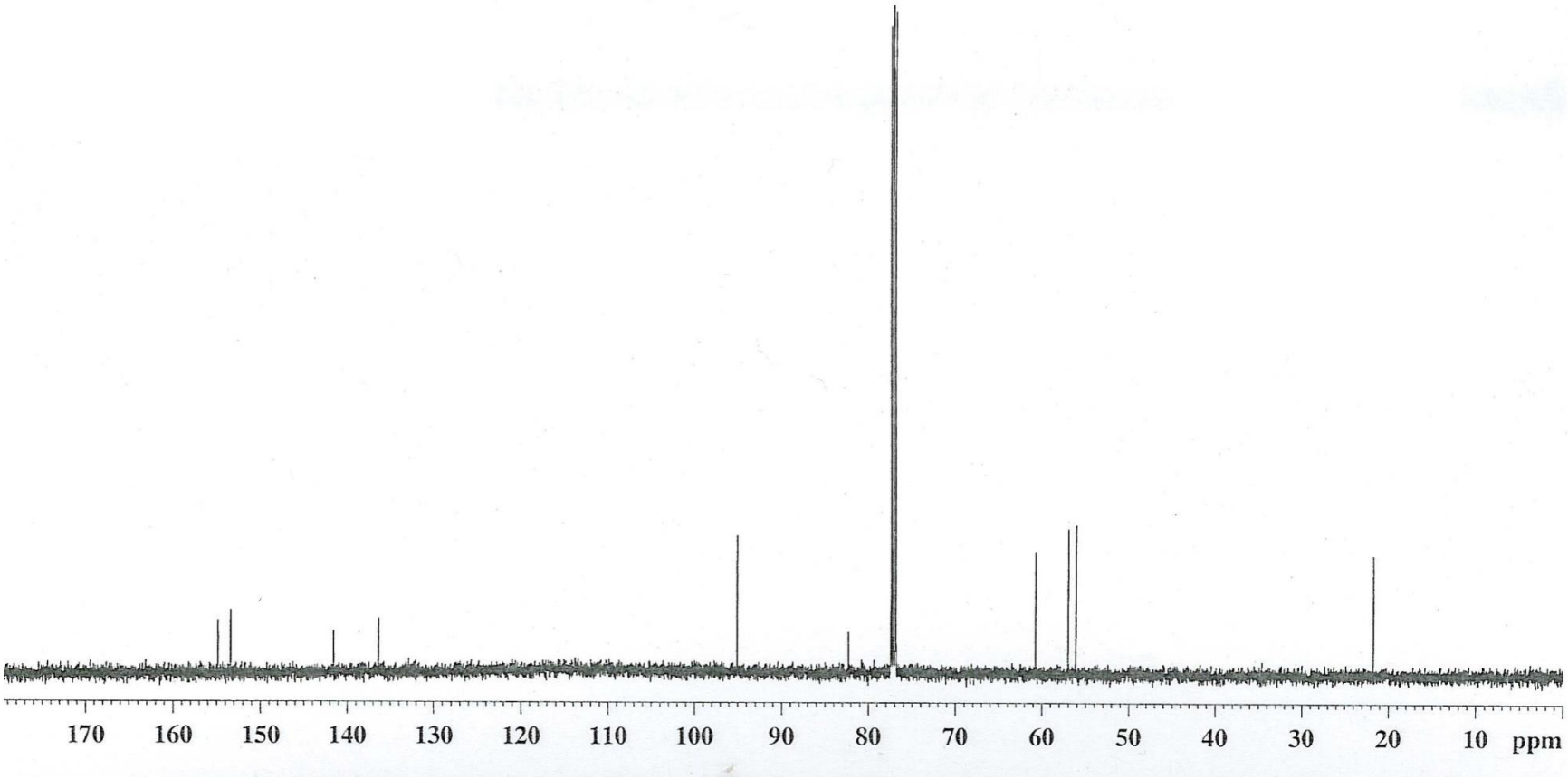
II. NMR SPECTRA

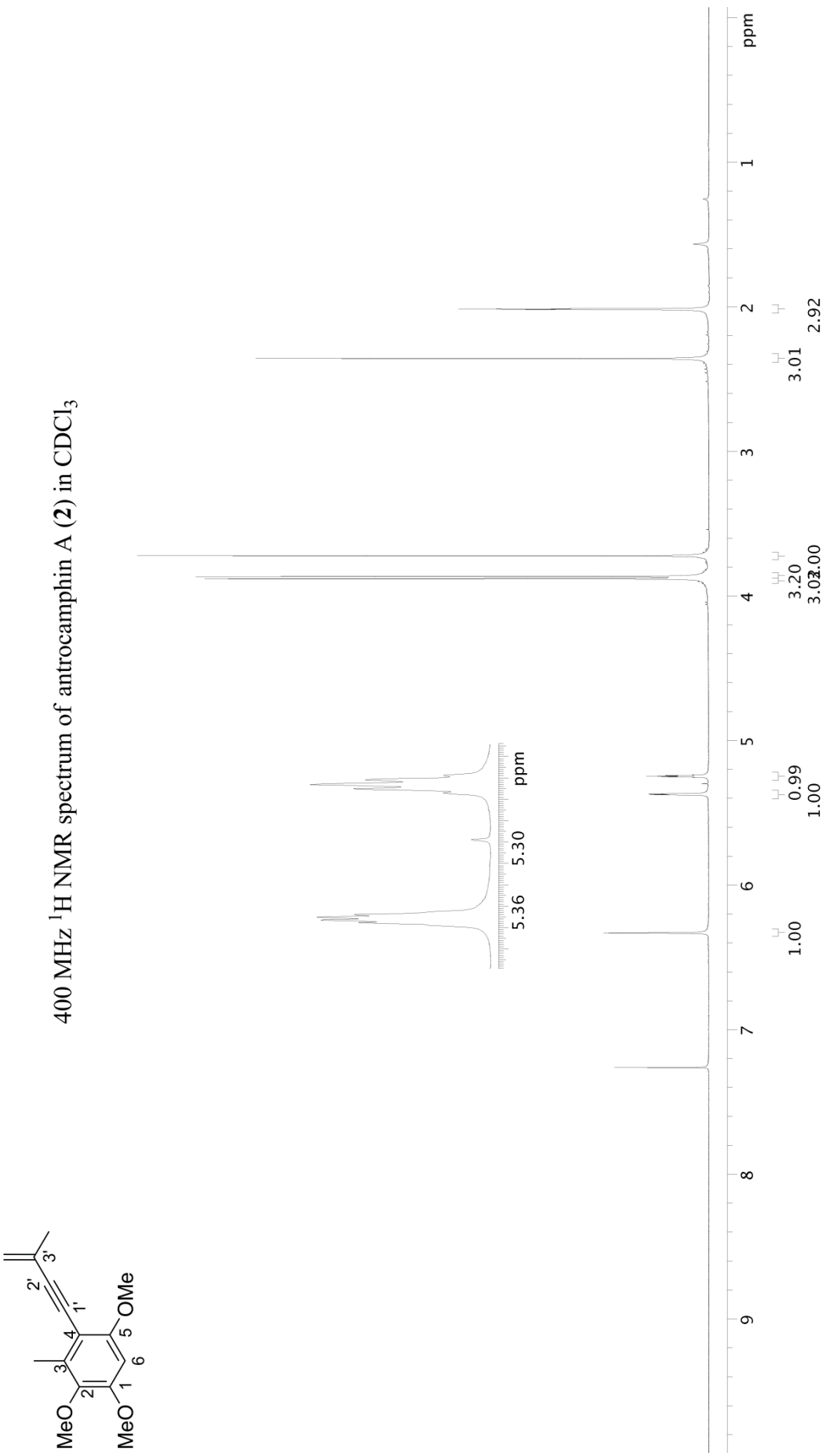
500 MHz ^{1}H NMR spectrum of **5** in $CDCl_3$



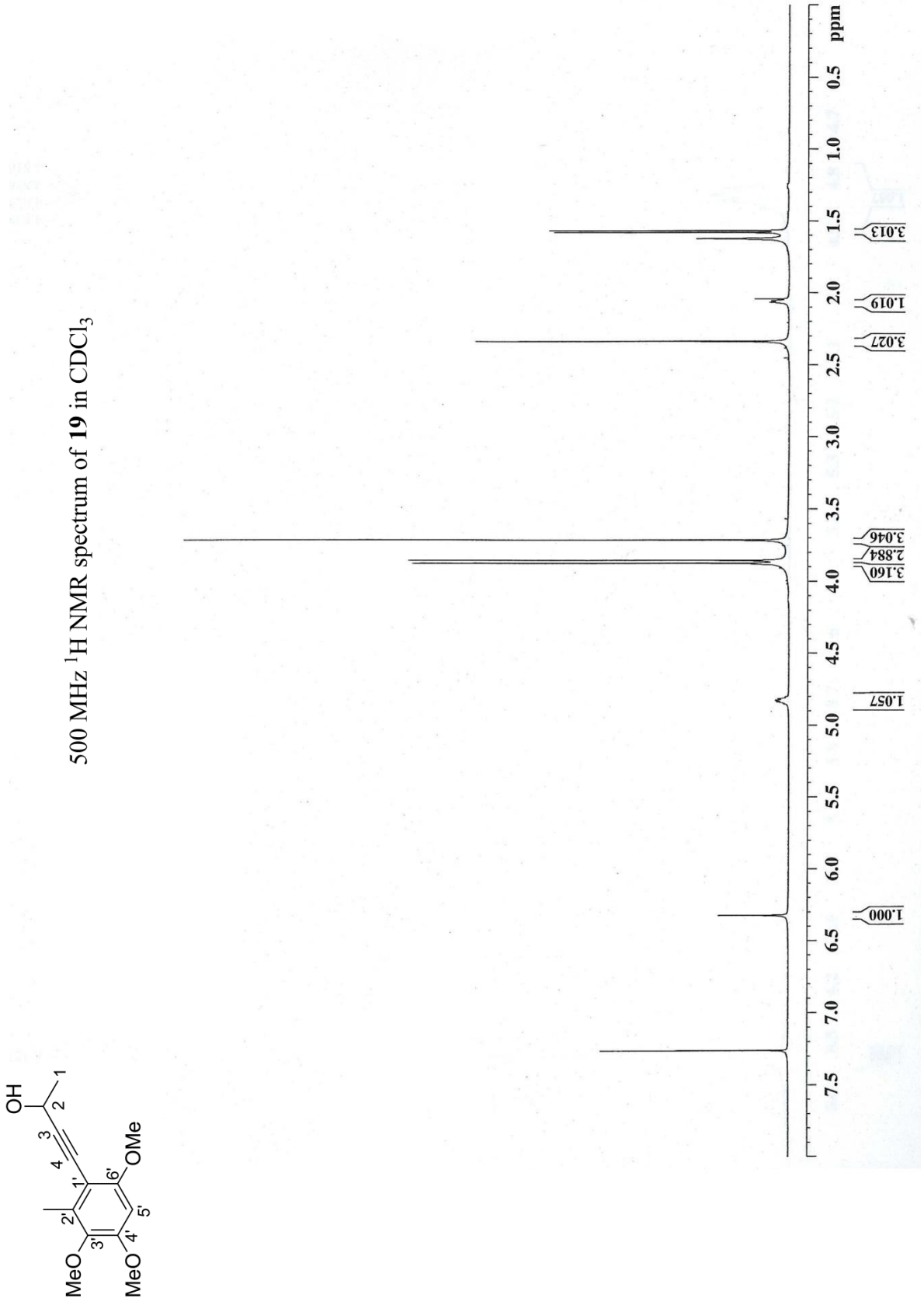


125 MHz ^{13}C NMR spectrum of **5** in $CDCl_3$

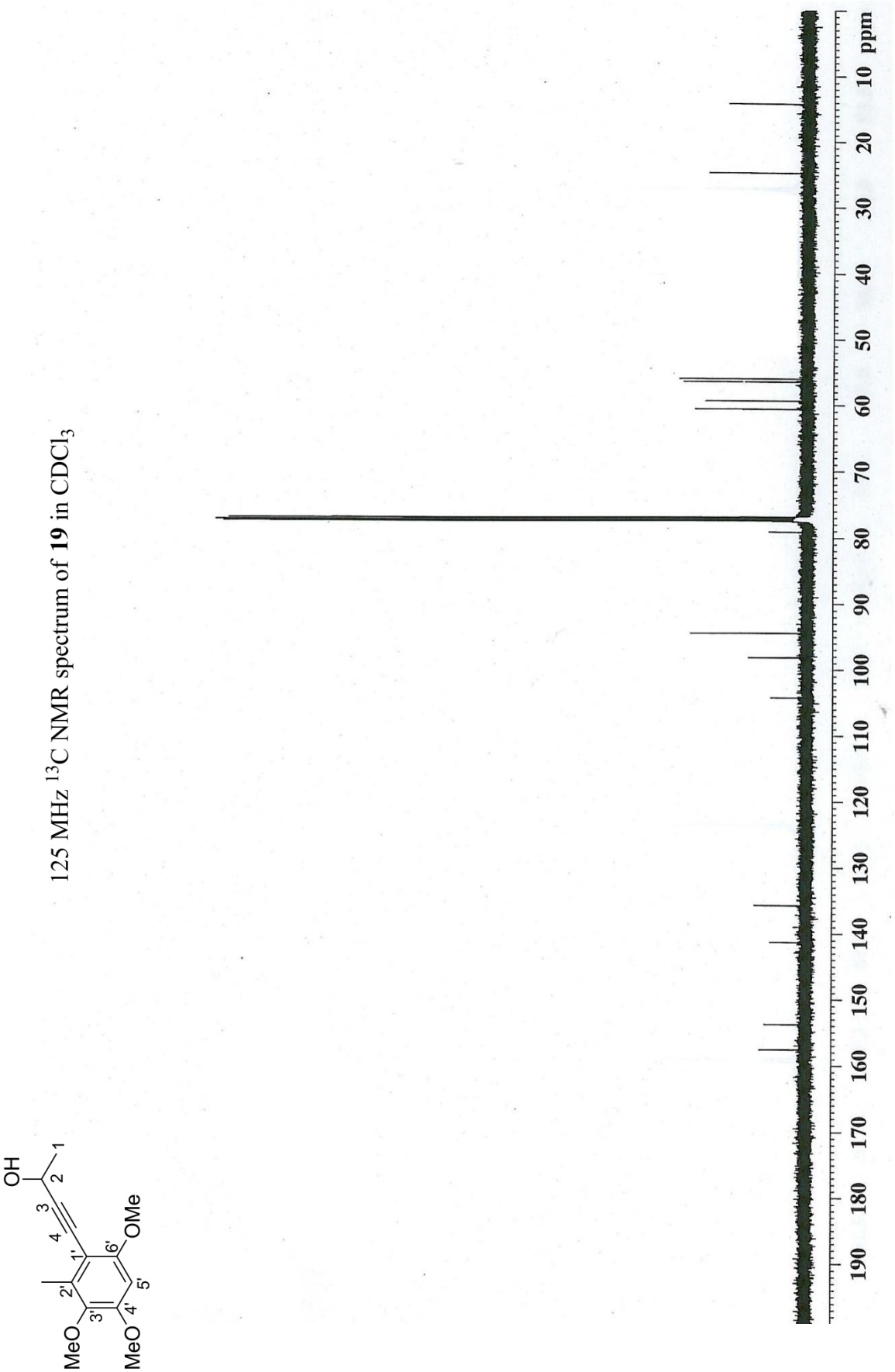



400 MHz ^{1}H NMR spectrum of antrocamphin A (**2**) in $CDCl_3$
MeO
MeO
OMe
1
2
3
4
5
6
1'
2'
3'
ppm
1.00
0.99
1.00
3.20
3.03
3.00
3.01
2.92
5.36
5.30

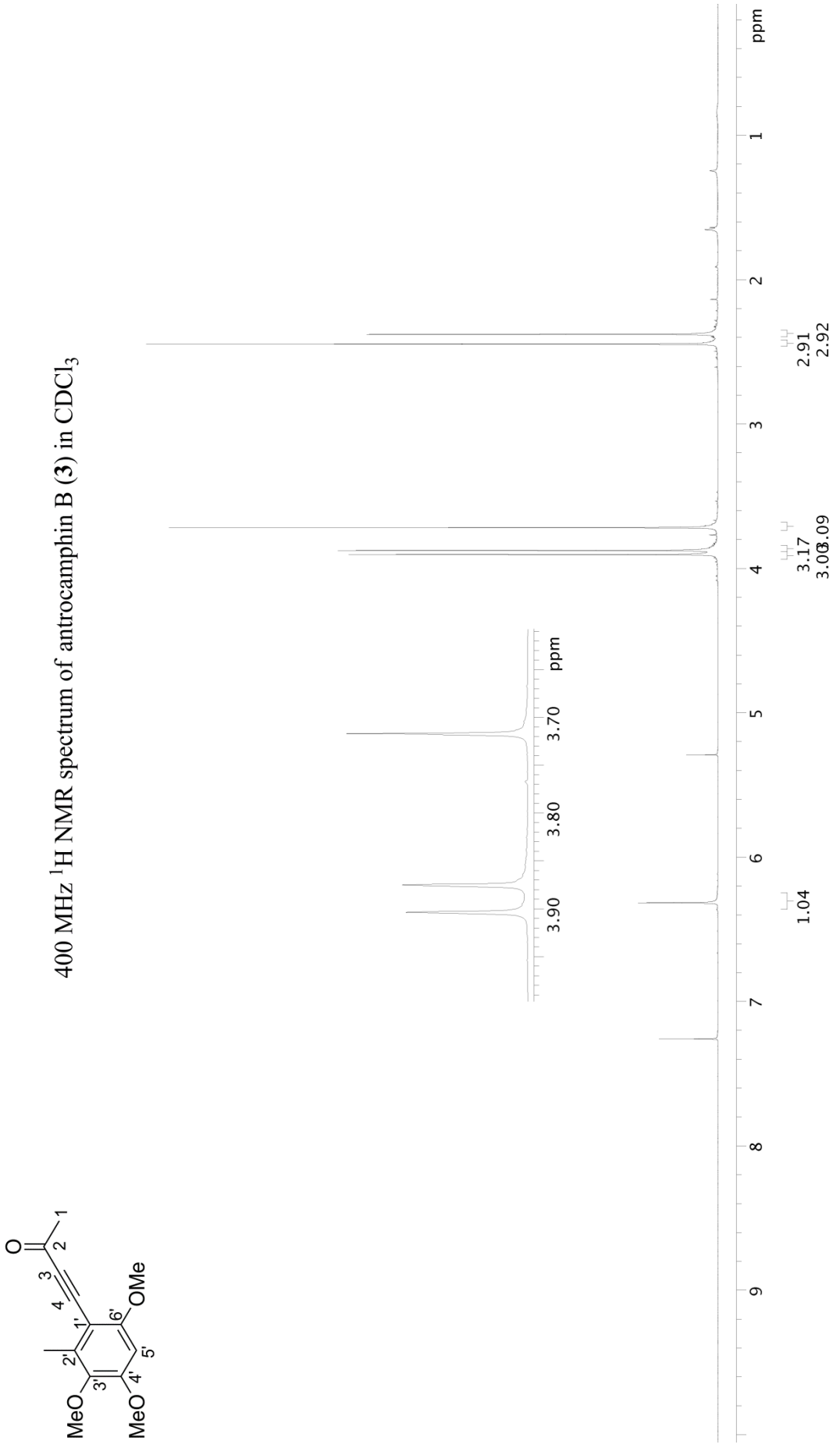
500 MHz ^{1}H NMR spectrum of **19** in $CDCl_3$



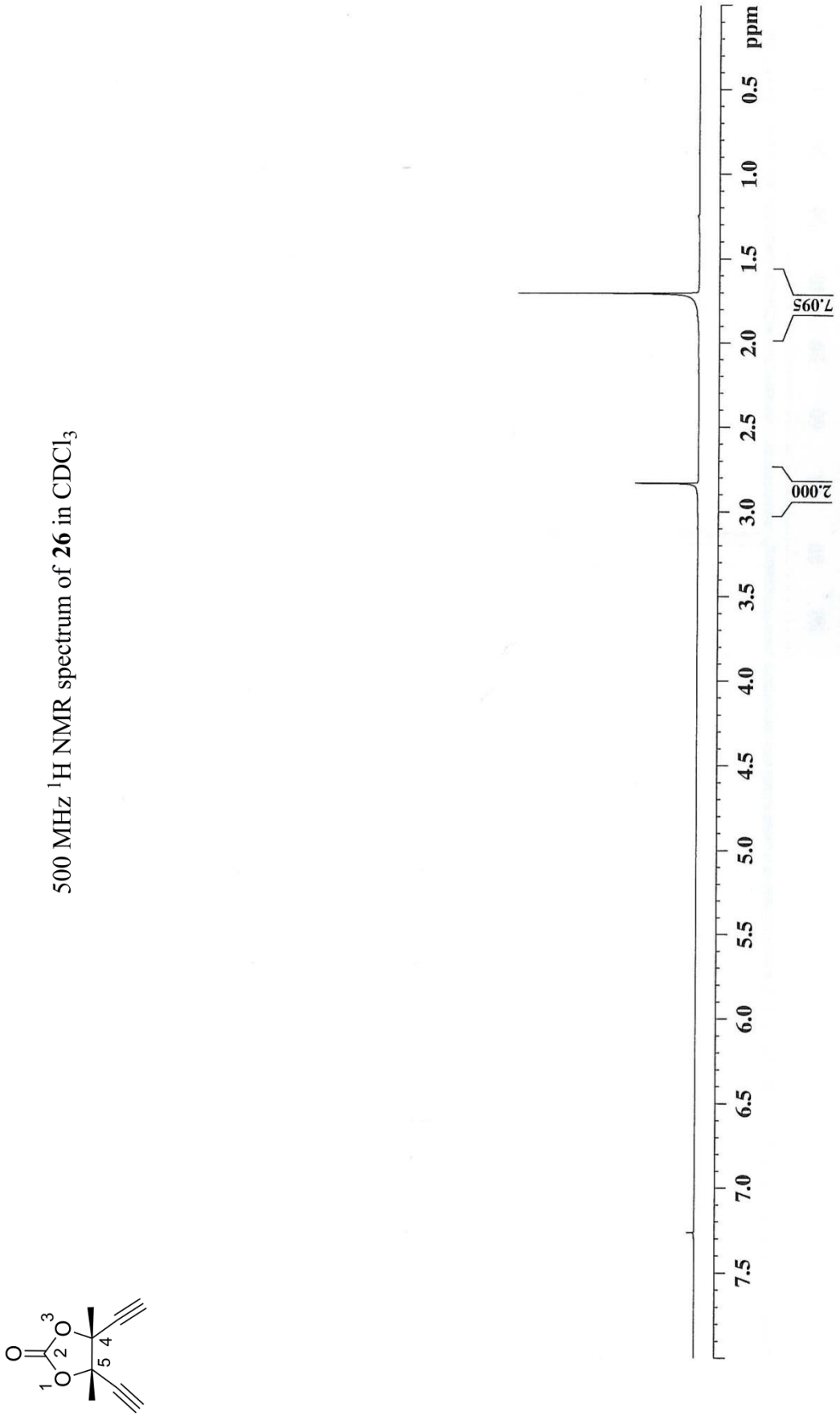
125 MHz ^{13}C NMR spectrum of **19** in $CDCl_3$



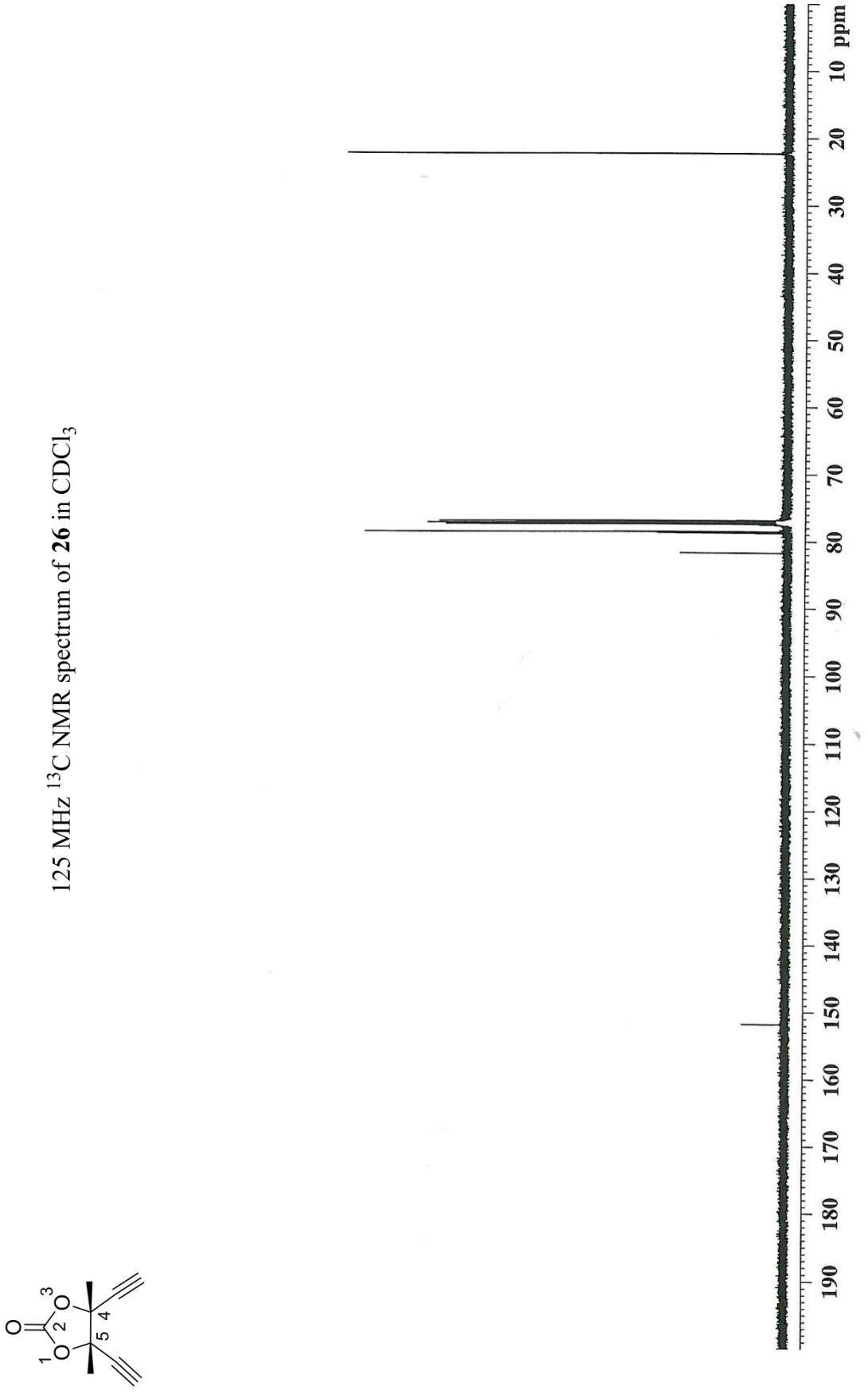
400 MHz ^{1}H NMR spectrum of antrocamphin B (**3**) in $CDCl_3$



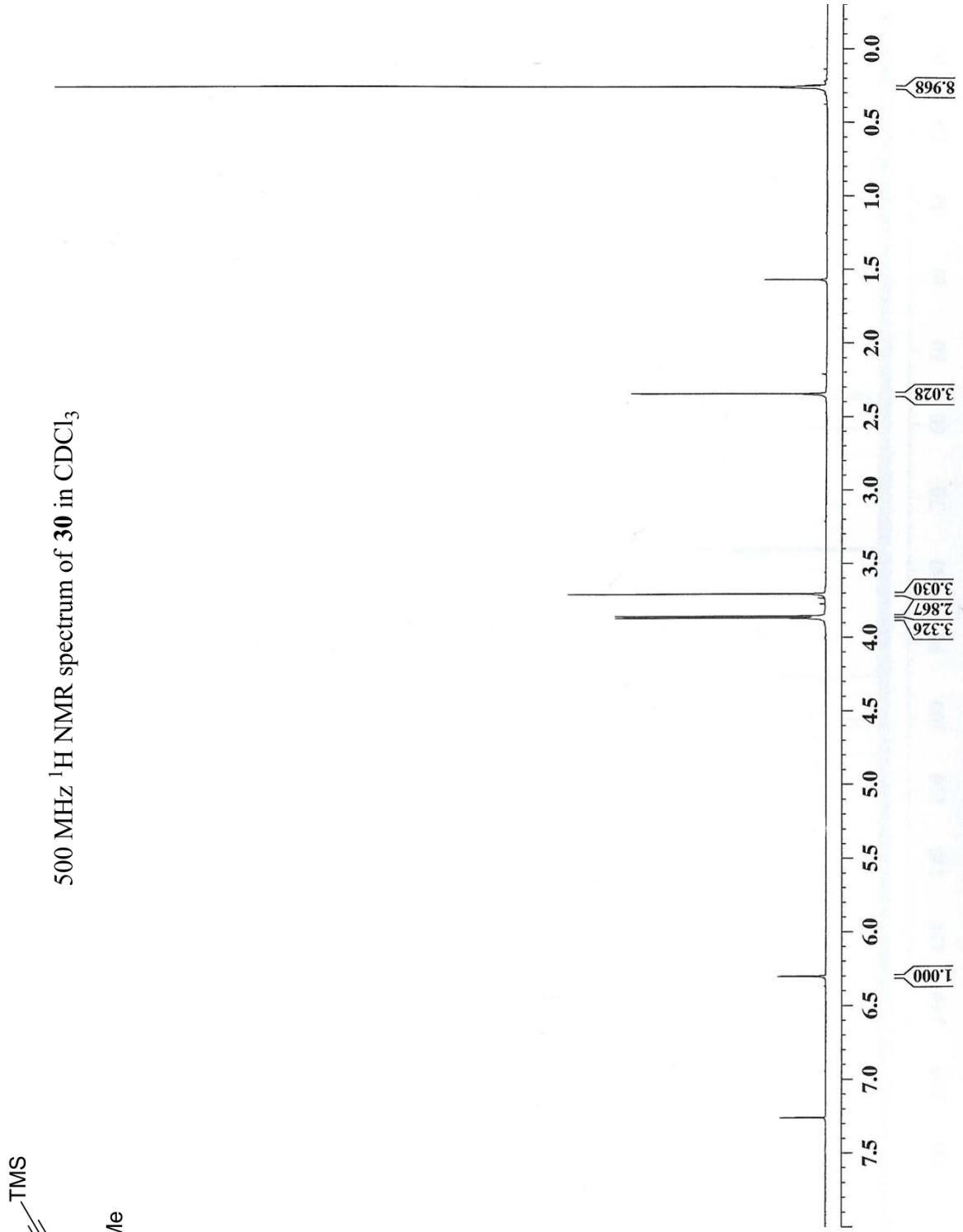
500 MHz ^{1}H NMR spectrum of **26** in $CDCl_3$

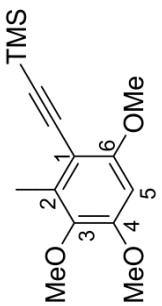


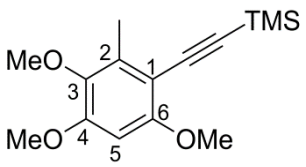
125 MHz ^{13}C NMR spectrum of **26** in $CDCl_3$



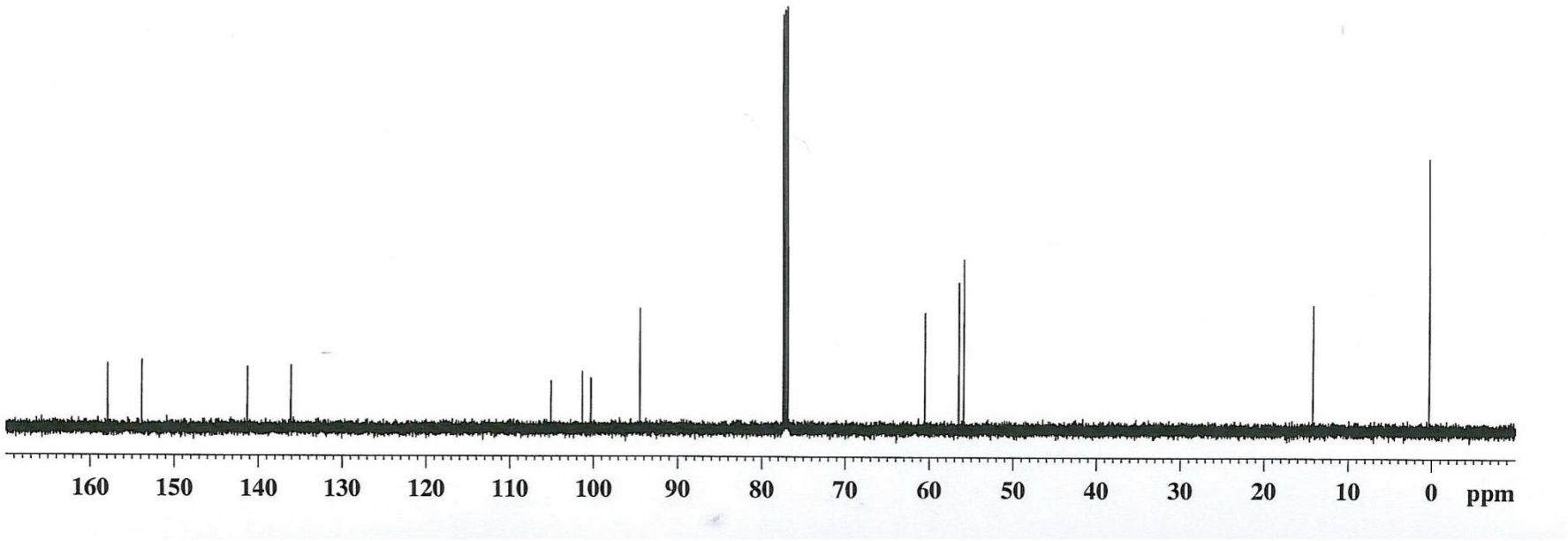
500 MHz ^{1}H NMR spectrum of **30** in $CDCl_3$



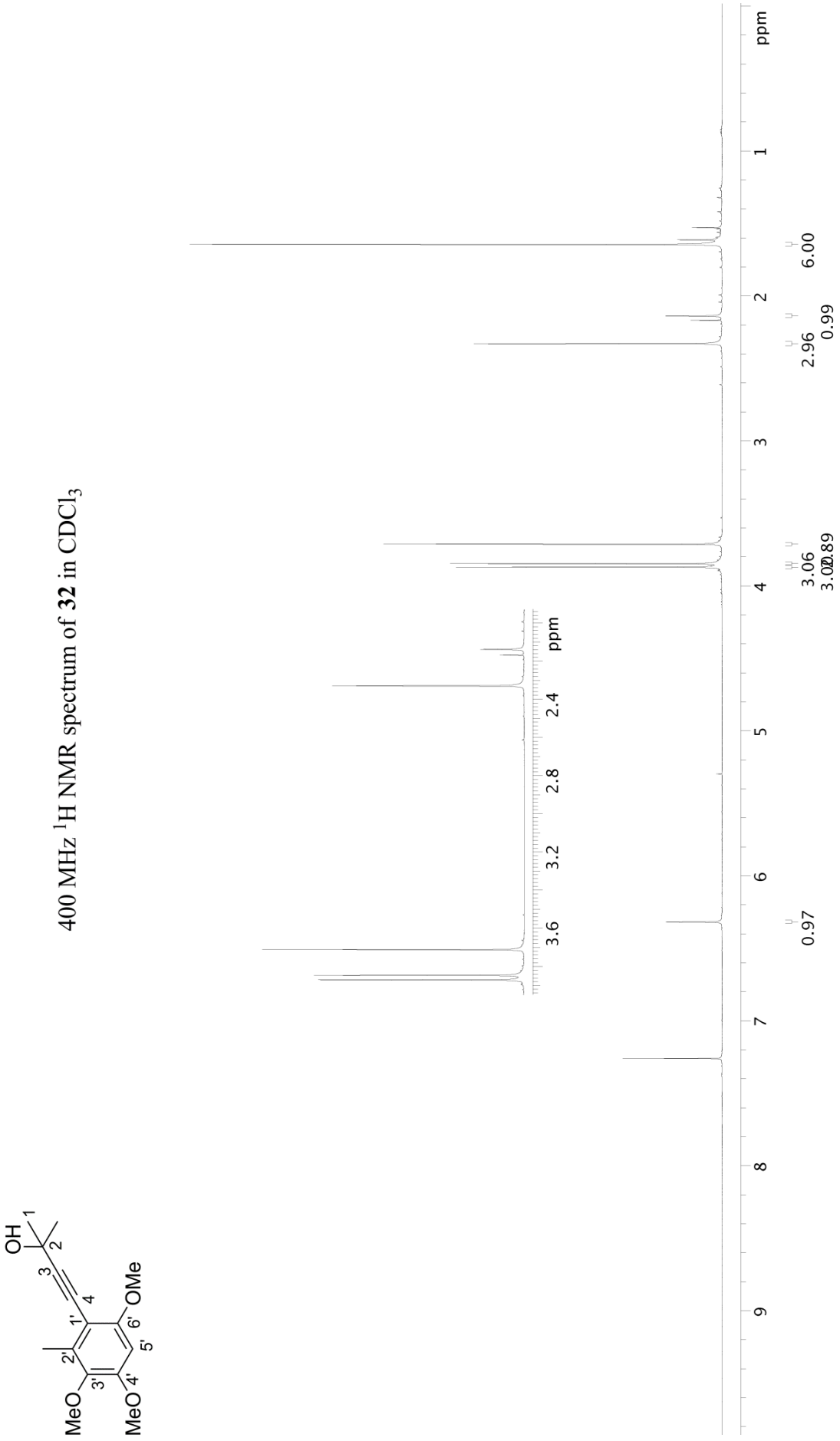




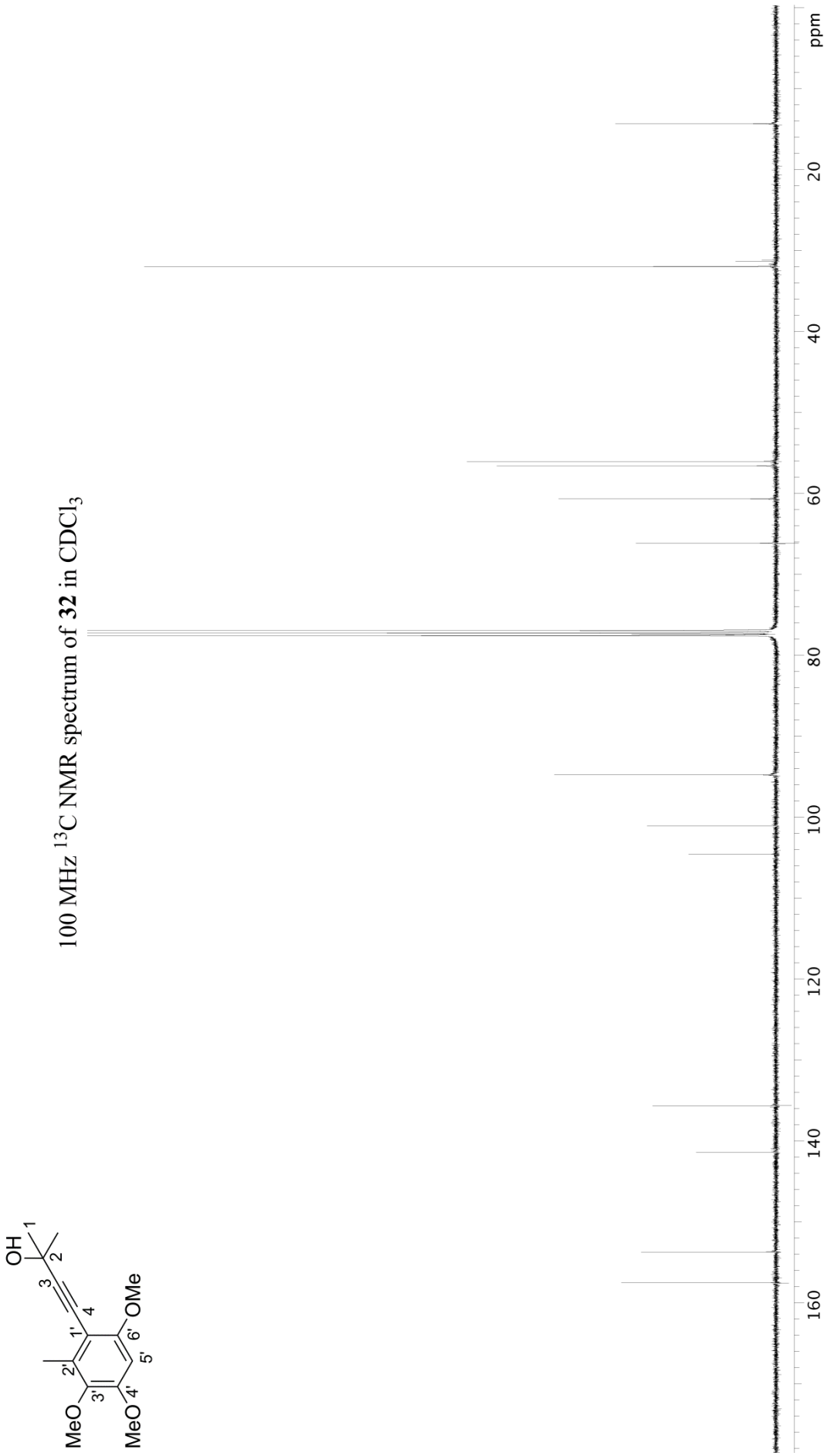
125 MHz ^{13}C NMR spectrum of **30** in $CDCl_3$



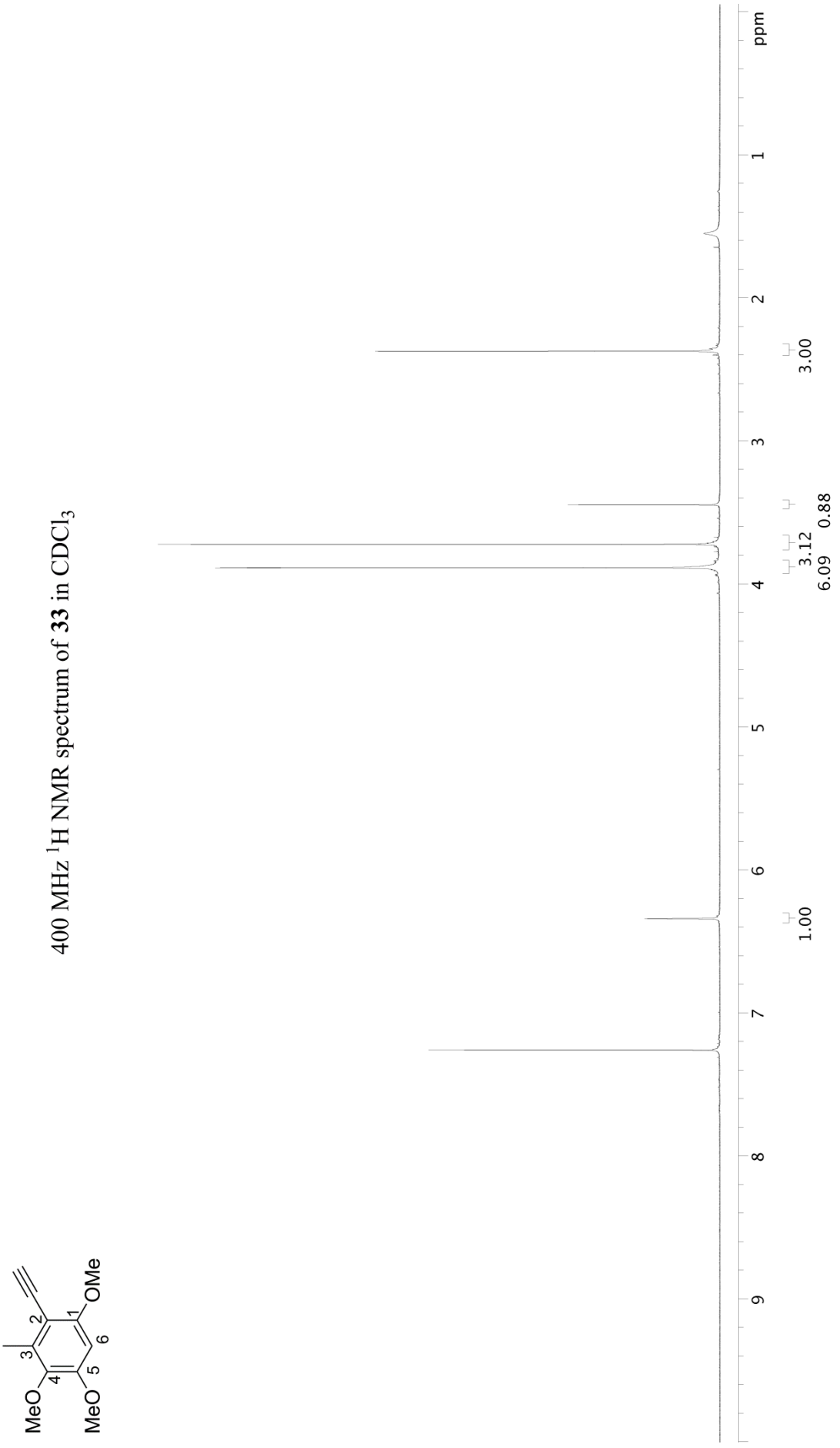
400 MHz ^{1}H NMR spectrum of **32** in $CDCl_3$



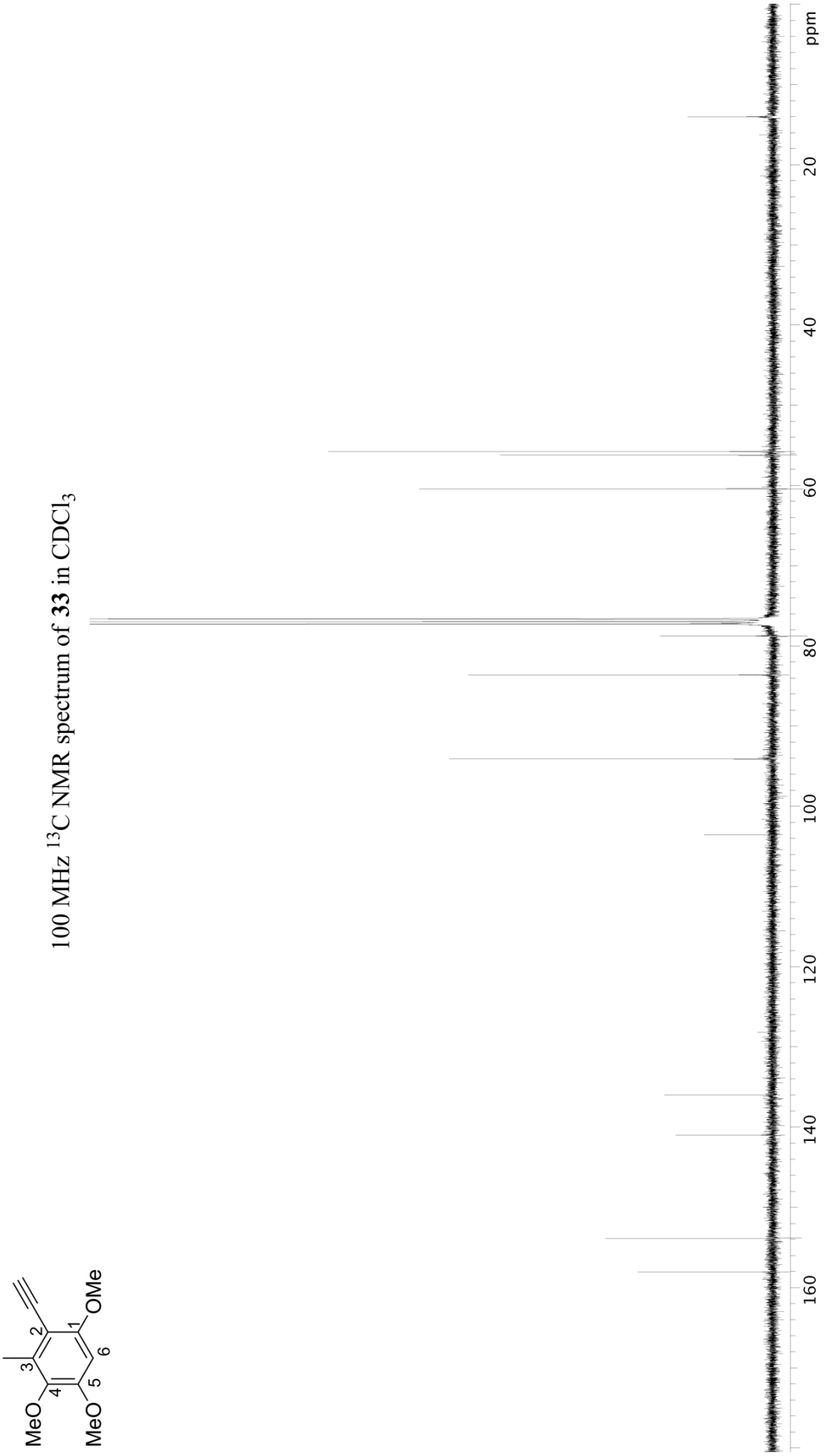
100 MHz ^{13}C NMR spectrum of **32** in $CDCl_3$



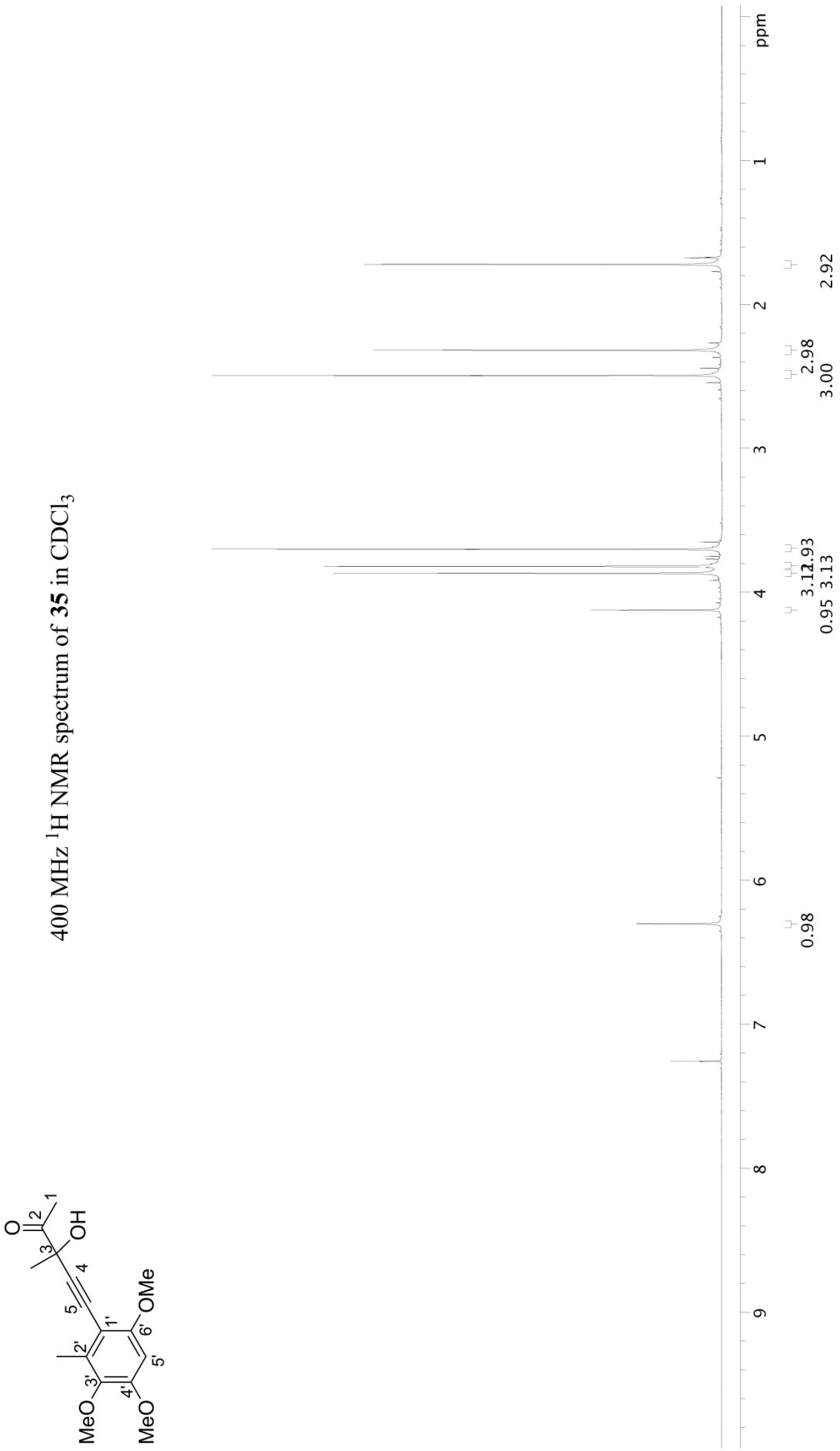
400 MHz ^{1}H NMR spectrum of **33** in $CDCl_3$



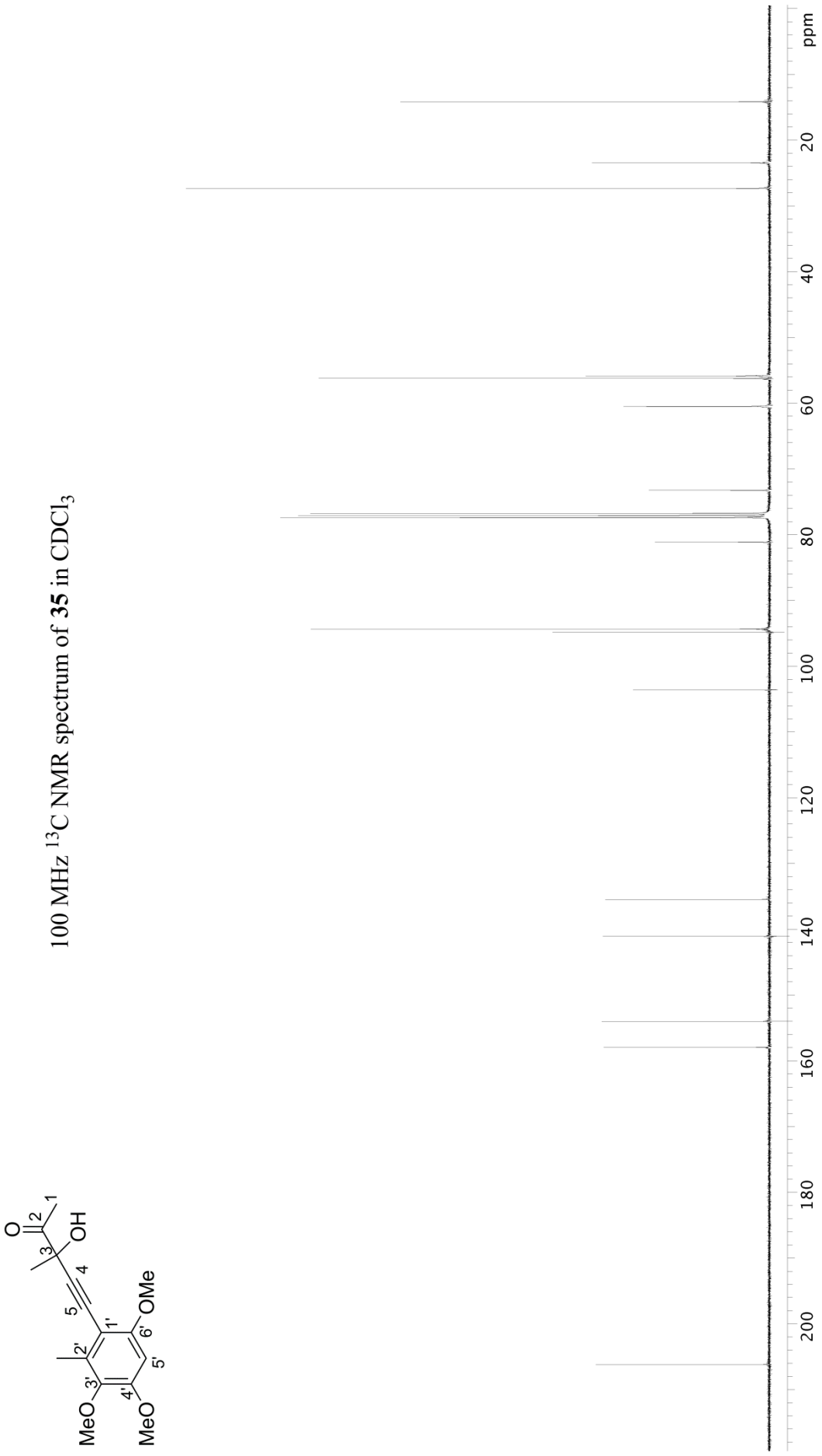
100 MHz ^{13}C NMR spectrum of **33** in $CDCl_3$



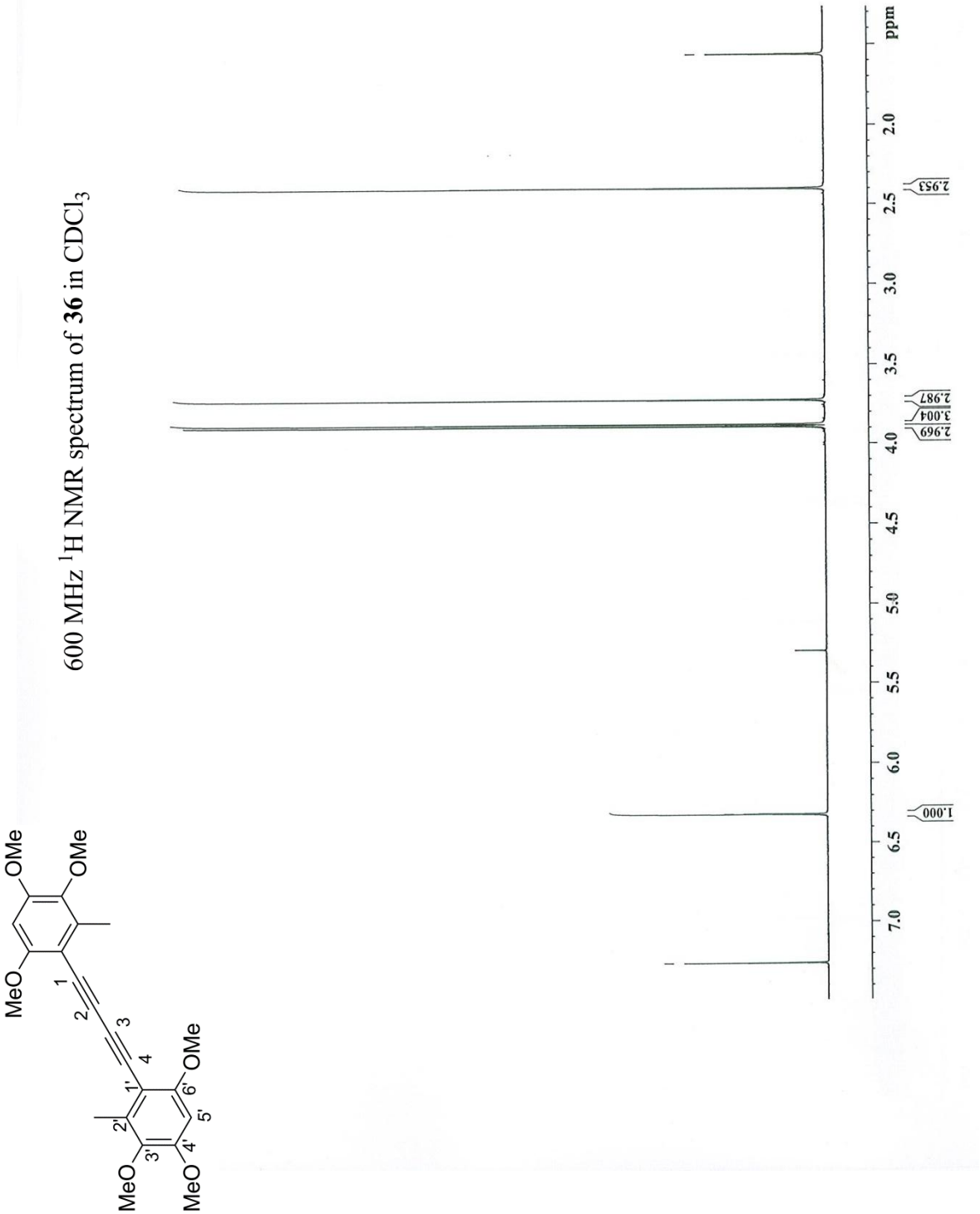
400 MHz ^{1}H NMR spectrum of **35** in $CDCl_3$



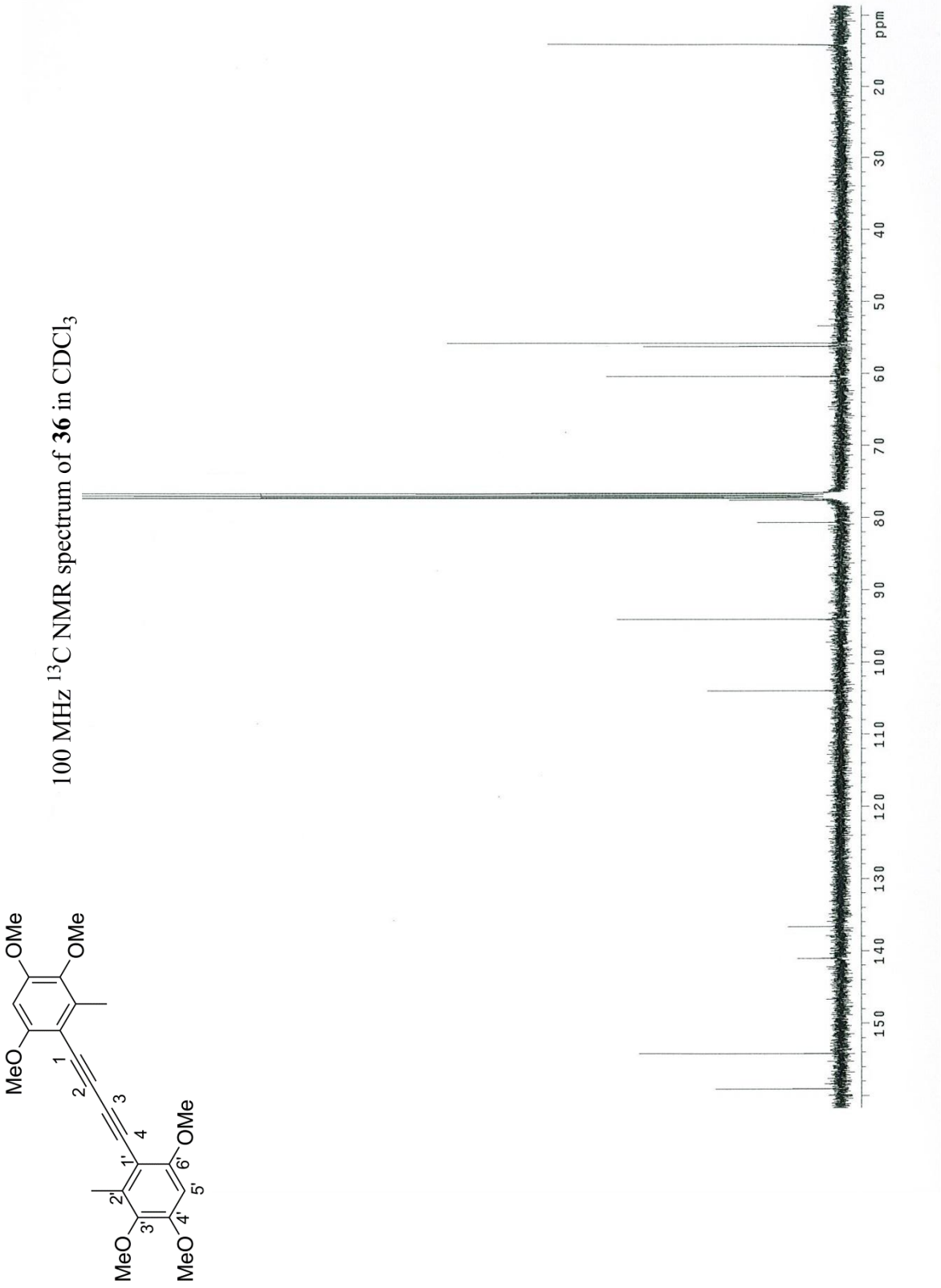
100 MHz ^{13}C NMR spectrum of **35** in $CDCl_3$



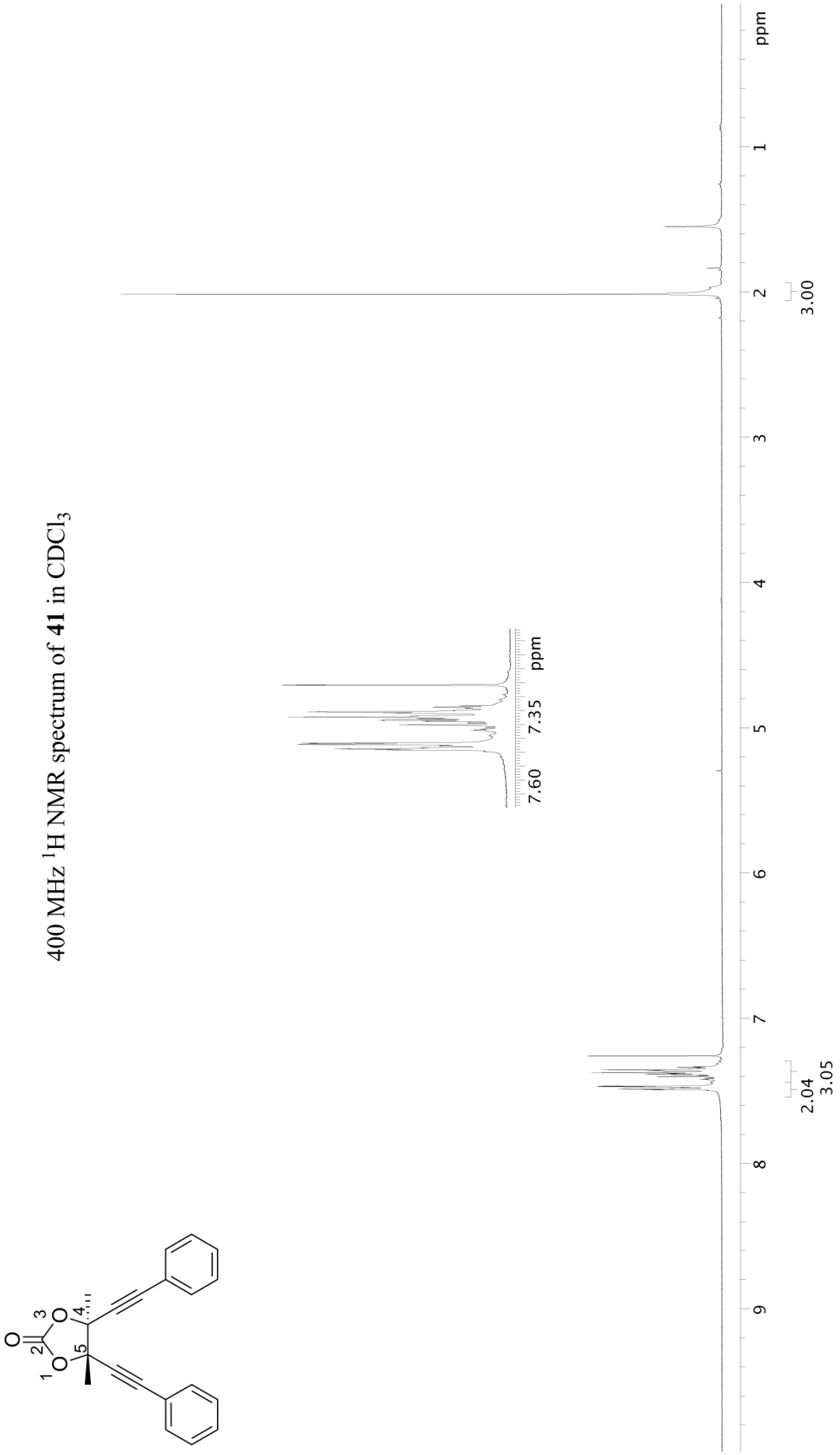
600 MHz ^{1}H NMR spectrum of **36** in $CDCl_3$



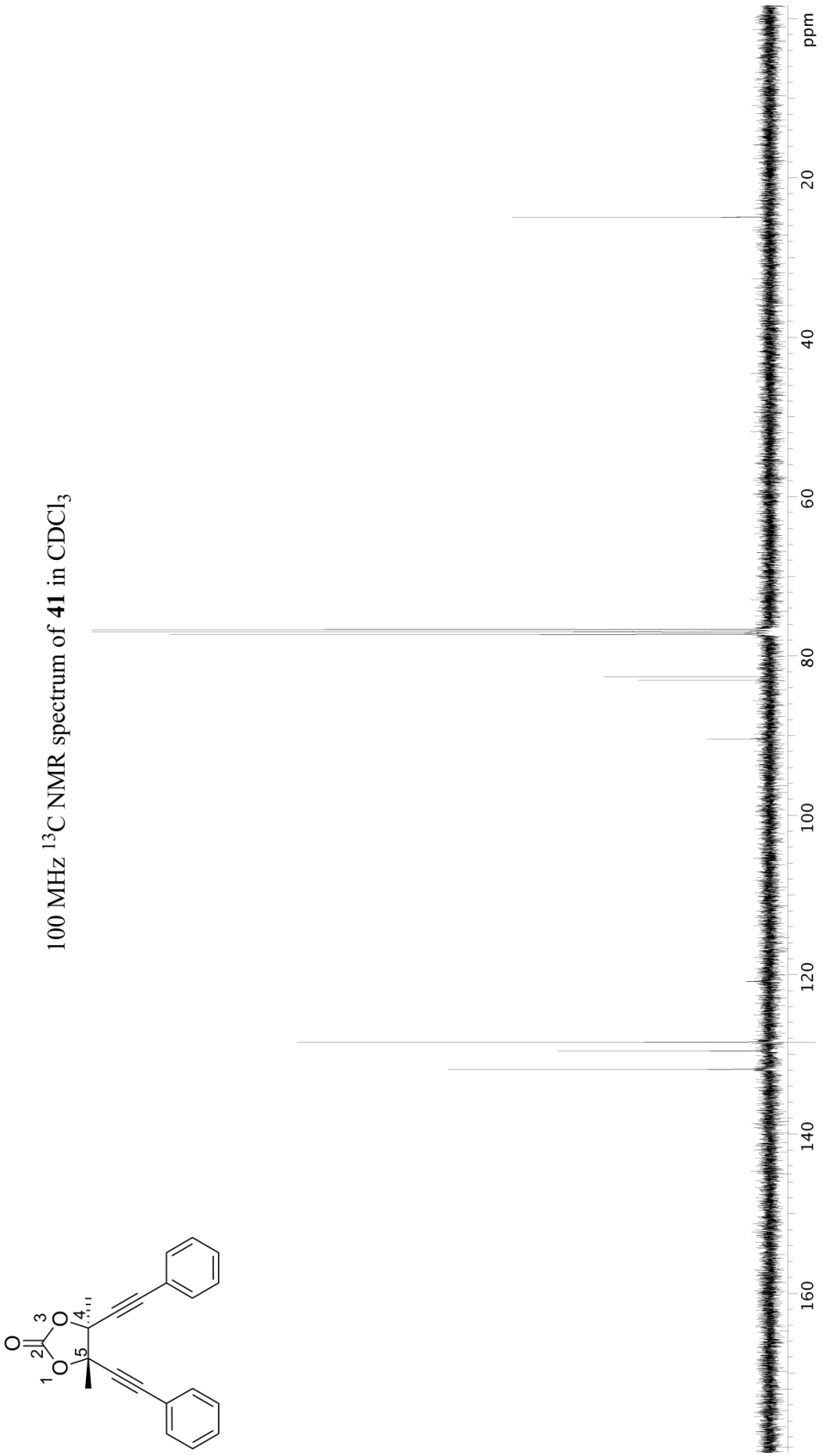
100 MHz ^{13}C NMR spectrum of **36** in $CDCl_3$



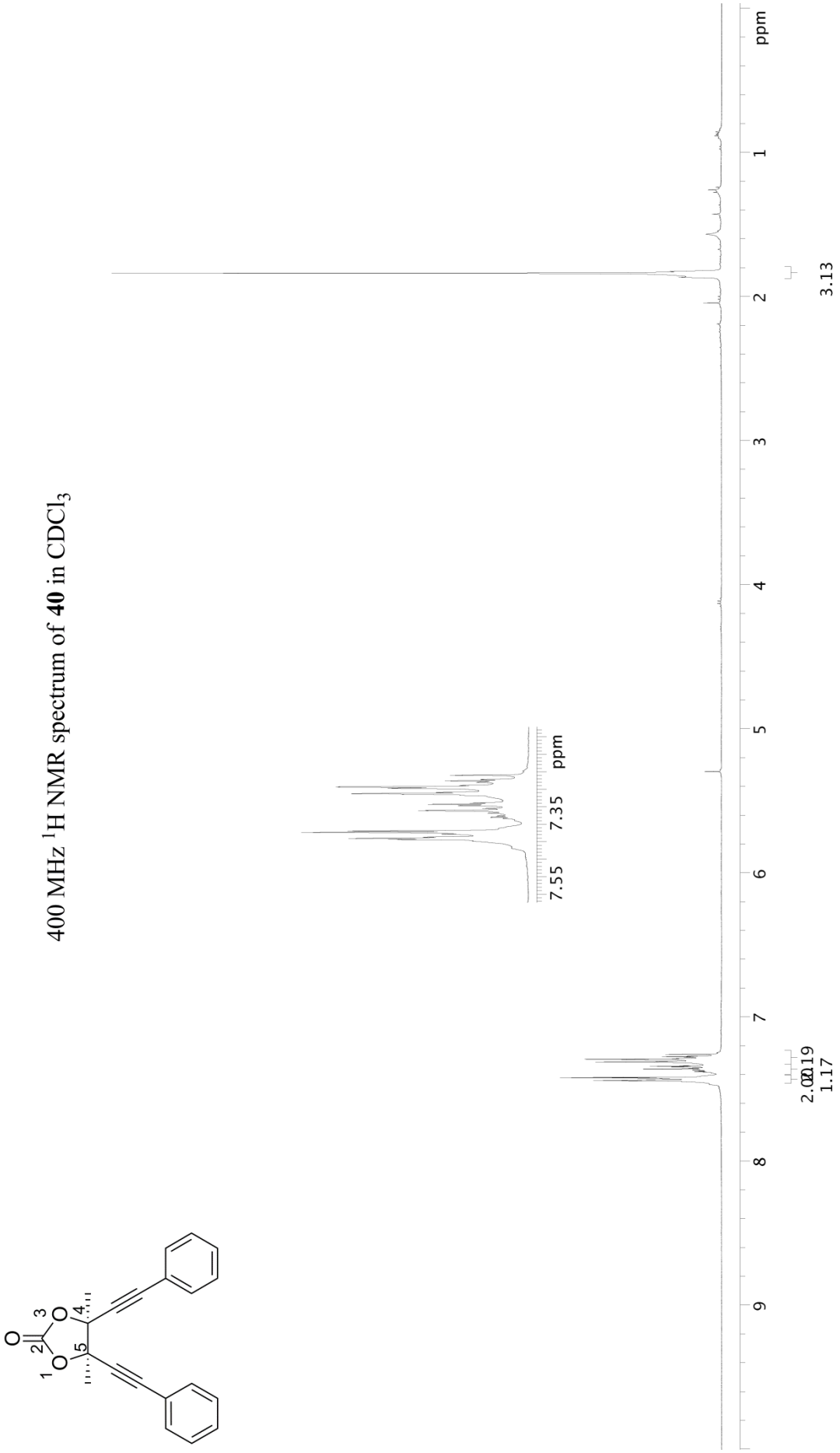
400 MHz ^{1}H NMR spectrum of **41** in $CDCl_3$



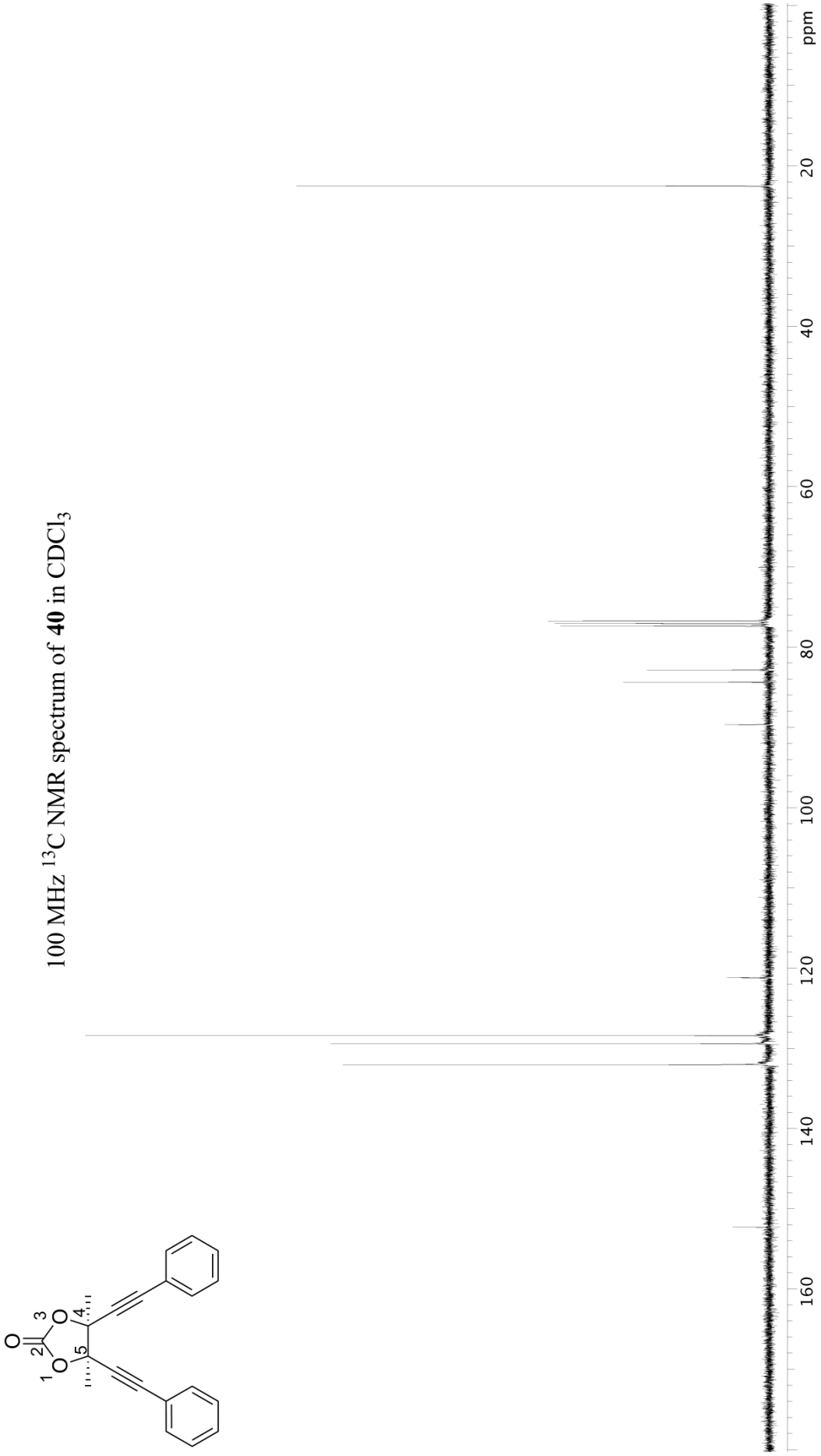
100 MHz ^{13}C NMR spectrum of **41** in $CDCl_3$

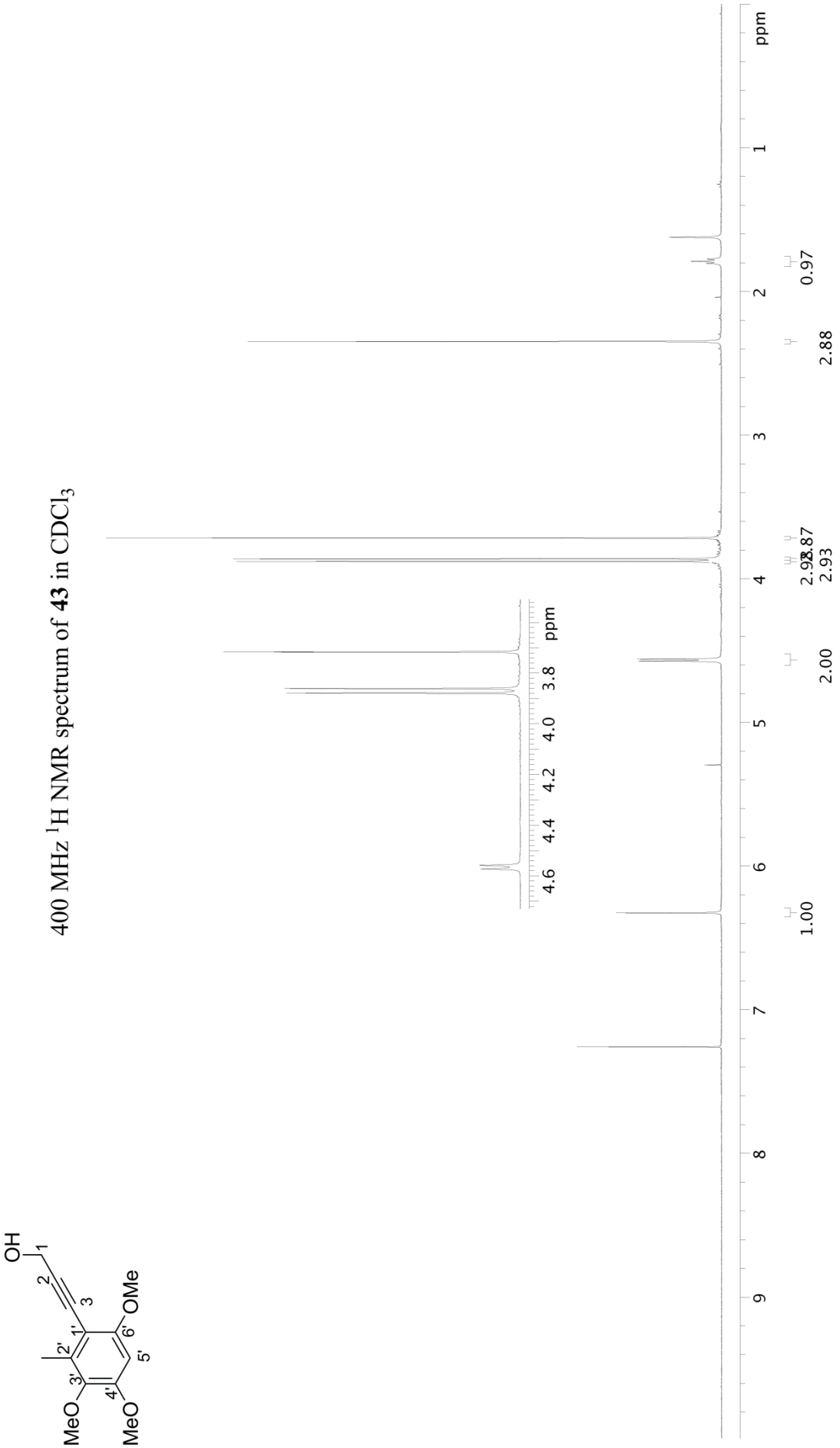


400 MHz ^{1}H NMR spectrum of **40** in $CDCl_3$

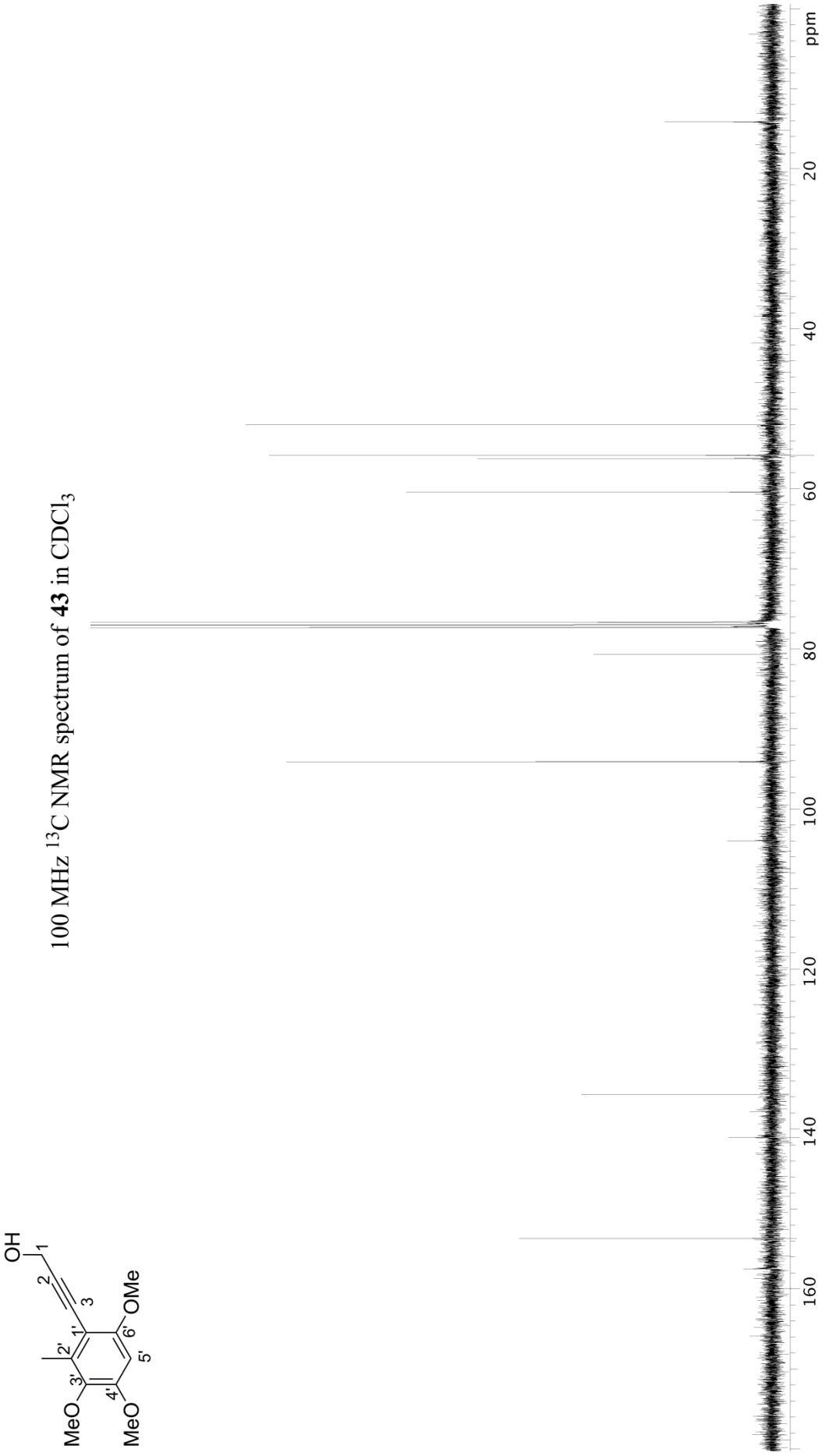


100 MHz ^{13}C NMR spectrum of **40** in $CDCl_3$

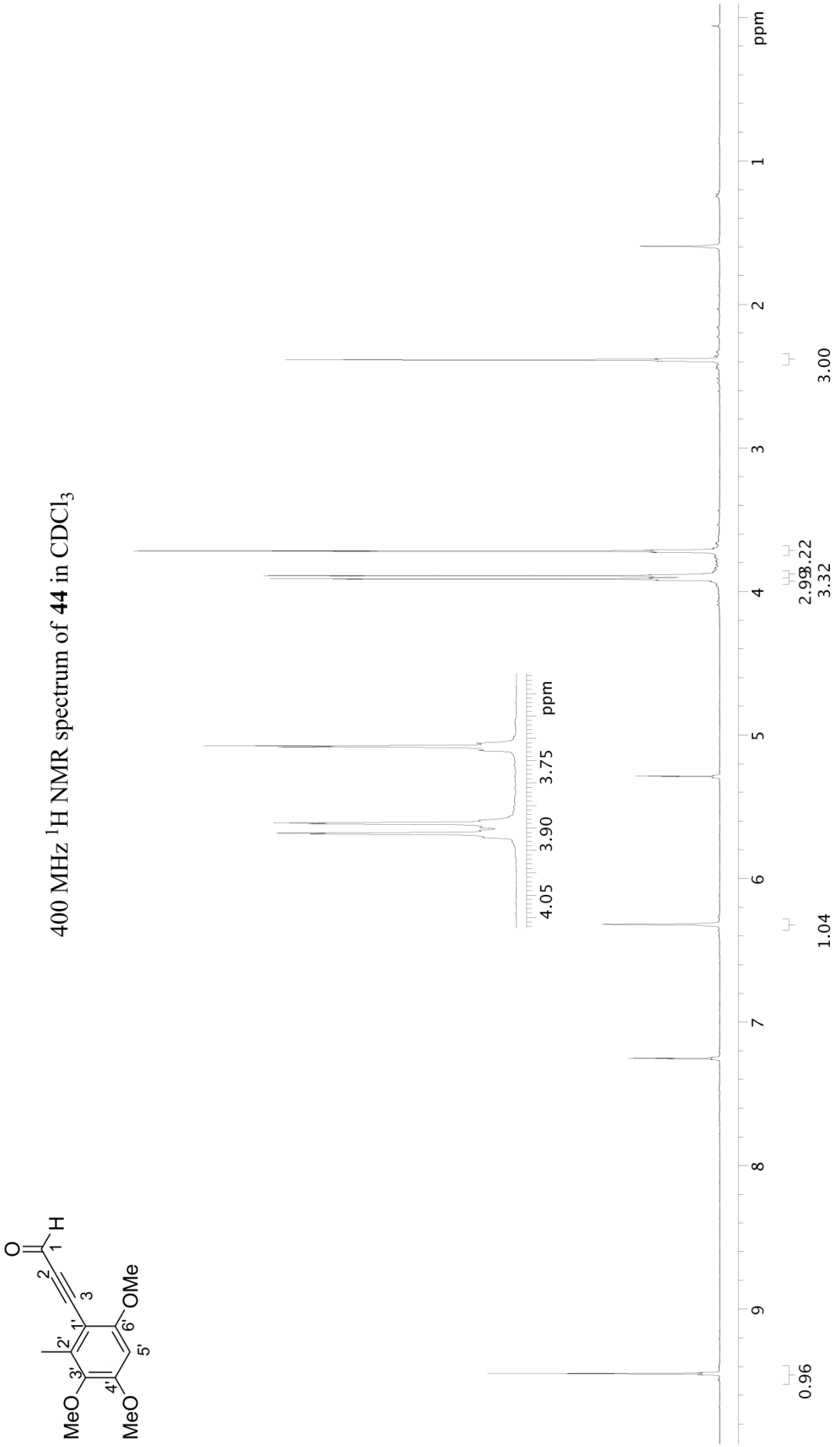



400 MHz ^{1}H NMR spectrum of **43** in $CDCl_3$
OH
1
2
3
1'
2'
3'
4'
5'
6'
OMe
MeO
MeO
ppm
1.00
2.00
2.93
2.87
2.88
0.97

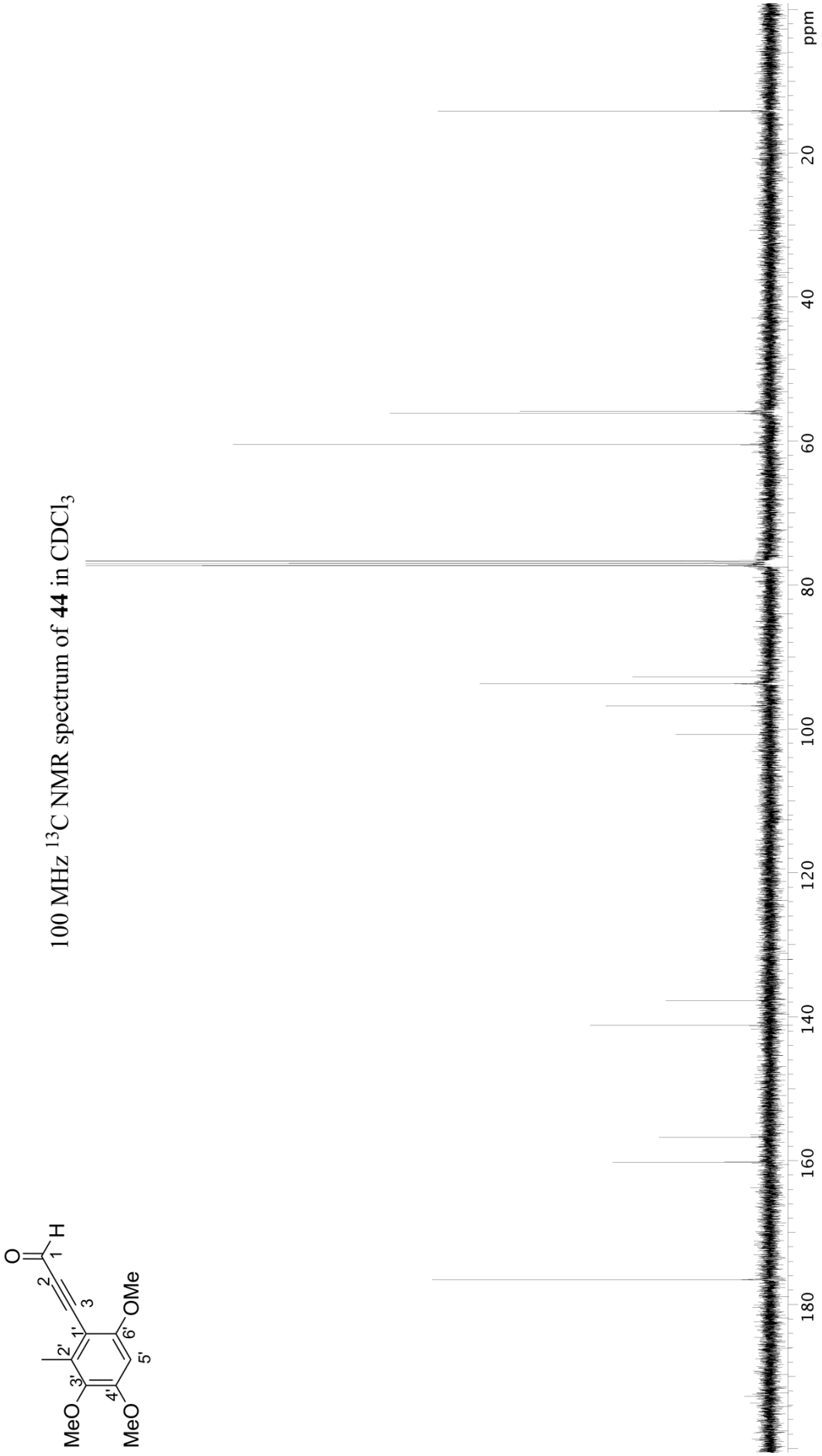
100 MHz ^{13}C NMR spectrum of **43** in $CDCl_3$



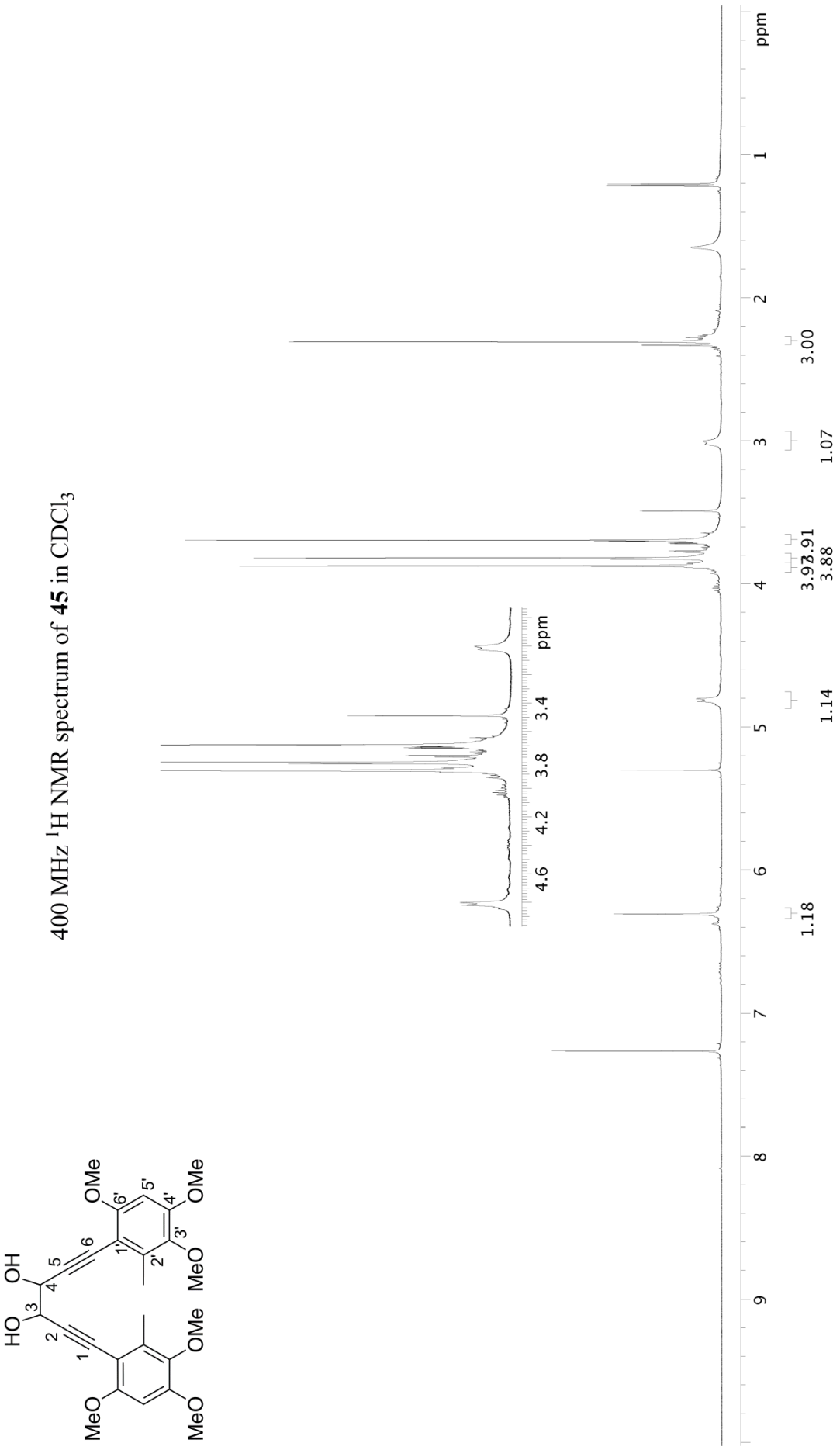
400 MHz ^{1}H NMR spectrum of **44** in $CDCl_3$



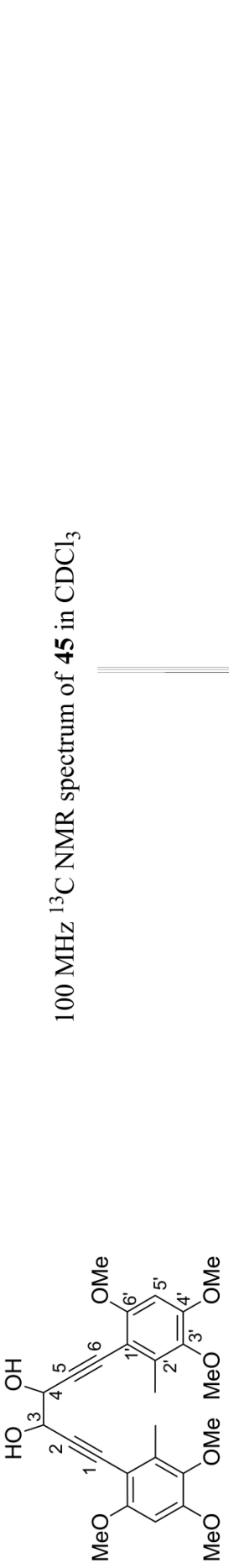
100 MHz ^{13}C NMR spectrum of **44** in $CDCl_3$

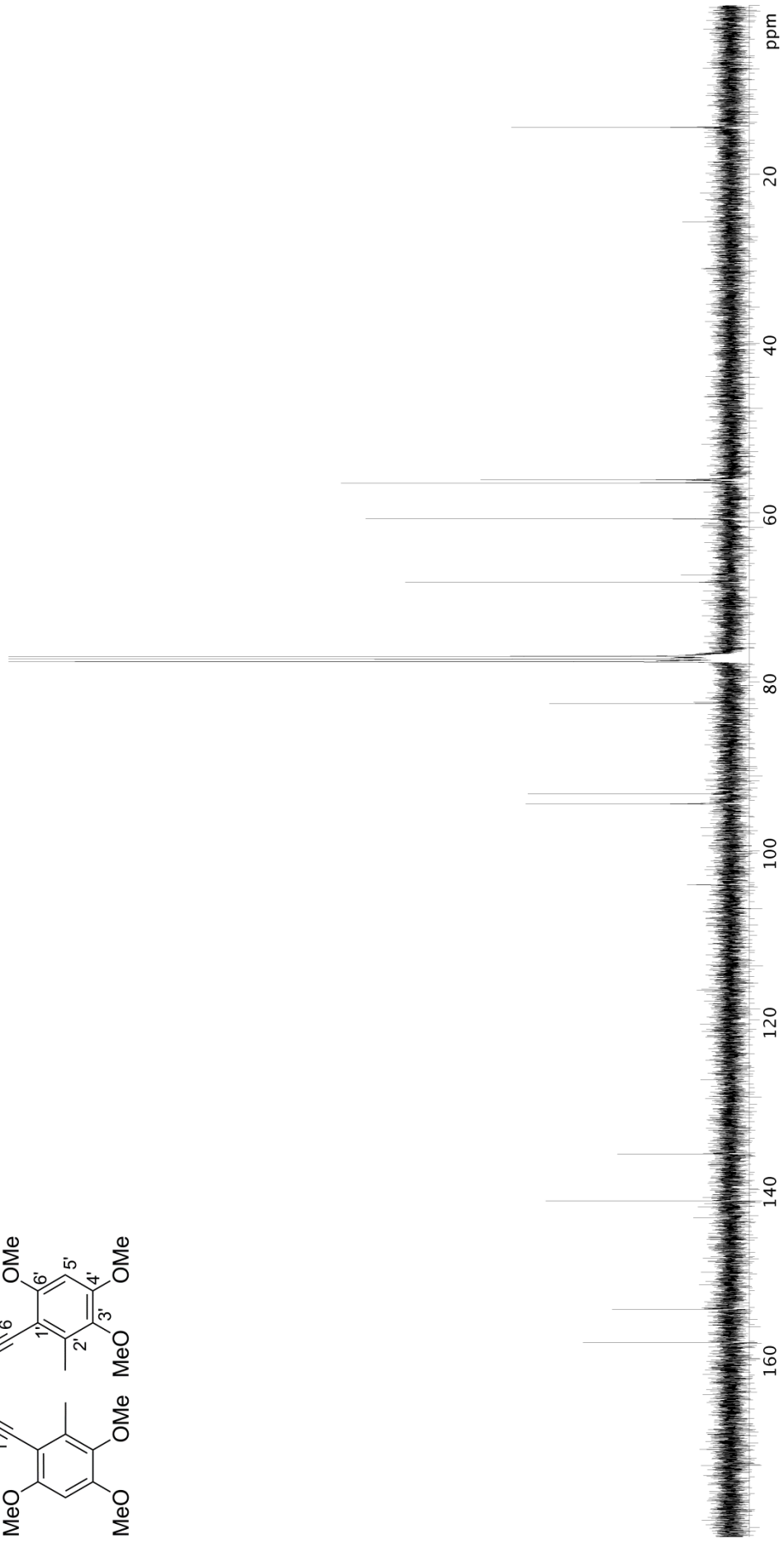


400 MHz ^{1}H NMR spectrum of **45** in $CDCl_3$

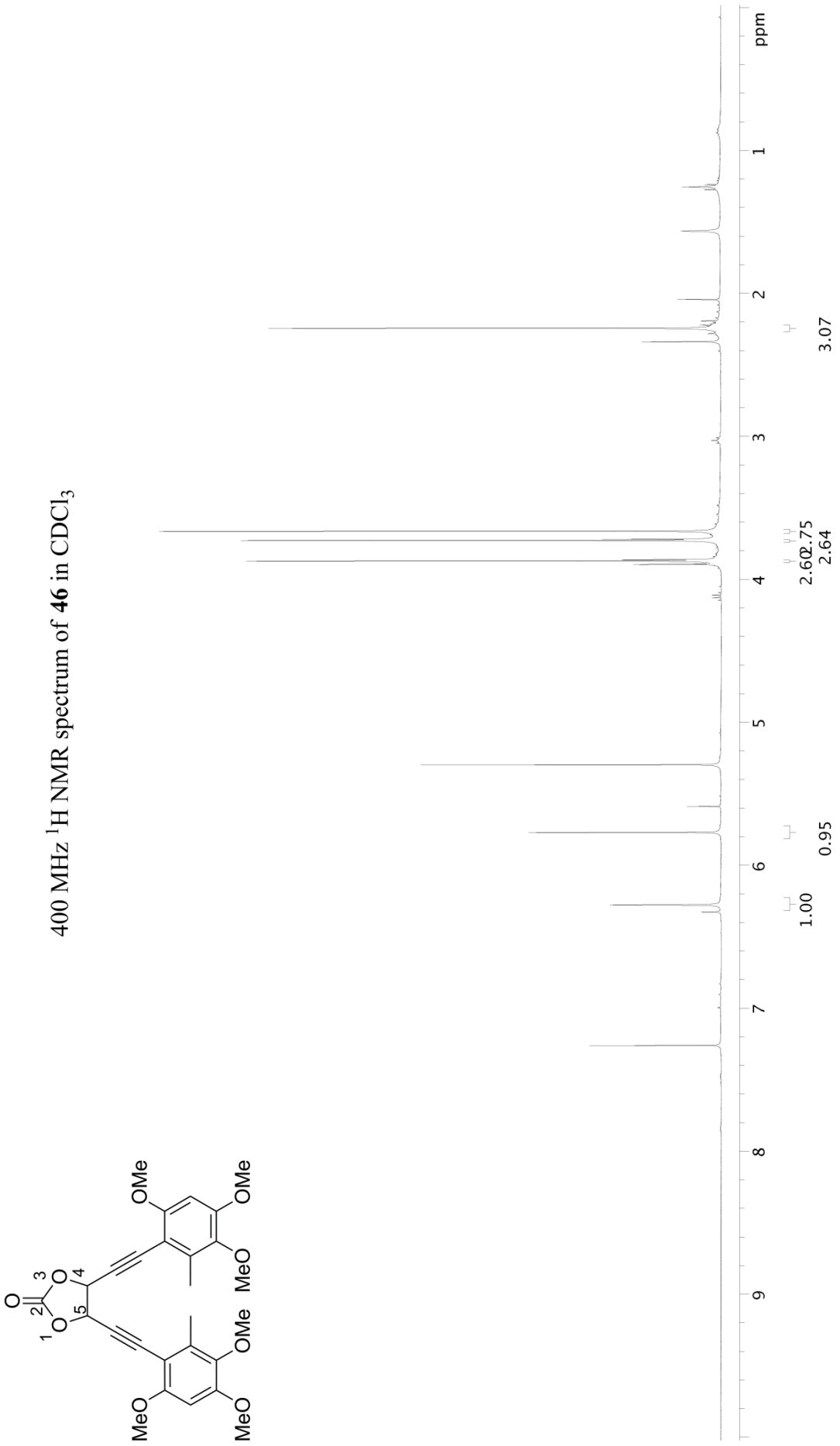


100 MHz ^{13}C NMR spectrum of **45** in $CDCl_3$

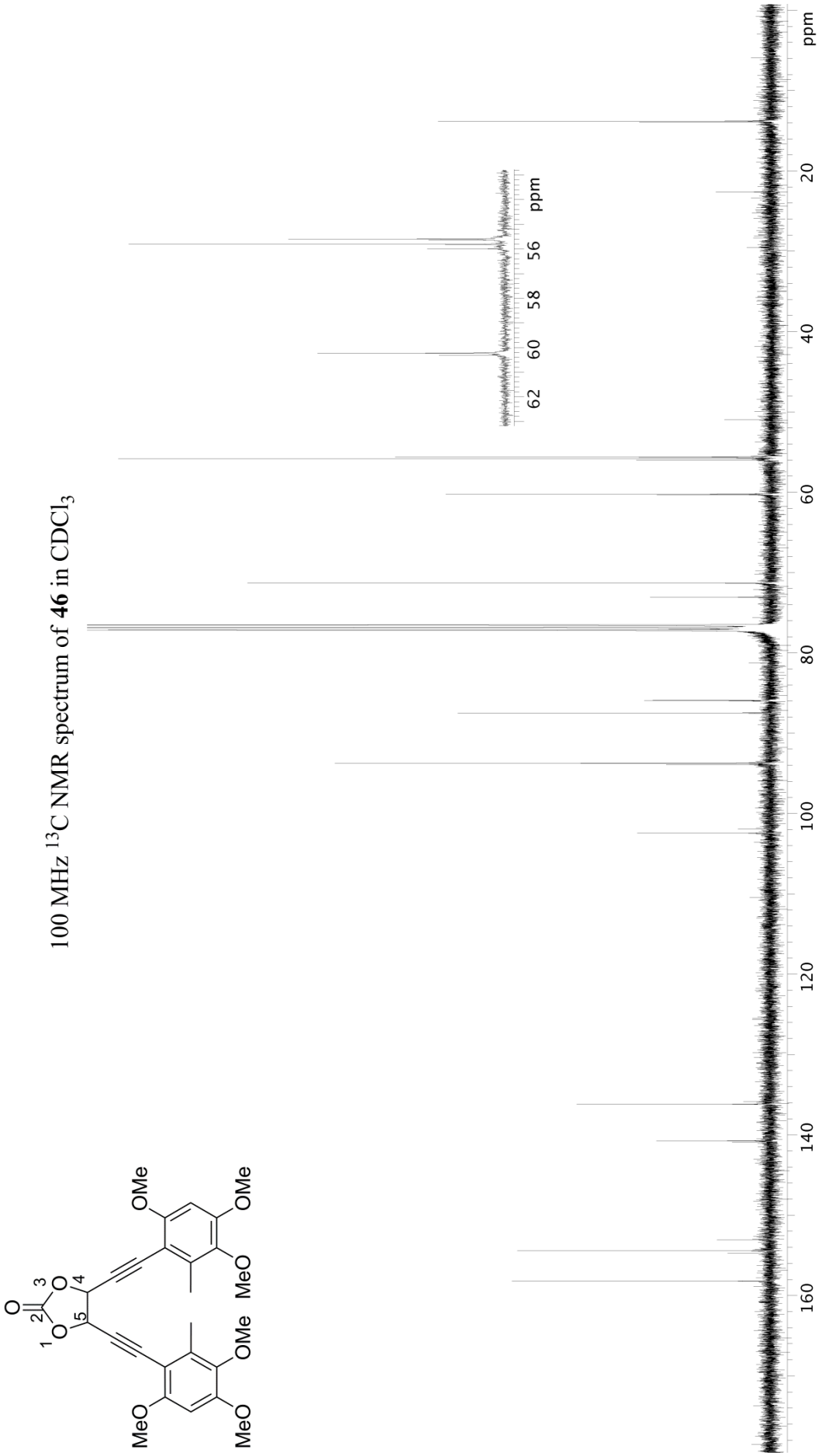




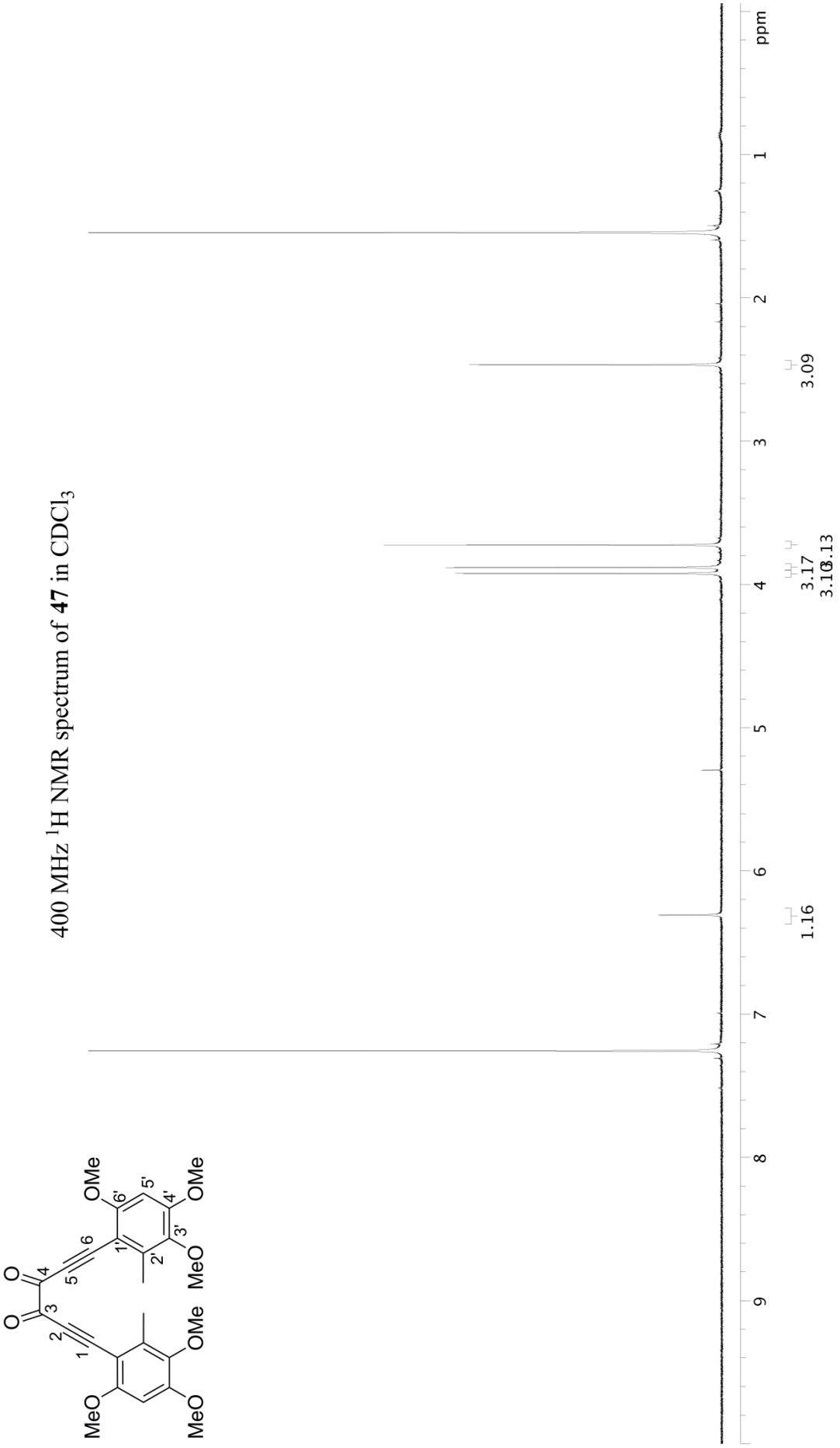
400 MHz ^{1}H NMR spectrum of **46** in $CDCl_3$



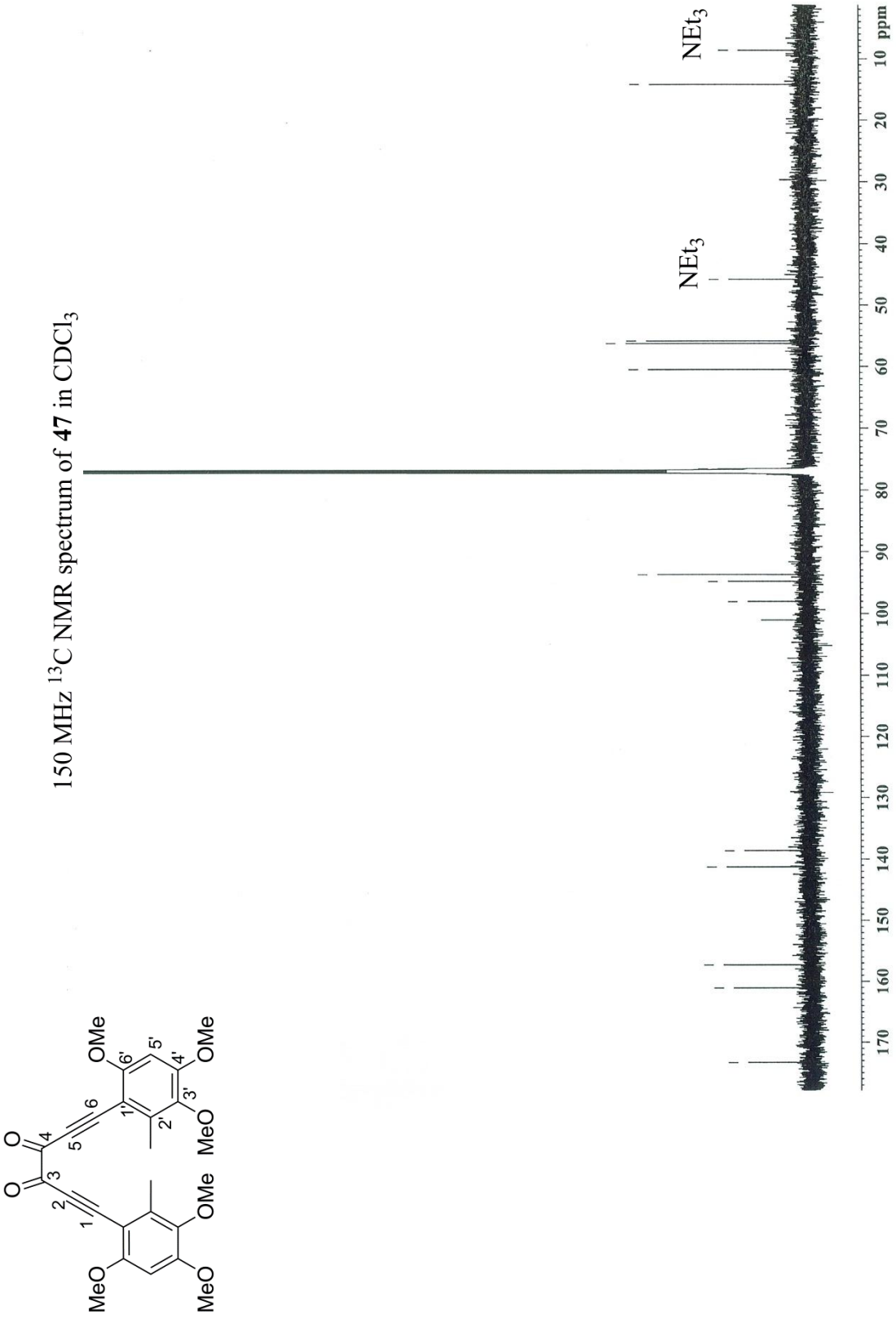
100 MHz ^{13}C NMR spectrum of **46** in $CDCl_3$



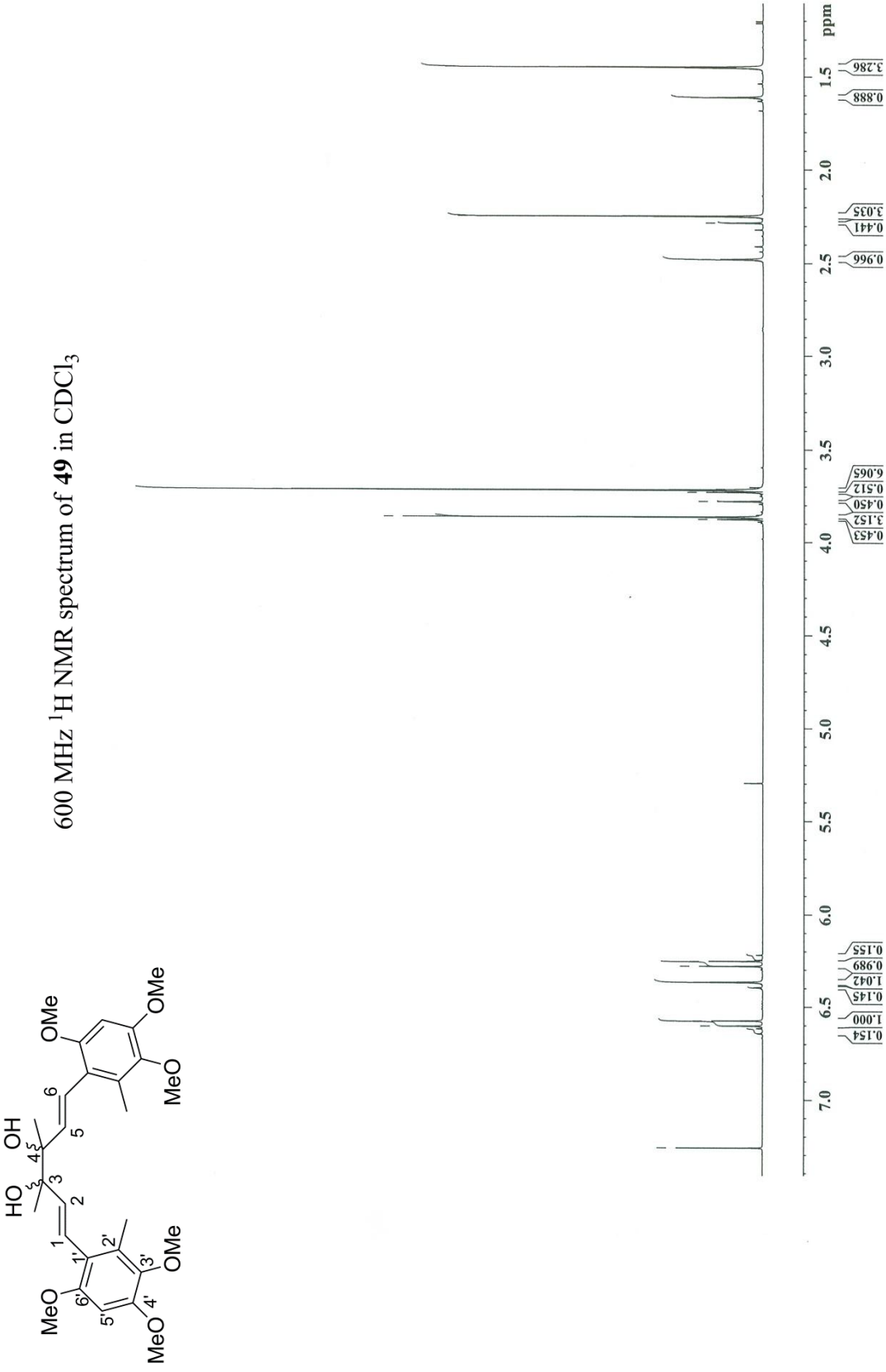
400 MHz ^{1}H NMR spectrum of **47** in $CDCl_3$



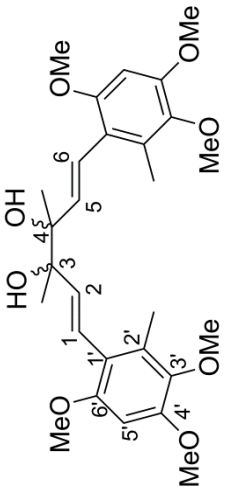
150 MHz ^{13}C NMR spectrum of **47** in $CDCl_3$

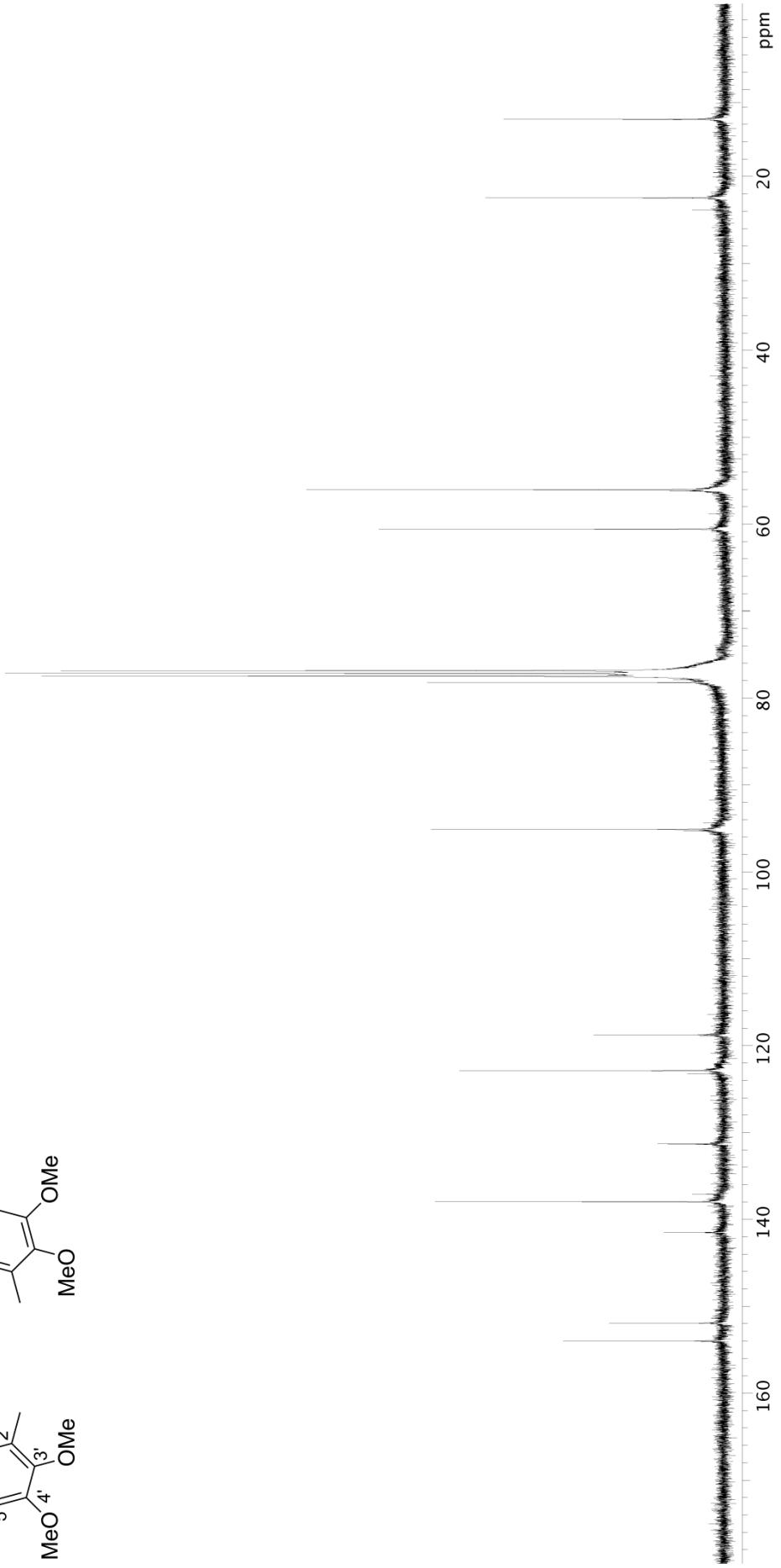


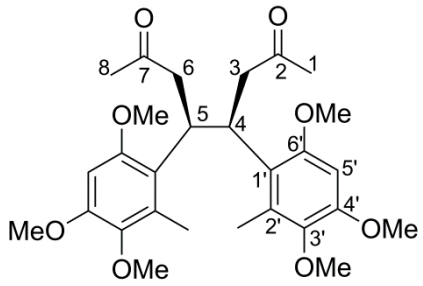
600 MHz ^{1}H NMR spectrum of **49** in $CDCl_3$



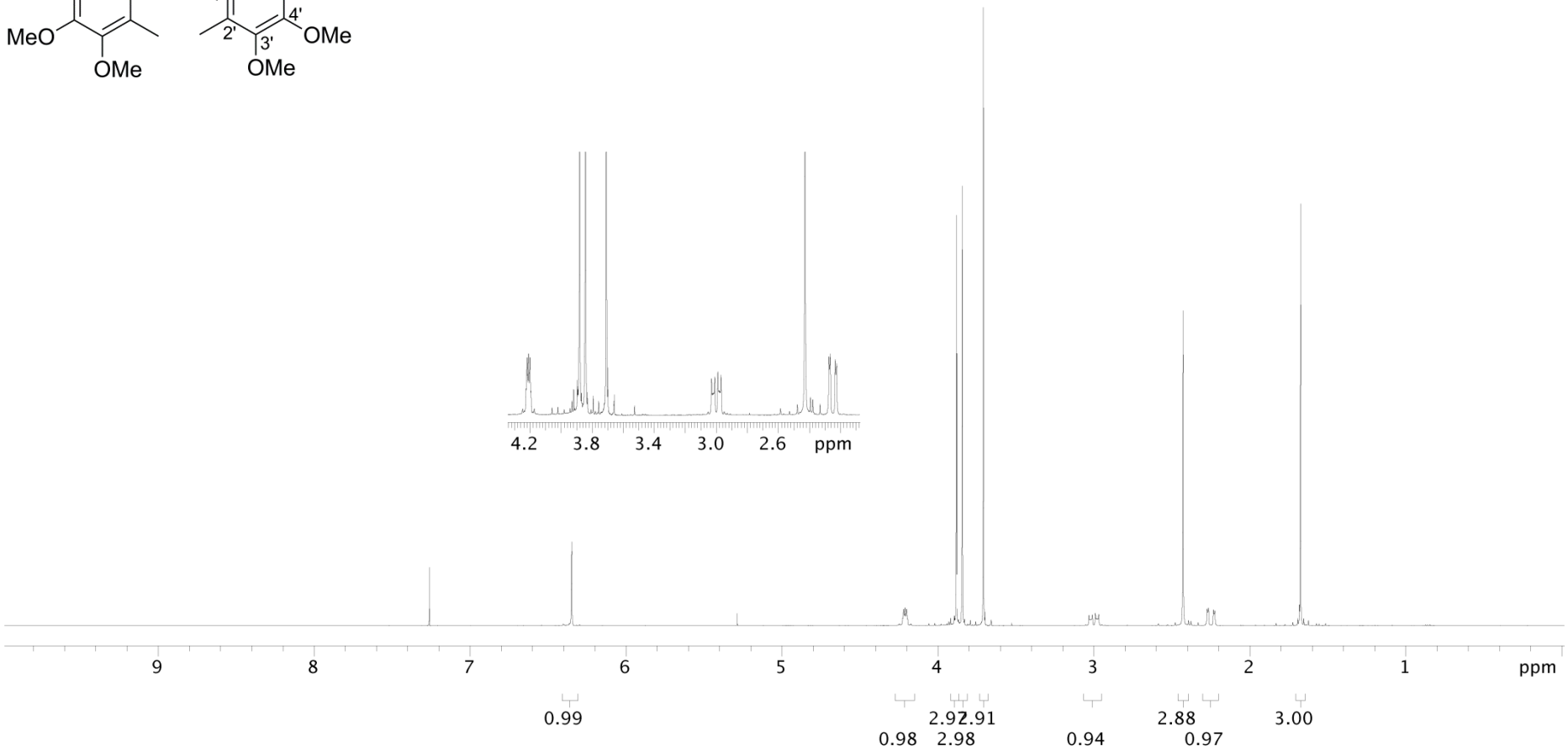
100 MHz ^{13}C NMR spectrum of **49** in $CDCl_3$



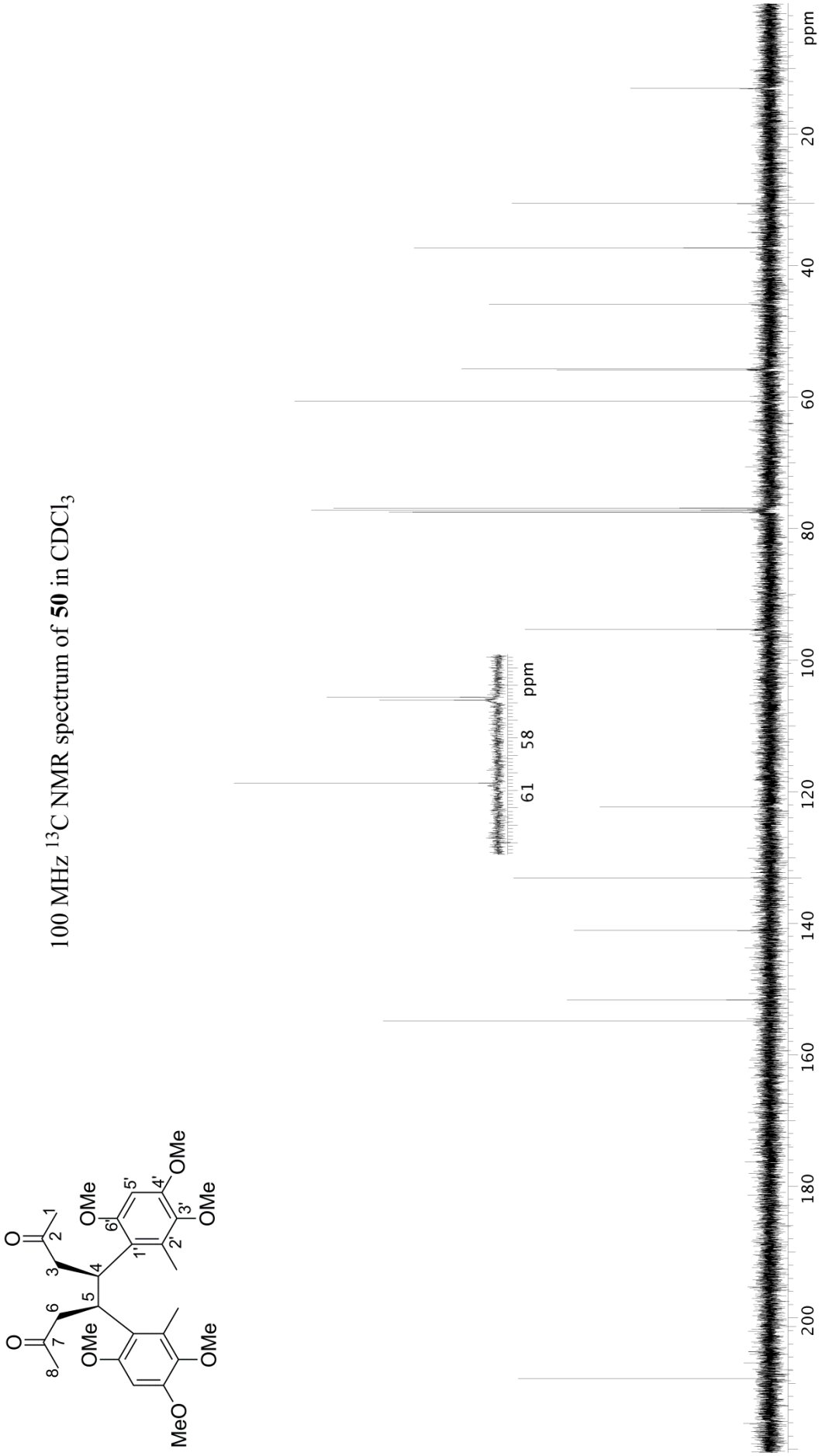




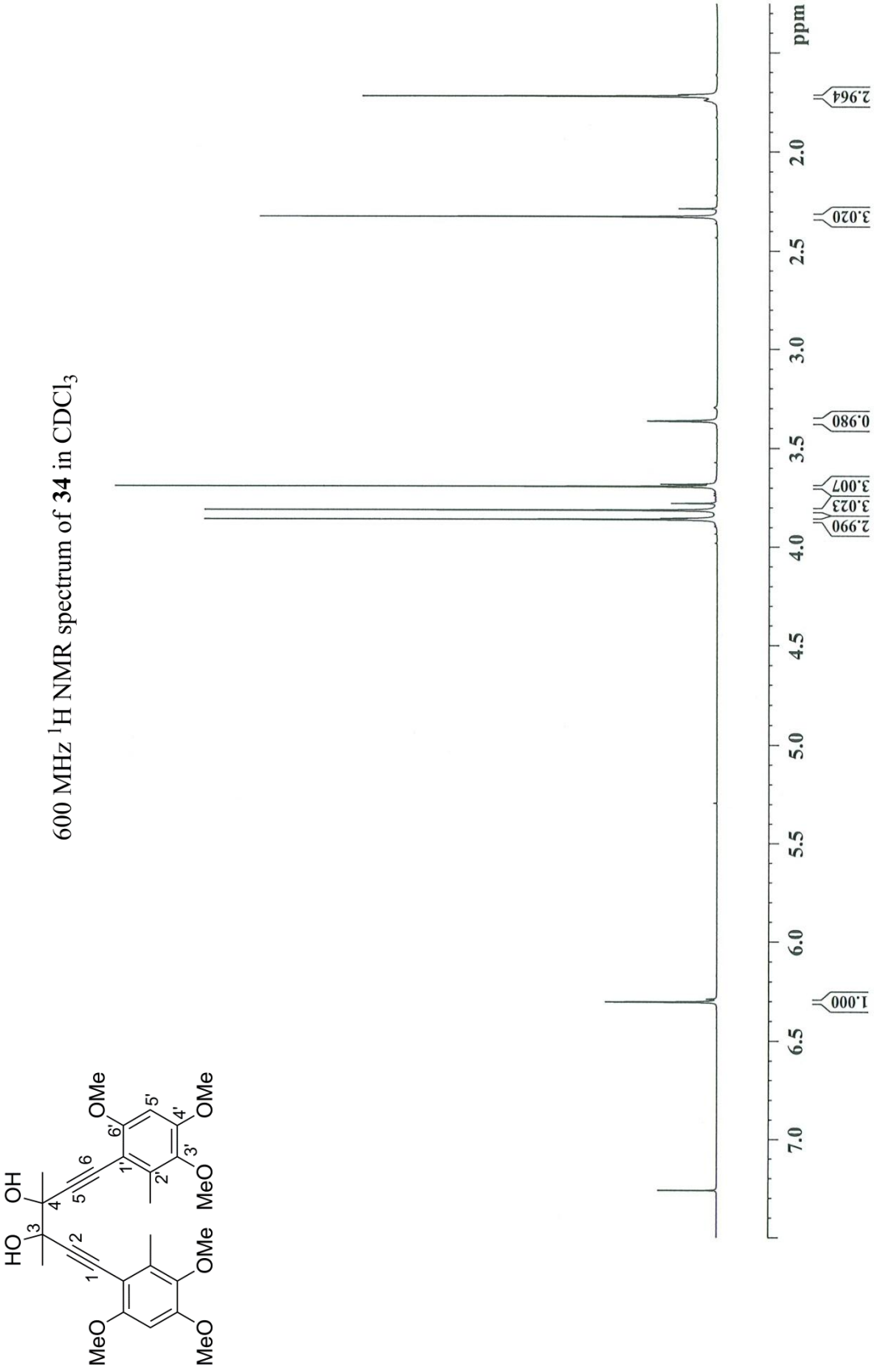
400 MHz ^{1}H NMR spectrum of **50** in $CDCl_3$

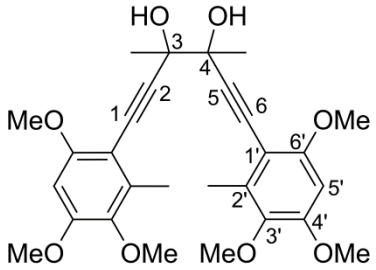


100 MHz ^{13}C NMR spectrum of **50** in $CDCl_3$

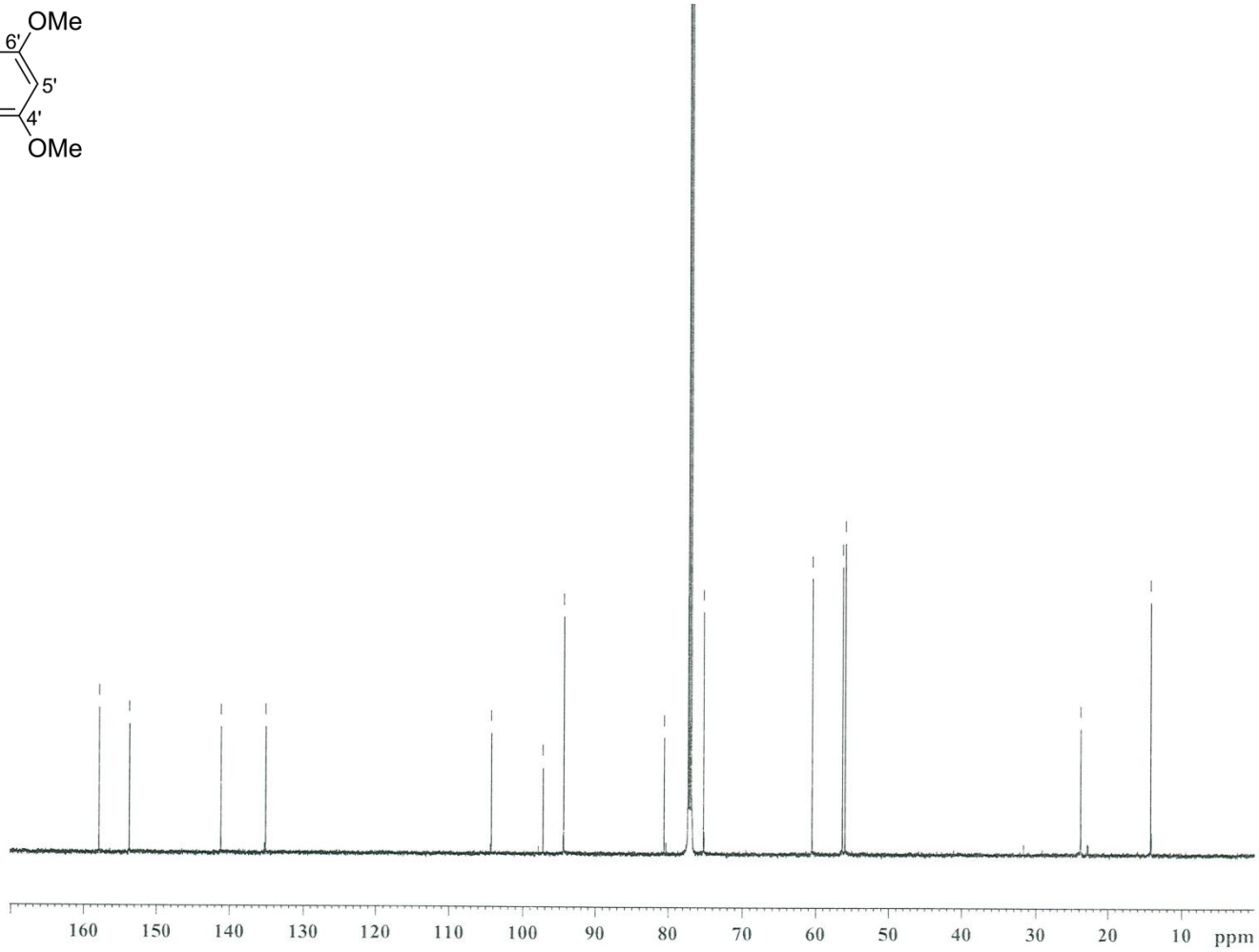


600 MHz ^{1}H NMR spectrum of **34** in $CDCl_3$

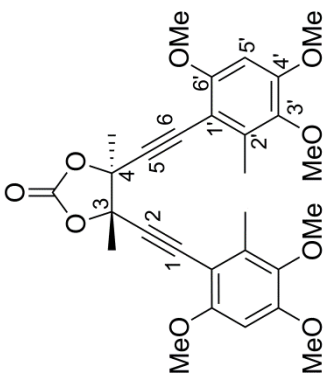


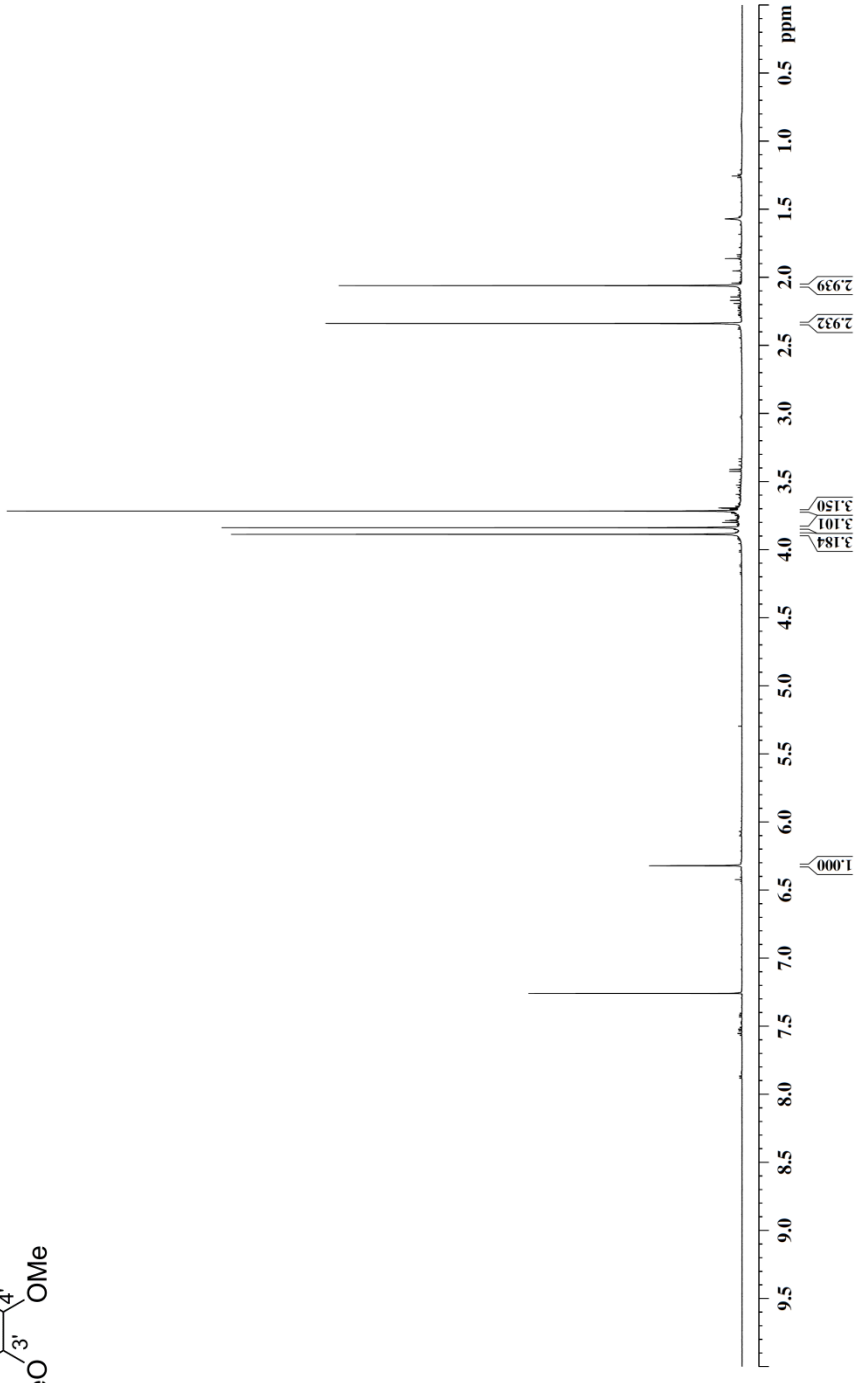


150 MHz ^{13}C NMR spectrum of **34** in $CDCl_3$



600 MHz ^{1}H NMR spectrum of **51** in $CDCl_3$





150 MHz ^{13}C NMR Spectrum of **51** in $CDCl_3$