Electronic Supplementary Material (ESI) for ChemComm.

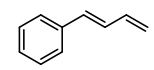
Base-tuned selective 1,2-dichloromethylhydroxylation and 1,2-peroxyhydroxylation of 1,3-dienes *via* tandem radical process

Jiantao Zhang, Weiming Zhu, Peng Zhou*, Cui Chen and Weibing Liu*

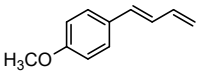
College of Chemistry, Guangdong University of Petrochemical Technology, Guandu Road, Maoming, 525000, P. R. China

General Information

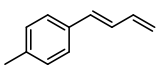
The substrates of 1,3-dienes **1** were synthesized according to the literature.[1-2] ^{1}H, ^{13}C NMR spectra were recorded on a Bruker AVANCE 400 (400 MHz for ^{1}H; 100 MHz for ^{13}C), ^{1}H NMR and ^{13}C NMR chemical shifts were determined relative to internal standard TMS at δ 0.0. Chemical shifts (δ) are reported in ppm, and coupling constants (J) are in Hertz (Hz). The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Mass spectra were obtained using *ESI* mass spectrometer. All reagents were used as received from commercial sources, unless specified otherwise, or prepared as described in the literature.



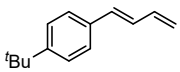
1a[1]



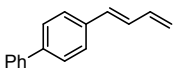
1b[1]



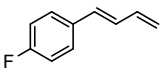
1c[2]



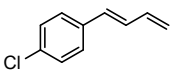
1d[2]



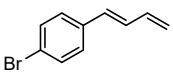
1e[2]



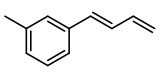
1f[1]



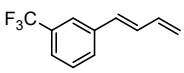
1g[1]



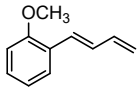
1h[1]



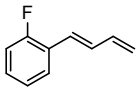
1i[2]



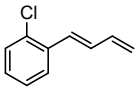
1j[1]



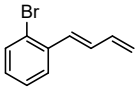
1k[1]



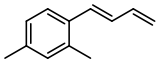
1l[2]



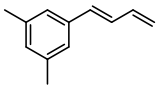
1m[1]



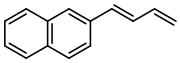
1n[1]



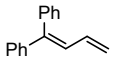
1o[2]



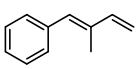
1p[2]



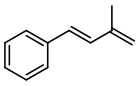
1q[2]



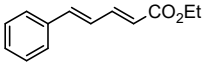
1r[1]



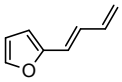
1s[1]



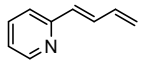
1t[1]



1u[2]

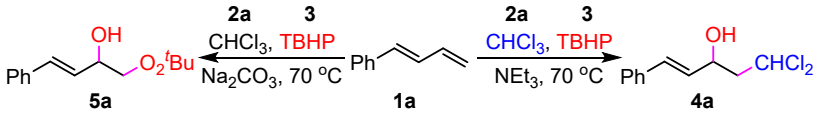


1v[1]



1w[2]

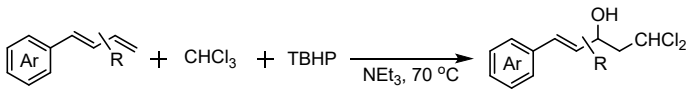
Table S1 Optimization of the reaction conditions[a]



Entry	Base	Solvent	**3**/equiv.	**4a**/%	**5a**/%
1	NEt_3	$CHCl_3$	2.0	23	-
2	NEt_3	$CHCl_3$	3.0	49	-
3	**NEt_3**	$CHCl_3$	**4.0**	**72**[b]	**-**
4	NEt_3	$CHCl_3$	5.0	70	-
5	NEt_3	$CHCl_3$	8.0	65	-
6[c]	NEt_3	$CHCl_3$	4.0	<5	-
7[d]	DABCO	$CHCl_3$	4.0	-	35
8[d]	DMAP	$CHCl_3$	4.0	-	10
9[d]	DBU	$CHCl_3$	4.0	-	<5
10[d]	^{t}BuOK	$CHCl_3$	4.0	-	52
11[d]	pyridine	$CHCl_3$	4.0	-	40
12[d]	**Na_2CO_3**	$CHCl_3$	**4.0**	**-**	**67**[b]
13[e]	Na_2CO_3	$CHCl_3$	4.0	-	64
14[f]	Na_2CO_3	$CHCl_3$	4.0	-	65
15	-	$CHCl_3$	4.0	-	-
16	Na_2CO_3	CH_3CN	4.0	-	45
17	Na_2CO_3	PhMe	4.0	-	59
18	Na_2CO_3	DMF	4.0	-	35
19	Na_2CO_3	DMSO	4.0	-	40
20	Na_2CO_3	PhCl	4.0	-	43

[a] Unless other noted, the reaction was performed in air by using **1a** (0.5 mmol), **2a** (0.5 mL), TBHP (2.0 mmol) in NEt_3 (0.5 mL) at 70 °C for 12 h. [b] Isolated yield. [c] The reaction was performed at room temperature for 24 h. [d] 2.0 equiv. of base was employed, and $CHCl_3$ was used as solvent. [e] 3.0 equiv. of Na_2CO_3 was used. [f] 4.0 equiv. of Na_2CO_3 was used. TBHP (70% solution in water). DABCO: 1,4-Diazabicyclo[2.2.2]octane; DMAP: 4-dimethylaminopyridine; DBU: 1,8-diazabicyclo[5.4.0]undec-7-ene.

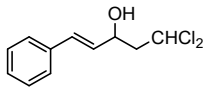
General Procedure for Products 4.



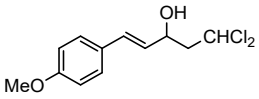
1 2a 3 4

To a solution of **1** (0.5 mmol) in NEt_3 (0.5 mL) was added $CHCl_3$ (0.5 mL) and TBHP (2.0 mmol) at room temperature. Then the solution mixture was stirred at 70℃ for 12 h, after completion of the reaction, the solvent was removed under reduced pressure and the crude product was subjected to silica gel column chromatography to afford **4**.

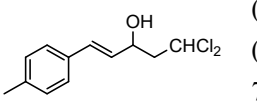
Characterization Data of Products.



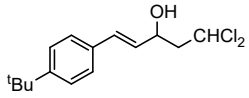
(*E*)-5,5-dichloro-1-phenylpent-1-en-3-ol (**4a**): Yield: 72%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 (dd, J = 8.3, 1.3 Hz, 2H), 7.36 – 7.31 (m, 2H), 7.28 (dt, J = 5.3, 2.1 Hz, 1H), 6.66 (d, J = 15.9 Hz, 1H), 6.20 (dd, J = 15.9, 6.9 Hz, 1H), 5.96 (dd, J = 8.9, 4.4 Hz, 1H), 4.57 (s, 1H), 2.60 – 2.49 (m, 1H), 2.43 (m, 1H), 1.86 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.0, 131.8, 130.0, 128.7, 128.2, 126.6, 70.5, 70.0, 50.5. HRMS (*ESI*) m/z: $[M+Cl]^-$ Calcd for $C_{11}H_{12}Cl_3O^-$ 264.9959; Found 265.0267.



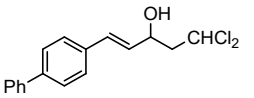
(*E*)-5,5-dichloro-1-(4-methoxyphenyl)pent-1-en-3-ol (**4b**): Yield: 78%, yellow oil liquid; R_f = 0.35 (EtOAc/petroleum ether = 1/15). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.30 (m, 2H), 6.99 – 6.79 (m, 2H), 6.61 (d, J = 15.8 Hz, 1H), 6.08 (dd, J = 15.9, 7.1 Hz, 1H), 5.97 (dd, J = 8.8, 4.5 Hz, 1H), 4.64 – 4.48 (m, 1H), 3.84 (s, 3H), 2.55 (ddd, J = 13.6, 6.7, 2.9 Hz, 1H), 2.48 – 2.34 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.6, 131.5, 128.7, 127.8, 127.7, 114.1, 70.6, 70.2, 55.3, 50.6. HRMS (*ESI*) m/z: $[M+Cl]^-$ Calcd for $C_{12}H_{14}Cl_3O_2^-$ 295.0163; Found 295.1581.



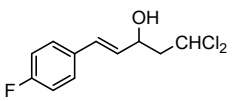
(*E*)-5,5-dichloro-1-(*p*-tolyl)pent-1-en-3-ol (**4c**): Yield: 74%, yellow oil liquid; R_f = 0.38 (EtOAc/petroleum ether = 1/20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 (d, J = 8.1 Hz, 2H), 7.17 (d, J = 8.0 Hz, 2H), 6.64 (d, J = 15.9 Hz, 1H), 6.16 (dd, J = 15.9, 7.0 Hz, 1H), 5.97 (dd, J = 8.8, 4.5 Hz, 1H), 4.57 (td, J = 8.2, 4.2 Hz, 1H), 2.59 – 2.51 (m, 1H), 2.44 (ddd, J = 14.3, 8.8, 4.1 Hz, 1H), 2.37 (s, 3H), 1.99 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.1, 133.2, 131.8, 129.4, 128.9, 126.5, 70.6, 70.1, 50.5, 25.8, 21.3. HRMS (*ESI*) m/z: $[M+Cl]^-$ Calcd for $C_{12}H_{14}Cl_3O^-$ 279.0116; Found 279.0203.



(*E*)-1-(4-(tert-butyl)phenyl)-5,5-dichloropent-1-en-3-ol (**4d**): Yield: 70%, yellow oil liquid; R_f = 0.40 (EtOAc/petroleum ether = 1/25). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.38 (m, 2H), 7.37 – 7.33 (m, 2H), 6.66 (d, J = 15.8 Hz, 1H), 6.18 (dd, J = 15.9, 7.0 Hz, 1H), 5.97 (dd, J = 8.8, 4.5 Hz, 1H), 4.64 – 4.45 (m, 1H), 2.55 (ddd, J = 13.6, 9.0, 4.5 Hz, 1H), 2.44 (ddd, J = 14.4, 8.8, 4.1 Hz, 1H), 1.93 (s, 1H), 1.35 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.4, 133.2, 131.6, 129.2, 126.4, 125.6, 70.6, 70.1, 50.5, 34.7, 31.3. HRMS (*ESI*) m/z: $[M+Cl]^-$ Calcd for $C_{15}H_{20}Cl_3O^-$ 321.0585; Found 321.0577.

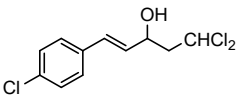


(*E*)-1-([1,1'-biphenyl]-4-yl)-5,5-dichloropent-1-en-3-ol (**4e**): Yield: 76%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/25). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 – 7.57 (m, 4H), 7.47 (t, J = 8.2 Hz, 4H), 7.38 (ddd, J = 7.4, 3.8, 1.1 Hz, 1H), 6.72 (d, J = 15.9 Hz, 1H), 6.27 (dd, J = 15.9, 6.9 Hz, 1H), 6.00 (dd, J = 8.8, 4.4 Hz, 1H), 4.85 – 4.44 (m, 1H), 2.58 (ddd, J = 13.6, 9.1, 4.4 Hz, 1H), 2.47 (ddd, J = 14.4, 8.8, 4.0 Hz, 1H), 1.93 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.0, 140.5, 135.0, 131.3, 130.0, 128.8, 127.5, 127.4, 127.04, 126.95, 70.6, 70.0, 50.5. HRMS (*ESI*) m/z: $[M+Cl]^-$ Calcd for $C_{17}H_{16}Cl_3O^-$ 341.1575; Found 341.1585.

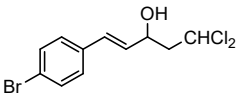


(*E*)-5,5-dichloro-1-(4-fluorophenyl)pent-1-en-3-ol (**4f**): Yield: 60%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 – 7.31

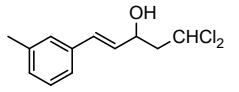
(m, 2H), 7.16 – 6.95 (m, 2H), 6.64 (d, J = 15.9 Hz, 1H), 6.14 (dd, J = 15.9, 6.9 Hz, 1H), 5.98 (dd, J = 8.9, 4.4 Hz, 1H), 4.67 – 4.49 (m, 1H), 2.55 (ddd, J = 13.6, 9.1, 4.4 Hz, 1H), 2.48 – 2.40 (m, 1H), 1.94 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.6 (d, J = 247.8 Hz), 132.2 (d, J = 3.4 Hz), 130.6, 129.8 (d, J = 2.2 Hz), 128.2 (d, J = 8.1 Hz), 115.7 (d, J = 21.7 Hz), 70.5, 69.9, 50.5. HRMS (*ESI*) m/z: $[M+Cl]^-$ Calcd for $C_{11}H_{11}Cl_3FO^-$ 282.9865; Found 282.9871.



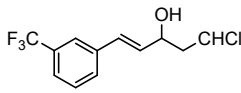
(*E*)-5,5-dichloro-1-(4-chlorophenyl)pent-1-en-3-ol (**4g**): Yield: 64%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (s, 4H), 6.64 (dd, J = 15.9, 1.0 Hz, 1H), 6.20 (dd, J = 15.9, 6.8 Hz, 1H), 5.98 (dd, J = 8.9, 4.3 Hz, 1H), 4.70 – 4.48 (m, 1H), 2.54 (ddd, J = 13.6, 9.1, 4.3 Hz, 1H), 2.44 (ddd, J = 14.4, 8.9, 3.9 Hz, 1H), 1.90 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.5, 133.8, 130.7, 130.4, 128.9, 127.8, 70.4, 69.8, 50.4. HRMS (*ESI*) m/z: $[M-H]^-$ Calcd for $C_{11}H_{10}Cl_3O^-$ 262.9803; Found 262.9805.



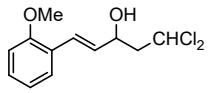
(*E*)-1-(4-bromophenyl)-5,5-dichloropent-1-en-3-ol (**4h**): Yield: 60%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 – 7.39 (m, 2H), 7.27 (d, J = 9.6 Hz, 2H), 6.62 (d, J = 15.9 Hz, 1H), 6.22 (ddd, J = 15.9, 6.7, 1.4 Hz, 1H), 6.05 – 5.92 (m, 1H), 4.77 – 4.35 (m, 1H), 4.89 – 4.52 (m, 1H), 2.58 – 2.49 (m, 1H), 2.48 – 2.39 (m, 1H), 1.90 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.0, 132.5, 131.8, 130.8, 130.5, 128.1, 70.4, 69.8, 50.4. HRMS (*ESI*) m/z: $[M+Cl]^-$ Calcd for $C_{11}H_{11}BrCl_3O^-$ 342.9064; Found 342.9061.



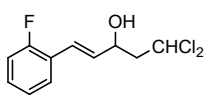
(*E*)-5,5-dichloro-1-(m-tolyl)pent-1-en-3-ol (**4i**): Yield: 70%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.18 (m, 3H), 7.13 (d, J = 7.3 Hz, 1H), 6.64 (d, J = 15.9 Hz, 1H), 6.21 (dd, J = 15.9, 7.0 Hz, 1H), 5.98 (dd, J = 8.8, 4.5 Hz, 1H), 4.58 (td, J = 8.0, 4.2 Hz, 1H), 2.55 (ddd, J = 13.6, 9.0, 4.5 Hz, 1H), 2.49 – 2.41 (m, 1H), 2.39 (s, 3H), 2.09 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.3, 136.0, 131.9, 129.8, 129.0, 128.6, 127.3, 123.8, 70.6, 70.0, 50.5, 21.4. HRMS (*ESI*) m/z: $[M+Cl]^-$ Calcd for $C_{12}H_{14}Cl_3O^-$ 279.0116; Found 279.0139.



(*E*)-5,5-dichloro-1-(3-(trifluoromethyl)phenyl)pent-1-en-3-ol (**4j**): Yield: 50%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (s, 1H), 7.60 – 7.52 (m, 2H), 7.47 (t, J = 7.7 Hz, 1H), 6.72 (d, J = 15.9 Hz, 1H), 6.31 (dd, J = 15.9, 6.5 Hz, 1H), 5.99 (dd, J = 8.9, 4.3 Hz, 1H), 4.62 (d, J = 9.5 Hz, 1H), 2.55 (ddd, J = 13.5, 9.1, 4.3 Hz, 1H), 2.50 – 2.42 (m, 1H), 2.02 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.9, 132.1, 131.3, 131.0, 130.1, 129.8, 129.2, 124.6 (q, J = 3.7 Hz), 123.2 (q, J = 3.7 Hz), 70.4, 69.7, 50.4. HRMS (*ESI*) m/z: $[M+Cl]^-$ Calcd for $C_{12}H_{11}Cl_3F_3O^-$ 332.9833; Found 332.9827.

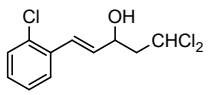


(*E*)-5,5-dichloro-1-(2-methoxyphenyl)pent-1-en-3-ol (**4k**): Yield: 62%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/15). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (dd, J = 7.6, 1.3 Hz, 1H), 7.28 (dd, J = 15.6, 1.5 Hz, 1H), 6.98 (dd, J = 11.7, 8.2 Hz, 2H), 6.92 (t, J = 8.6 Hz, 1H), 6.25 (dd, J = 16.0, 7.1 Hz, 1H), 5.99 (dd, J = 8.9, 4.4 Hz, 1H), 4.68 – 4.47 (m, 1H), 3.88 (s, 3H), 2.56 (ddd, J = 13.7, 9.1, 4.4 Hz, 1H), 2.51 – 2.39 (m, 1H), 1.91 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.9, 130.6, 129.3, 127.1, 126.8, 125.0, 120.7, 110.9, 70.7, 70.5, 55.4, 50.6. HRMS (*ESI*) m/z: $[M+Cl]^-$ Calcd for $C_{12}H_{14}Cl_3O_2^-$ 295.0065; Found 295.0068.

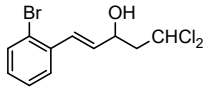


(*E*)-5,5-dichloro-1-(2-fluorophenyl)pent-1-en-3-ol (**4l**): Yield: 53%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (td, J = 7.7, 1.6 Hz, 1H), 7.26 (ddd, J = 7.2, 5.5, 1.8 Hz, 1H), 7.16 – 7.11 (m, 1H), 7.10 – 7.04 (m, 1H), 6.83 (d, J = 16.1 Hz, 1H), 6.32 (dd, J = 16.1, 6.7 Hz, 1H), 5.99 (dd, J = 8.9, 4.3 Hz, 1H), 4.61 (dd, J = 15.2, 4.3 Hz, 1H), 2.55 (ddd, J = 13.6, 9.2, 4.3 Hz, 1H), 2.49 – 2.42 (m, 1H), 1.95 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.39 (d, J = 249.9 Hz), 132.73 (d, J = 5.1 Hz), 129.43 (d, J = 8.5 Hz), 127.66 (d, J = 3.6 Hz), 124.22 (dd, J =

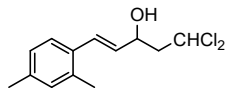
3.4, 1.0 Hz), 115.87 (d, *J* = 22.0 Hz), 70.48, 70.10, 50.40. HRMS (*ESI*) m/z: $[M+Cl]^-$ Calcd for $C_{11}H_{11}Cl_3FO^-$ 282.9865; Found 282.9873.



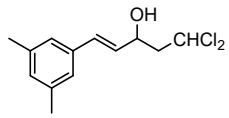
(*E*)-5,5-dichloro-1-(2-chlorophenyl)pent-1-en-3-ol (**4m**): Yield: 59%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (dd, *J* = 7.2, 2.1 Hz, 1H), 7.39 (dd, *J* = 7.4, 1.8 Hz, 1H), 7.24 (tt, *J* = 9.3, 3.8 Hz, 2H), 7.07 (d, *J* = 15.9 Hz, 1H), 6.22 (dd, *J* = 15.9, 6.7 Hz, 1H), 6.00 (dd, *J* = 8.9, 4.3 Hz, 1H), 4.85 – 4.49 (m, 1H), 2.56 (m, 1H), 2.51 – 2.41 (m, 1H), 2.12 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.3, 133.3, 133.0, 129.8, 129.1, 127.8, 127.0, 70.5, 69.9, 50.4. HRMS (*ESI*) m/z: $[M+Cl]^-$ Calcd for $C_{11}H_{11}Cl_4O^-$ 298.9569; Found 298.9576.



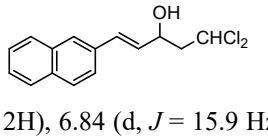
(*E*)-1-(2-bromophenyl)-5,5-dichloropent-1-en-3-ol (**4n**): Yield: 56%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (dd, *J* = 8.0, 1.2 Hz, 1H), 7.52 (dd, *J* = 7.8, 1.6 Hz, 1H), 7.32 (d, *J* = 7.3 Hz, 1H), 7.16 (td, *J* = 7.8, 1.6 Hz, 1H), 7.03 (d, *J* = 15.8 Hz, 1H), 6.17 (dd, *J* = 15.8, 6.7 Hz, 1H), 6.00 (dd, *J* = 8.9, 4.4 Hz, 1H), 4.79 – 4.47 (m, 1H), 2.56 (ddd, *J* = 13.5, 9.1, 4.4 Hz, 1H), 2.47 (ddd, *J* = 14.4, 8.9, 4.0 Hz, 1H), 1.95 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.0, 133.09, 133.05, 130.5, 129.4, 127.6, 127.2, 123.8, 70.4, 69.8, 50.3. HRMS (*ESI*) m/z: $[M+Cl]^-$ Calcd for $C_{11}H_{11}BrCl_3O^-$ 342.9064; Found 342.9073.



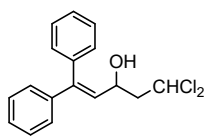
(*E*)-5,5-dichloro-1-(2,4-dimethylphenyl)pent-1-en-3-ol (**4o**): Yield: 52%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/25). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.4 Hz, 1H), 7.02 (d, *J* = 7.1 Hz, 2H), 6.87 (d, *J* = 15.7 Hz, 1H), 6.07 (dd, *J* = 15.7, 7.1 Hz, 1H), 5.98 (dd, *J* = 8.8, 4.5 Hz, 1H), 4.84 – 4.48 (m, 1H), 2.56 (ddd, *J* = 13.8, 9.0, 4.5 Hz, 1H), 2.50 – 2.41 (m, 1H), 2.35 (s, 3H), 2.34 (s, 3H), 1.92 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.9, 135.6, 132.2, 131.2, 130.3, 129.6, 127.0, 125.6, 70.6, 70.3, 50.6, 21.1, 19.7. HRMS (*ESI*) m/z: $[M+Cl]^-$ Calcd for $C_{13}H_{16}Cl_3O^-$ 293.0821; Found 293.1780.



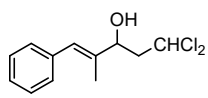
(*E*)-5,5-dichloro-1-(3,5-dimethylphenyl)pent-1-en-3-ol (**4p**): Yield: 62%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/25). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.04 (s, 2H), 6.95 (s, 1H), 6.61 (d, *J* = 15.9 Hz, 1H), 6.19 (dd, *J* = 15.9, 7.0 Hz, 1H), 5.97 (dd, *J* = 8.7, 4.5 Hz, 1H), 4.57 (td, *J* = 8.1, 4.3 Hz, 1H), 2.55 (ddd, *J* = 13.7, 9.0, 4.5 Hz, 1H), 2.49 – 2.40 (m, 1H), 2.34 (s, 6H), 1.95 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.2, 135.9, 132.0, 129.9, 129.5, 124.5, 70.6, 70.1, 50.5, 21.3. HRMS (*ESI*) m/z: $[M+Cl]^-$ Calcd for $C_{13}H_{16}Cl_3O^-$ 293.0272; Found 293.0280.



(*E*)-5,5-dichloro-1-(naphthalen-2-yl)pent-1-en-3-ol (**4q**): Yield: 48%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/25). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 – 7.80 (m, 3H), 7.77 (s, 1H), 7.60 (dd, *J* = 8.6, 1.6 Hz, 1H), 7.50 (ddt, *J* = 11.0, 7.4, 3.4 Hz, 2H), 6.84 (d, *J* = 15.9 Hz, 1H), 6.35 (dd, *J* = 15.9, 6.9 Hz, 1H), 6.01 (dd, *J* = 8.8, 4.4 Hz, 1H), 4.65 (dt, *J* = 9.8, 4.1 Hz, 1H), 2.60 (ddd, *J* = 13.7, 9.1, 4.4 Hz, 1H), 2.53 – 2.45 (m, 1H), 1.95 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 133.52, 133.46, 133.2, 131.9, 130.3, 128.4, 128.1, 127.7, 126.9, 126.5, 126.2, 123.4, 70.6, 70.1, 50.6. HRMS (*ESI*) m/z: $[M+H]^+$ Calcd for $C_{15}H_{15}Cl_2O^+$ 281.0494; Found 281.0486.

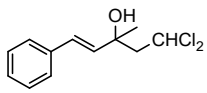


5,5-dichloro-1,1-diphenylpent-1-en-3-ol (**4r**): Yield: 37%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/25). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.37 (m, 3H), 7.32 (dd, *J* = 9.7, 4.5 Hz, 3H), 7.29 – 7.25 (m, 2H), 7.23 – 7.17 (m, 2H), 6.07 (dd, *J* = 9.1, 1.1 Hz, 1H), 5.92 (m, 1H), 4.53 (td, *J* = 9.0, 2.4 Hz, 1H), 2.71 – 2.53 (m, 1H), 2.50 – 2.36 (m, 1H), 1.70 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.0, 141.1, 138.6, 129.5, 128.7, 128.5, 128.3, 128.0, 127.9, 127.5, 70.4, 66.7, 50.7. HRMS (*ESI*) m/z: $[M+Cl]^-$ Calcd for $C_{17}H_{16}Cl_3O$- 341.0272; Found 341.0265.

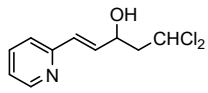


(*E*)-5,5-dichloro-2-methyl-1-phenylpent-1-en-3-ol (**4s**: 4.5/1.0): Yield: 45%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.32 (m, 2H), 7.27 (d, *J* = 7.2 Hz, 2H), 7.23 (d, *J* = 7.3 Hz, 1H), 6.58 (s, 1H), 5.96 (dd, *J* =

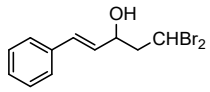
9.1, 4.1 Hz, 1H), 4.45 (d, *J* = 9.1 Hz, 1H), 2.57 – 2.48 (m, 1H), 2.47 – 2.39 (m, 1H), 1.89 (d, *J* = 1.3 Hz, 3H), 1.82 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.5, 136.9, 129.0, 128.7, 128.3, 128.2, 126.9, 126.6, 74.6, 71.1, 49.0, 13.6. HRMS (*ESI*) m/z: $[M-H]^-$ Calcd for $C_{12}H_{13}Cl_2O^-$ 243.0349; Found 243.0363.



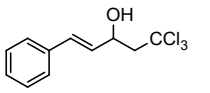
(*E*)-5,5-dichloro-3-methyl-1-phenylpent-1-en-3-ol (**4t**: 5.0/1.0): Yield: 36%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (dd, *J* = 6.6, 5.2 Hz, 2H), 7.36 (dd, *J* = 10.1, 4.8 Hz, 2H), 7.31 – 7.27 (m, 1H), 6.69 (d, *J* = 16.0 Hz, 1H), 6.28 (d, *J* = 16.0 Hz, 1H), 6.09 – 5.86 (m, 1H), 2.78 – 2.58 (m, 2H), 2.21 (s, 1H), 1.49 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.36, 134.12, 128.68, 128.39, 127.84, 126.56, 72.56, 69.84, 55.35, 29.40. HRMS (*ESI*) m/z: $[M+Cl]^-$ Calcd for $C_{12}H_{14}Cl_3O^-$ 279.0116; Found 279.0157.



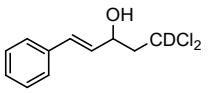
(*E*)-5,5-dichloro-1-(pyridin-2-yl)pent-1-en-3-ol (**4w**): Yield: 42%, yellow oil liquid; R_f = 0.35 (EtOAc/petroleum ether = 1/5). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 – 8.44 (m, 1H), 7.68 (td, *J* = 7.7, 1.8 Hz, 1H), 7.31 (d, *J* = 7.9 Hz, 1H), 7.19 (ddd, *J* = 7.5, 4.9, 1.0 Hz, 1H), 6.88 – 6.60 (m, 2H), 6.03 (dd, *J* = 8.9, 4.3 Hz, 1H), 4.77 – 4.56 (m, 1H), 2.54 (ddd, *J* = 13.5, 9.2, 4.3 Hz, 1H), 2.47 (ddd, *J* = 14.5, 9.0, 4.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.6, 149.4, 136.9, 135.3, 130.4, 122.6, 122.0, 70.6, 69.1, 50.4. HRMS (*ESI*) m/z: $[M+H]^+$ Calcd for $C_{10}H_{12}Cl_2NO^+$ 232.0290; Found 232.0283.



(*E*)-5,5-dibromo-1-phenylpent-1-en-3-ol (**4x**): Yield: 65%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.39 (m, 2H), 7.39 – 7.33 (m, 2H), 7.32 – 7.28 (m, 1H), 6.69 (d, *J* = 15.9 Hz, 1H), 6.23 (dd, *J* = 15.9, 6.9 Hz, 1H), 5.88 (dd, *J* = 9.2, 4.6 Hz, 1H), 4.59 – 4.49 (m, 1H), 2.81 – 2.70 (m, 1H), 2.69 – 2.59 (m, 1H), 1.84 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.0, 131.9, 129.7, 128.7, 128.2, 126.6, 71.4, 52.1, 42.1. HRMS (*ESI*) m/z: $[M+Cl]^-$ Calcd for $C_{11}H_{12}Br_2ClO^-$ 352.8949; Found 352.8956.

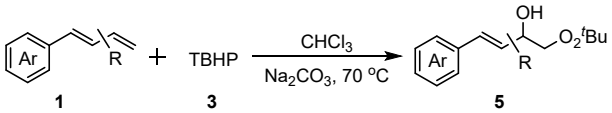


(*E*)-5,5,5-trichloro-1-phenylpent-1-en-3-ol (**4y**): Yield: 52%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, *J* = 7.3 Hz, 2H), 7.36 (t, *J* = 7.5 Hz, 2H), 7.30 (d, *J* = 7.2 Hz, 1H), 6.75 (d, *J* = 15.9 Hz, 1H), 6.28 (dd, *J* = 15.9, 6.5 Hz, 1H), 4.91 (q, *J* = 6.2 Hz, 1H), 3.15 (dd, *J* = 15.1, 6.8 Hz, 1H), 3.06 (dd, *J* = 15.2, 3.8 Hz, 1H), 2.25 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.1, 131.5, 129.8, 128.7, 128.1, 126.6, 124.3, 70.6, 61.2. HRMS (*ESI*) m/z: $[M+Cl]^-$ Calcd for $C_{11}H_{11}Cl_4O^-$ 298.9569; Found 298.9729.



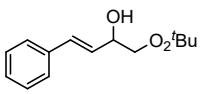
(*E*)-5,5-dichloro-1-phenylpent-1-en-5-d-3-ol (**4aa**): Yield: 68%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.38 (m, 2H), 7.36 (t, *J* = 7.4 Hz, 2H), 7.32 – 7.29 (m, 1H), 6.68 (d, *J* = 15.9 Hz, 1H), 6.23 (dd, *J* = 15.9, 6.9 Hz, 1H), 4.60 (t, *J* = 9.7 Hz, 1H), 2.55 (dd, *J* = 14.4, 9.1 Hz, 1H), 2.45 (dd, *J* = 14.3, 3.9 Hz, 1H), 1.86 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.0, 131.8, 130.0, 128.7, 128.2, 126.6, 70.0, 50.4. HRMS (*ESI*) m/z: $[M+Cl]^-$ Calcd for $C_{11}H_{11}DCl_3O^-$ 266.0022; Found 266.0030.

General Procedure for Products 5.



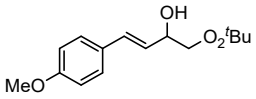
1 **3** **5**

To a solution of **1** (0.5 mmol) in $CHCl_3$ (0.5 mL) was added Na_2CO_3 (1.0 mmol) and TBHP (2.0 mmol) at room temperature. Then the solution mixture was stirred at 70℃ for 12 h, after completion of the reaction, the solvent was removed under reduced pressure and the crude product was subjected to silica gel column chromatography to afford **5**.

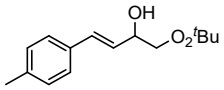


(*E*)-1-(tert-butylperoxy)-4-phenylbut-3-en-2-ol (**5a**): Yield: 67%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, *J* = 7.4 Hz,

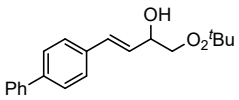
2H), 7.34 (t, *J* = 7.5 Hz, 2H), 7.30 – 7.24 (m, 1H), 6.75 (d, *J* = 15.9 Hz, 1H), 6.19 (dd, *J* = 16.0, 6.1 Hz, 1H), 4.88 – 4.59 (m, 1H), 4.12 (dd, *J* = 12.5, 2.8 Hz, 1H), 3.93 (dd, *J* = 12.5, 8.2 Hz, 1H), 2.80 (s, 1H), 1.31 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.6, 131.8, 128.6, 127.8, 127.3, 126.5, 81.0, 79.1, 71.0, 26.3. HRMS (*ESI*) m/z: $[M+Na]^+$ Calcd for $C_{14}H_{20}NaO_3^+$ 259.1305; Found 259.1312.



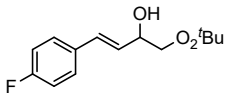
(*E*)-1-(tert-butylperoxy)-4-(4-methoxyphenyl)but-3-en-2-ol (**5b**): Yield: 74%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/15). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 (d, *J* = 8.7 Hz, 2H), 6.87 (d, *J* = 8.7 Hz, 2H), 6.68 (d, *J* = 15.9 Hz, 1H), 6.04 (dd, *J* = 15.9, 6.4 Hz, 1H), 4.67 (t, *J* = 6.6 Hz, 1H), 4.10 (dd, *J* = 12.5, 2.8 Hz, 1H), 3.96 – 3.88 (m, 1H), 3.83 (s, 3H), 1.30 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.4, 131.4, 129.4, 127.7, 125.0, 114.0, 80.9, 79.2, 71.2, 55.3, 26.3. HRMS (*ESI*) m/z: $[M+Na]^+$ Calcd for $C_{15}H_{22}NaO_4^+$ 289.1410; Found 289.1418.



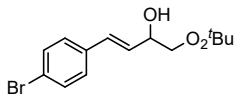
(*E*)-1-(tert-butylperoxy)-4-(p-tolyl)but-3-en-2-ol (**5c**): Yield: 72%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 (d, *J* = 8.1 Hz, 2H), 7.14 (d, *J* = 7.9 Hz, 2H), 6.71 (d, *J* = 15.9 Hz, 1H), 6.13 (dd, *J* = 16.0, 6.3 Hz, 1H), 4.73 – 4.65 (m, 1H), 4.10 (dd, *J* = 12.5, 2.8 Hz, 1H), 3.92 (dd, *J* = 12.5, 8.3 Hz, 1H), 2.42 (dd, *J* = 21.0, 7.7 Hz, 1H), 2.36 (s, 3H), 1.31 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.6, 133.8, 131.8, 129.3, 126.4, 126.2, 79.2, 71.1, 26.3, 21.2. HRMS (*ESI*) m/z: $[M+Na]^+$ Calcd for $C_{15}H_{22}NaO_3^+$ 273.1461; Found 273.1468.



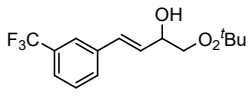
(*E*)-4-([1,1'-biphenyl]-4-yl)-1-(tert-butylperoxy)but-3-en-2-ol (**5d**): Yield: 66%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/25). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 – 7.61 (m, 2H), 7.59 (d, *J* = 8.3 Hz, 2H), 7.51 – 7.47 (m, 2H), 7.45 (d, *J* = 7.8 Hz, 2H), 7.39 – 7.32 (m, 1H), 6.79 (d, *J* = 16.0 Hz, 1H), 6.24 (dd, *J* = 16.0, 6.1 Hz, 1H), 4.88 – 4.66 (m, 1H), 4.14 (dd, *J* = 12.5, 2.8 Hz, 1H), 3.95 (dd, *J* = 12.5, 8.2 Hz, 1H), 2.81 (s, 1H), 1.32 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.7, 140.7, 140.5, 135.6, 131.3, 128.8, 127.4, 127.3, 127.3, 126.9, 81.0, 79.1, 71.1, 26.4. HRMS (*ESI*) m/z: $[M+Na]^+$ Calcd for $C_{20}H_{24}NaO_3^+$ 335.1618; Found 335.1626.



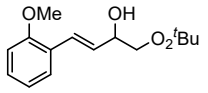
(*E*)-1-(tert-butylperoxy)-4-(4-fluorophenyl)but-3-en-2-ol (**5e**): Yield: 57%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (dd, *J* = 8.7, 5.4 Hz, 2H), 7.02 (t, *J* = 8.7 Hz, 2H), 6.71 (d, *J* = 15.8 Hz, 1H), 6.10 (dd, *J* = 16.0, 6.1 Hz, 1H), 4.69 (t, *J* = 6.5 Hz, 1H), 4.11 (dd, *J* = 12.5, 2.8 Hz, 1H), 3.93 (d, *J* = 8.2 Hz, 1H), 2.80 (s, 1H), 1.31 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.40 (d, *J* = 247.2 Hz), 132.77 (d, *J* = 3.4 Hz), 130.60, 128.02 (d, *J* = 8.0 Hz), 127.04 (d, *J* = 2.2 Hz), 115.48 (d, *J* = 21.6 Hz), 81.00, 79.06, 70.97, 26.33. HRMS (*ESI*) m/z: $[M+Na]^+$ Calcd for $C_{14}H_{19}FNaO_3^+$ 277.1210; Found 277.1217.



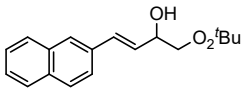
(*E*)-4-(4-bromophenyl)-1-(tert-butylperoxy)but-3-en-2-ol (**5f**): Yield: 60%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 (d, *J* = 8.4 Hz, 2H), 7.36 – 7.18 (m, 2H), 6.69 (d, *J* = 16.0 Hz, 1H), 6.18 (dd, *J* = 16.0, 6.0 Hz, 1H), 4.69 (t, *J* = 6.5 Hz, 1H), 4.11 (dd, *J* = 12.6, 2.8 Hz, 1H), 3.91 (dd, *J* = 12.6, 8.2 Hz, 1H), 2.82 (s, 1H), 1.30 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.6, 131.7, 130.5, 128.1, 128.0, 121.5, 81.0, 79.0, 70.9, 26.3. HRMS (*ESI*) m/z: $[M+Na]^+$ Calcd for $C_{14}H_{19}BrNaO_3^+$ 337.0410; Found 337.0405.



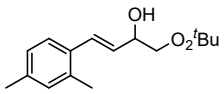
(*E*)-1-(tert-butylperoxy)-4-(3-(trifluoromethyl)phenyl)but-3-en-2-ol (**5g**): Yield: 44%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (s, 1H), 7.56 (d, *J* = 7.5 Hz, 1H), 7.51 (d, *J* = 7.7 Hz, 1H), 7.44 (t, *J* = 7.7 Hz, 1H), 6.79 (dd, *J* = 16.0, 1.2 Hz, 1H), 6.28 (dd, *J* = 16.0, 5.8 Hz, 1H), 4.73 (s, 1H), 4.13 (dd, *J* = 12.6, 2.9 Hz, 1H), 3.93 (dd, *J* = 12.6, 8.1 Hz, 1H), 2.92 (s, 1H), 1.31 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.43, 131.14, 130.82, 130.14, 129.69, 129.46, 129.02, 124.22 (dd, *J* = 7.5, 3.7 Hz), 123.09 (q, *J* = 3.9 Hz), 81.08, 78.94, 70.78, 26.31. HRMS (*ESI*) m/z: $[M+H]^+$ Calcd for $C_{15}H_{20}F_3O_3^+$ 305.1359; Found 305.1512.



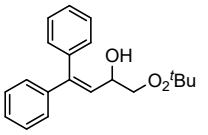
(*E*)-1-(tert-butylperoxy)-4-(2-methoxyphenyl)but-3-en-2-ol (**5h**): Yield: 52%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/15). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (dd, J = 7.6, 1.5 Hz, 1H), 7.27 – 7.21 (m, 1H), 7.04 (dd, J = 16.6, 6.8 Hz, 1H), 6.94 (t, J = 7.1 Hz, 1H), 6.89 (d, J = 8.2 Hz, 1H), 6.21 (dd, J = 16.1, 6.4 Hz, 1H), 4.71 (dd, J = 6.9, 6.0 Hz, 1H), 4.12 (dd, J = 12.4, 2.9 Hz, 1H), 3.95 (dd, J = 7.8, 4.6 Hz, 1H), 3.92 – 3.89 (m, 1H), 3.86 (s, 3H), 2.77 (s, 1H), 1.31 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.8, 128.8, 128.0, 127.0, 126.8, 125.6, 120.6, 110.9, 80.9, 79.2, 71.4, 55.4, 26.4. HRMS (*ESI*) m/z: $[M+Na]^+$ Calcd for $C_{15}H_{22}NaO_4^+$ 289.1410; Found 289.1407.



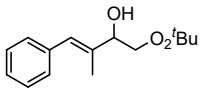
(*E*)-1-(tert-butylperoxy)-4-(naphthalen-2-yl)but-3-en-2-ol (**5i**): Yield: 49%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/25). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.75 (m, 4H), 7.62 (dd, J = 8.6, 1.8 Hz, 1H), 7.53 – 7.43 (m, 2H), 6.91 (dd, J = 16.0, 1.4 Hz, 1H), 6.32 (dd, J = 15.9, 6.1 Hz, 1H), 4.77 (m, 1H), 4.16 (dd, J = 12.5, 2.9 Hz, 1H), 3.97 (dd, J = 12.6, 8.2 Hz, 1H), 2.85 (s, 1H), 1.32 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.1, 133.6, 133.1, 131.9, 128.2, 128.0, 127.69, 127.66, 126.6, 126.3, 125.9, 123.6, 81.0, 79.1, 71.1, 26.4. HRMS (*ESI*) m/z: $[M+Na]^+$ Calcd for $C_{18}H_{22}NaO_3^+$ 309.1461; Found 309.1457.



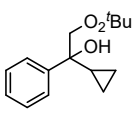
(*E*)-1-(tert-butylperoxy)-4-(2,4-dimethylphenyl)but-3-en-2-ol (**5j**): Yield: 41%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/25). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, J = 7.8 Hz, 1H), 7.00 (d, J = 7.2 Hz, 2H), 6.92 (d, J = 15.8 Hz, 1H), 6.03 (dd, J = 15.8, 6.2 Hz, 1H), 4.70 (t, J = 6.3 Hz, 1H), 4.15 – 4.05 (m, 1H), 3.98 – 3.90 (m, 1H), 2.80 (s, 1H), 2.35 (s, 3H), 2.32 (s, 3H), 1.31 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.4, 135.4, 132.9, 131.1, 129.6, 127.7, 126.8, 125.6, 80.9, 79.2, 71.3, 26.4, 21.1, 19.74. HRMS (*ESI*) m/z: $[M+Na]^+$ Calcd for $C_{16}H_{24}NaO_3^+$ 287.1618; Found 287.1610.



1-(tert-butylperoxy)-4,4-diphenylbut-3-en-2-ol (**5k**): Yield: 35%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/25). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (dt, J = 6.7, 1.9 Hz, 2H), 7.39 – 7.34 (m, 2H), 7.29 (s, 4H), 7.27 – 7.24 (m, 2H), 6.07 (dd, J = 9.1, 1.7 Hz, 1H), 4.63 – 4.52 (m, 1H), 4.09 (dt, J = 12.4, 2.1 Hz, 1H), 4.02 – 3.93 (m, 1H), 1.25 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.4, 141.6, 139.1, 129.7, 128.3, 128.21, 128.17, 127.7, 127.6, 126.4, 80.9, 79.0, 68.4, 26.3. HRMS (*ESI*) m/z: $[M+Na]^+$ Calcd for $C_{20}H_{24}NaO_3^+$ 335.1618; Found 335.1611.

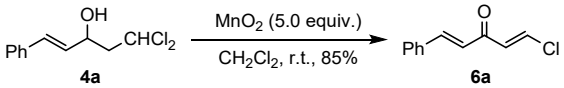


1-(tert-butylperoxy)-3-methyl-4-phenylbut-3-en-2-ol (**5l**: 3.3/1.0): Yield: 38%, yellow oil liquid; R_f = 0.37 (EtOAc/petroleum ether = 1/20). major: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.33 (m, 2H), 7.32 – 7.29 (m, 2H), 7.27 – 7.24 (m, 1H), 6.67 (s, 1H), 4.56 (dd, J = 8.4, 2.1 Hz, 1H), 4.15 (dd, J = 12.4, 2.6 Hz, 1H), 3.98 (dd, J = 12.4, 8.5 Hz, 1H), 2.82 (s, 1H), 1.91 (s, 3H), 1.31 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.5, 136.1, 129.0, 128.1, 126.7, 126.5, 80.9, 78.5, 75.4, 26.4, 26.3, 14.5. Minor: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.33 (m, 2H), 7.32 – 7.29 (m, 1H), 7.23 (d, J = 7.1 Hz, 2H), 6.50 (s, 1H), 5.14 – 5.08 (m, 1H), 4.10 (d, J = 5.6 Hz, 2H), 1.94 (d, J = 1.2 Hz, 3H), 1.27 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.2, 136.5, 129.2, 128.8, 128.1, 126.6, 78.2, 68.1, 65.2, 31.5, 18.5. HRMS (*ESI*) m/z: $[M+Na]^+$ Calcd for $C_{15}H_{22}NaO_3^+$ 273.1461; Found 273.1462.



2-(tert-butylperoxy)-1-cyclopropyl-1-phenylethan-1-ol (**5o**): Yield: 37%, yellow oil liquid; R_f = 0.35 (EtOAc/petroleum ether = 1/20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.93 (s, 1H), 7.37 (d, J = 4.3 Hz, 4H), 7.31 (dd, J = 8.8, 4.7 Hz, 1H), 4.80 – 4.57 (m, 2H), 1.51 – 1.38 (m, 1H), 1.34 (s, 9H), 0.54 – 0.38 (m, 2H), 0.32 – 0.25 (m, 1H), 0.08 (dq, J = 9.9, 4.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.71, 127.92, 127.62, 126.72, 87.98, 81.79, 77.15, 26.36, 15.77, 1.08, 0.95. HRMS (*ESI*) m/z: $[M+Na]^+$ Calcd for $C_{15}H_{22}NaO_3^+$ 273.1461; Found 273.1467.

Procedure for the Synthesis of Products 6a and 7a.



To a solution of **4a** (0.2 mmol) in CH_2Cl_2 (2.0 mL) was added MnO_2 (1.0 mmol) at room temperature. Then the solution mixture was stirred at the same temperature, after completion of the reaction, the solvent was removed under reduced pressure and the crude product was subjected to silica gel column chromatography to afford **6a**.

1-chloro-5-phenylpenta-1,4-dien-3-one (**6a**)[3]: Yield: 85%; R_f = 0.4 (EtOAc/petroleum ether = 1/25) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (d, J = 16.0 Hz, 1H), 7.60 – 7.55 (m, 2H), 7.42 (dd, J = 7.3, 5.4 Hz, 4H), 6.89 (d, J = 13.4 Hz, 1H), 6.85 (d, J = 16.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.4, 144.6, 137.0, 134.3, 131.1, 130.9, 129.1, 128.5, 124.9.

References:

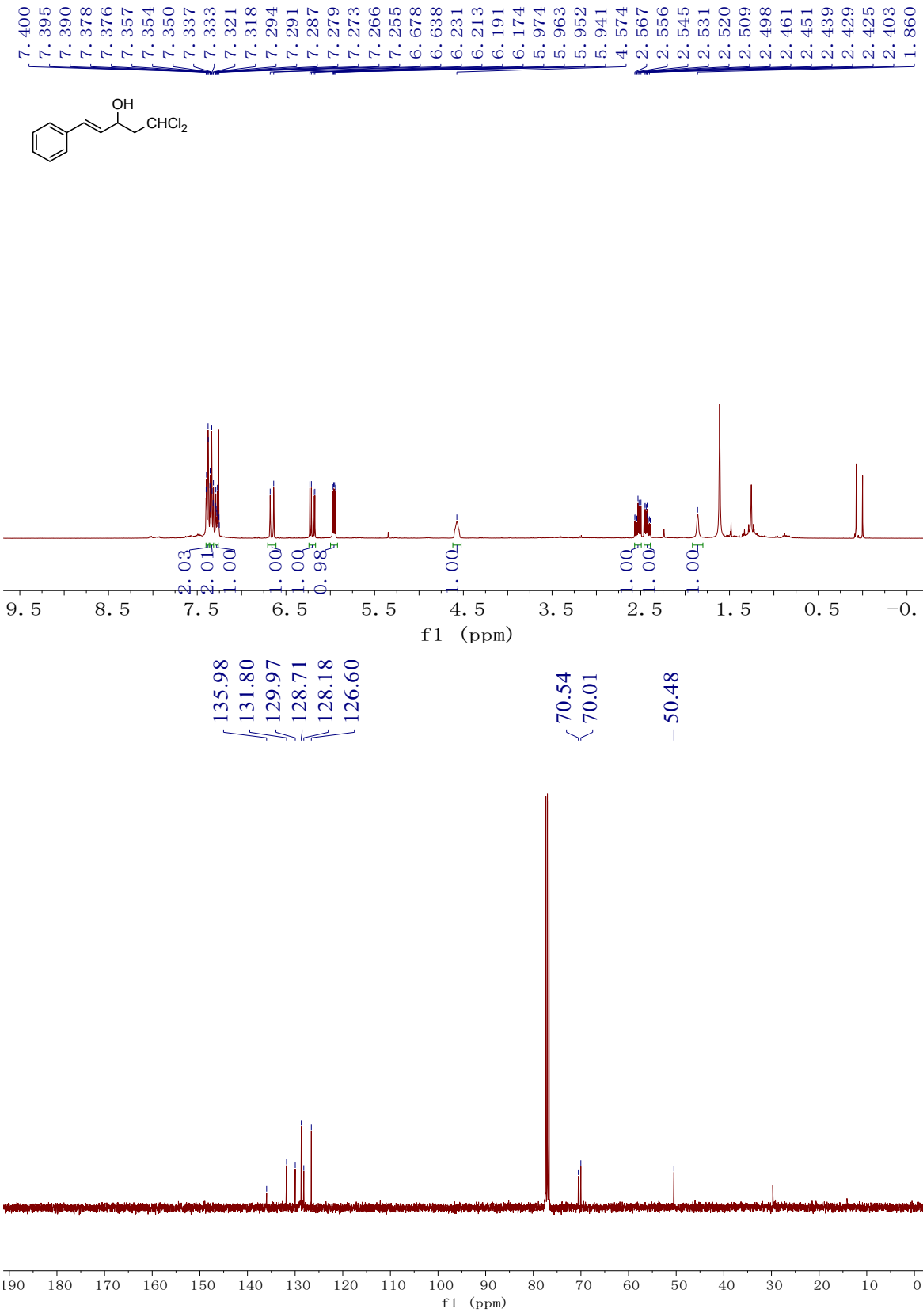
[1] X.-J. Wei, D.-T. Yang, L. Wang, T. Song, L.-Z. Wu, and Q. Liu, *Org. Lett.* 2013, **15**, 6054–6057.

[2] R. A. Fernandes, P. Kumar, A. Bhowmik, and D. A. Gorve, *Org. Lett.* 2022, **24**, 3435−3439.

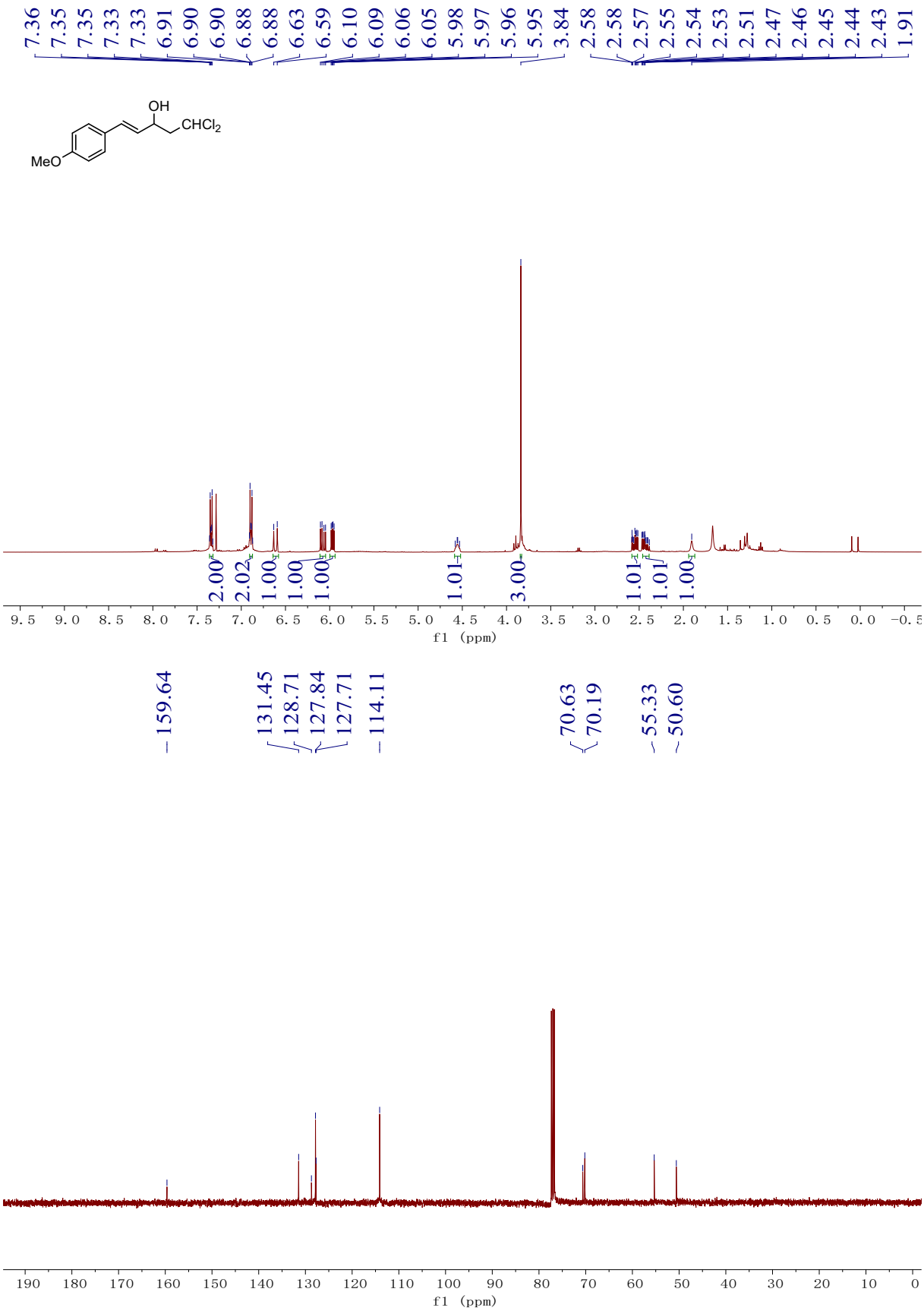
[3] A. S. K. Hashmi, and L. Grundl, *Tetrahedron* 2005, **61**, 6231–6236.

Copies of ^{1}H, ^{13}C NMR spectra

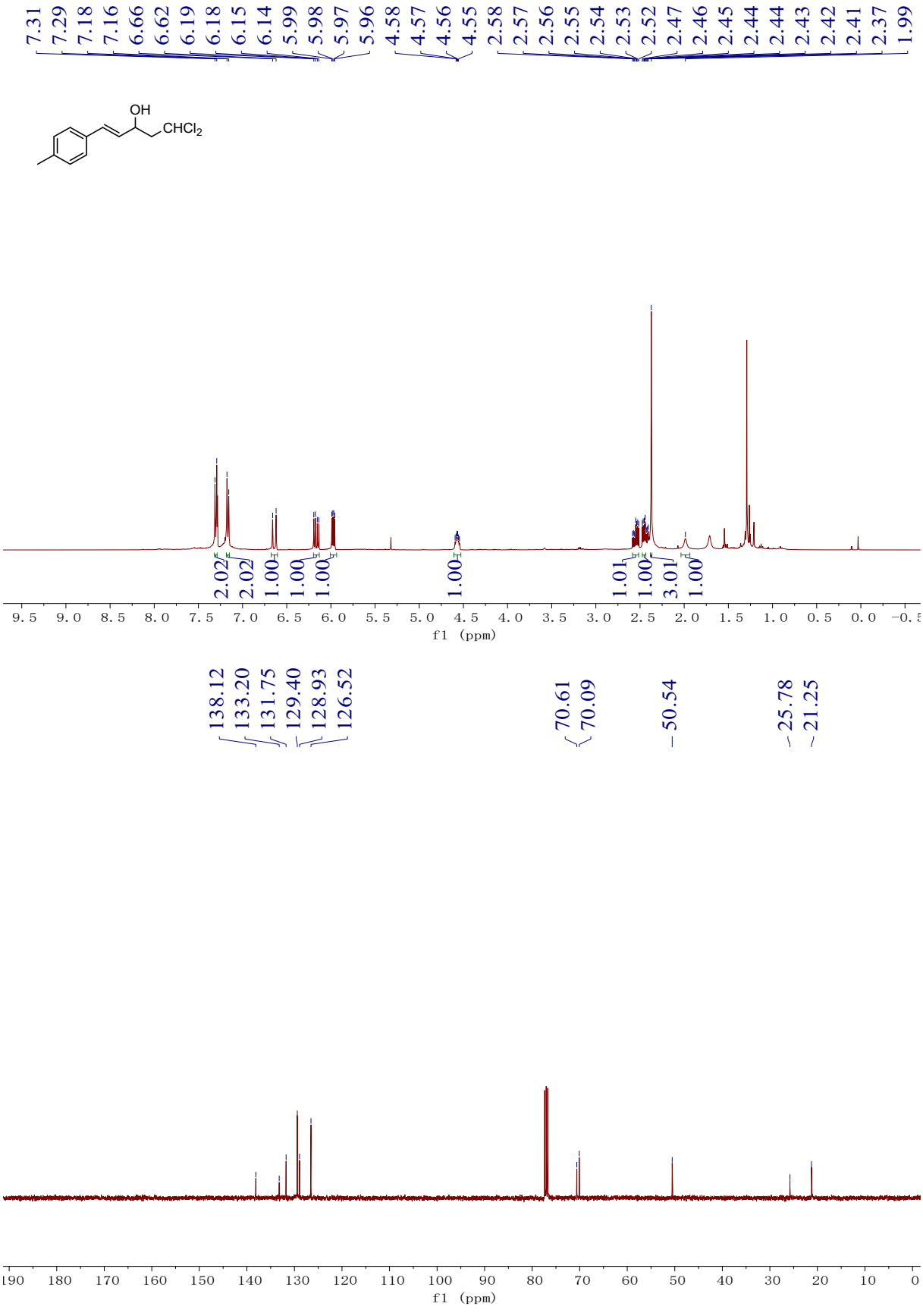
4a



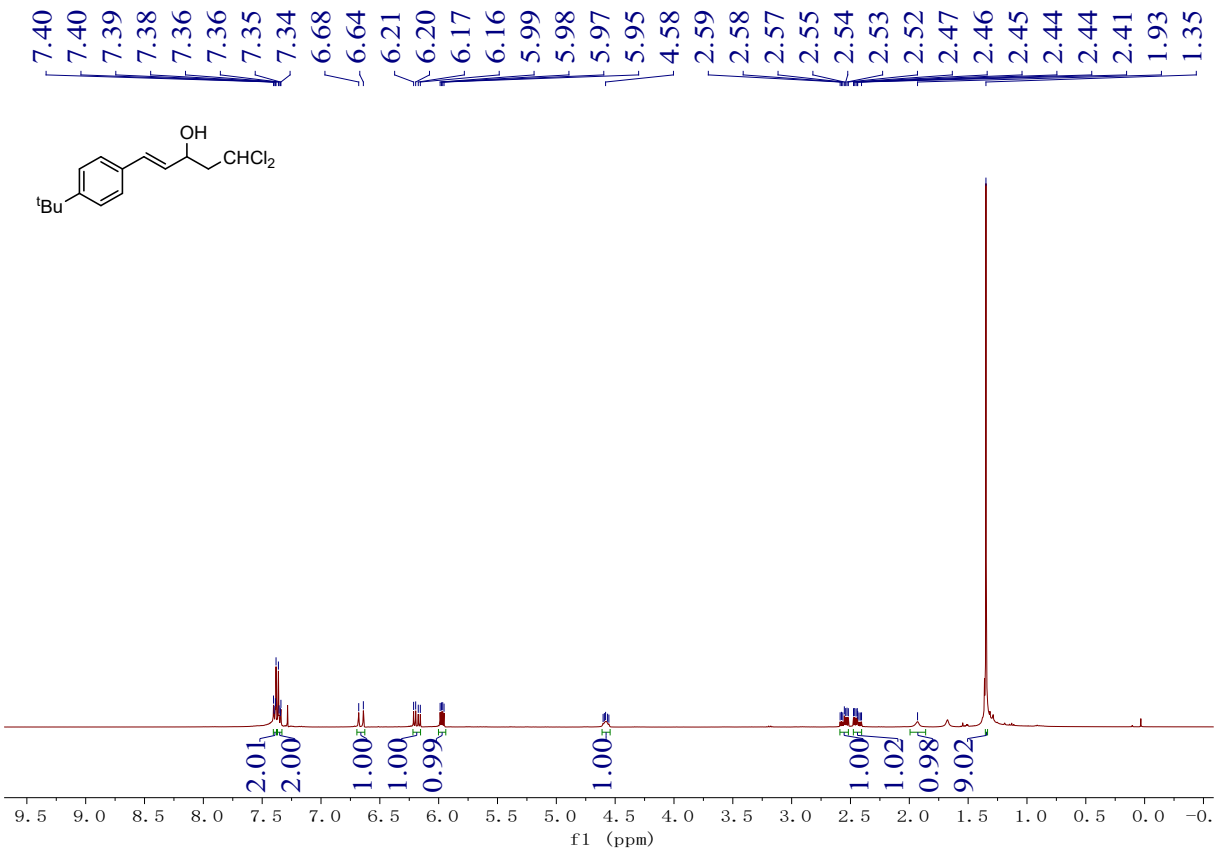
4b

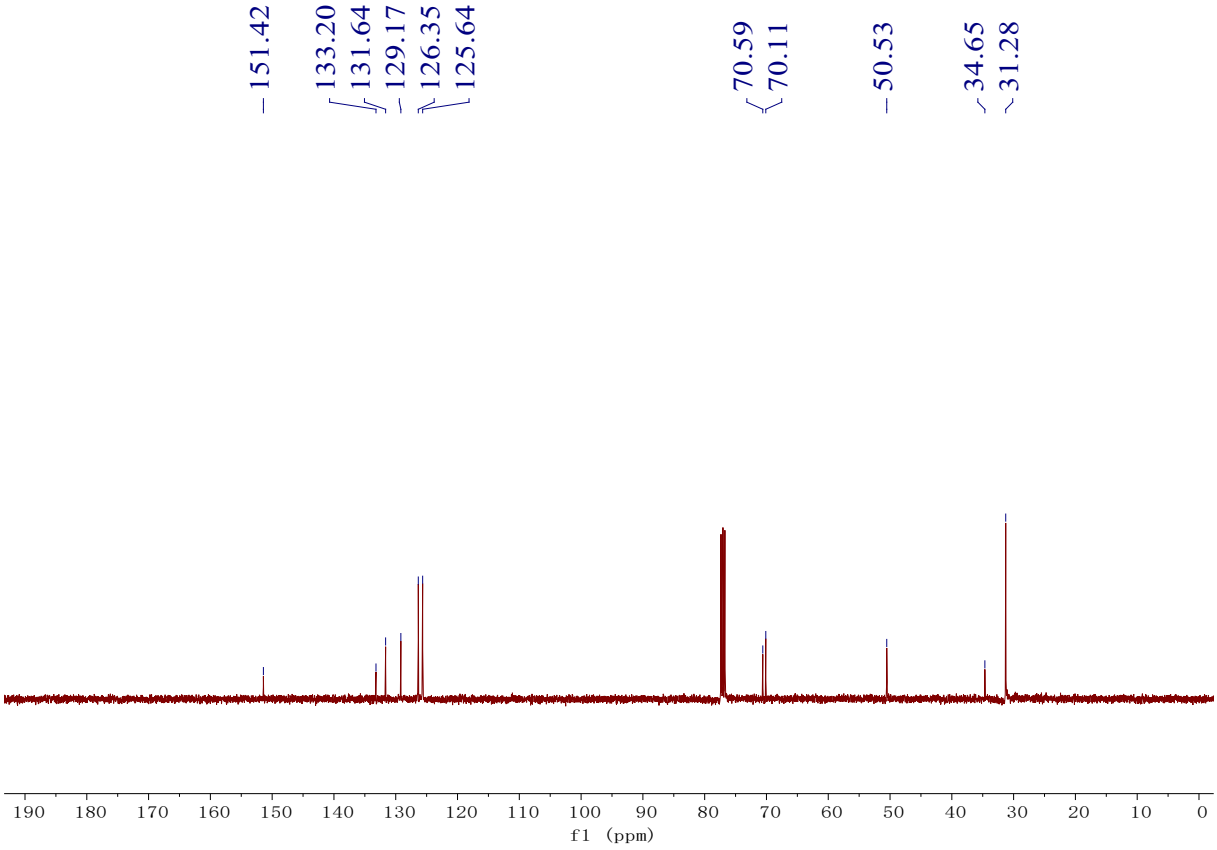

7.36
7.35
7.35
7.33
7.33
6.91
6.90
6.90
6.88
6.88
6.63
6.59
6.10
6.09
6.06
6.05
5.98
5.97
5.96
5.95
3.84
2.58
2.58
2.57
2.55
2.54
2.53
2.51
2.47
2.46
2.45
2.44
2.43
1.91
OH
CHCl2
MeO
2.00
2.02
1.00
1.00
1.00
1.01
3.00
1.01
1.01
1.00
f1 (ppm)
159.64
131.45
128.71
127.84
127.71
114.11
70.63
70.19
55.33
50.60
f1 (ppm)

4c

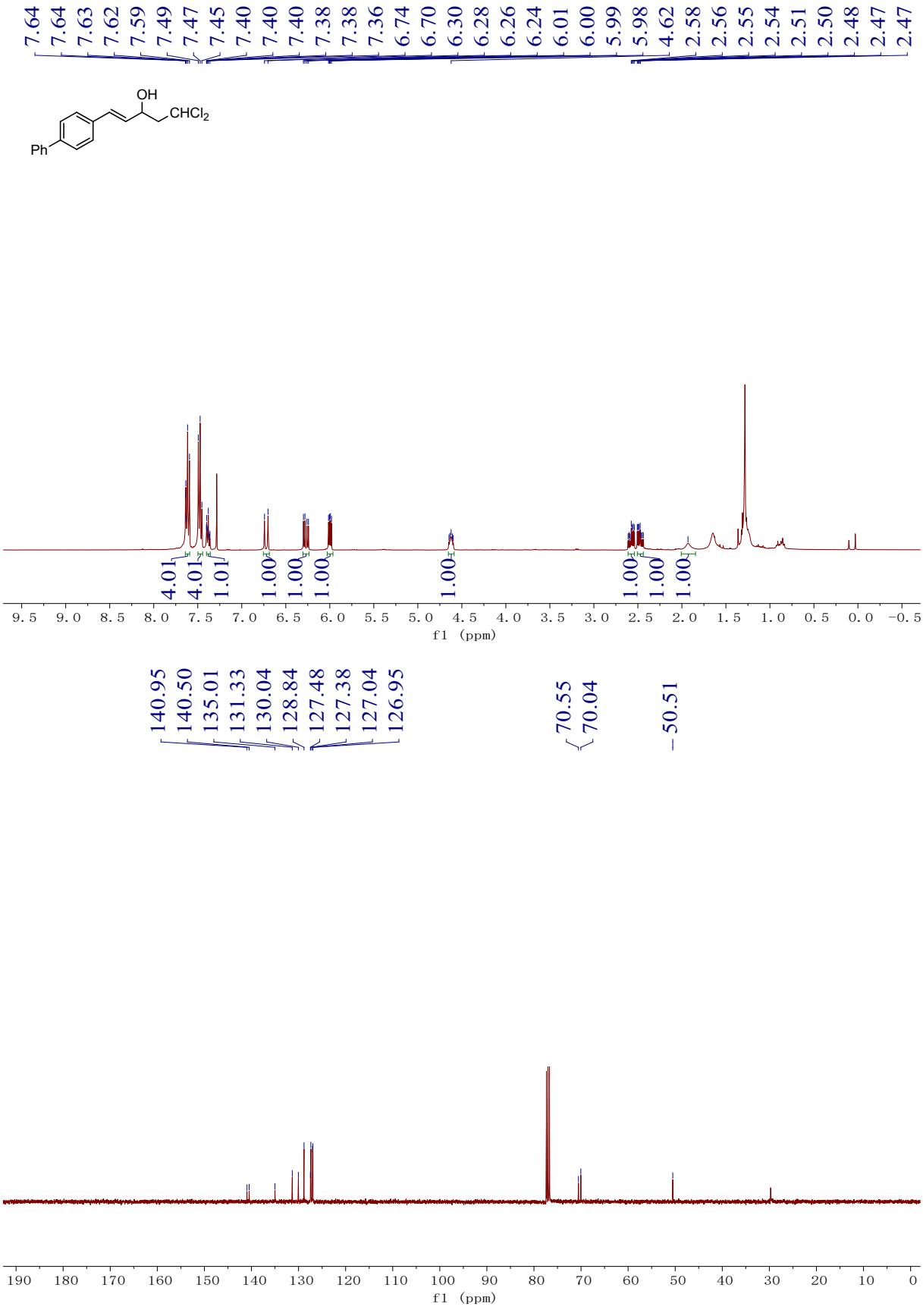

7.31
7.29
7.18
7.16
6.66
6.62
6.19
6.18
6.15
6.14
5.99
5.98
5.97
5.96
4.58
4.57
4.56
4.55
2.58
2.57
2.56
2.55
2.54
2.53
2.52
2.47
2.46
2.45
2.44
2.44
2.43
2.42
2.41
2.37
1.99
OH
CHCl2
2.02
2.02
1.00
1.00
1.00
1.00
1.01
1.00
3.01
1.00
f1 (ppm)
138.12
133.20
131.75
129.40
128.93
126.52
70.61
70.09
50.54
25.78
21.25
f1 (ppm)

4d

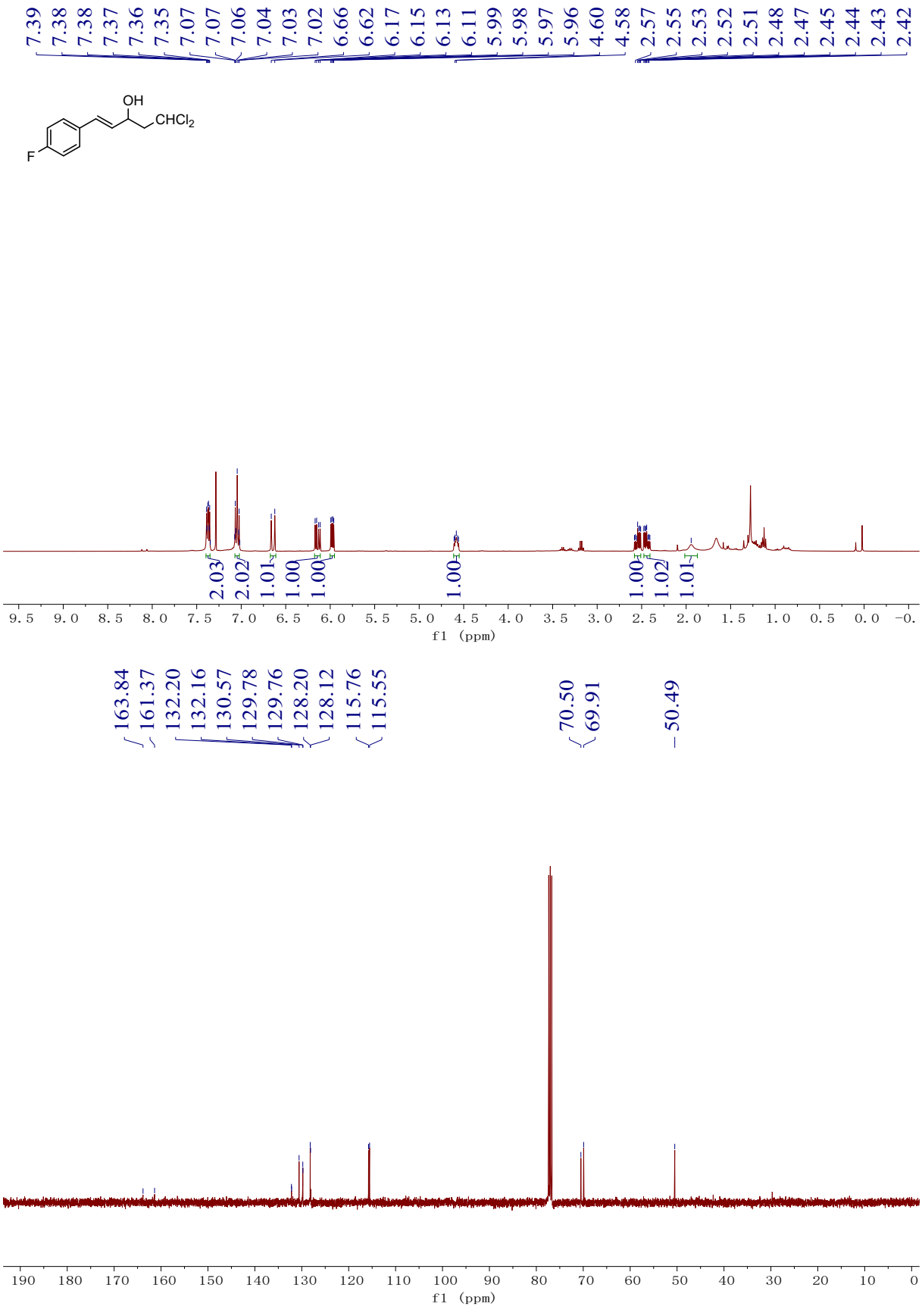

7.40
7.40
7.39
7.38
7.36
7.36
7.35
7.34
6.68
6.64
6.21
6.20
6.17
6.16
5.99
5.98
5.97
5.95
4.58
2.59
2.58
2.57
2.55
2.54
2.53
2.52
2.47
2.46
2.45
2.44
2.44
2.41
1.93
1.35
OH
CHCl2
tBu
2.01
2.00
1.00
1.00
0.99
1.00
1.00
1.02
0.98
9.02
f1 (ppm)


151.42
133.20
131.64
129.17
126.35
125.64
70.59
70.11
50.53
34.65
31.28
f1 (ppm)

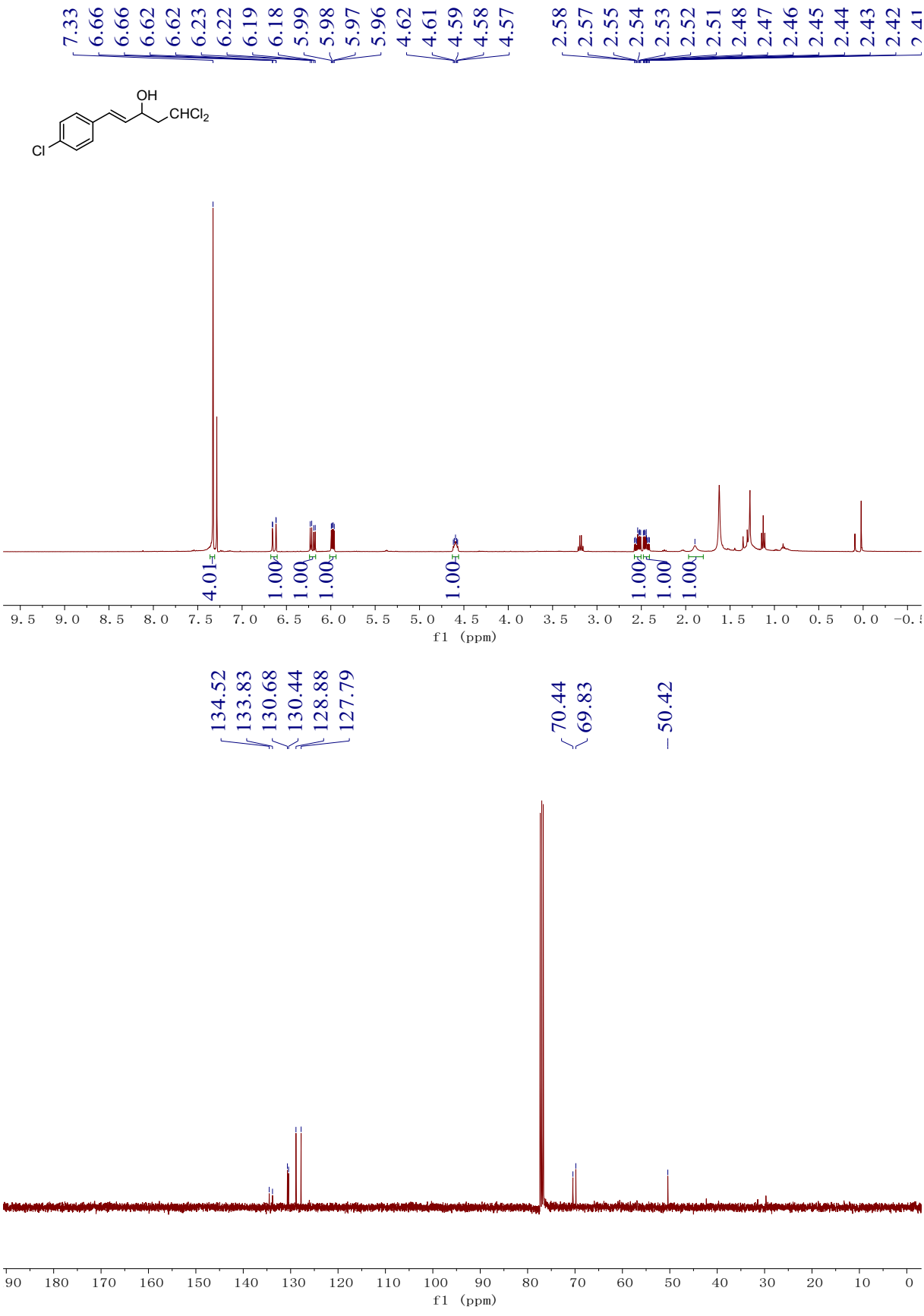
4e


7.64
7.64
7.63
7.62
7.59
7.49
7.47
7.45
7.40
7.40
7.40
7.38
7.38
7.36
6.74
6.70
6.30
6.28
6.26
6.24
6.01
6.00
5.99
5.98
4.62
2.58
2.56
2.55
2.54
2.51
2.50
2.48
2.47
2.47
OH
CHCl2
Ph
4.01
4.01
1.01
1.00
1.00
1.00
1.00
1.00
1.00
1.00
f1 (ppm)
140.95
140.50
135.01
131.33
130.04
128.84
127.48
127.38
127.04
126.95
70.55
70.04
50.51
f1 (ppm)

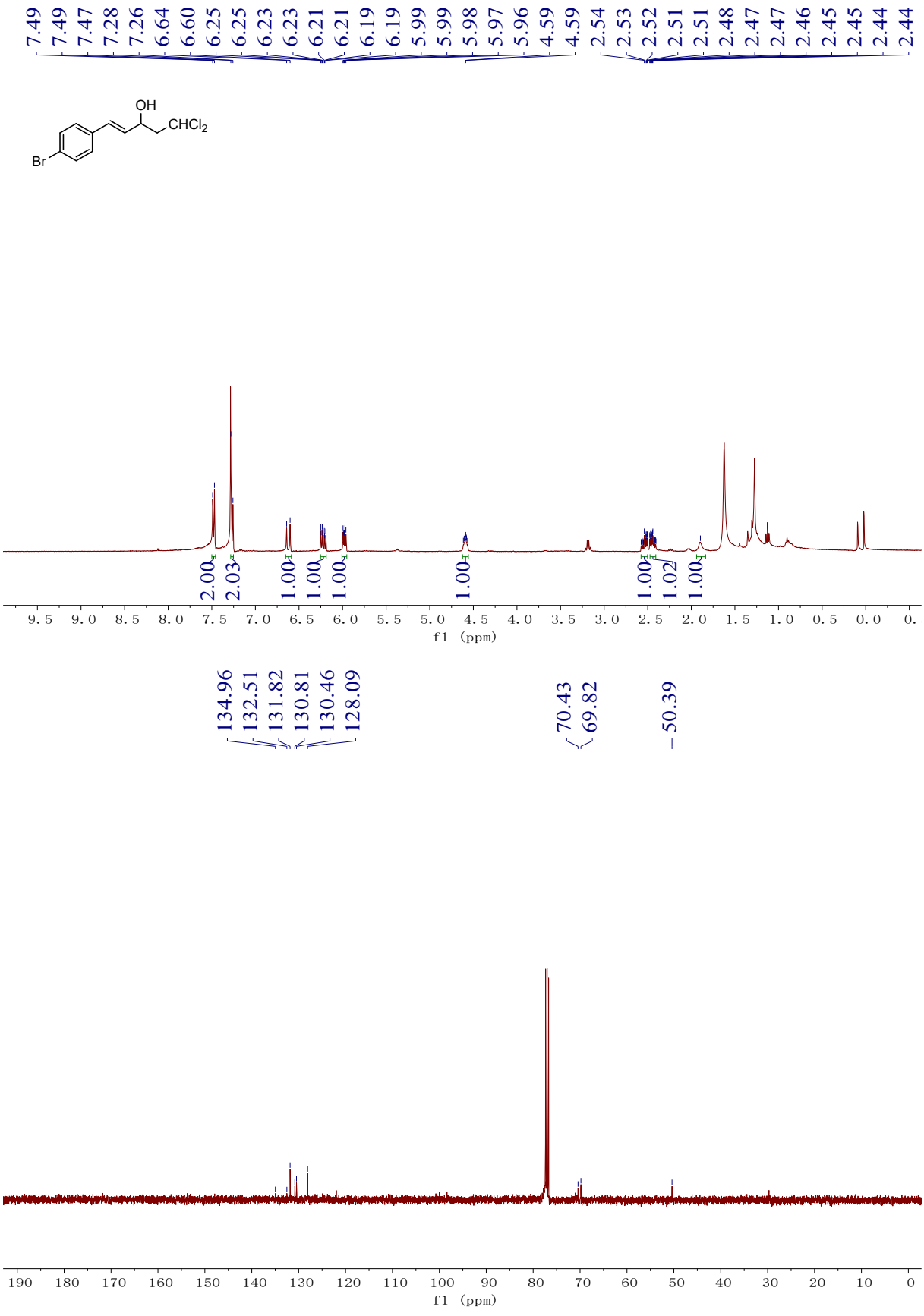
4f


7.39
7.38
7.38
7.37
7.36
7.35
7.07
7.07
7.06
7.04
7.03
7.02
6.66
6.62
6.17
6.15
6.13
6.11
5.99
5.98
5.97
5.96
4.60
4.58
2.57
2.55
2.53
2.52
2.51
2.48
2.47
2.45
2.44
2.43
2.42
OH
CHCl2
F
2.03
2.02
1.01
1.00
1.00
1.00
1.00
1.02
1.01
f1 (ppm)
163.84
161.37
132.20
132.16
130.57
129.78
129.76
128.20
128.12
115.76
115.55
70.50
69.91
50.49
f1 (ppm)

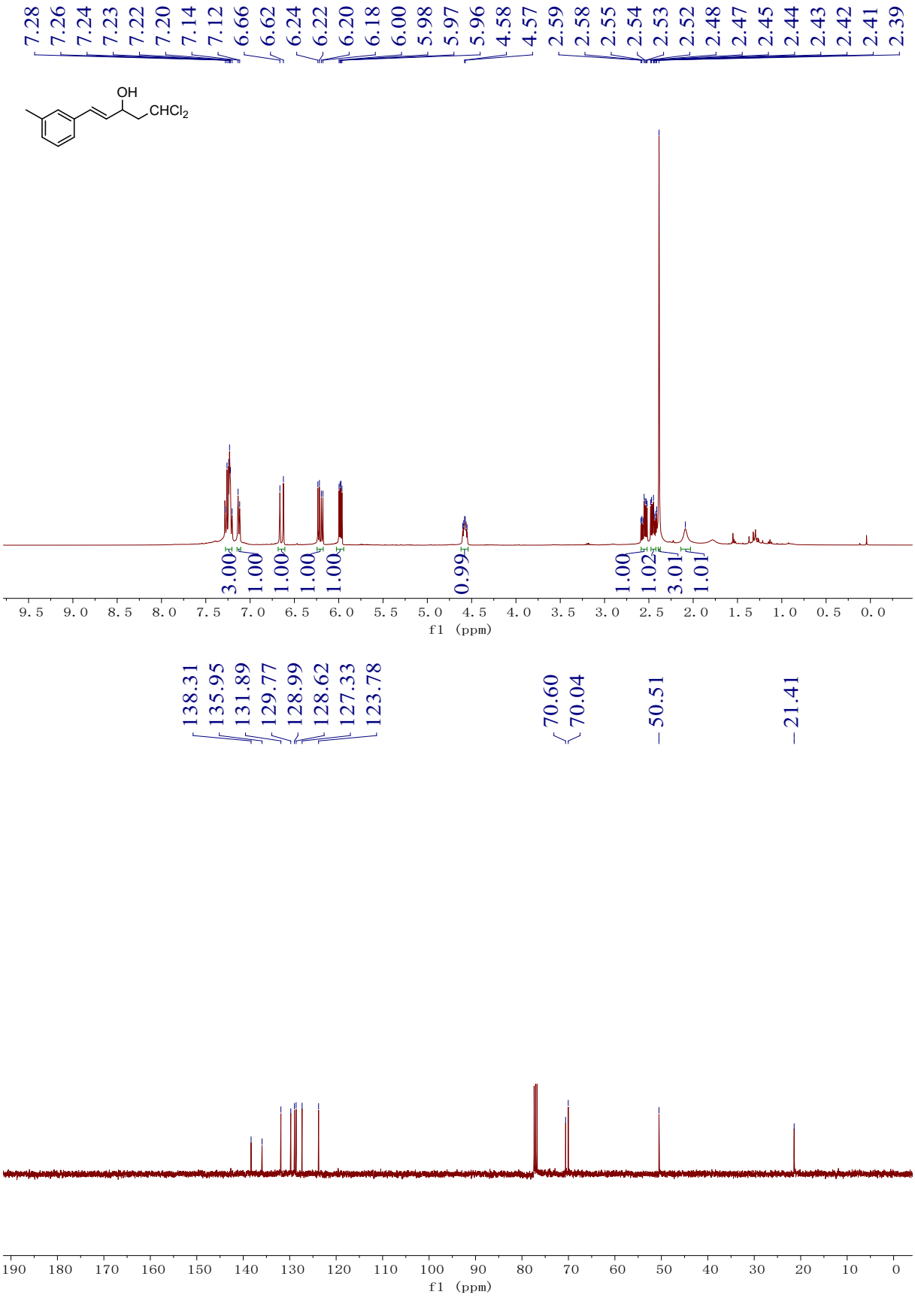
4g


7.33
6.66
6.66
6.62
6.62
6.23
6.22
6.19
6.18
5.99
5.98
5.97
5.96
4.62
4.61
4.59
4.58
4.57
2.58
2.57
2.55
2.54
2.53
2.52
2.51
2.48
2.47
2.46
2.45
2.44
2.43
2.42
OH
CHCl2
Cl
4.01
1.00
1.00
1.00
1.00
1.00
1.00
f1 (ppm)
134.52
133.83
130.68
130.44
128.88
127.79
70.44
69.83
50.42
f1 (ppm)

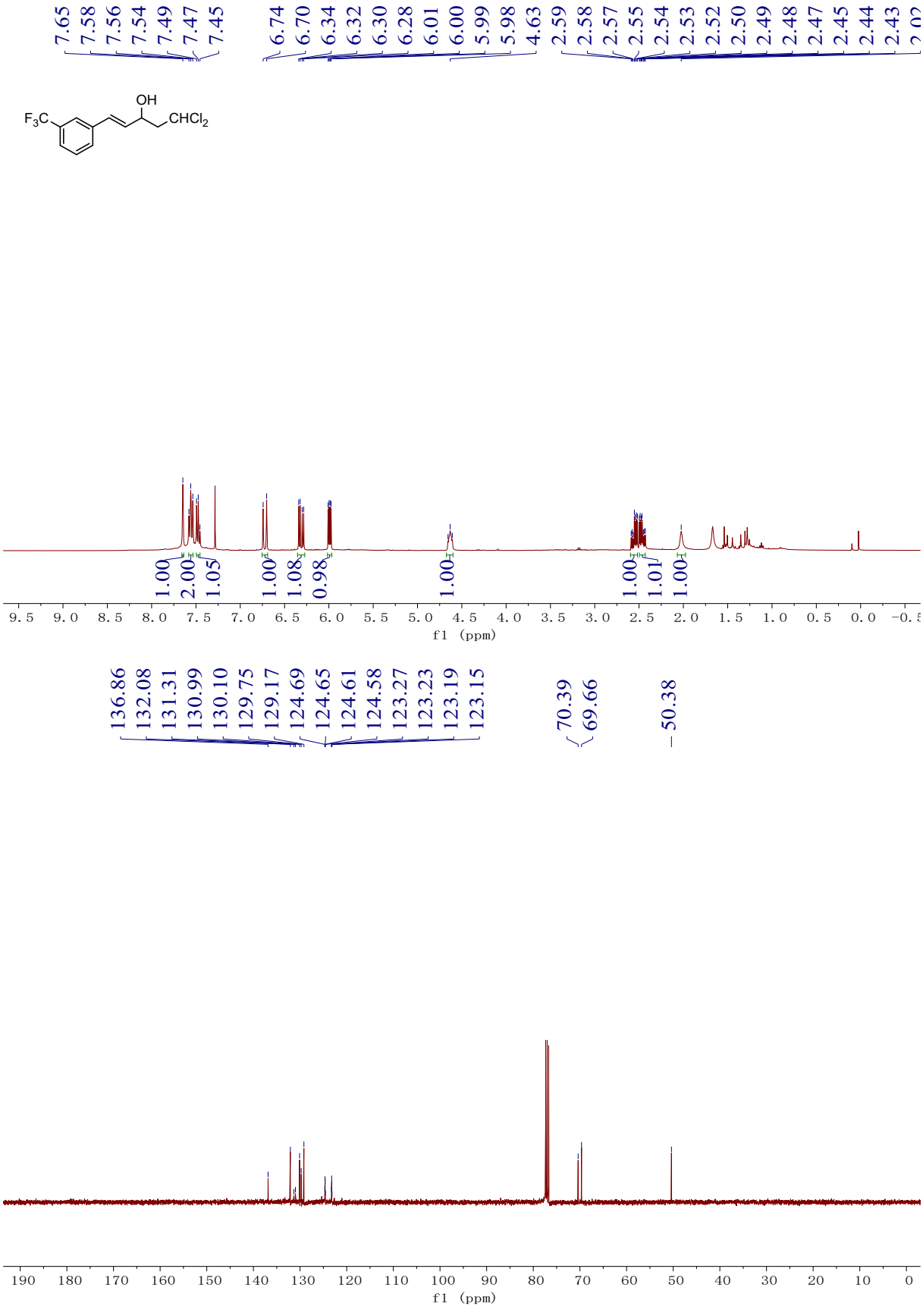
4h


7.49
7.49
7.47
7.28
7.26
6.64
6.60
6.25
6.25
6.23
6.23
6.21
6.21
6.19
6.19
5.99
5.99
5.98
5.97
5.96
4.59
4.59
2.54
2.53
2.52
2.51
2.51
2.48
2.47
2.47
2.46
2.45
2.45
2.44
2.44
OH
CHCl2
Br
2.00
2.03
1.00
1.00
1.00
1.00
1.00
1.02
1.00
f1 (ppm)
134.96
132.51
131.82
130.81
130.46
128.09
70.43
69.82
50.39
f1 (ppm)

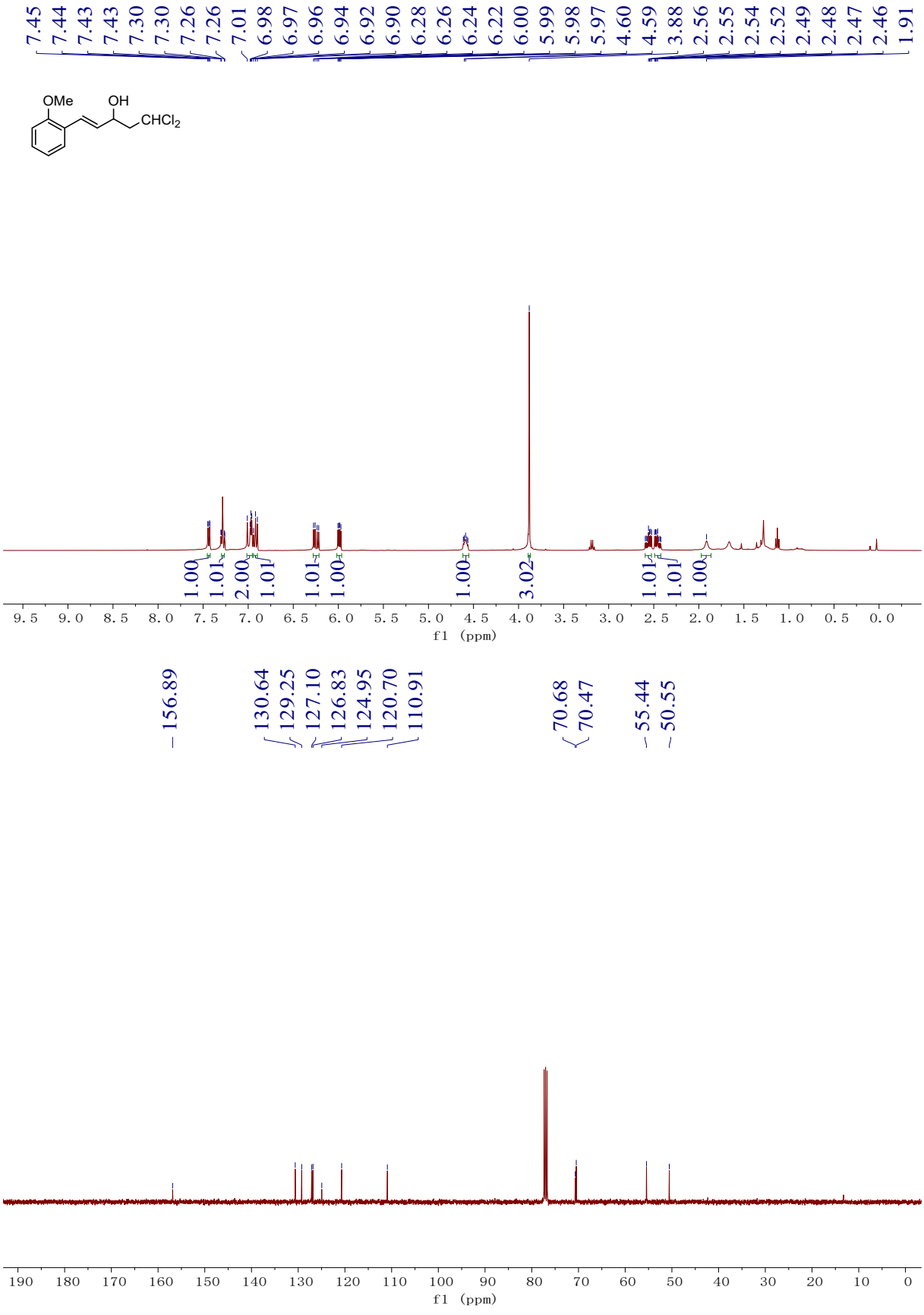
4i


7.28
7.26
7.24
7.23
7.22
7.20
7.14
7.12
6.66
6.62
6.24
6.22
6.20
6.18
6.00
5.98
5.97
5.96
4.58
4.57
2.59
2.58
2.55
2.54
2.53
2.52
2.48
2.47
2.45
2.44
2.43
2.42
2.41
2.39
OH
CHCl2
3.00
1.00
1.00
1.00
1.00
0.99
1.00
1.02
3.01
1.01
f1 (ppm)
138.31
135.95
131.89
129.77
128.99
128.62
127.33
123.78
70.60
70.04
50.51
21.41
f1 (ppm)

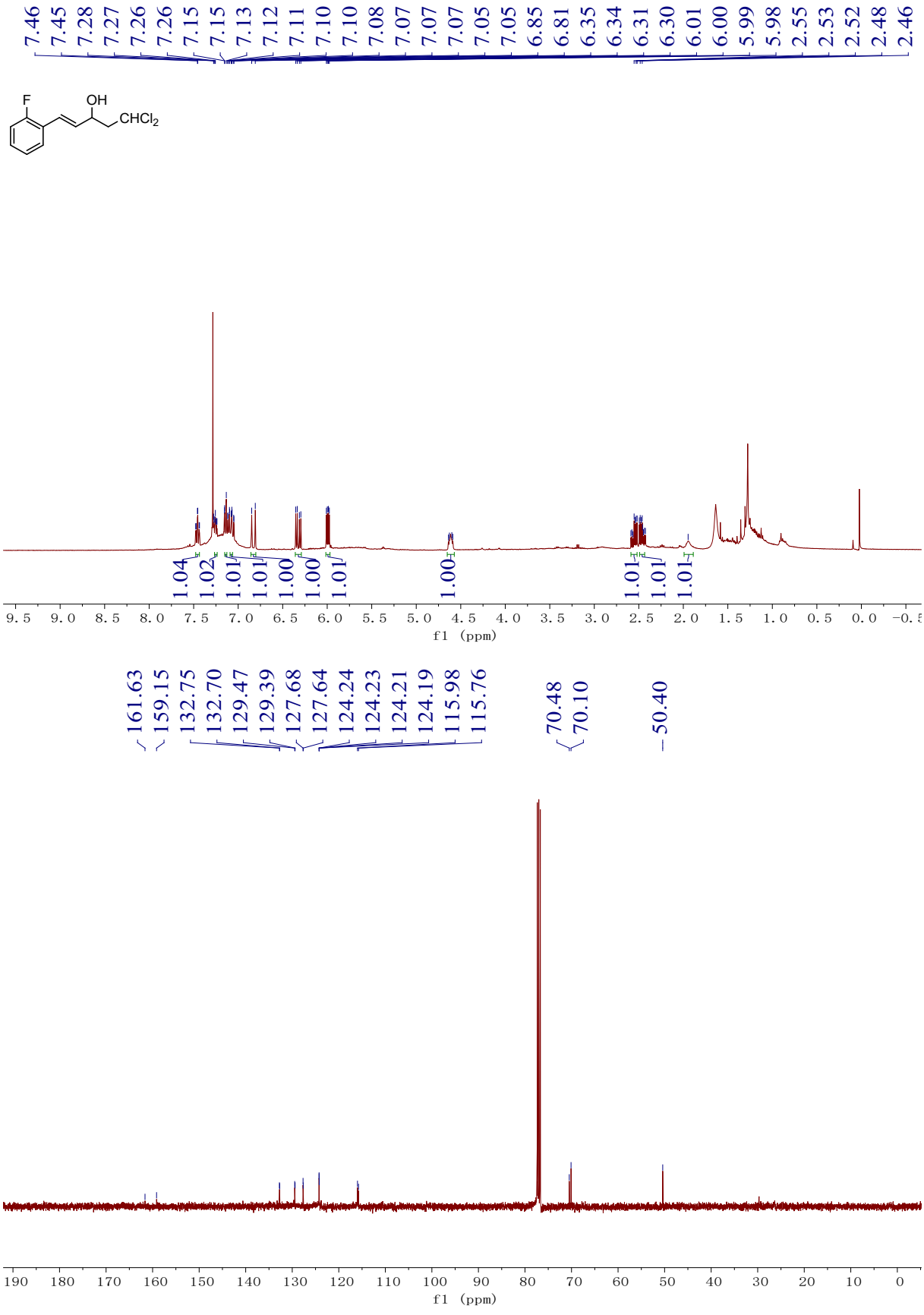
4j


7.65
7.58
7.56
7.54
7.49
7.47
7.45
6.74
6.70
6.34
6.32
6.30
6.28
6.01
6.00
5.99
5.98
4.63
2.59
2.58
2.57
2.55
2.54
2.53
2.52
2.50
2.49
2.48
2.47
2.45
2.44
2.43
OH
F3C
CHCl2
1.00
2.00
1.05
1.00
1.08
0.98
1.00
1.00
1.01
1.00
f1 (ppm)
136.86
132.08
131.31
130.99
130.10
129.75
129.17
124.69
124.65
124.61
124.58
123.27
123.23
123.19
123.15
70.39
69.66
50.38
f1 (ppm)

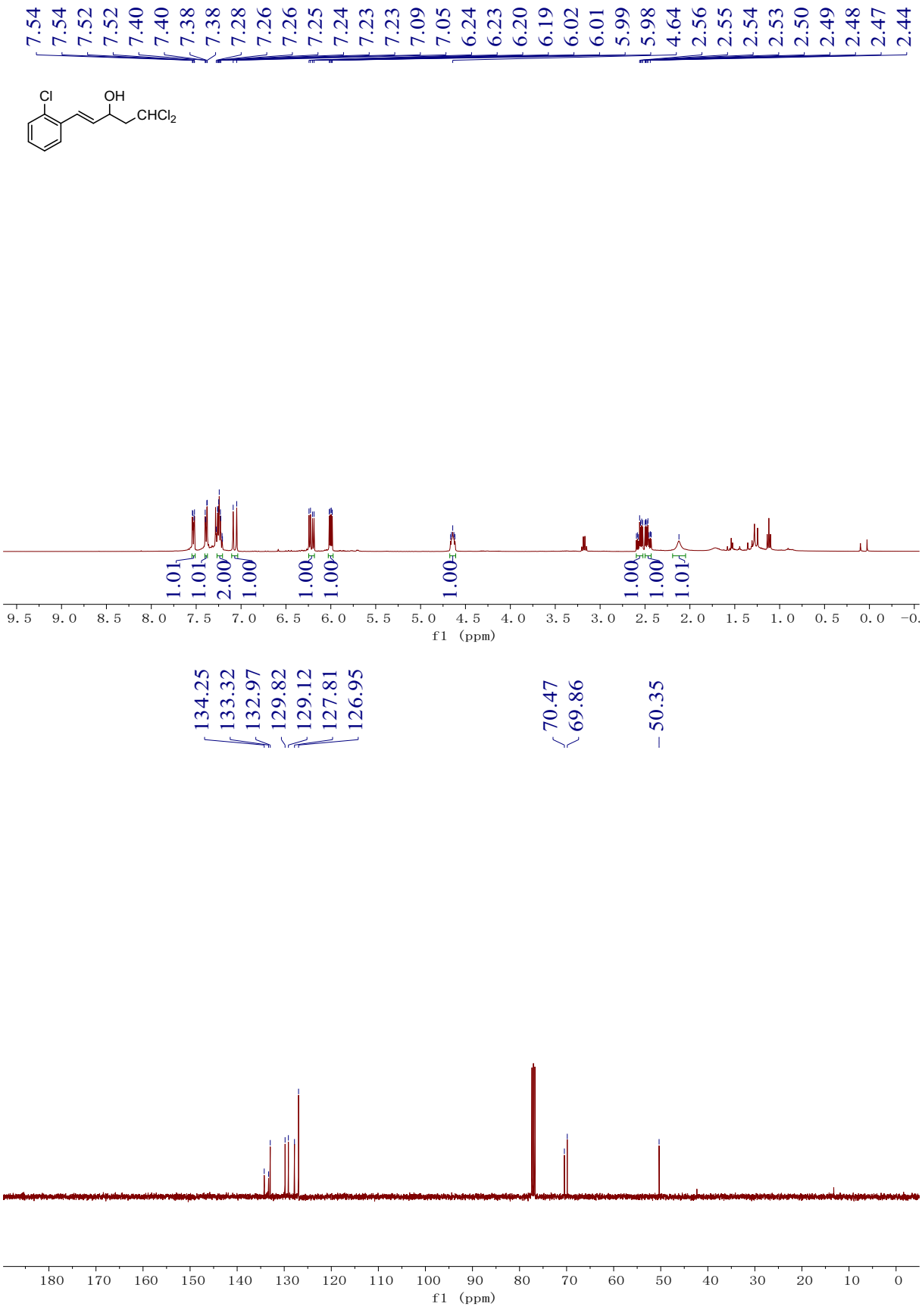
4k


7.45
7.44
7.43
7.43
7.30
7.30
7.26
7.26
7.01
6.98
6.97
6.96
6.94
6.92
6.90
6.28
6.26
6.24
6.22
6.00
5.99
5.98
5.97
4.60
4.59
3.88
2.56
2.55
2.54
2.52
2.49
2.48
2.47
2.46
1.91
OMe
OH
CHCl2
1.00
1.01
2.00
1.01
1.01
1.00
1.00
3.02
1.01
1.01
1.00
f1 (ppm)
156.89
130.64
129.25
127.10
126.83
124.95
120.70
110.91
70.68
70.47
55.44
50.55
f1 (ppm)

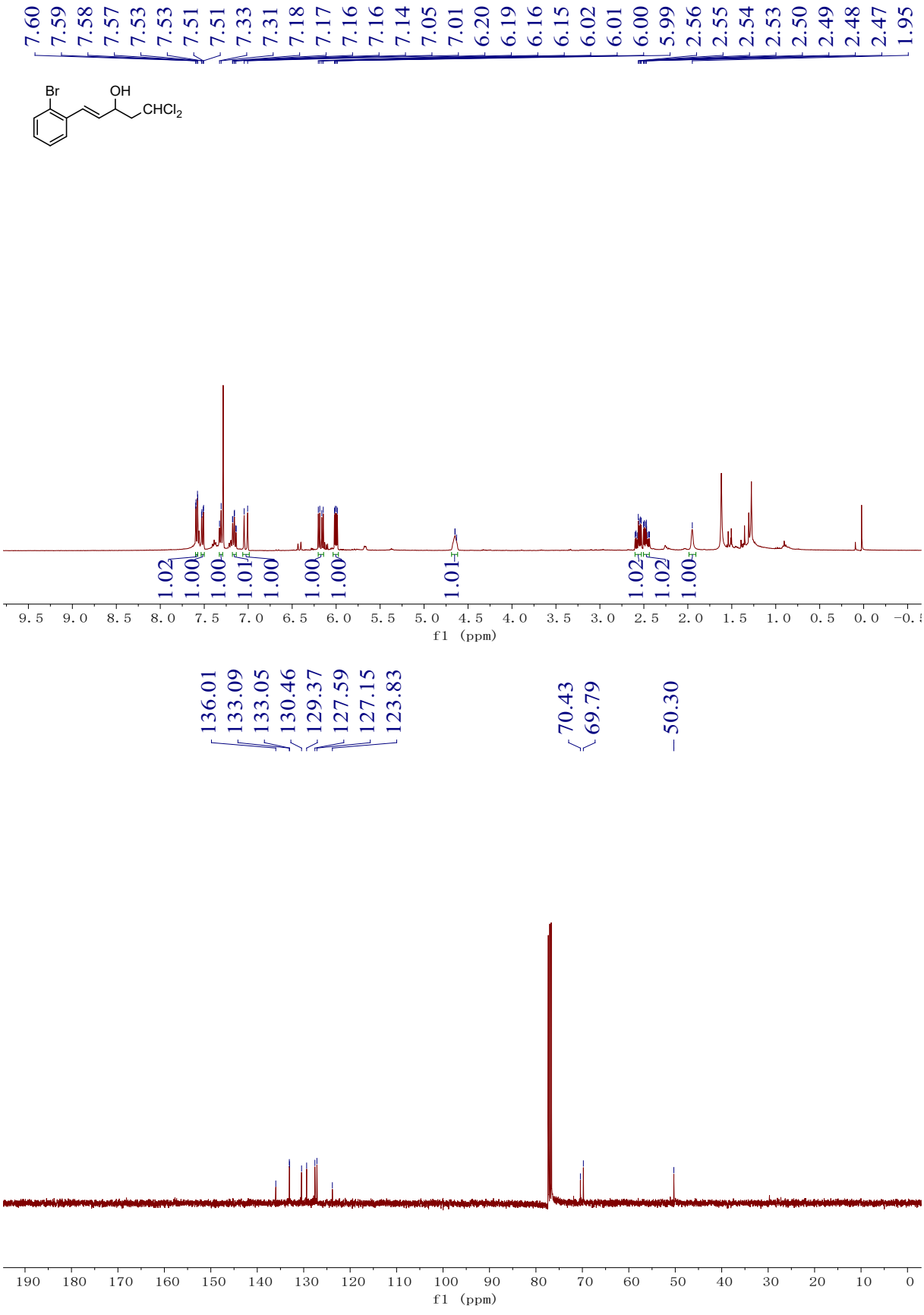
4l


7.46
7.45
7.28
7.27
7.26
7.26
7.15
7.15
7.13
7.12
7.11
7.10
7.10
7.08
7.07
7.07
7.07
7.05
7.05
6.85
6.81
6.35
6.34
6.31
6.30
6.01
6.00
5.99
5.98
2.55
2.53
2.52
2.48
2.46
F
OH
CHCl2
1.04
1.02
1.01
1.01
1.00
1.00
1.01
1.00
1.01
1.01
1.01
f1 (ppm)
161.63
159.15
132.75
132.70
129.47
129.39
127.68
127.64
124.24
124.23
124.21
124.19
115.98
115.76
70.48
70.10
50.40
f1 (ppm)

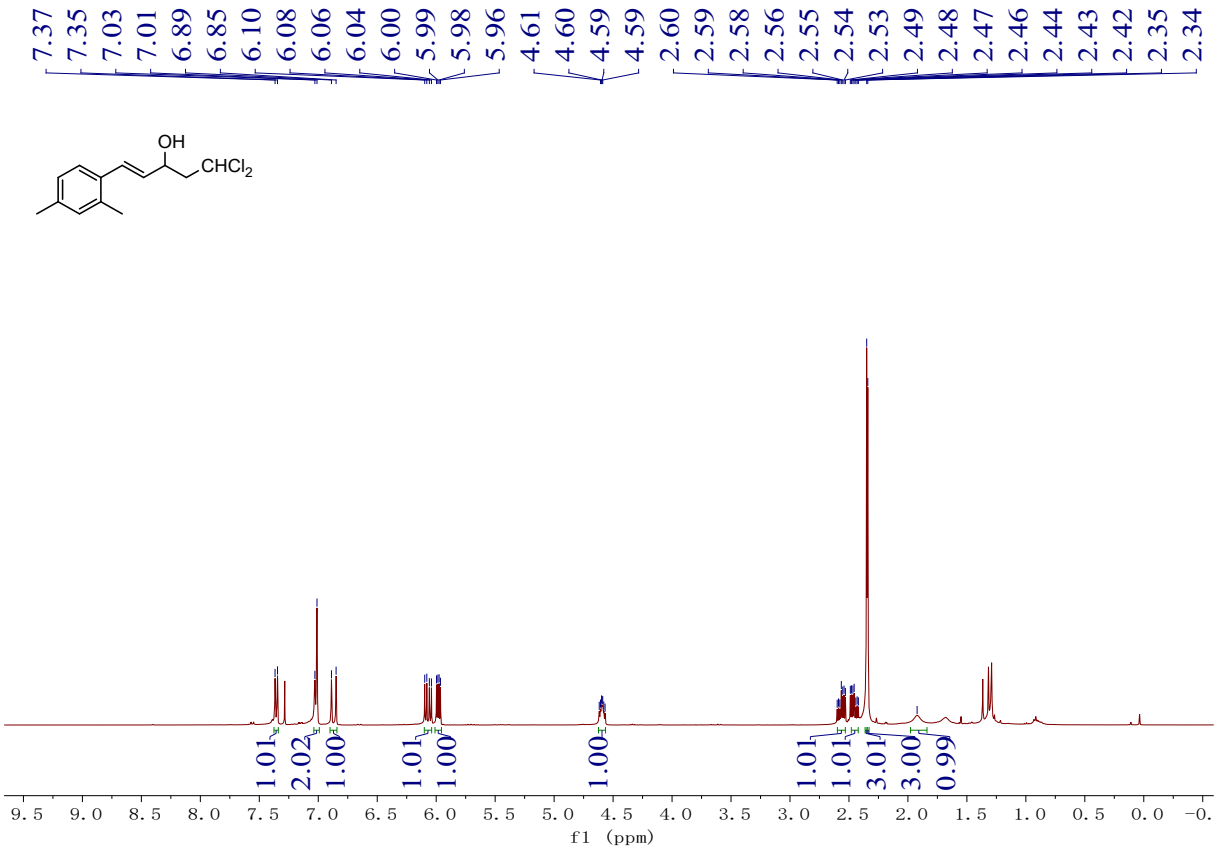
4m

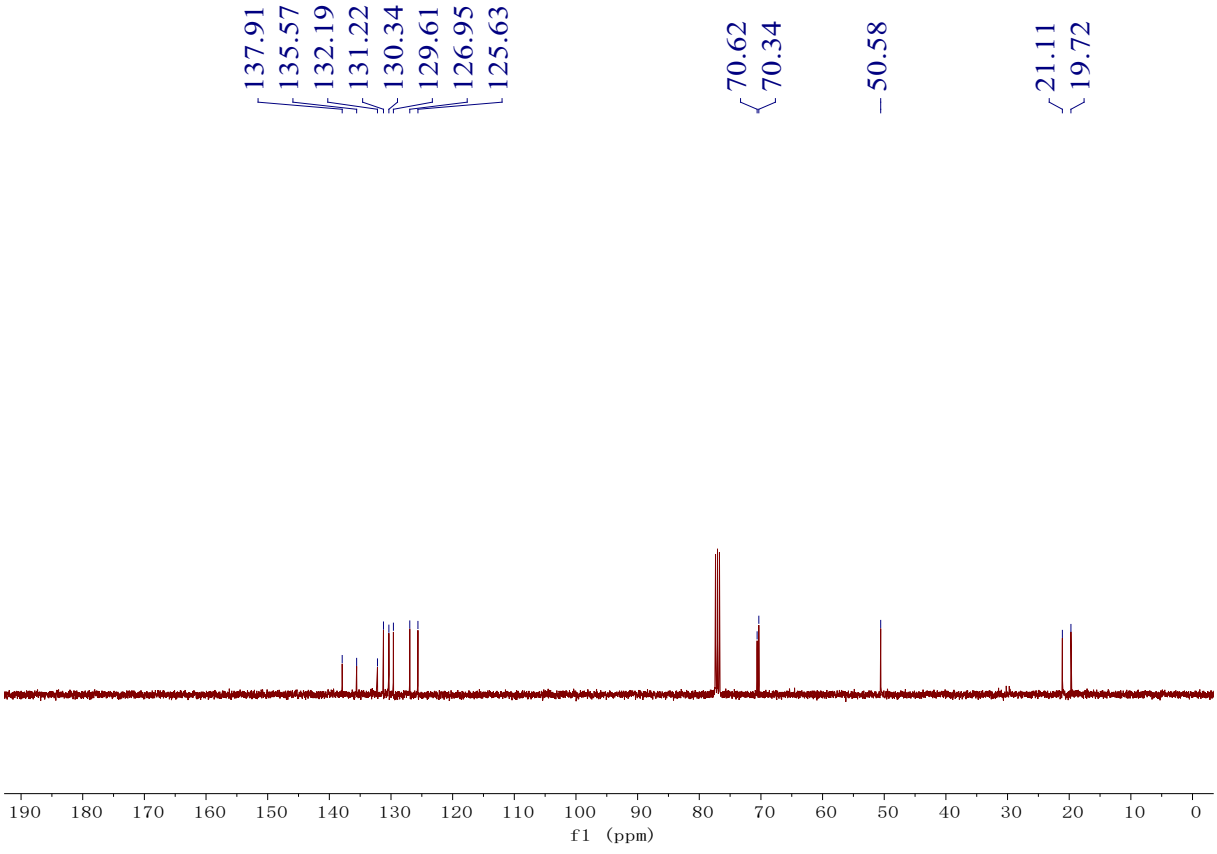

7.54
7.54
7.52
7.52
7.40
7.40
7.38
7.38
7.28
7.26
7.26
7.25
7.24
7.23
7.23
7.09
7.05
6.24
6.23
6.20
6.19
6.02
6.01
5.99
5.98
4.64
2.56
2.55
2.54
2.53
2.50
2.49
2.48
2.47
2.44
Cl
OH
CHCl2
1.01
1.01
2.00
1.00
1.00
1.00
1.00
1.00
1.00
1.01
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.
f1 (ppm)
134.25
133.32
132.97
129.82
129.12
127.81
126.95
70.47
69.86
50.35
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

4n

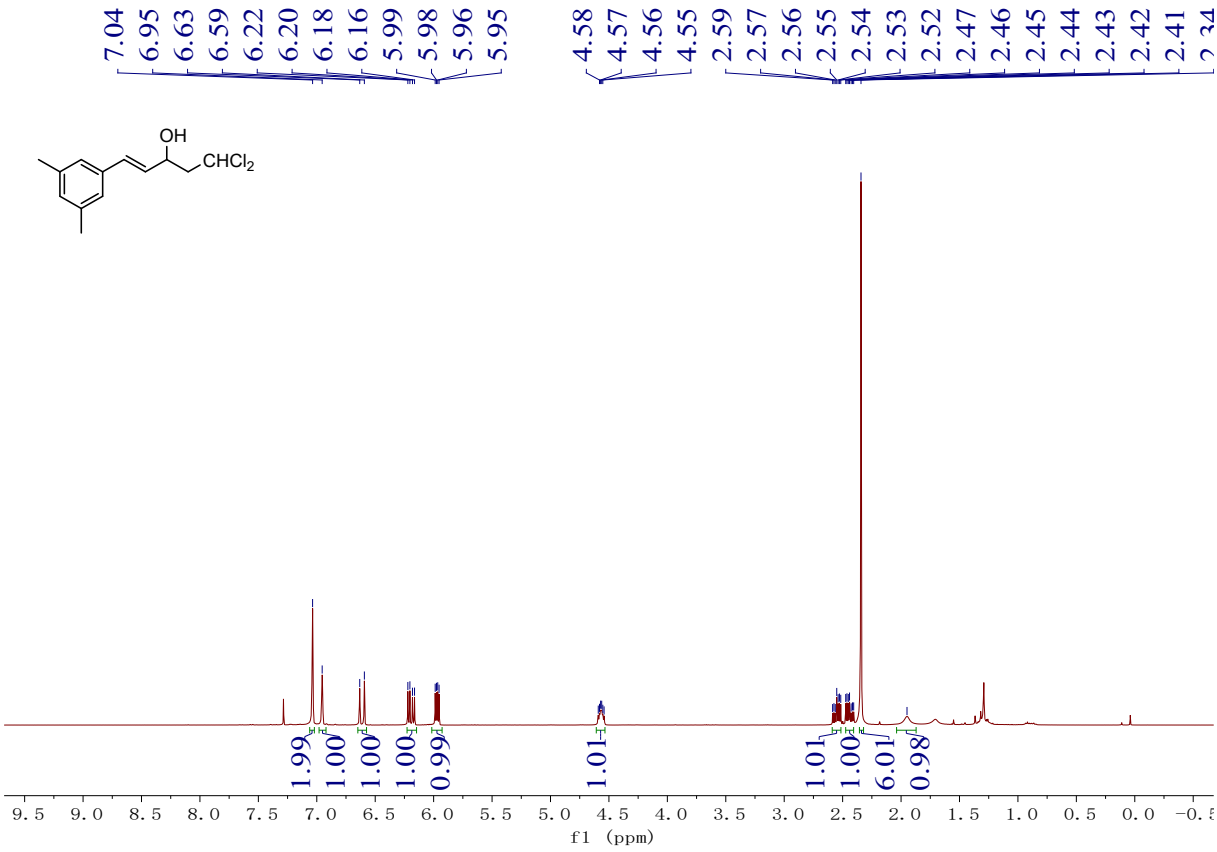

7.60
7.59
7.58
7.57
7.53
7.53
7.51
7.51
7.33
7.31
7.18
7.17
7.16
7.16
7.14
7.05
7.01
6.20
6.19
6.16
6.15
6.02
6.01
6.00
5.99
2.56
2.55
2.54
2.53
2.50
2.49
2.48
2.47
1.95
Br
OH
CHCl2
1.02
1.00
1.00
1.01
1.00
1.00
1.00
1.01
1.02
1.02
1.00
f1 (ppm)
136.01
133.09
133.05
130.46
129.37
127.59
127.15
123.83
70.43
69.79
50.30
f1 (ppm)

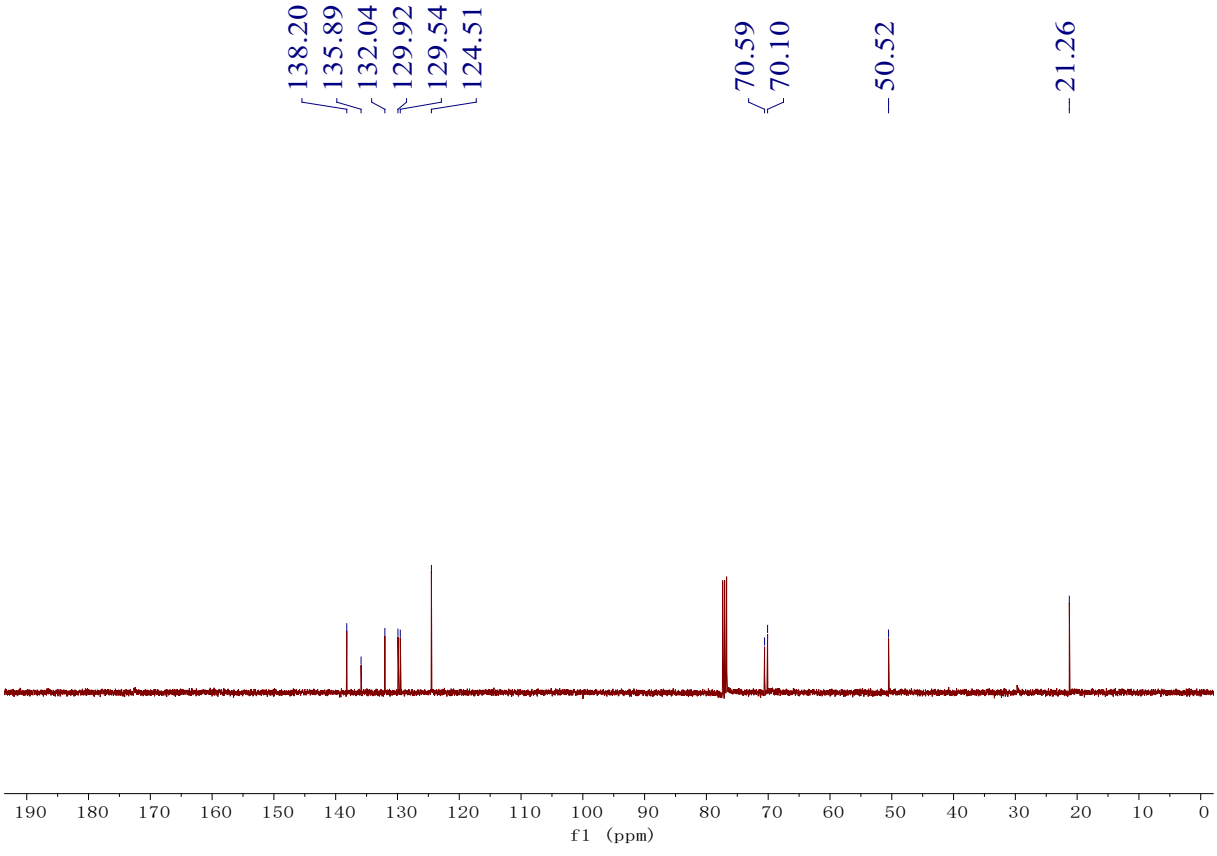
4o


7.37
7.35
7.03
7.01
6.89
6.85
6.10
6.08
6.06
6.04
6.00
5.99
5.98
5.96
4.61
4.60
4.59
4.59
2.60
2.59
2.58
2.56
2.55
2.54
2.53
2.49
2.48
2.47
2.46
2.44
2.43
2.42
2.35
2.34
OH
CHCl2
1.01
2.02
1.00
1.01
1.00
1.00
1.01
1.01
3.01
3.00
0.99
f1 (ppm)

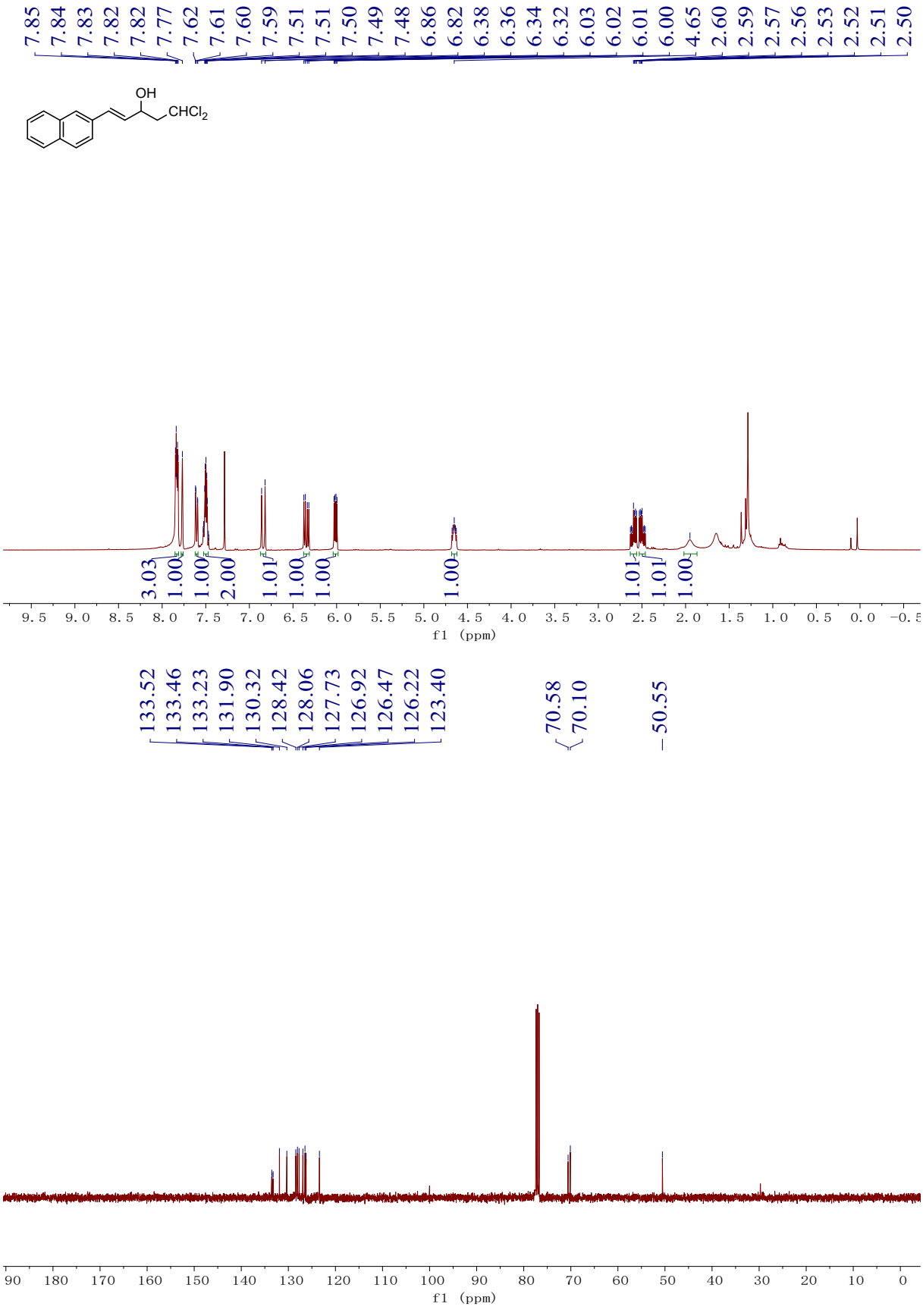

137.91
135.57
132.19
131.22
130.34
129.61
126.95
125.63
70.62
70.34
50.58
21.11
19.72
f1 (ppm)

4p

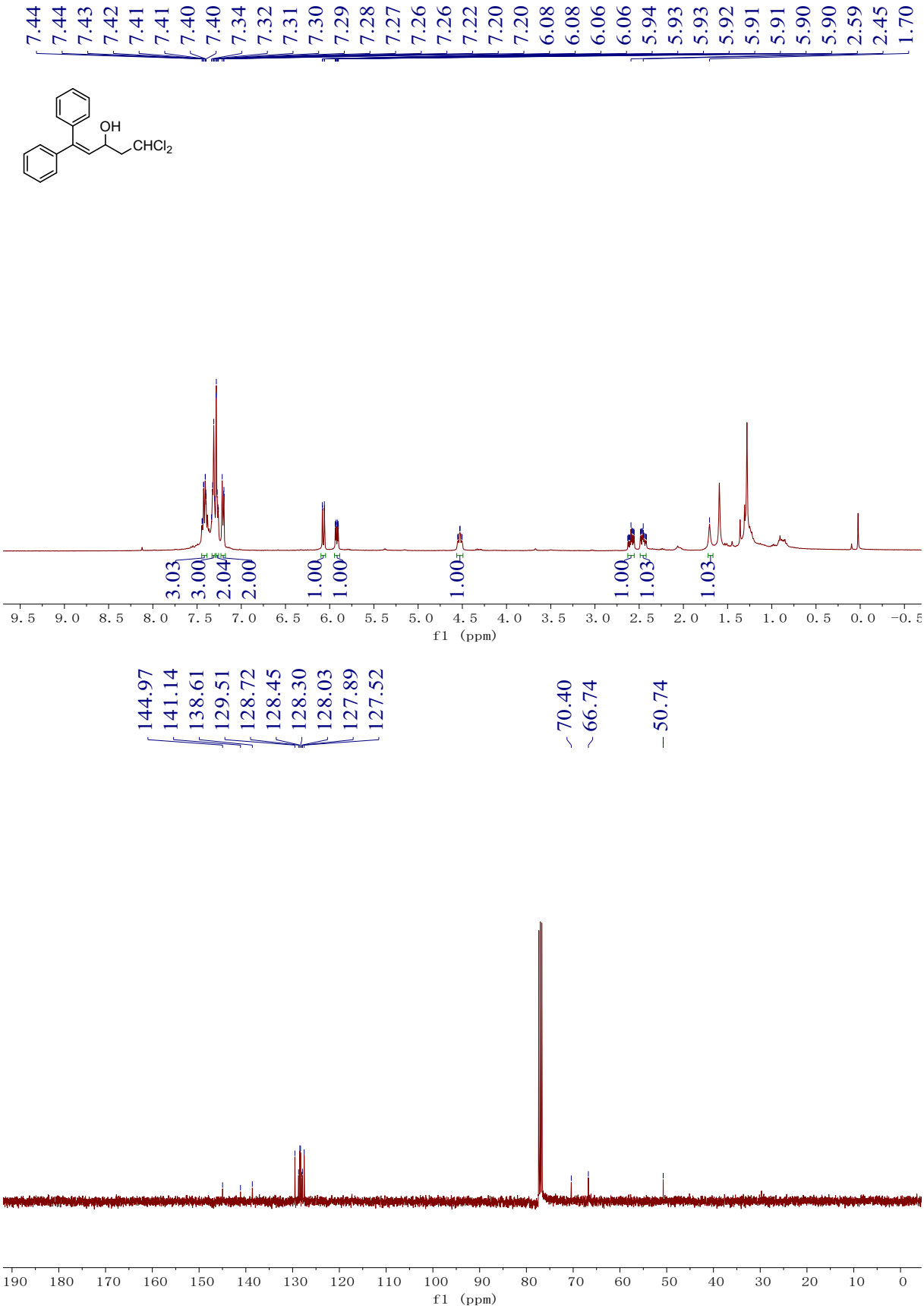

7.04
6.95
6.63
6.59
6.22
6.20
6.18
6.16
5.99
5.98
5.96
5.95
4.58
4.57
4.56
4.55
2.59
2.57
2.56
2.55
2.54
2.53
2.52
2.47
2.46
2.45
2.44
2.43
2.42
2.41
OH
CHCl2
1.99
1.00
1.00
1.00
0.99
1.01
1.01
1.00
6.01
0.98
f1 (ppm)


138.20
135.89
132.04
129.92
129.54
124.51
70.59
70.10
50.52
21.26
f1 (ppm)

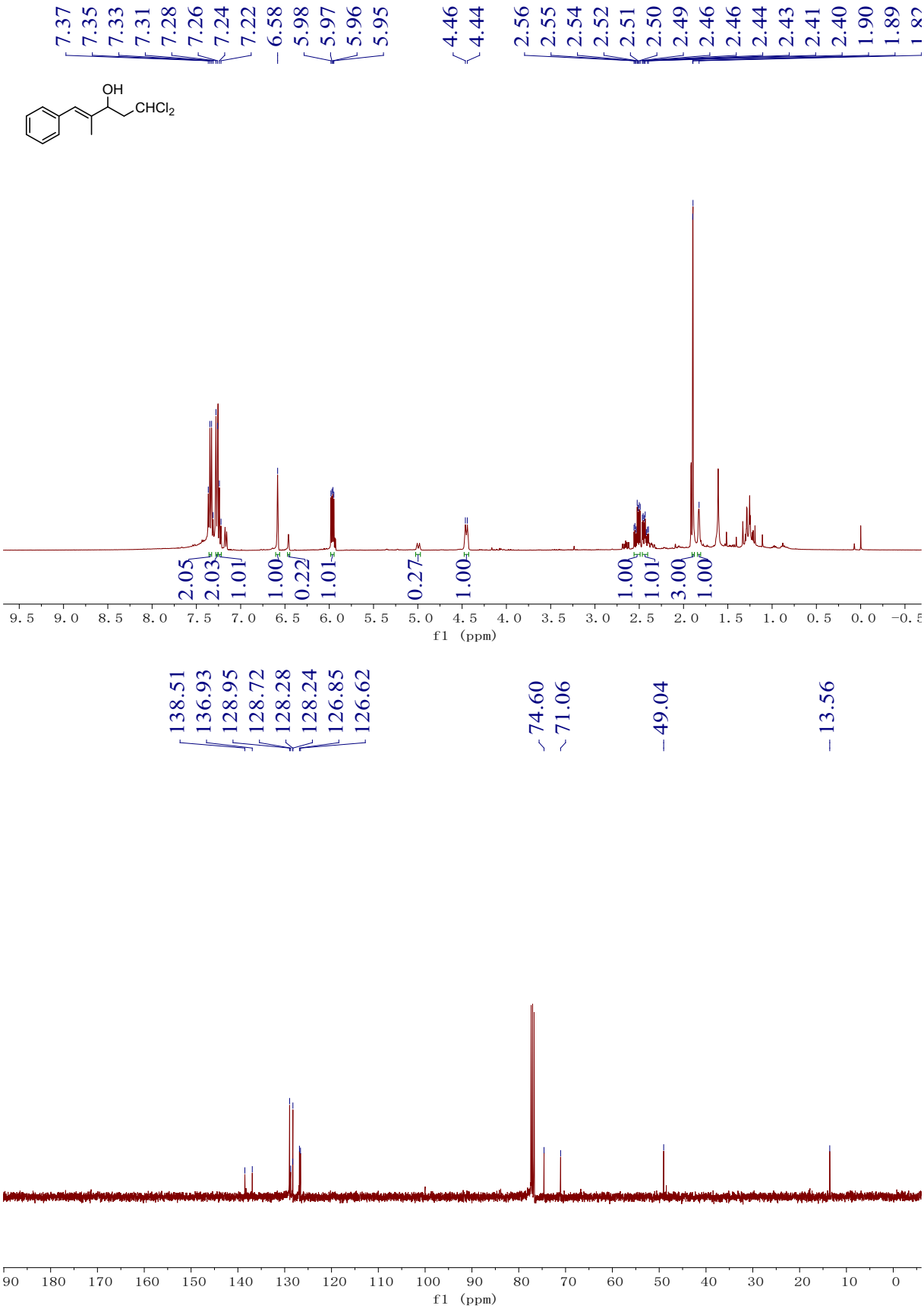
4q


7.85
7.84
7.83
7.82
7.82
7.77
7.62
7.61
7.60
7.59
7.51
7.51
7.50
7.49
7.48
6.86
6.82
6.38
6.36
6.34
6.32
6.03
6.02
6.01
6.00
4.65
2.60
2.59
2.57
2.56
2.53
2.52
2.51
2.50
OH
CHCl2
3.03
1.00
1.00
2.00
1.01
1.00
1.00
1.00
1.01
1.01
1.00
f1 (ppm)
133.52
133.46
133.23
131.90
130.32
128.42
128.06
127.73
126.92
126.47
126.22
123.40
70.58
70.10
50.55
f1 (ppm)

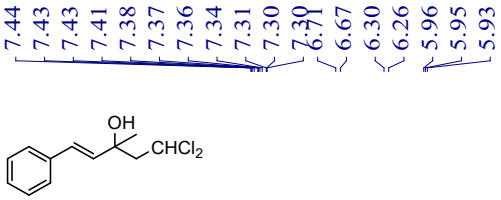
4r

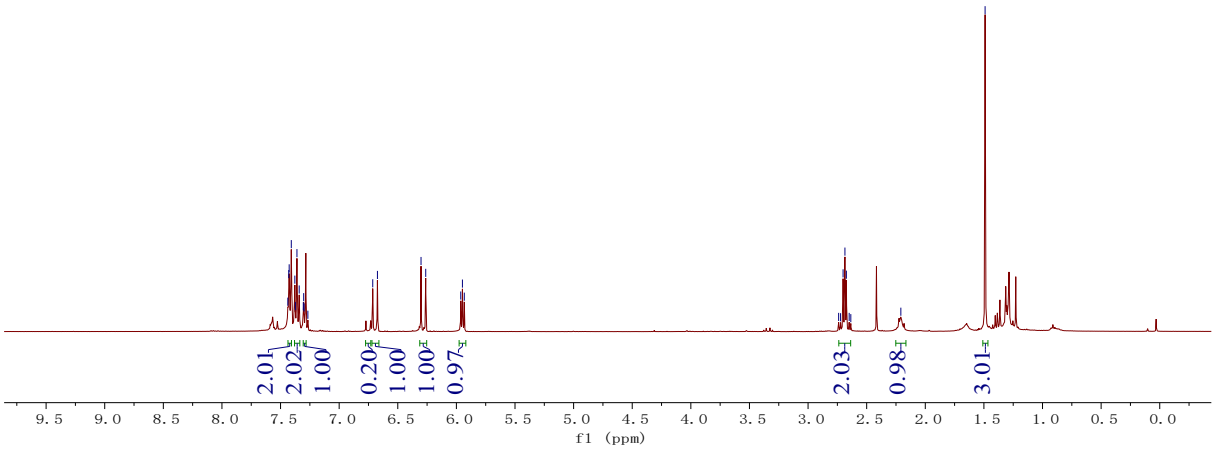

7.44
7.44
7.43
7.42
7.41
7.41
7.40
7.40
7.34
7.32
7.31
7.30
7.29
7.28
7.27
7.26
7.26
7.22
7.20
7.20
6.08
6.08
6.06
6.06
5.94
5.93
5.93
5.92
5.91
5.91
5.90
5.90
2.59
2.45
1.70
OH
CHCl2
3.03
3.00
2.04
2.00
1.00
1.00
1.00
1.00
1.03
1.03
f1 (ppm)
144.97
141.14
138.61
129.51
128.72
128.45
128.30
128.03
127.89
127.52
70.40
66.74
50.74
f1 (ppm)

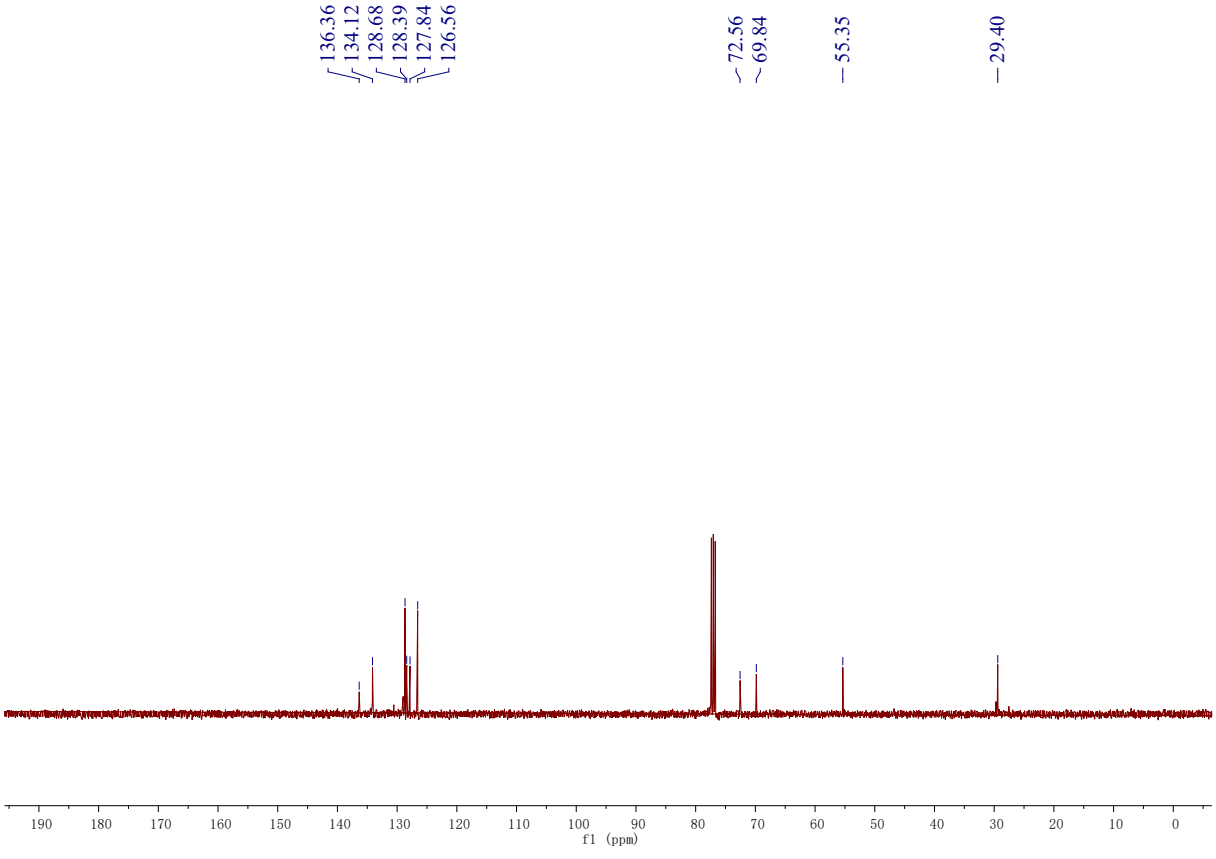
4s


7.37
7.35
7.33
7.31
7.28
7.26
7.24
7.22
6.58
5.98
5.97
5.96
5.95
4.46
4.44
2.56
2.55
2.54
2.52
2.51
2.50
2.49
2.46
2.46
2.44
2.43
2.41
2.40
1.90
1.89
OH
CHCl2
2.05
2.03
1.01
1.00
0.22
1.01
0.27
1.00
1.00
1.01
3.00
1.00
f1 (ppm)
138.51
136.93
128.95
128.72
128.28
128.24
126.85
126.62
74.60
71.06
49.04
13.56
f1 (ppm)

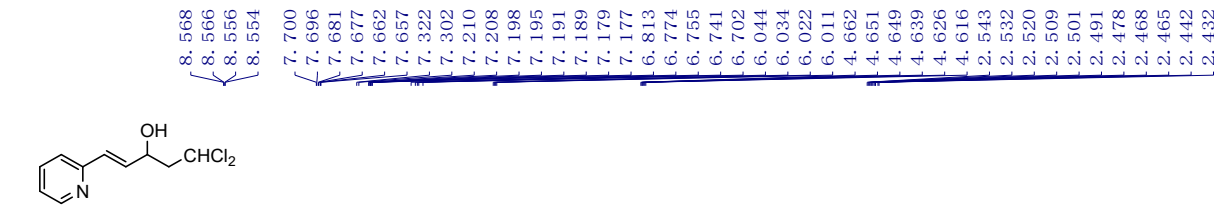
4t


7.44
7.43
7.43
7.41
7.38
7.37
7.36
7.34
7.31
7.30
7.30
6.71
6.67
6.30
6.26
5.96
5.95
5.93
2.74
2.72
2.70
2.69
2.67
2.65
2.64
2.21
1.49
OH
CHCl2

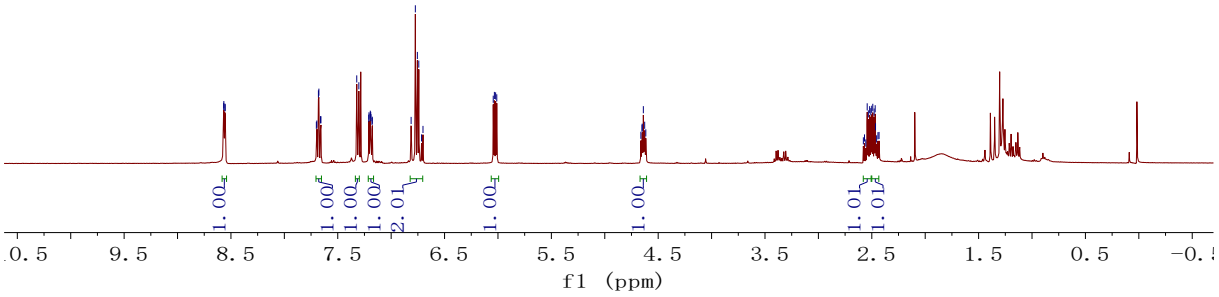

2.01
2.02
1.00
0.20
1.00
1.00
0.97
2.03
0.98
3.01
f1 (ppm)

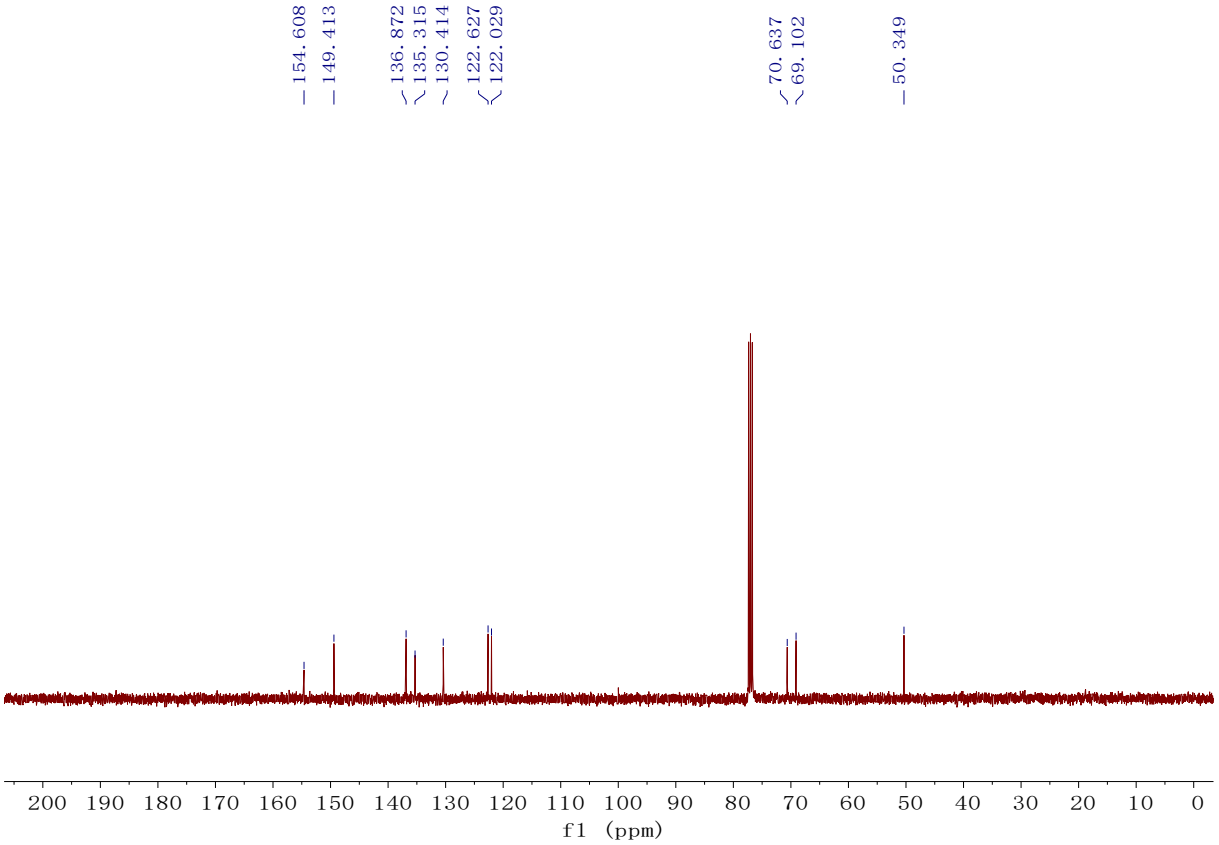

136.36
134.12
128.68
128.39
127.84
126.56
72.56
69.84
55.35
29.40
f1 (ppm)

4w

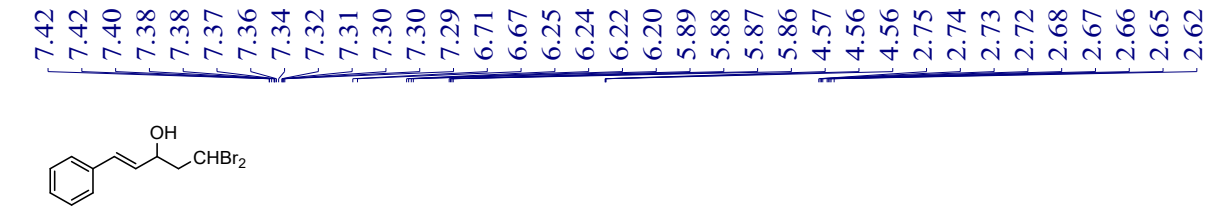

8.568
8.566
8.556
8.554
7.700
7.696
7.681
7.677
7.662
7.657
7.322
7.302
7.210
7.208
7.198
7.195
7.191
7.189
7.179
7.177
6.813
6.774
6.755
6.741
6.702
6.044
6.034
6.022
6.011
4.662
4.651
4.649
4.639
4.626
4.616
2.543
2.532
2.520
2.509
2.501
2.491
2.478
2.468
2.465
2.442
2.432

OH
CHCl2
N

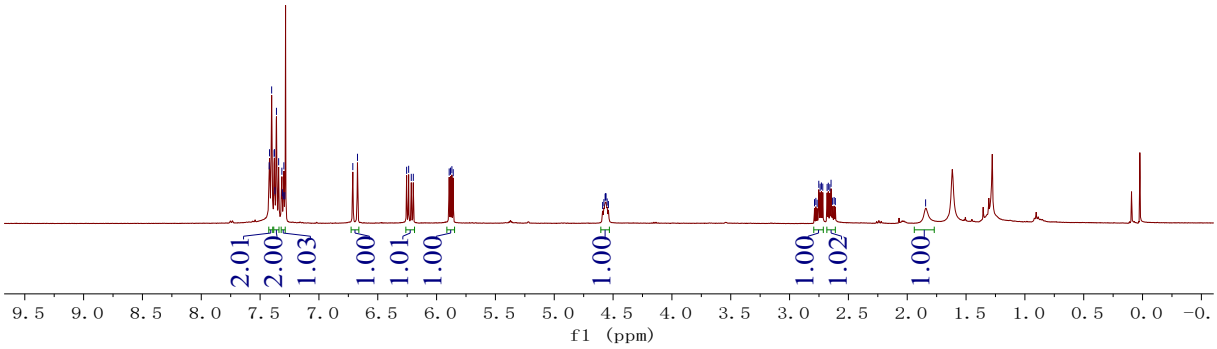

1.00
1.00
1.00
1.00
2.01
1.00
1.00
1.01
1.01
f1 (ppm)


154.608
149.413
136.872
135.315
130.414
122.627
122.029
70.637
69.102
50.349
f1 (ppm)

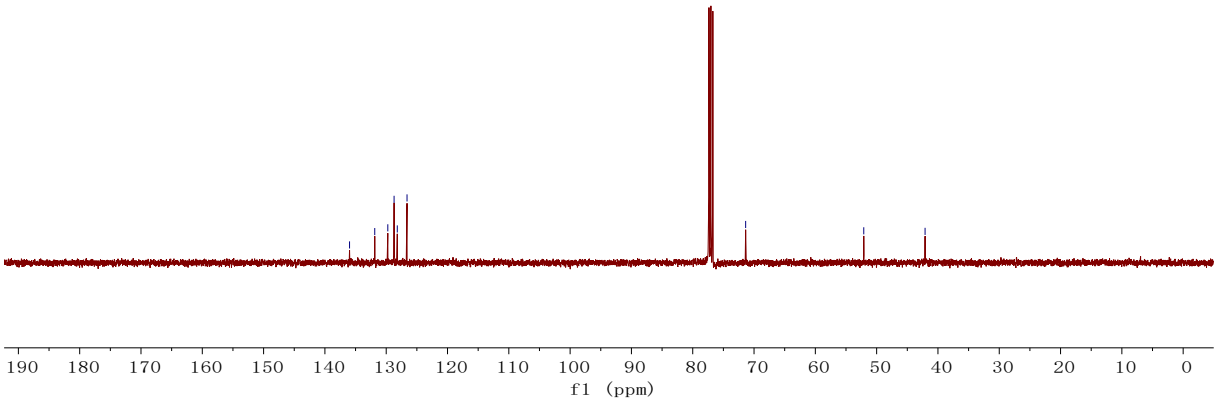
4x


7.42
7.42
7.40
7.38
7.38
7.37
7.36
7.34
7.32
7.31
7.30
7.30
7.29
6.71
6.67
6.25
6.24
6.22
6.20
5.89
5.88
5.87
5.86
4.57
4.56
4.56
2.75
2.74
2.73
2.72
2.68
2.67
2.66
2.65
2.62

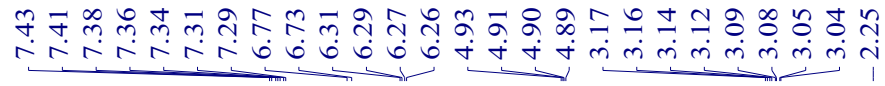
OH
CHBr2

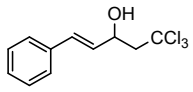

2.01
2.00
1.03
1.00
1.01
1.00
1.00
1.00
1.02
1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.
f1 (ppm)

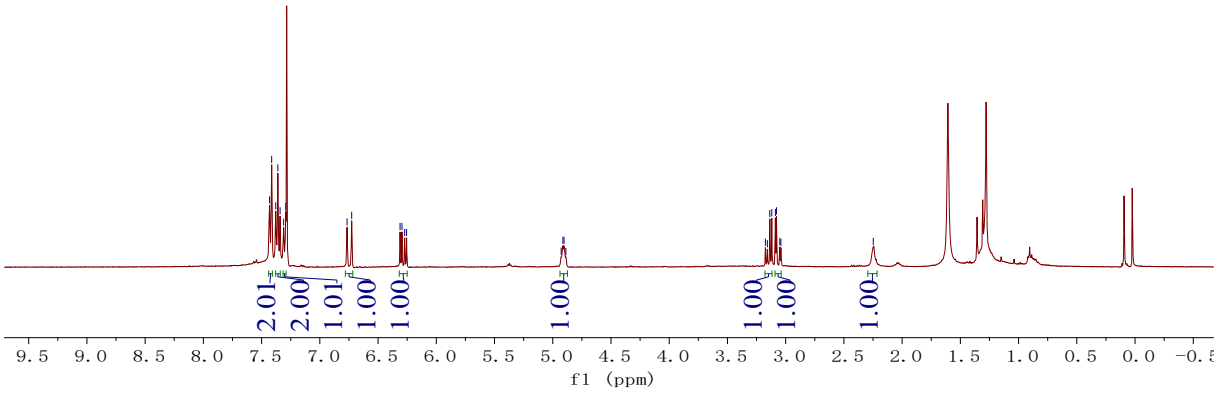

135.98
131.87
129.74
128.72
128.19
126.60
71.37
52.10
42.10

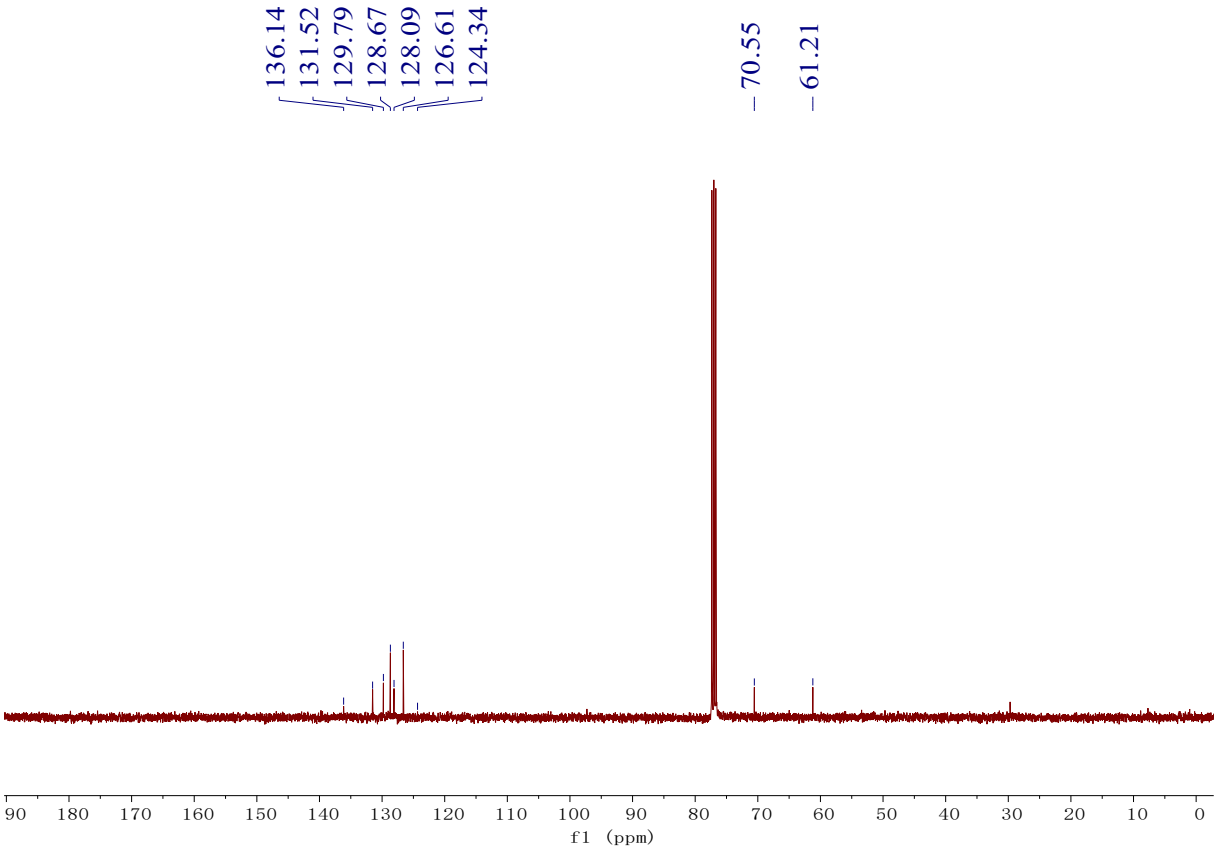

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

4y

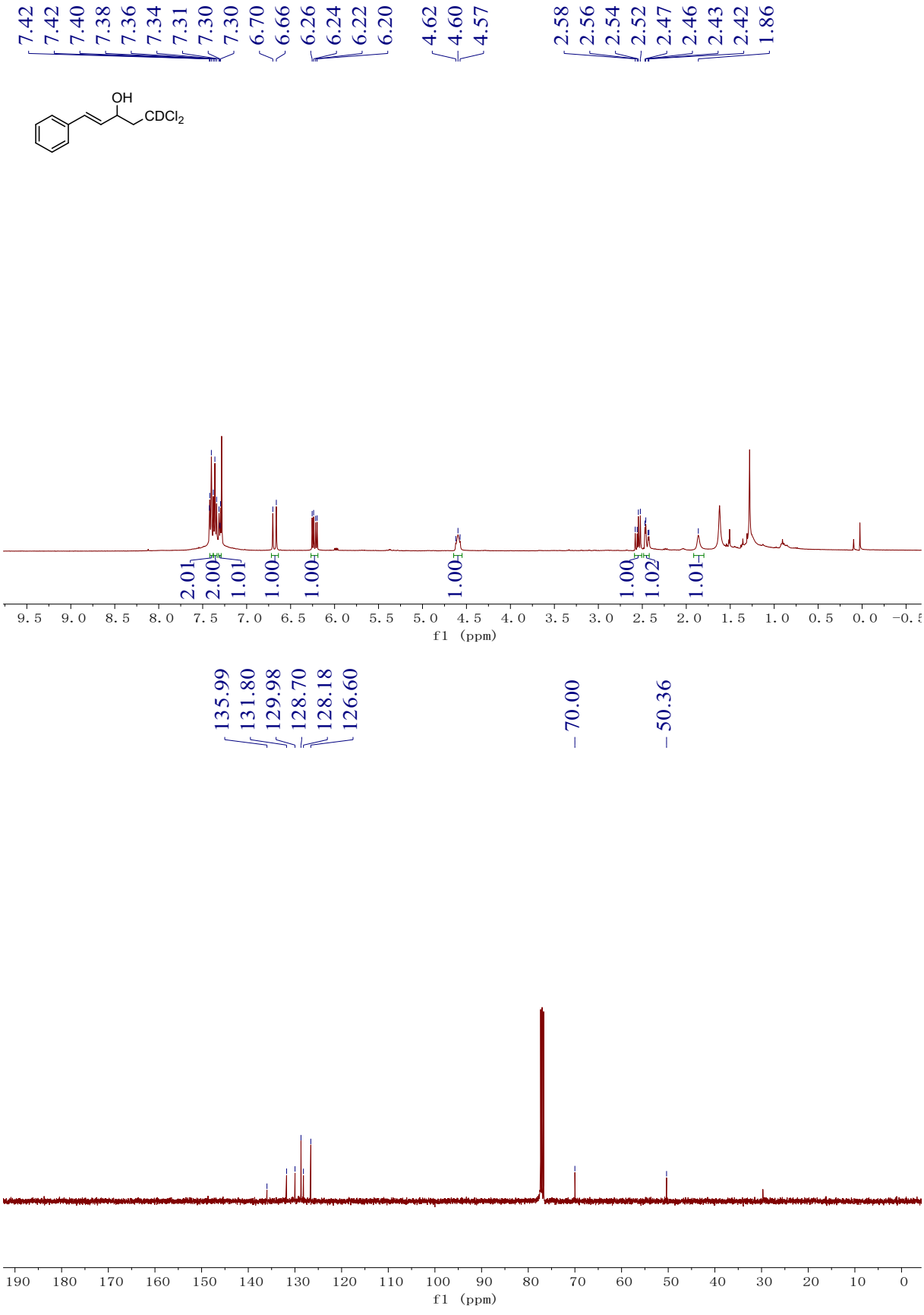

7.43
7.41
7.38
7.36
7.34
7.31
7.29
6.77
6.73
6.31
6.29
6.27
6.26
4.93
4.91
4.90
4.89
3.17
3.16
3.14
3.12
3.09
3.08
3.05
3.04
2.25


OH
CCl3

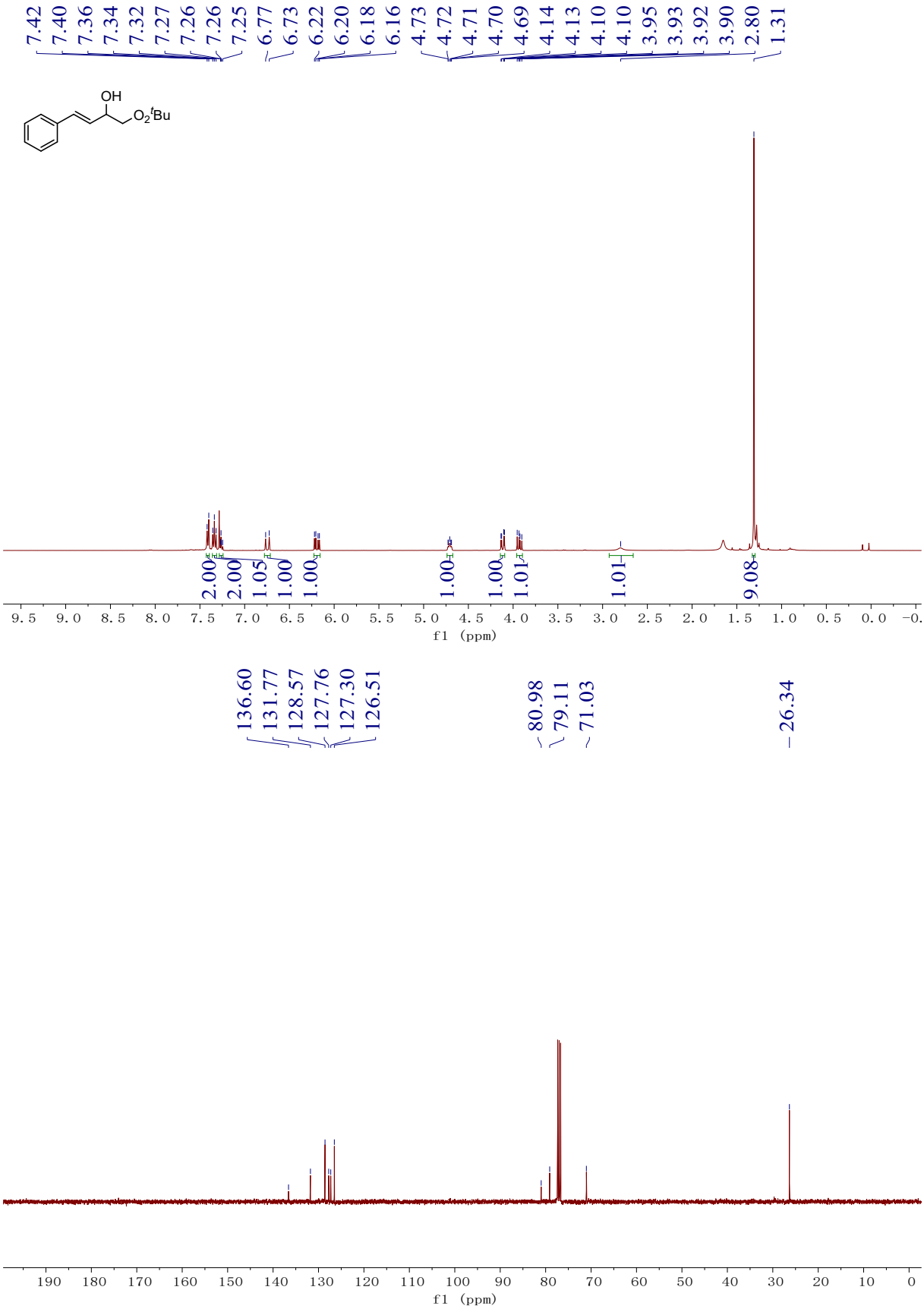

2.01
2.00
1.01
1.00
1.00
1.00
1.00
1.00
1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


136.14
131.52
129.79
128.67
128.09
126.61
124.34
70.55
61.21
90 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

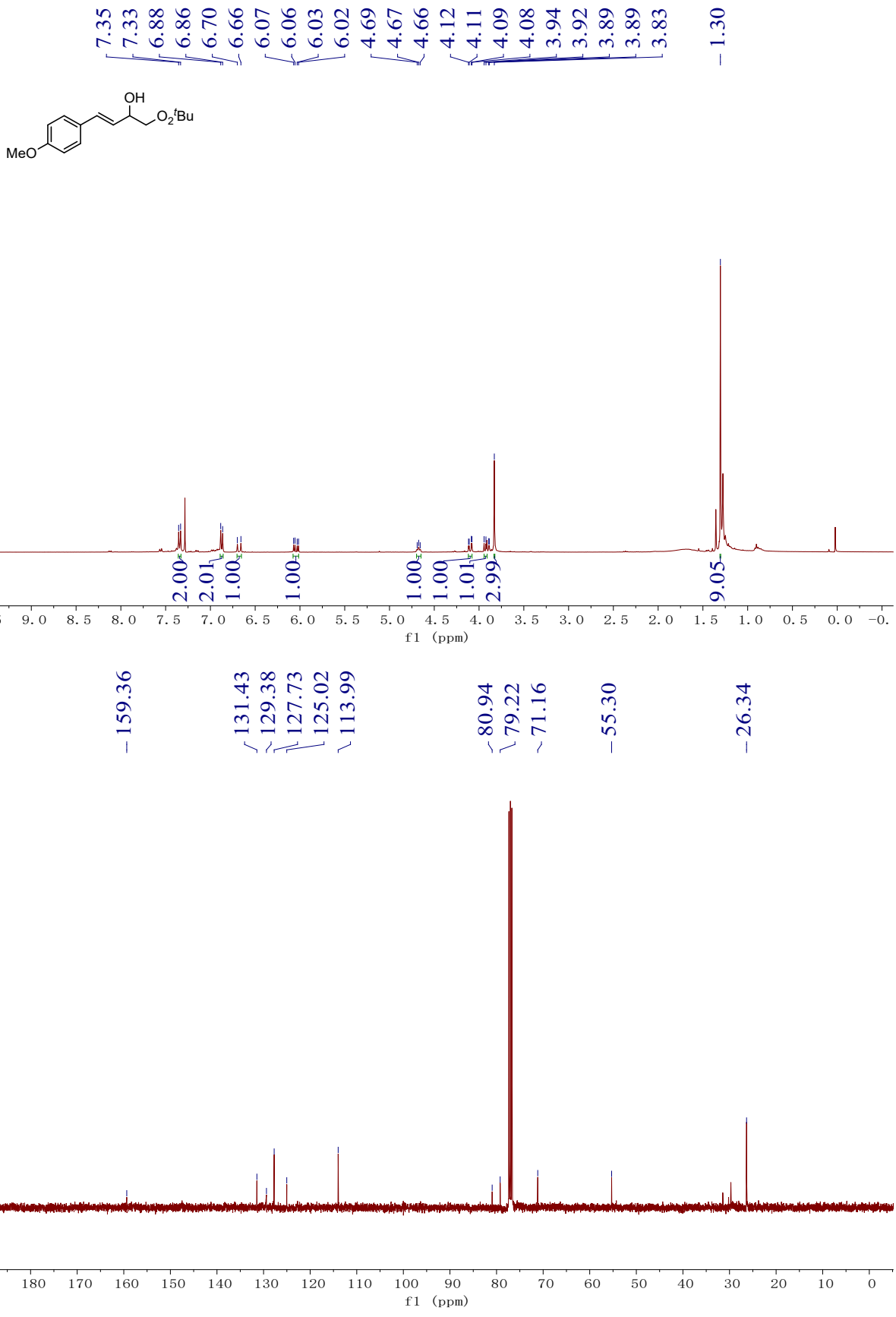
4aa


7.42
7.42
7.40
7.38
7.36
7.34
7.31
7.30
7.30
6.70
6.66
6.26
6.24
6.22
6.20
4.62
4.60
4.57
2.58
2.56
2.54
2.52
2.47
2.46
2.43
2.42
1.86
OH
CDCl2
2.01
2.00
1.01
1.00
1.00
1.00
1.00
1.02
1.01
f1 (ppm)
135.99
131.80
129.98
128.70
128.18
126.60
70.00
50.36
f1 (ppm)

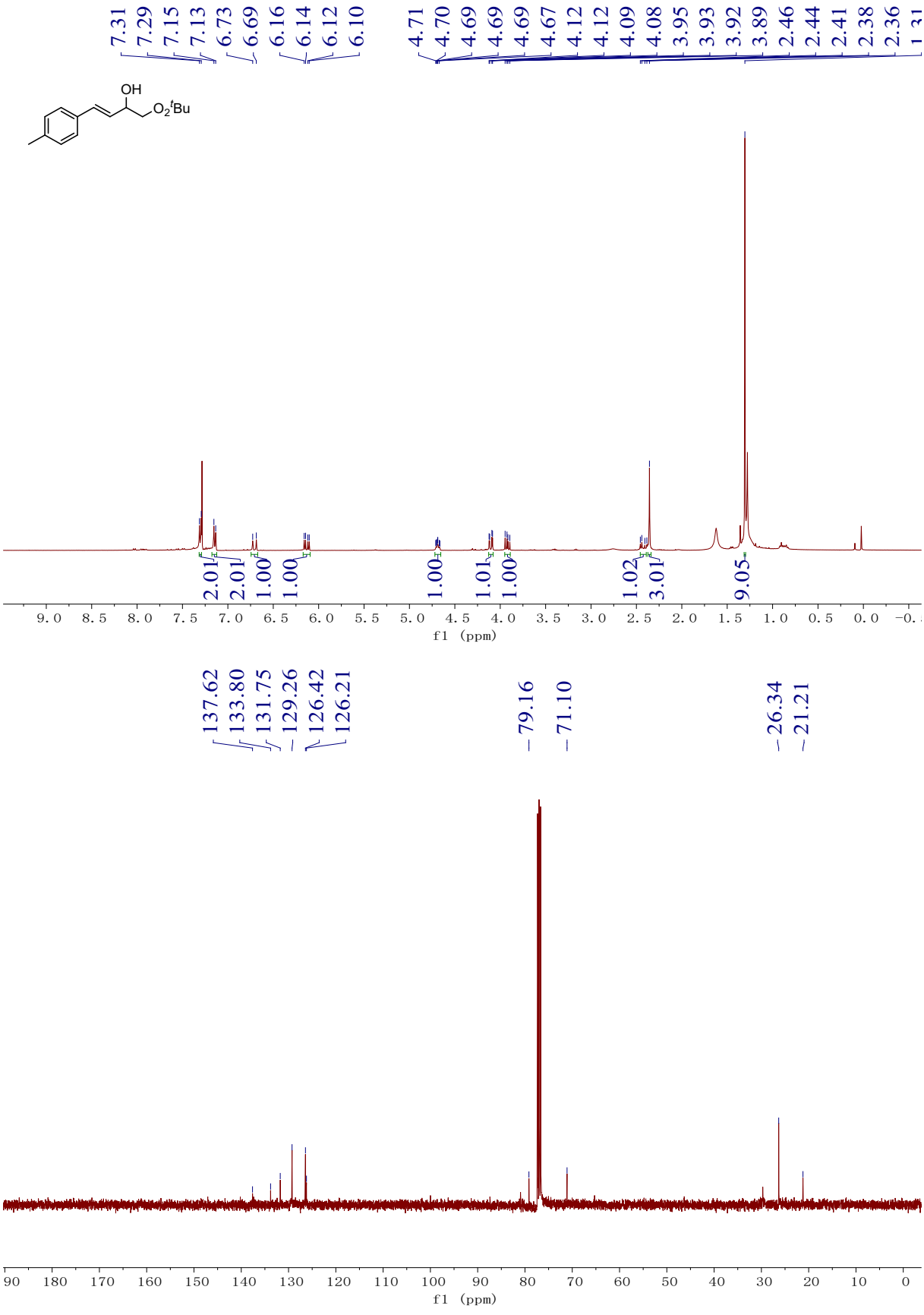
5a


7.42
7.40
7.36
7.34
7.32
7.27
7.26
7.26
7.25
6.77
6.73
6.22
6.20
6.18
6.16
4.73
4.72
4.71
4.70
4.69
4.14
4.13
4.10
4.10
3.95
3.93
3.92
3.90
2.80
1.31
OH
O2tBu
2.00
2.00
1.05
1.00
1.00
1.00
1.00
1.01
1.01
9.08
f1 (ppm)
136.60
131.77
128.57
127.76
127.30
126.51
80.98
79.11
71.03
26.34
f1 (ppm)

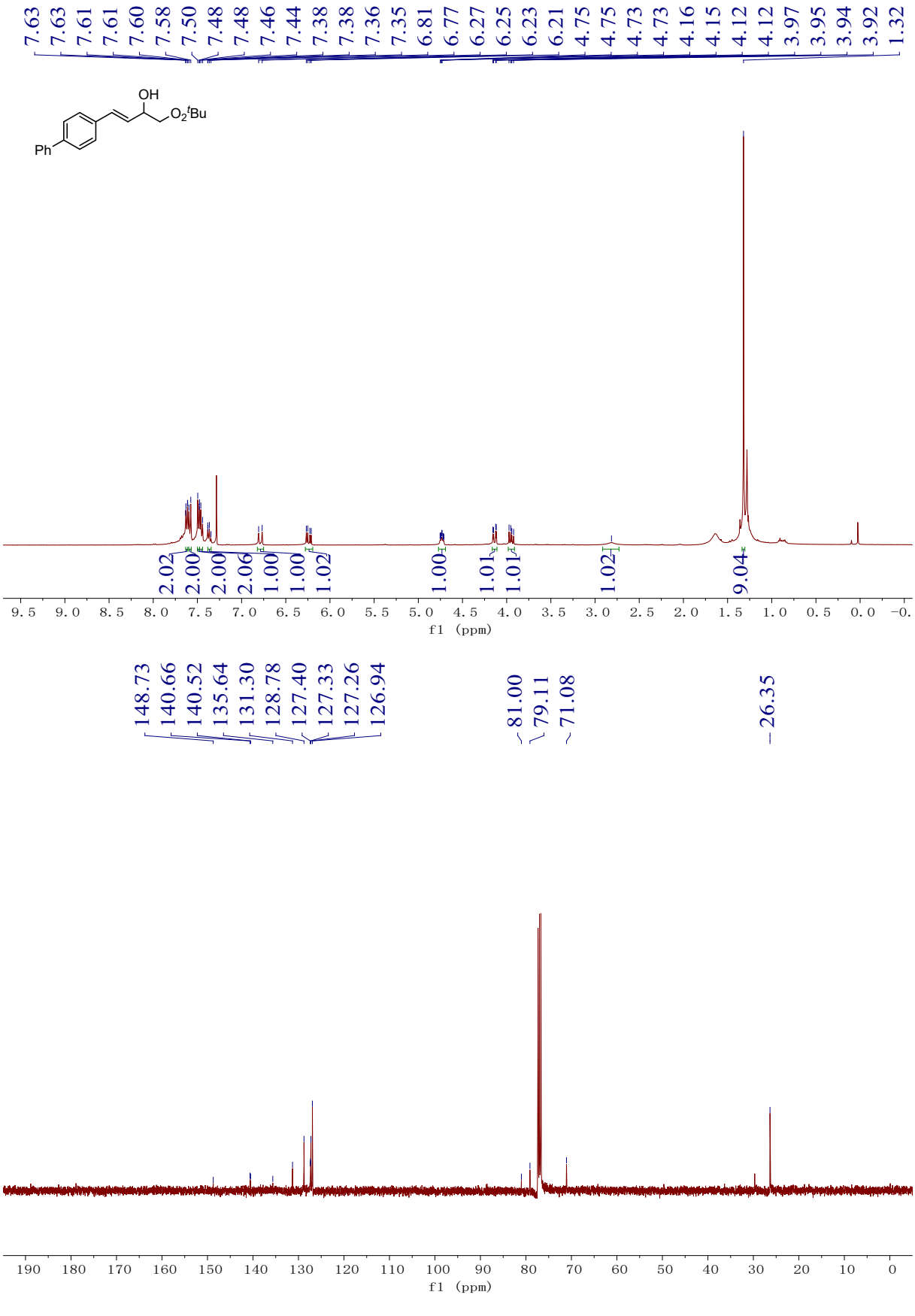
5b


7.35
7.33
6.88
6.86
6.70
6.66
6.07
6.06
6.03
6.02
4.69
4.67
4.66
4.12
4.11
4.09
4.08
3.94
3.92
3.89
3.89
3.83
1.30
OH
O2tBu
MeO
2.00
2.01
1.00
1.00
1.00
1.00
1.01
2.99
9.05
f1 (ppm)
159.36
131.43
129.38
127.73
125.02
113.99
80.94
79.22
71.16
55.30
26.34
f1 (ppm)

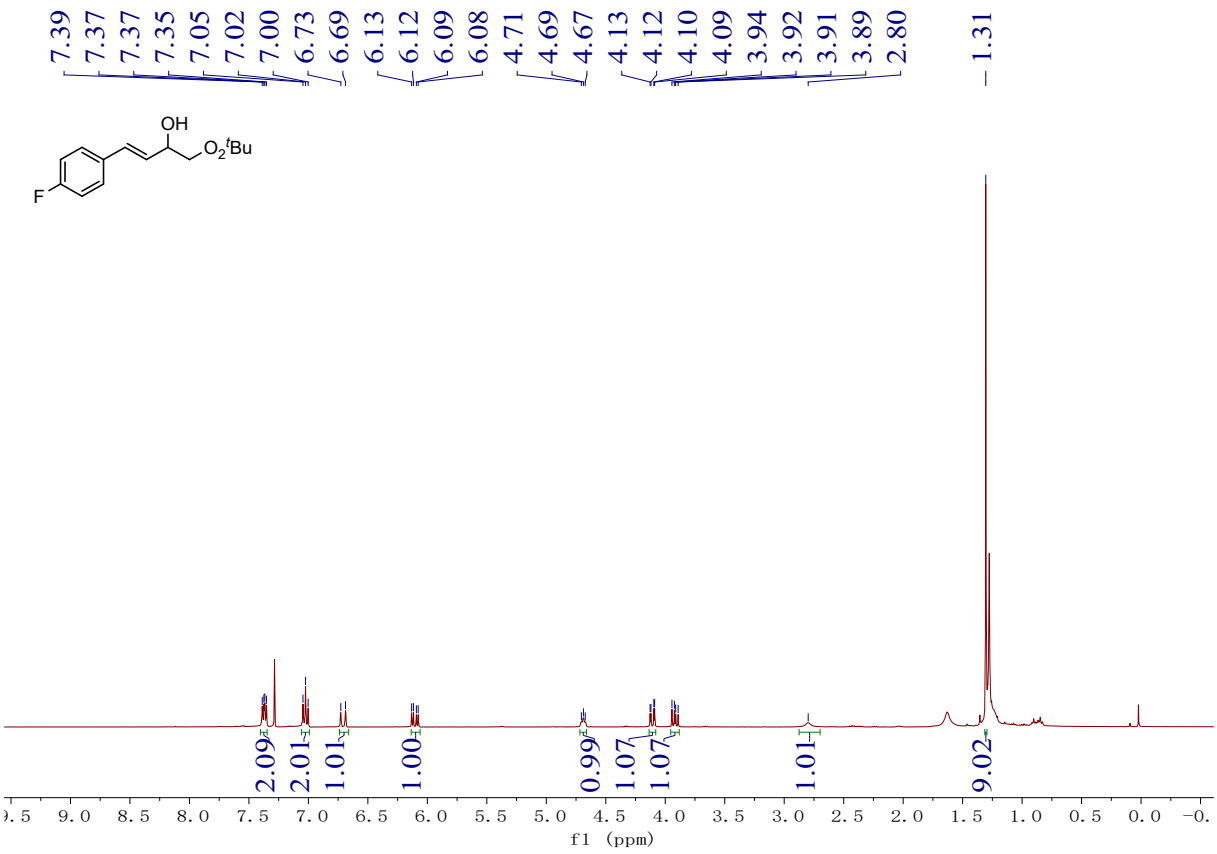
5c

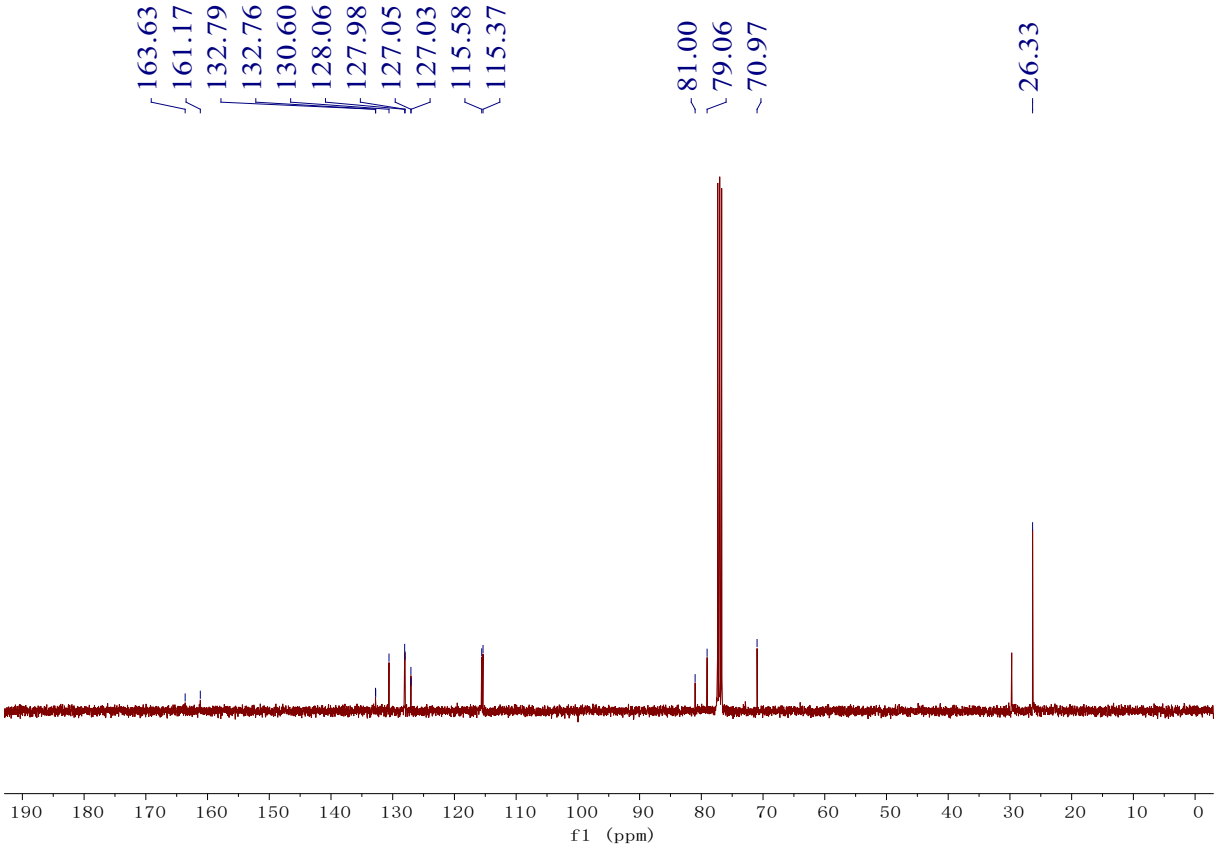

7.31
7.29
7.15
7.13
6.73
6.69
6.16
6.14
6.12
6.10
4.71
4.70
4.69
4.69
4.69
4.67
4.12
4.12
4.09
4.08
3.95
3.93
3.92
3.89
2.46
2.44
2.41
2.38
2.36
OH
O2tBu
2.01
2.01
1.00
1.00
1.00
1.01
1.00
1.02
3.01
9.05
f1 (ppm)
137.62
133.80
131.75
129.26
126.42
126.21
79.16
71.10
26.34
21.21
f1 (ppm)

5d

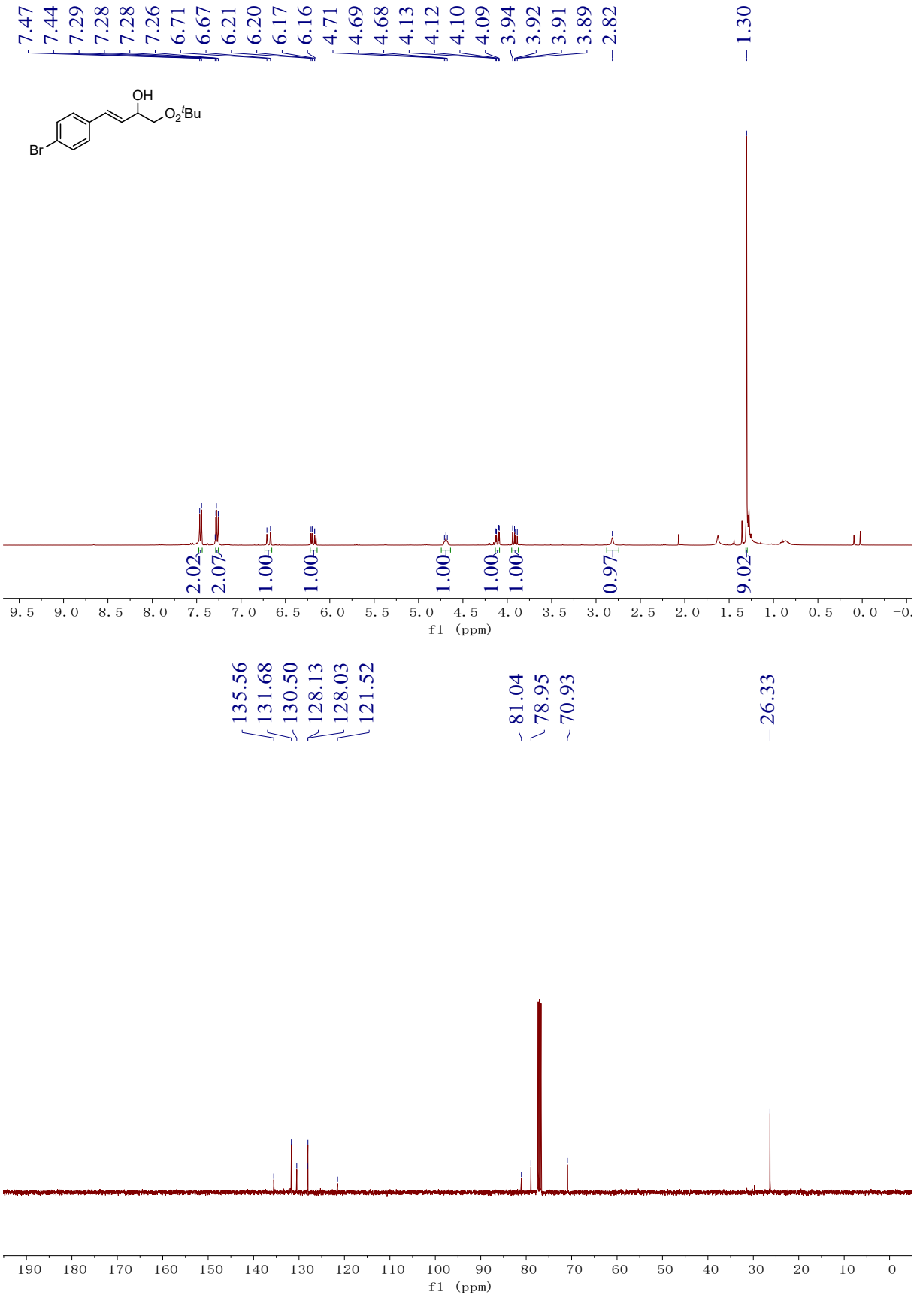

7.63
7.63
7.61
7.61
7.60
7.58
7.50
7.48
7.48
7.46
7.44
7.38
7.38
7.36
7.35
6.81
6.77
6.27
6.25
6.23
6.21
4.75
4.75
4.73
4.73
4.16
4.15
4.12
4.12
3.97
3.95
3.94
3.92
1.32
OH
O2tBu
Ph
2.02
2.00
2.00
2.06
1.00
1.00
1.02
1.00
1.01
1.01
1.02
9.04
f1 (ppm)
148.73
140.66
140.52
135.64
131.30
128.78
127.40
127.33
127.26
126.94
81.00
79.11
71.08
26.35
f1 (ppm)

5e

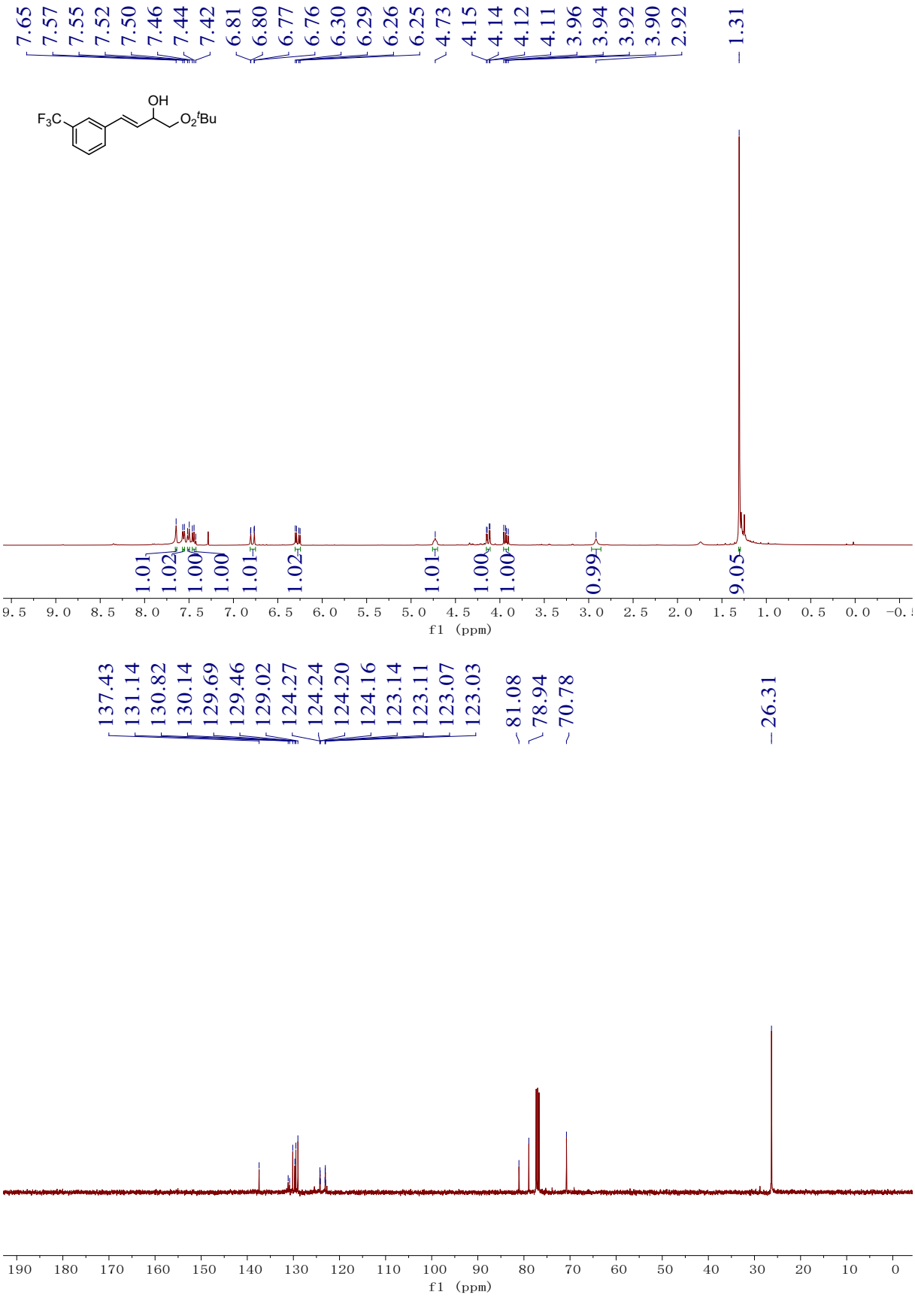

7.39
7.37
7.37
7.35
7.05
7.02
7.00
6.73
6.69
6.13
6.12
6.09
6.08
4.71
4.69
4.67
4.13
4.12
4.10
4.09
3.94
3.92
3.91
3.89
2.80
1.31
OH
O2tBu
F
2.09
2.01
1.01
1.00
0.99
1.07
1.07
1.01
9.02
f1 (ppm)


163.63
161.17
132.79
132.76
130.60
128.06
127.98
127.05
127.03
115.58
115.37
81.00
79.06
70.97
26.33
f1 (ppm)

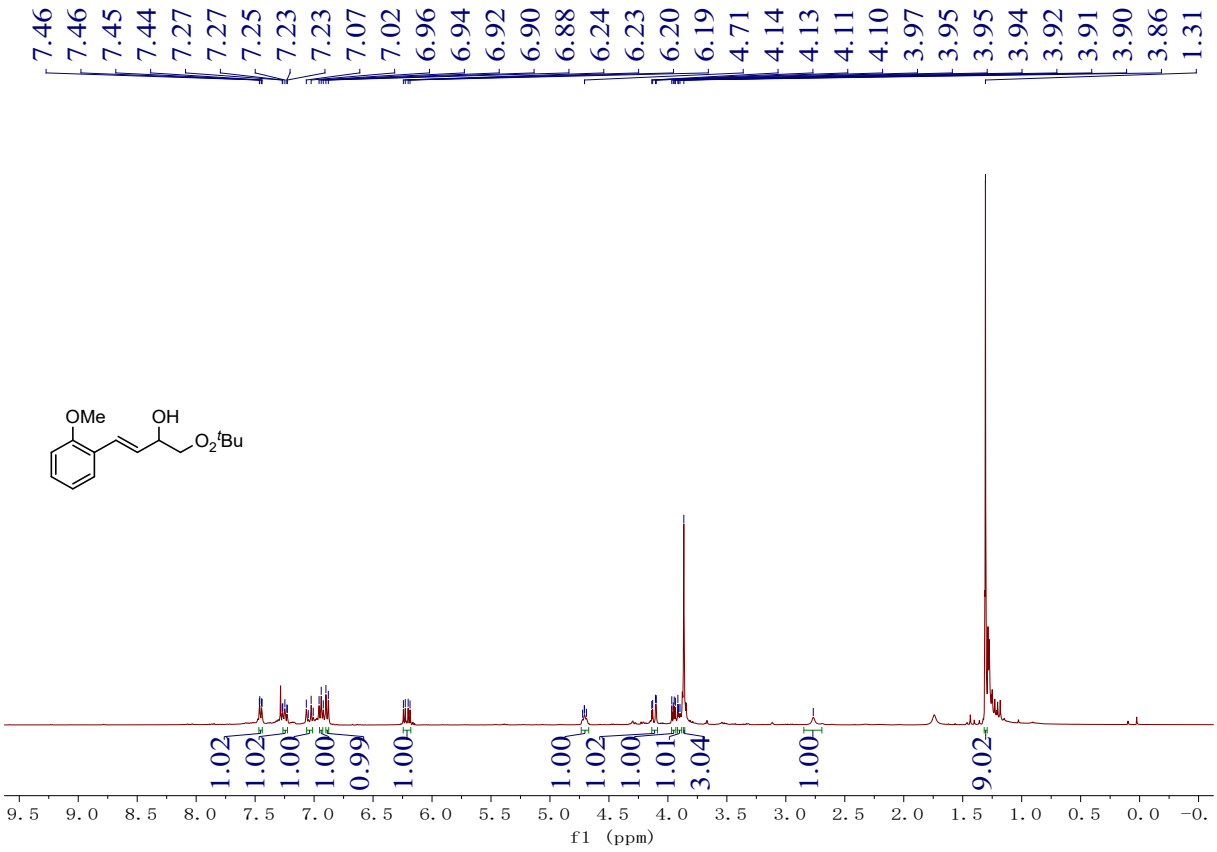
5f

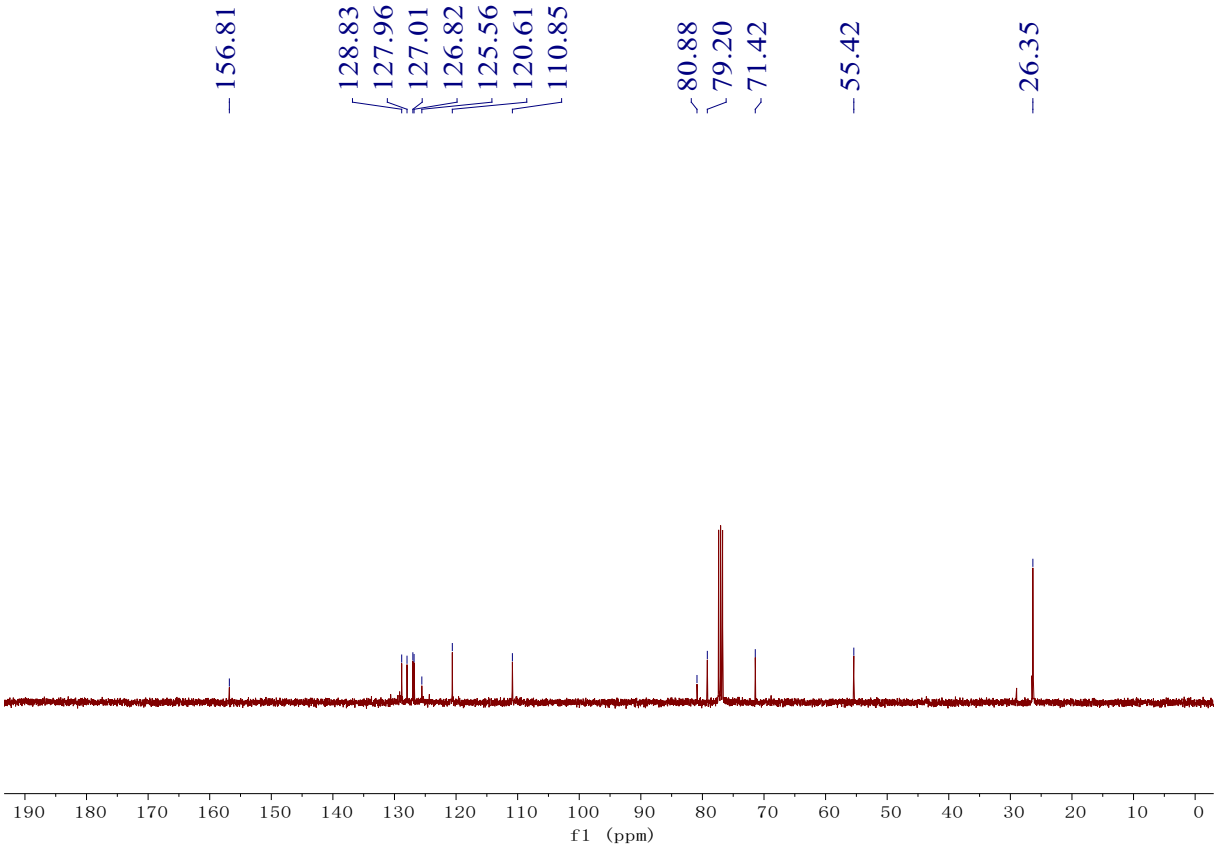

7.47
7.44
7.29
7.28
7.28
7.26
6.71
6.67
6.21
6.20
6.17
6.16
4.71
4.69
4.68
4.13
4.12
4.10
4.09
3.94
3.92
3.91
3.89
2.82
1.30
OH
O2tBu
Br
2.02
2.07
1.00
1.00
1.00
1.00
1.00
0.97
9.02
f1 (ppm)
135.56
131.68
130.50
128.13
128.03
121.52
81.04
78.95
70.93
26.33
f1 (ppm)

5g

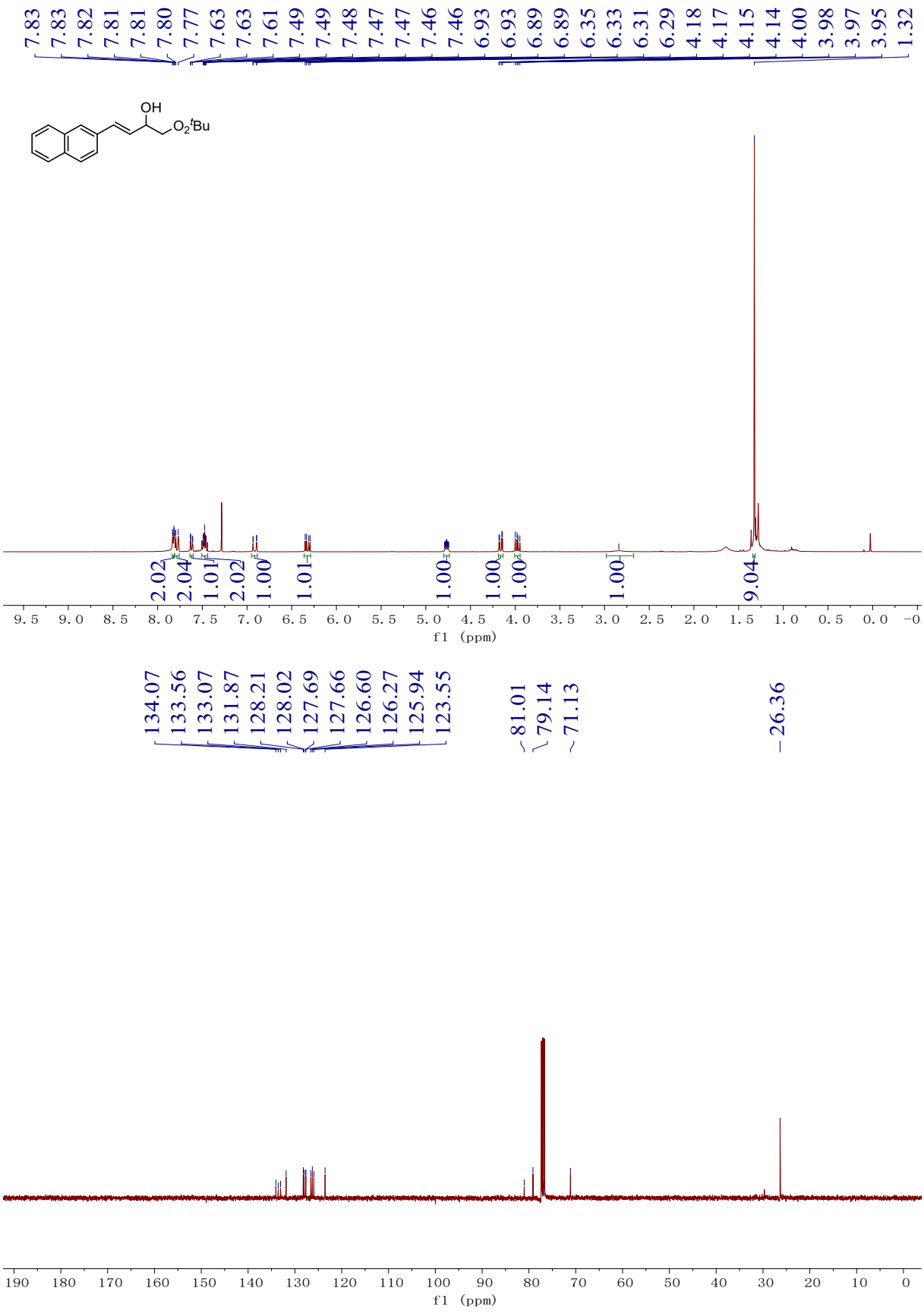

7.65
7.57
7.55
7.52
7.50
7.46
7.44
7.42
6.81
6.80
6.77
6.76
6.30
6.29
6.26
6.25
4.73
4.15
4.14
4.12
4.11
3.96
3.94
3.92
3.90
2.92
1.31
OH
F3C
O2tBu
1.01
1.02
1.00
1.00
1.01
1.02
1.01
1.00
1.00
0.99
9.05
f1 (ppm)
137.43
131.14
130.82
130.14
129.69
129.46
129.02
124.27
124.24
124.20
124.16
123.14
123.11
123.07
123.03
81.08
78.94
70.78
26.31
f1 (ppm)

5h

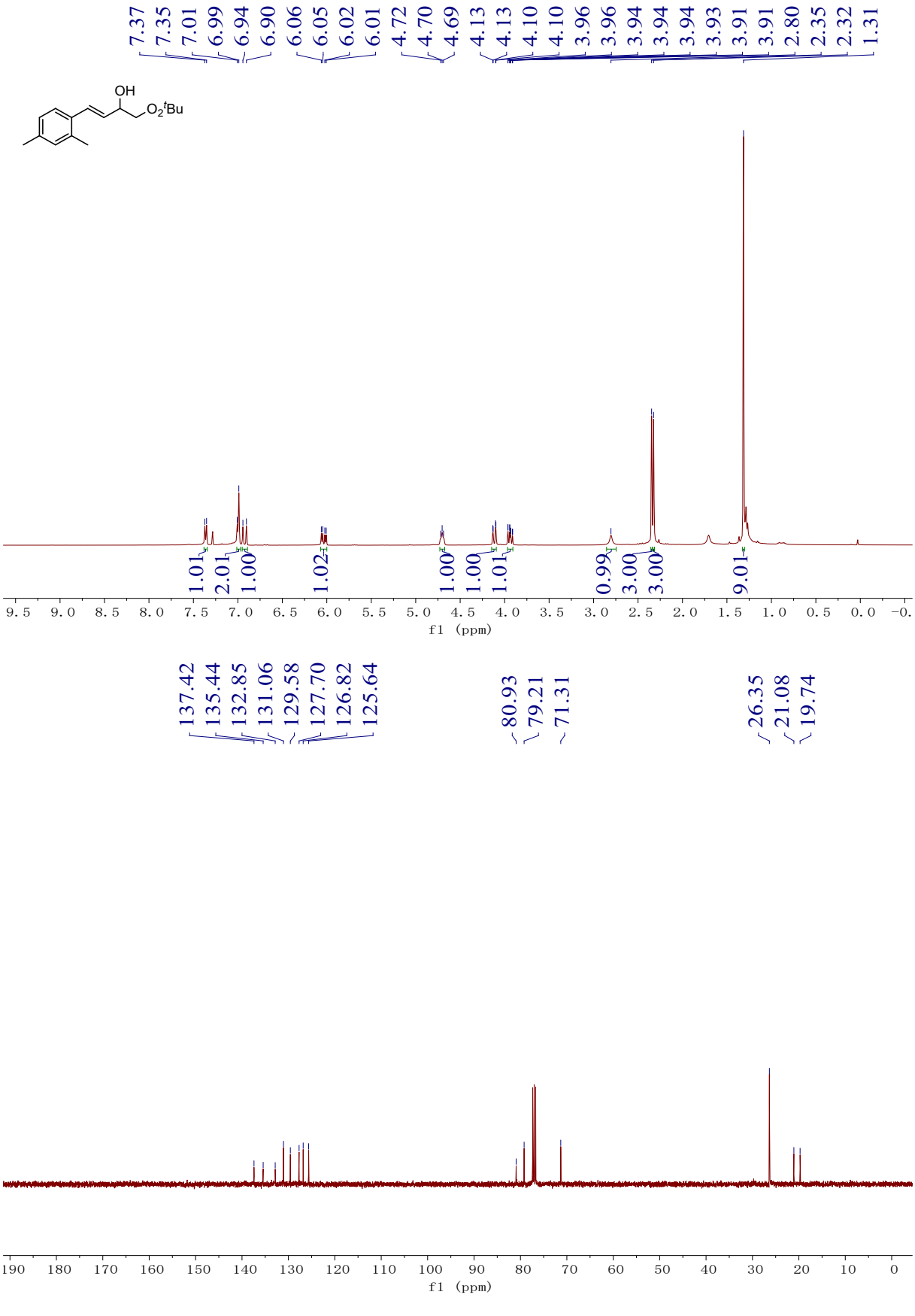

7.46 7.46 7.45 7.44 7.27 7.27 7.25 7.23 7.23 7.07 7.02 6.96 6.94 6.92 6.90 6.88 6.24 6.23 6.20 6.19 4.71 4.14 4.13 4.11 4.10 3.97 3.95 3.95 3.94 3.92 3.91 3.90 3.86 1.31
OMe
OH
O2tBu
1.02 1.02 1.00 1.00 0.99 1.00 1.00 1.02 1.00 1.01 3.04 1.00 9.02
f1 (ppm)


156.81
128.83 127.96 127.01 126.82 125.56 120.61 110.85
80.88 79.20 71.42
55.42
26.35
f1 (ppm)

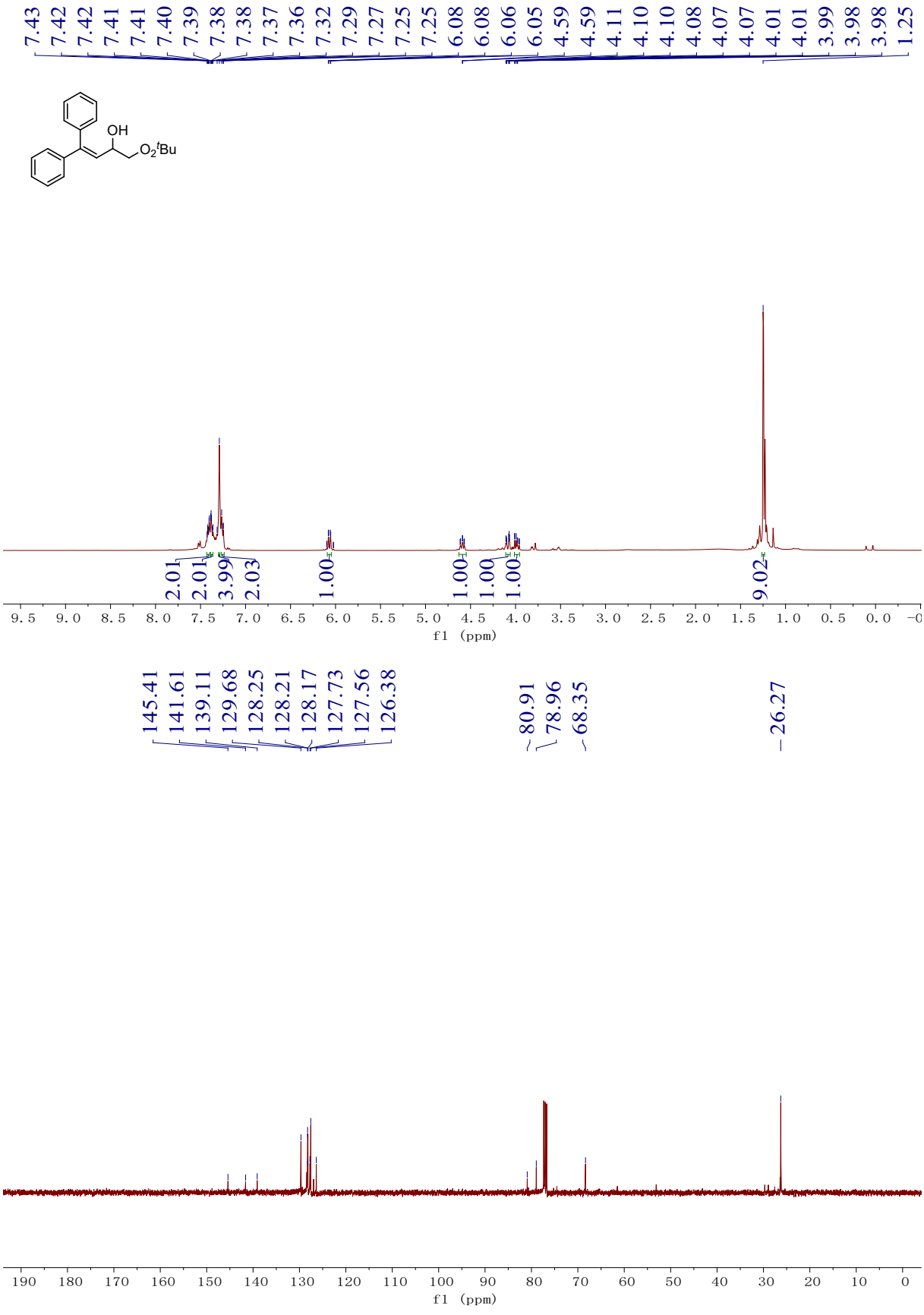
5i


7.83
7.83
7.82
7.81
7.81
7.80
7.77
7.63
7.63
7.61
7.49
7.49
7.48
7.47
7.47
7.46
7.46
6.93
6.93
6.89
6.89
6.35
6.33
6.31
6.29
4.18
4.17
4.15
4.14
4.00
3.98
3.97
3.95
1.32
OH
O2tBu
2.02
2.04
1.01
2.02
1.00
1.01
1.00
1.00
1.00
1.00
9.04
f1 (ppm)
134.07
133.56
133.07
131.87
128.21
128.02
127.69
127.66
126.60
126.27
125.94
123.55
81.01
79.14
71.13
26.36
f1 (ppm)

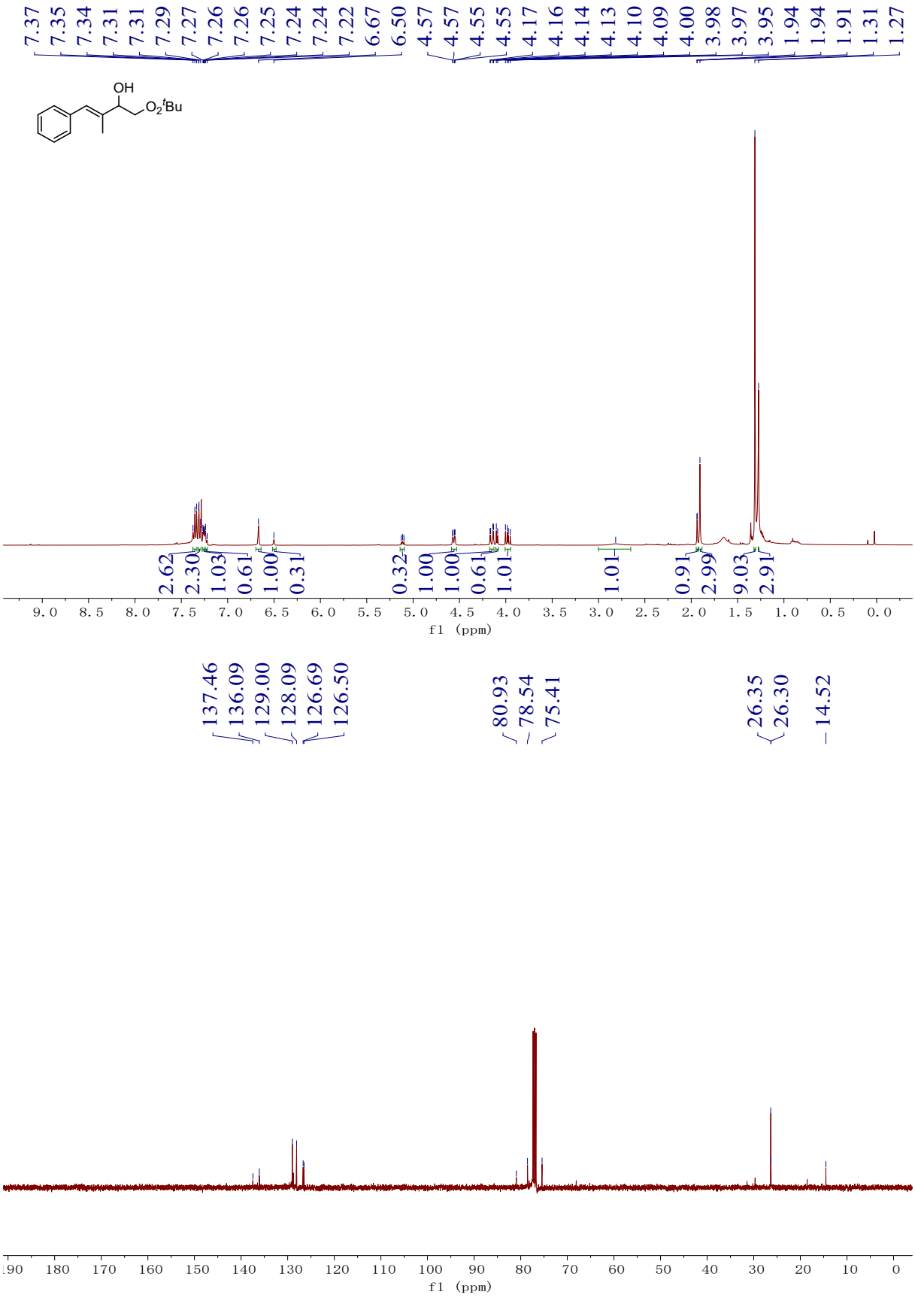
5j


7.37
7.35
7.01
6.99
6.94
6.90
6.06
6.05
6.02
6.01
4.72
4.70
4.69
4.13
4.13
4.10
4.10
3.96
3.96
3.94
3.94
3.94
3.93
3.91
3.91
2.80
2.35
2.32
1.31
OH
O2tBu
1.01
2.01
1.00
1.02
1.00
1.00
1.01
0.99
3.00
3.00
9.01
f1 (ppm)
137.42
135.44
132.85
131.06
129.58
127.70
126.82
125.64
80.93
79.21
71.31
26.35
21.08
19.74
f1 (ppm)

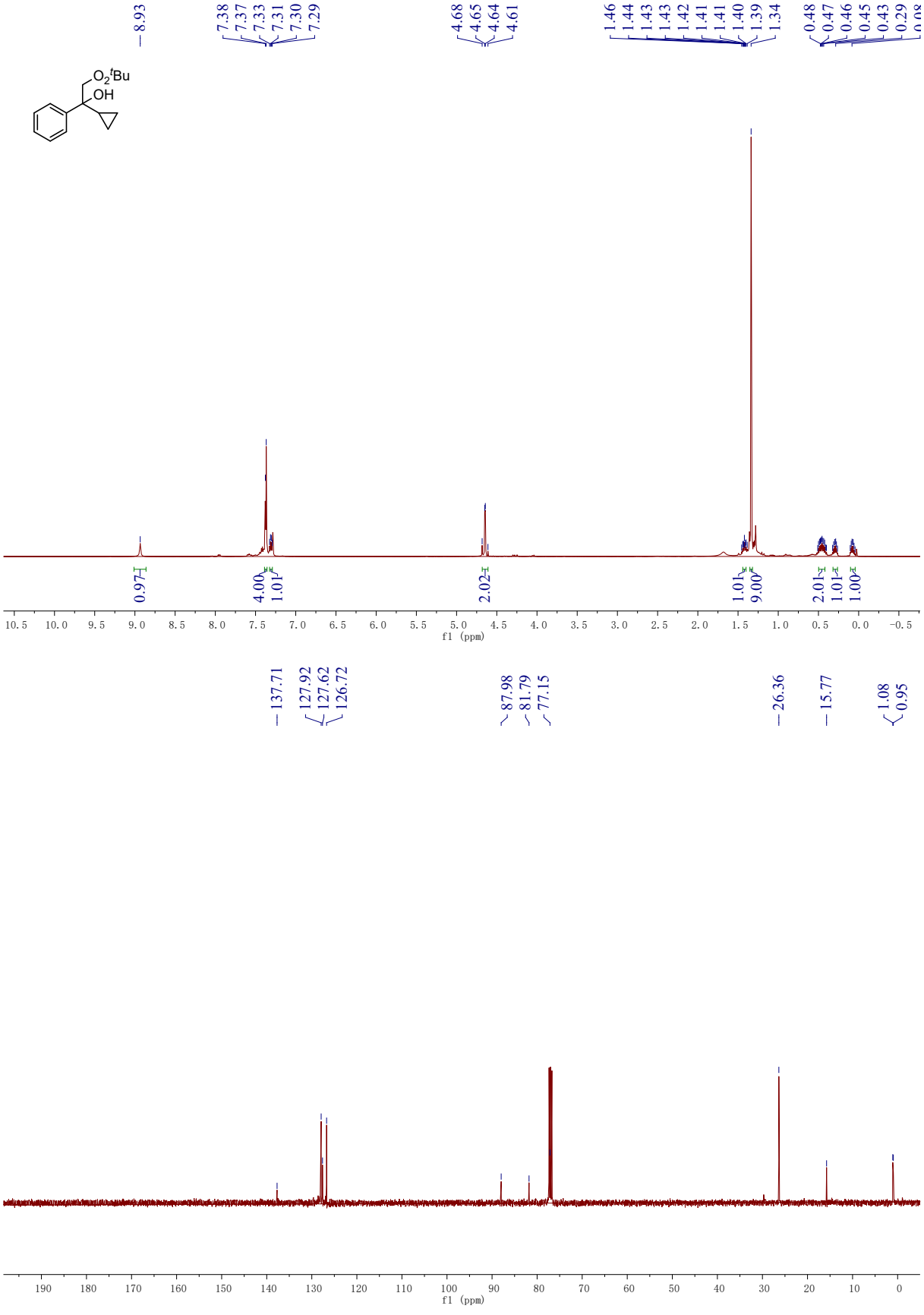
5k


7.43
7.42
7.42
7.41
7.41
7.40
7.39
7.38
7.38
7.37
7.36
7.32
7.29
7.27
7.25
7.25
6.08
6.08
6.06
6.05
4.59
4.59
4.11
4.10
4.10
4.08
4.07
4.07
4.01
4.01
3.99
3.98
3.98
1.25
OH
O2tBu
2.01
2.01
3.99
2.03
1.00
1.00
1.00
1.00
9.02
f1 (ppm)
145.41
141.61
139.11
129.68
128.25
128.21
128.17
127.73
127.56
126.38
80.91
78.96
68.35
26.27
f1 (ppm)

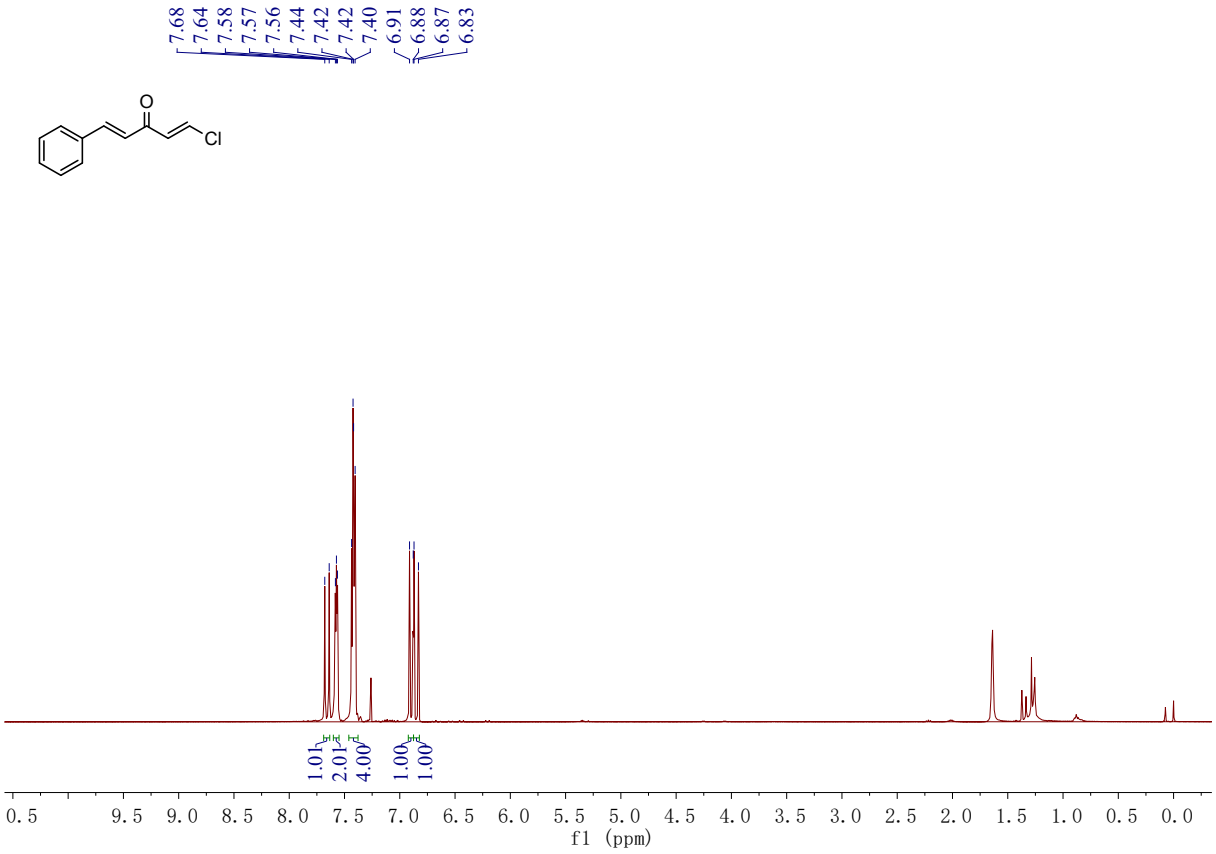
5l

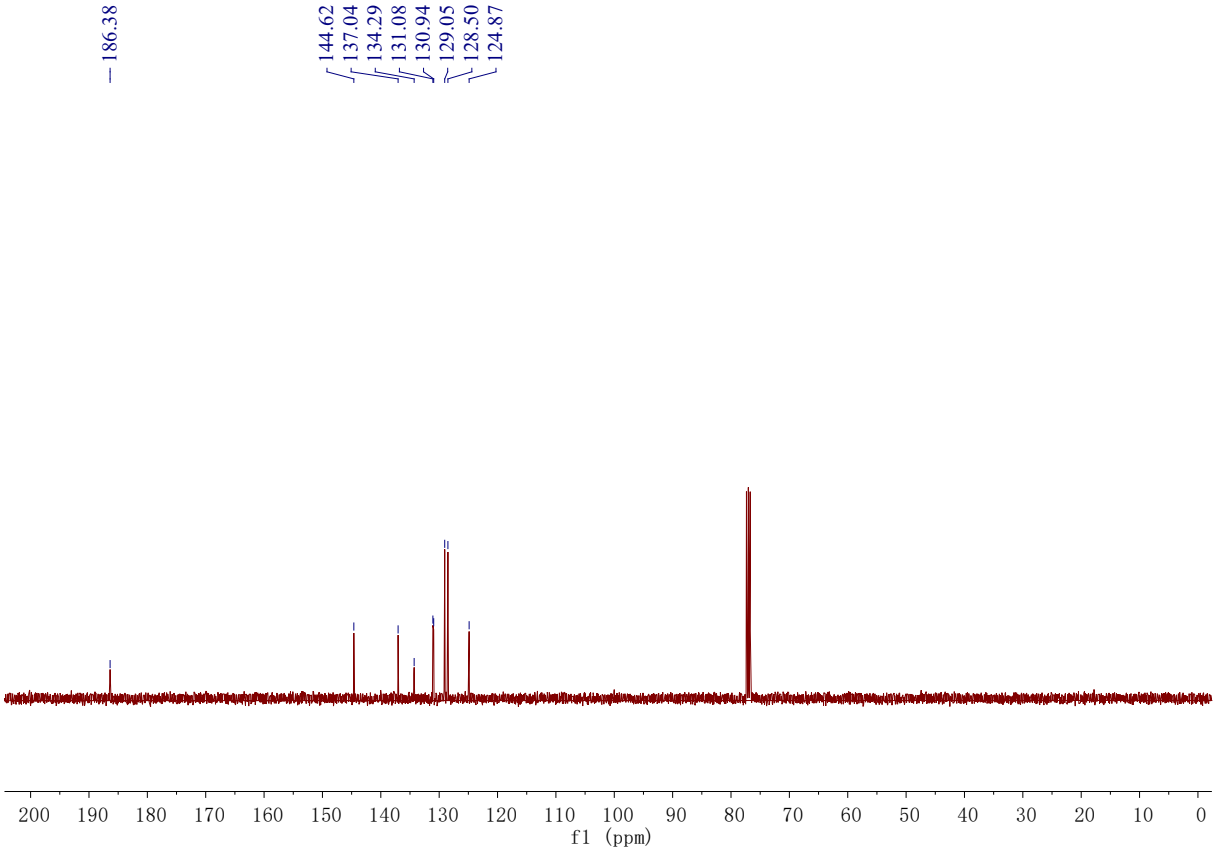

7.37
7.35
7.34
7.31
7.31
7.29
7.27
7.26
7.26
7.25
7.24
7.24
7.22
6.67
6.50
4.57
4.57
4.55
4.55
4.17
4.16
4.14
4.13
4.10
4.09
4.00
3.98
3.97
3.95
1.94
1.94
1.91
1.31
1.27
OH
O2tBu
2.62
2.30
1.03
0.61
1.00
0.31
0.32
1.00
1.00
0.61
1.01
1.01
0.91
2.99
9.03
2.91
f1 (ppm)
137.46
136.09
129.00
128.09
126.69
126.50
80.93
78.54
75.41
26.35
26.30
14.52
f1 (ppm)

5o


8.93
7.38
7.37
7.33
7.31
7.30
7.29
4.68
4.65
4.64
4.61
1.46
1.44
1.43
1.43
1.42
1.41
1.41
1.40
1.39
1.34
0.48
0.47
0.46
0.45
0.43
0.29
O2tBu
OH
0.97
4.00
1.01
2.02
1.01
9.00
2.01
1.01
1.00
f1 (ppm)
137.71
127.92
127.62
126.72
87.98
81.79
77.15
26.36
15.77
1.08
0.95
f1 (ppm)

6a


7.68
7.64
7.58
7.57
7.56
7.44
7.42
7.42
7.40
6.91
6.88
6.87
6.83
O
Cl
1.01
2.01
4.00
1.00
1.00
f1 (ppm)


186.38
144.62
137.04
134.29
131.08
130.94
129.05
128.50
124.87
f1 (ppm)