Supplementary information for

Regioselective Oxidation of Nonactivated Alkyl C-H Groups Using Highly Structured Non-Heme Iron Catalysts

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1.- Experimental section

Materials

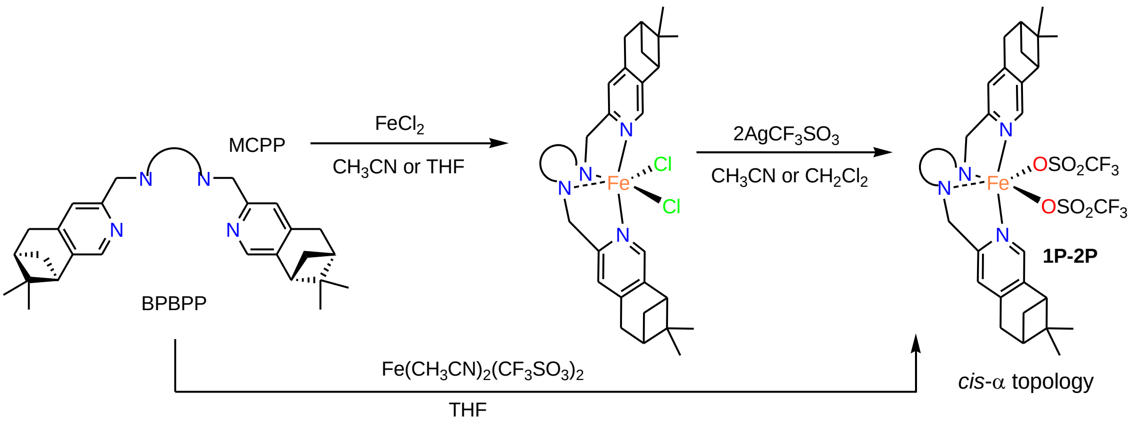
Reagents and solvents used were commercially available reagent quality unless otherwise stated. Acetonitrile for catalysis was HPLC grade. Preparation and handling of air-sensitive materials were carried out in a N_2 drybox with O_2 and H_2O concentrations < 1 ppm. $[Fe(CF_3SO_3)_2(BPBP)]$,[1] $[Fe(BPBP)(CH_3CN)_2](SbF_6)_2$[2], $[Fe(CF_3SO_3)_2$((*S,S*)-MCP)],[3] $[Fe(CF_3SO_3)_2$((*S,S,R*)-MCPP)],[4] ((*S,S,R*)-BPBPP)[5] and ((*R,R,R*)-BPBPP),[5] were prepared according to published procedures.

Instrumentation

IR spectra were taken in a FT-IR spectrophotometer using a single reflection ATR system. NMR spectra were taken on a 300 or 400 MHz spectrometer. ^{1}H-NMR spectra of paramagnetic compounds were performed with the following special parameters; relaxation delay = 0.03 s, acquisition time = 0.064 s, line broadening = 30 Hz, sweep width = 100-250 ppm. Spectra were referenced to the residual solvent peaks or TMS (tetramethylsilane) for ^{1}H-NMR. Elemental analyses were performed using a CHNS-O elemental analyzer. ESI-MS experiments were performed using methanol or acetonitrile as a mobile phase. HRMS were performed on time-of-flight mass spectrometer (University of Girona) with an ESI source using methanol as mobile phase. Product analyses were performed on a gas chromatograph (HP5 column, 30m or Cyclosil-B column, 30 m) and a flame ionization detector. GC-MS spectral analyses were performed on a gas chromatograph interfaced with an mass spectrometer. A 50% NH_3/CH_4 mix was used as the ionization gas for chemical ionization analyses.

Synthesis of complexes

Scheme S1. Synthesis of complexes.



Complex **Δ-1P** was prepared following a two step procedure that involves initial reaction of $FeCl_2$ with **(*R*,*R*,*R*)-MCPP** to yield chloride complex $[Fe(Cl_2)$(**(*R*,*R*,*R*)-MCPP**)].[6] This was subsequently treated with 2 equiv of $AgCF_3SO_3$ to afford triflato complex $[Fe(CF_3SO_3)_2($**(*R*,*R*,*R*)-MCPP**)] that could be isolated in crystalline form by diffusion of diethyl ether to dichloromethane solution of the complex. **Λ-2P** and **Δ-2P** were prepared by direct reaction of the ligand and $Fe(CF_3SO_3)_2(CH_3CN)_2$. Attempts to prepare **Λ-1P** and **Δ-1P** by this method yielded a mixture of products according to ^{1}H-NMR analysis, probably reflecting a mixture of topological isomeric species.[6-8] For complex **Δ-1P**, the standard procedure described for **1** and **Λ-1P** resulted in non crystalline material. Changing acetonitrile by THF in the reaction of **(*R*,*R*,*R*)-MCPP** with $FeCl_2$ and by CH_2Cl_2 in the subsequent reaction to obtain the triflato complex produced highly pure complex **Δ-1P** in crystalline form from CH_2Cl_2/diethyl ether.

Crystal data

Table S1. Crystal data and structure refinement for **Δ-1P**, **Λ-2P** and **Δ-2P**.

Compound	**Δ-1P**	**Λ-2P**	**Δ-2P**
Empirical formula	$C_{36}H_{48}F_6FeN_4O_6S_2$	$C_{36}H_{46}F_6FeN_4O_6S_2$	$C_{36}H_{46}F_6FeN_4O_6S_2$
Formula weight	866.75	864.74	864.74
Temperature	100(2) K	300(2) K	300(2) K
Wavelength	0.71073 Å	0.71073 Å	0.71073 Å
Cristal system	Trigonal	Orthorhombic	Orthorhombic
Space group	P3121	P2(1)2(1)2(1)	P2(1)2(1)2(1)
Unit cell dimensions	a = 18.797(3) Å	a = 14.954(11) Å	a = 15.398(2) Å
	α = 90°	α = 90°	α = 90°
	b = 18.797(3) Å	b = 16.340(12) Å	b = 26.204(3) Å
	β = 90°	β = 90°	β = 90°
	c = 12.998(12) Å	c = 16.575(12) Å	c = 10.6011(14) Å
	γ = 120°	γ = 90°	γ = 90°
Volume	3977.2(16) $Å^3$	4050(5) $Å^3$	4277.4(10) $Å^3$
Z	3	4	4
Density (calculated)	1.271 Mg/m^3	1.418 Mg/m^3	1.610 Mg/m^3
Absorption coefficient	0.434 mm^{-1}	0.551 mm^{-1}	0.537 mm^{-1}
F(000)	1608	1800	2152
Reflections collected	38824	63774	67855
Independent reflections	6235 [R(int) = 0.0581]	10101 [R(int) = 0.0808]	10554 [R(int) = 0.1084]
Final R indices [I>2sigma(I)]	R1 = 0.0406, wR2 = 0.1029	R1 = 0.0549, wR2 = 0.1440	R1 = 0.0596, wR2 = 0.1301
R indices (all data)	R1 = 0.0450, wR2 = 0.1044	R1 = 0.0890, wR2 = 0.1610	R1 = 0.1112, wR2 = 0.1467

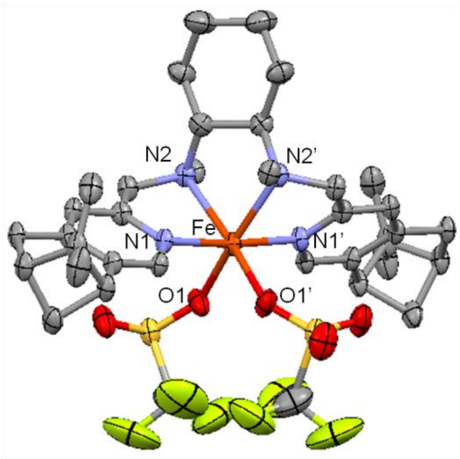
For the submitted single crystal data DF044 and LGL9P95 “A” alerts are reported when checked with the International Union of Crystallography's checkcif service. The alerts being:
CHEMW03 Type_2 The ratio of given/expected molecular weight as calculated from the _atom_site* data lies outside the range 0.90 <> 1.10

PLAT043 Type_1 Test for MolWeight

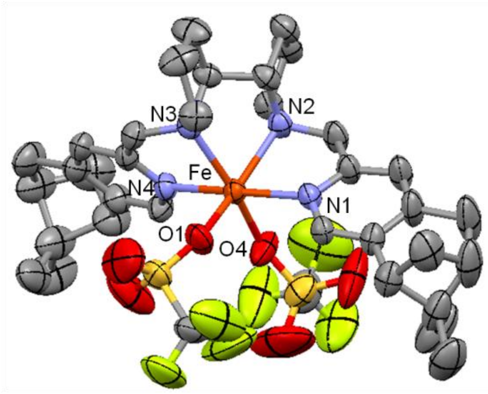
In Both structures, Electron density attributable to partially disordered solvent molecules was removed using the SQUEEZE option in PLATON[9]; a water molecule for the LGL9P95 structure and an ethyl ether molecule for the DF044 structure. These solvent molecules are, however, included in the reported chemical formula and derived values (e.g. formula weight, etc).

Table S2. Selected bond lengths [Å] and angles [°] for **Δ-1P**, **Λ-2P** and **Δ-2P**.

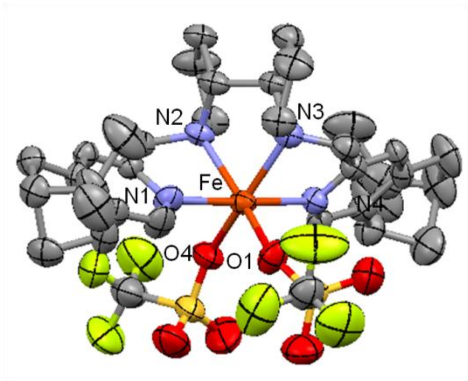
Δ-1P		**Λ-2P**		**Δ-2P**	
Fe1 N1’	2.1407	Fe1 N1	2.176(3)	Fe1 N1	2.180(3)
Fe1 N1	2.1407	Fe1 N2	2.220(3)	Fe1 N2	2.218(3)
Fe1 N2’	2.2335	Fe1 N3	2.210(3)	Fe1 N3	2.228(3)
Fe1 N2	2.2335	Fe1 N4	2.184(3)	Fe1 N4	2.175(3)
Fe1 O1’	2.1229	Fe1 O1A	2.132(3)	Fe1 O1	2.121(3)
Fe1 O1	2.1229	Fe1 O1B	2.116(3)	Fe1 O4	2.127(3)
N1 Fe1 N2	98.38	N1 Fe1 N2	77.49(11)	N1 Fe1 N2	77.79(12)
N1 Fe1 N2’	77.57	N1 Fe1 N3	99.18(11)	N1 Fe1 N3	98.47(11)
N1 Fe1 N1’	174.80	N1 Fe1 N4	172.96(11)	N1 Fe1 N4	175.75(12)
N2 Fe1 N2’	79.85	N2 Fe1 N3	80.80(12)	N2 Fe1 N3	79.93(12)
N2 Fe1 N1’	77.57	N2 Fe1 N4	107.20(11)	N2 Fe1 N4	102.19(13)
N2’ Fe1 N1’	98.38	N3 Fe1 N4	76.75(11)	N3 Fe1 N4	77.38(11)
O1’ Fe1 N1	95.16	O1B Fe1 N1	86.46(11)	N1 Fe1 O1	88.46(12)
O1’ Fe1 N2	164.39	O1B Fe1 N2	162.98(11)	N2 Fe1 O1	161.73(12)
O1’ Fe1 N2’	95.60	O1B Fe1 N3	96.42(14)	N3 Fe1 O1	90.42(11)
O1‘ Fe1 N1’	88.44	O1B Fe1 N4	88.28(11)	N4 Fe1 O1	90.64(13)
O1 Fe1 N1	88.44	O1A Fe1 N1	95.94(12)	N1 Fe1 O4	96.35(11)
O1 Fe1 N2	95.60	O1A Fe1 N2	85.23(13)	N2 Fe1 O4	92.48(13)
O1 Fe1 N2’	164.39	O1A Fe1 N3	156.62(12)	N3 Fe1 O4	161.49(11)
O1 Fe1 N1’	95.16	O1A Fe1 N4	89.75(11)	N4 Fe1 O4	87.90(12)
O1’ Fe1 O1	92.48	O1B Fe1 O1A	102.24(15)	O1 Fe1 O4	101.04(13)



Δ-1P



Λ-2P



Δ-2P

Figure S1. X-Ray structures **Δ-1P**, **Λ-2P** and **Δ-2P**. Hydrogen atoms and solvents of crystallization omitted for clarity.

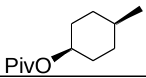
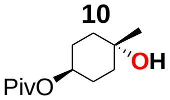
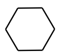
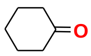
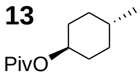
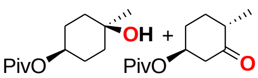
Reaction conditions for catalysis

Single addition versus iterative addition results

Our previous experimental conditions allowed high conversion of substrate to product but they involved several subsequent additions of catalyst and H_2O_2.[4] Depending on the substrate, up to three were necessary. For convenience, a simpler protocol was evaluated, which consisted in a single syringe pump addition of H_2O_2 (17 min) over a CH_3CN solution containing the catalyst (3 mol %), AcOH (1.5 equiv) and substrate at 0 ºC. *Cis*-4-methyl-1-cyclohexylpivalate (**9**) and cyclohexane (**11**) were taken as model substrates for optimization because the results from these reactions allow a straightforward comparison with the literature. Table S3 shows that this simpler set of conditions, which do not involve the most common use of excess of substrate, provides preparative useful yields based on substrate into oxidation products (51-75%) using low catalyst loadings (3 mol %) of pinene containing complexes **Λ-1P**, **Δ-1P**, **Λ-2P** and **Δ-2P**. Most significantly, yields attained with the pinene containing catalysts are substantially higher than those obtained with **1** and **2** both in the oxidation of secondary (**11**) and tertiary (**9**) C-H bonds.

It is also interesting to notice that higher differences among catalysts efficiencies arise when using the iterative protocol. **Λ-1P** is the only complex that provides better yields when the iterative protocol is employed. Instead, all other complexes performed better with the simpler protocol. Particularly remarkable is that in the case of **Δ-1P**, the new synthesis of the complex in combination with this new protocol increased substantially the efficiency of the catalyst in comparison with the previously reported one.[4] In conclusion, the advantage of this new protocol is not only its simplicity, but also that it allows better performance of most catalysts.

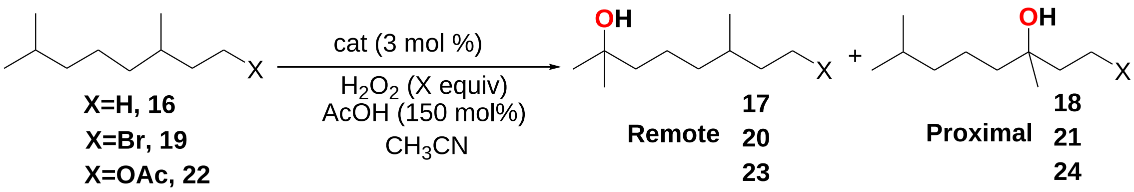
Table S3. Oxidation of cyclohexane and *cis*- and *trans*-4-methylcyclohexyl pivalate.[a]

			Product yield %[b] (substrate conversion) [14/15][c]					
Substrate	**Products**	**Protocol**	**Λ-1P**	**Δ-1P**	**Λ-2P**	**Δ-2P**	**1**	**2**
9 	**10** 	Iterative[d]	57[h] (75)	9[j] (19)	62 (92)	52 (75)	20 (33)	43 (45)
		Single[e]	51 (70)	53 (65)	65[i] (87)	57 (70)	38 (53)	53 (69)
11 	**12** 	Iterative[f]	67 (92)	21 (48)	64 (91)	56 (84)	47 (77)	47 (79)
		Single[g]	72 (93)	75 (87)	71 (92)	67 (87)	61 (79)	61 (79)
13 	 **14** **15**	Single[e]	60 (67) [71/29]	58 (60) [69/31]	61 (80) [80/20]	57 (71) [77/23]	-	-

[a] Except otherwise indicated for oxidation at terciary C-H bonds RC>95% (RC = retention of configuration in the oxidation of the tertiary C-H bonds, expressed as the ratio of the tertiary alcohols: |[(*trans* – *cis*) / (*trans* + *cis*)] x 100|). [b] % GC yield based on substrate. [c] Normalized (100) ratio of products. [d] Conditions: Cat:H_2O_2:substrate:AcOH; 1x3:120x3:100:50x3; [e] 3:200:100:150; [f] 1x2:120x2:100:50x2; [g] 3:280:100:150. [h] Isolated by flash chromatography. [i] 55% isolated by flash chrnormatography. [j] 82% RC.

Single addition results

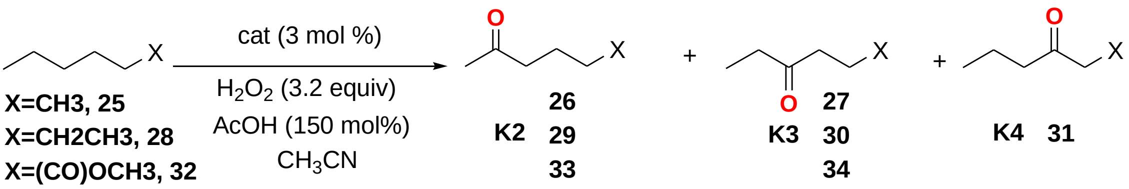
Table S4. Evaluation of electronic effects in the oxidation of tertiary C-H bonds.



Substrate	Yield[a] % (conversion %) [Remote/Proximal]			
	Λ-1P	**Δ-1P**	**Λ-2P**	**Δ-2P**
16[b]	31 (78) [55/45]	13 (54) [57/43]	32 (75) [51/49]	20 (44) [54/46]
16[c]	32 (86) [55/45]	31 (72) [55/45]	31 (82) [48/52]	29 (64) [51/49]
19[c]	48 (83) [88/12]	41 (66) [90/10]	43 (78) [85/15]	29 (44) [85/15]
22[c]	49 (68) [79/21]	44 (65) [81/19]	44 (69) [77/23]	33 (51) [76/24]

[a] GC yield based on substrate. [b] Cat:H_2O_2:substrate:AcOH 1:180:100:50. [c] 3:200:100:150.

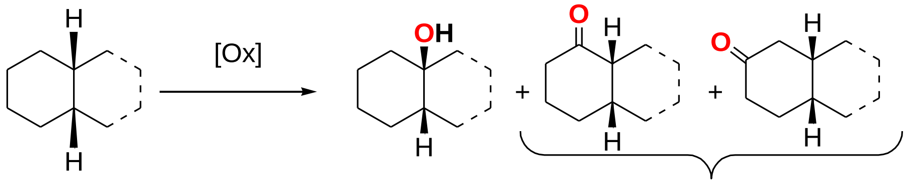
Table S5. Evaluation of electronic effects in the oxidation of secondary C-H bonds



Substrate:	**25**	**28**	**32**
	Yield[a] % (conversion %)		
Catalyst	**[K2/K3]**	**[K2/K3/K4]**	**[K2/K3]**
Λ-1P	50 (94) [57/43]	57 (79) [51/35/14]	55 (62) [76/24]
Δ-1P	46 (72) [59/41]	53 (72) [47/36/17]	26 (36) [74/26]
Λ-2P	51 (94) [50/50]	56 (71) [42/40/18]	56 (66) [63/37]
Δ-2P	44 (74) [50/50]	50 (56) [40/40/20]	31 (34) [63/37]
2	50 (69) [53/47]	41 (61) [43/39/18]	-

[a] GC yield based on substrate.

Table S6. Oxidation of *cis* isomers of dimethylcyclohexane and decalin.



cis-1,2-dimethylcyclohexane, **6**
cis-decalin, **35**

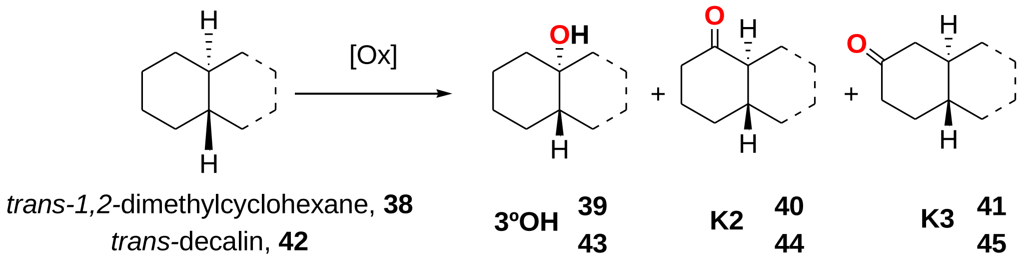
3ºOH **7** **36**

K **8** **37**

	Substrate					
	6[a]			**35**[b]		
Catalyst	**Yield[c] % (conversion)**	**[7/8]**	**RC[d]**	**Yield[c] % (conversion)**	**[36/37]**	**RC[d]**
Λ-1P	57 (80)	[84/16]	99	46 (86)	[69/31]	99
Δ-1P	58 (81)	[83/17]	99	51 (88)	[71/29]	99
Λ-2P	58 (80)	[88/12]	99	48 (82)	[77/23]	99
Δ-2P	55 (72)	[86/14]	99	47 (91)	[74/26]	99

[a] Cat:H_2O_2:substrate:AcOH 1:120:100:50. [b]Cat:H_2O_2:substrate:AcOH 3:200:100:150 [c] GC yield based on substrate. [d] RC = retention of configuration in the oxidation of the tertiary C-H bonds, expressed as the ratio of the tertiary alcohols: [(*trans* – *cis*) / (*trans* + *cis*)] x 100.

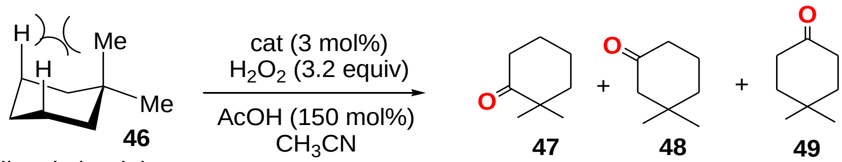
Table S7. Oxidation of *trans* isomers of dimethylcyclohexane and decalin.



	Substrate					
	38[a]			42[b]		
Catalyst	**Yield[c] % (conversion)**	**[39/40/41]**	**RC[d]**	**Yield[c] % (conversion)**	**[43/44/45]**	**RC[d]**
Λ-1P	47±1 (77)	[26±1/29±1/45±1]	99	47±2 (85)	[4±1/37±1/59±1]	99
Δ-1P	52±2 (74)	[28±1/26±1/46±1]	98	50±1 (70)	[7±1/37±1/56±1]	98
Λ-2P	49±1 (87)	[48±1/24±1/28±1]	99	48±1 (83)	[13±1/40±1/46±1]	99
Δ-2P	43±2 (77)	[50±1/21±1/29±1]	99	52±2 (88)	[16±1/39±1/45±1]	99
2	36±2 (65)	[48±1/22±1/30±1]	99	48±2 (88)	[15±1/38±1/46±1]	99

[a] Cat:H_2O_2:substrate:AcOH 3:200:100:150. [b] Cat:H_2O_2:substrate:AcOH 3:280:100:150 [c] GC yield based on substrate. [d] RC = retention of configuration in the oxidation of the tertiary C-H bonds, expressed as the ratio of the tertiary alcohols: [(*trans* – *cis*) / (*trans* + *cis*)] x 100.

Table S8. Steric and electronic factors in the oxidation of cycloalkanes.

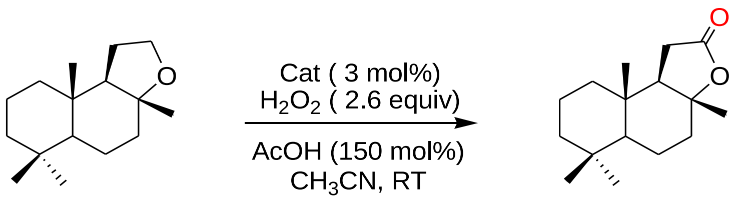


1,1-dimethylcyclohexane

Catalyst	Yield[a] % (conversion)	[47/48/49]
Λ-1P	67 (94)	[23/54/23]
Δ-1P	66 (90)	[21/57/22]
Λ-2P	68 (81)	[37/44/19]
Δ-2P	69 (92)	[32/47/21]
2	58 (68)	[36/43/21]

[a] GC yield based on substrate.

Table S9. Oxidation of ambroxide.

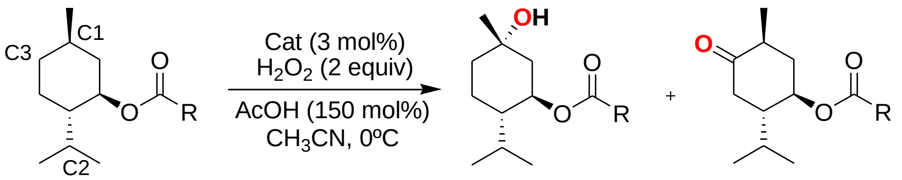


(-)-ambroxide, 50 **(+)-sclareolide, 51**

Catalyst	Λ-1P	Δ-1P	Λ-2P	Δ-2P	Λ-2	Λ-2
Product yield %[a] (substrate conversion)	73[b] (99)	73 (99)	60 (95)	72 (99)	58 (99)	66 (95)

[a] GC yield based on substrate. [b] 70% isolated by flash chromatography. After reaction, 1 equivalent of HCl (2M) was added and the crude mixture heated to 40ºC for 4 hours to reverse partial lactone ring-opening.

Table S10. Oxidation of menthol derivatives.



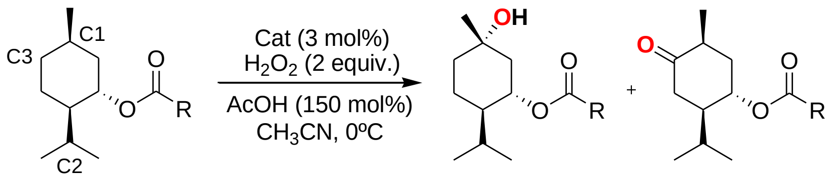
menthol derivatives

(+) and R=CH_3, **54** — **55** — **56**
(-) and R=CH_3, **3** — **M-OH1** **4** — **M-K** **5**
(-) and R=CF_3, **57** — **58** — **59**

	Menthol derivatives		
	54	**3**	**57**
Catalyst	**Yield %[a] [M-OH1 / M-K][b]**		
Λ-1P	37 [86/13]	63 (65)[c] [95/5]	51 [93/7]
Δ-1P	58 [90/10]	42 [76/24]	29 [73/27]
Λ-2P	47 [85/15]	59 [88/12]	53 [75/25]
Δ-2P	52 [85/15]	47[79/21]	48 [70/30]
Λ-2SbF$_6$	42 [78/22]	47 [83/17]	
Λ-2	45 [77/23]	48 [83/17]	37 [69/31]
Δ-2	48 [84/16]	47 [78/22]	39 [68/32]

[a] Combined GC yield based on substrate. [b] Normalized (100) ratio of products. [c] Isolated by flash chromatography. [d] Arrows connecting reactions of **Λ-2** and **Δ-2** with substrates **54** and **3** correspond to enantiomerically related reactions.

Table S11. Oxidation of (+)-isomenthol derivatives.



isomenthol derivatives

R=CH_3, **60**
R=tBu, **63**

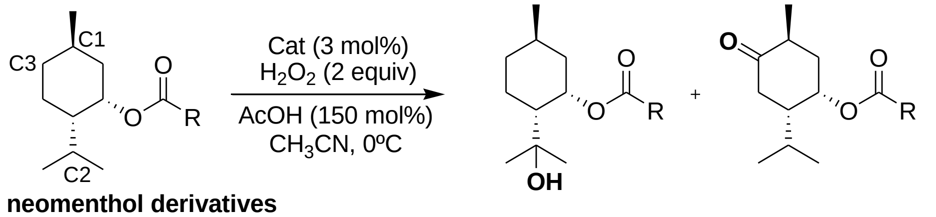
I-OH1 **61** **64**

I-K **62** **65**

Catalyst	**Substrate 60**	**Substrate 63**
	Yield %[a] [I-OH1% / I-K%][b]	
Λ-1P	75 [86/14]	68 [87/13]
Δ-1P	83 [89/11]	82 (71)[c] [91/9]
Λ-2P	70 (63)[c] [83/17]	67 [83/17]
Δ-2P	65 [85/15]	67 [86/15]

[a] Combined GC yield based on substrate. [b] Normalized (100) ratio of products. [c] Isolated by flash chromatography.

Table S12. Oxidation of neomenthol derivatives.



neomenthol derivatives

neomenthol derivatives	N-OH2	N-K
(-) and R=CH_3, **66**	**67**	**68**
(+) and R=CH_3, **69**	**70**	**71**
(+) and R=CF_3, **72**	**73**	**74**
(+) and R=tBu, **75**	**76**	**77**

	Substrate			
	66	**69**	**72**	**75**
Catalyst	**Yield %[a] (conversion) [N-OH2 / N-K][b]**			
Λ-1P	40, 42[c] (53) [55/45]	43 (77) [83/17]	35, 35[c] (64)[83/17]	54 (71) [80/20]
Δ-1P	44 (59) [64/36]	41 (51) [44/56]	34 (40) [61/39]	40 (44) [34/66]
Λ-2P	47 (72) [58/42]	43 (74) [69/31]	41 (96) [53/47]	46, 45[c] (66) [52/48]
Δ-2P	43 (69) [48/52]	44 (68) [44/56]	34 (72) [42/58]	35 (48) [34/66]
Λ-2SbF_6	43 (70) [41/59]	46 (66) [48/52]		
Λ-2	43 (69) [42/58]	44 (66) [49/51]	33 (61) [40/60]	38 (50) [40/60]
Δ-2	44 (72) [48/52]	44 (72) [42/58]	33 (64) [37/63]	34 (43) [35/65]

[a] Combined GC yield based on substrate. [b] Normalized (100) ratio of products. [c] Isolated by flash chromatography.

Table S13. Oxidation of cyclohexane and sclareolide with catalyst **Λ-1P**. Comparison of alcohol/ketone ration using standard workup or adding PPh_3.[10]

Cyclohexane	**Yield**	**[Cyclohexanol/Cyclohexanone]**
Sample 1[a]	76	[1/75]
Sample 2[b]	76	[1/75]
Sclareolide	**Yield**	**[78/79/79OH/80]**
Sample 1[a]	74	[33/36/4/27]
Sample 2[b]	67	[33/36/5/26]

[a] Sample passed though silica plug. [b] Sample treated with 1 equiv *versus* H_2O_2 of PPh_3.

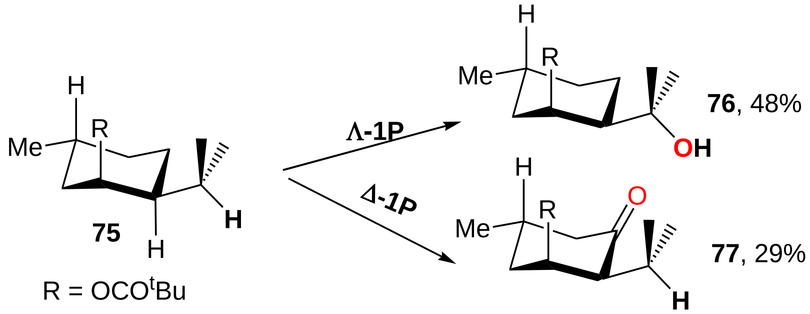
Product isolation

Purity of isolated products was checked by ^{1}H-NMR and GC, and yields corrected based on the value of purity.

Mmol scale reactions

(+)-Neomenthyl pivalate (**75**):

Scheme S2. mmol scale oxidation of (+)-neomenthyl pivalate.



76 favoured conditions: Catalyst **Λ-1P**, temperature: 0ºC.

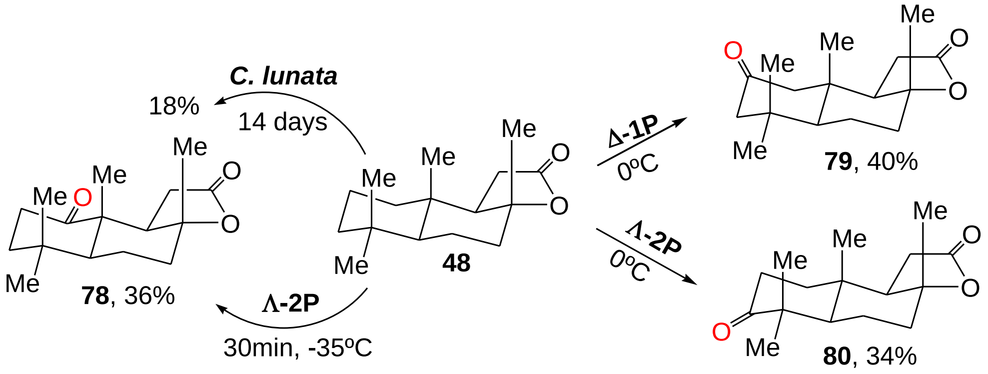
	Yield %				
Substrate	**76**	**77**	**Total**	**76/77**	**Conversion (%)**
672 mmol	**34**	7	41	83/17	64
241 mmol	**38**	9	47	81/19	76
TOTAL	**48**	**10**	**58**	**83/17**	**91**

77 favoured conditions: Catalyst **Δ-1P**, temperature: 0ºC.

	Yield %				
Substrate	**76**	**77**	**Total**	**76/77**	**Conversion (%)**
709 mmol	13	18	31	41/59	52
340 mmol	9	13	22	42/58	44
189 mmol	12	19	31	40/60	59
TOTAL	**20**	**29**	**49**	**41/59**	**89**

(+)-Sclareolide (**51**):

Scheme S3. mmol scale oxidation of sclareolide.



78 favoured conditions: Catalyst **Λ-2P**, temperature: -35ºC.

	Yield %				
Substrate	**78**	**79**	**80**	**Total**	**Conversion (%)**
627 mmol	**26**	4	14	44	58
252 mmol	**26**	-	13	39	43
TOTAL	**36**	4	19	59	77

79 favoured conditions: catalyst **Δ-1P**, temperature: 0ºC.

	Product yield %				
Substrate	**78**	**79**	**80**	**Total**	**Conversion %**
664 mmol	5	**31**	21	57	70
198 mmol	6	**26**	18	50	64
TOTAL	7	**40**	26	73	89

80 favoured conditions: catalyst **Λ-2P**, temperature: 0ºC.

	Product yield %				
Substrate	**78**	**79**	**80**	**Total**	**Conversion %**
709 mmol	23	16	**34**	73	87

2.- References

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[9] A. L. P. Spek, A Multipurpose Crystallographic Tool, Utrecht University, Utrecht, THe Netherlands.
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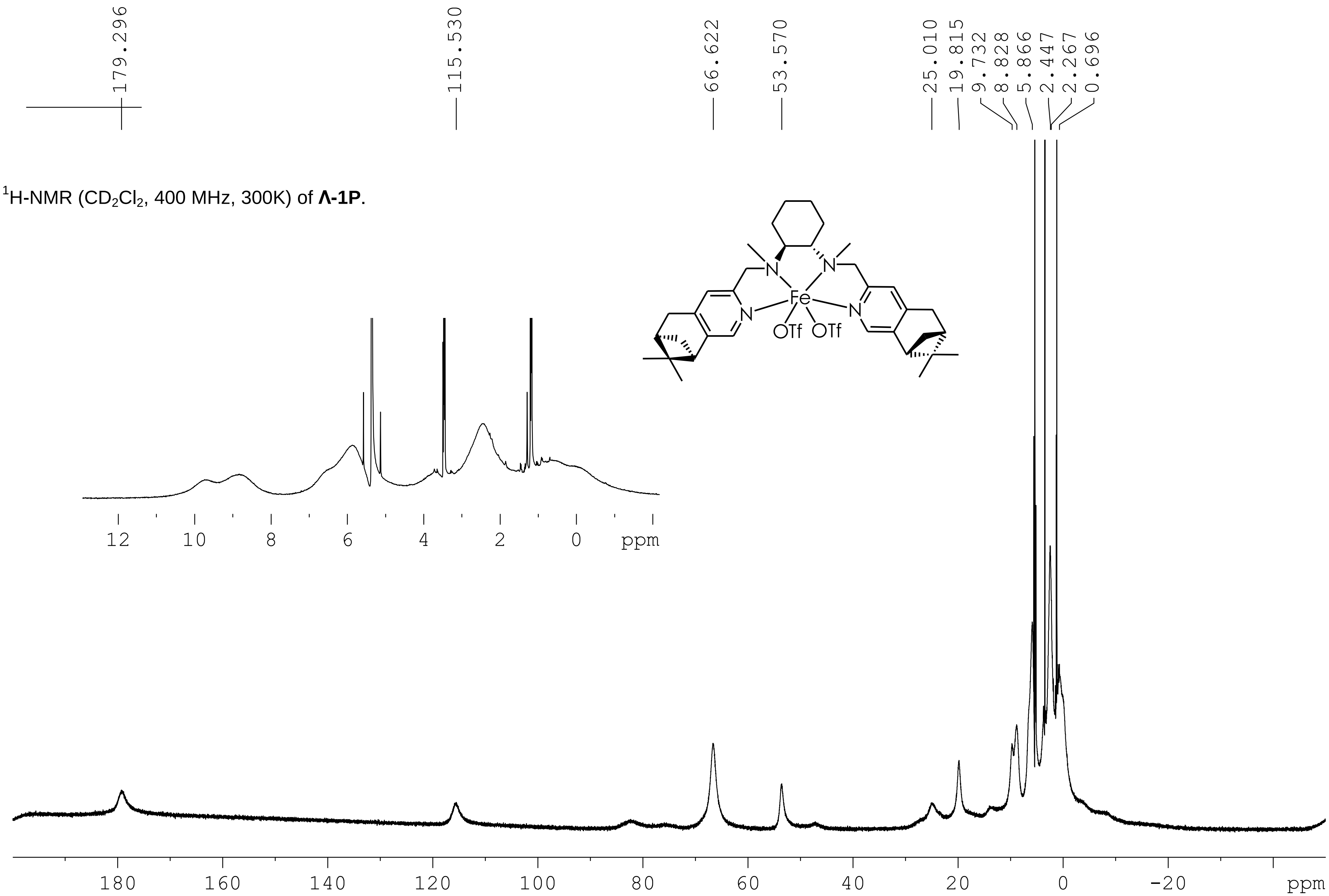
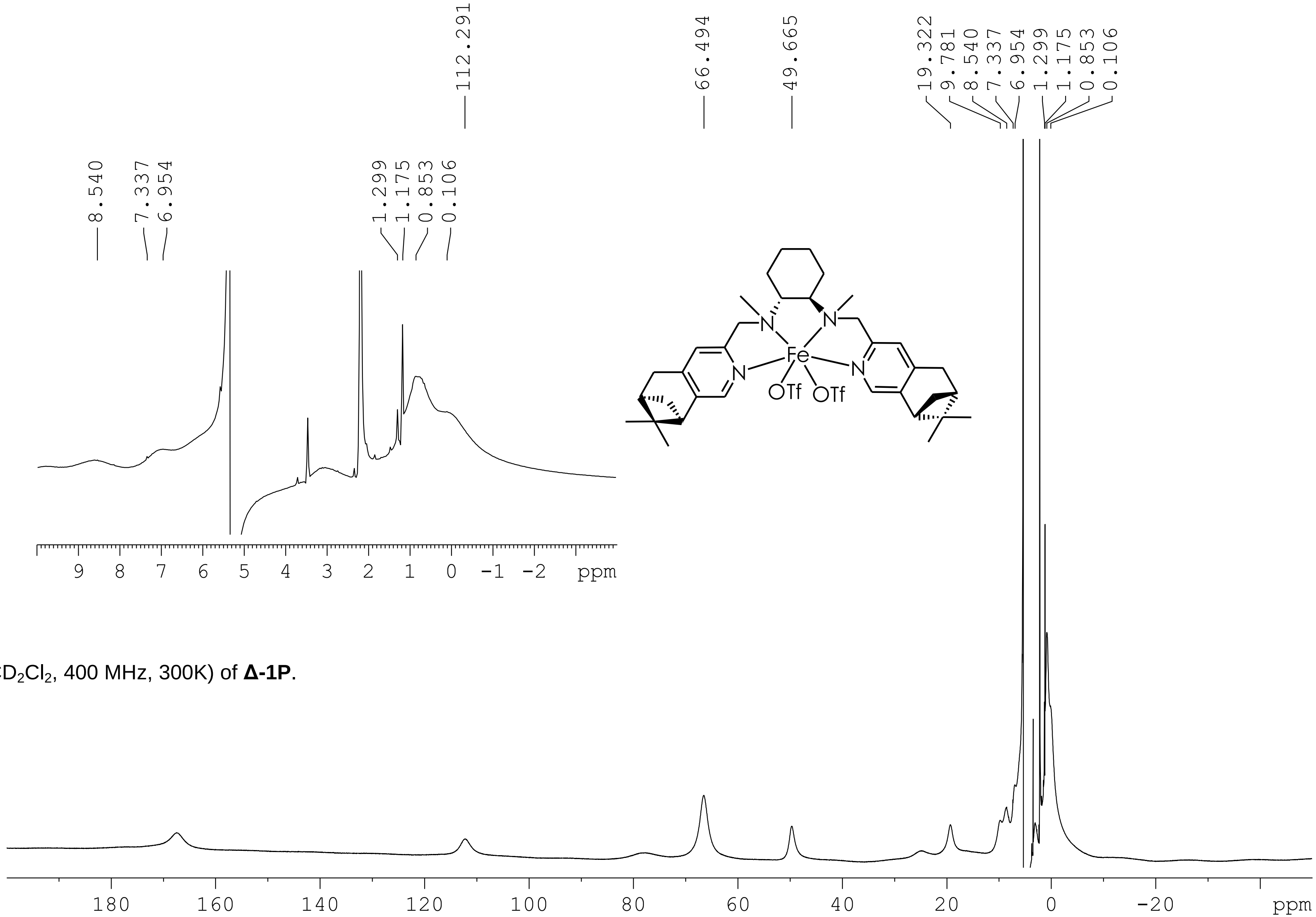
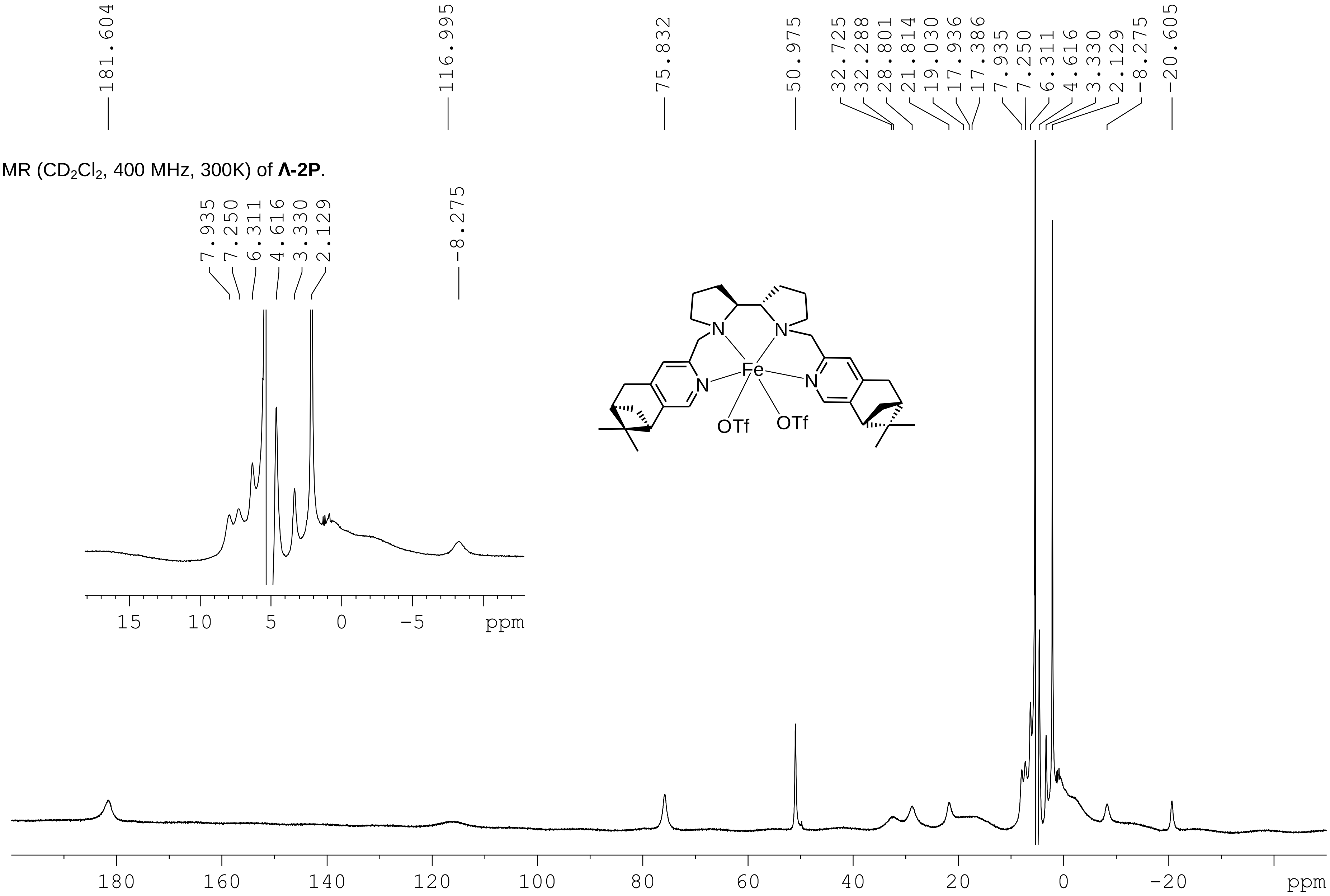


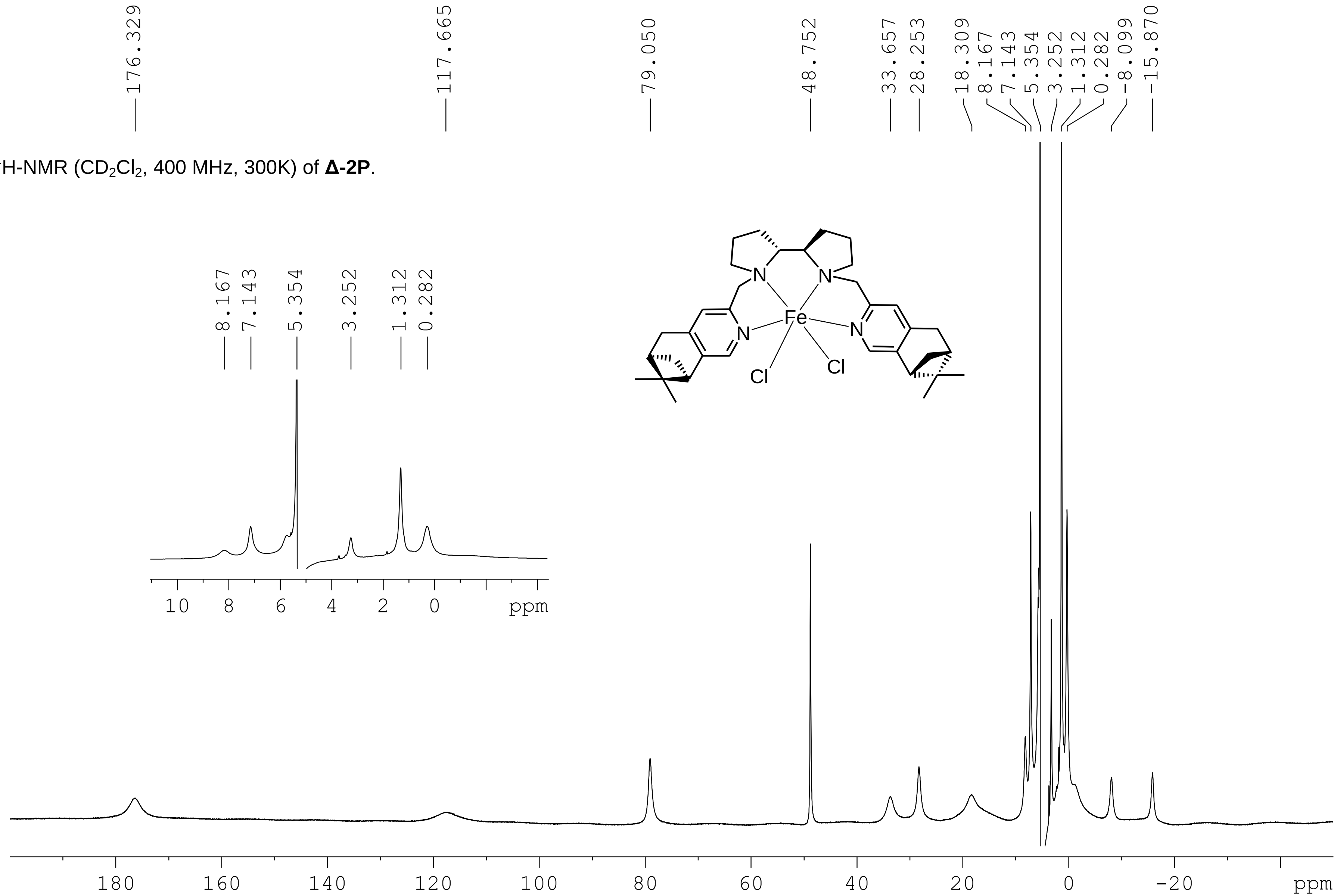
[1]H-NMR (CD_2Cl_2, 400 MHz, 300K) of **Λ-1P**.



^{1}H-NMR (CD_2Cl_2, 400 MHz, 300K) of **Δ-1P**.

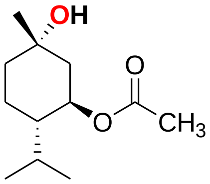
^{1}H-NMR (CD_2Cl_2, 400 MHz, 300K) of **Λ-2P**.

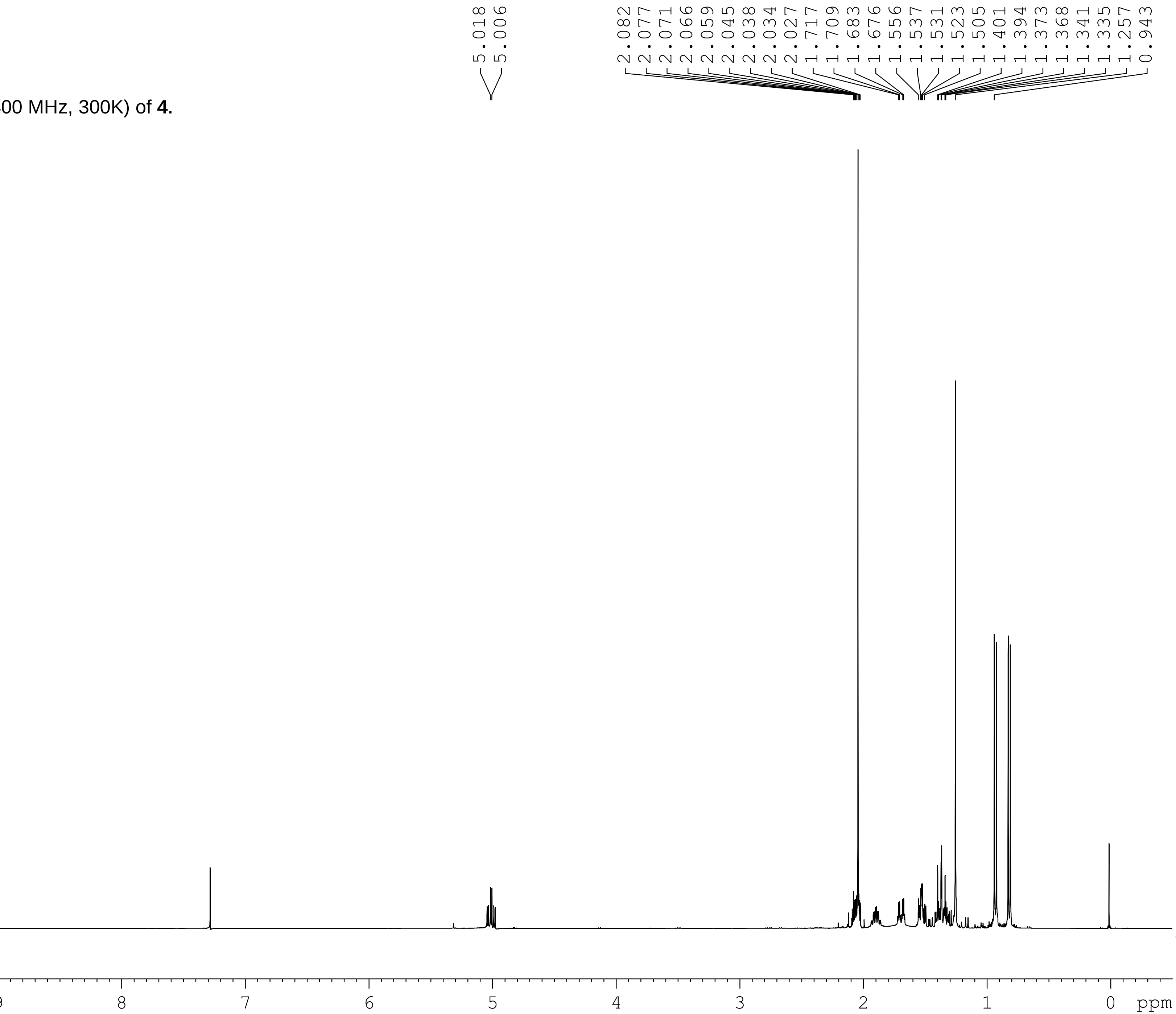




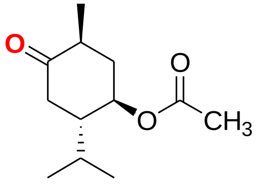
[1]H-NMR (CD_2Cl_2, 400 MHz, 300K) of **Δ-2P**.

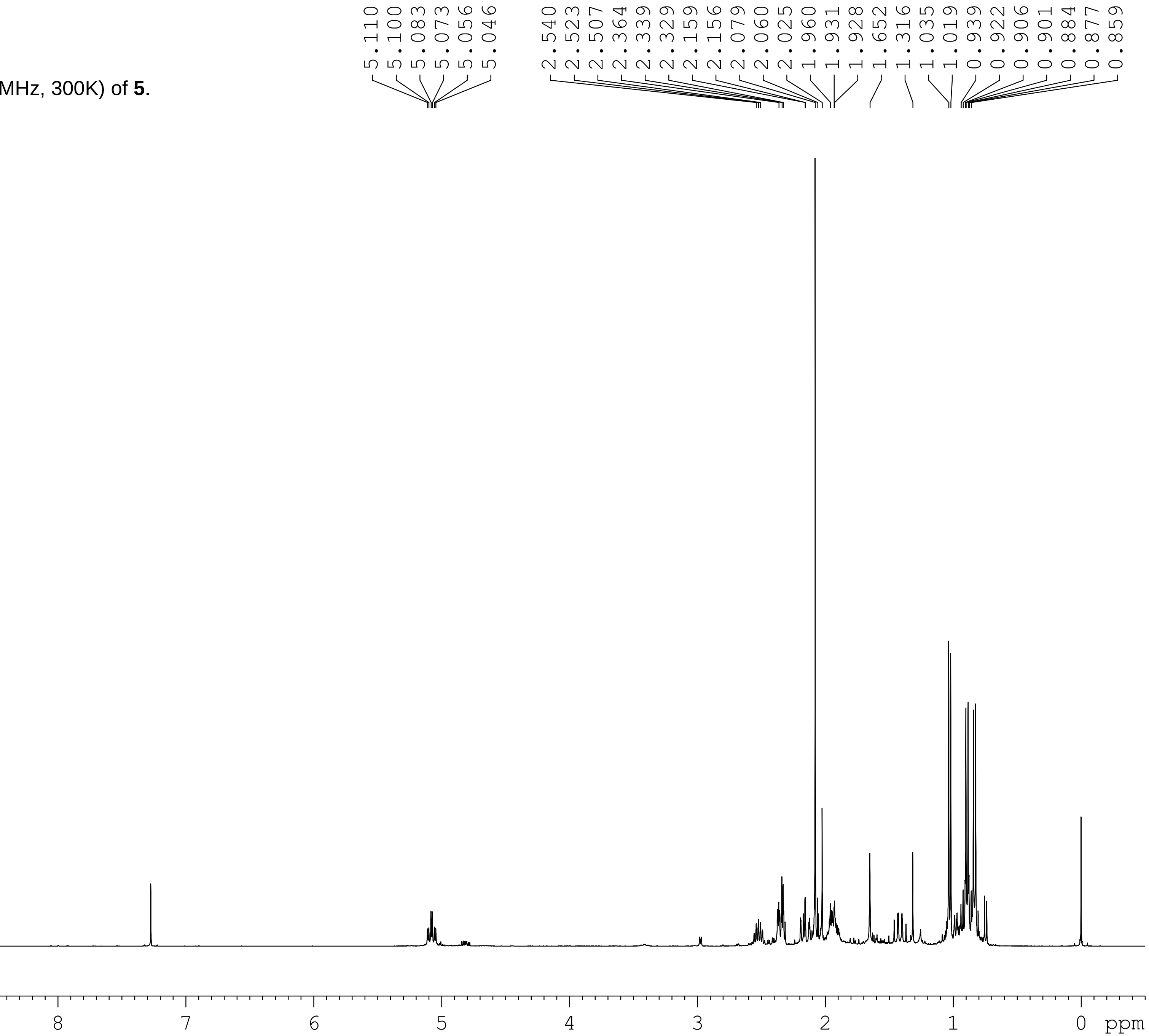
[1]H-NMR ($CDCl_3$, 400 MHz, 300K) of **4.**



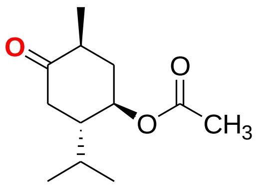


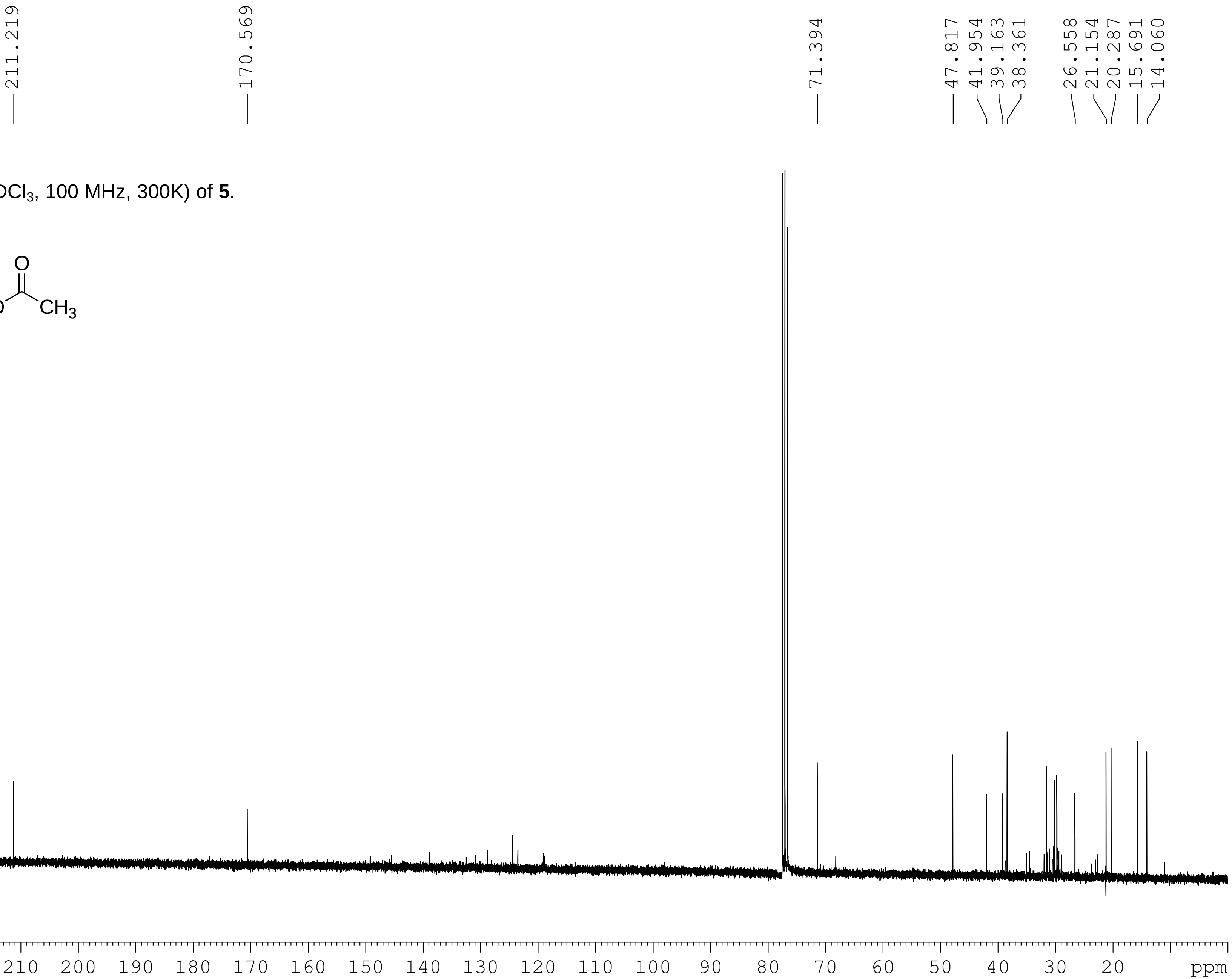
[1]H-NMR ($CDCl_3$, 400 MHz, 300K) of **5**.



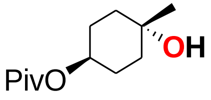


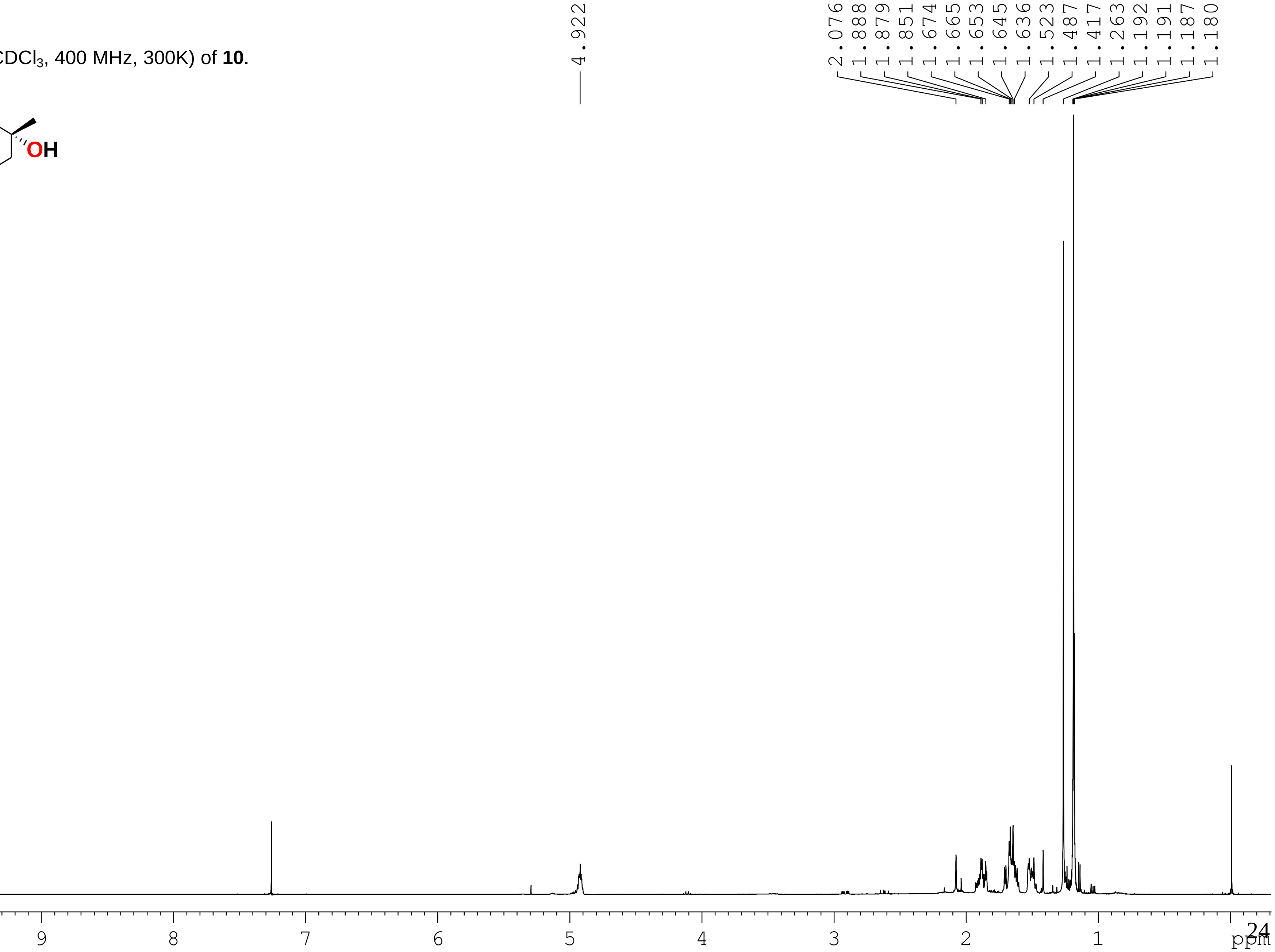
^{13}C-NMR ($CDCl_3$, 100 MHz, 300K) of **5**.



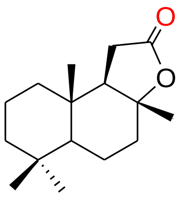


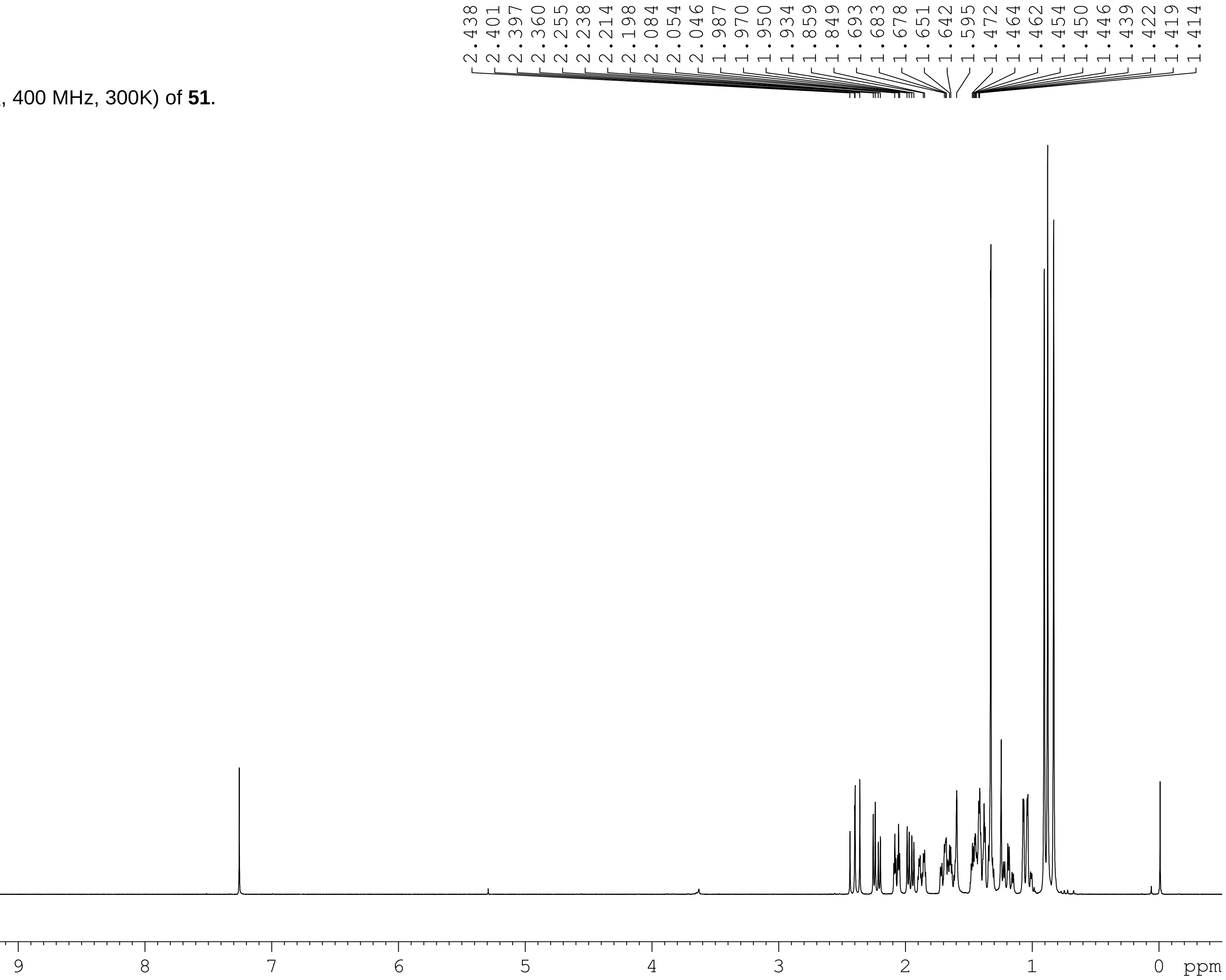
^{1}H-NMR ($CDCl_3$, 400 MHz, 300K) of **10**.



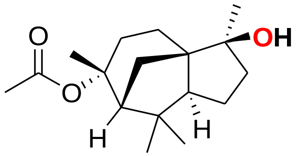


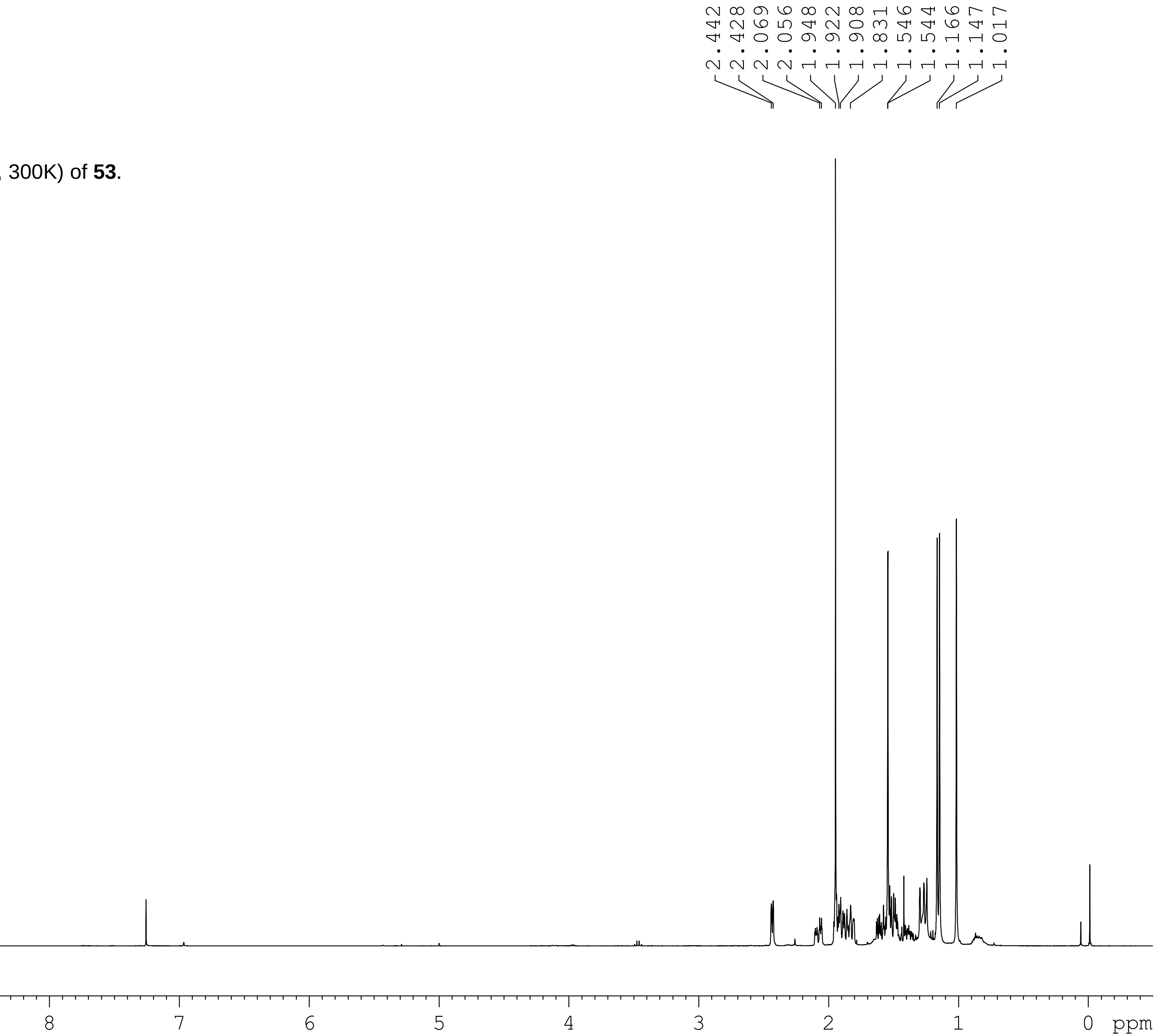
[1]H-NMR ($CDCl_3$, 400 MHz, 300K) of **51**.



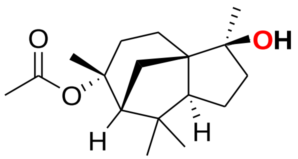


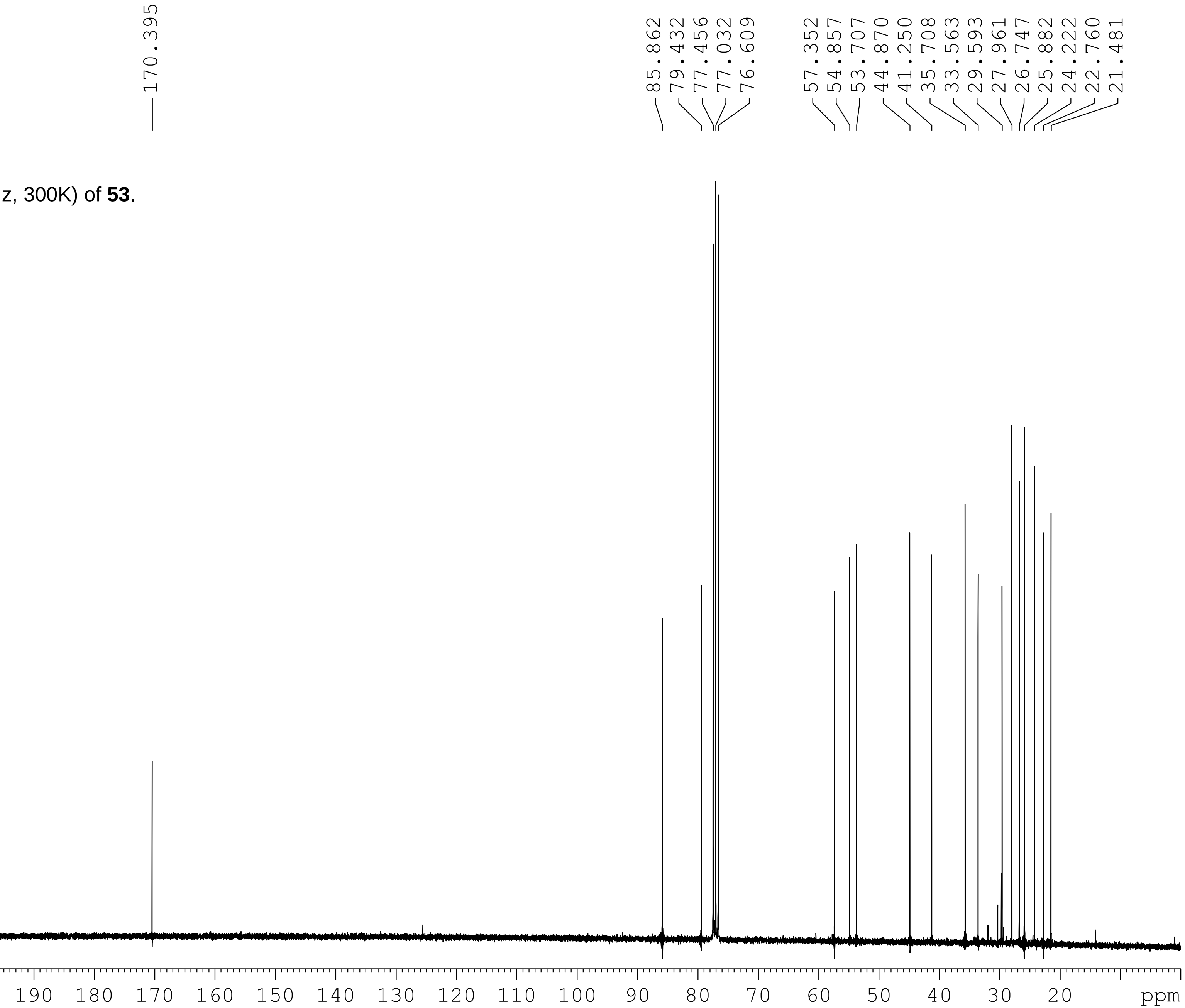
^{1}H-NMR ($CDCl_3$, 400 MHz, 300K) of **53**.



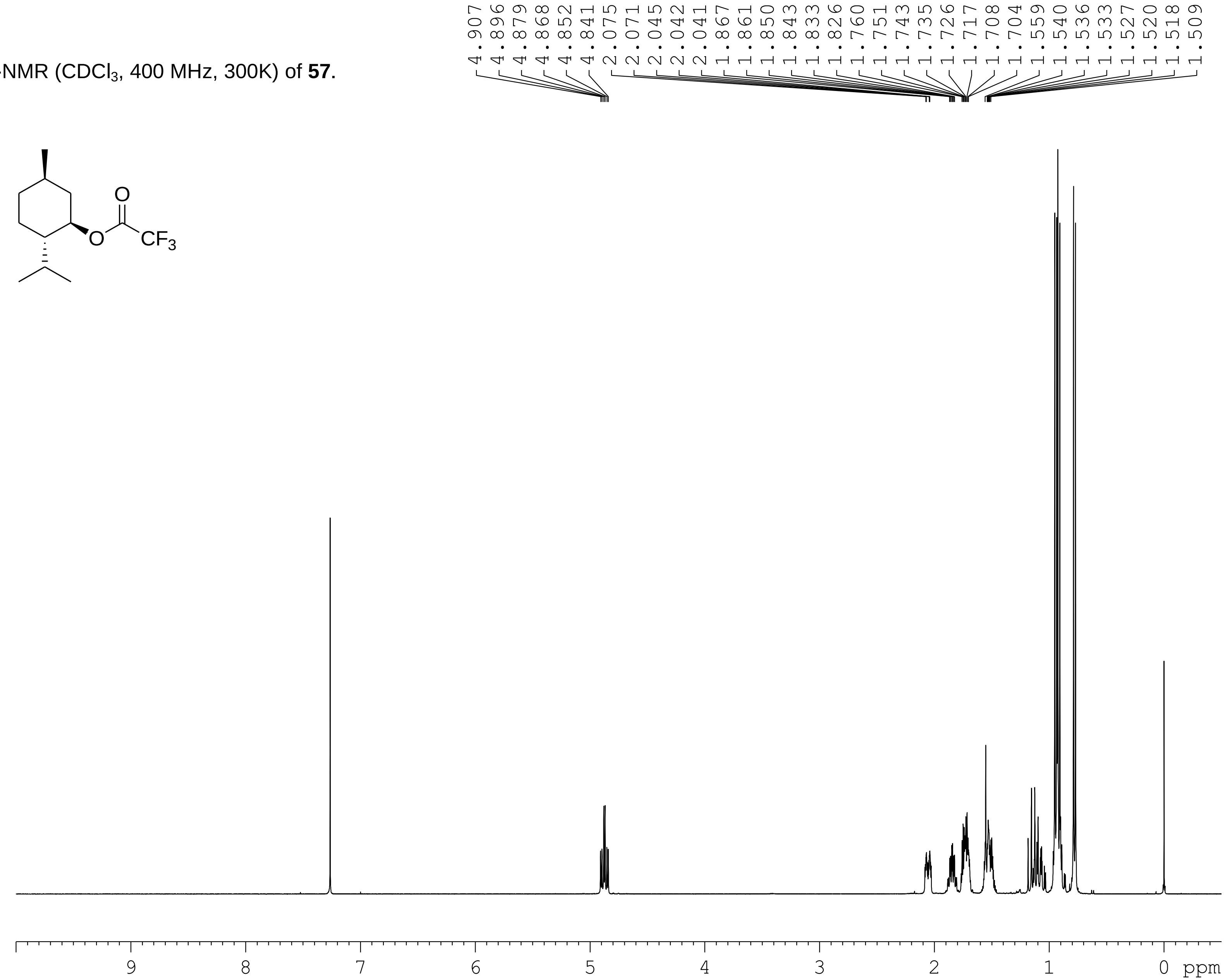


^{13}C-NMR ($CDCl_3$, 100 MHz, 300K) of **53**.

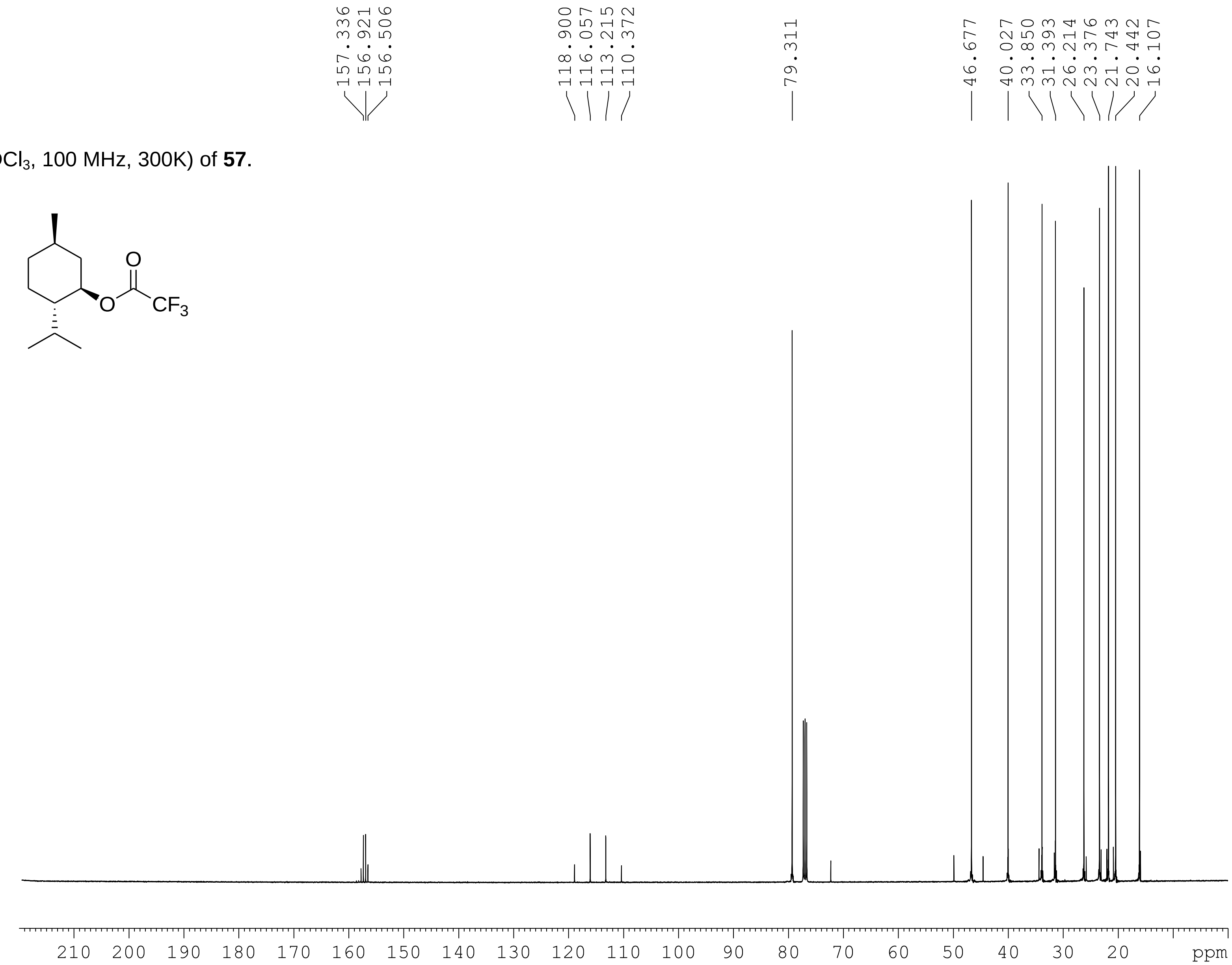




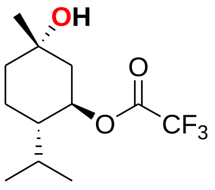
^{1}H-NMR ($CDCl_3$, 400 MHz, 300K) of **57**.

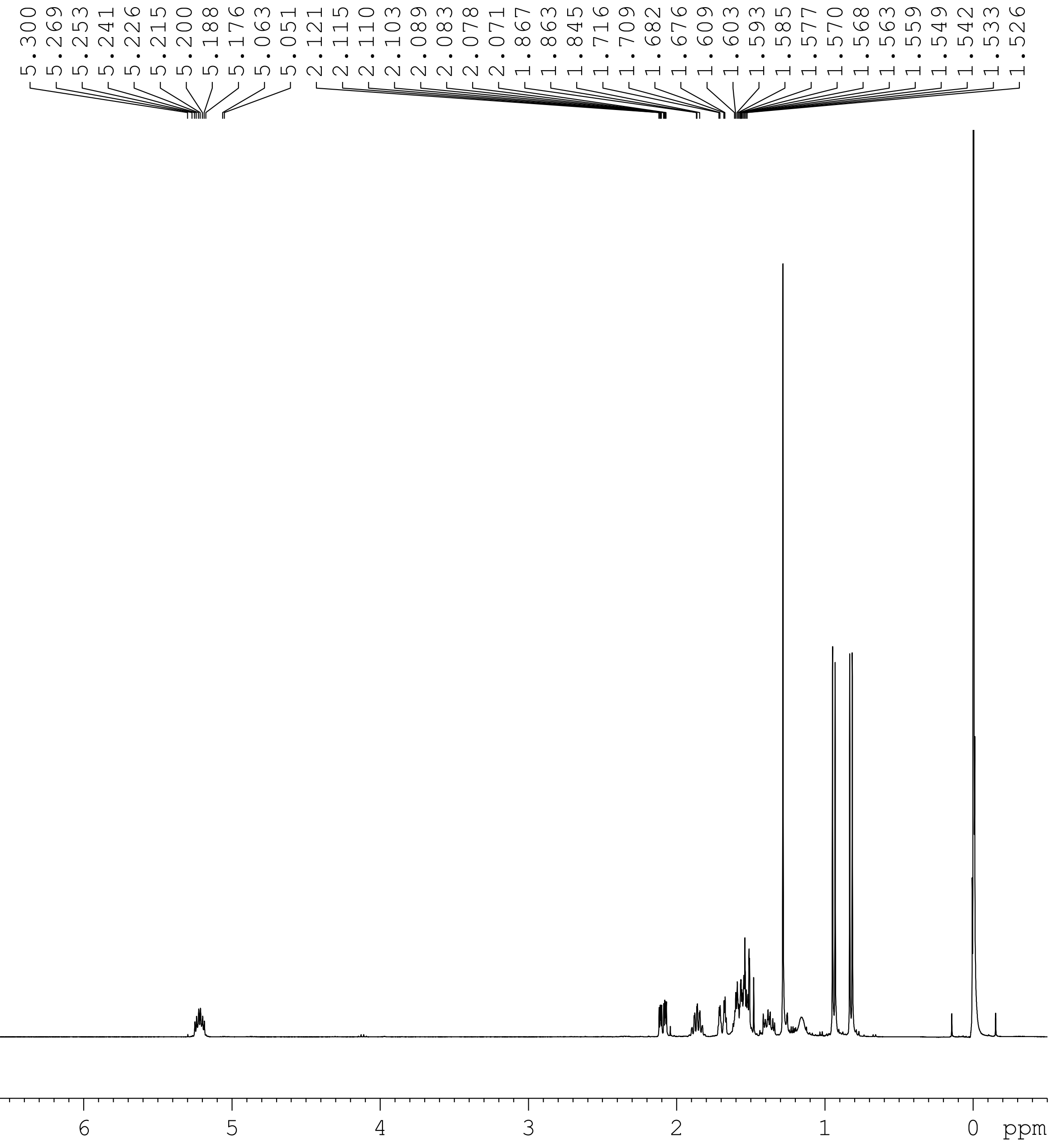


^{13}C-NMR ($CDCl_3$, 100 MHz, 300K) of **57**.



[1]H-NMR ($CDCl_3$, 400 MHz, 300K) of **58**.





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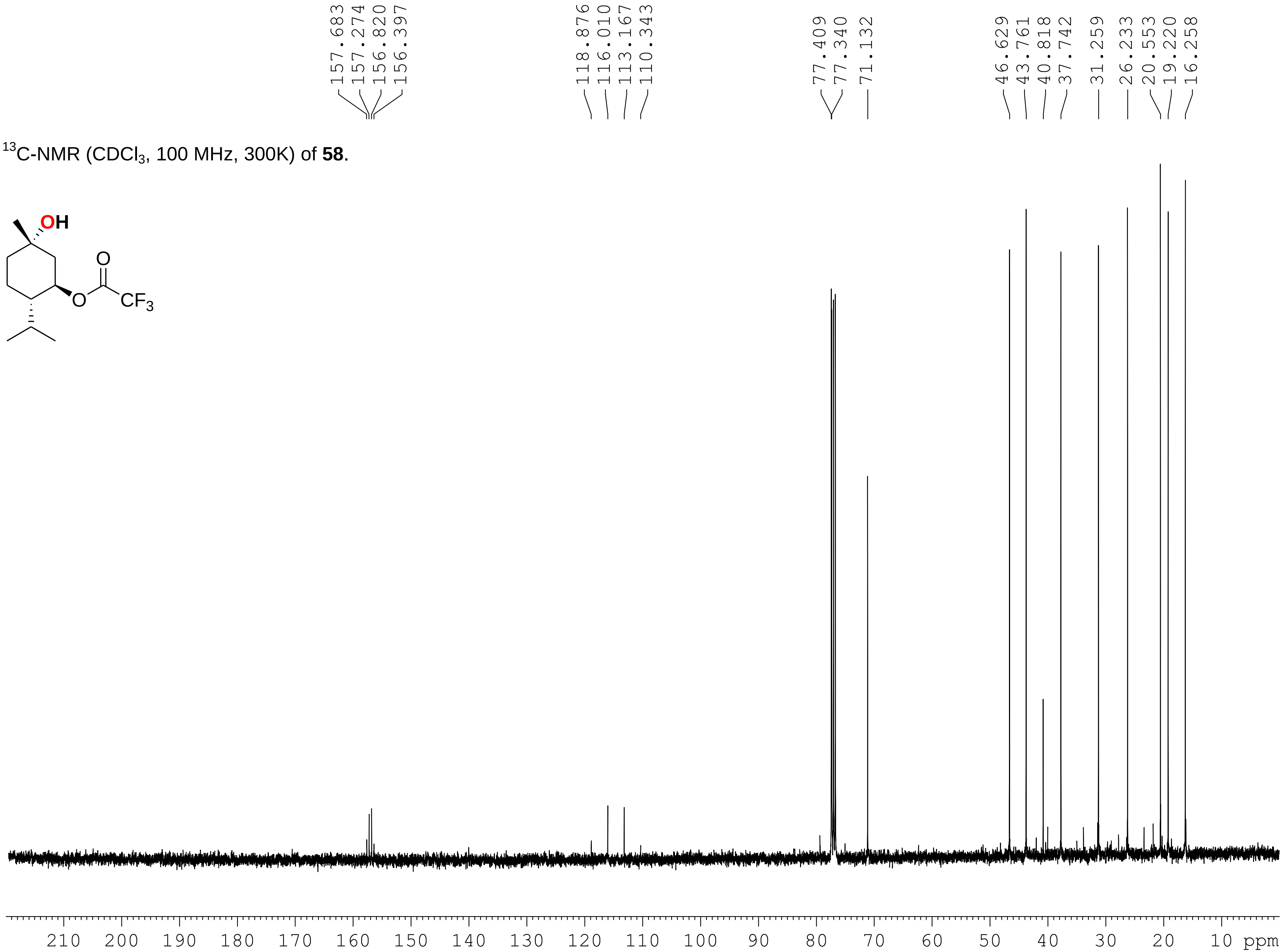
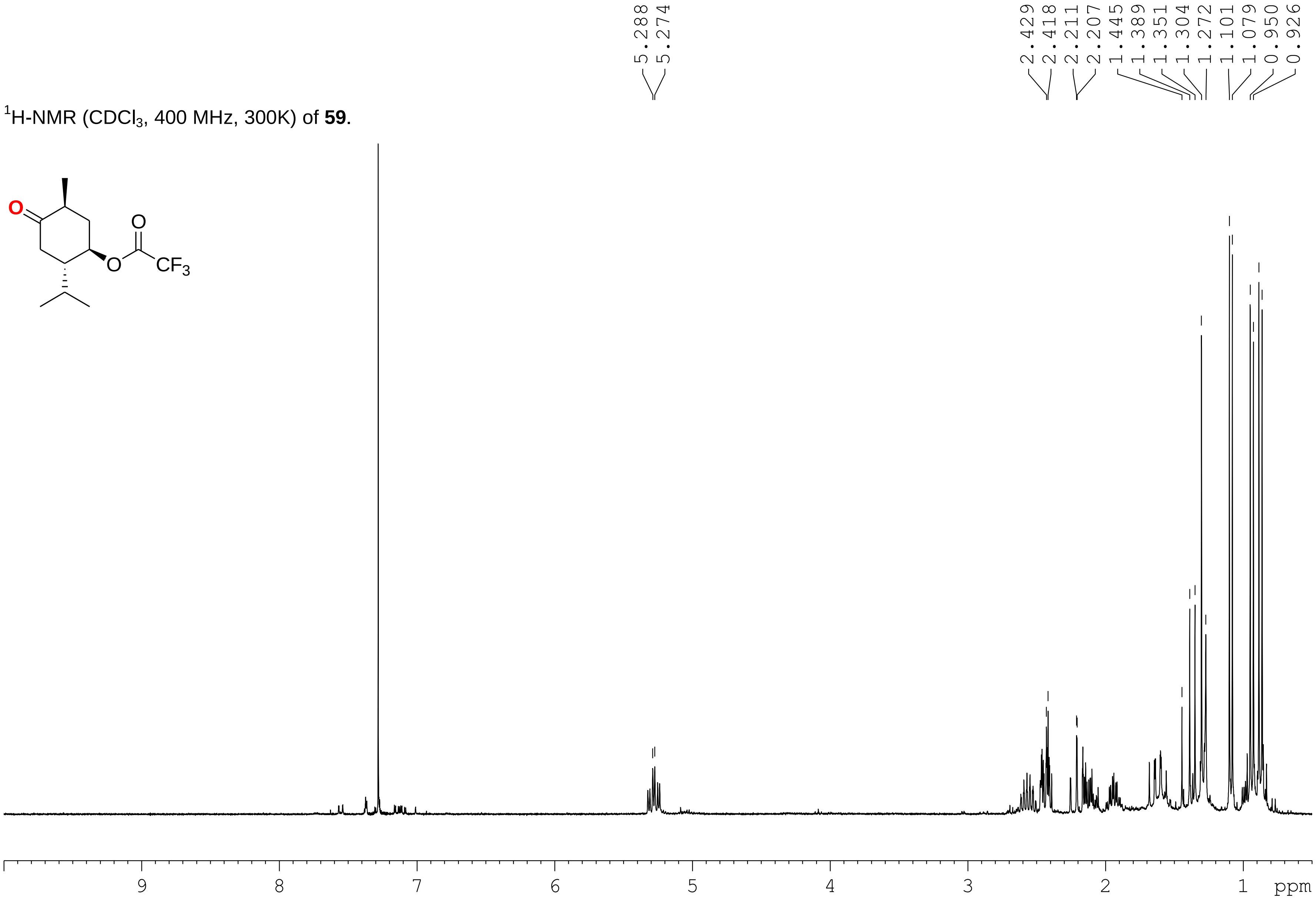
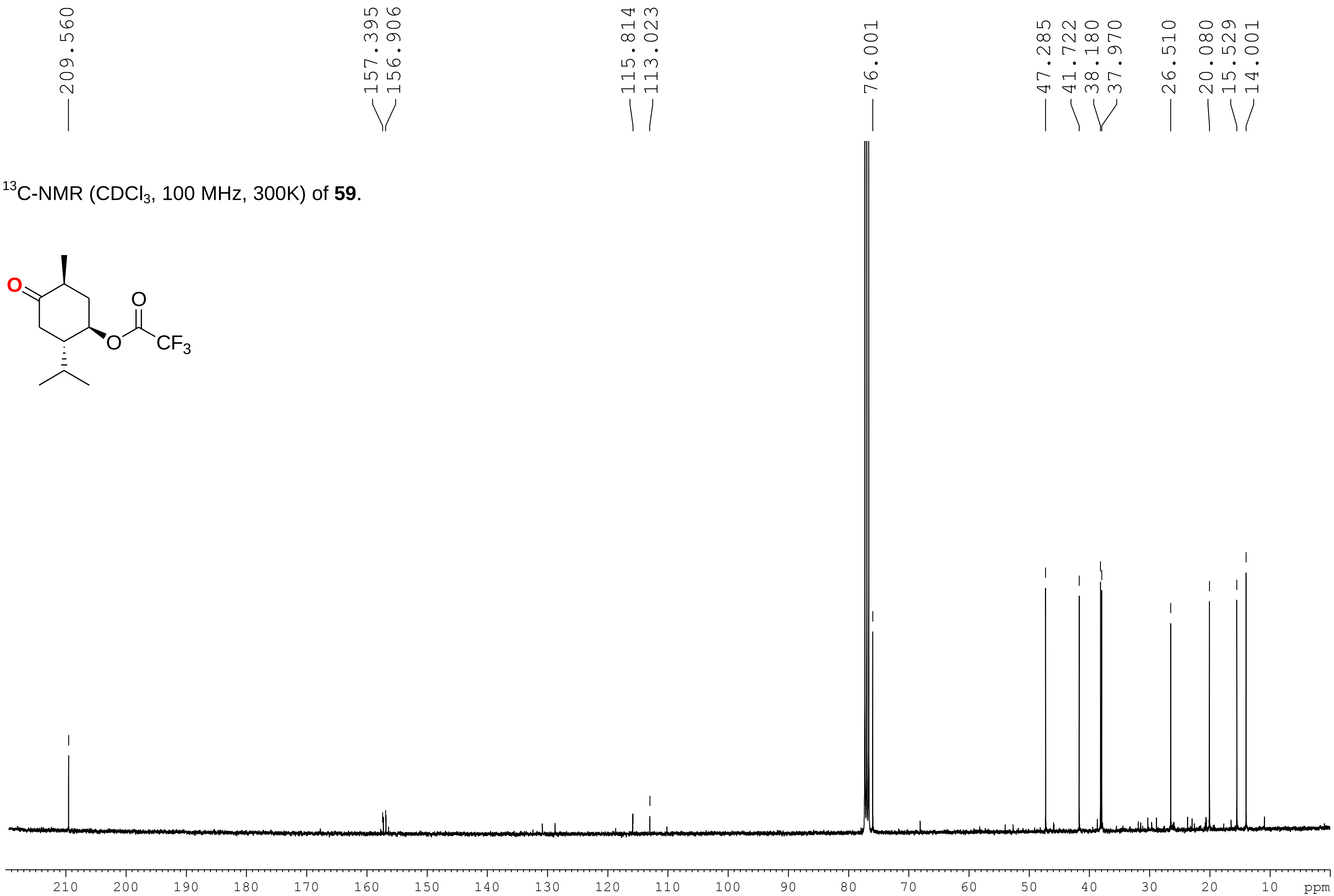


^{1}H-NMR ($CDCl_3$, 400 MHz, 300K) of **59**.





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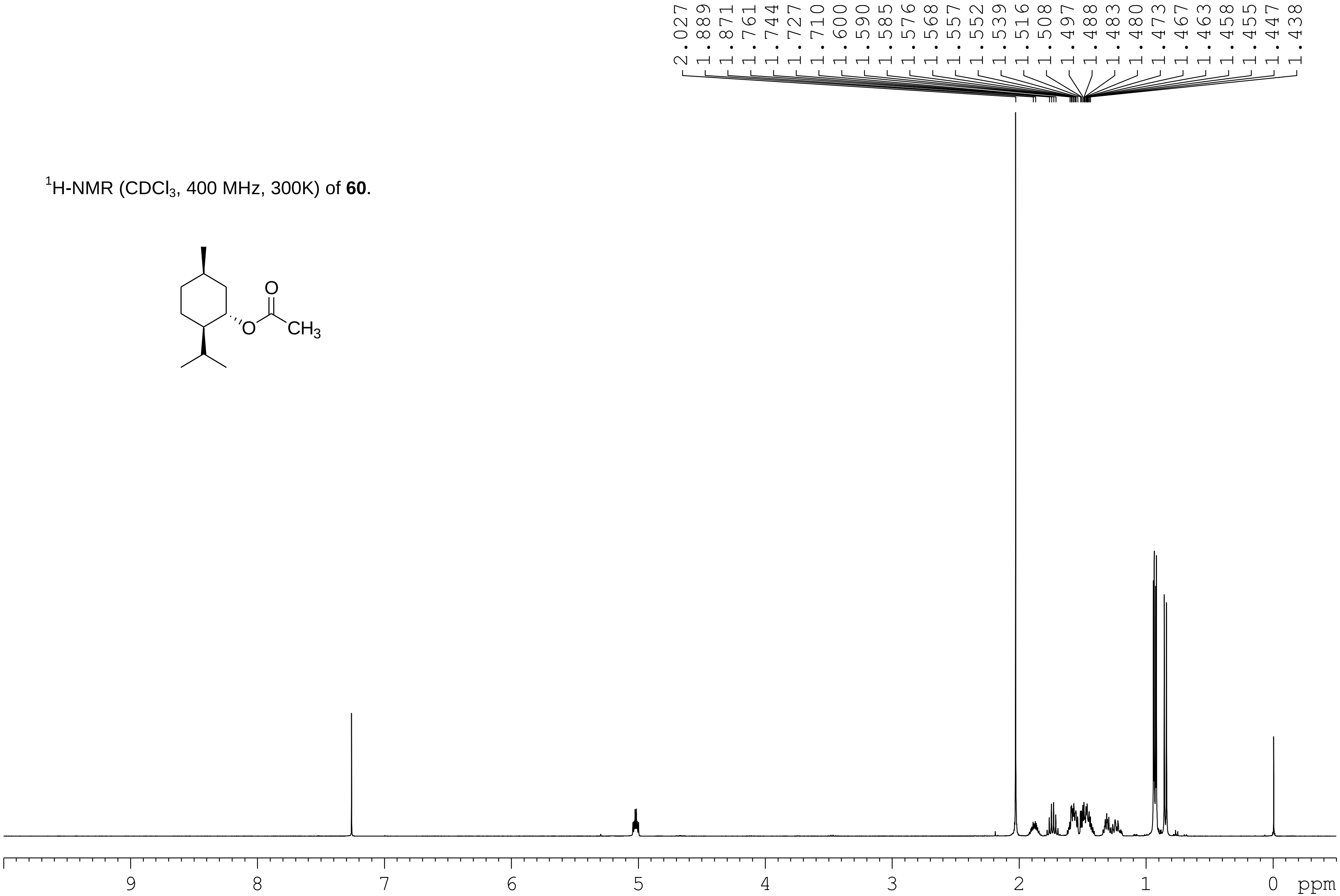
^{1}H-NMR ($CDCl_3$, 400 MHz, 300K) of **60**.



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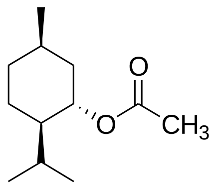
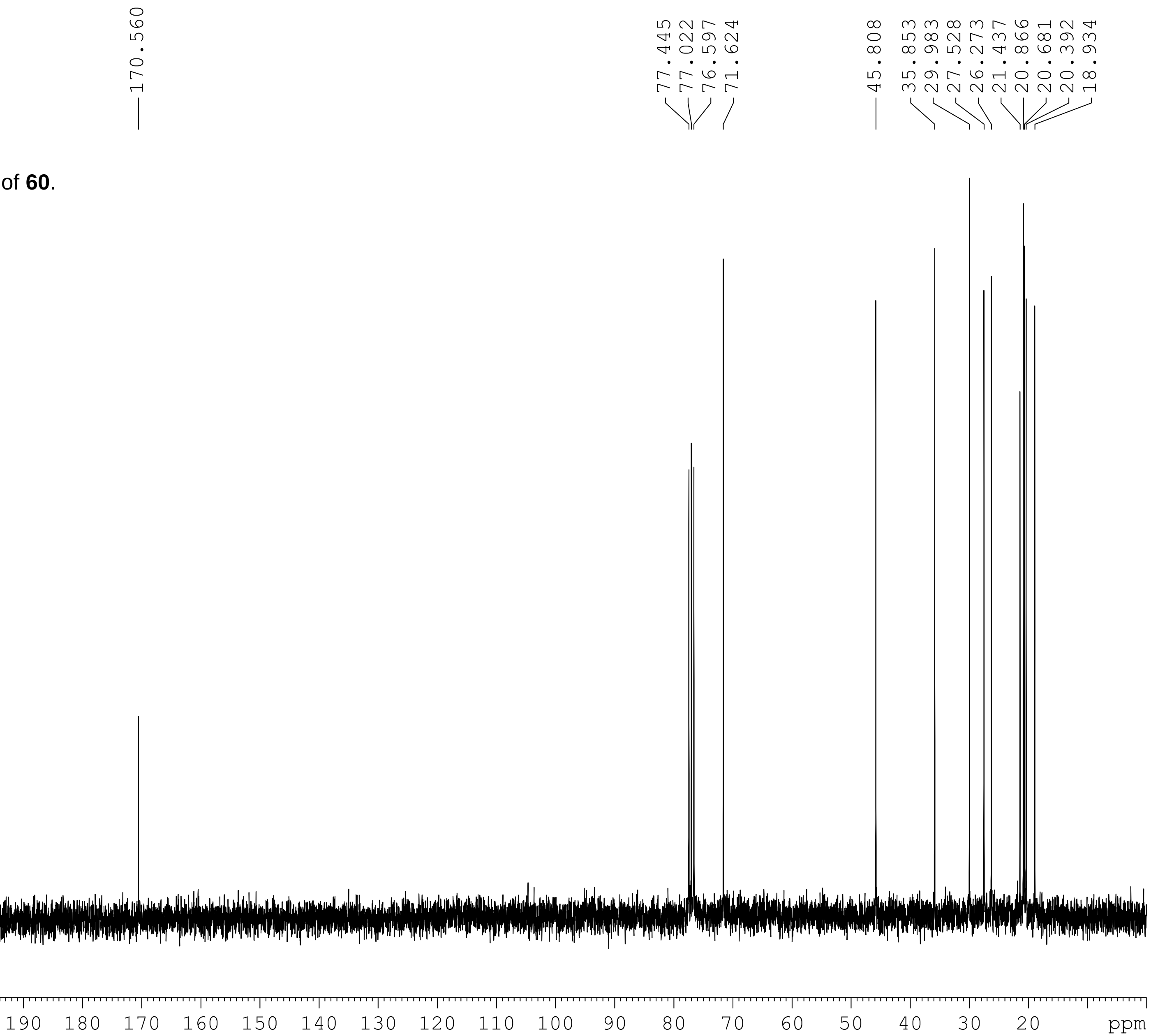
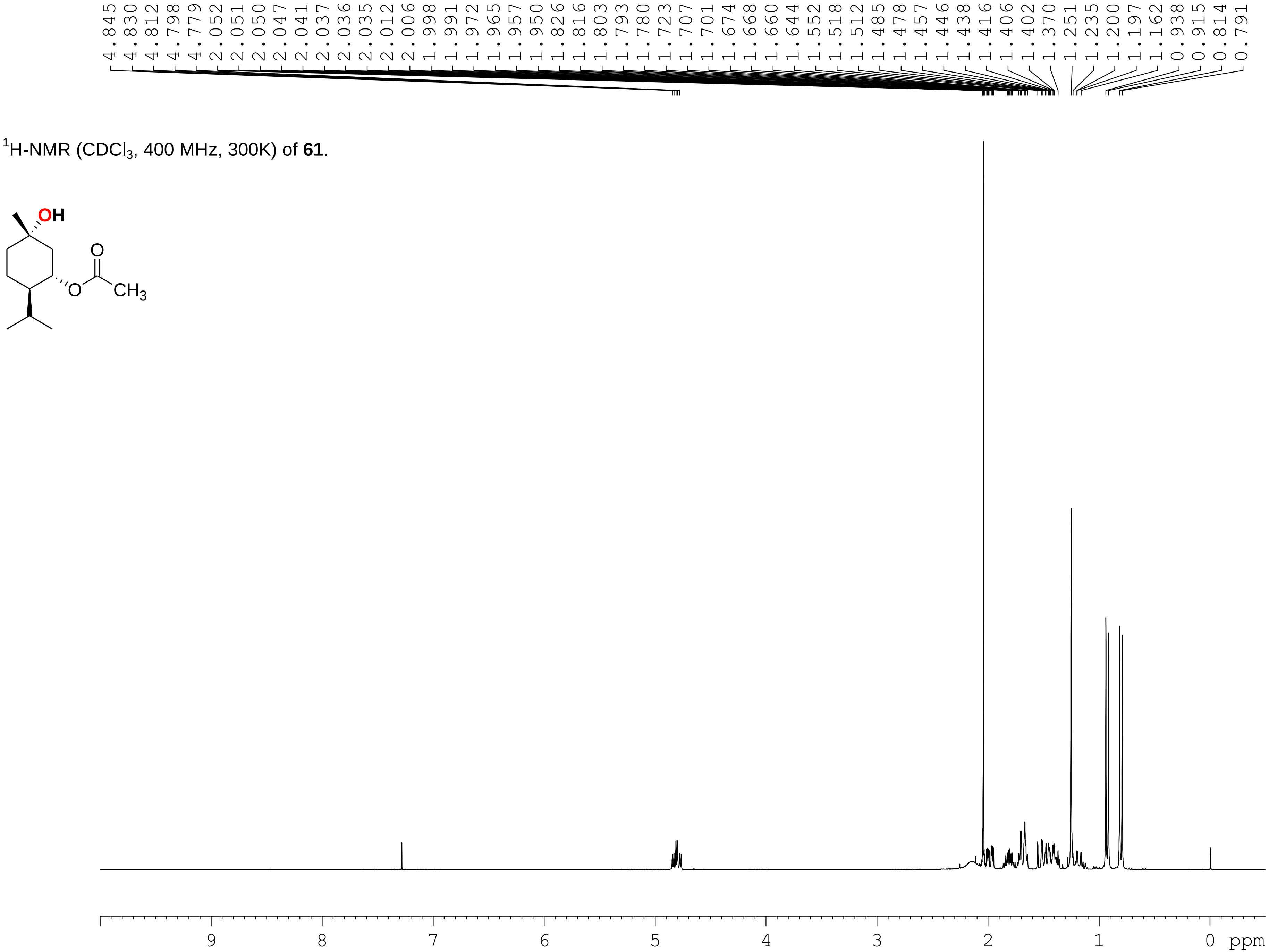
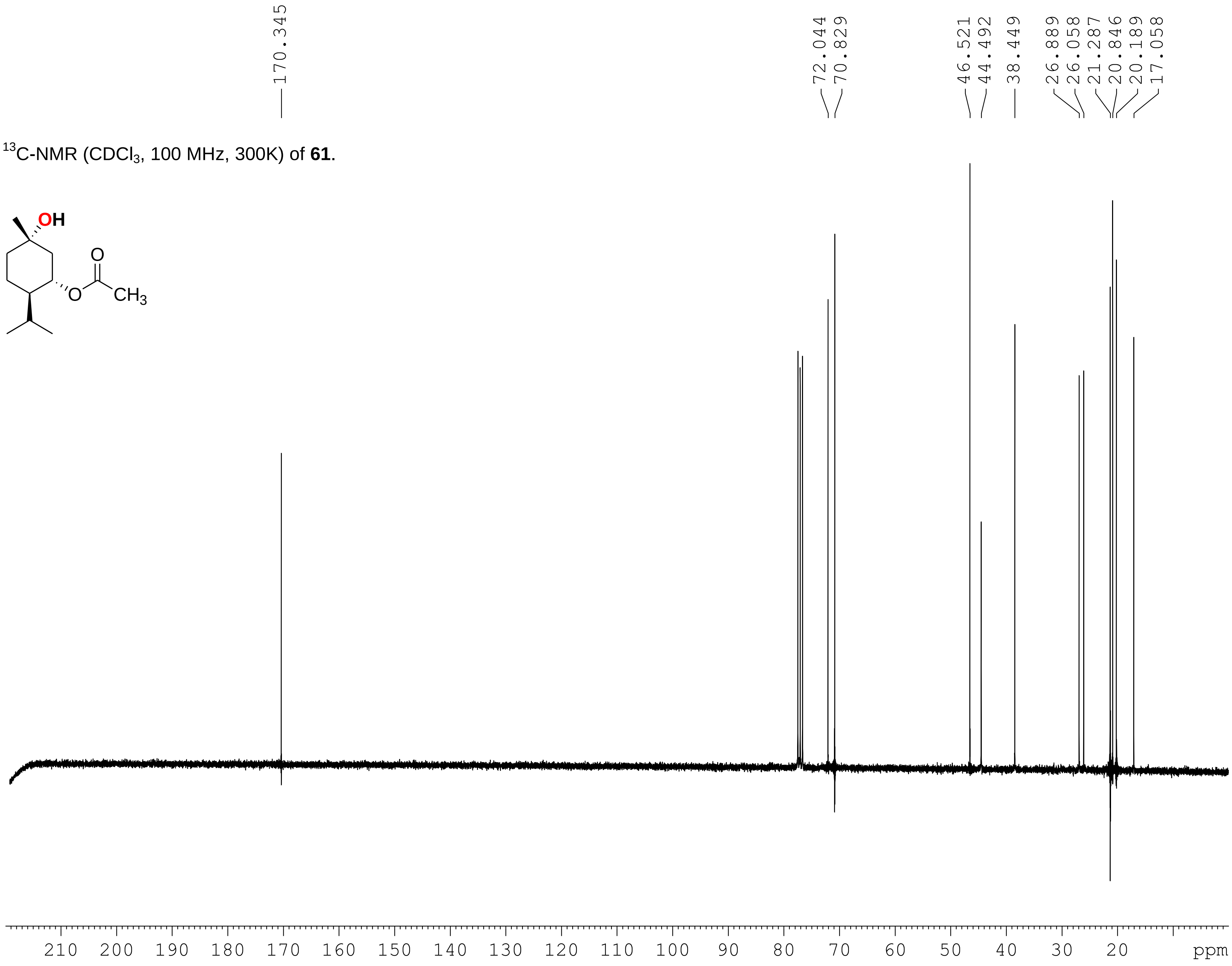




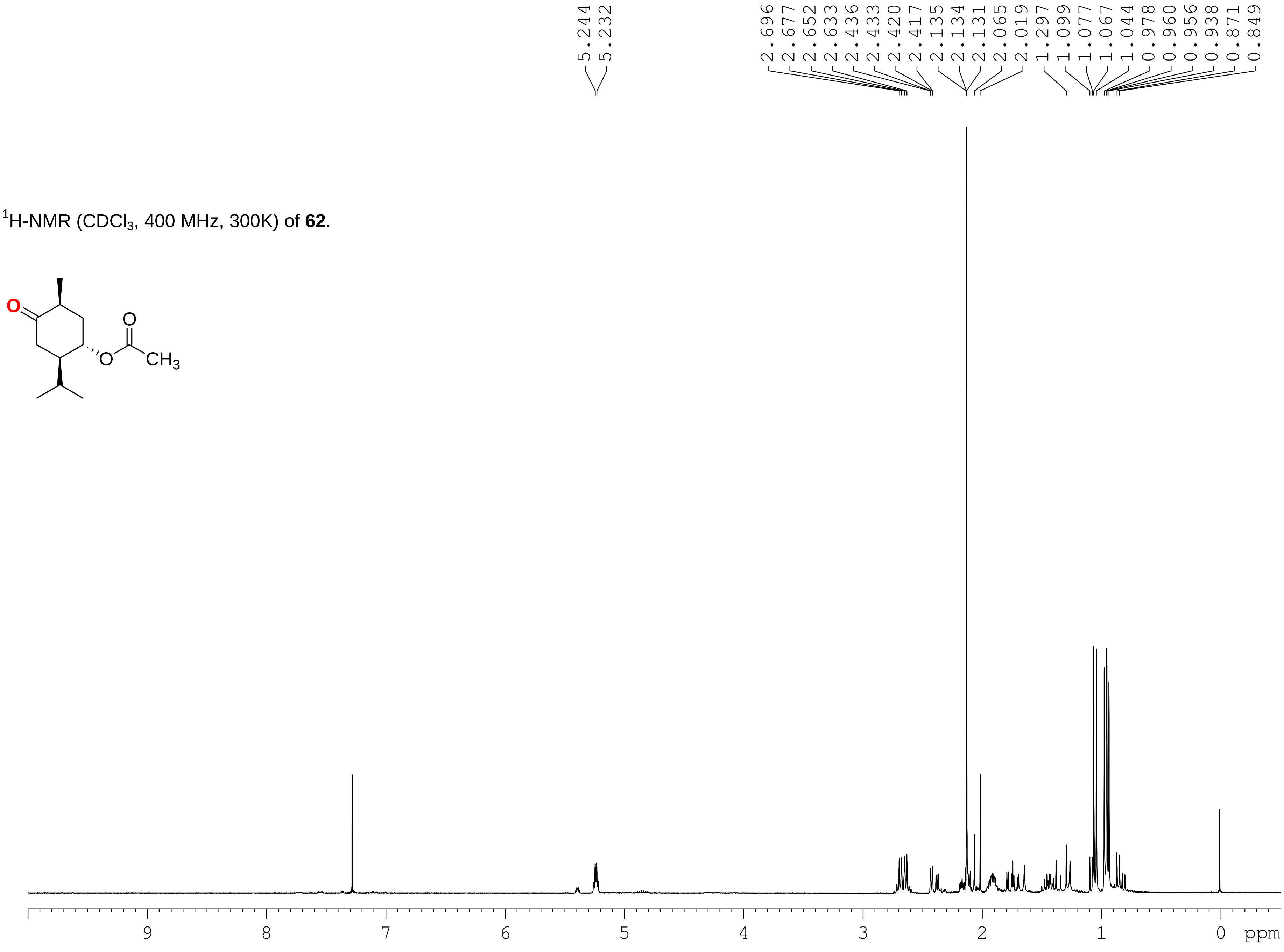


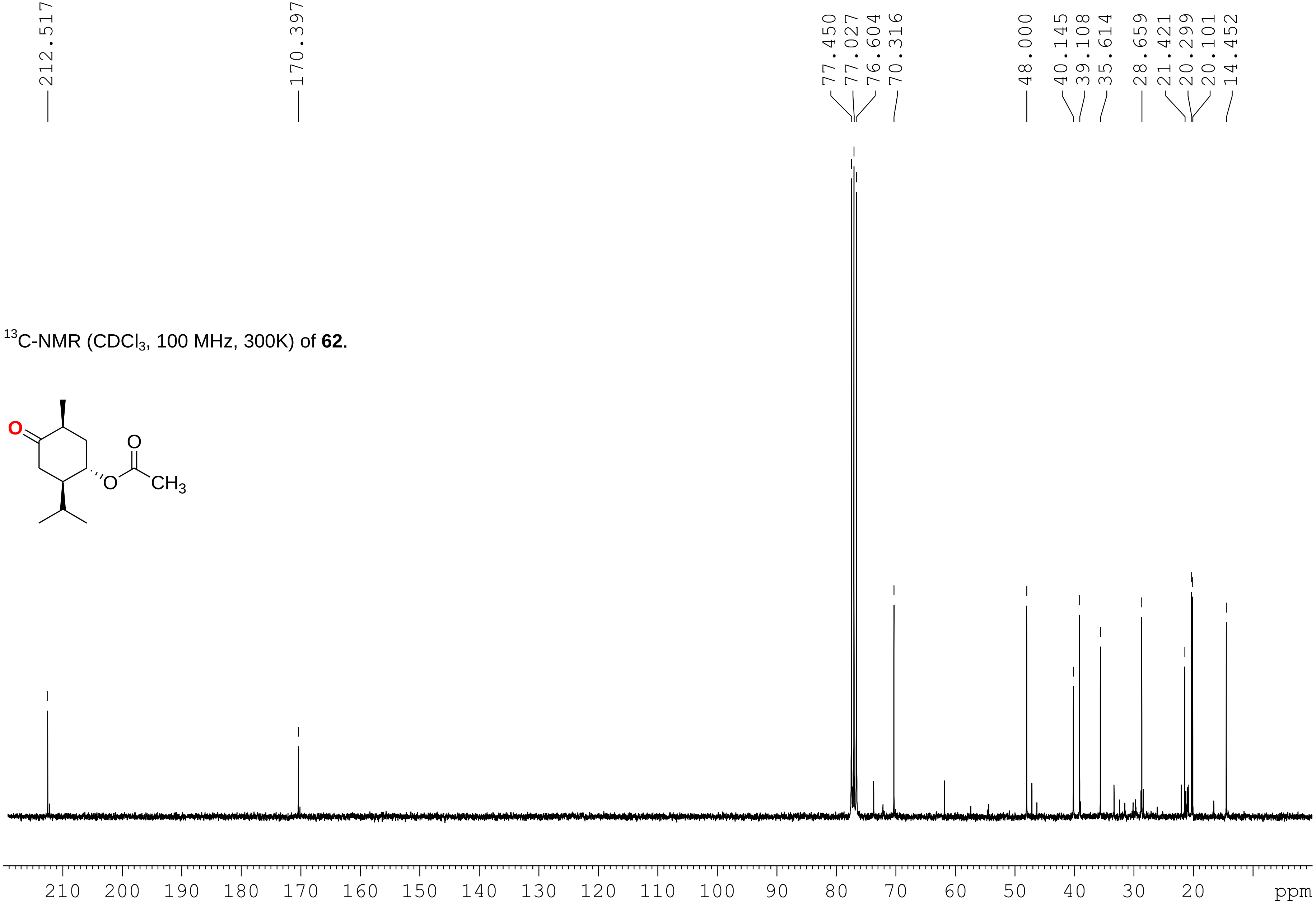
^{1}H-NMR ($CDCl_3$, 400 MHz, 300K) of **61**.

^{13}C-NMR ($CDCl_3$, 100 MHz, 300K) of **61**.



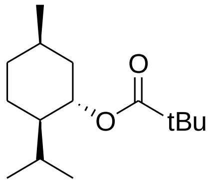
[1]H-NMR ($CDCl_3$, 400 MHz, 300K) of **62**.

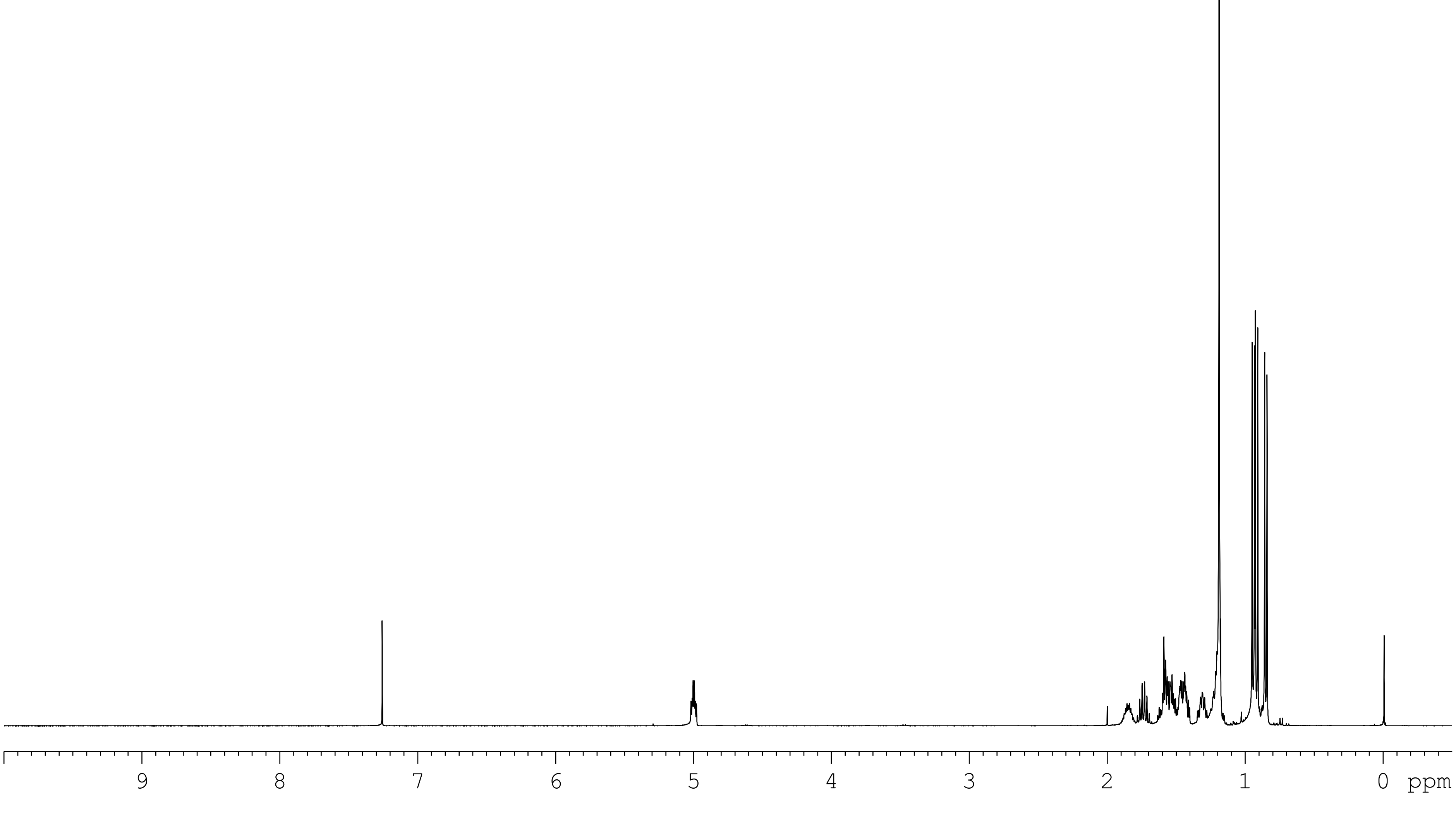




^{13}C-NMR ($CDCl_3$, 100 MHz, 300K) of **62**.

[1]H-NMR ($CDCl_3$, 400 MHz, 300K) of **63**.





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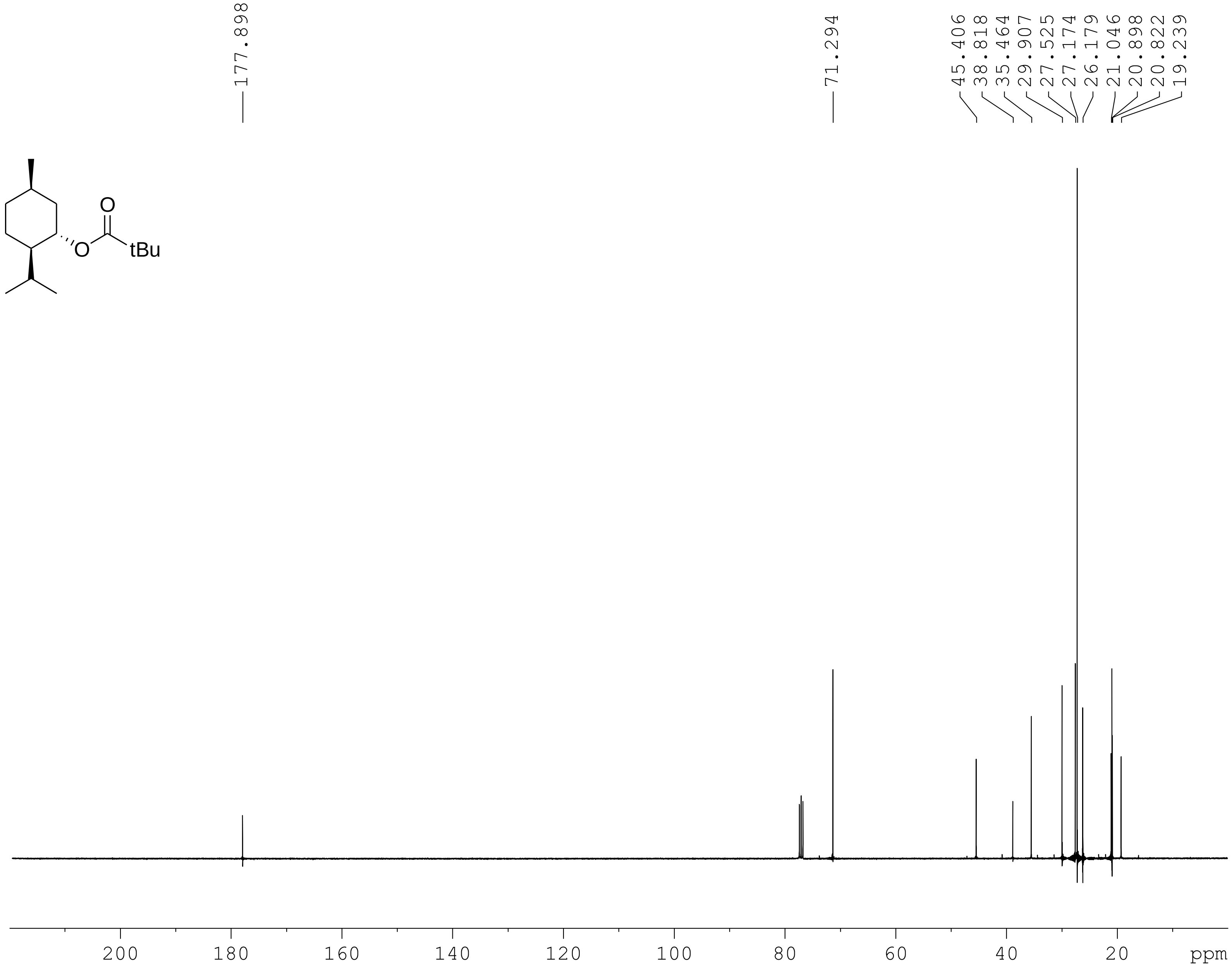
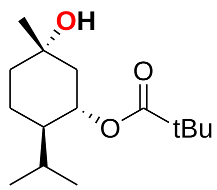
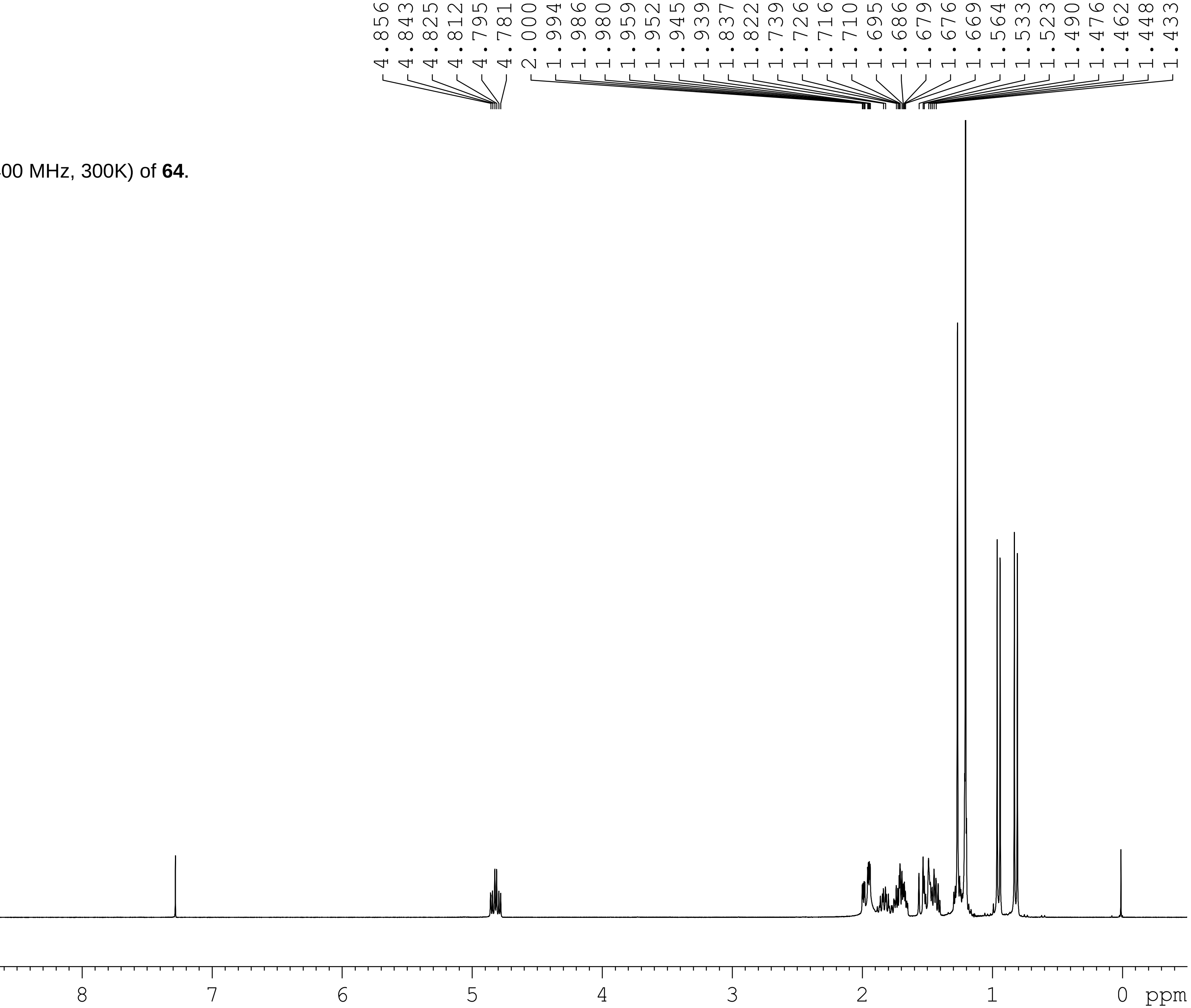


^{1}H-NMR ($CDCl_3$, 400 MHz, 300K) of **64**.





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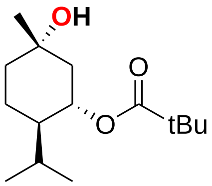
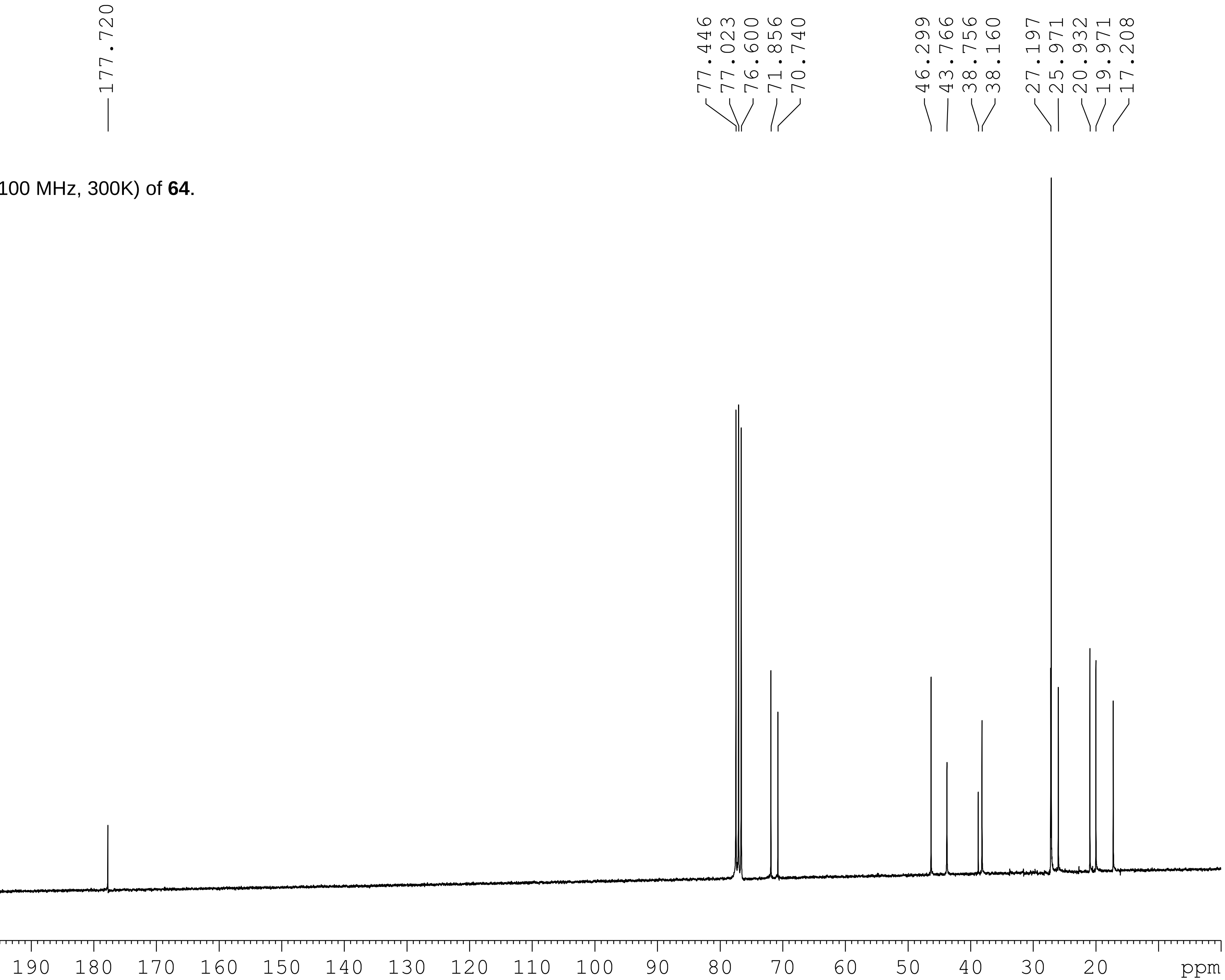
^{13}C-NMR ($CDCl_3$, 100 MHz, 300K) of **64**.





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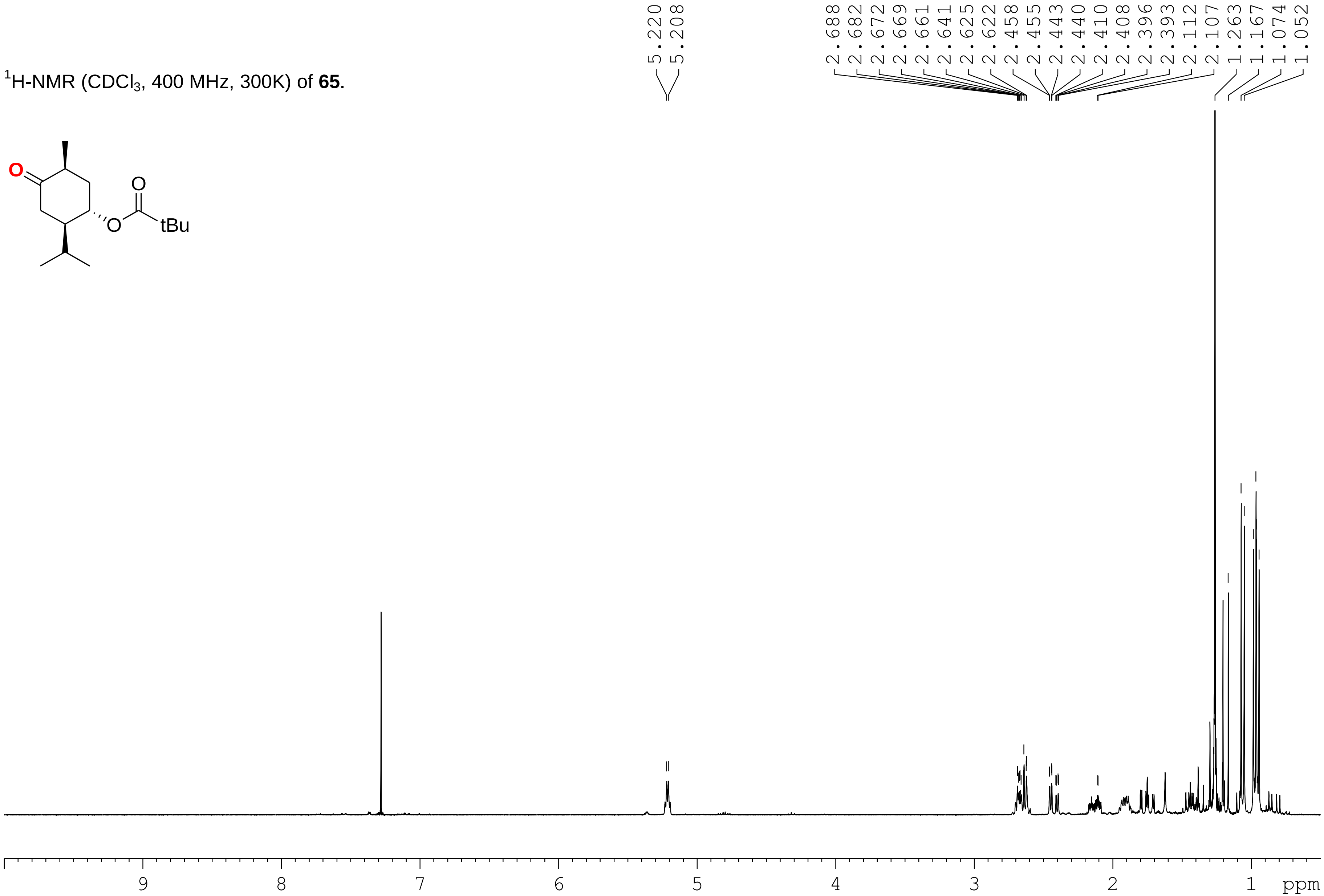

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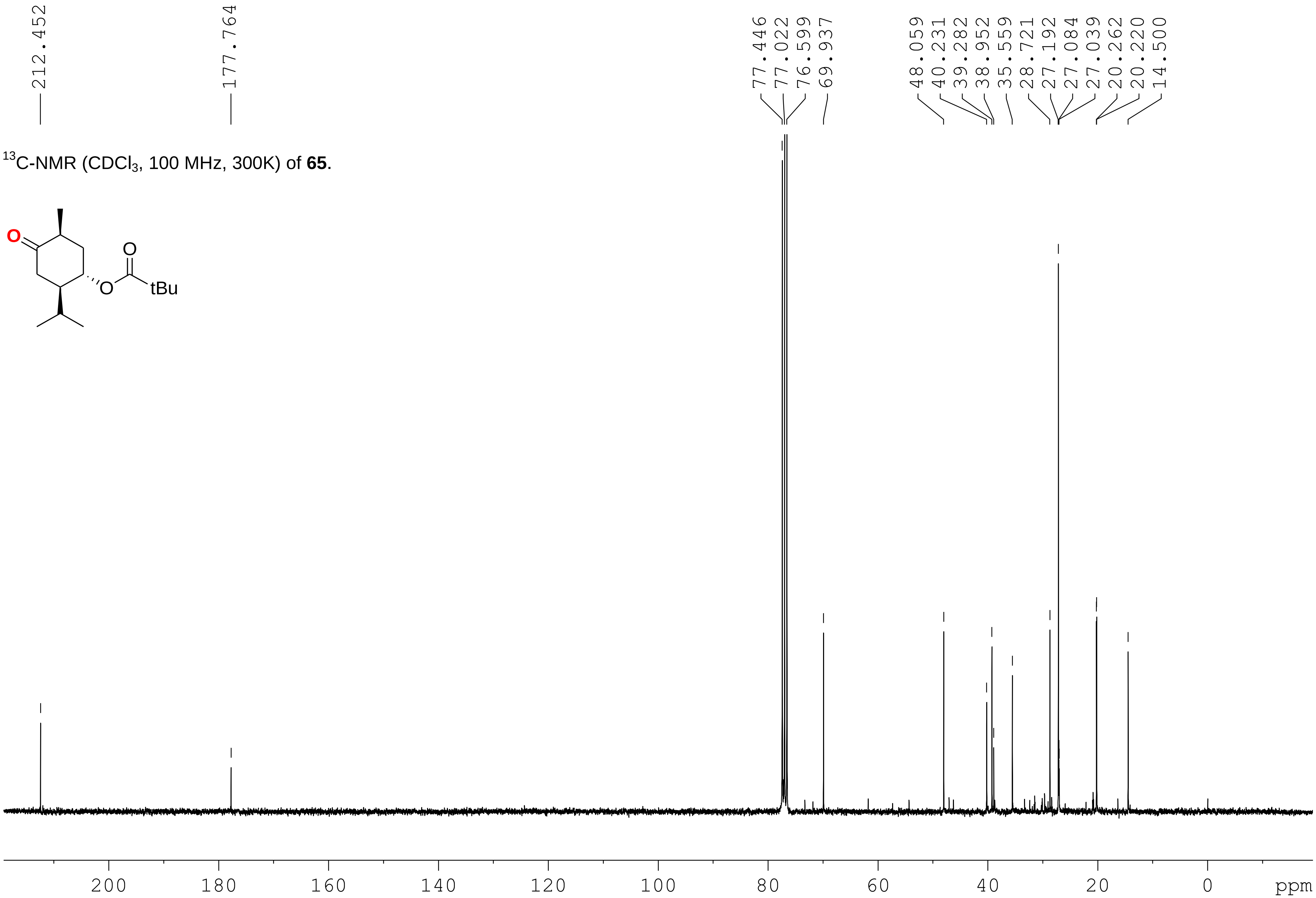
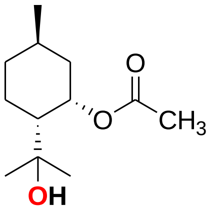
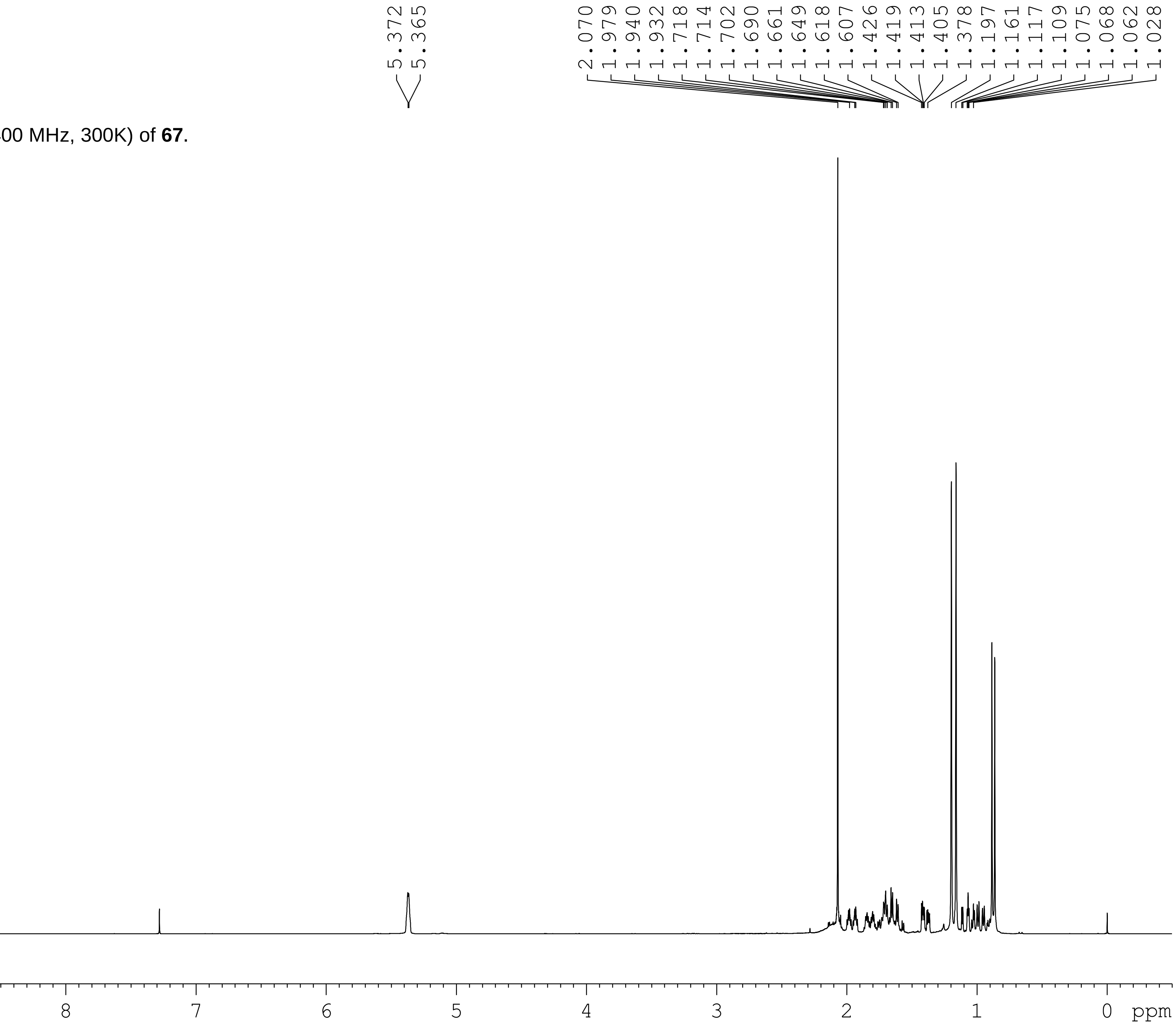
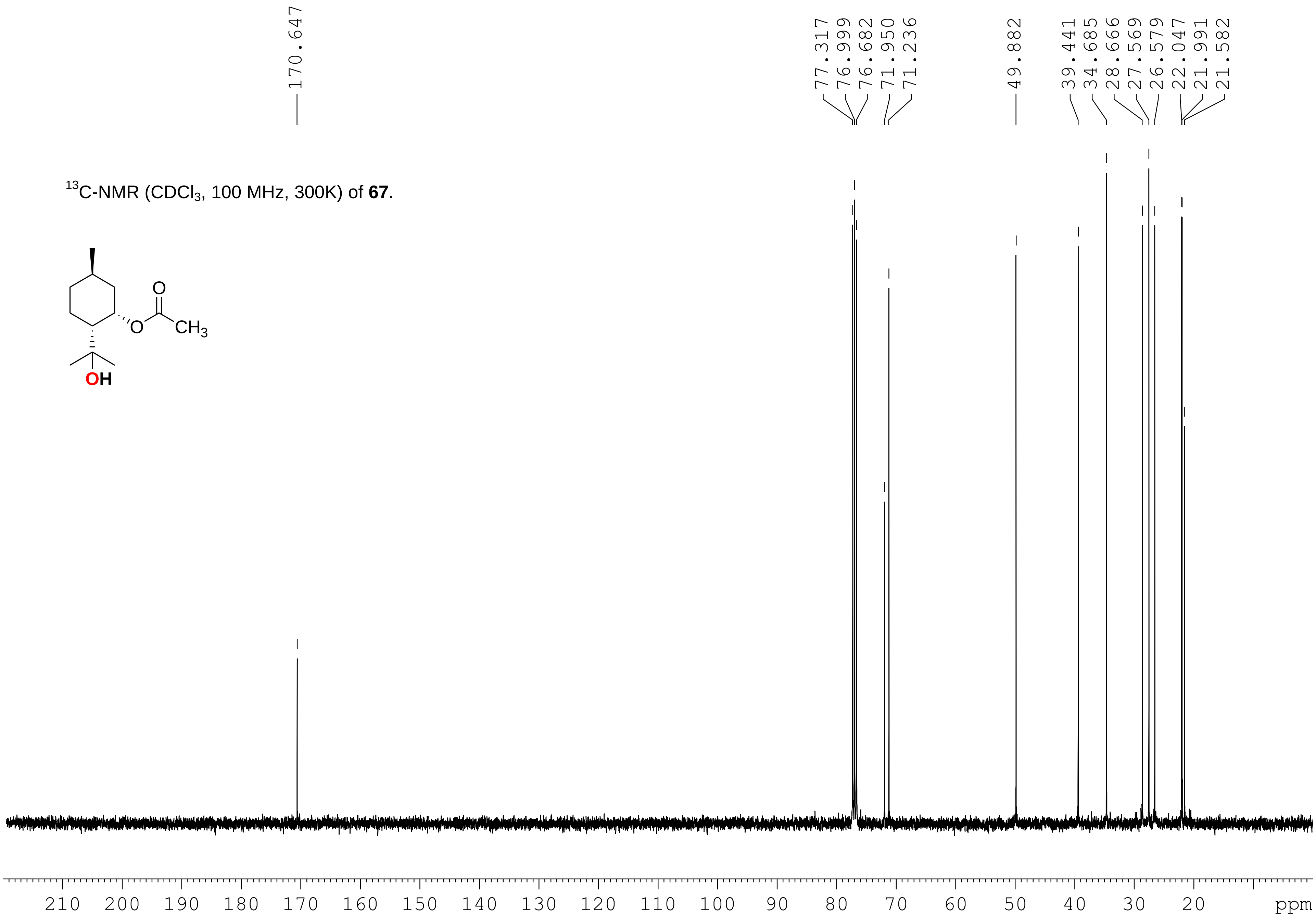


[1]H-NMR ($CDCl_3$, 400 MHz, 300K) of **67**.







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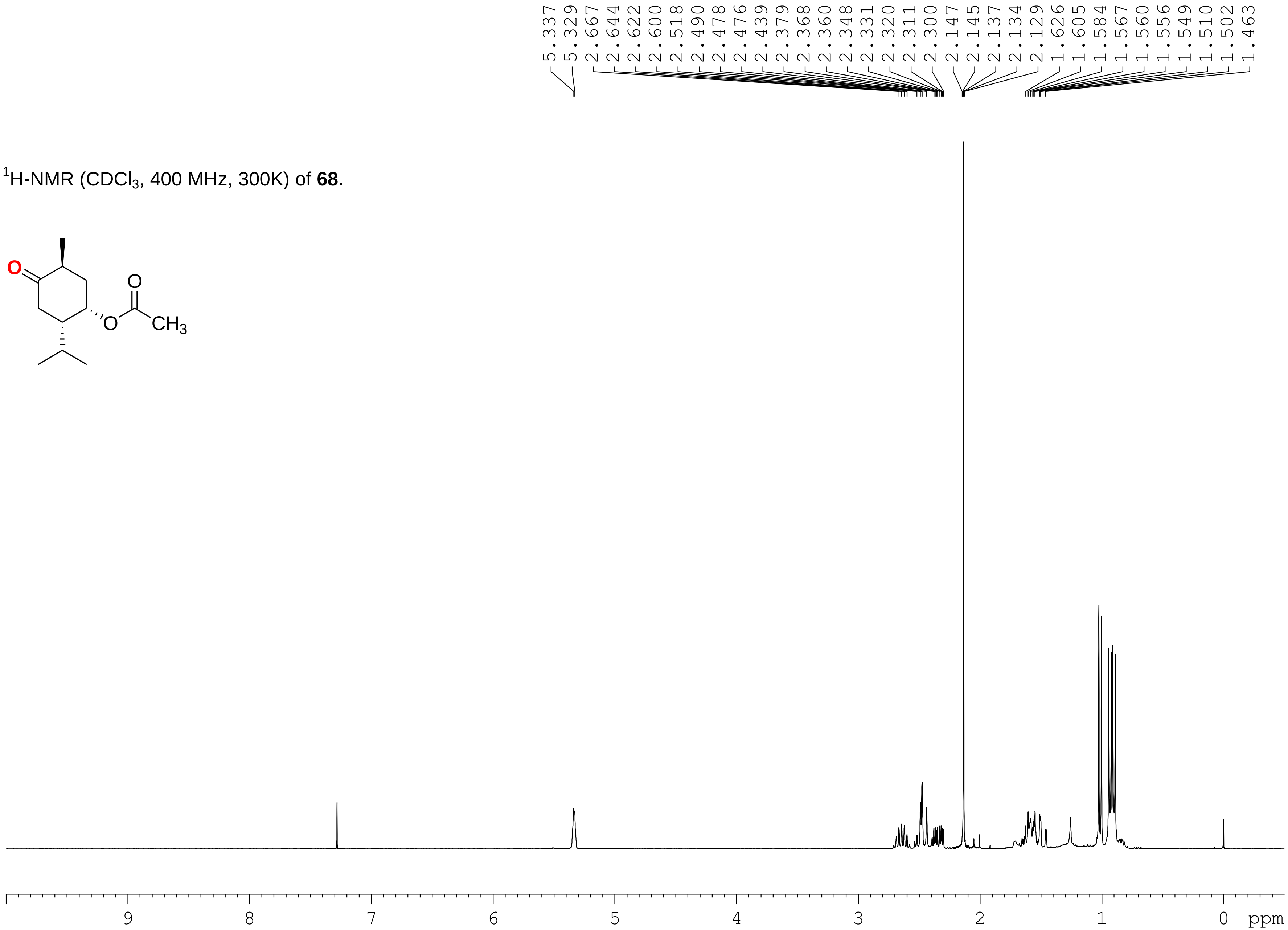
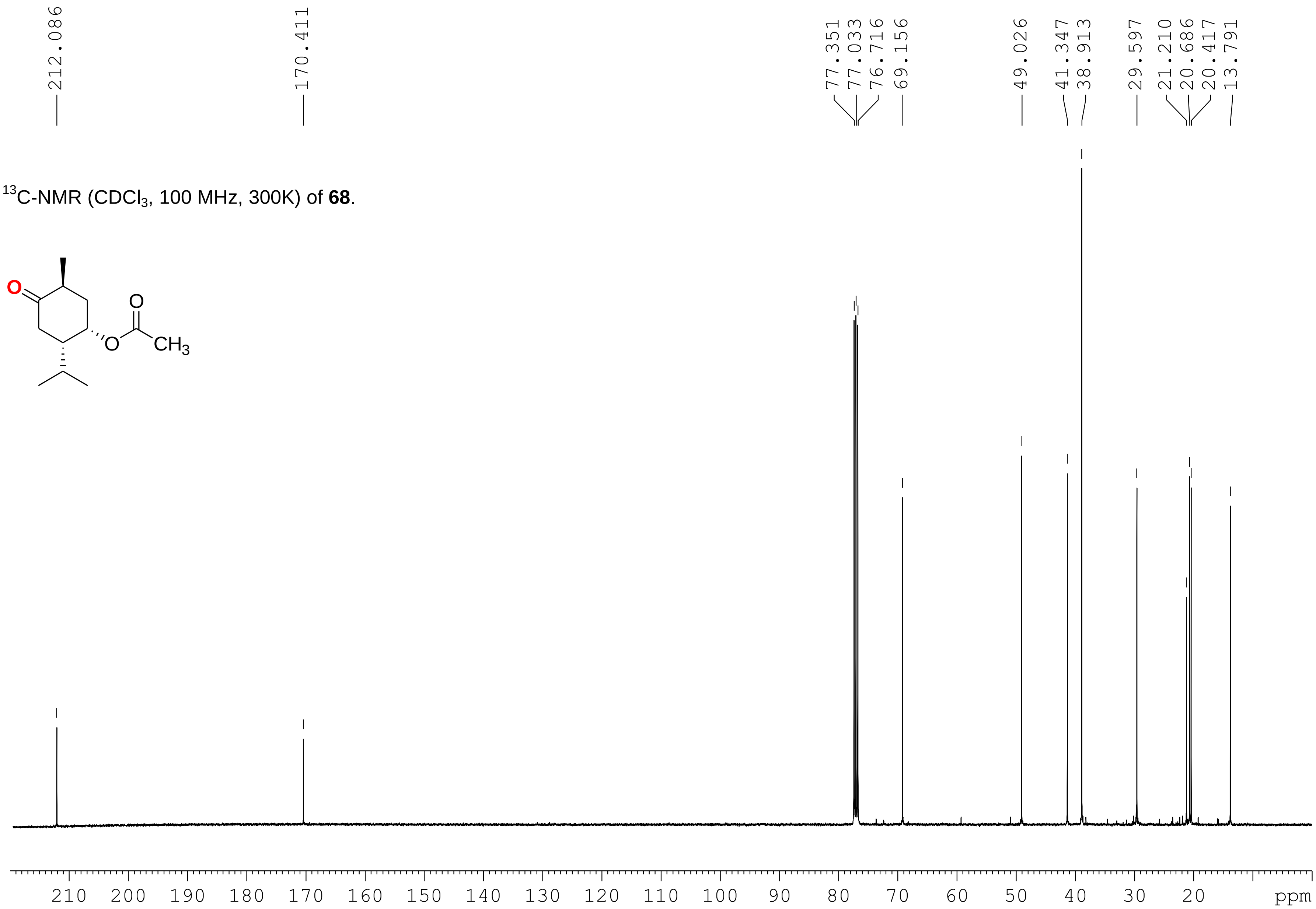
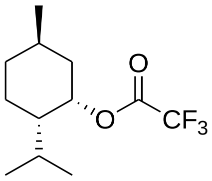


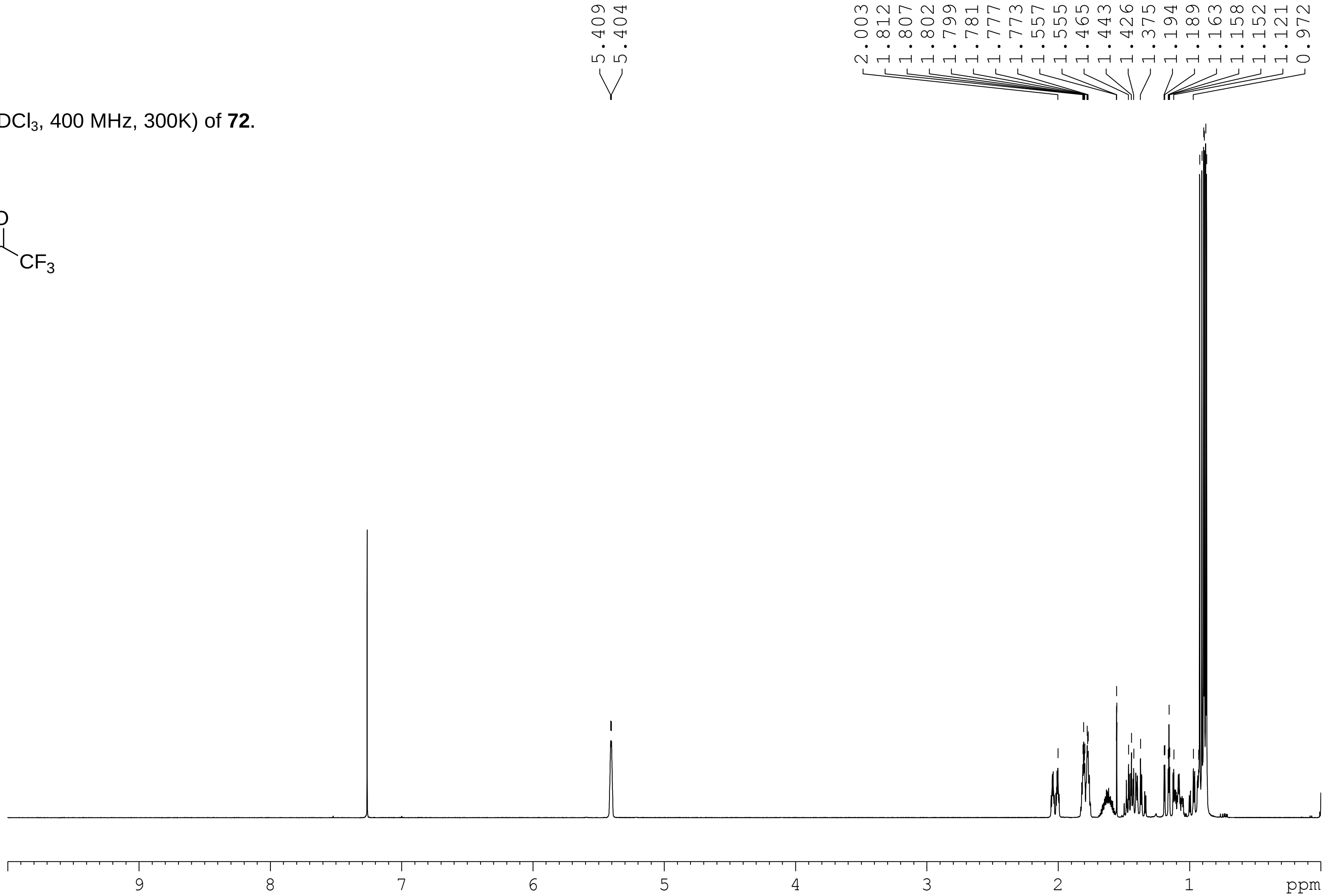
^{1}H-NMR ($CDCl_3$, 400 MHz, 300K) of **68**.

^{13}C-NMR ($CDCl_3$, 100 MHz, 300K) of **68**.



[1]H-NMR ($CDCl_3$, 400 MHz, 300K) of **72**.





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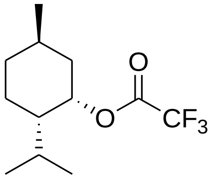
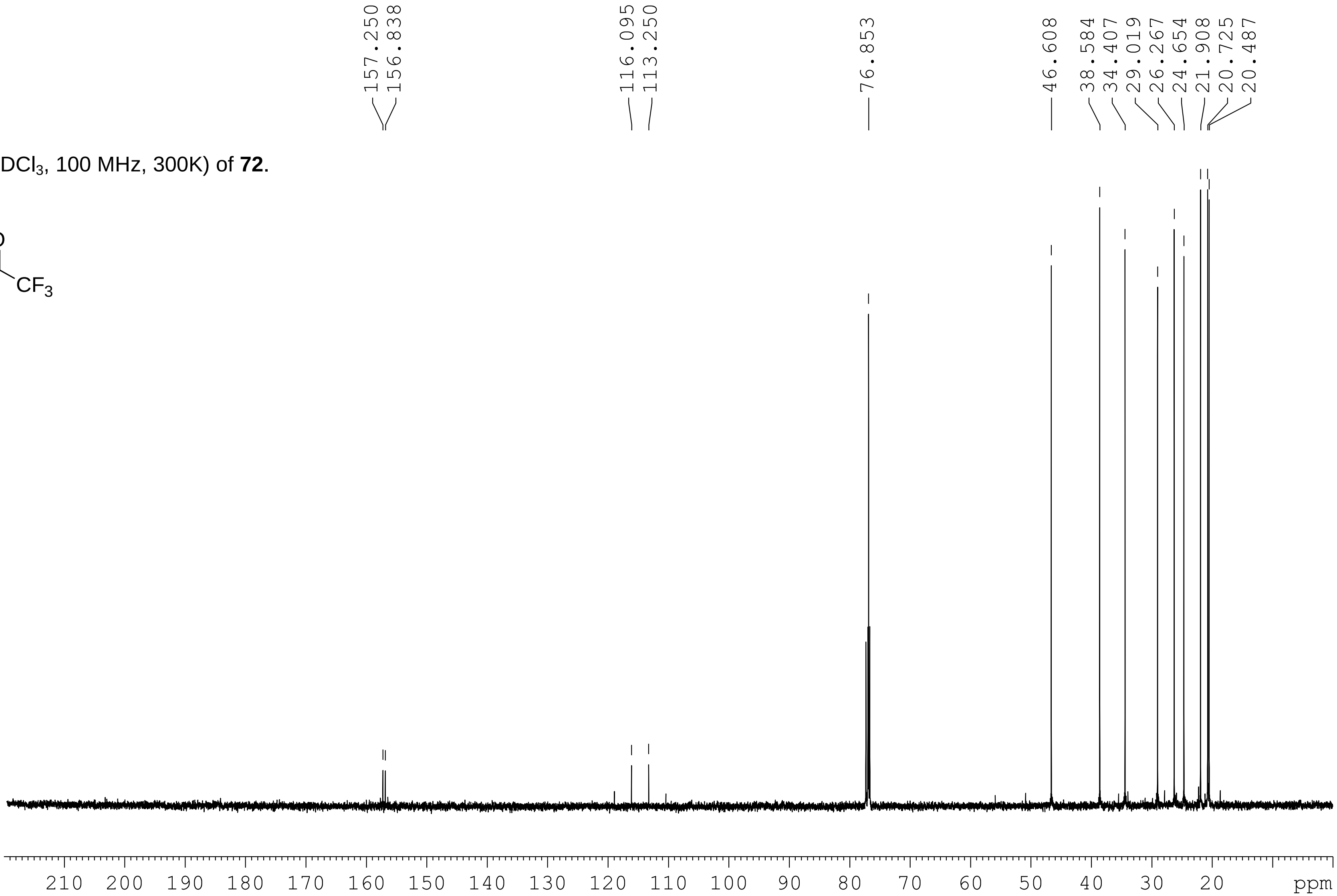
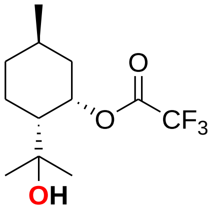
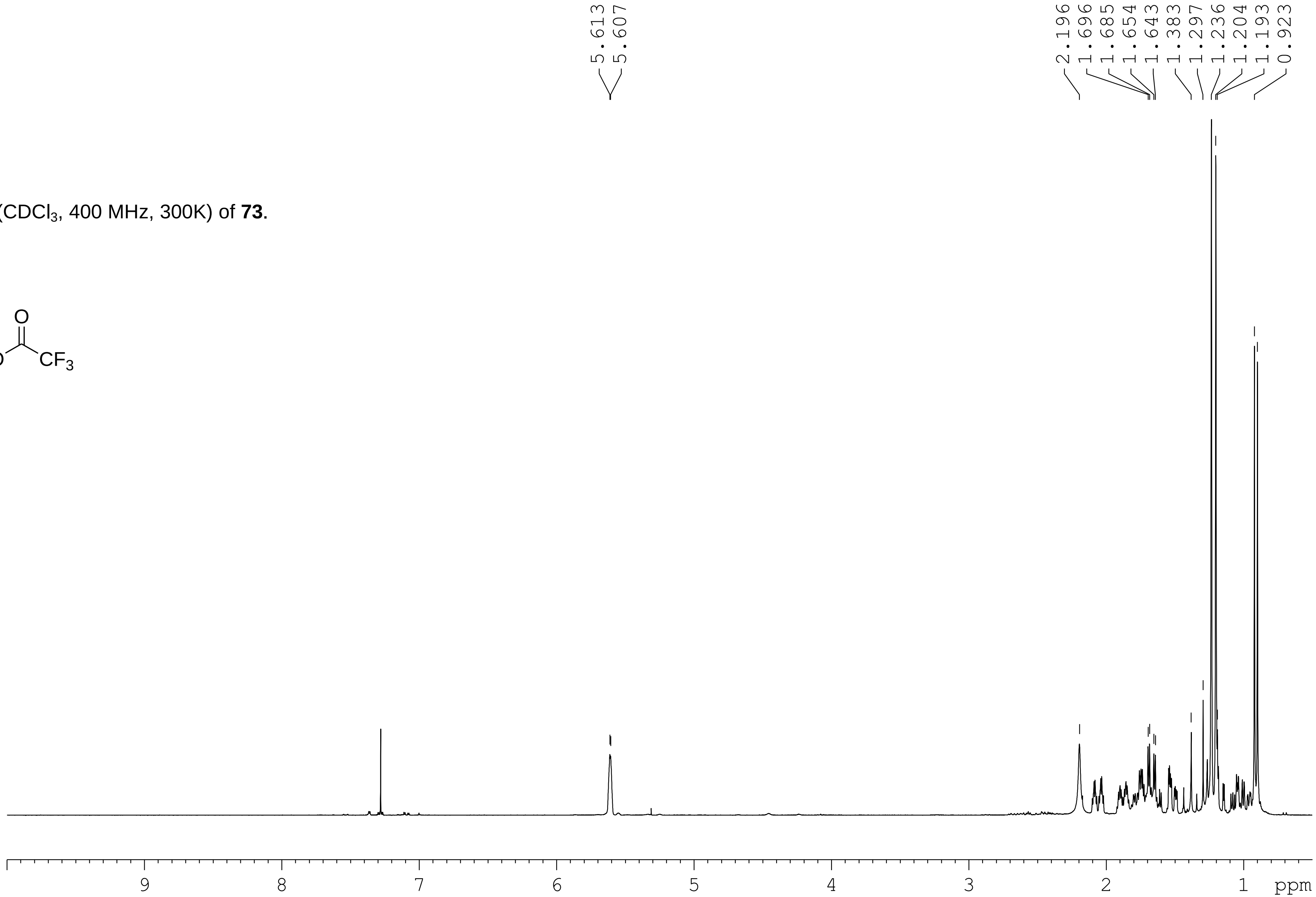


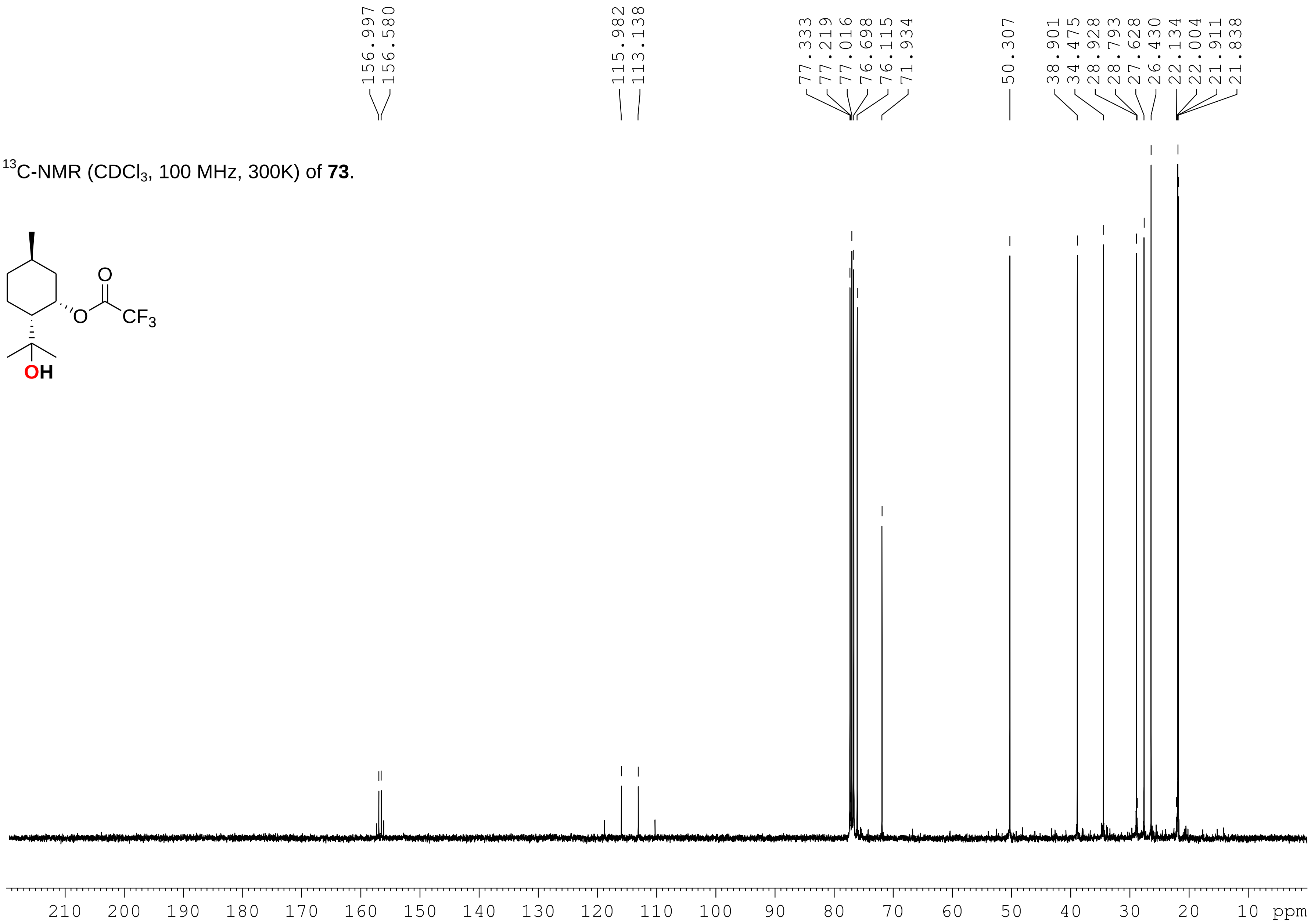


^{1}H-NMR ($CDCl_3$, 400 MHz, 300K) of **73**.

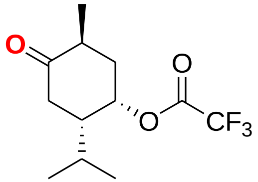


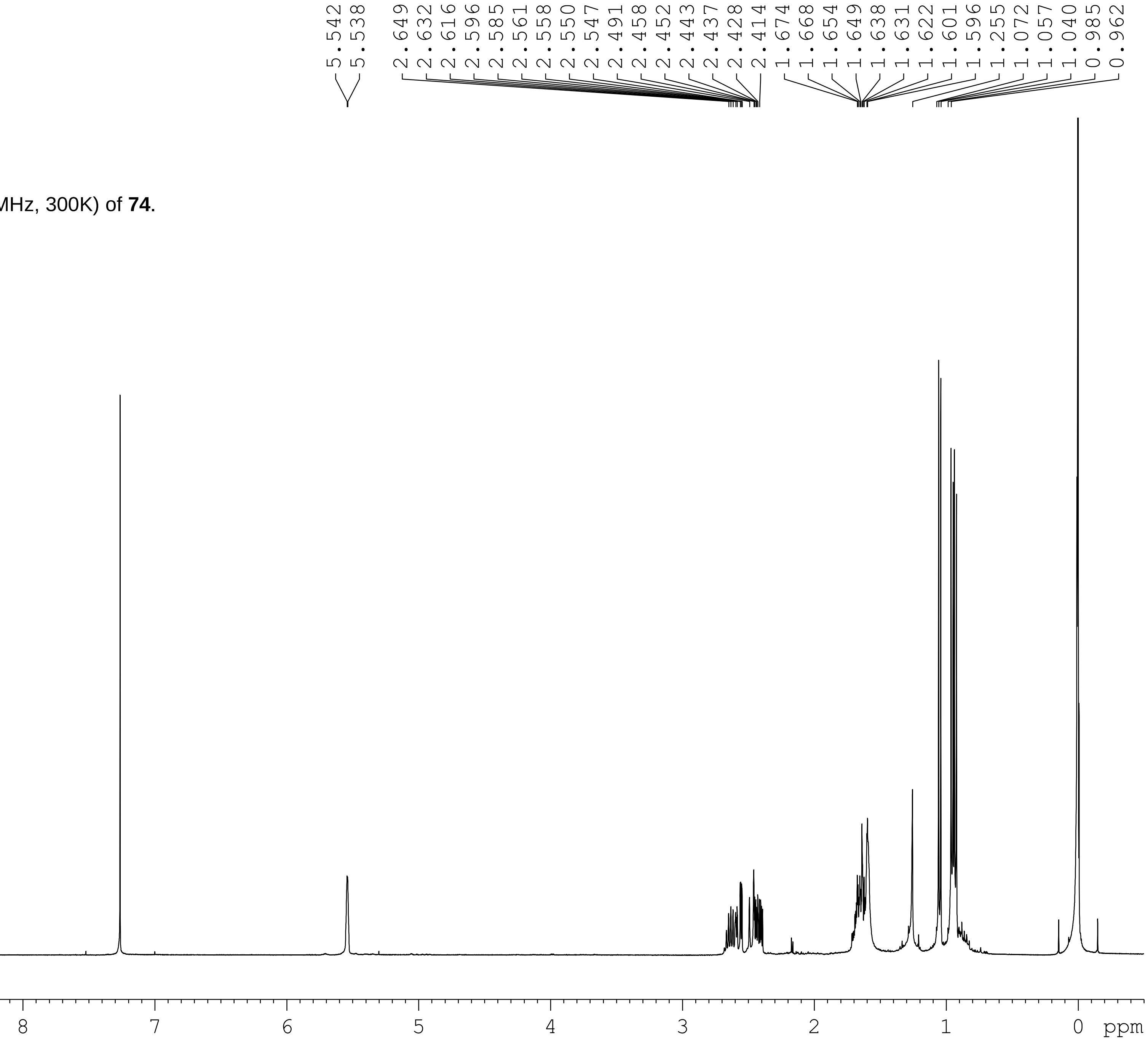


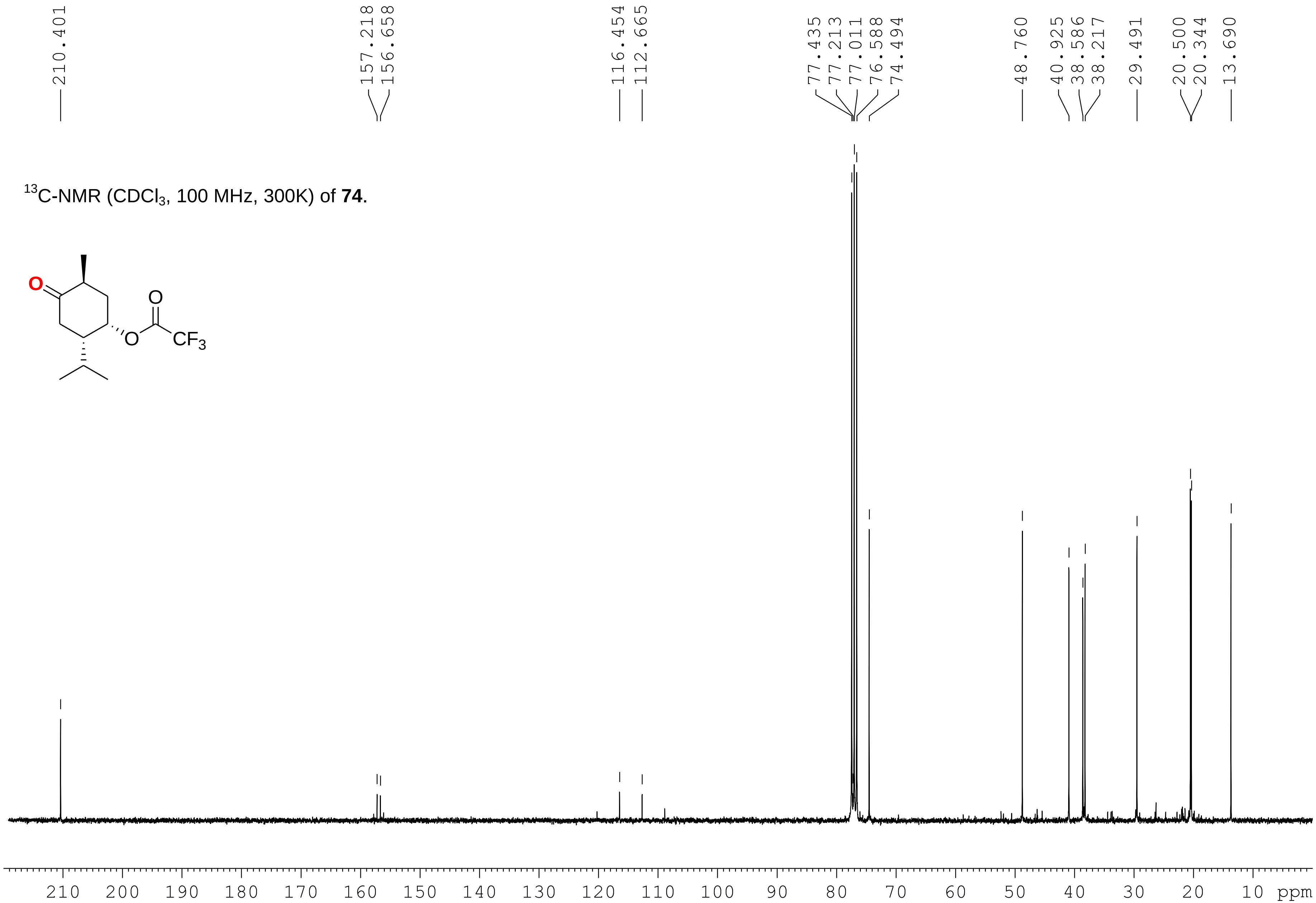
^{13}C-NMR ($CDCl_3$, 100 MHz, 300K) of **73**.



^{1}H-NMR ($CDCl_3$, 400 MHz, 300K) of **74**.

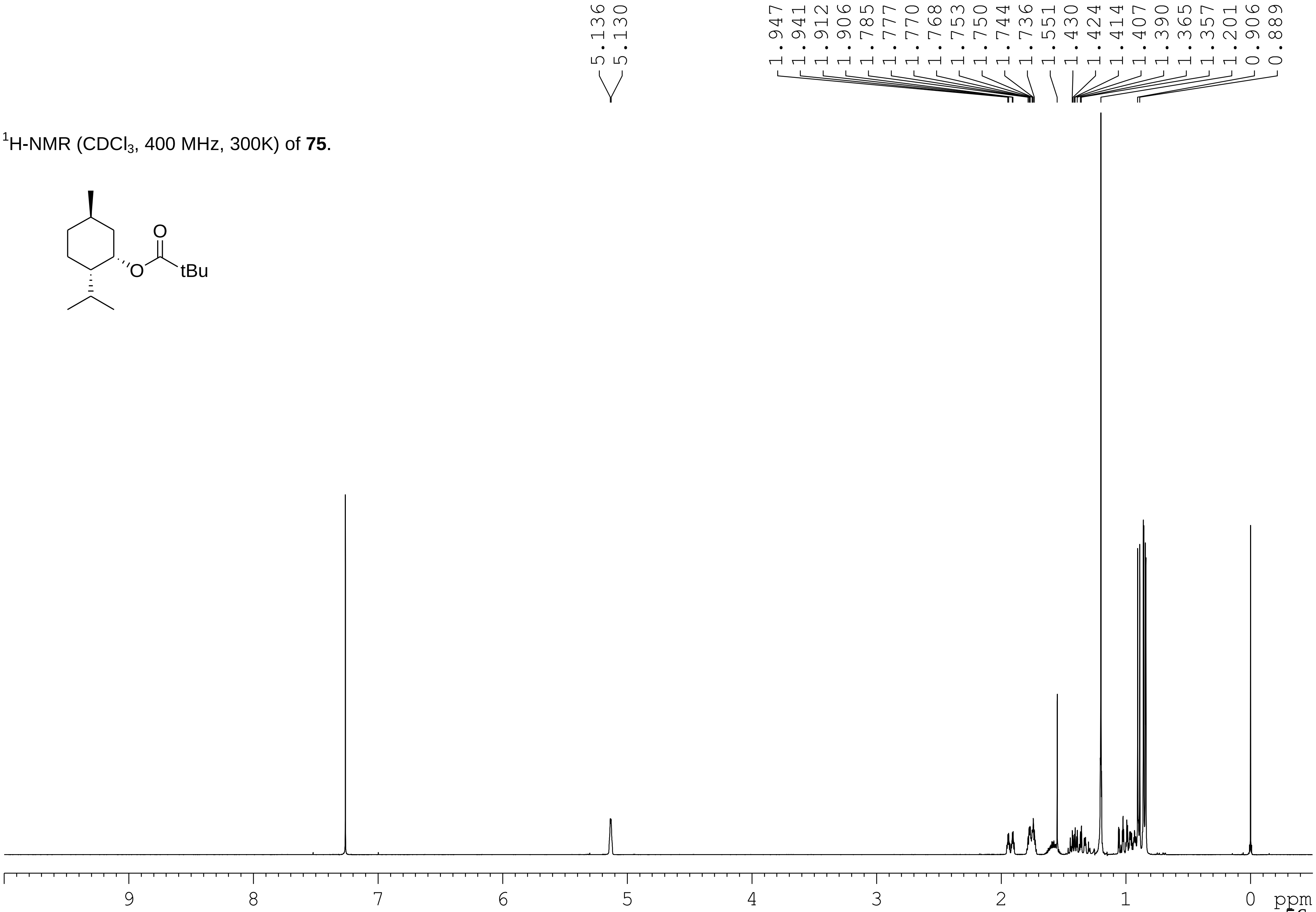




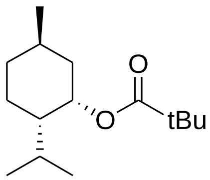


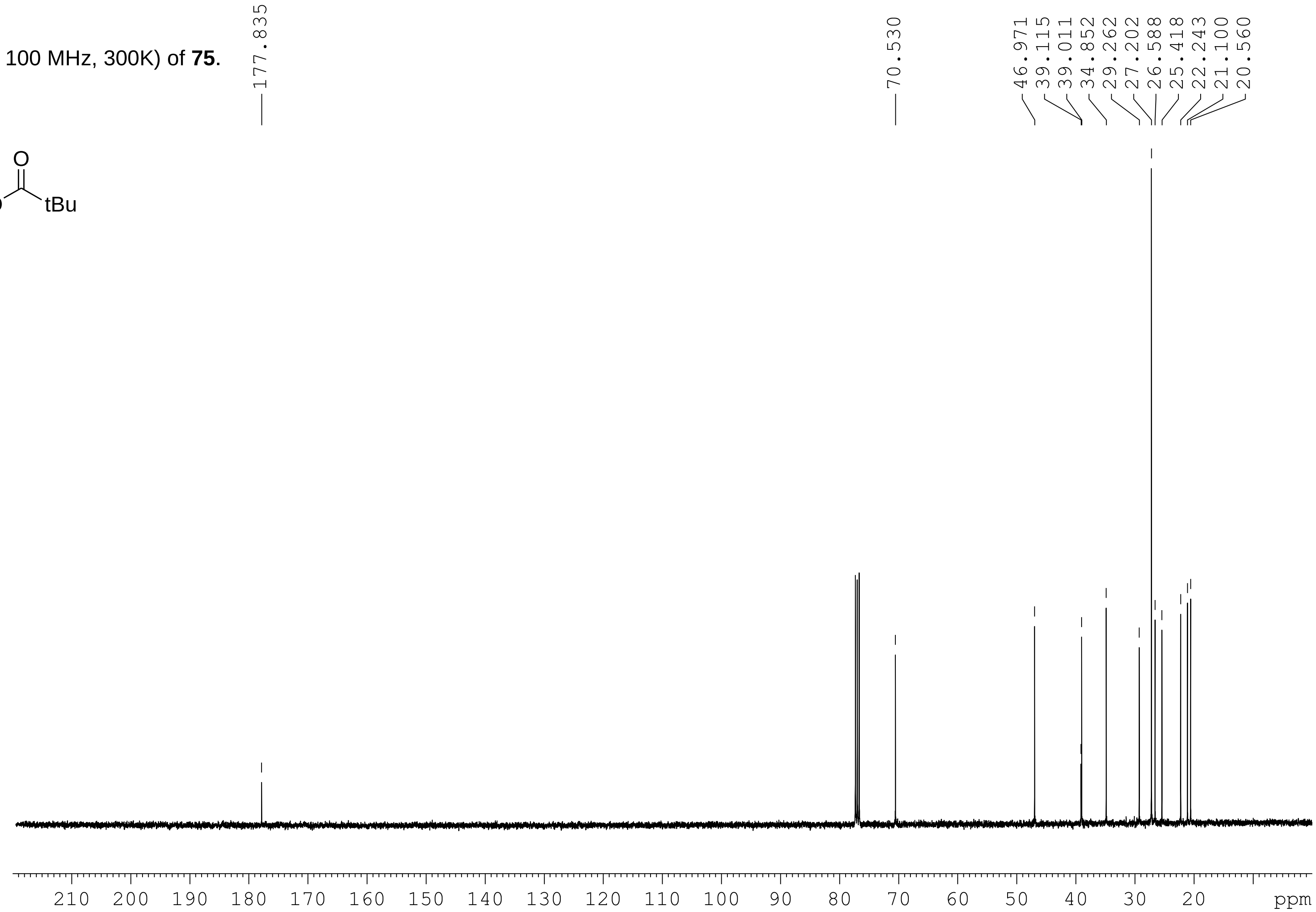
^{13}C-NMR ($CDCl_3$, 100 MHz, 300K) of **74**.

^{1}H-NMR ($CDCl_3$, 400 MHz, 300K) of **75**.

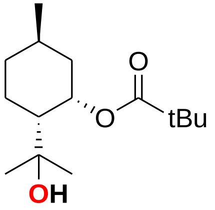


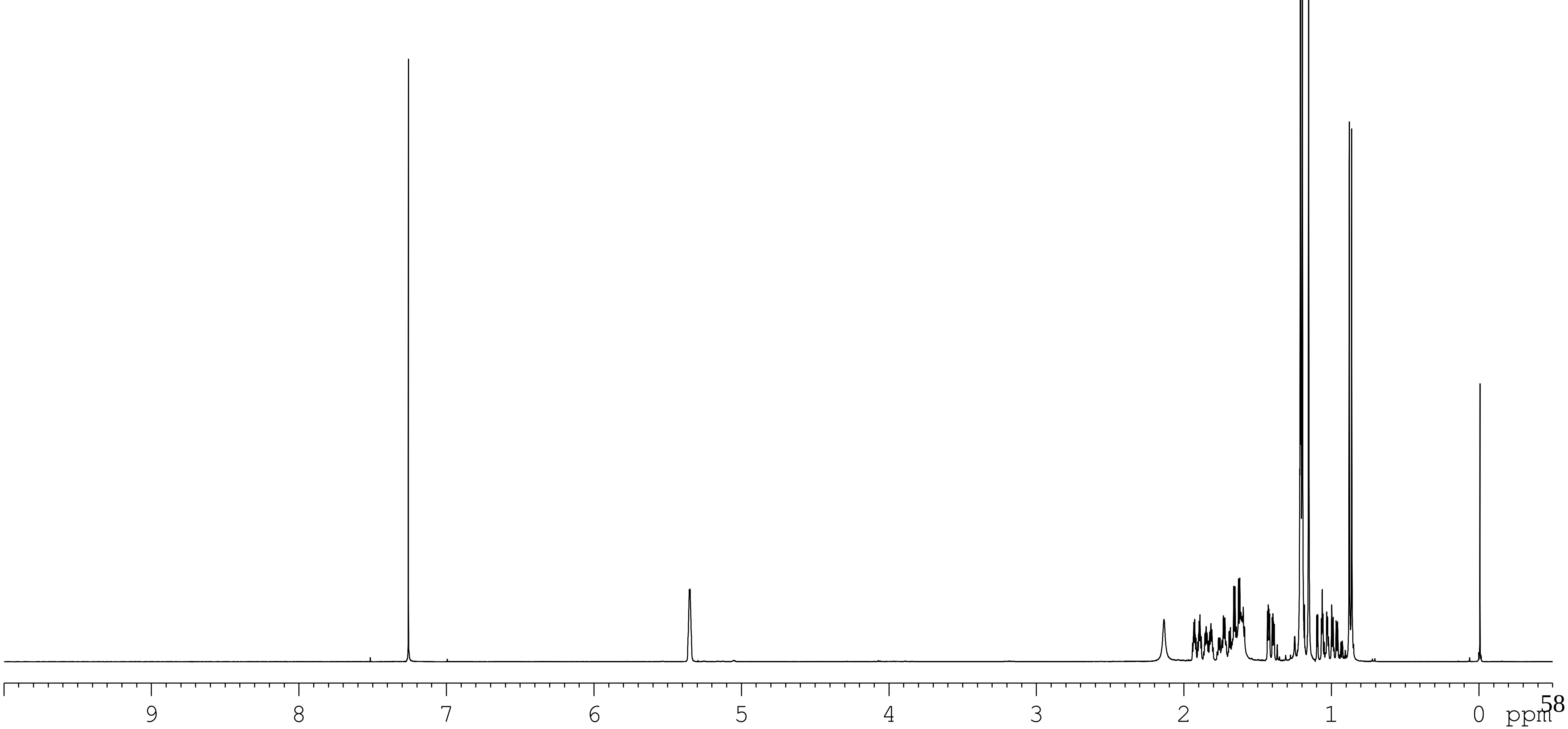
^{13}C-NMR ($CDCl_3$, 100 MHz, 300K) of **75**.



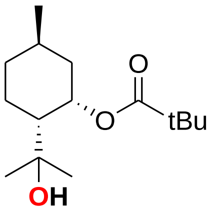


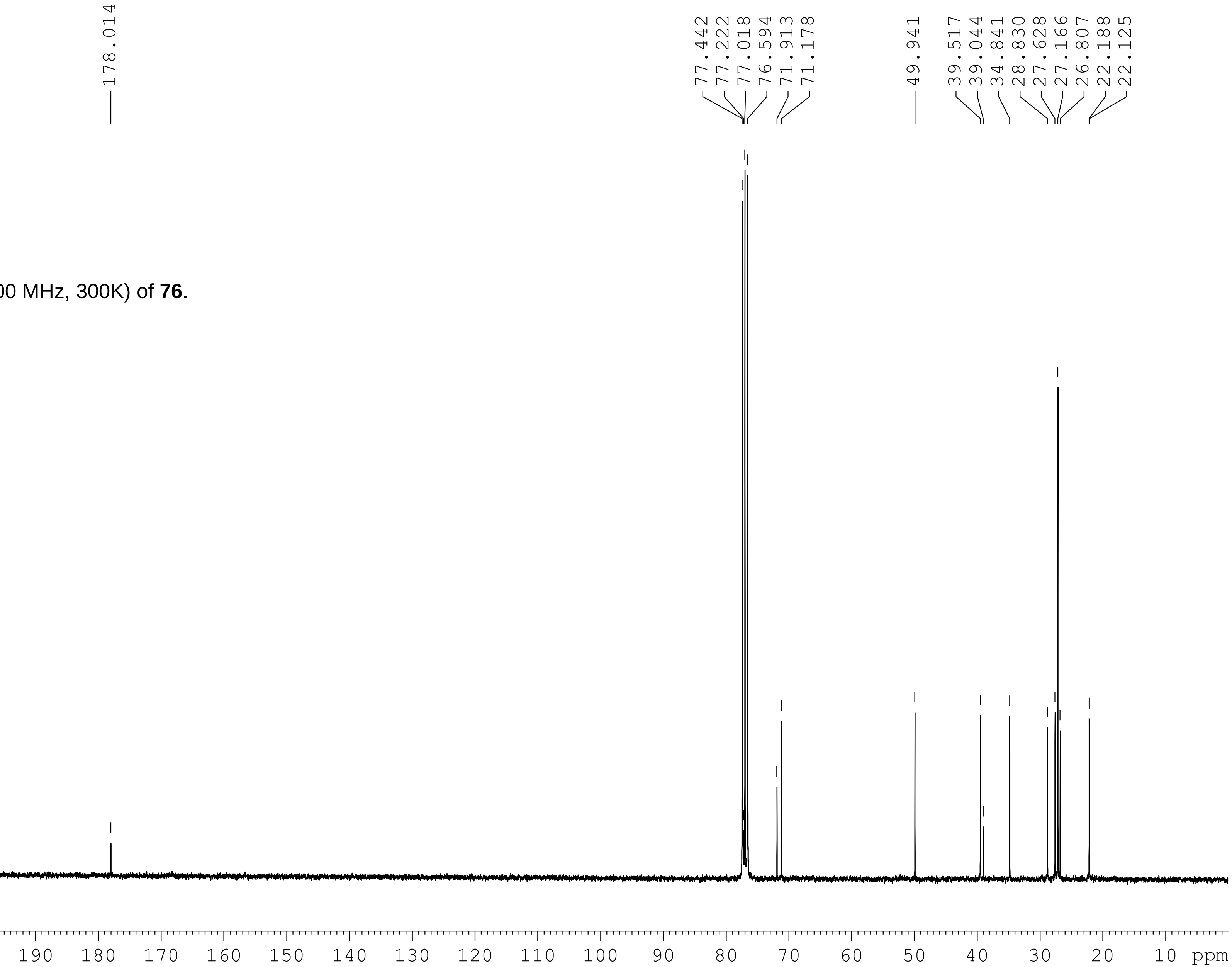
[1]H-NMR ($CDCl_3$, 400 MHz, 300K) of **76**.



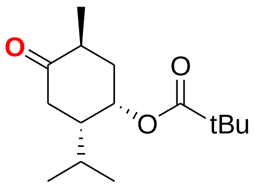


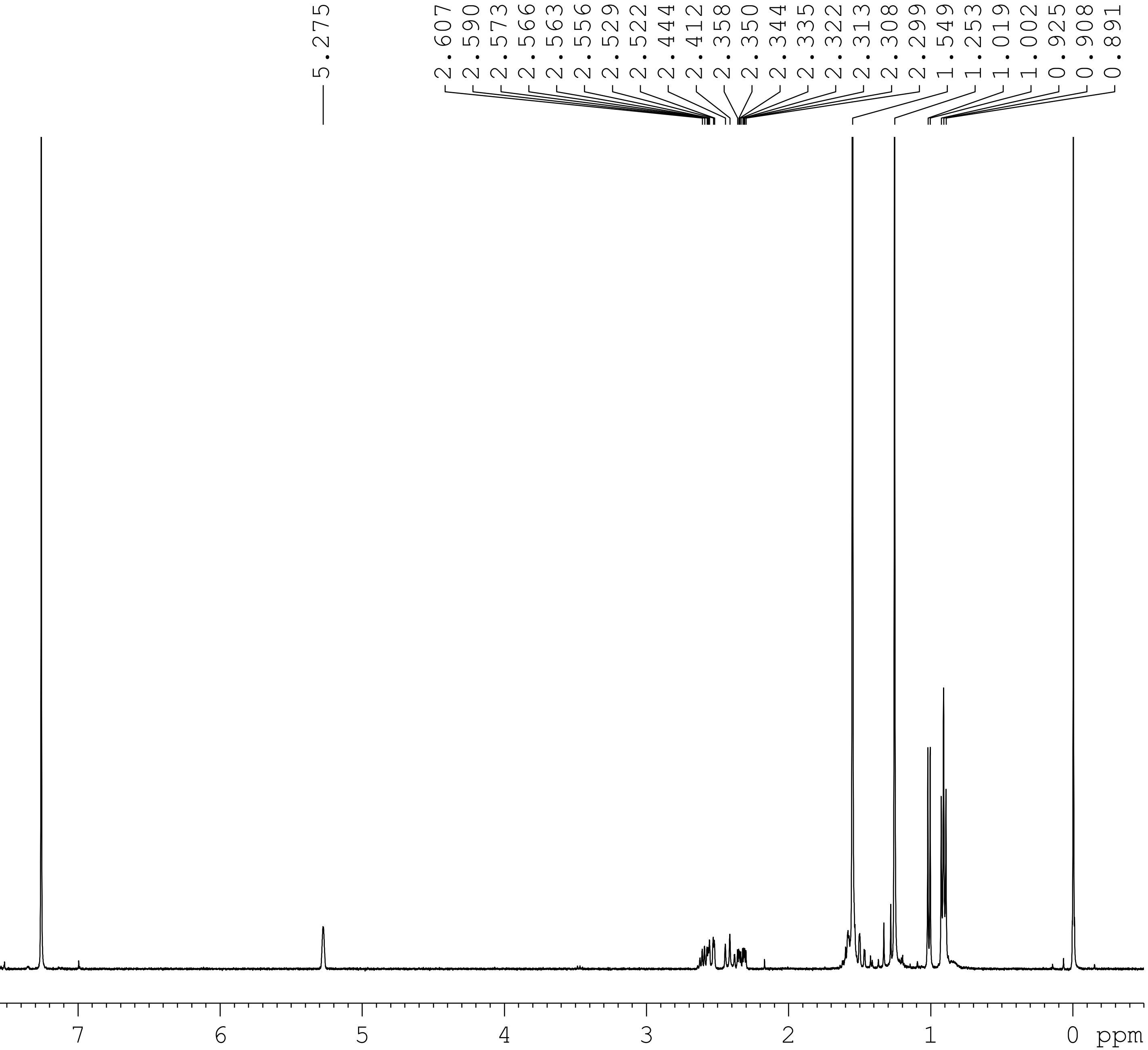
^{13}C-NMR ($CDCl_3$, 100 MHz, 300K) of **76**.



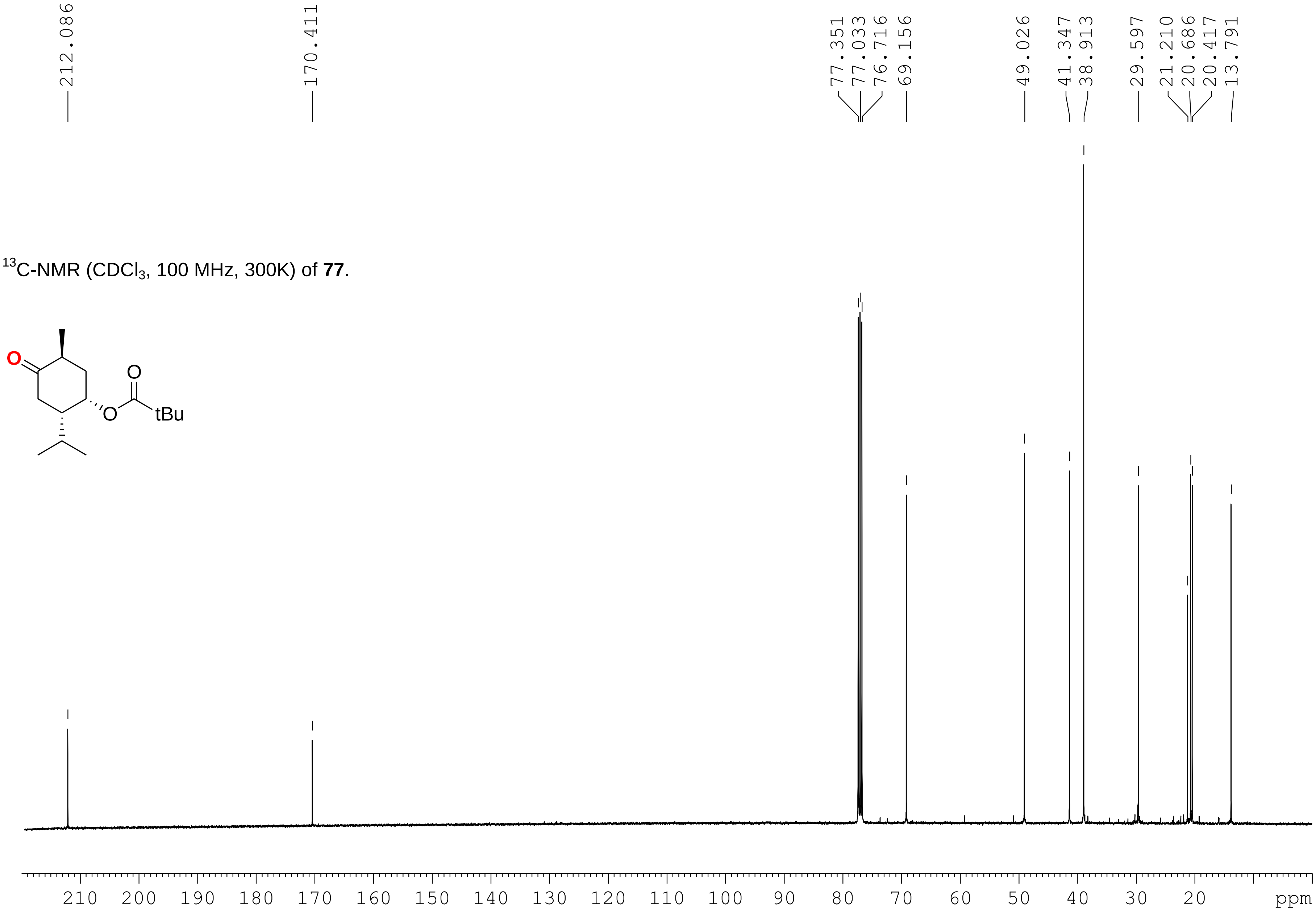


^{1}H-NMR ($CDCl_3$, 400 MHz, 300K) of **77**.

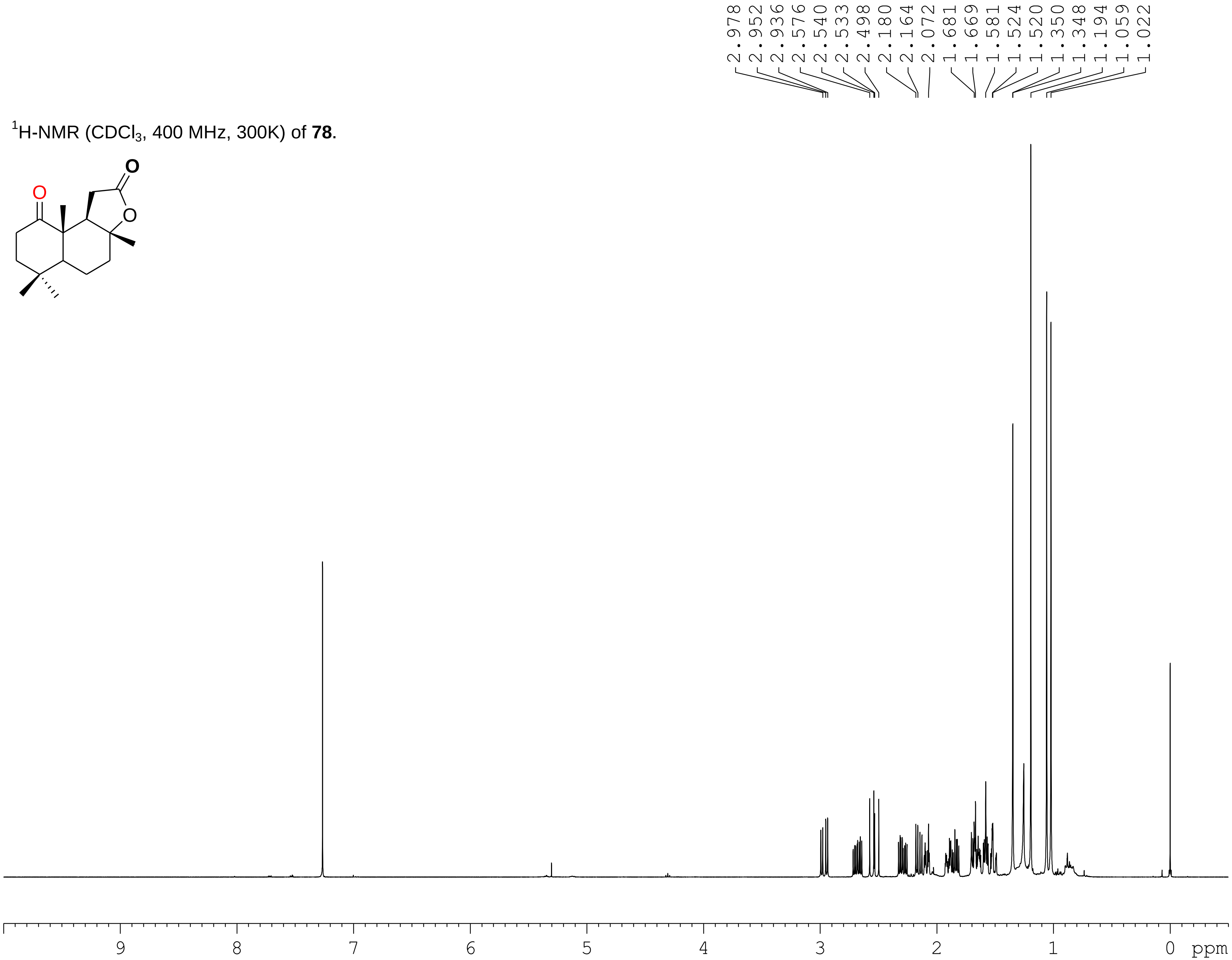




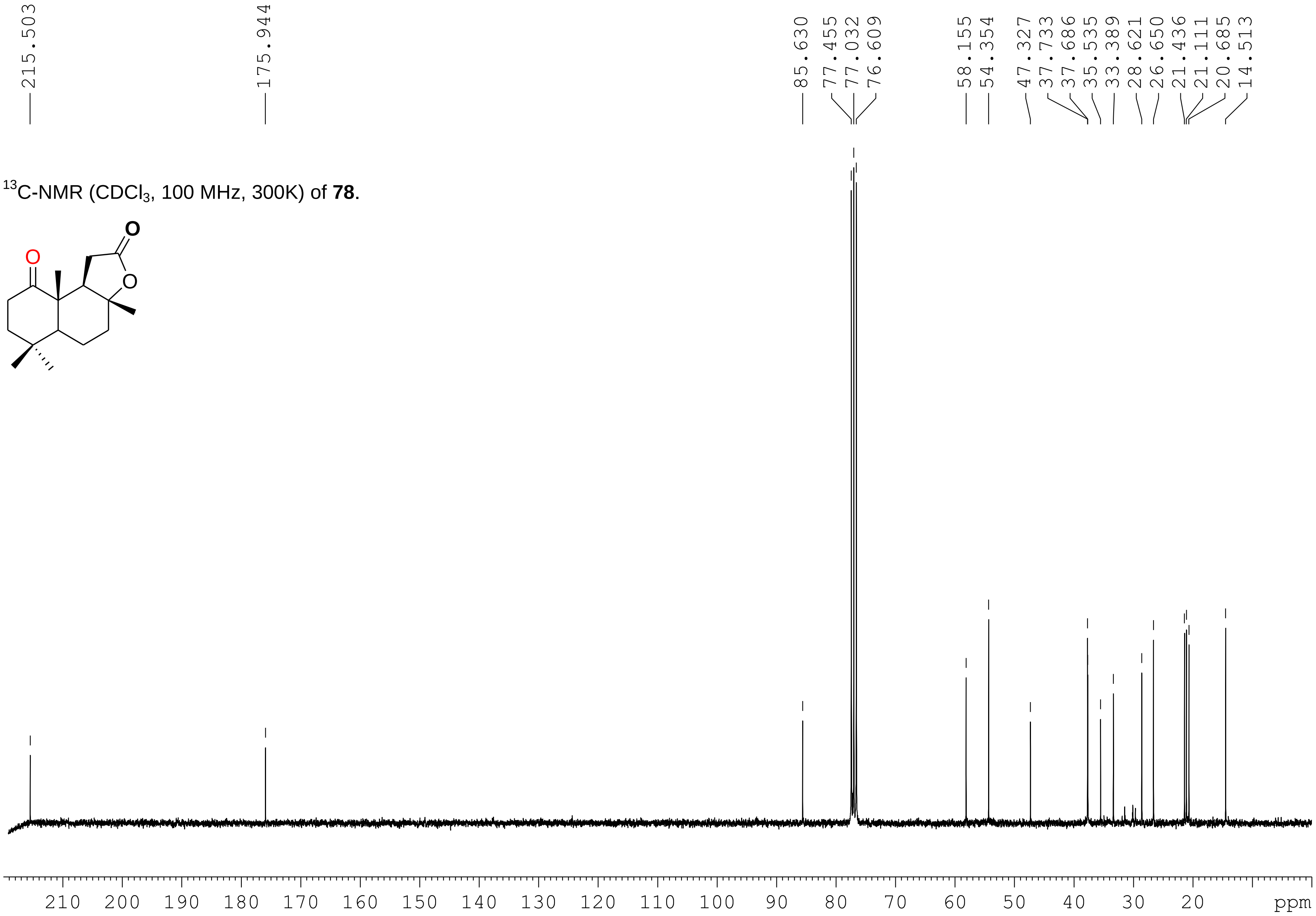
^{13}C-NMR ($CDCl_3$, 100 MHz, 300K) of **77**.



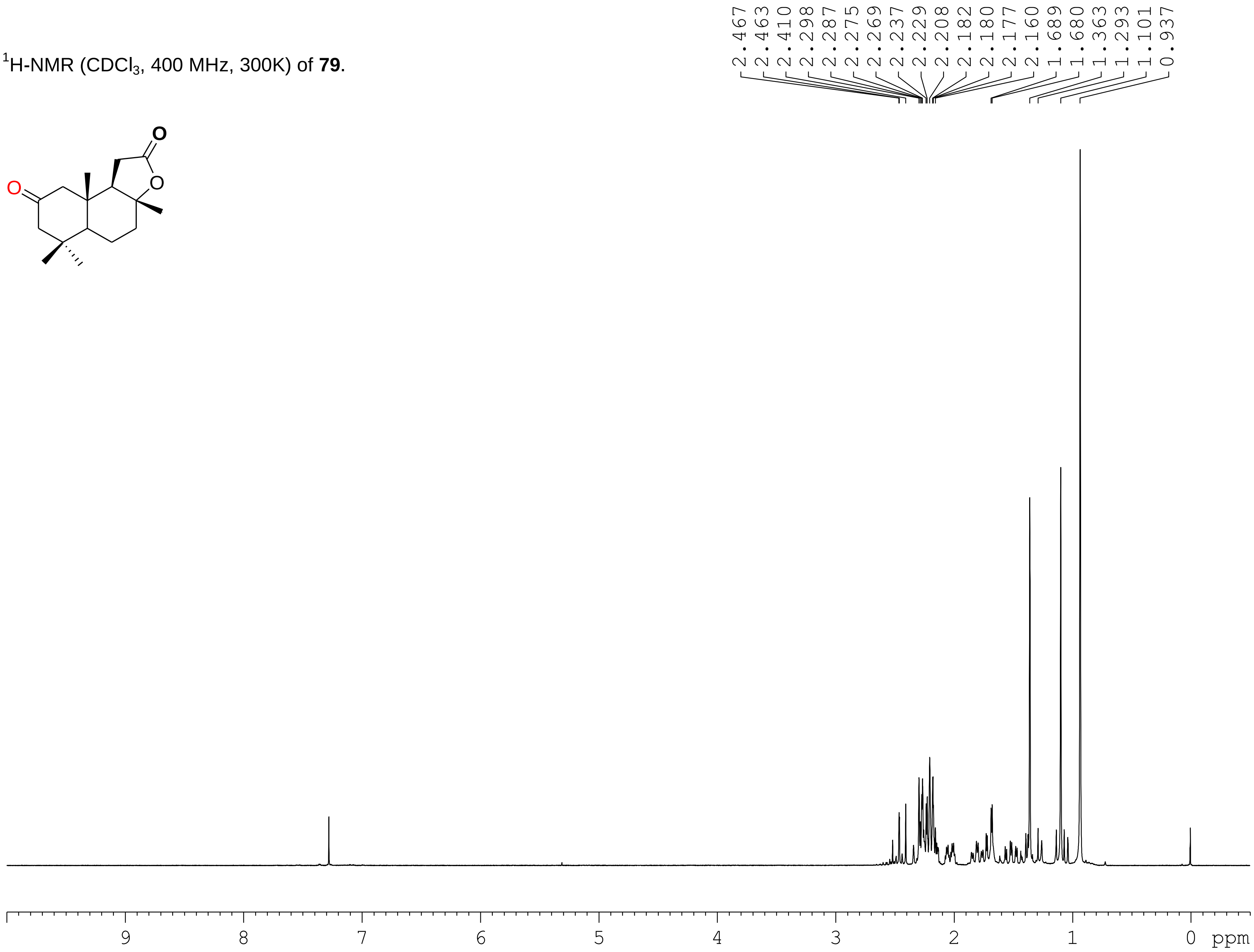
^{1}H-NMR ($CDCl_3$, 400 MHz, 300K) of **78**.

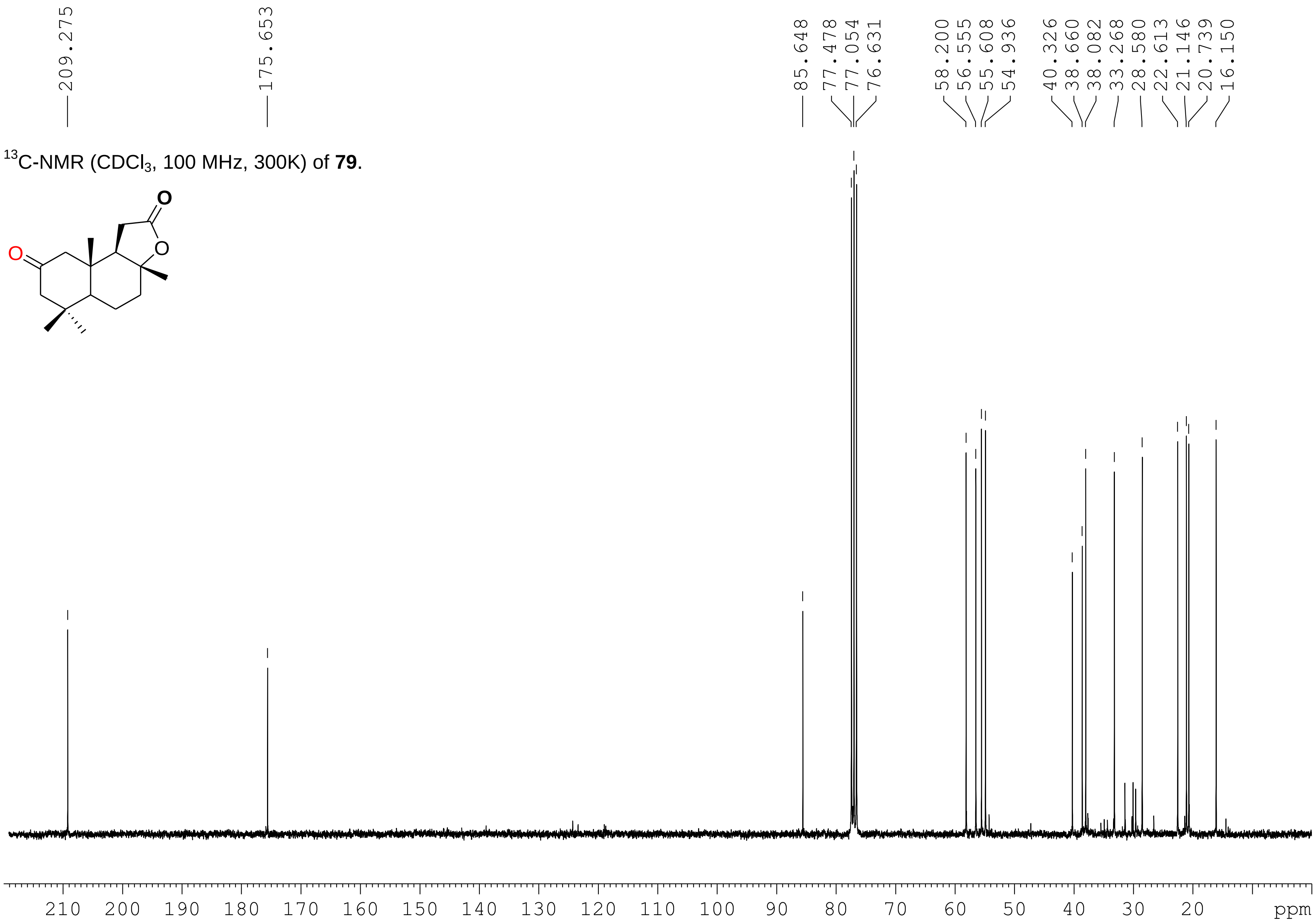


^{13}C-NMR ($CDCl_3$, 100 MHz, 300K) of **78**.



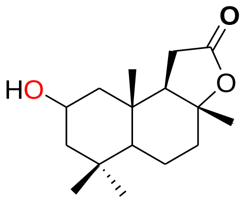
[1]H-NMR ($CDCl_3$, 400 MHz, 300K) of **79**.

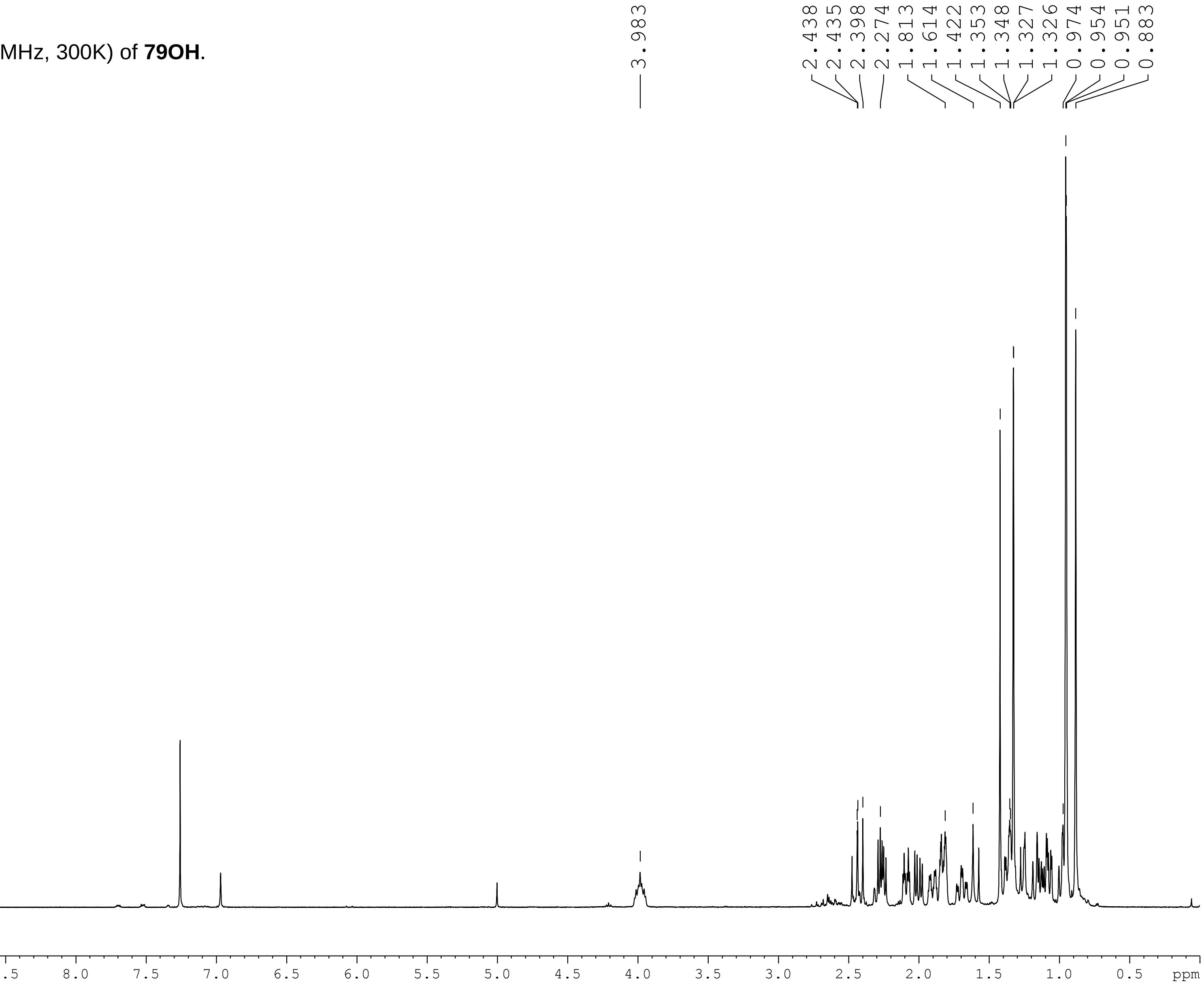




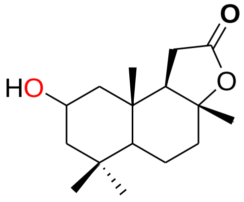
[13]C-NMR ($CDCl_3$, 100 MHz, 300K) of **79**.

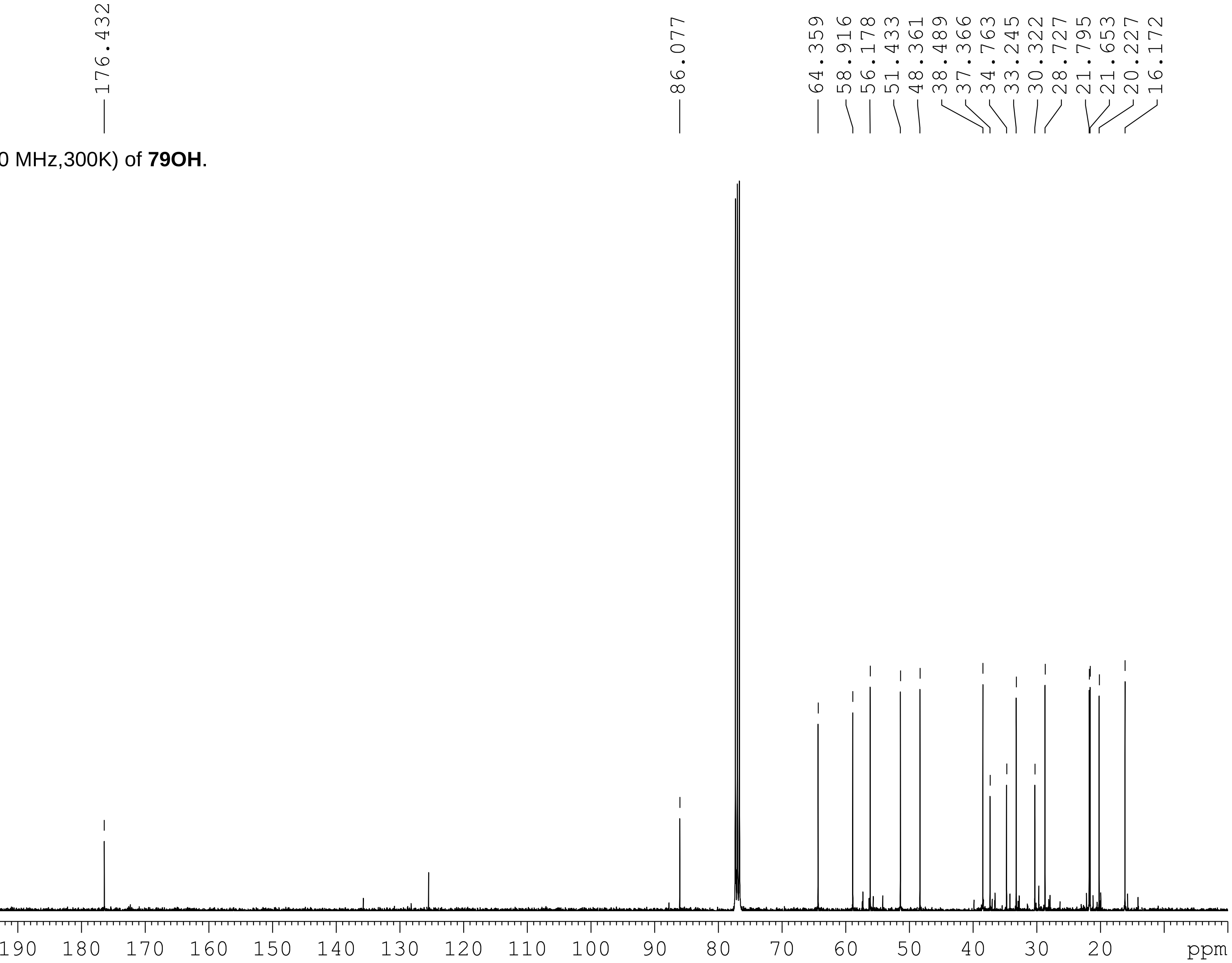
[1]H-NMR ($CDCl_3$, 400 MHz, 300K) of **79OH**.



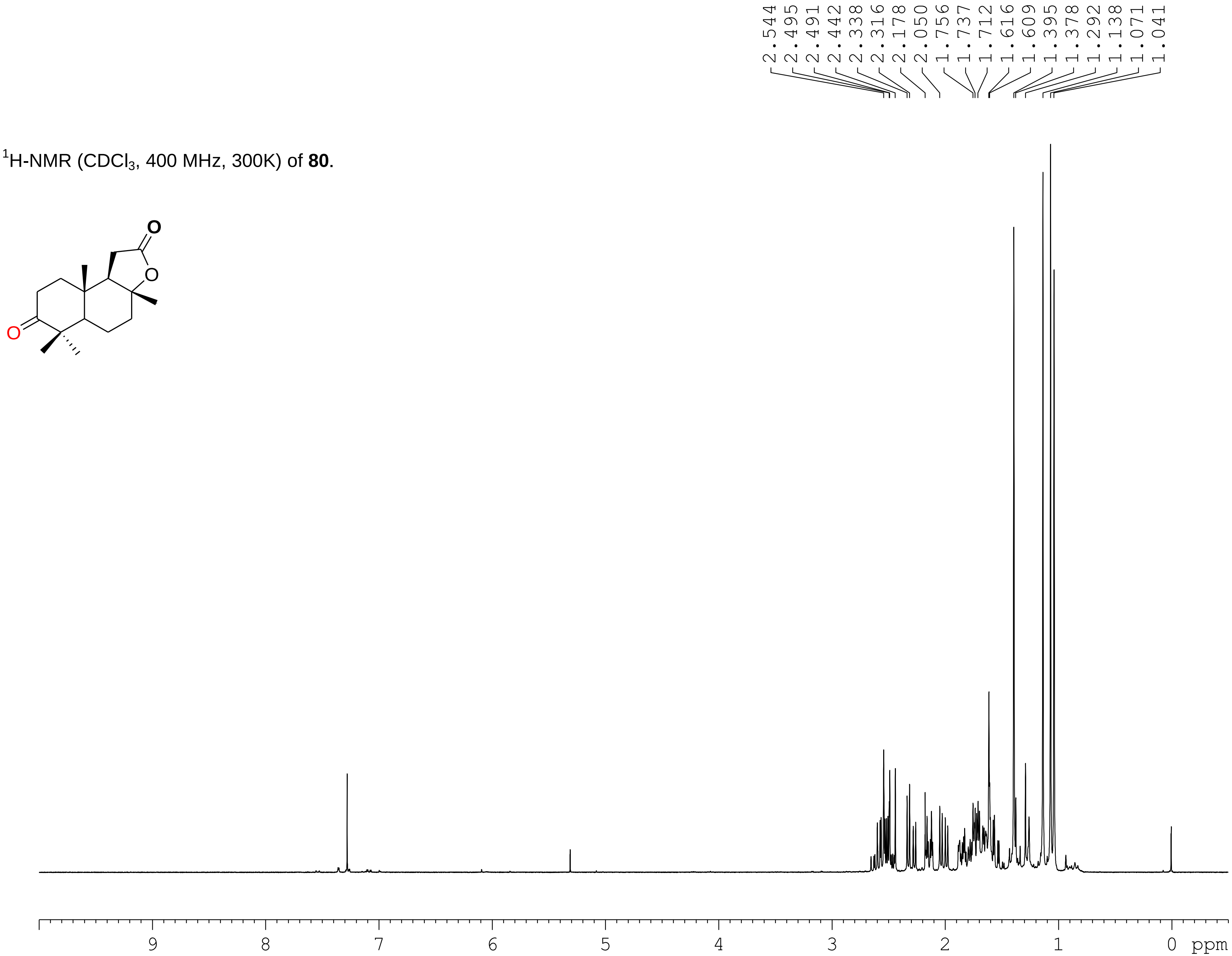


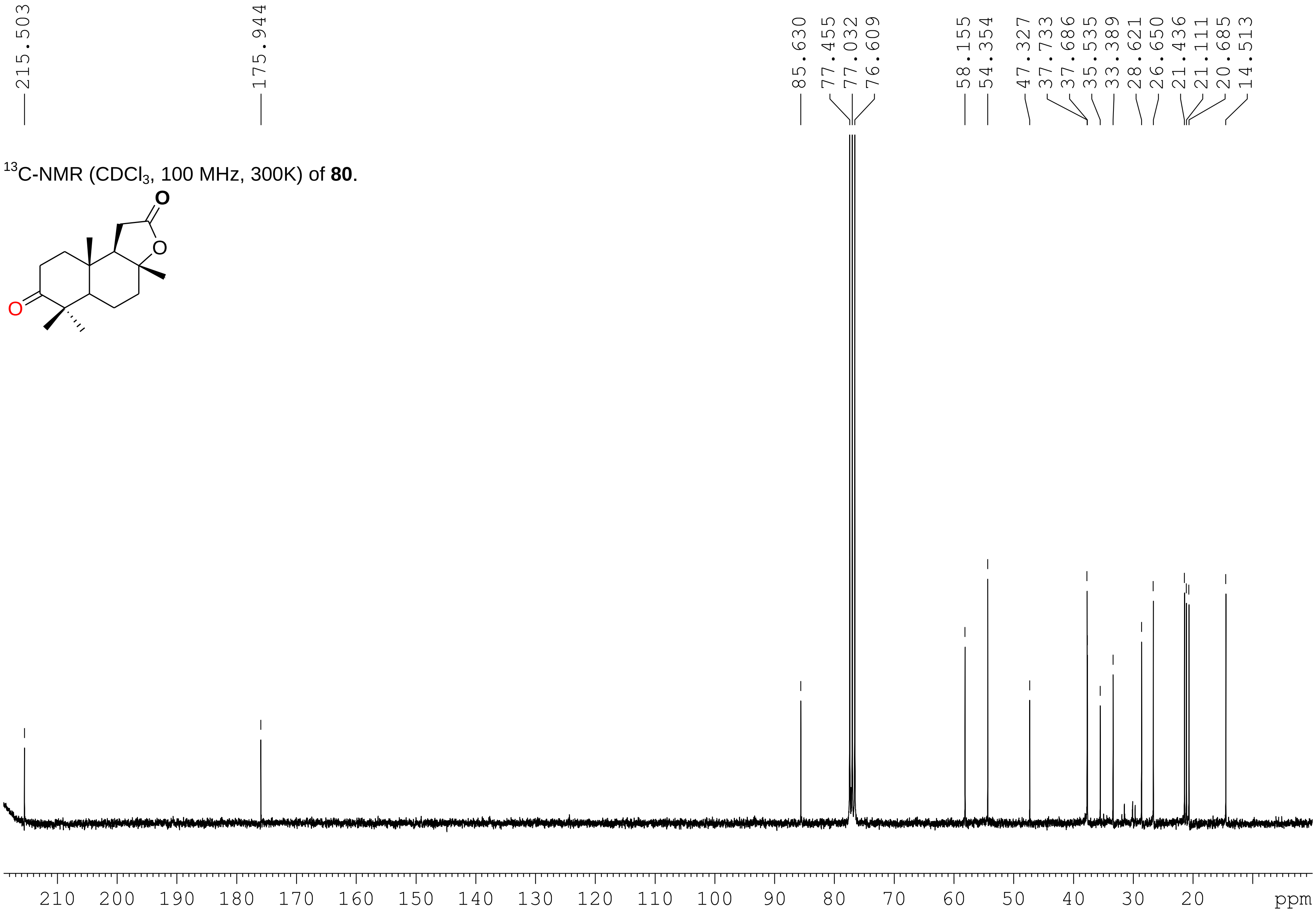
^{13}C-NMR ($CDCl_3$, 100 MHz,300K) of **79OH**.





^{1}H-NMR ($CDCl_3$, 400 MHz, 300K) of **80**.





^{13}C-NMR ($CDCl_3$, 100 MHz, 300K) of **80**.