Supporting information

Palladium-Catalyzed Asymmetric Allylic Alkylation of Alkyl Substituted Allyl Reagents with Acyclic Amides

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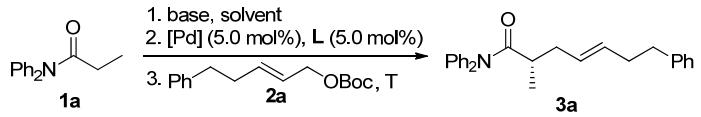
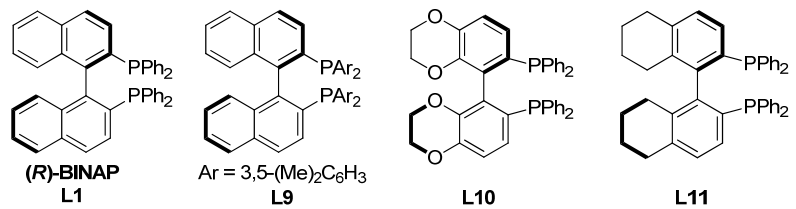
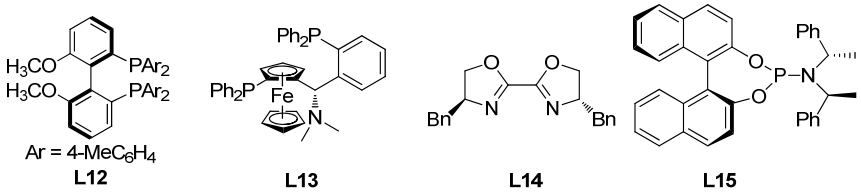
1. General methods

Commercially available reagents were used without further purification. Solvents were purified prior to use according to the standard methods. Column chromatography was performed on silica gel (300–400 mesh) using a forced flow of eluent. NMR spectra were recorded at room temperature on an NMR instrument operating at 400 MHz. Chemical shifts for ^{1}H NMR are reported in parts per million with the solvent resonance as the internal standard (7.26 ppm for $CHCl_3$). The data were reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, sep = septet, bs = broad singlet, m = multiplet), coupling constants (Hz), and integration. ^{13}C NMR spectra were recorded on an NMR instrument operating at 100 MHz with complete proton decoupling. Chemical shifts were reported in parts per million with the solvent resonance as the internal standard (77.1 ppm for $CDCl_3$). MS and HRMS were measured in EI, ESI, or DART (direct analysis in real-time) mode and the mass analysis mode of the HRMS was TOF. Infrared spectra were recorded from thin films of pure samples. Melting points were measured on an XT-4 micromelting point apparatus. Thin layer chromatography was performed on precoated glass-backed plates and visualized with UV light at 254 nm. Enantiomeric ratios were determined by chiral HPLC analysis in comparison with authentic racemic materials.

2. Screening of reaction conditions for Pd-catalyzed asymmetric allylic alkylation of amide 1a with allyl reagent 2a[a]



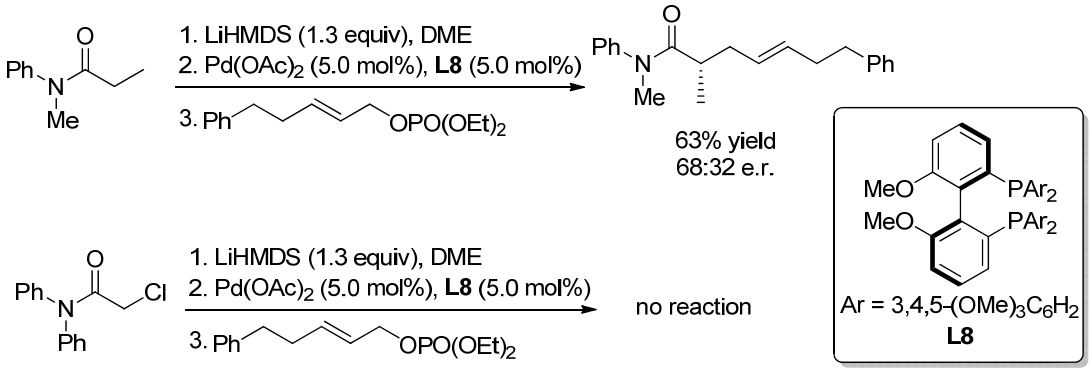




entry	base	solvent	[Pd]	**L**	yield %[b]	ee%[c]
1	LDA	THF	$[Pd(C_3H_5)Cl]_2$	**L1**	50	32
2	LDA	THF	$Pd_2(dba)_3$	**L1**	90	34
3	LDA	Et_2O	$Pd(OAc)_2$	**L1**	90	32
4	LDA	toluene	$Pd(OAc)_2$	**L1**	79	37
5	LDA	DME	$Pd(OAc)_2$	**L1**	91	42
6	LDA	DCM	$Pd(OAc)_2$	**L1**	NR	—
7	LiHMDS	DME	$Pd(OAc)_2$	**L1**	94	46
8	NaHMDS	DME	$Pd(OAc)_2$	**L1**	90	29
9	KHMDS	DME	$Pd(OAc)_2$	**L1**	trace	—
10	^{t}BuOK	DME	$Pd(OAc)_2$	**L1**	trace	—
11	Cs_2CO_3	DME	$Pd(OAc)_2$	**L1**	NR	—
12	LiHMDS	DME	$Pd(OAc)_2$	**L9**	94	48
13	LiHMDS	DME	$Pd(OAc)_2$	**L10**	98	41
14	LiHMDS	DME	$Pd(OAc)_2$	**L11**	99	51
15	LiHMDS	DME	$Pd(OAc)_2$	**L12**	72	23
16	LiHMDS	DME	$Pd(OAc)_2$	**L13**	75	12
17	LiHMDS	DME	$Pd(OAc)_2$	**L14**	NR	—
18	LiHMDS	DME	$Pd(OAc)_2$	**L15**	NR	—

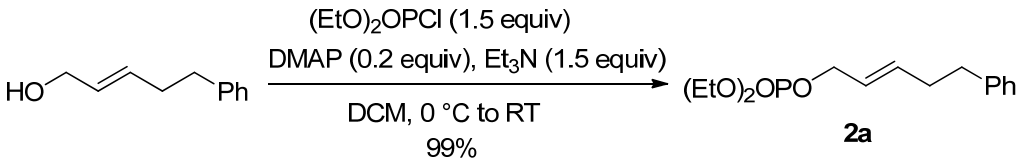
[a]Molar ratio of **1a**/**2a**/base/[Pd]/ligand/additive = 130/100/130/5/5/100. [b]The yield determined by ^{1}H NMR using mesitylene as internal standard. [c]Determined by chiral HPLC.

3. Effect of amide on the Pd-catalyzed asymmetric allylic alkylation



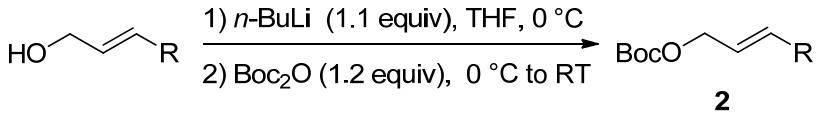
4. Synthesis of allylic reagents 2

Experimental procedure for the preparation of allylic reagent 2a[1]



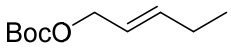
4-Dimethylaminopyridine (257 mg, 2.1 mmol) and triethylamine (2.2 mL, 15.7 mmol) were added to a solution of (*E*)-5-phenylpent-2-en-1-ol (1.7 g, 10.5 mmol) in CH_2Cl_2 (35 mL). The resulting solution was cooled to 0 °C. Diethyl chlorophosphate (2.3 mL, 15.7 mmol) was added dropwise, and the reaction mixture was stirred overnight at room temperature. The reaction was quenched with saturated aqueous NH_4Cl and extracted with EtOAc. The organic layer was dried over anhydrous $MgSO_4$, filtered, and concentrated under vacuum. The residue was purified by chromatography on silica gel with hexane/EtOAc = 1/1 to afford **2a**[2] as a colorless oil (3.12g, 99% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (dd, *J* = 8.1, 6.3 Hz, 2H), 7.21–7.14 (m, 3H), 5.89–5.79 (m, 1H), 5.69–5.59 (m, 1H), 4.52–4.43 (m, 2H), 4.16–4.03 (m, 4H), 2.77–2.65 (m, 2H), 2.39 (dd, *J* = 15.0, 7.1 Hz, 2H), 1.33 (td, *J* = 7.1, 0.9 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 141.40, 135.26, 128.37, 128.33, 125.92, 125.05 (d, *J* = 6.9 Hz), 67.97 (d, *J* = 5.4 Hz), 63.73 (d, *J* = 5.6 Hz), 35.21, 33.90, 16.17 (d, *J* = 6.9 Hz).

General experimental procedure for the preparation of allylic *tert*-butyl carbonates[3]



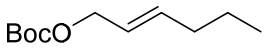
2

To a solution of allylic alcohol (10 mmol) in THF (45 mL) under a nitrogen atmosphere was added *n*-BuLi (2.0 M in hexane, 11 mmol) at 0 °C and the mixture was stirred for 30 min. Boc_2O (12 mmol) was added at 0 °C and the reaction mixture was stirred overnight at room temperature. The reaction was quenched with H_2O and the solvent was removed in vacuo. The residue was diluted with ethanol (15 mL) and imidazole (3 mmol) was added to remove the unreacted Boc_2O. After stirring for 1 h at room temperature, the solvent was removed and the residue was purified by flash column chromatography (petroleum ether: ethyl acetate = 100:1) to give the corresponding allylic *tert*-butyl carbonate.



2b

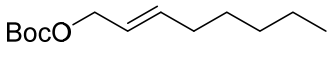
(*E*)-*tert*-butyl pent-2-en-1-yl carbonate: General procedure was followed on a 10.0 mmol scale, a colorless oil (1.7 g, 95%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.91–5.77 (m, 1H), 5.65–5.51 (m, 1H), 4.50 (d, *J* = 6.6 Hz, 2H), 2.14–2.01 (m, 2H), 1.49 (s, 9H), 1.00 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.30, 138.28, 122.52, 81.69, 67.60, 27.67, 25.14, 12.94; IR: 2967, 1738, 1458, 1369, 1272, 1251, 1159, 1102, 857, 793; MS (EI): m/z (%) 69 (58), 57 (100), 55 (5), 42 (4); HRMS (DART) m/z Calcd for $C_{10}H_{19}O_3$ $[M+H]^+$: 187.1329, found 187.1329.



2c

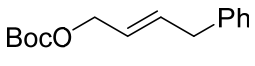
(*E*)-*tert*-butyl hex-2-en-1-yl carbonate: General procedure was followed on a 8.5 mmol scale, a colorless oil (1.6 g, 95%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.84–5.74 (m, 1H), 5.58 (ddd, *J* = 13.9, 7.2, 5.9 Hz, 1H), 4.50 (d, *J* = 6.6 Hz, 2H), 2.03 (q, *J* = 7.1 Hz, 2H), 1.48 (s, 9H), 1.46–1.35 (m, 2H), 0.90 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101

MHz, $CDCl_3$) δ 153.36, 136.86, 123.60, 81.87, 67.71, 34.26, 27.74, 21.96, 13.60; IR: 2962, 1738, 1458, 1369, 1273, 1251, 1159, 1104, 971, 928, 858, 793, 761; MS (EI): m/z (%) 200 (M^+, 0.1), 144 (13), 83 (53), 57 (100), 56 (21), 43 (7); HRMS (DART) m/z Calcd for $C_{11}H_{24}O_3N$ $[M+NH_4]^+$: 218.1751, found 218.1751.



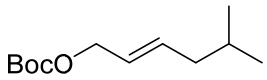
2d

(*E*)-*tert*-butyl oct-2-en-1-yl carbonate: General procedure was followed on a 13.3 mmol scale, a colorless oil (2.8 g, 92%). ^1H NMR (400 MHz, $CDCl_3$) δ 5.85–5.74 (m, 1H), 5.63–5.53 (m, 1H), 4.50 (d, *J* = 6.6 Hz, 2H), 2.04 (q, *J* = 7.0 Hz, 2H), 1.49 (s, 9H), 1.43–1.33 (m, 2H), 1.33–1.22 (m, 4H), 0.88 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.37, 137.17, 123.38, 81.89, 67.75, 32.18, 31.30, 28.47, 27.75, 22.47, 14.00; IR: 2858, 1739, 1458, 1369, 1272, 1251, 1159, 1102, 1036, 972, 928, 859, 793; MS (EI): m/z (%) 228 (M^+, 0.03), 144 (2), 117 (1), 84 (2), 71 (3), 57 (100), 41 (10); HRMS (DART) m/z Calcd for $C_{13}H_{28}O_3N$ $[M+NH_4]^+$: 246.2064, found 246.2064.



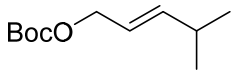
2e

(*E*)-*tert*-butyl (4-phenylbut-2-en-1-yl) carbonate: General procedure was followed on a 2.4 mmol scale, a colorless oil (0.45 g, 77%). ^1H NMR (400 MHz, $CDCl_3$) δ 7.28 (dd, *J* = 13.8, 6.1 Hz, 2H), 7.24–7.14 (m, 3H), 5.95 (dt, *J* = 13.9, 6.7 Hz, 1H), 5.65 (dt, *J* = 13.6, 6.5 Hz, 1H), 4.53 (d, *J* = 6.4 Hz, 2H), 3.40 (d, *J* = 6.7 Hz, 2H), 1.48 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.33, 139.45, 135.02, 128.60, 128.45, 126.21, 124.97, 82.08, 67.34, 38.61, 27.76; IR: 2979, 1737, 1454, 1369, 1273, 1251, 1157, 1090, 971, 857, 793, 747, 698, 461; MS (EI): m/z (%) 248 (M^+, 0.02), 131 (45), 104 (4), 91 (23), 77 (4), 57 (30); HRMS (DART) m/z Calcd for $C_{15}H_{21}O_3$ $[M+H]^+$: 249.1485, found 249.1485.



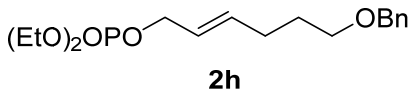
2f

(*E*)-*tert*-butyl (5-methylhex-2-en-1-yl) carbonate: General procedure was followed on a 3.7 mmol scale, a colorless oil (0.73 g, 92%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.77 (dt, *J* = 14.4, 7.2 Hz, 1H), 5.62–5.52 (m, 1H), 4.50 (d, *J* = 6.5 Hz, 2H), 1.94 (t, *J* = 6.9 Hz, 2H), 1.68–1.60 (m, 1H), 1.49 (s, 9H), 0.88 (d, *J* = 6.6 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.32, 135.63, 124.62, 81.75, 67.58, 41.51, 28.00, 27.70, 22.19; IR: 2957, 1738, 1462, 1368, 1274, 1251, 1159, 1100, 972, 858, 793, 758; MS (DART): m/z (%) 214 (M^+, 0.2), 97 (60), 57 (100), 43 (24); HRMS (EI) m/z Calcd for $C_{12}H_{26}O_3N$ $[M+NH_4]^+$: 232.1907, found 232.1907.



2g

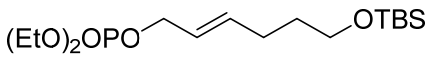
(*E*)-*tert*-butyl (4-methylpent-2-en-1-yl) carbonate: General procedure was followed on a 2.6 mmol scale, a colorless oil (0.42 g, 80%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.76 (ddt, *J* = 15.4, 6.4, 1.1 Hz, 1H), 5.53 (dtd, *J* = 15.4, 6.6, 1.4 Hz, 1H), 4.52–4.48 (m, 2H), 2.31 (dq, *J* = 13.6, 6.8 Hz, 1H), 1.49 (s, 9H), 0.99 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 153.36, 143.73, 120.55, 81.96, 67.89, 30.75, 27.77, 21.93; IR: 2961, 1738, 1462, 1368, 1273, 1251, 1159, 1090, 971, 927, 860, 793, 763; MS (EI): m/z (%) 200 (M^+, 1.1), 83 (14), 69 (7), 57 (100), 56 (25), 43 (28); HRMS (DART) m/z Calcd for $C_{11}H_{24}O_3N$ $[M+NH_4]^+$: 218.1751, found 218.1751.



2h

(*E*)-6-(benzyloxy)hex-2-en-1-yl diethyl phosphate: Procedure was followed as the preparation of **2a** on a 4.85mmol scale, a colorless oil (1.68g, 99%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42–7.25 (m, 5H), 5.85–5.75 (m, 1H), 5.66–5.57 (m, 1H), 4.53–4.42 (m, 4H), 4.10 (p, *J* = 7.2 Hz, 4H), 3.47 (t, *J* = 6.4 Hz, 2H), 2.17 (q, *J* = 7.1 Hz, 2H),

1.78–1.64 (m, 2H), 1.33 (t, J = 7.1 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 138.47, 135.58, 128.34, 127.59, 127.52, 124.88 (d, J = 6.7 Hz), 72.88, 69.48, 67.99 (d, J = 5.1 Hz), 63.67 (d, J = 6.6 Hz), 28.89, 28.79, 16.16 (d, J = 6.7 Hz); ^{31}P NMR (162 MHz, $CDCl_3$) δ -0.83; IR: 2982, 2936, 2855, 1453, 1392, 1367, 1263, 1166, 1099, 1024, 967, 845, 800, 736, 698, 610, 515; MS (ESI) m/z: 365.1 $(M+Na)^+$; HRMS (ESI) m/z Calcd for $C_{17}H_{31}NO_5P$ $(M+NH_4)^+$: 360.1934, found 360.1932.

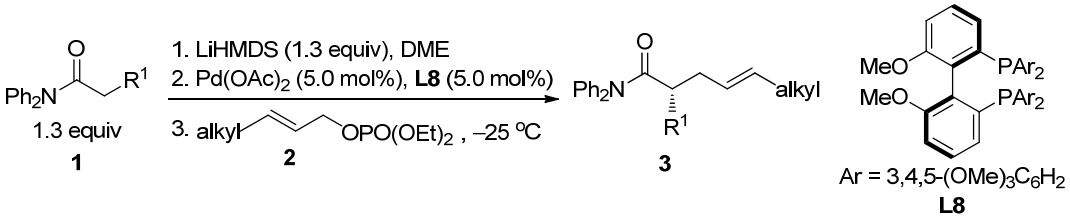


2i

(*E*)-6-((tert-butyldimethylsilyl)oxy)hex-2-en-1-yl diethyl phosphate: Procedure was followed as the preparation of **2a** on a 2.6 mmol scale, a colorless oil (0.95g, 99%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.87–5.76 (m, 1H), 5.68–5.57 (m, 1H), 4.48 (t, J = 7.2 Hz, 2H), 4.11 (q, J = 7.3 Hz, 4H), 3.61 (t, J = 6.3 Hz, 2H), 2.13 (dd, J = 14.7, 7.2 Hz, 2H), 1.66–1.56 (m, 2H), 1.34 (t, J = 7.1 Hz, 6H), 0.89 (s, 9H), 0.04 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 136.01, 124.62 (d, J = 7.0 Hz), 68.10 (d, J = 4.7 Hz), 63.70 (d, J = 5.8 Hz), 62.36, 31.87, 28.49, 25.93, 16.18 (d, J = 6.5 Hz), -5.30; ^{31}P NMR (162 MHz, $CDCl_3$) δ -0.83; IR: 2930, 1470, 1254, 1098, 967, 834, 774, 662, 526; MS (ESI) m/z: 389.1 $(M+Na)^+$; HRMS (ESI) m/z Calcd for $C_{16}H_{39}NO_5PSi$ $(M+NH_4)^+$: 384.233, found 384.2327.

5 General experimental procedure and characterization of products

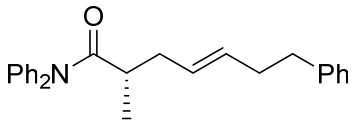
5.1 General experimental procedure



A dry Schlenk tube was flame dried and flushed with Argon. LiCl (8.4 mg), amide **1** (0.26 mmol) and DME (1.8 mL) were added into the dry Schlenk tube. LiHMDS (1.0 M in THF, 0.26 mL, 0.26 mmol) were added at 0 °C, and stirred at room temperature for 30 min. In a separated flushed flask, $Pd(OAc)_2$ (2.25 mg, 0.01 mmol), **L8** (9.43, 0.01 mmol) and DME (1.0 mL) were added. The resulting mixture was stirred at room temperature for 30 min. Then the in-situ generated catalyst was added to the enolate solution. The allylic substrates **2** (0.2 mmol) was then added and the mixture was stirred at -25 ^{o}C. After the reaction was complete (monitored by TLC), the reaction mixture was quenched by H_2O (0.3 mL). The solution was dried (anhydrous Na_2SO_4) and then filtered through a 0.5 inch plug of silica gel (eluting with EtOAc) to remove the solid. The crude reaction mixture was concentrated under reduced pressure. The resulting crude product was purified by flash column chromatography on silica gel (eluting with petroleum ether/ethyl acetate 10/1) to yield the product.

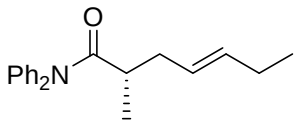
Special Note: ^{13}C NMR spectra of products **3** show broad peaks around 125–130 ppm, which was assigned as peaks of phenyl groups on nitrogen of the amide. To better understand these phenomena, the ^{13}C NMR spectrum of *N*,*N*-diphenylpropionamide **1a** was measured at 50 ^{o}C and -30 ^{o}C respectively. It revealed that the broad peaks around 125–130 ppm were observed for ^{13}C NMR spectrum measured at 50 ^{o}C, while six single peaks around 125–130 ppm were observed for ^{13}C NMR spectrum measured at -30 ^{o}C (these spectra was included in the spectra section). These phenomena may attributed to rapid rotation of two phenyl groups on nitrogen of the amide at room temperature and 50 ^{o}C, while the rotation was significantly slowed down at -30 ^{o}C.

5.2 characterization of products



3a

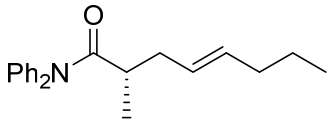
colorless oil, 69.5 mg, yield: 95%, 95:5 er. $[\alpha]_D^{31}$ = 63.6 (1.0, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.40–7.14 (m, 15H), 5.58–5.47 (m, 1H), 5.40–5.29 (m, 1H), 2.71–2.64 (m, 2H), 2.59 (dt, *J* = 13.1, 6.6 Hz, 1H), 2.43 (dt, *J* = 14.7, 7.5 Hz, 1H), 2.33 (dd, *J* = 14.9, 7.2 Hz, 2H), 2.09–1.98 (m, 1H), 1.10 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ 176.61, 143.00, 141.95, 131.81, 129.68, 128.80, 128.42, 128.31, 127.97, 126.49, 125.81, 37.81, 37.72, 35.98, 34.52, 17.68; IR: 3062, 3026, 2969, 2930, 2849, 1669, 1591, 1490, 1452, 1379, 1254, 968, 755, 693, 620 cm^{-1}; MS (ESI) m/z: 370.1 $(M+H)^+$, 392.1 $(M+Na)^+$; HRMS (ESI) m/z Calcd for $C_{26}H_{28}NO$ $(M+H)^+$: 370.2165, found 370.2164; HPLC (Chiralpak OD-H, Hexane:*i*-Propanol = 99:1,1 mL/min, 214 nm): t_{major} = 21.22 min, t_{minor} = 24.70 min.



3b

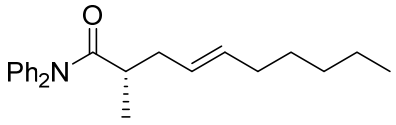
colorless oil, 48.9 mg, yield: 84%, 95.7:4.3 er. $[\alpha]_D^{31}$ = 97.5 (1.0, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.26 (dd, *J* = 28.0, 20.2 Hz, 10H), 5.59–5.49 (m, 1H), 5.37–5.27 (m, 1H), 2.75–2.55 (m, 1H), 2.52–2.37 (m, 1H), 2.11–1.96 (m, 3H), 1.14 (d, *J* = 6.7 Hz, 3H), 1.00 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.68, 143.05, 134.33, 128.83, 126.19, 37.88, 37.84, 25.59, 17.73, 13.80; IR; 3063, 3029, 2962, 2931, 2873, 2850, 1669, 1591, 1490, 1452, 1379, 1256, 1162, 1074, 1029, 967, 755, 691, 665, 649, 620 cm^{-1}; MS (ESI) m/z: 294.1 $(M+H)^+$, 316.1 $(M+Na)^+$; HRMS (ESI) m/z

Calcd for $C_{20}H_{24}NO$ $(M+H)^+$: 294.1852, found 294.1852; HPLC (Chiralpak OD-H, Hexane:*i*-Propanol = 99:1, 1 mL/min, 214 nm): t_{major} = 9.88 min, t_{minor} = 11.17 min.



3c

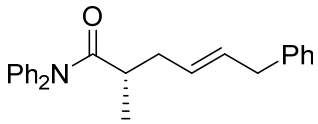
colorless oil, 56.9 mg, yield: 92%, 96:4 er. $[\alpha]_D^{31}$ = 79.5 (1.0, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.34-7.22 (m, 10H), 5.54–5.44 (m, 1H), 5.38–5.28 (m, 1H), 2.75–2.55 (m, 1H), 2.52–2.30 (m, 1H), 2.15–1.95 (m, 3H), 1.48–1.34 (m, 2H), 1.14 (d, *J* = 6.7 Hz, 3H), 0.91 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.68, 143.03, 132.67, 129.59, 128.77, 127.58, 127.35, 126.48, 126.23, 125.89, 125.48, 37.86, 37.85, 34.74, 22.61, 17.74, 13.78; IR: 3063, 3037, 2959, 2929, 2872, 2841, 1671, 1591, 1490，1452, 1379, 1254, 967, 755, 691, 620 cm^{-1}; MS (ESI) m/z: 308.1 $(M+H)^+$, 330.1 $(M+Na)^+$; HRMS (ESI) m/z Calcd for $C_{21}H_{26}NO$ $(M+H)^+$: 308.2009, found 308.2009; HPLC (Chiralpak OD-H, Hexane:*i*-Propanol = 99:1, 1 mL/min, 214 nm): t_{major} = 9.58 min, t_{minor} = 10.58 min.



3d

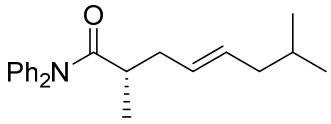
colorless oil, 55.0 mg, yield: 82%, 94.6:5.4 er. $[\alpha]_D^{31}$ = 76.3 (1.0, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.33-7.21 (m, 10H), 5.54–5.43 (m, 1H), 5.37–5.26 (m, 1H), 2.75–2.52 (m, 1H), 2.53–2.35 (m, 1H), 2.11–1.93 (m, 3H), 1.47–1.21 (m, 6H), 1.12 (d, *J* = 6.7 Hz, 3H), 0.88 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.68, 143.04, 132.92, 129.60, 128.77, 127.69, 127.12, 126.52, 125.95, 37.87, 32.62, 31.45, 29.20, 22.58, 17.75, 14.08; IR: 3063, 3037, 2957, 2925, 2872, 2854, 1672, 1591, 1490,

1452，1378, 1253, 1160, 1074, 968, 902, 755, 699, 691, 665, 649, 620 cm^{-1}; MS (ESI) m/z: 336.2 $(M+H)^+$, 358.1 $(M+Na)^+$; HRMS(ESI) m/z Calcd for $C_{23}H_{30}NO$ $(M+H)^+$: 336.2322, found 336.2322; HPLC (Chiralpak IG, Hexane:*i*-Propanol = 90:10, 0.7 mL/min,214 nm): t_{major} = 13.12 min, t_{minor} = 14.82 min.



3e

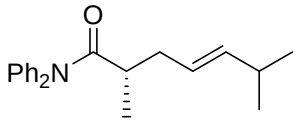
colorless oil, 59.5 mg, yield: 83%, 95.5:4.5 er. $[\alpha]_D^{31}$ = 59.1 (1.0, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.39–7.12 (m, 15H), 5.73–5.62 (m, 1H), 5.46–5.35 (m, 1H), 3.34 (d, *J* = 6.6 Hz, 2H), 2.69–2.55 (m, 1H), 2.55–2.42 (m, 1H), 2.13–1.97 (m, 1H), 1.13 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.44, 142.97, 140.59, 131.69, 131.22, 128.84, 128.60, 128.43, 125.99, 39.01, 37.79, 37.71, 17.97; IR：3055，3028，2975，2960，2926，2868，2834，1658，1590，1490，1452，1427, 1386，1277，1180，1154，1072，1028，969，907，773，747，715，692，648，621 cm^{-1}; MS (ESI) m/z: 356.1 $(M+H)^+$, 378.1 $(M+Na)^+$; HRMS (ESI) m/z Calcd for $C_{25}H_{26}NO$ $(M+H)^+$: 356.2009, found 356.2007; HPLC (Chiralpak OD-H, Hexane:*i*-Propanol = 99:1, 1 mL/min, 214 nm): t_{major} = 18.60 min, t_{minor} = 22.79 min.



3f

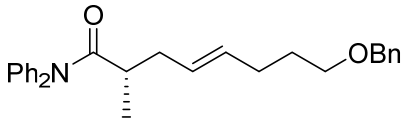
colorless oil, 57.8 mg, yield: 89%, 96.4:3.6 er. $[\alpha]_D^{31}$ = 68.5 (0.8, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.33-7.22 (m,, 10H), 5.46 (dt, *J* = 14.3, 7.1 Hz, 1H), 5.35–5.25 (m, 1H), 2.73–2.54 (m, 1H), 2.45 (dt, *J* = 14.7, 7.6 Hz, 1H), 2.04 (dt, *J* = 13.2, 6.6 Hz, 1H), 1.99–1.80 (m, 2H), 1.70–1.50 (m, 1H), 1.12 (d, *J* = 6.7 Hz, 3H), 0.87 (d, *J* = 6.7

Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.68, 142.99, 131.66, 131.58, 128.78, 128.31, 126.49, 42.06, 37.88, 37.82, 28.43, 22.42, 22.32, 17.75; IR: 3063, 3038, 2955, 2930, 2899, 2869, 2837, 1671, 1591, 1490, 1452, 1379, 1252, 1163, 969, 755, 700, 691, 665, 649， 620 cm^{-1}; MS (ESI) m/z: 322.1 $(M+H)^+$, 344.1 $(M+Na)^+$; HRMS (ESI) m/z Calcd for $C_{22}H_{28}NO$ $(M+H)^+$: 322.2165, found 322.2164; HPLC (Chiralpak OD-H, Hexane:*i*-Propanol = 99:1, 1mL/min, 214nm): t_{major} = 9.51 min, t_{minor} = 10.63 min.



3g

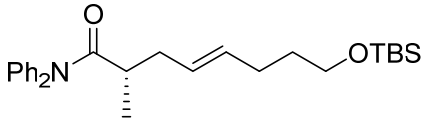
colorless oil, 55.2 mg, yield: 90%, 96:4 er. $[\alpha]_D^{25}$ = 94.9 (1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50–7.09 (m, 10H), 5.47 (dd, *J* = 15.3, 6.5 Hz, 1H), 5.33–5.22 (m, 1H), 2.68–2.55 (m, 1H), 2.50–2.38 (m, 1H), 2.26 (dq, *J* = 13.4, 6.7 Hz, 1H), 2.08–1.95 (m, 1H), 1.13 (d, *J* = 6.7 Hz, 3H), 0.98 (d, *J* = 6.8 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.73, 142.99, 139.84, 134.86, 128.73, 126.49, 124.14, 119.82, 37.92, 31.03, 22.62, 22.55, 17.87; IR: 2958, 2930, 1671, 1591, 1490, 1452, 1379, 1257, 970, 756, 692, 620, 522; MS (ESI) m/z: 308.2 $(M+H)^+$, 330.1 $(M+Na)^+$; HRMS (ESI) m/z Calcd for $C_{21}H_{26}NO$ $(M+H)^+$: 308.2009, found 308.2011; HPLC (Chiralpak OD-H, Hexane:*i*-Propanol = 99:1, 1 mL/min, 214 nm): t_{major} = 9.25 min, t_{minor} = 10.79 min.



3h

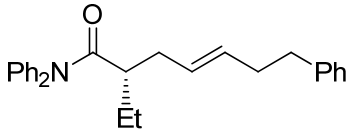
colorless oil, 74.9 mg, yield: 91%, 96:4 er. $[\alpha]_D^{27}$ = 60.80 (1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52–7.06 (m, 15H), 5.53–5.42 (m, 1H), 5.38–5.27 (m, 1H), 4.48 (s, 2H), 3.46 (t, *J* = 6.5 Hz, 2H), 2.73–2.52 (m, 1H), 2.51–2.35 (m, 1H), 2.10 (dd, *J* =

14.4, 7.1 Hz, 2H), 2.07–1.98 (m, 1H), 1.76–1.60 (m, 2H), 1.11 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.61, 143.01, 138.61, 132.02, 128.82, 128.37, 127.80, 127.64, 127.52, 126.49, 72.86, 69.75, 37.81, 37.79, 29.52, 29.22, 17.72; IR: 2932, 2850, 1670, 1590, 1490, 1452, 1378, 1258, 1101, 968, 756, 697, 620, 522 cm^{-1}; MS (ESI) m/z: 414.2 $(M+H)^+$, 436.2 $(M+Na)^+$; HRMS (ESI) m/z Calcd for $C_{28}H_{32}NO_2$ $(M+H)^+$: 414.2428, found 414.2425; HPLC (Chiralpak OD-H, Hexane:*i*-Propanol = 95:5, 0.7 mL/min, 214 nm): t_{major} = 17.53min, t_{minor} = 19.26 min.



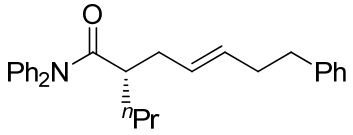
3i

colorless oil, 76.2 mg, yield: 88%, 92.8:7.2 er. $[\alpha]_D^{27}$ = 55.3 (1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33-7.21 (m, 10H), 5.54–5.44 (m, 1H), 5.39–5.27 (m, 1H), 3.60 (t, *J* = 6.4 Hz, 2H), 2.70–2.55 (m, 1H), 2.51–2.35 (m, 1H), 2.13–1.97 (m, 3H), 1.67–1.51 (m, 2H), 1.12 (d, *J* = 6.7 Hz, 3H), 0.89 (s, 9H), 0.04 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.63, 143.00, 132.23, 129.63, 128.77, 127.52, 126.51, 62.59, 37.81, 32.59, 28.86, 25.97, 18.34, 17.68, -5.24; IR: 2929, 2855, 1673, 1592, 1491, 1380, 1251, 1098, 968, 834, 774, 755, 700, 620, 521 cm^{-1}; MS (ESI) m/z: 438.3 $(M+H)^+$, 460.2 $(M+Na)^+$; HRMS (ESI) m/z Calcd for $C_{27}H_{40}NO_2Si$ $(M+H)^+$: 438.2823, found 438.2819; HPLC (Chiralpak OD-H, Hexane:*i*-Propanol=99:1, 1.0 mL/min, 214 nm): t_{major} = 9.63 min, t_{minor} = 11.16 min.



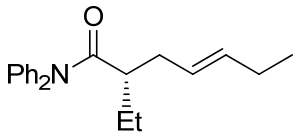
3j

colorless oil, 68.6 mg, yield: 91%, 94.7:5.3 er. $[\alpha]_D^{31}$ = -22.2 (1.0, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.50–7.14 (m, 15H), 5.62–5.51 (m, 1H), 5.46–5.34 (m, 1H), 2.72 (t, *J* = 7.8 Hz, 2H), 2.51 (dq, *J* = 8.3, 5.6 Hz, 1H), 2.45–2.32 (m, 3H), 2.25–2.08 (m, 1H), 1.75 (tt, *J* = 15.4, 7.5 Hz, 1H), 1.57–1.37 (m, 1H), 0.95 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.93, 143.03, 141.94, 131.72, 129.61, 129.26, 128.82, 128.43, 128.32, 128.10, 127.74, 126.55, 125.82, 44.65, 36.30, 35.96, 34.56, 25.97, 12.12; IR: 3026，2961, 2927, 2854, 1667, 1591, 1490, 1453, 1385, 1275, 1239, 969, 754, 693, 619 cm^{-1}; MS (ESI) m/z: 384.2 $(M+H)^+$, 406.1 $(M+Na)^+$; HRMS (ESI) m/z Calcd for $C_{27}H_{30}NO$ $(M+H)^+$: 384.2322, found 384.2321; HPLC (Chiralpak IG, Hexane:*i*-Propanol = 90:10, 0.7 mL/min, 214 nm): t_{major} = 19.09 min, t_{minor} = 25.35 min.



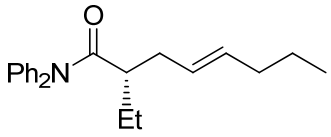
3k

colorless oil, 76.2 mg, yield: 97%, 95:5 er. $[\alpha]_D^{31}$ = -27.6 (1.0, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.53–7.11 (m, 15H), 5.61–5.50 (m, 1H), 5.45–5.34 (m, 1H), 2.71 (t, *J* = 7.8 Hz, 2H), 2.66–2.50 (m, 1H), 2.49–2.31 (m, 3H), 2.13 (dt, *J* = 13.2, 6.4 Hz, 1H), 1.82–1.65 (m, 1H), 1.47–1.24 (m, 3H), 0.86 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.07, 143.01, 141.94, 134.77, 131.72, 129.59, 129.22, 128.81, 128.43, 128.32, 128.11, 127.74, 126.54, 125.82, 43.00, 36.51, 35.96, 35.21, 34.54, 20.79, 14.31; IR: 3027, 2956, 2927, 2856, 1668, 1591, 1490, 1452, 1383, 1259, 970, 754, 697, 662，620 cm^{-1}; MS (ESI) m/z: 398.1 $(M+H)^+$, 420.1 $(M+Na)^+$; HRMS (ESI) m/z Calcd for $C_{28}H_{32}NO$ $(M+H)^+$: 398.2478, found 398.2477; HPLC (Chiralpak IC, Hexane:*i*-Propanol = 99:1, 1 mL/min, 214 nm): t_{minor} = 30.59 min, t_{major} = 33.32 min.



3l

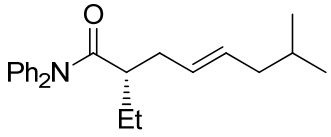
colorless oil, 45.8 mg, yield: 74%, 92.4:7.6 er. $[\alpha]_D^{28}$ = -7.7 (1.0, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.49–7.11 (m, 10H), 5.60–5.48 (m, 1H), 5.39–5.28 (m, 1H), 2.58–2.44 (m, 1H), 2.44–2.34 (m, 1H), 2.17–2.00 (m, 3H), 1.74 (td, *J* = 14.9, 7.7 Hz, 1H), 1.54–1.39 (m, 1H), 1.01 (t, *J* = 7.4 Hz, 3H), 0.94 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.99, 134.22, 129.54, 128.27, 126.55, 126.31, 44.71, 36.38, 26.09, 25.61, 13.77, 12.10; IR: 2961, 2930, 2873, 1667, 1591, 1490, 1454, 1383, 1275, 1239, 1161, 1074, 1029, 968, 754, 693, 662, 619, 521, 464 cm^{-1}; MS (ESI) m/z: 308.2 $(M+H)^+$, 330.1 $(M+Na)^+$; HRMS (ESI) m/z Calcd for $C_{21}H_{26}NO$ $(M+H)^+$: 308.2009, found 308.2003; HPLC (Chiralpak IG, Hexane:*i*-Propanol = 90:10, 0.7 mL/min, 214 nm): t_{major} = 15.69 min, t_{minor} = 20.15 min.



3m

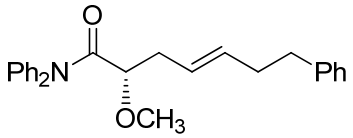
colorless oil, 50.1 mg, yield: 80%, 91.3:8.7 er. $[\alpha]_D^{28}$ = -18.8 (1.0, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ 7.48–7.06 (m, 10H), 5.52–5.42 (m, 1H), 5.37–5.27 (m, 1H), 2.53–2.44 (m, 1H), 2.43–2.33 (m, 1H), 2.15–2.05 (m, 1H), 1.99 (dd, *J* = 14.0, 7.1 Hz, 2H), 1.84–1.66 (m, 1H), 1.52–1.32 (m, 3H), 0.91 (q, *J* = 7.7 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.97, 143.05, 132.56, 129.26, 128.76, 127.45, 126.56, 44.69, 36.37, 34.76, 26.06, 22.60, 13.79, 12.08; IR: 2959, 2927, 2872, 1668, 1591, 1490, 1453, 1382, 1274, 1239, 1161, 1074, 1029, 968, 754, 693, 662, 618, 520 cm^{-1}; MS (ESI) m/z: 322.2 $(M+H)^+$, 344.2 $(M+Na)^+$; HRMS (ESI) m/z Calcd for $C_{22}H_{28}NO$ $(M+H)^+$:

322.2165, found 322.2159; HPLC (Chiralpak IE, Hexane:*i*-Propanol = 95:5, 0.7 mL/min, 214 nm): t_{major} = 24.67 min, t_{minor} = 26.57 min.



3n

colorless oil, 47.8 mg, yield: 72%, 92.9:7.1 er. $[\alpha]_D^{28}$ = -34.4 (1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55–6.98 (m, 10H), 5.46 (dt, *J* = 14.3, 6.9 Hz, 1H), 5.37–5.27 (m, 1H), 2.54–2.45 (m, 1H), 2.40 (dt, *J* = 14.6, 7.4 Hz, 1H), 2.22–2.07 (m, 1H), 1.98–1.83 (m, 2H), 1.74 (tt, *J* = 15.0, 7.5 Hz, 1H), 1.61 (tt, *J* = 13.2, 6.7 Hz, 1H), 1.53–1.41 (m, 1H), 0.99–0.78 (m, 9H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.93, 143.05, 131.46, 129.23, 128.77, 128.40, 126.54, 44.68, 42.06, 36.32, 28.44, 26.04, 22.40, 22.33, 12.06; IR: 2957, 2926, 2870, 2837, 1669, 1592, 1490, 1454, 1383, 1273, 1239, 1162, 1074, 1029, 970, 754, 693, 662, 619, 520 cm^{-1}; MS (ESI) m/z: 336.2 $(M+H)^+$, 358.2 $(M+Na)^+$; HRMS (ESI) m/z Calcd for $C_{23}H_{30}NO$ $(M+H)^+$: 336.2322, found 336.2315; HPLC (Chiralpak IE, Hexane:*i*-Propanol = 95:5, 0.7 mL/min, 214 nm): t_{major} = 21.89 min, t_{minor} = 23.85 min.

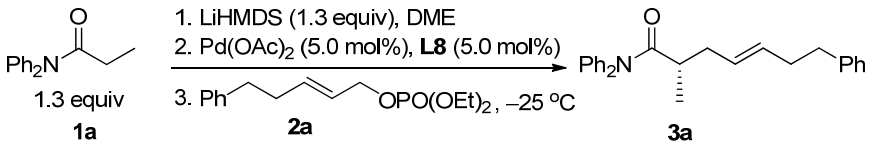


3o

colorless oil, 59.7 mg, yield: 79%, 94.4:5.6 er. $[\alpha]_D^{26}$ = 54.4 (1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46–7.11 (m, 15H), 5.58–5.47 (m, 1H), 5.45–5.35 (m, 1H), 3.92 (t, *J* = 6.4 Hz, 1H), 3.30 (s, 3H), 2.65 (dd, J = 8.5, 6.8 Hz, 2H), 2.53–2.35 (m, 2H), 2.32 (dd, *J* = 14.9, 7.1 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.52, 141.86, 132.95, 128.96, 128.44, 128.30, 125.81, 125.36, 78.31, 57.06, 35.78, 35.65, 34.47; IR:

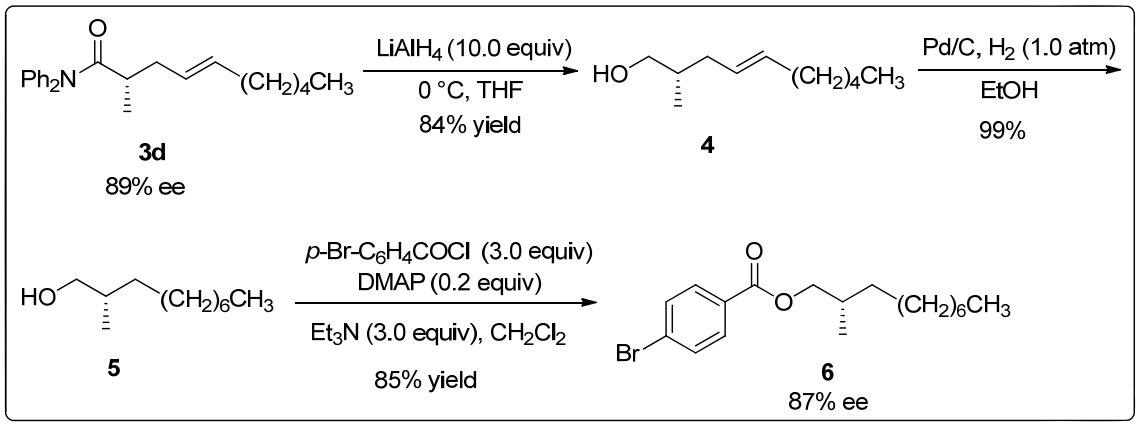
3061, 2923, 1679, 1591, 1490, 1379, 1270, 1114, 969, 753, 698, 618, 568 cm^{-1}; MS (ESI) m/z: 386.2 $(M+H)^+$, 408.2 $(M+Na)^+$; HRMS (ESI) m/z Calcd for $C_{26}H_{28}NO_2$ $(M+H)^+$: 386.2115, found 386.2125; HPLC (Chiralpak OD-H, Hexane:*i*-Propanol = 90:10, 1.0 mL/min, 214 nm): t_{major}= 13.04 min, t_{minor} = 17.16 min.

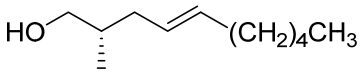
6. Pd-Catalyzed Asymmetric Allylic Alkylation of Amide 1a with Allyl Reagents 2a on 1.0 mmol Scale.



A dry Schlenk tube was flame dried and flushed with Argon. LiCl (42.9 mg), amide **1a** (292.9 mg, 1.3 mmol) and DME (9.0 mL) were added into the dry Schlenk tube. LiHMDS (1.0 M in THF, 1.3 mL, 1.3 mmol) were added at 0 °C, and stirred at room temperature for 30 min. In a separated flushed flask, $Pd(OAc)_2$ (11.2 mg, 0.05 mmol), **L8** (47.2 mg, 0.05 mmol) and DME (5.0 mL) were added. The resulting mixture was stirred at room temperature for 30 min. Then the in-situ generated catalyst was added to the enolate solution. Allylic substrate **2a** (298.3 mg, 1.0 mmol) was then added and the mixture was stirred at -25 °C. After the reaction was complete (monitored by TLC), the reaction mixture was quenched by H_2O (1.0 mL). The solution was dried (anhydrous Na_2SO_4) and then filtered through a 2.0 inch plug of silica gel (eluting with EtOAc) to remove the solid. The crude reaction mixture was concentrated under reduced pressure. The resulting crude product was purified by flash column chromatography on silica gel (eluting with petroleum ether/ethyl acetate 10/1) to yield the product **3a** (0.35 g, 93%, 93.5:6.5 er).

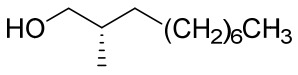
7. Transformation of the allylated product 3d and its determination of absolute configuration





4

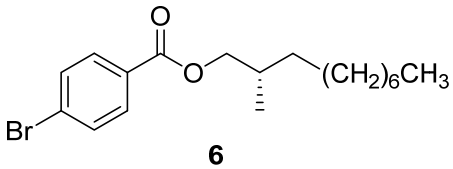
To a solution of $LiAlH_4$ (62.2 mg,1.64 mmol) in THF (12 mL) at 0 °C, was added dropwise a solution of **3d** (50 mg, 0.164 mmol, 89% ee) in THF (4 mL). After stirring for 1 h, the reaction was quenched by the addition of water (3 mL). The mixture was filtered through Celite, which was washed with THF. The filtrate was concentrated, and the residue was purified by flash column chromatography on silica gel (eluting with petroleum ether/ethyl acetate 10/1) to furnished the product **4**[4] (colorless oil, 21.4 mg, 84%). $[\alpha]_D^{21}$ = -0.62 (1.0, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.60–5.26 (m, 2H), 3.50 (dd, *J* = 10.5, 6.1 Hz, 1H), 3.42 (dd, *J* = 10.6, 6.2 Hz, 1H), 2.16–2.03 (m, 1H), 1.99 (dd, *J* = 13.3, 6.5 Hz, 2H), 1.94–1.83 (m, 2H), 1.68 (dq, *J* = 13.1, 6.6 Hz, 1H), 1.42–1.20 (m, 6H), 0.89 (dd, *J* = 10.3, 6.9 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 132.36, 127.95, 67.91, 36.56, 35.96, 32.56, 31.37, 29.24, 22.51, 16.41, 14.06.



5

Palladium-charcoal (2.0 mg, 10%) was added to a solution of **4** (18 mg, 0.106 mmol) in EtOH (1mL). The suspension was stirred under H_2 (balloon) for 24 h at room temperature. The catalyst was filtered off through Celite, and the solid was washed

with EtOH. The combined solution was concentrated in vacuo to provide **5**[5] (colorless oil, 18 mg, 99%). $[\alpha]_D^{26}$ = -7.2 (1.0, CH_2Cl_2){(*S*)-**5**: $[\alpha]_D$ = -9.4 (1.0, CH_2Cl_2)}[5]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.51 (dd, *J* = 10.5, 5.8 Hz, 1H), 3.41 (dd, *J* = 10.4, 6.6 Hz, 1H), 1.60 (dt, *J* = 12.6, 6.1 Hz, 1H), 1.47 (s, 1H), 1.40–1.05 (m, 14H), 0.97–0.83 (m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 68.40, 35.74, 33.13, 31.90, 29.94, 29.61, 29.33, 26.98, 22.68, 16.58, 14.12.

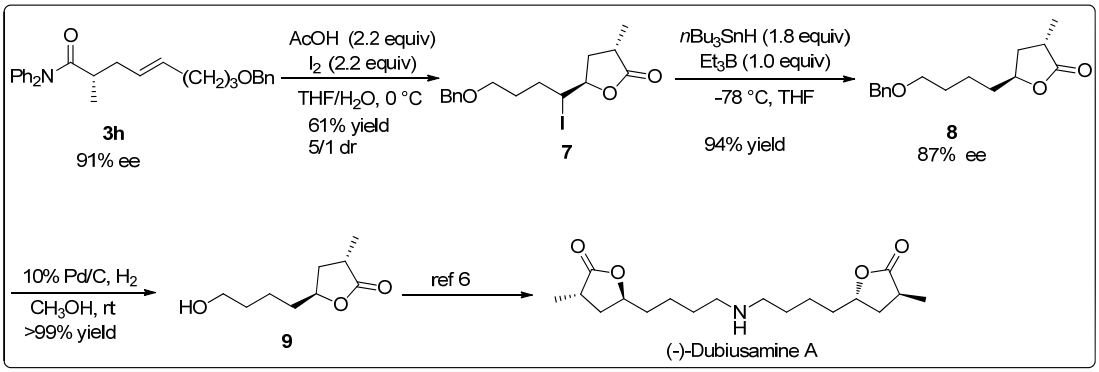


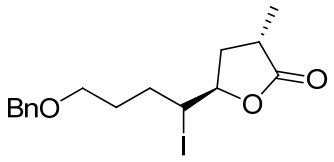
6

The alcohol **5** (15 mg, 0.087 mmol) and DCM (1.5 mL) was added to a dry Schlenk tube, and then Et_3N (36 μL, 0.26mmol), DMAP (2.1 mg, 0.0174 mmol), and 4-bromobenzoyl chloride (57.3 mg, 0.26 mmol) were added. The mixture was stirred at room temperature until **5** was fully consumed (monitored by TLC). After the reaction mixture was diluted with DCM (3 mL), the organic phase was washed with 1M HCl (3 mL), sat. aq. Na_2CO_3 solution (3 mL), and brine (3 mL). After the organic phase was dried over anhydrous $MgSO_4$, the solvent was removed under reduced pressure and the residue was purified by flash column chromatography on silica gel (eluting with petroleum ether/ethyl acetate 10/1) to give the product **6** (colorless oil, 36.7 mg, 85%, 93.7:6.3 er). $[\alpha]_D^{26}$ = 1.5 (1.0, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, *J* = 8.6 Hz, 2H), 7.60 (d, *J* = 8.5 Hz, 2H), 4.22 (dd, *J* = 10.7, 5.8 Hz, 1H), 4.12 (dd, *J* = 10.7, 6.8 Hz, 1H), 2.11–1.78 (m, 1H), 1.48–1.25 (m, 14H), 1.03 (d, *J* = 6.7 Hz, 3H), 0.90 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.93, 131.66, 131.05, 129.40, 127.88, 70.14, 33.42, 32.65, 31.89, 29.82, 29.55, 29.30, 26.84, 22.67, 17.03, 14.12; IR: 2923, 2853, 1721, 1591, 1266, 1172, 1012, 847, 756, 722, 469; MS (EI): m/z (%) 354 (M^+, 0.4), 200 (11), 185 (71), 183 (73), 155 (29), 141 (1), 113 (3), 99 (5),

85 (12), 79 (3), 71 (17), 57 (66), 43 (40); HRMS (EI) Calcd for $C_{18}H_{27}O_2Br$ $[M]^+$: 354.1194, found 354.1187; HPLC (Chiralpak OJ-H, Hexane: *i*-Propanol=98:2, 0.7 mL/min, 214nm): t_{minor} = 6.87 min, t_{minor} = 7.52 min.

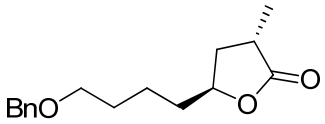
8. Enantioselective formal synthesis of enantiomer of Dubiusamine A





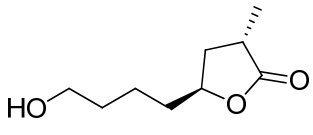
To a solution of **3h** (70 mg, 0.17mmol) and AcOH (21.3 μL, 0.37mmol) in THF/H_2O (2:1, 2.4 mL) was added iodine (93.9 mg, 0.37 mmol) in THF (1.6 mL) at 0 °C. After being stirred at 0 °C for 48 h, the reaction mixture was poured into ice-cold 40% $NaHSO_3$ and extracted with Et_2O. The combined organic layers were washed with 1 N HCl, saturated $NaHCO_3$, and saturated NaCl. The organic phase was dried over anhydrous Na_2SO_4, filtered, and concentrated under vacuum. The diastereoselectivity was determined to be 5/1 by ^1H NMR spectroscopy. Then, the resulting crude product was purified by flash column chromatography on silica gel (eluting with petroleum ether/ethyl acetate 10/1) to yield the pure major isomer of **7** (colorless oil, 40.2 mg, 61%) and the minor isomer was completely removed. $[\alpha]_D^{28}$ = -11.6 (1.0, $CHCl_3$); ^1H NMR (600 MHz, $CDCl_3$) δ 7.39–7.25 (m, 5H), 4.53–4.46 (m, 2H), 4.41 (td, *J* = 7.7, 5.6 Hz, 1H), 4.13 (ddd, *J* = 10.1, 7.9, 3.5 Hz, 1H), 3.56–3.46 (m, 2H), 2.76 (dp, *J* =

9.5, 7.3 Hz, 1H), 2.35 (ddd, *J* = 13.5, 9.6, 5.5 Hz, 1H), 2.17–2.08 (m, 1H), 2.08–2.00 (m, 1H), 1.94 (dtd, *J* = 19.9, 10.1, 6.0 Hz, 1H), 1.90–1.82 (m, 1H), 1.70 (ddt, *J* = 15.6, 11.9, 5.8 Hz, 1H), 1.30 (d, *J* = 7.4 Hz, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 179.29, 138.29, 128.39, 127.65, 127.61, 80.19, 72.96, 69.03, 39.07, 35.80, 34.48, 32.76, 29.41, 16.41; IR: 2935, 2855, 1772, 1495, 1453, 1360, 1174, 1096, 1022, 919, 736, 697, 597, 459 cm^{-1}; MS (ESI) m/z: 389.0 $(M+H)^+$, 406.1 $(M+NH_4)^+$; HRMS (ESI) m/z Calcd for $C_{16}H_{25}INO_3$ $(M+NH_4)^+$: 406.0874, found 406.0872.



8

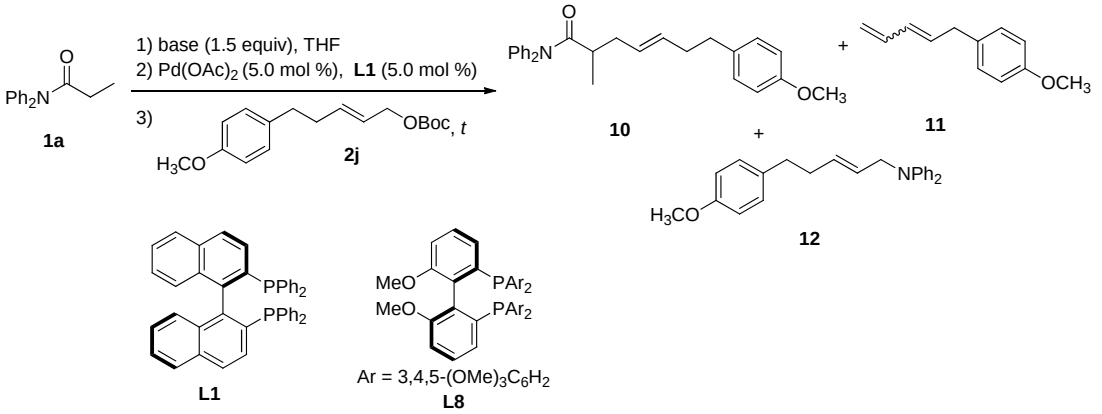
To compound **7** (40 mg, 0.10 mmol) in THF (3 mL) was added *n*Bu_3SnH (51.3 μL, 0.19 mmol) and Et_3B (1.0 M in THF, 0.1 mL, 0.1 mmol) at – 78 °C, and stirred at -78 °C for 2 hours. After quenching the reaction with saturated aqueous KF (3 mL), work-up with Et_2O (5 mL × 3) and chromatographic separation (Et_2O/hexane = 1/20) provided **8** (colorless oil, 25.4 mg, 94 %, 93.8:6.2 er). $[\alpha]_D^{26}$ = -22.2 (1.0, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43–7.23 (m, 5H), 4.55–4.45 (m, 3H), 3.47 (t, *J* = 6.3 Hz, 2H), 2.80–2.57 (m, 1H), 2.10 (ddd, *J* = 13.5, 8.9, 4.9 Hz, 1H), 1.98 (dt, *J* = 12.9, 7.5 Hz, 1H), 1.73–1.57 (m, 4H), 1.51 (ddd, *J* = 19.3, 9.6, 4.9 Hz, 2H), 1.27 (d, *J* = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 180.09, 138.44, 128.37, 127.65, 127.57, 78.33, 72.96, 69.93, 35.39, 35.20, 34.00, 29.41, 22.20, 15.90; IR: 2936, 2861, 1765, 1454, 1357, 1186, 1098, 996, 925, 736, 698, 610, 528cm^{-1}; MS (ESI) m/z: 263.1 $(M+H)^+$, 285.1 $(M+Na)^+$; HRMS (ESI) m/z Calcd for $C_{16}H_{26}NO_3$ $(M+NH_4)^+$: 280.1907, found 280.1907; HPLC (Chiralpak OD-H, Hexane:*i*-Propanol = 95:5, 0.7 mL/min, 214 nm): t_{major} = 27.66 min, t_{minor} = 29.62 min.



9

Palladium-charcoal (4 mg, 10%) was added to a solution of compound **8** (25 mg, 0.095 mmol) in EtOH (2 mL). The suspension was stirred under H_2 (balloon) for 24 h at room temperature. The catalyst was filtered off through Celite, and the solid was washed with EtOH. The combined solution was concentrated in vacuo to provide **9**. (colorless oil, 16.4 mg, quant). $[\alpha]_D^{30}$ = -29.9 (1.0, $CHCl_3$) {(*3R,5R*)-**9**: $[\alpha]_D^{22}$ = +40 (0.01, $CHCl_3$)}[6]; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.53 (tt, *J* = 7.7, 5.0 Hz, 1H), 3.67 (dd, *J* = 12.8, 6.6 Hz, 2H), 2.83–2.61 (m, 1H), 2.18–2.08 (m, 1H), 2.02 (dt, *J* = 12.8, 7.5 Hz, 1H), 1.82–1.69 (m, 2H), 1.61 (dd, *J* = 13.3, 5.9 Hz, 2H), 1.57–1.45 (m, 2H), 1.28 (d, *J* = 7.3 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 180.17, 78.37, 62.48, 35.39, 35.17, 34.02, 32.23, 21.73, 15.89; HRMS (ESI) m/z Calcd for $C_9H_{20}NO_3$ $(M+NH_4)^+$: 190.1438, found 190.1437.

9. Preliminary studies of the beta-hydride elimination for Pd-catalyzed allylic alkylation of amide 1a with allyl reagent 2j[a]

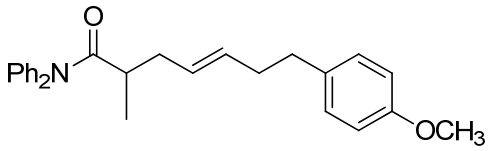


entry	base	*t* (°C)	yield (**3p**,%)[b]	yield (**10**,%)[b]	yield (**11**,%)[b]	yield(**2g**,%)[c]
1	LDA	0	76	trace	6	10

2	LDA	25	79	9	8	0
3	LDA	50	63	28	8	0
4[d]	LDA	25	78	0	4	18
5[d]	LDA	50	81	0	4	15
6[e]	LDA	50	74	14	8	4
7[f]	LDA	25	76	14	6	0
8	LiHMDS	25	97	0	0	0

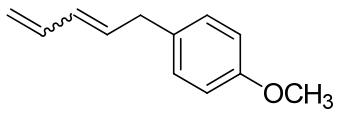
[a]Molar ratio of **1a**/**2g**/base/$Pd(OAc)_2$/**L1** = 150/100/150/5/5. [b]Isolated yield. [c]Isolated yield of **2g** recovered. [d]LiCl (1.0 equiv) added as additive. [e]**L8** used as ligand. [f]DME used as solvent.

General experimental procedure for the Pd-catalyzed allylic alkylation of amide **1a** with allyl reagent **2j** under reaction condition as shown in above table: a dry Schlenk tube was flame dried and flushed with argon. Amide **1a** (67.59 mg, 0.3 mmol) and solvent (1.7 mL) were added into the dry Schlenk tube. Base (0.3 mmol) were added at 0 °C, and stirred at room temperature for 30 min. In a separated flushed flask, $Pd(OAc)_2$ (2.25 mg, 0.01 mmol), ligand (0.01 mmol) and solvent (1.0 mL) were added. The resulting mixture was stirred at room temperature for 30 min. Then the in-situ generated catalyst was added to the enolate solution. The allylic substrate **2j** (58.48 mg, 0.2 mmol) was then added and the mixture was stirred at the corresponding temperature. After the reaction was complete (monitored by TLC), the reaction mixture was quenched by H_2O (0.3 mL). The solution was dried (anhydrous Na_2SO_4) and then filtered through a 0.5 inch plug of silica gel (eluting with EtOAc) to remove the solid. The crude reaction mixture was concentrated under reduced pressure. The resulting crude product was purified by flash column chromatography on silica gel (eluting with petroleum ether/ethyl acetate 10/1) to yield the product.



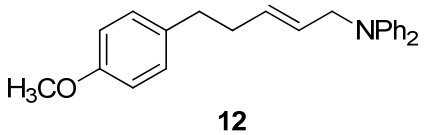
10

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38–7.18 (m, 10H), 7.08 (d, *J* = 8.4 Hz, 2H), 6.81 (d, *J* = 8.4 Hz, 2H), 5.57–5.46 (m, 1H), 5.39–5.29 (m, 1H), 3.77 (s, 3H), 2.60 (q, *J* = 8.0 Hz, 3H), 2.49–2.37 (m, 1H), 2.35–2.22 (m, 2H), 2.11–1.98 (m, 1H), 1.10 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.60, 157.71, 142.99, 134.03, 131.87, 129.26, 127.87, 113.69, 55.25, 37.80, 37.71, 35.03, 34.73, 17.64; IR: 3033, 2930, 1670, 1592, 1511, 1491, 1379, 1246, 1033, 804, 758, 698, 522cm^{-1}; MS(ESI) m/z: 400.2($M+H^+$), 422.2($M+Na^+$); HRMS(ESI) m/z Calcd for $C_{27}H_{30}NO_2(M+H)^+$: 400.2271, found 400.2267.



11[7]

E/Z = 62:38. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14–7.07 (m, 2.0H), 6.88–6.80 (m, 2.0H), 6.33 (dt, *J* = 17.0, 10.3 Hz, 0.52H), 6.17–6.03 (m, 0.84H), 5.89–5.76 (m, 0.61H), 5.14 (ddd, *J* = 65.9, 45.6, 13.7 Hz, 1.84H), 3.79 (d, *J* = 2.0 Hz, 3.0H), 3.48 (d, *J* = 8.0 Hz, 0.70H), 3.36 (d, *J* = 6.9 Hz,1.12H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.98, 136.96, 133.94, 132.07, 131.73, 131.03, 129.67, 129.52, 129.28, 117.87, 115.58, 113.89, 113.84, 55.27, 38.01, 33.00. HRMS (EI^+) Calcd for $C_{12}H_{14}O$: 174.1045, found 174.1046.



12

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 (dd, *J* = 10.6, 5.0 Hz, 4H), 6.99 (d, *J* = 8.5 Hz, 6H), 6.93 (t, *J* = 7.3 Hz, 2H), 6.78 (d, *J* = 8.5 Hz, 2H), 5.71–5.59 (m, 1H), 5.53 (dt, *J*

= 15.5, 5.0 Hz, 1H), 4.27 (d, *J* = 4.9 Hz, 2H), 3.77 (s, 3H), 2.56 (t, *J* = 7.6 Hz, 2H), 2.28 (q, *J* = 7.1 Hz, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.68, 147.88, 133.82, 131.87, 129.40, 129.15, 129.07, 126.31, 122.88, 121.70, 121.10, 120.77, 113.79, 113.63, 55.24, 53.99, 34.85, 34.42; HRMS(ESI) m/z Calcd for $C_{24}H_{26}NO$ $(M+H)^+$: 344.2009, found 344.2002.

10. References

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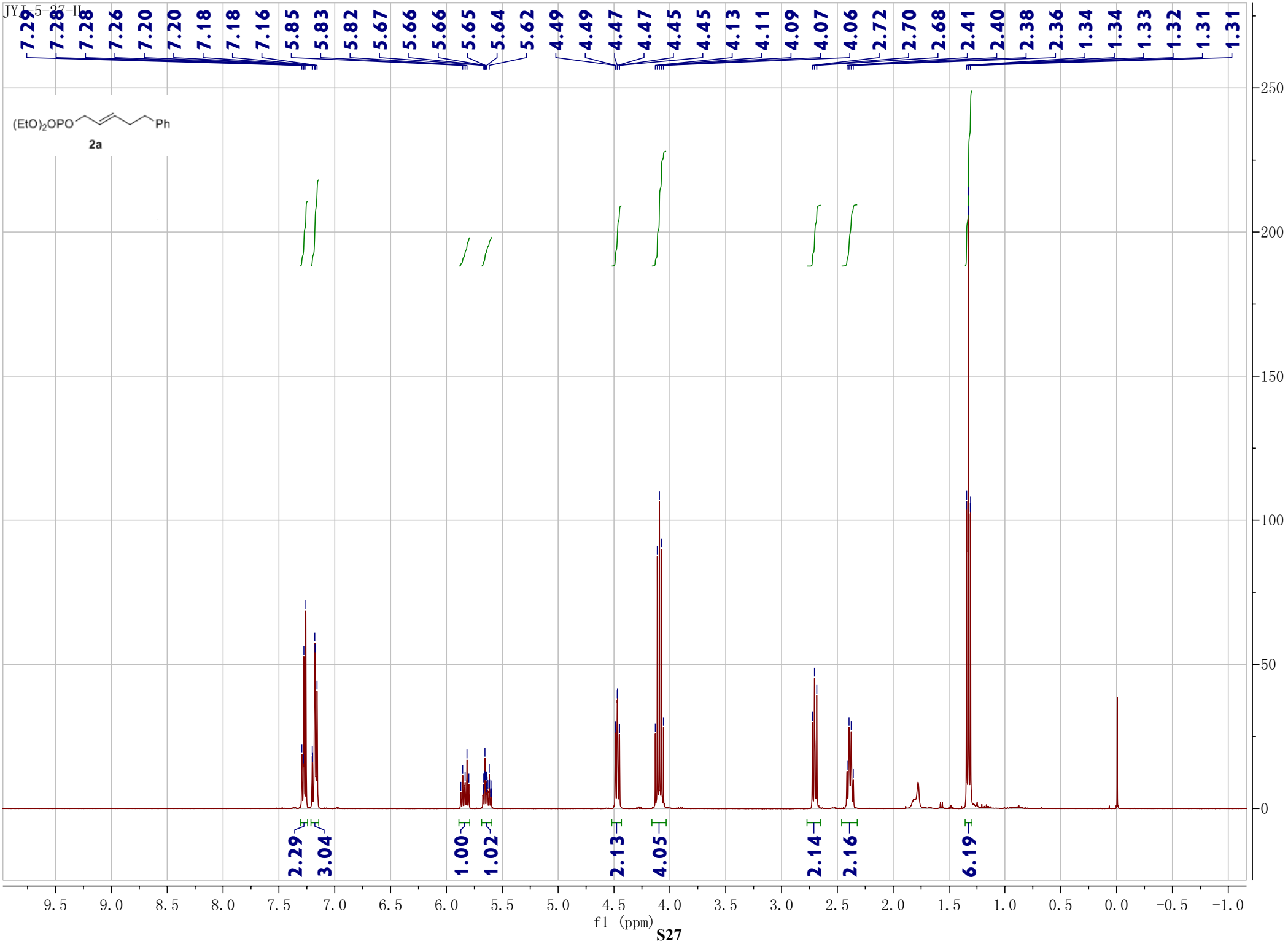
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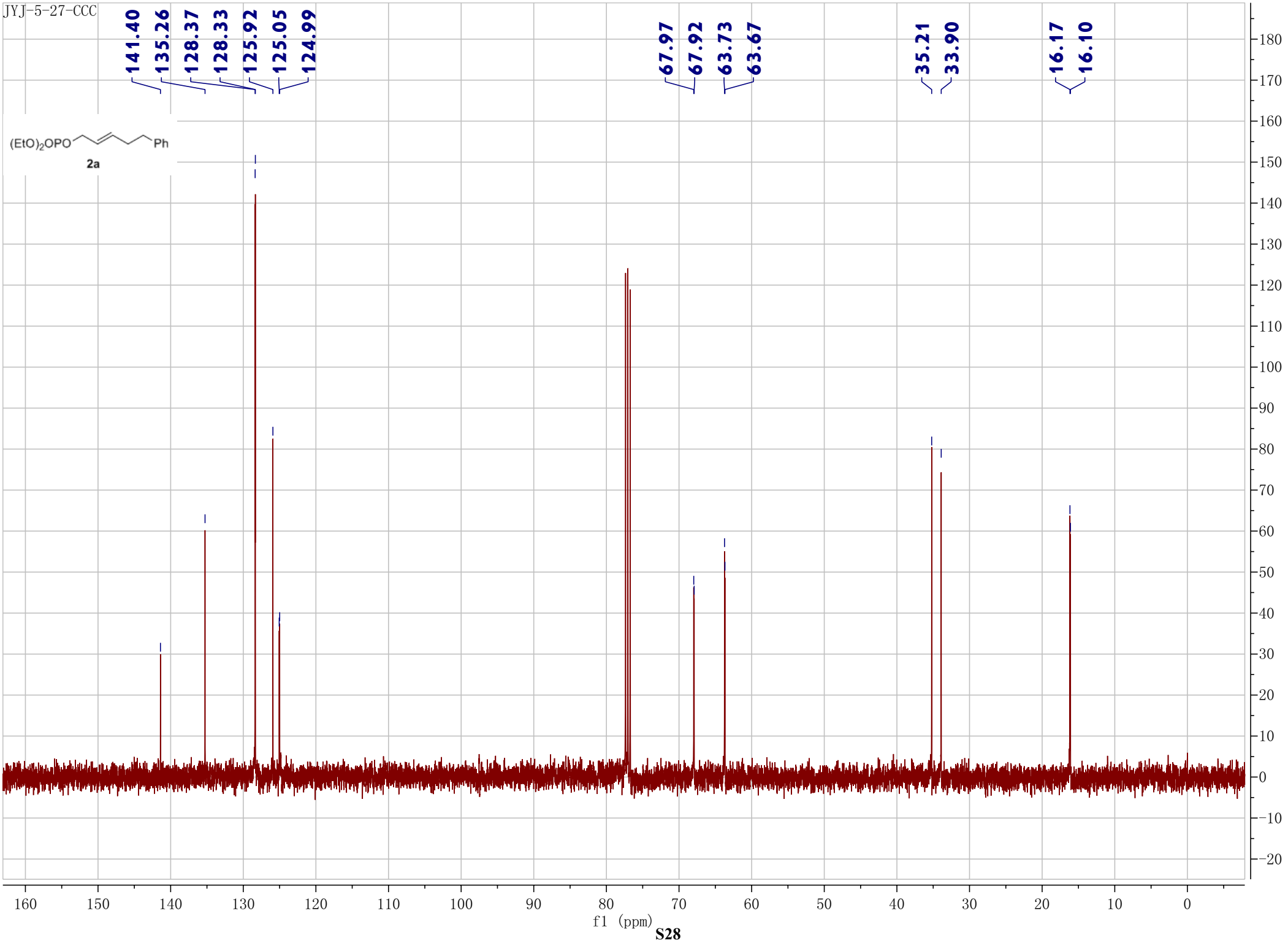
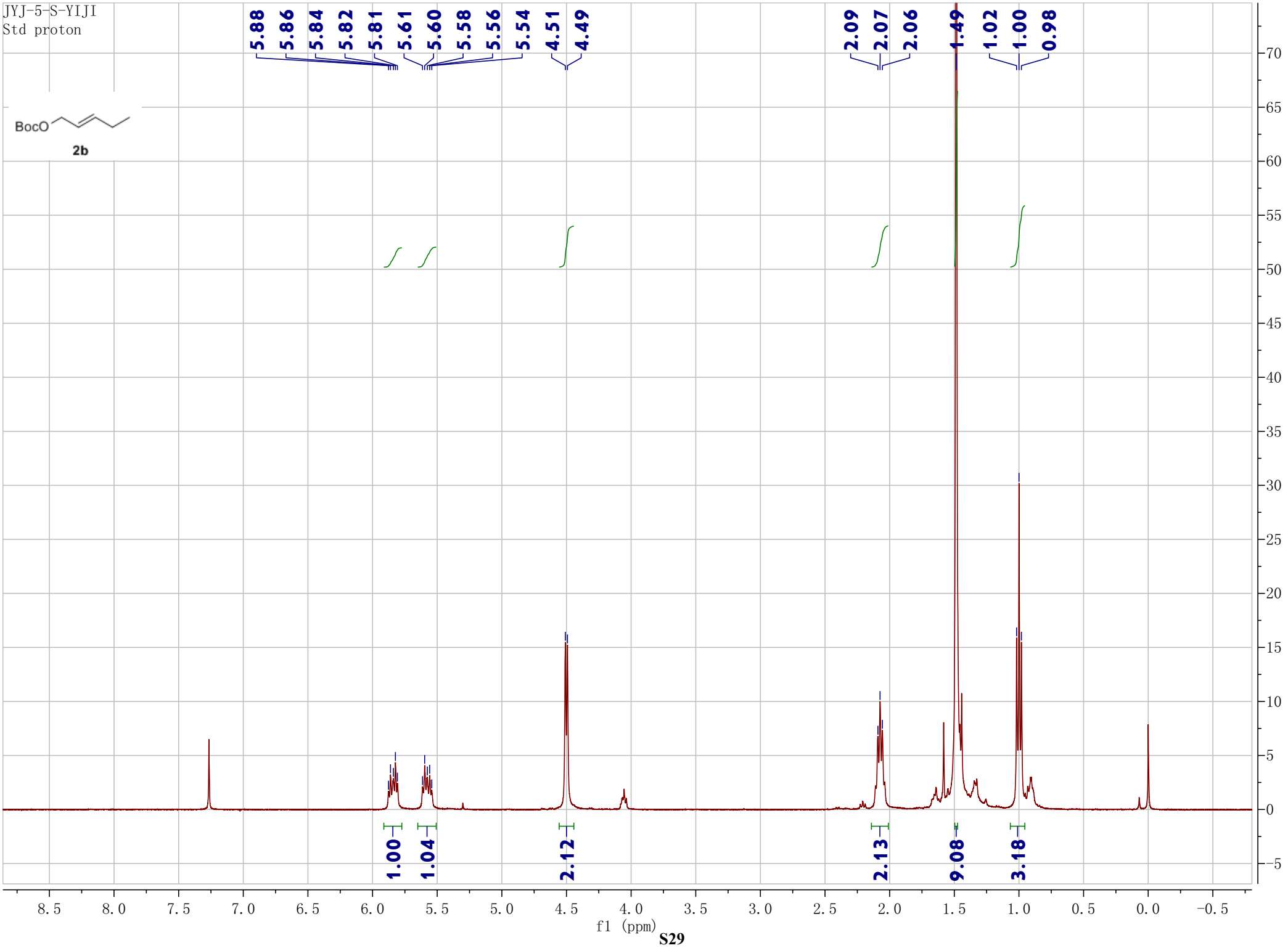
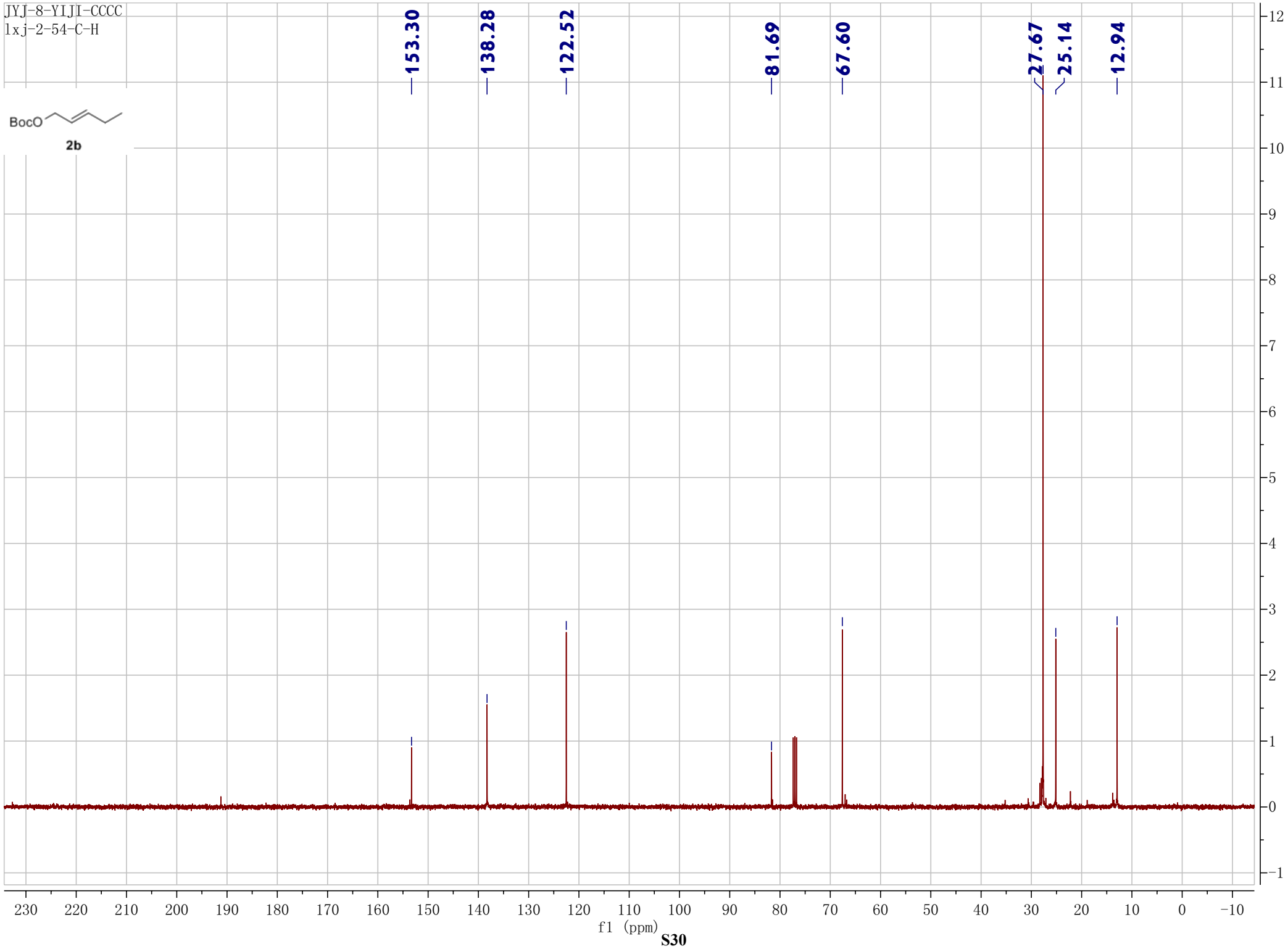
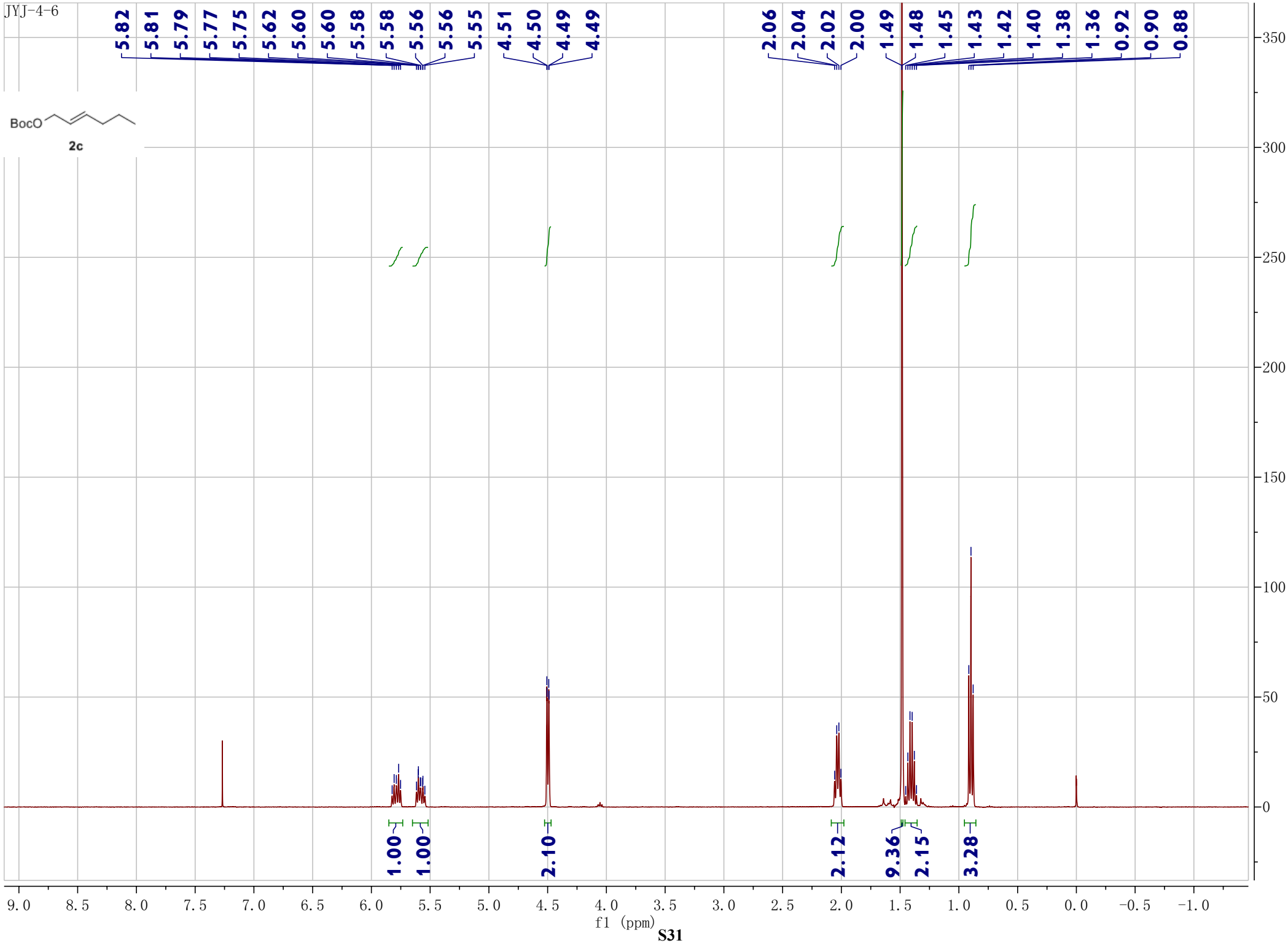
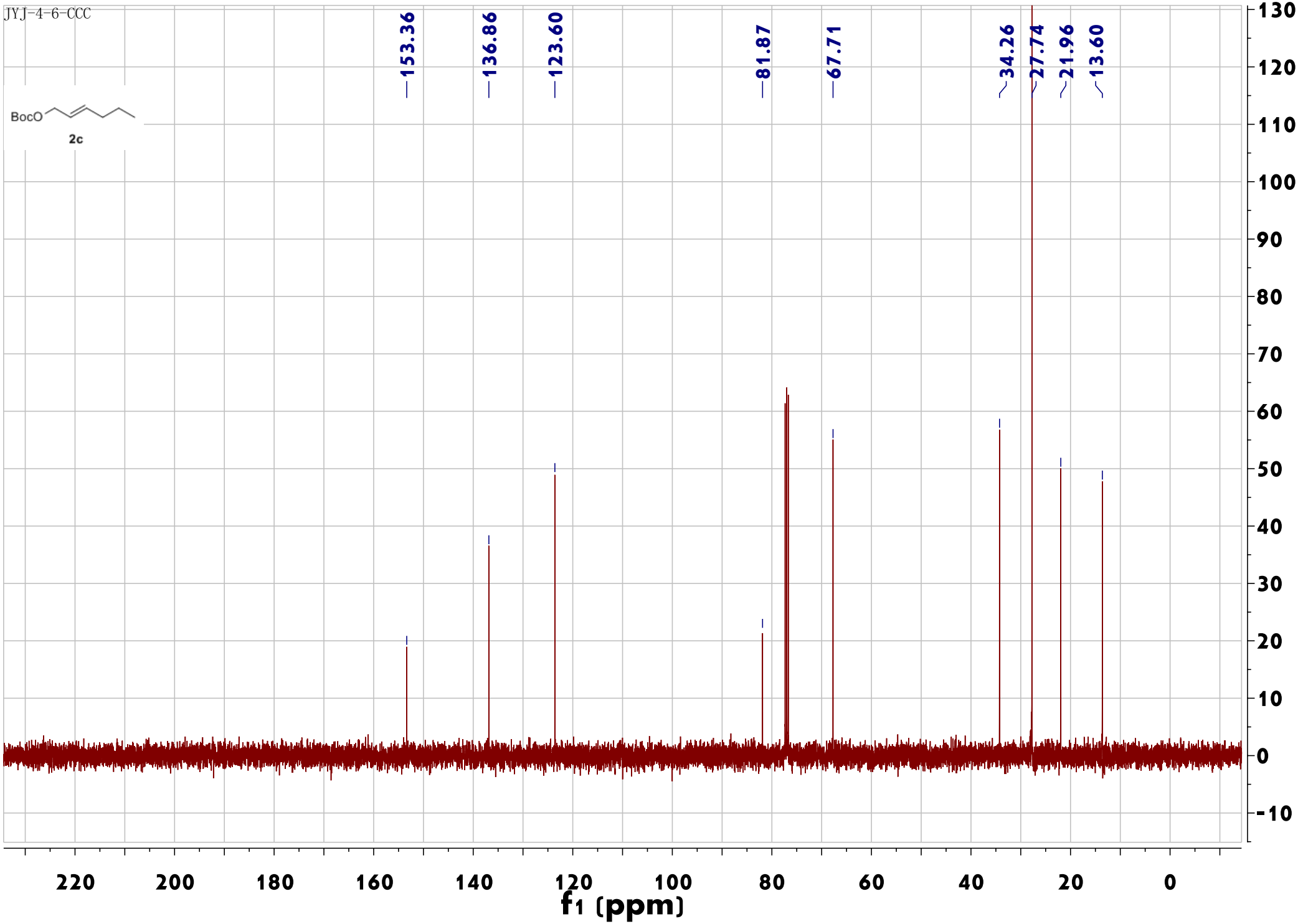
7.29 7.28 7.28 7.26 7.20 7.20 7.18 7.18 7.16 5.85 5.83 5.82 5.67 5.66 5.66 5.65 5.64 5.62 4.49 4.49 4.47 4.47 4.45 4.45 4.13 4.11 4.09 4.07 4.06 2.72 2.70 2.68 2.41 2.40 2.38 2.36 1.34 1.34 1.33 1.32 1.31 1.31
(EtO)2OPO
Ph
2a
2.29 3.04 1.00 1.02 2.13 4.05 2.14 2.16 6.19
f1 (ppm)

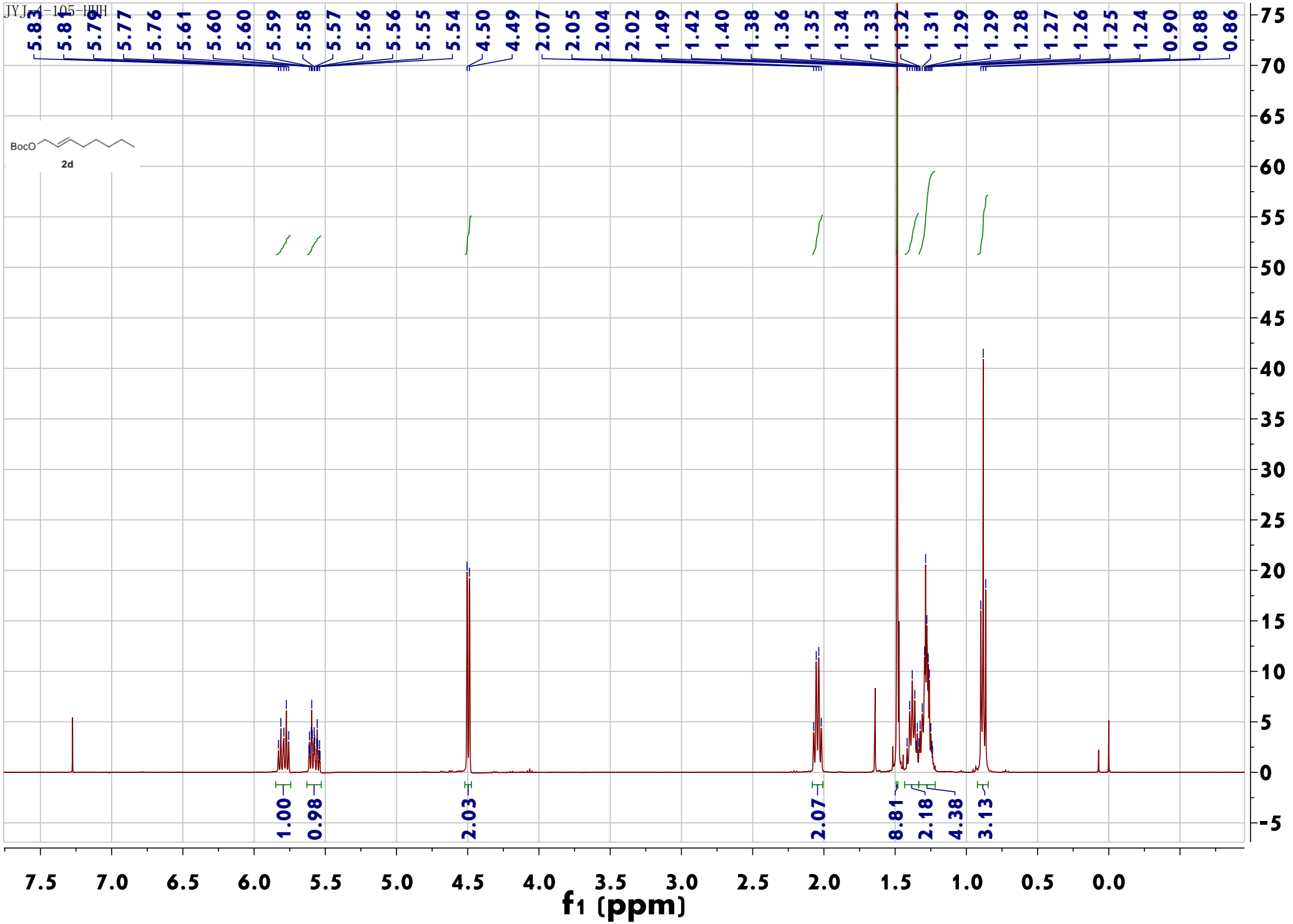

JYJ-5-27-CCC
141.40
135.26
128.37
128.33
125.92
125.05
124.99
67.97
67.92
63.73
63.67
35.21
33.90
16.17
16.10
(EtO)2OPO
Ph
2a
f1 (ppm)

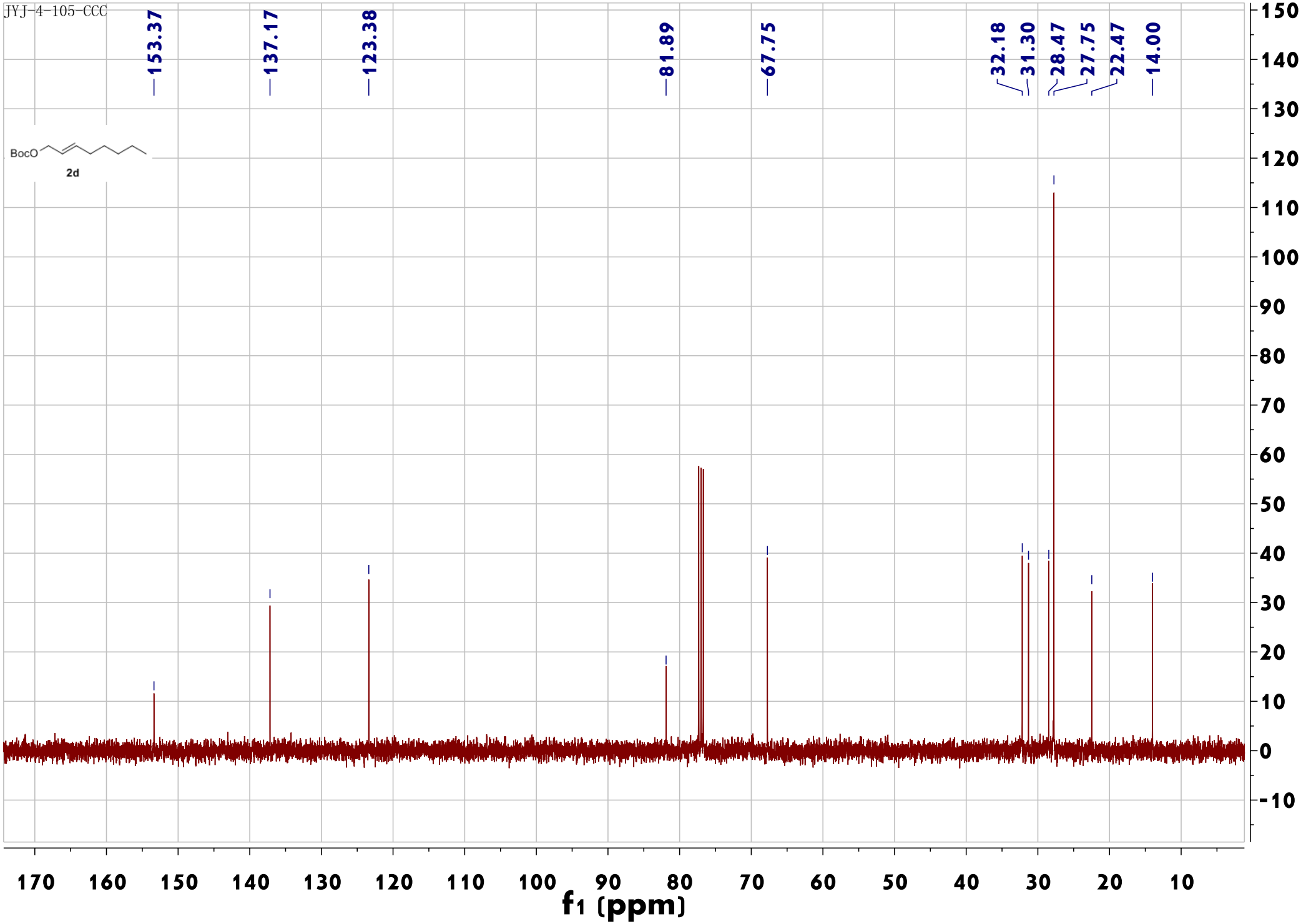

JYJ-5-S-YIJI
Std proton
5.88
5.86
5.84
5.82
5.81
5.61
5.60
5.58
5.56
5.54
4.51
4.49
2.09
2.07
2.06
1.49
1.02
1.00
0.98
BocO
2b
1.00
1.04
2.12
2.13
9.08
3.18
f1 (ppm)

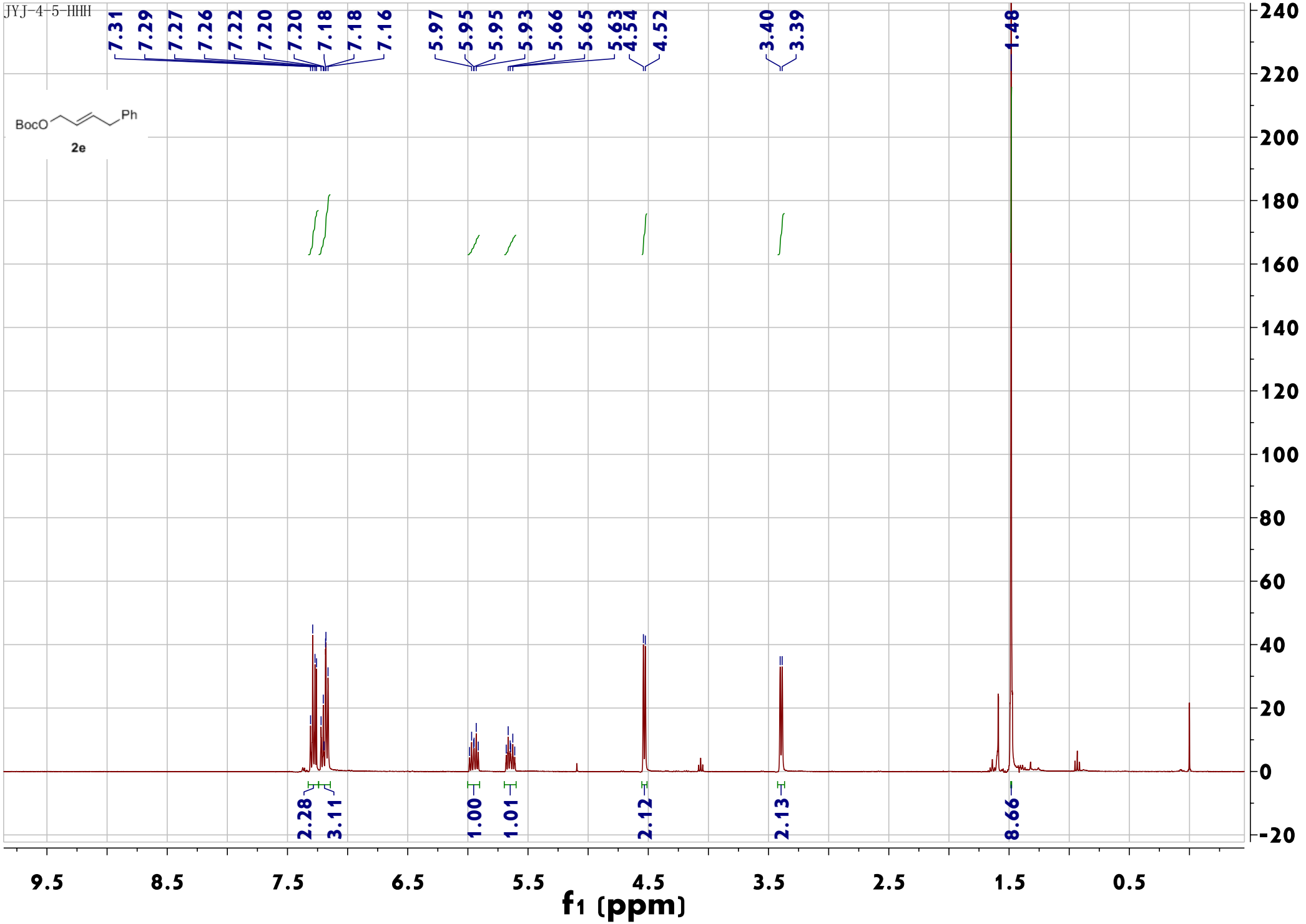

JYJ-8-YIJI-CCCC
lxj-2-54-C-H
153.30
138.28
122.52
81.69
67.60
27.67
25.14
12.94
BocO
2b
f1 (ppm)

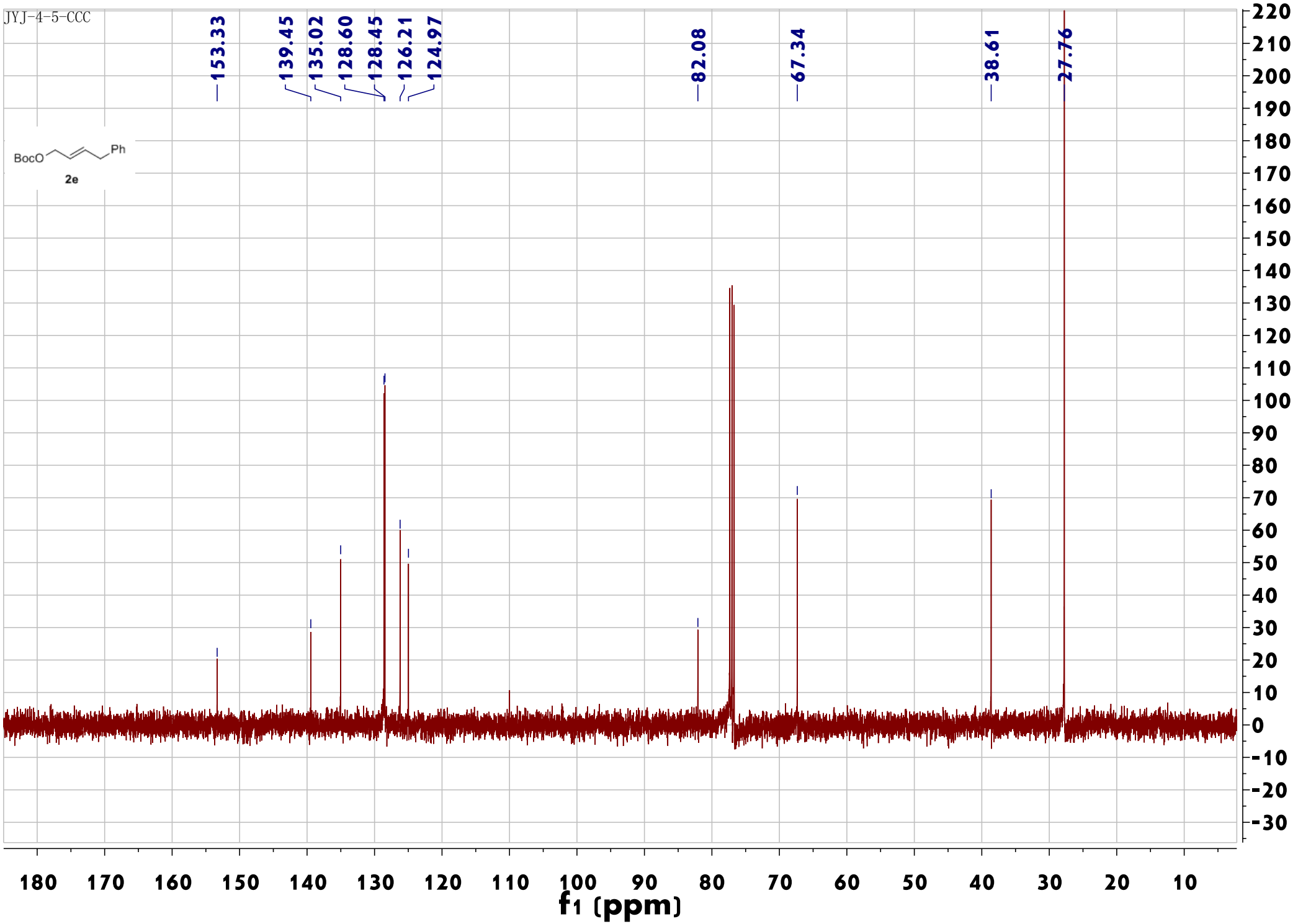

JYJ-4-6
5.82
5.81
5.79
5.77
5.75
5.62
5.60
5.60
5.58
5.58
5.56
5.56
5.55
4.51
4.50
4.49
4.49
2.06
2.04
2.02
2.00
1.49
1.48
1.45
1.43
1.42
1.40
1.38
1.36
0.92
0.90
0.88
BocO
2c
1.00
1.00
2.10
2.12
9.36
2.15
3.28
f1 (ppm)

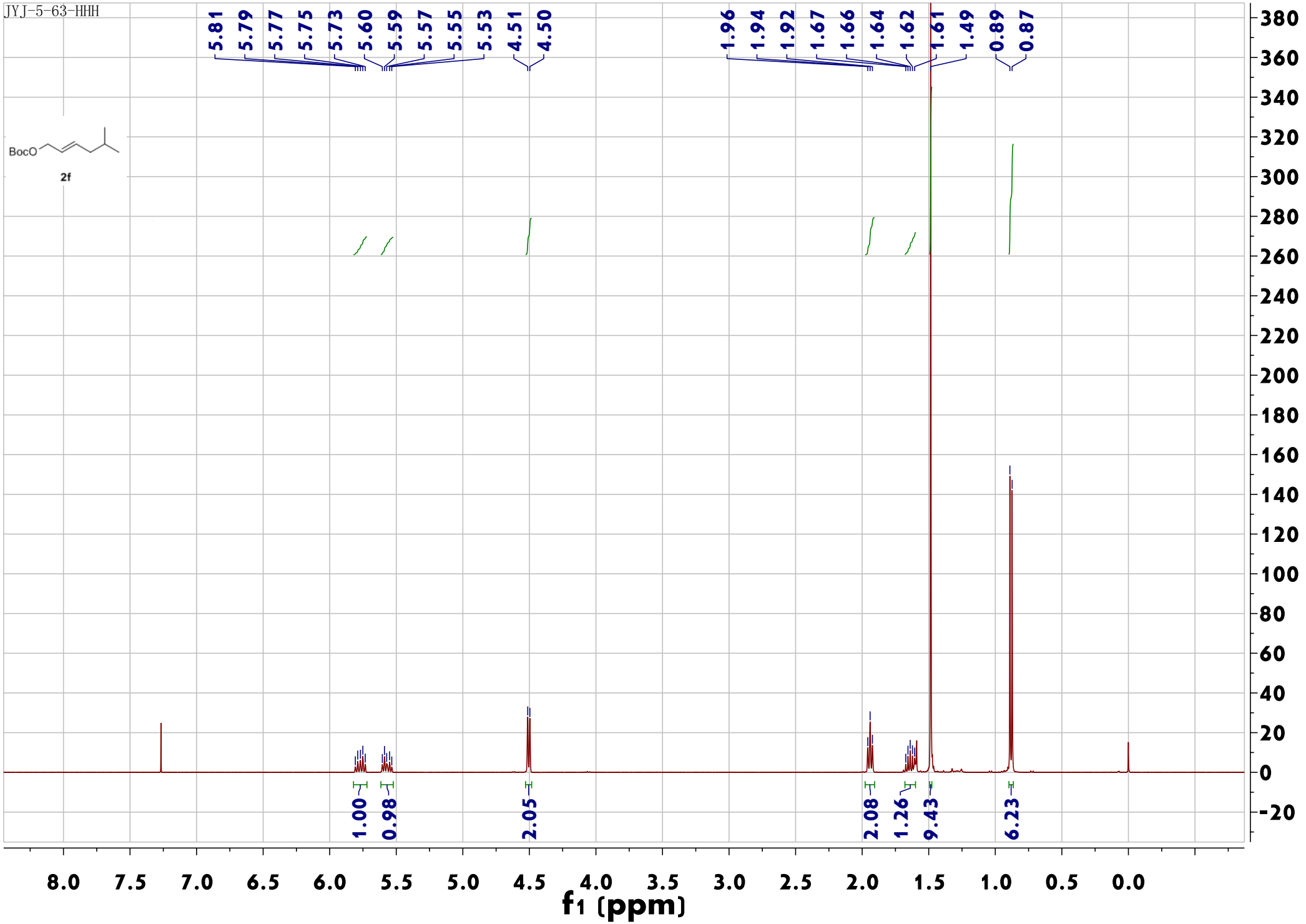

JYJ-4-6-CCC
153.36
136.86
123.60
81.87
67.71
34.26
27.74
21.96
13.60
BocO
2c
f1 (ppm)

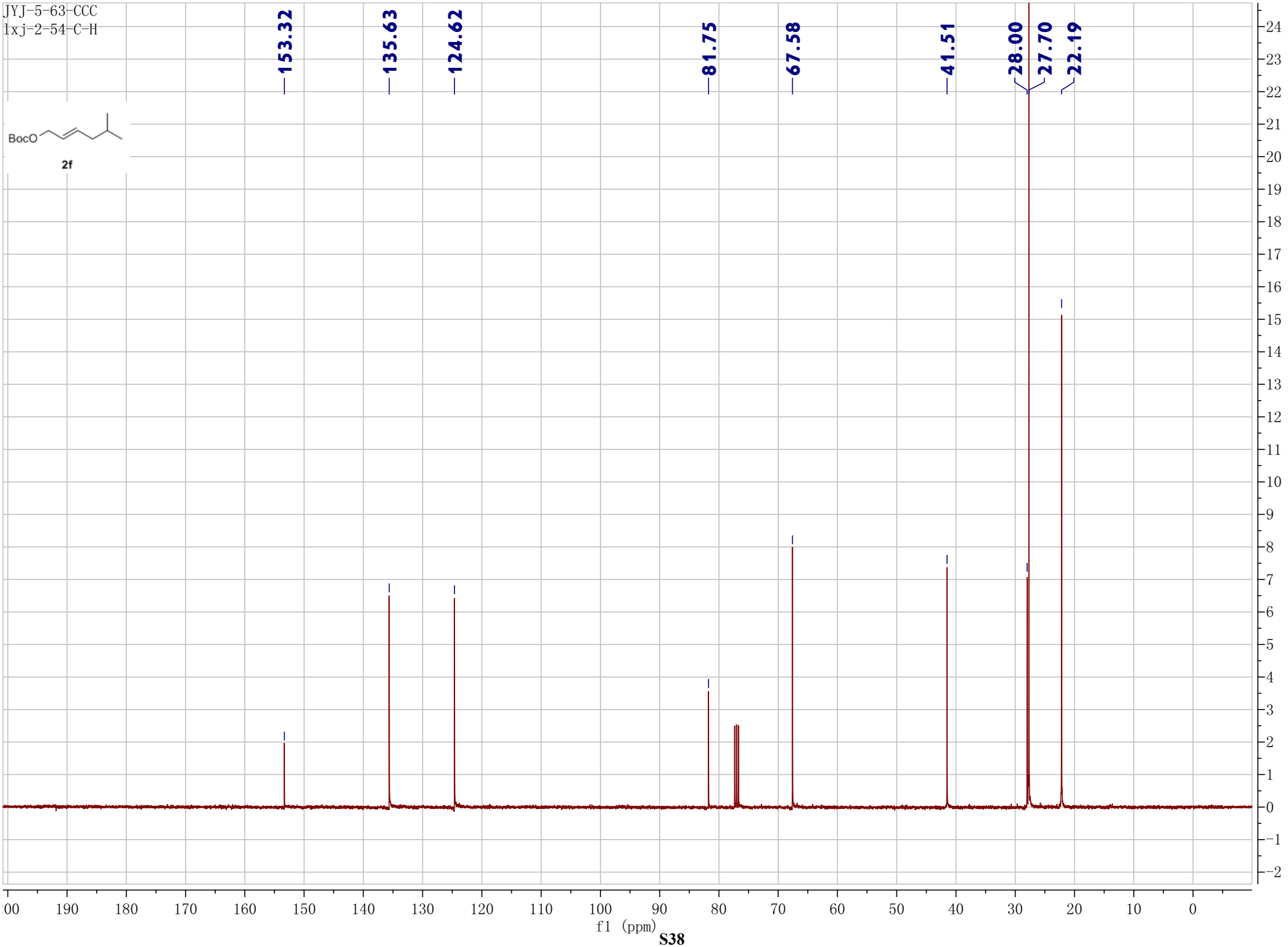

JYJ-4-105-HH
5.83
5.81
5.79
5.77
5.76
5.61
5.60
5.60
5.59
5.58
5.57
5.56
5.56
5.55
5.54
4.50
4.49
2.07
2.05
2.04
2.02
1.49
1.42
1.40
1.38
1.36
1.35
1.34
1.33
1.32
1.31
1.29
1.29
1.28
1.27
1.26
1.25
1.24
0.90
0.88
0.86
BocO
2d
1.00
0.98
2.03
2.07
8.81
2.18
4.38
3.13
f1 (ppm)

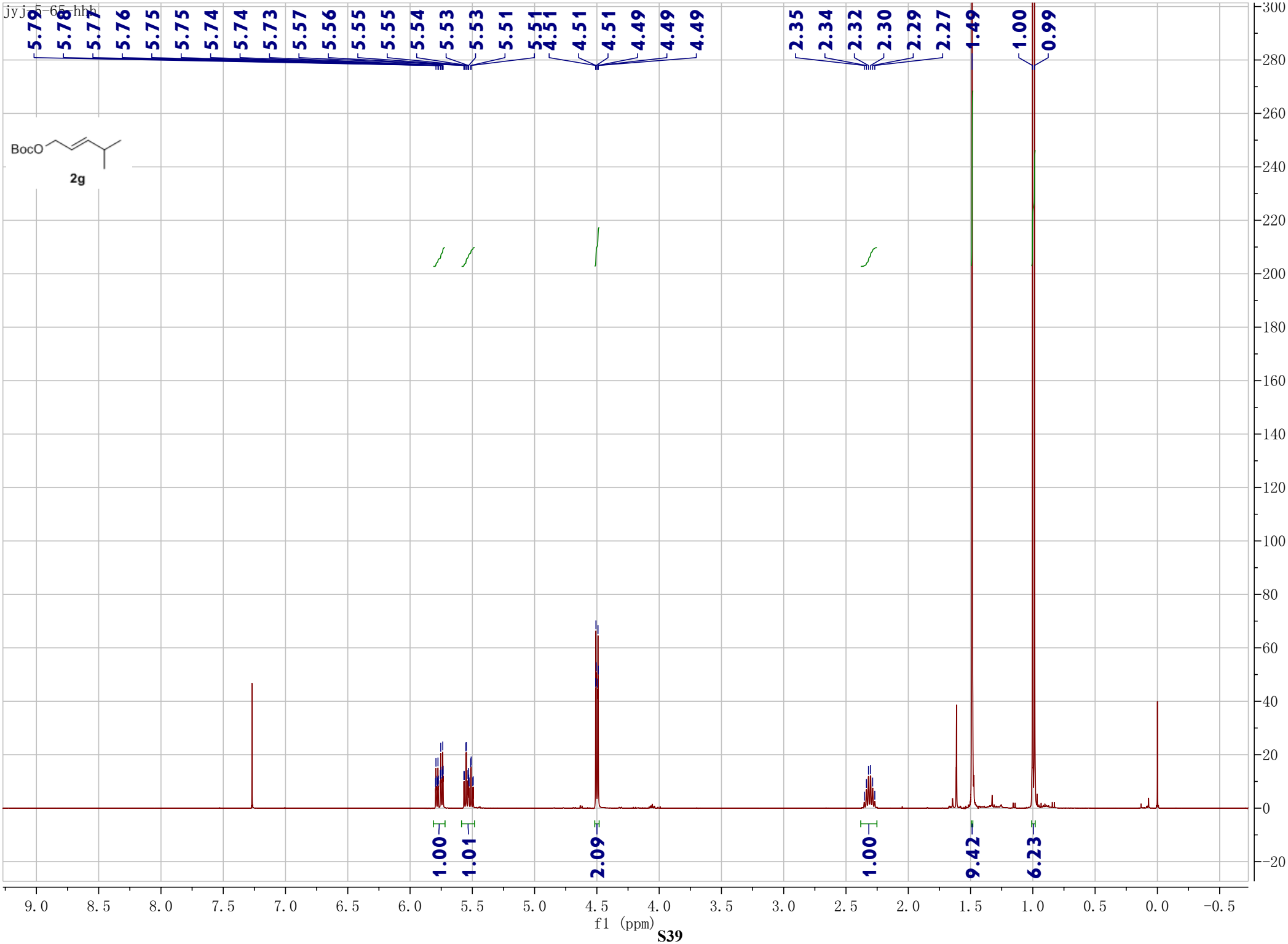

JYJ-4-105-CCC
153.37
137.17
123.38
81.89
67.75
32.18
31.30
28.47
27.75
22.47
14.00
BocO
2d
f1 (ppm)

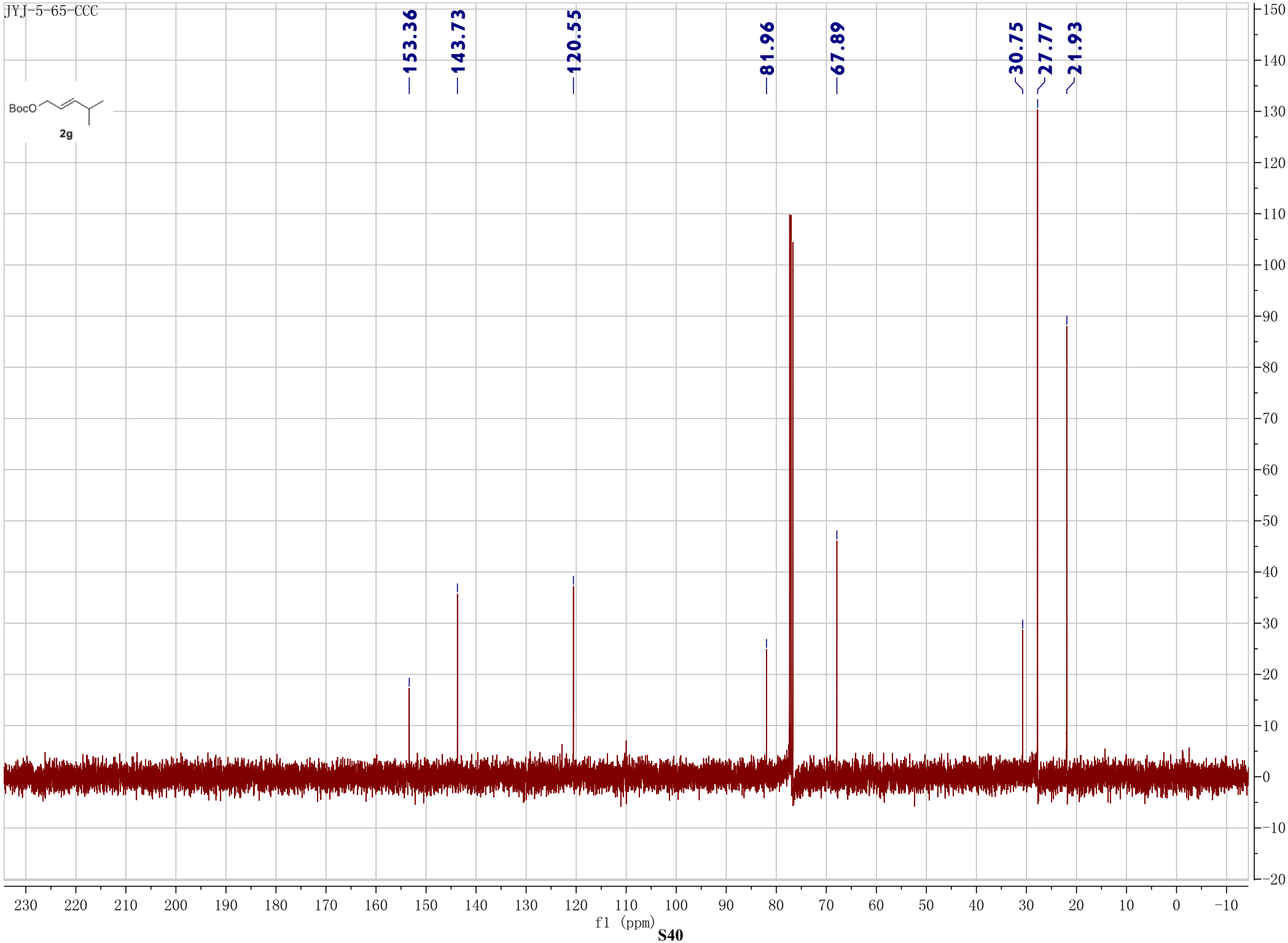

JYJ-4-5-HHH
7.31
7.29
7.27
7.26
7.22
7.20
7.20
7.18
7.18
7.16
5.97
5.95
5.95
5.93
5.66
5.65
5.63
4.54
4.52
3.40
3.39
1.48
BocO
Ph
2e
2.28
3.11
1.00
1.01
2.12
2.13
8.66
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)

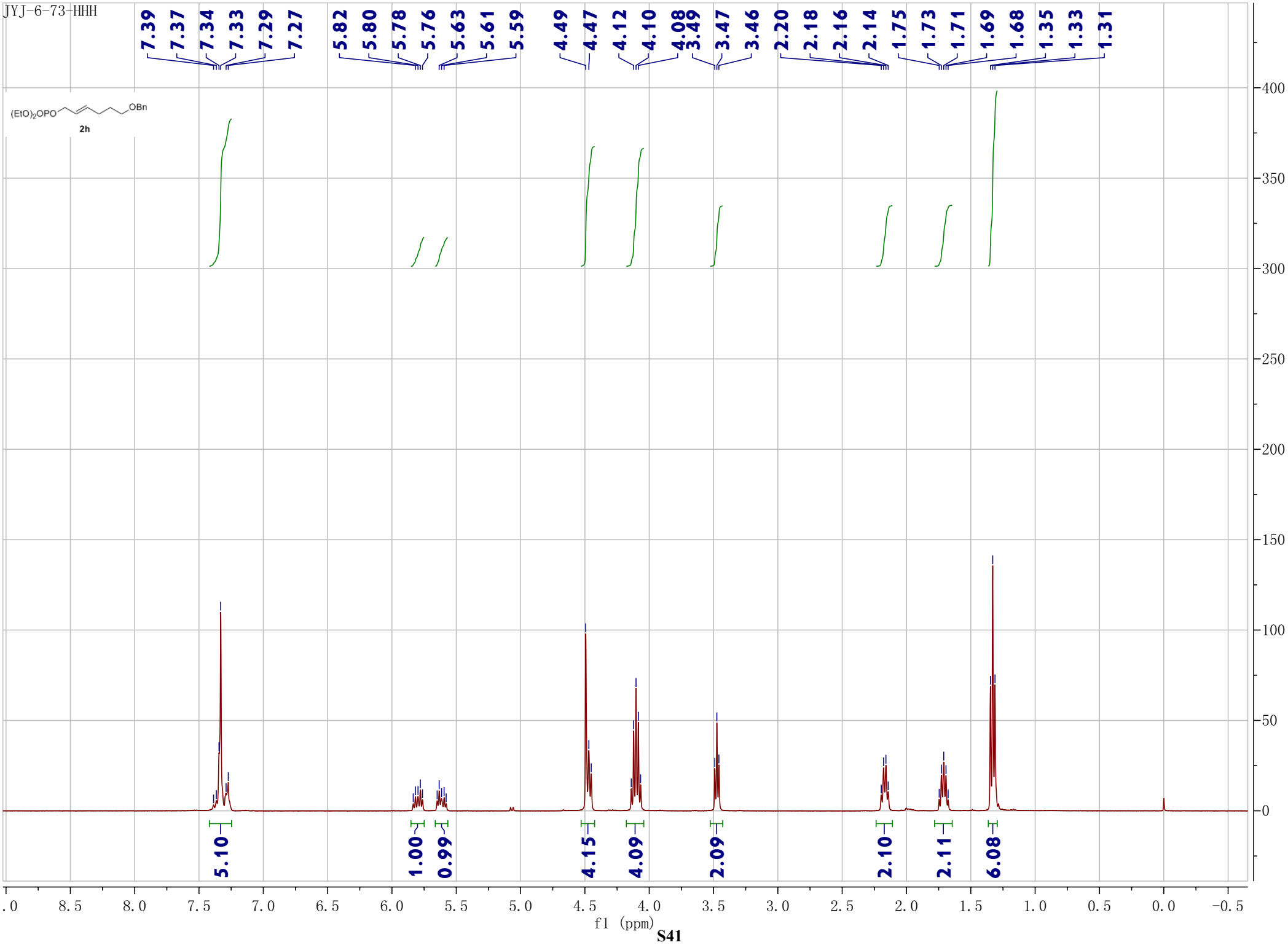

JYJ-4-5-CCC
153.33
139.45
135.02
128.60
128.45
126.21
124.97
82.08
67.34
38.61
27.76
BocO
Ph
2e
f1 (ppm)

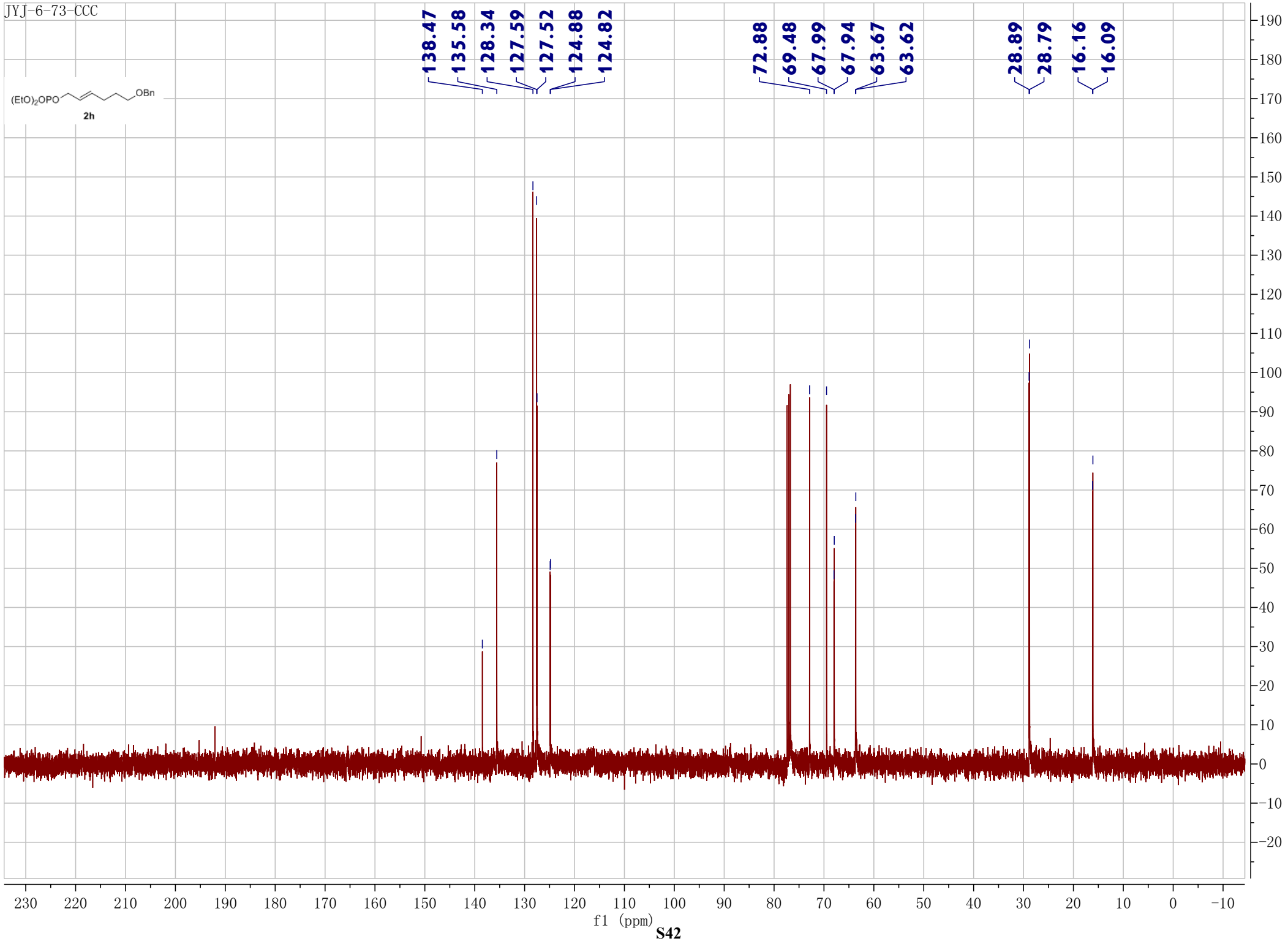

JYJ-5-63-HHH
5.81
5.79
5.77
5.75
5.73
5.60
5.59
5.57
5.55
5.53
4.51
4.50
1.96
1.94
1.92
1.67
1.66
1.64
1.62
1.61
1.49
0.89
0.87
BocO
2f
1.00
0.98
2.05
2.08
1.26
9.43
6.23
f1 (ppm)

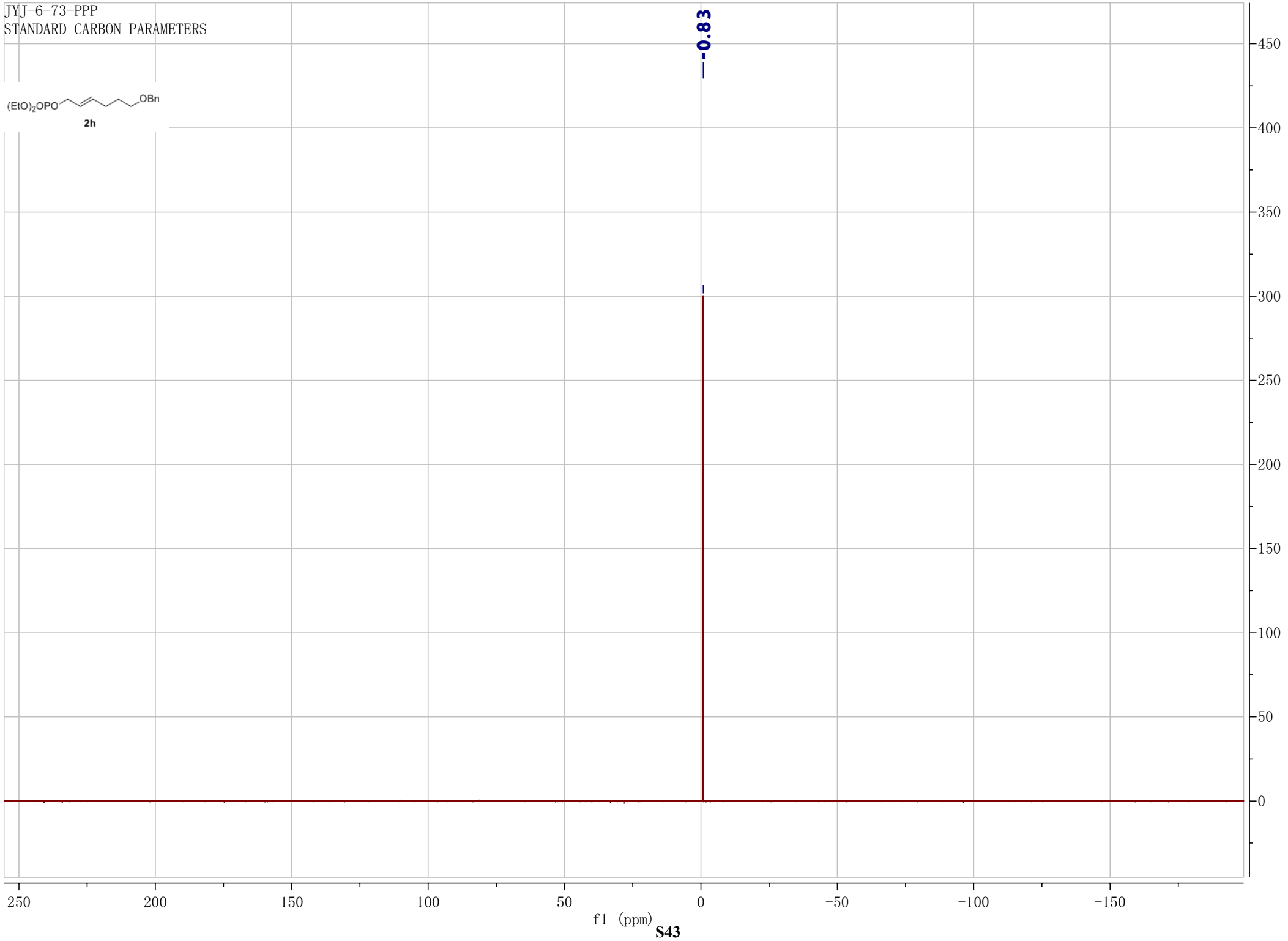

JYJ-5-63-CCC
1xj-2-54-C-H
153.32
135.63
124.62
81.75
67.58
41.51
28.00
27.70
22.19
BocO
2f
f1 (ppm)

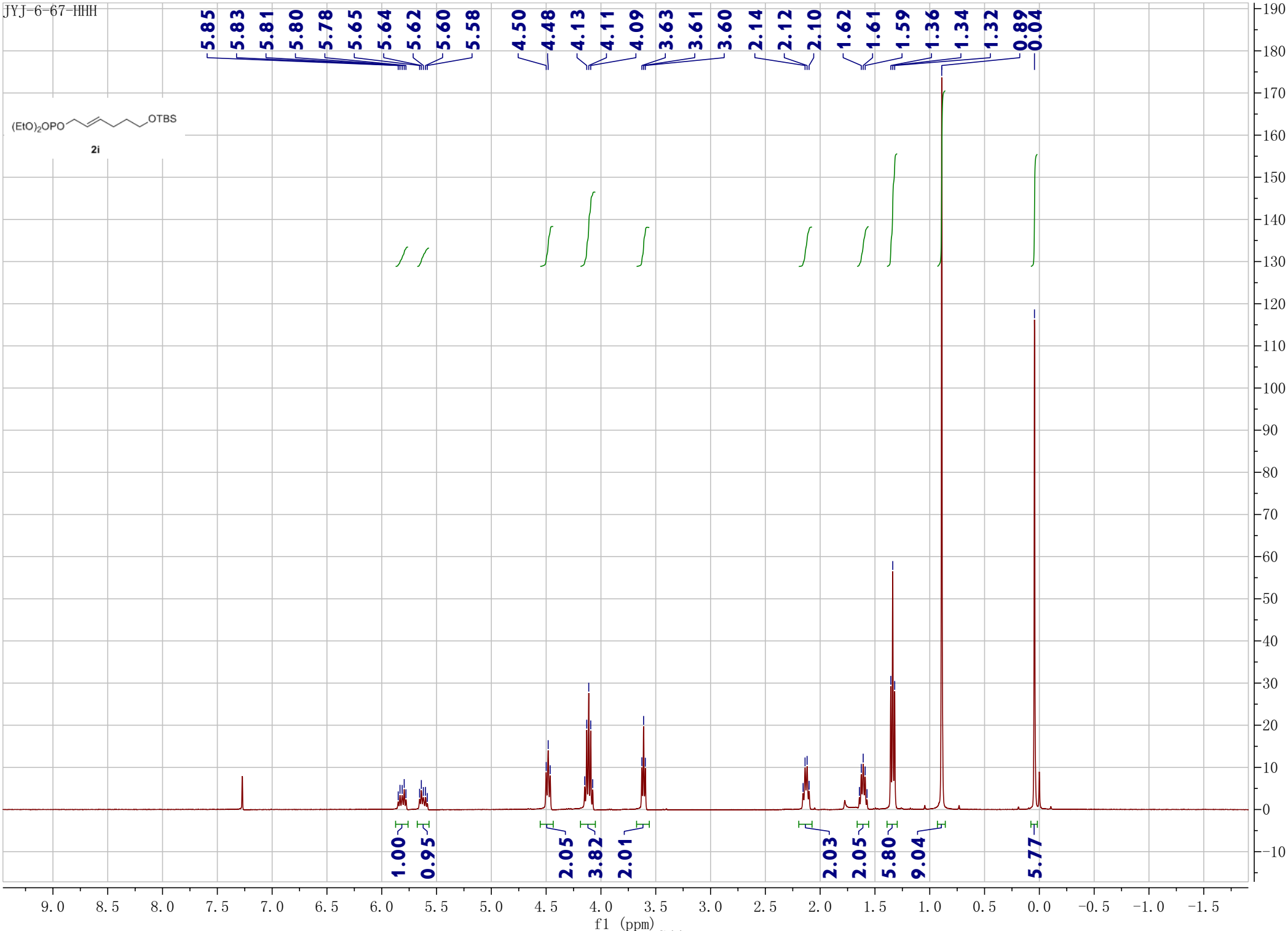

jyj-5-65-hhh
5.79
5.78
5.77
5.76
5.75
5.75
5.74
5.74
5.73
5.57
5.56
5.55
5.55
5.54
5.53
5.53
5.51
5.51
4.51
4.51
4.51
4.49
4.49
4.49
2.35
2.34
2.32
2.30
2.29
2.27
1.49
1.00
0.99
BocO
2g
1.00
1.01
2.09
1.00
9.42
6.23
f1 (ppm)

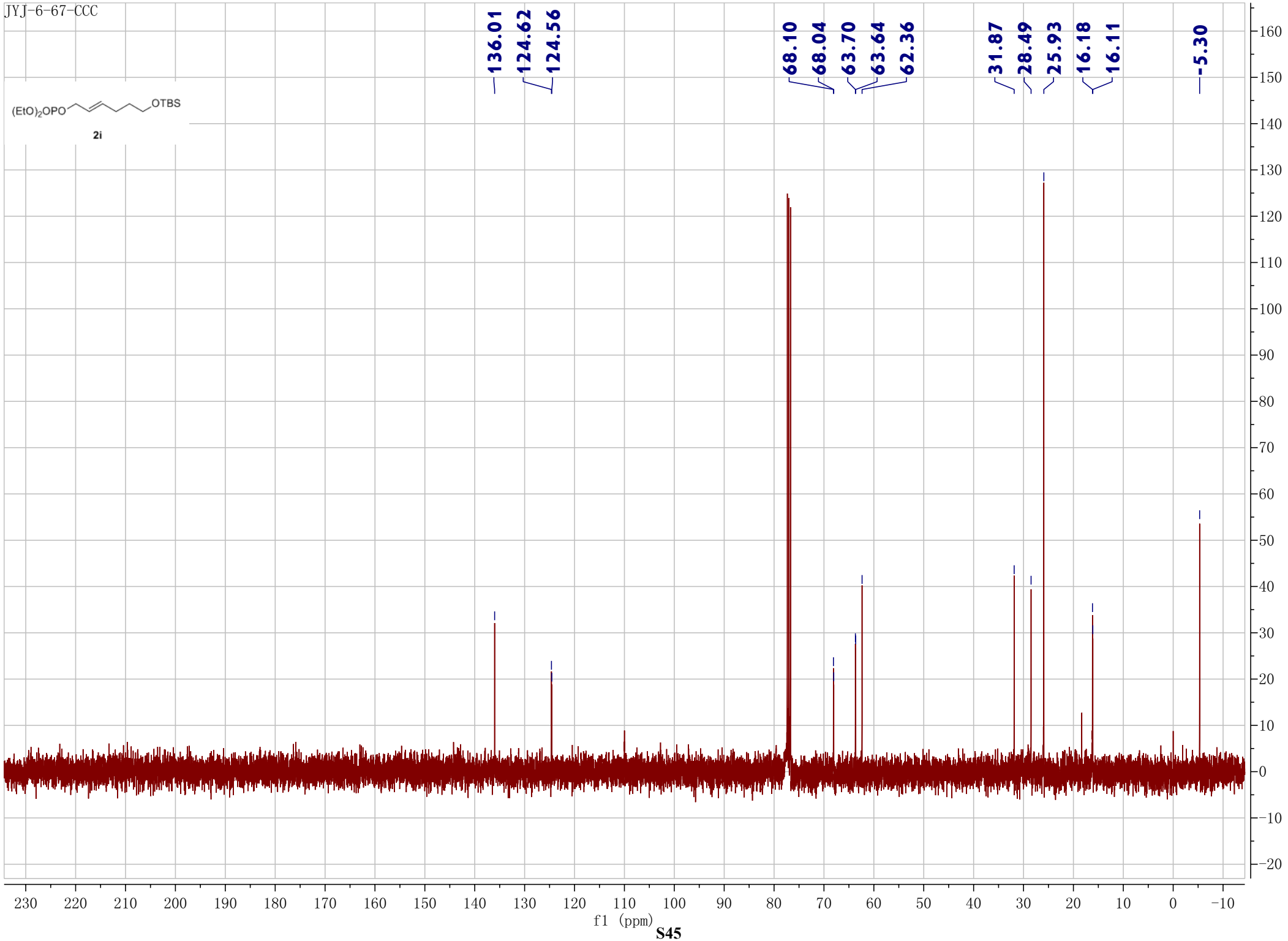

JYJ-5-65-CCC
153.36
143.73
120.55
81.96
67.89
30.75
27.77
21.93
BocO
2g
f1 (ppm)

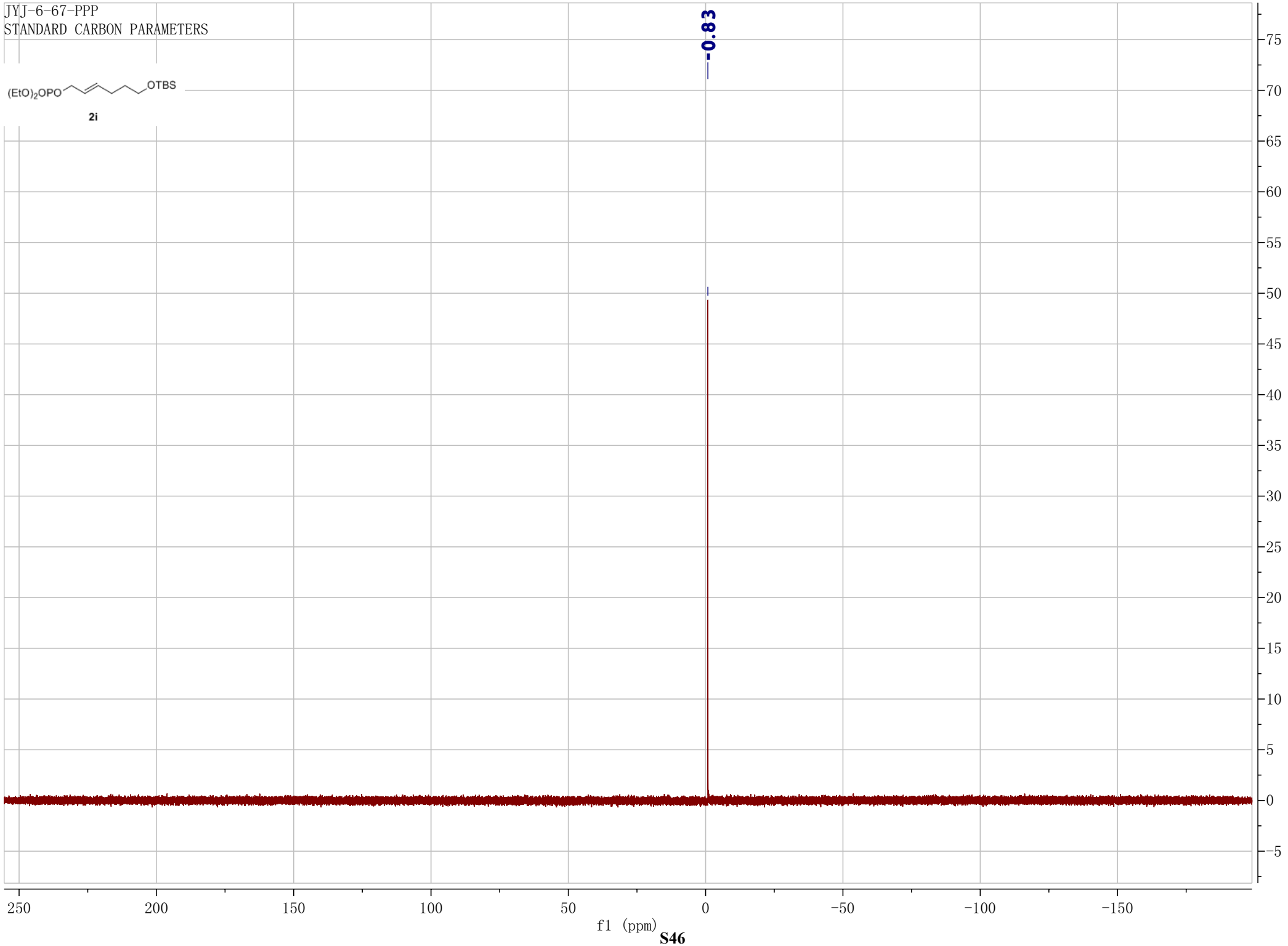

JYJ-6-73-HHH
7.39
7.37
7.34
7.33
7.29
7.27
5.82
5.80
5.78
5.76
5.63
5.61
5.59
4.49
4.47
4.12
4.10
4.08
3.49
3.47
3.46
2.20
2.18
2.16
2.14
1.75
1.73
1.71
1.69
1.68
1.35
1.33
1.31
(EtO)2OPO
OBn
2h
5.10
1.00
0.99
4.15
4.09
2.09
2.10
2.11
6.08
f1 (ppm)

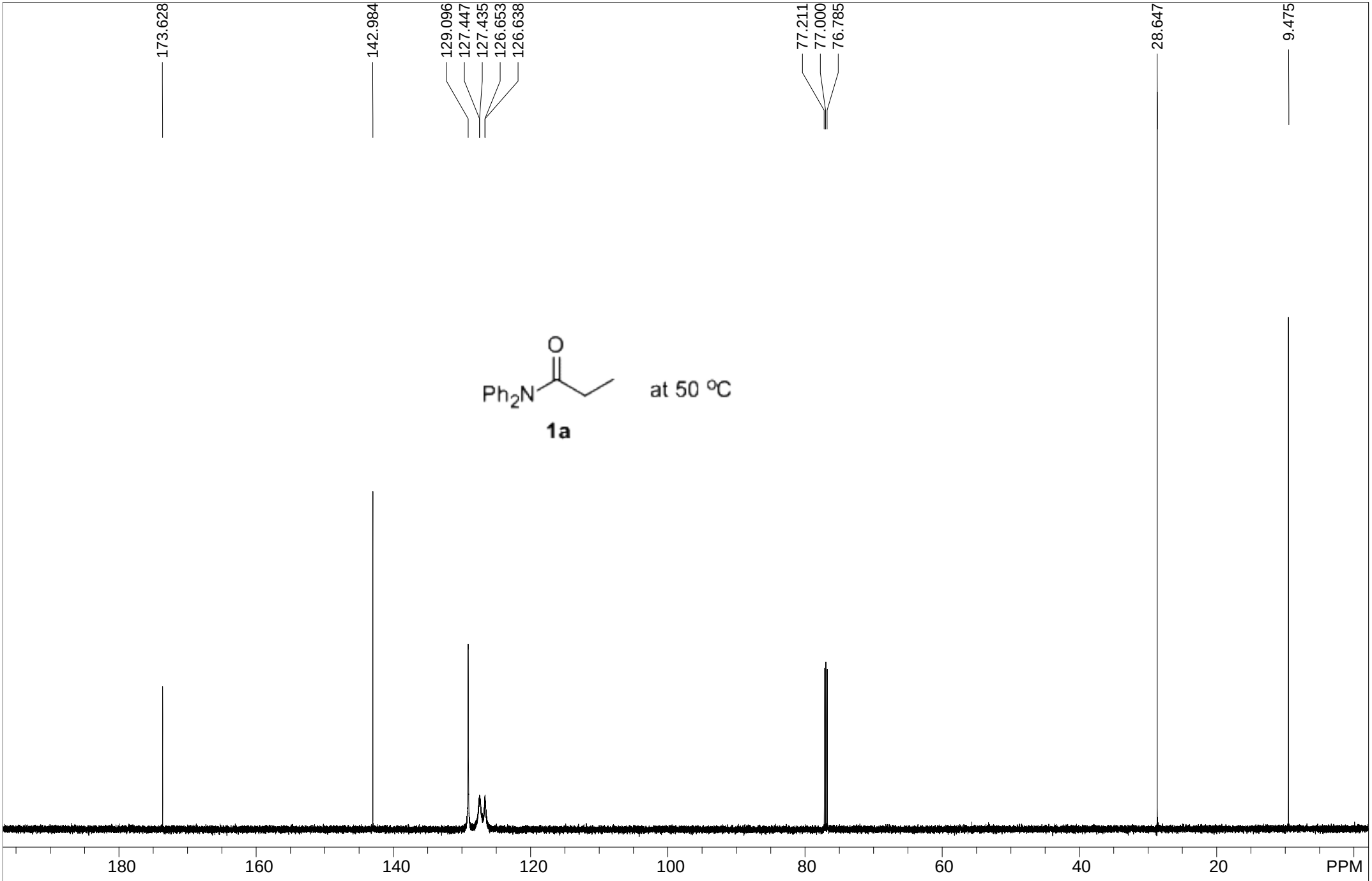

JYJ-6-73-CCC
138.47
135.58
128.34
127.59
127.52
124.88
124.82
72.88
69.48
67.99
67.94
63.67
63.62
28.89
28.79
16.16
16.09
(EtO)2OPO
OBn
2h
f1 (ppm)


JYJ-6-73-PPP
STANDARD CARBON PARAMETERS
(EtO)2OPO
OBn
2h
-0.83
450
400
350
300
250
200
150
100
50
0
250
200
150
100
50
0
-50
-100
-150
f1 (ppm)


JYJ-6-67-HHH
5.85
5.83
5.81
5.80
5.78
5.65
5.64
5.62
5.60
5.58
4.50
4.48
4.13
4.11
4.09
3.63
3.61
3.60
2.14
2.12
2.10
1.62
1.61
1.59
1.36
1.34
1.32
0.89
0.04
(EtO)2OPO
OTBS
2i
1.00
0.95
2.05
3.82
2.01
2.03
2.05
5.80
9.04
5.77
f1 (ppm)


JYJ-6-67-CCC
136.01
124.62
124.56
68.10
68.04
63.70
63.64
62.36
31.87
28.49
25.93
16.18
16.11
-5.30
(EtO)2OPO
OTBS
2i
f1 (ppm)


JYJ-6-67-PPP
STANDARD CARBON PARAMETERS
(EtO)2OPO
OTBS
2i
-0.83
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
-5
250
200
150
100
50
0
-50
-100
-150
f1 (ppm)



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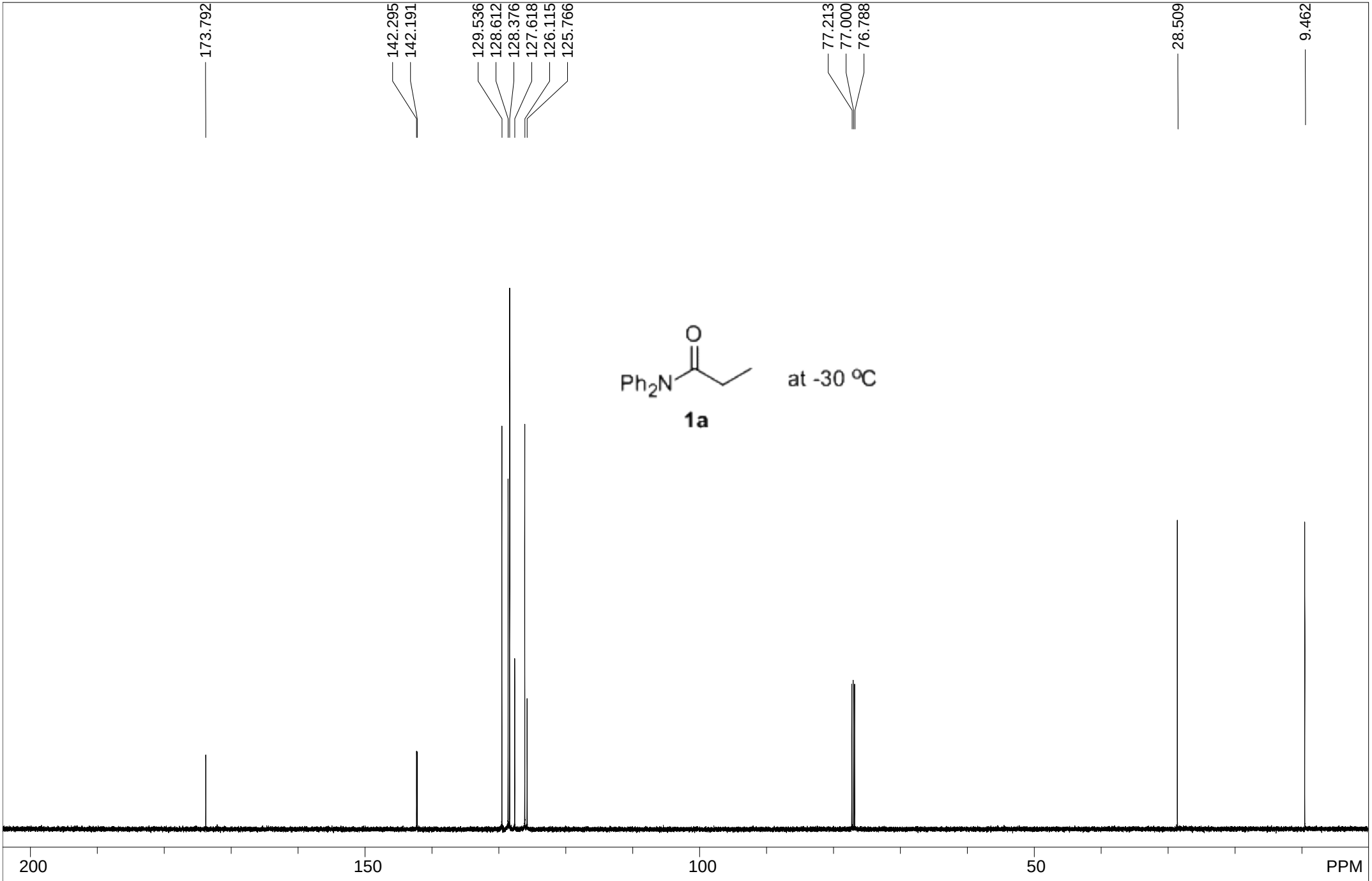
USER: -- DATE: Sep 23 2014

F1: 150.829	F2: 599.778	SW1: 35714		OF1: 15396.1		PTS1d: 65536	
EX: s2pul		PW: 5.2 usec	PD: 1.0 sec	NA: 96	LB: 0.0		Nuts - $50oC-C.fid



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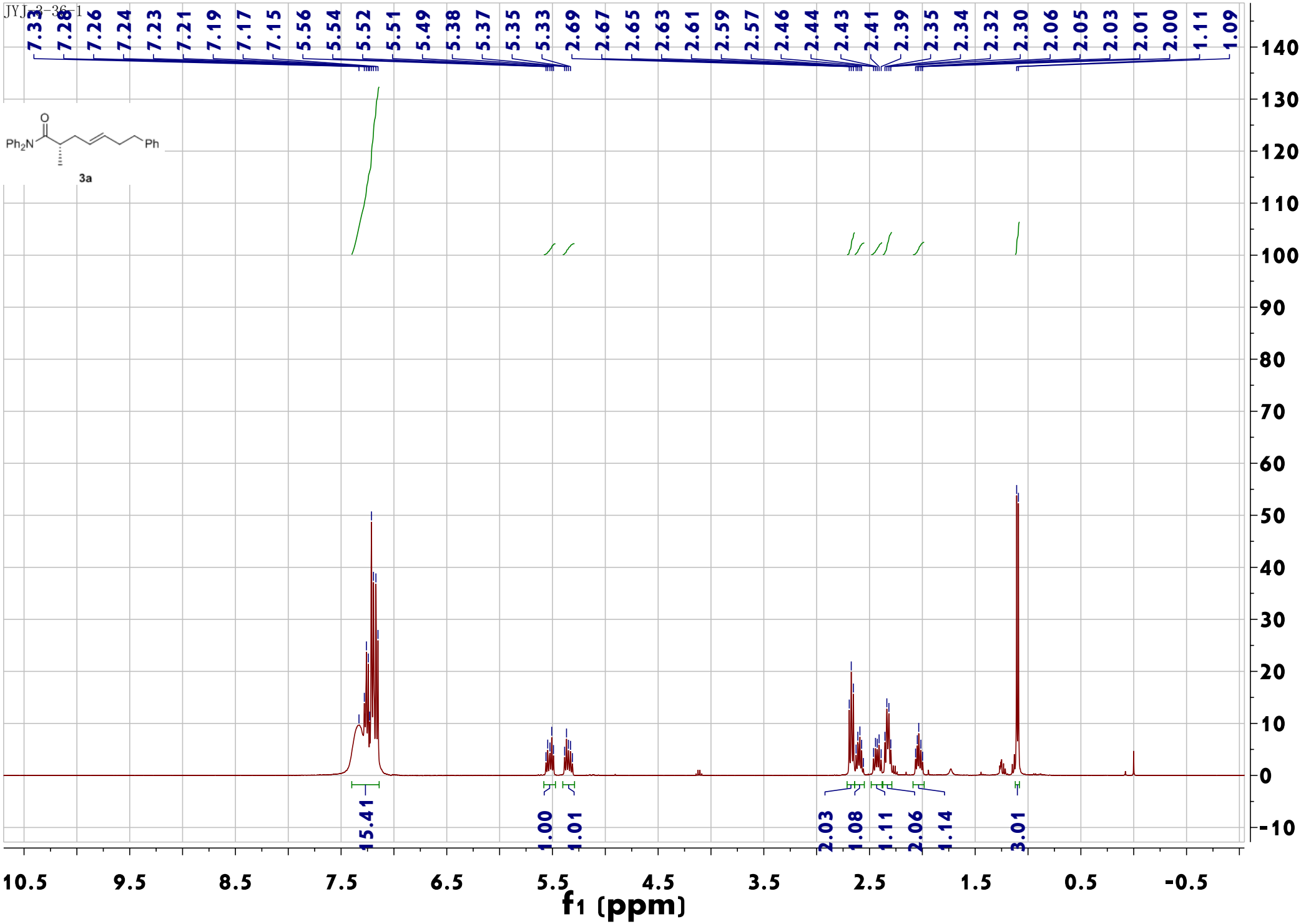
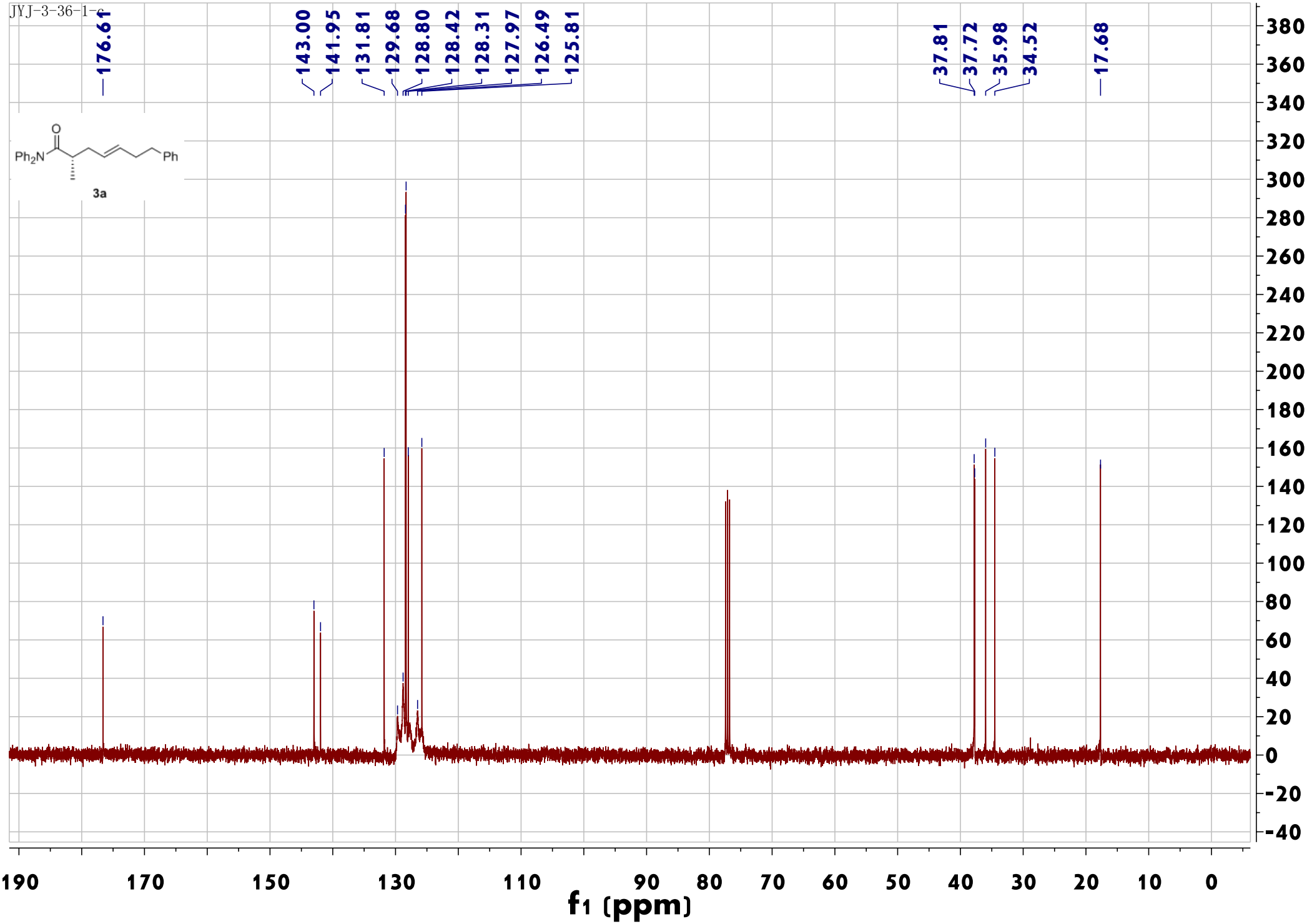
USER: -- DATE: Sep 23 2014

F1: 150.829	F2: 599.778	SW1: 35714		OF1: 15348.4		PTS1d: 65536	
EX: s2pul		PW: 5.2 usec	PD: 1.0 sec	NA: 32	LB: 0.0		Nuts - $-30oC-C.fid

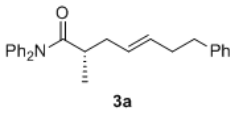

JYJ-3-36-1
7.33
7.28
7.26
7.24
7.23
7.21
7.19
7.17
7.15
5.56
5.54
5.52
5.51
5.49
5.38
5.37
5.35
5.33
2.69
2.67
2.65
2.63
2.61
2.59
2.57
2.46
2.44
2.43
2.41
2.39
2.35
2.34
2.32
2.30
2.06
2.05
2.03
2.01
2.00
1.11
1.09
Ph2N
O
Ph
3a
15.41
1.00
1.01
2.03
1.08
1.11
2.06
1.14
3.01
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)


JYJ-3-36-1-c
176.61
143.00
141.95
131.81
129.68
128.80
128.42
128.31
127.97
126.49
125.81
37.81
37.72
35.98
34.52
17.68
Ph2N
Ph
3a
f1 (ppm)

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\5\JYJ-5-36-RAC.lcd

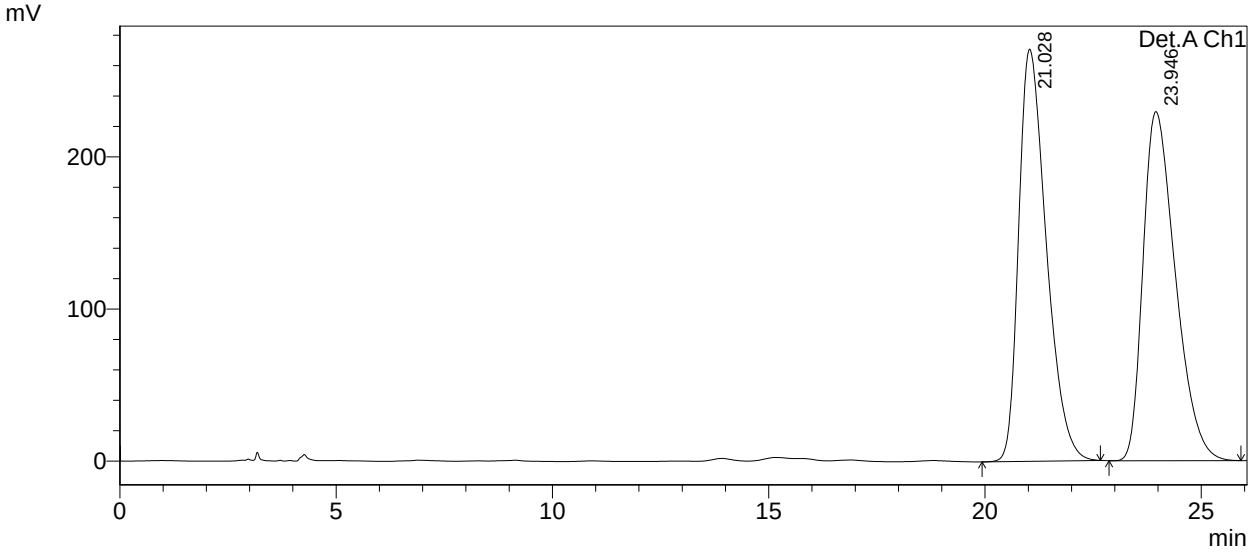
Acquired by	: Admin
Sample Name	: JYJ-5-36-RAC
Sample ID	: OD-H,99/1,1,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: JYJ-5-36-RAC.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2016-10-13 9:51:10
Data Processed	: 2016-10-13 10:46:36



3a

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\5\JYJ-5-36-RAC.lcd



1 Det.A Ch1/214nm

PeakTable

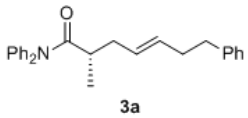
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.028	12015408	271024	50.173	54.143
2	23.946	11932609	229549	49.827	45.857
Total		23948017	500573	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\5\JYJ-5-36.lcd

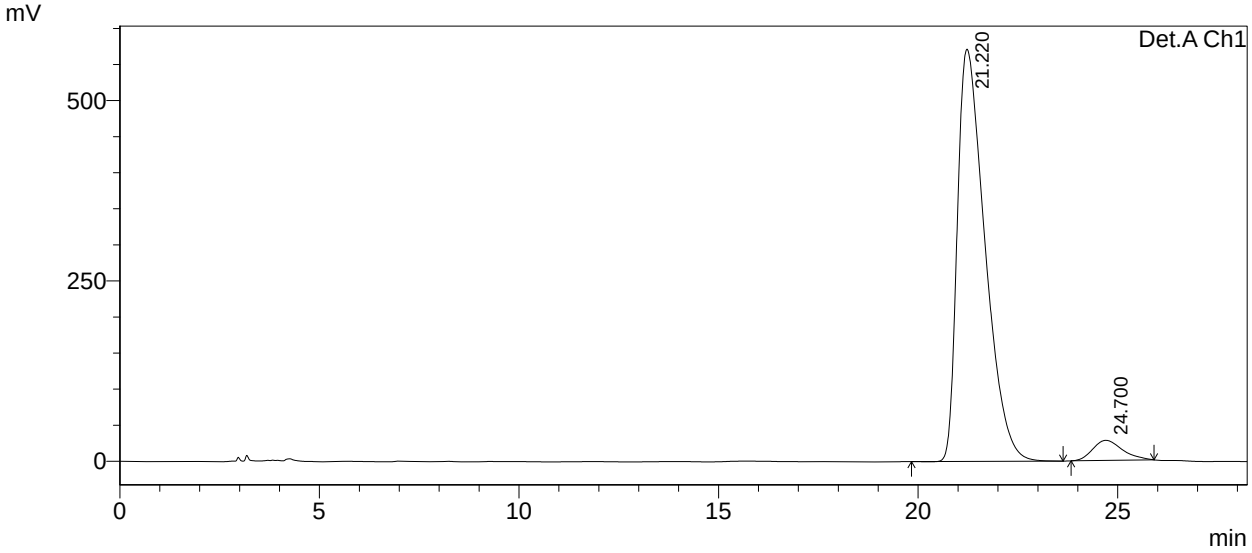
Acquired by	: Admin
Sample Name	: JYJ-5-36-1
Sample ID	: OD-H,99/1,1,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: JYJ-5-36.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2016-10-13 10:18:14
Data Processed	: 2016-10-13 10:52:12



3a

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\5\JYJ-5-36.lcd

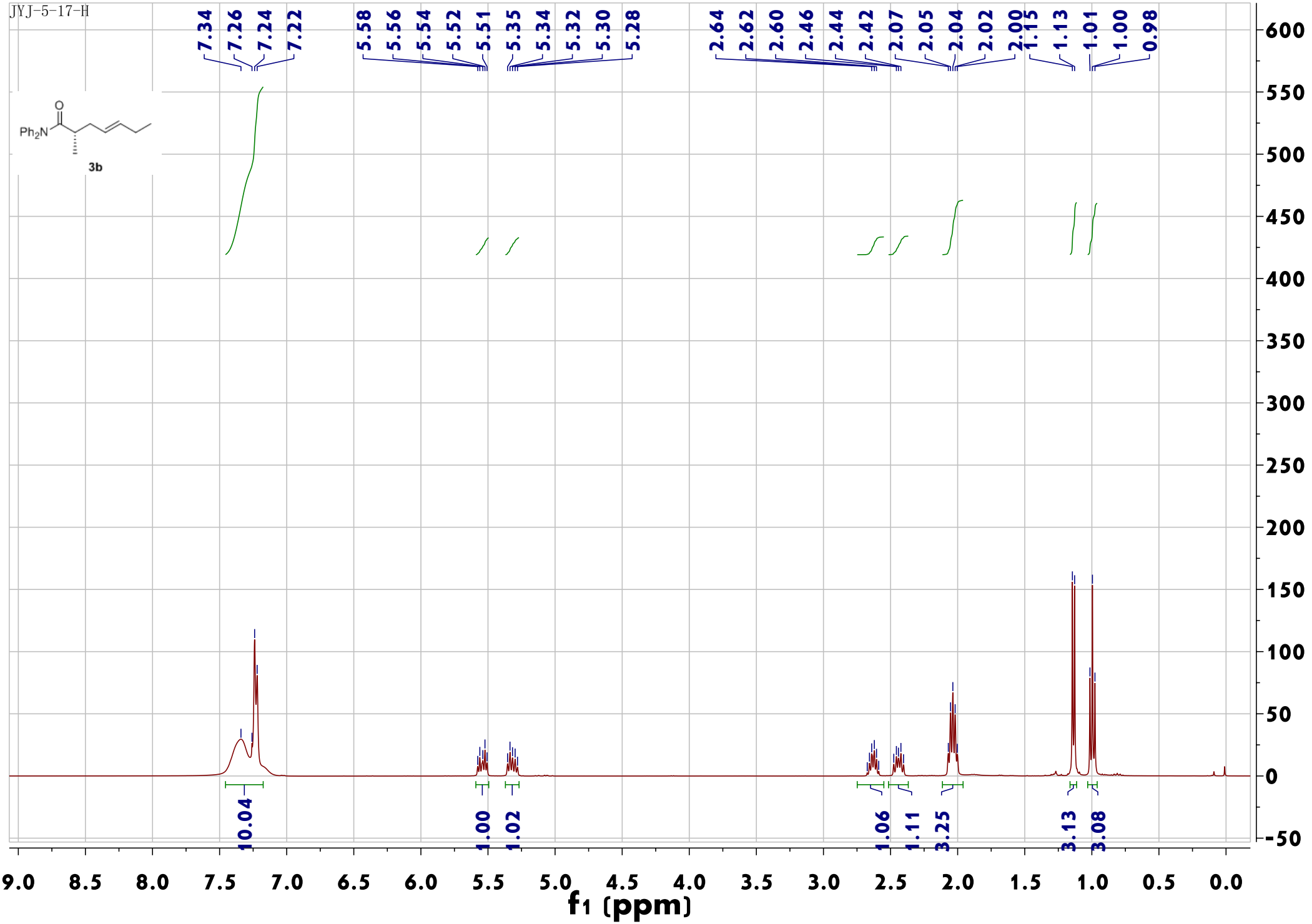


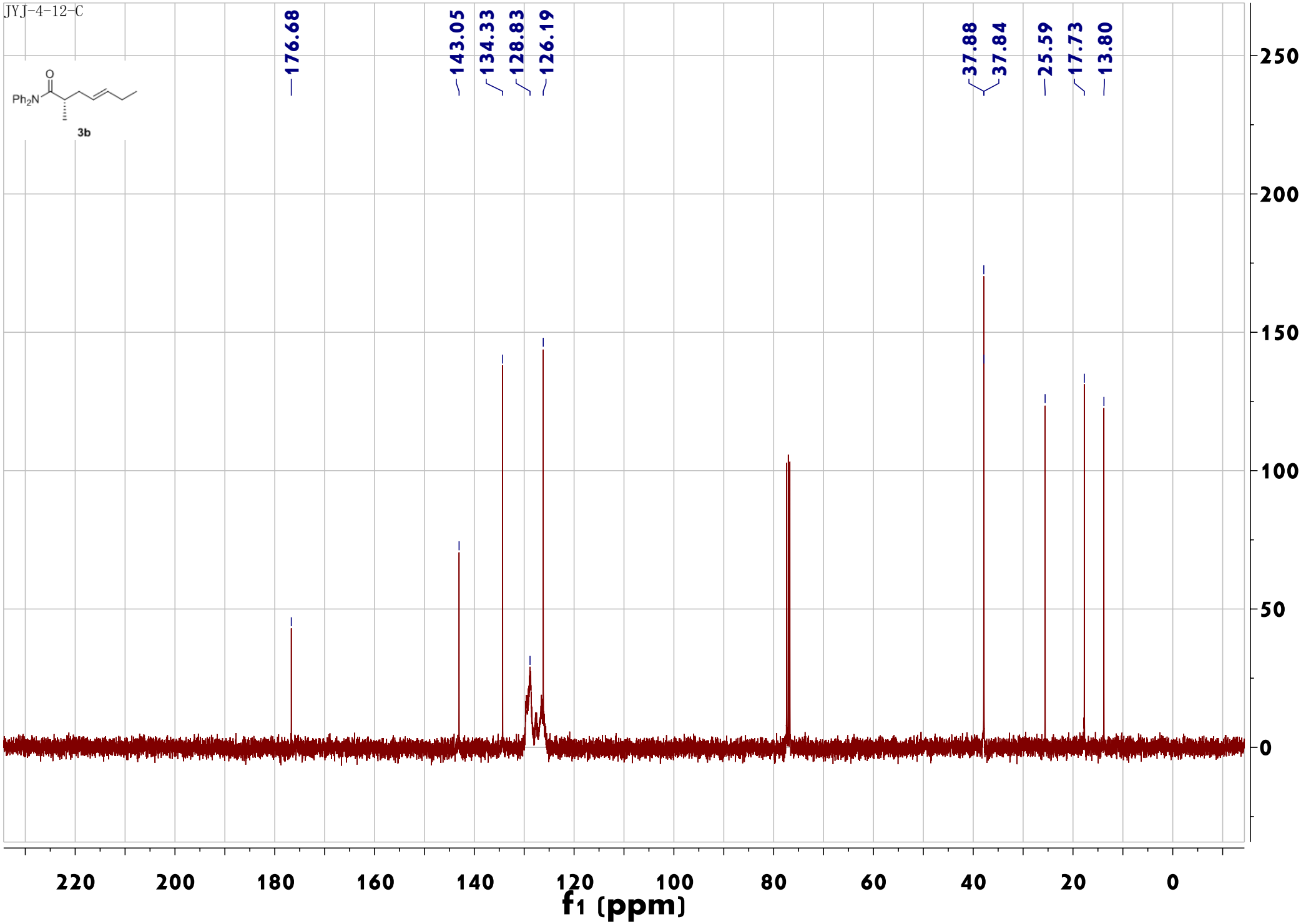
1 Det.A Ch1/214nm

PeakTable

Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.220	27462370	571498	94.985	95.345
2	24.700	1449818	27899	5.015	4.655
Total		28912188	599397	100.000	100.000

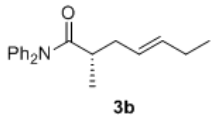

JYJ-5-17-H
7.34
7.26
7.24
7.22
5.58
5.56
5.54
5.52
5.51
5.35
5.34
5.32
5.30
5.28
2.64
2.62
2.60
2.46
2.44
2.42
2.07
2.05
2.04
2.02
2.00
1.15
1.13
1.01
1.00
0.98
Ph2N
3b
10.04
1.00
1.02
1.06
1.11
3.25
3.13
3.08
f1 (ppm)


JYJ-4-12-C
Ph2N
3b
176.68
143.05
134.33
128.83
126.19
37.88
37.84
25.59
17.73
13.80
250
200
150
100
50
0
220
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\5\jyj-5-46-rac.lcd

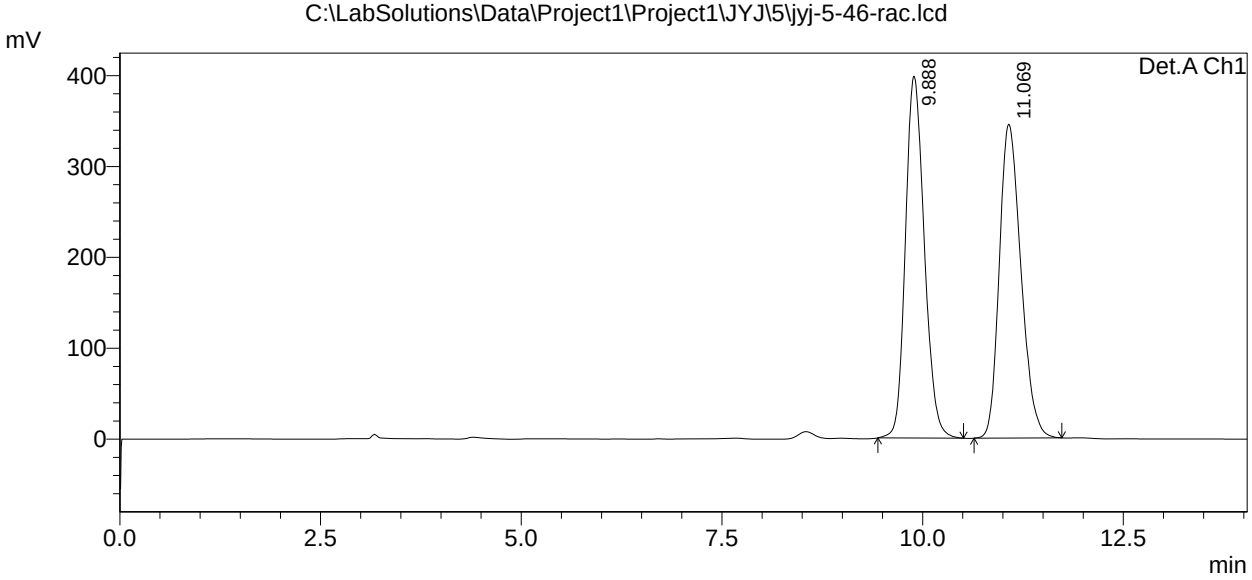
Acquired by	: Admin
Sample Name	: jyj-5-46-rac
Sample ID	: OD-H,99/1,1,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: jyj-5-46-rac.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2016-9-11 9:34:21
Data Processed	: 2016-9-11 10:08:41



3b

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\5\jyj-5-46-rac.lcd



1 Det.A Ch1/214nm

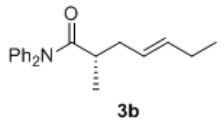
PeakTable

Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.888	6525634	398221	50.195	53.560
2	11.069	6474938	345290	49.805	46.440
Total		13000572	743511	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\5\jyj-5-60.lcd

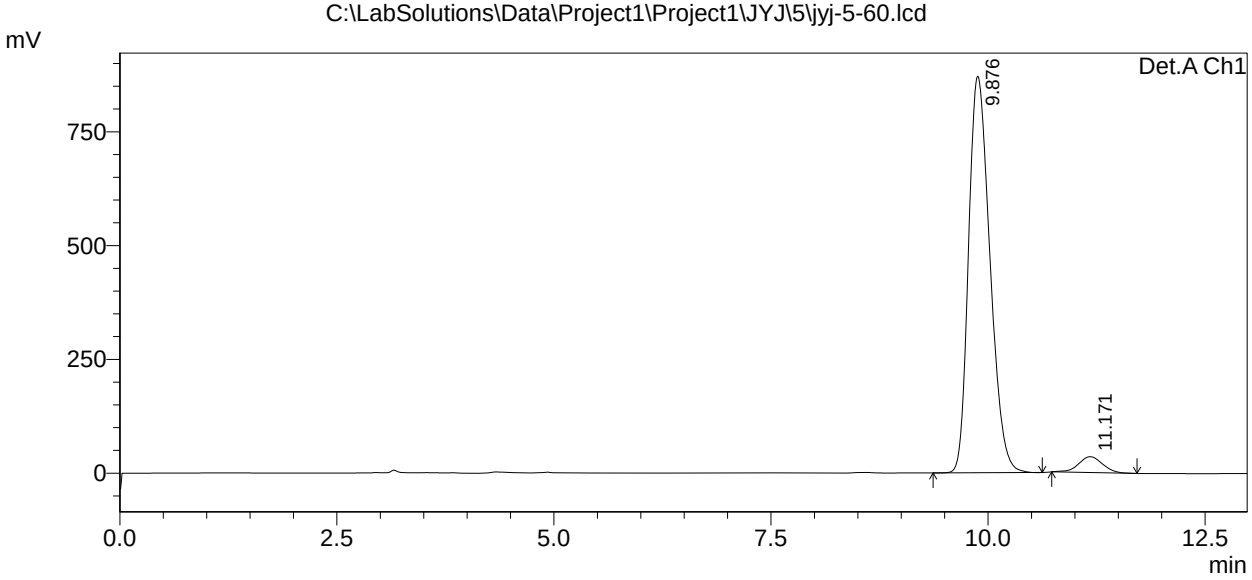


3b

Acquired by	: Admin
Sample Name	: jyj-5-60
Sample ID	: OD-H,99/1,1,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: jyj-5-60.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2016-9-24 22:18:14
Data Processed	: 2016-9-24 22:44:39

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\5\jyj-5-60.lcd

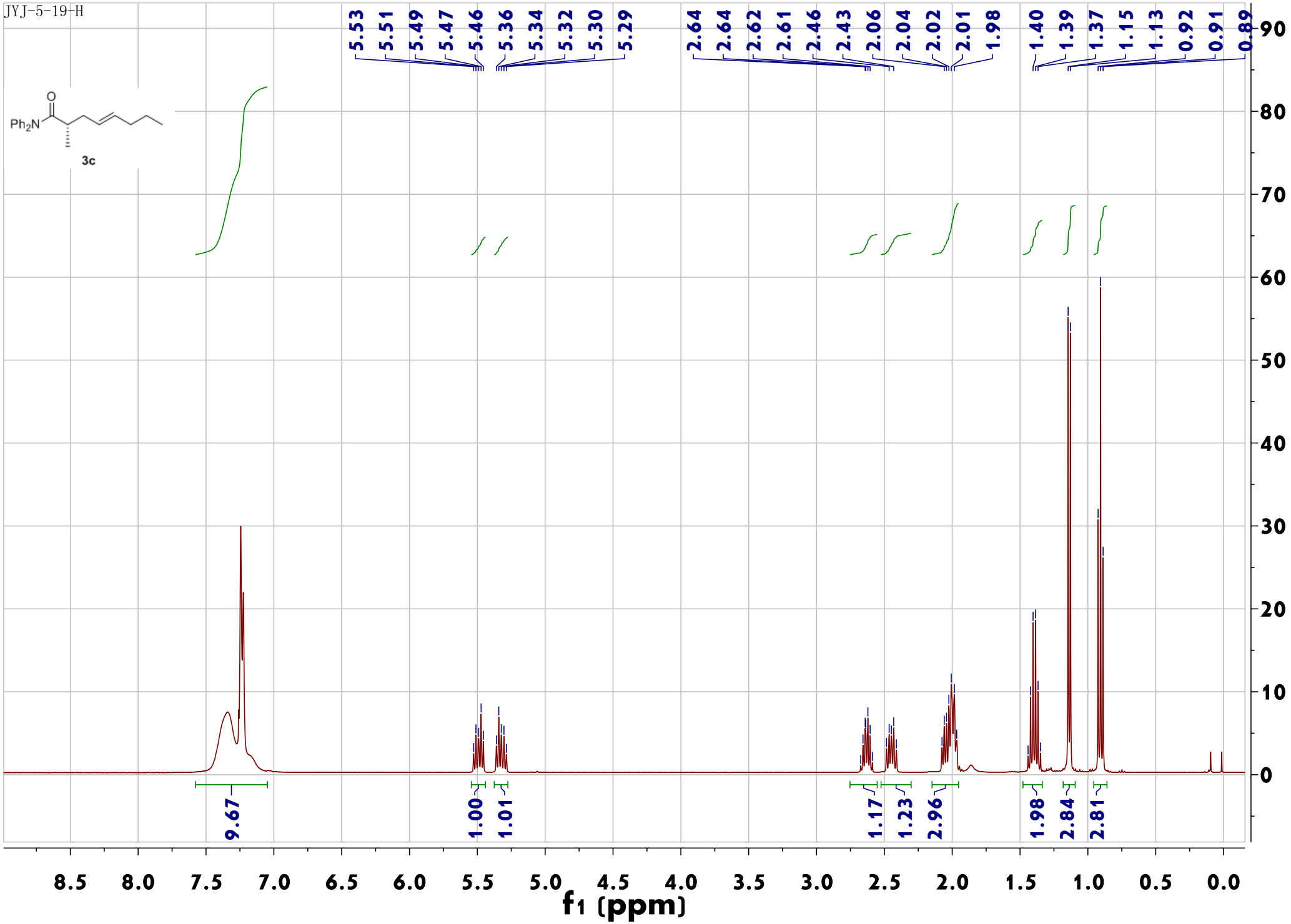


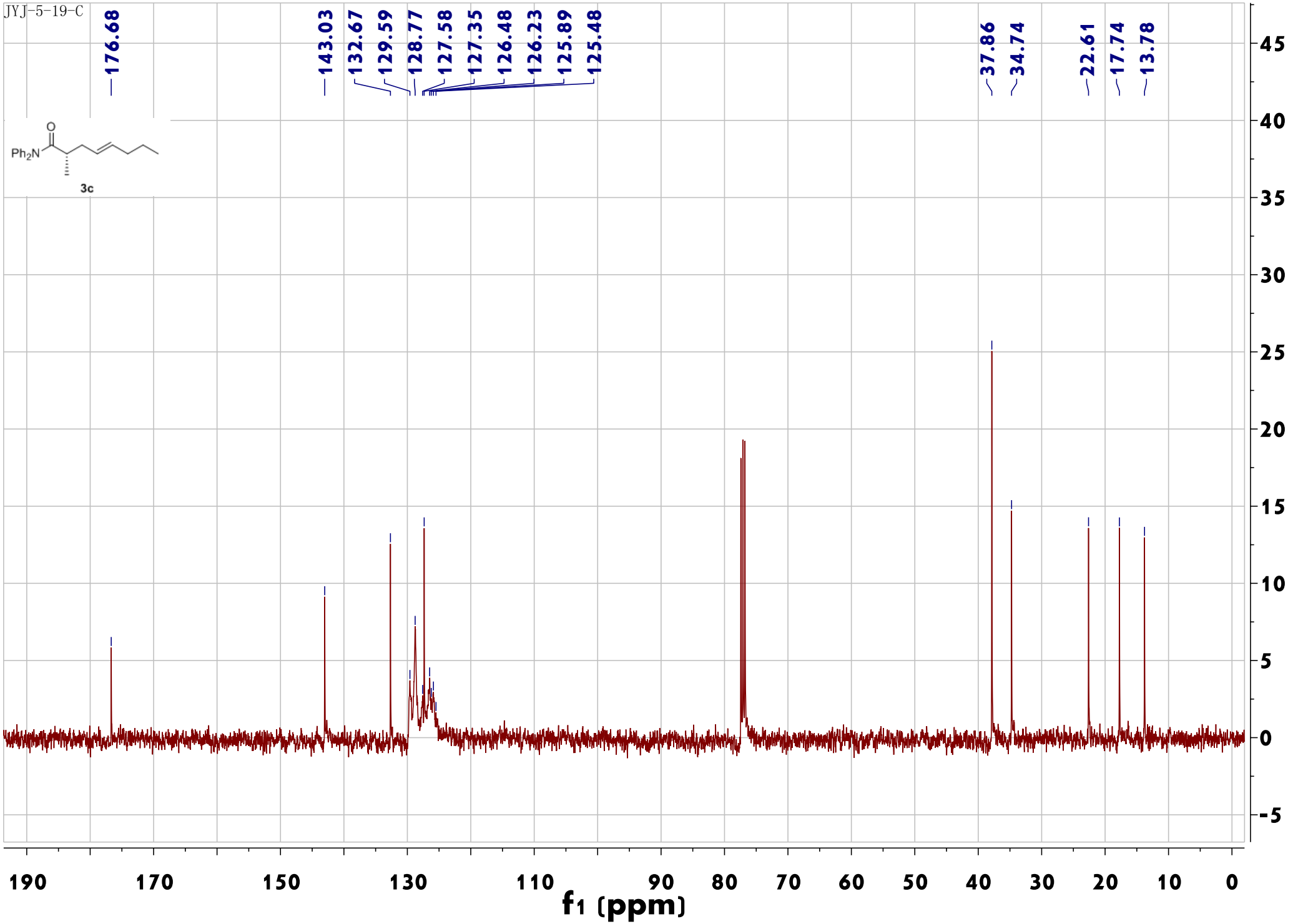
1 Det.A Ch1/214nm

PeakTable

Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.876	14917162	871371	95.676	96.136
2	11.171	674226	35023	4.324	3.864
Total		15591388	906394	100.000	100.000

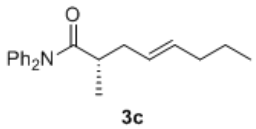

JYJ-5-19-H
5.53
5.51
5.49
5.47
5.46
5.36
5.34
5.32
5.30
5.29
2.64
2.64
2.62
2.61
2.46
2.43
2.06
2.04
2.02
2.01
1.98
1.40
1.39
1.37
1.15
1.13
0.92
0.91
0.89
Ph2N
O
3c
9.67
1.00
1.01
1.17
1.23
2.96
1.98
2.84
2.81
f1 (ppm)


JYJ-5-19-C
176.68
143.03
132.67
129.59
128.77
127.58
127.35
126.48
126.23
125.89
125.48
37.86
34.74
22.61
17.74
13.78
Ph2N
3c
f1 (ppm)

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\5\jyj-5-47-RAC.lcd

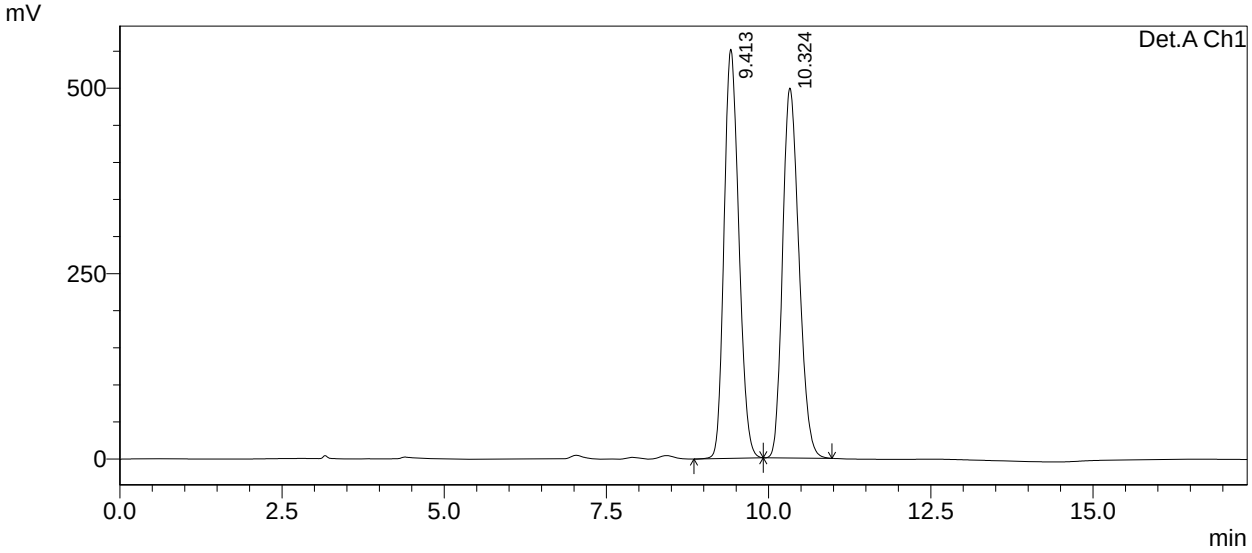
Acquired by	: Admin
Sample Name	: jyj-5-47-RAC
Sample ID	: OD-H,99/1,1,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: jyj-5-47-RAC.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2016-9-11 10:13:03
Data Processed	: 2016-9-11 10:44:03



3c

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\5\jyj-5-47-RAC.lcd



1 Det.A Ch1/214nm

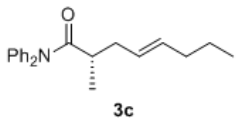
PeakTable

Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.413	8753559	551879	49.937	52.524
2	10.324	8775757	498831	50.063	47.476
Total		17529316	1050710	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\5\jyj-5-61.lcd

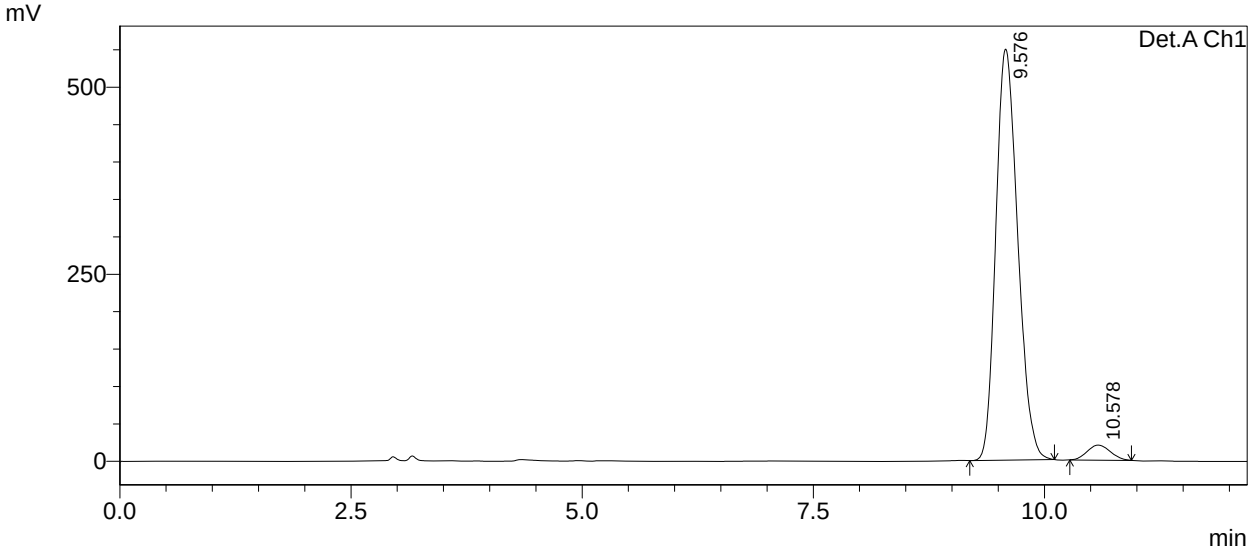


3c

Acquired by	: Admin
Sample Name	: jyj-5-61
Sample ID	: OD-H,99/1,1,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: jyj-5-61.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2016-9-24 22:32:18
Data Processed	: 2016-9-25 18:41:11

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\5\jyj-5-61.lcd

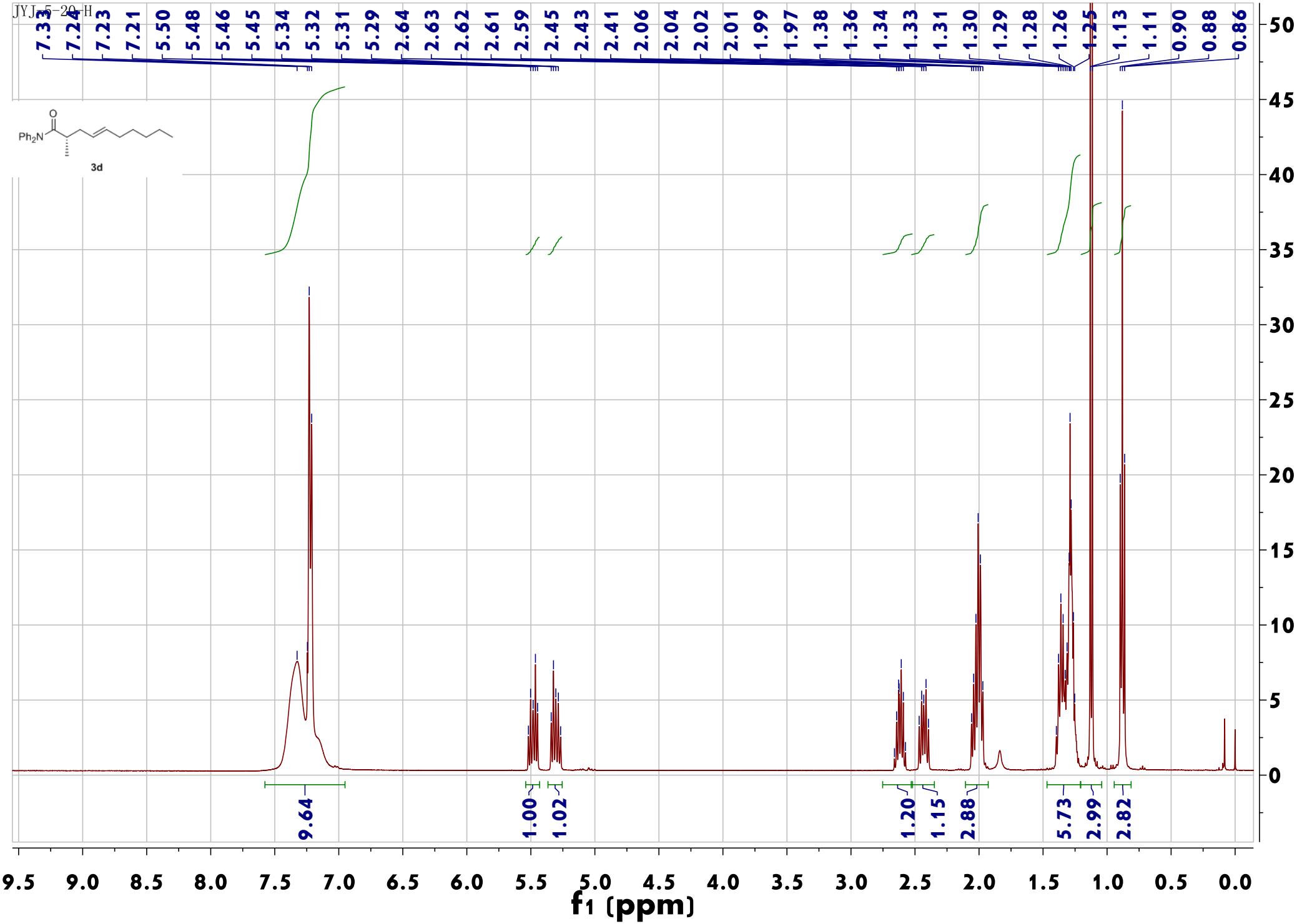


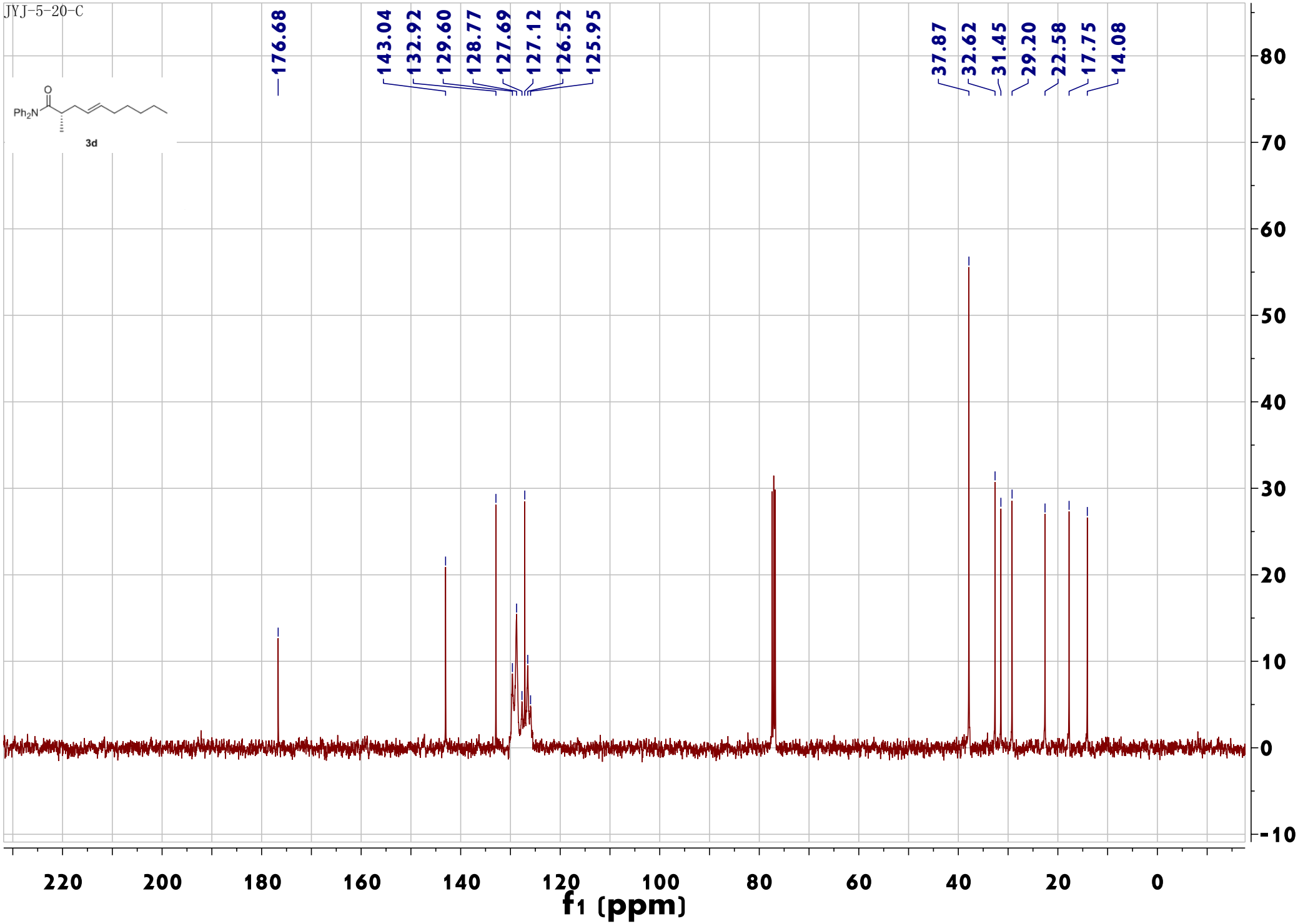
1 Det.A Ch1/214nm

PeakTable

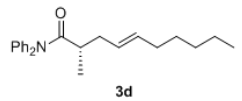
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.576	9049392	549273	96.230	96.440
2	10.578	354551	20275	3.770	3.560
Total		9403943	569548	100.000	100.000


JYJ-5-20-H
7.33
7.24
7.23
7.21
5.50
5.48
5.46
5.45
5.34
5.32
5.31
5.29
2.64
2.63
2.62
2.61
2.59
2.45
2.43
2.41
2.06
2.04
2.02
2.01
1.99
1.97
1.38
1.36
1.34
1.33
1.31
1.30
1.29
1.28
1.26
1.25
1.13
1.11
0.90
0.88
0.86
Ph2N
3d
9.64
1.00
1.02
1.20
1.15
2.88
5.73
2.99
2.82
f1 (ppm)

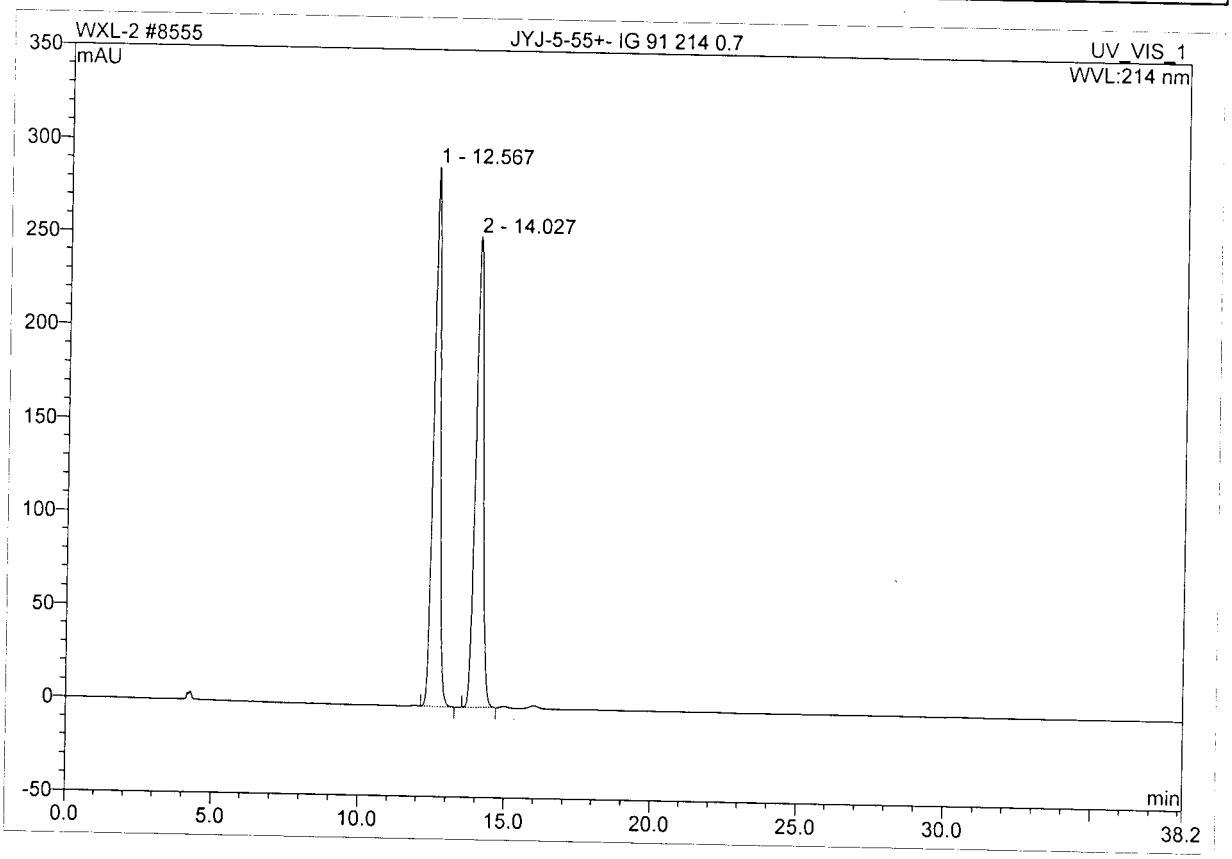

JYJ-5-20-C
176.68
143.04
132.92
129.60
128.77
127.69
127.12
126.52
125.95
37.87
32.62
31.45
29.20
22.58
17.75
14.08
Ph2N
3d
f1 (ppm)

8555 JYJ-5-55+- IG 91 214 0.7



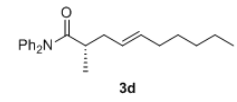
3d

Sample Name:	**JYJ-5-55+- IG 91 214 0.7**	*Injection Volume:*	**3.0**
Vial Number:	**RC6**	*Channel:*	**UV_VIS_1**
Sample Type:	**unknown**	*Wavelength:*	**214**
Control Program:	**WXL-2014-4**	*Bandwidth:*	**n.a.**
Quantif. Method:	**WXL**	*Dilution Factor:*	**1.0000**
Recording Time:	**2016/9/21 13:09**	*Sample Weight:*	**1.0000**
Run Time (min):	**38.15**	*Sample Amount:*	**1.0000**



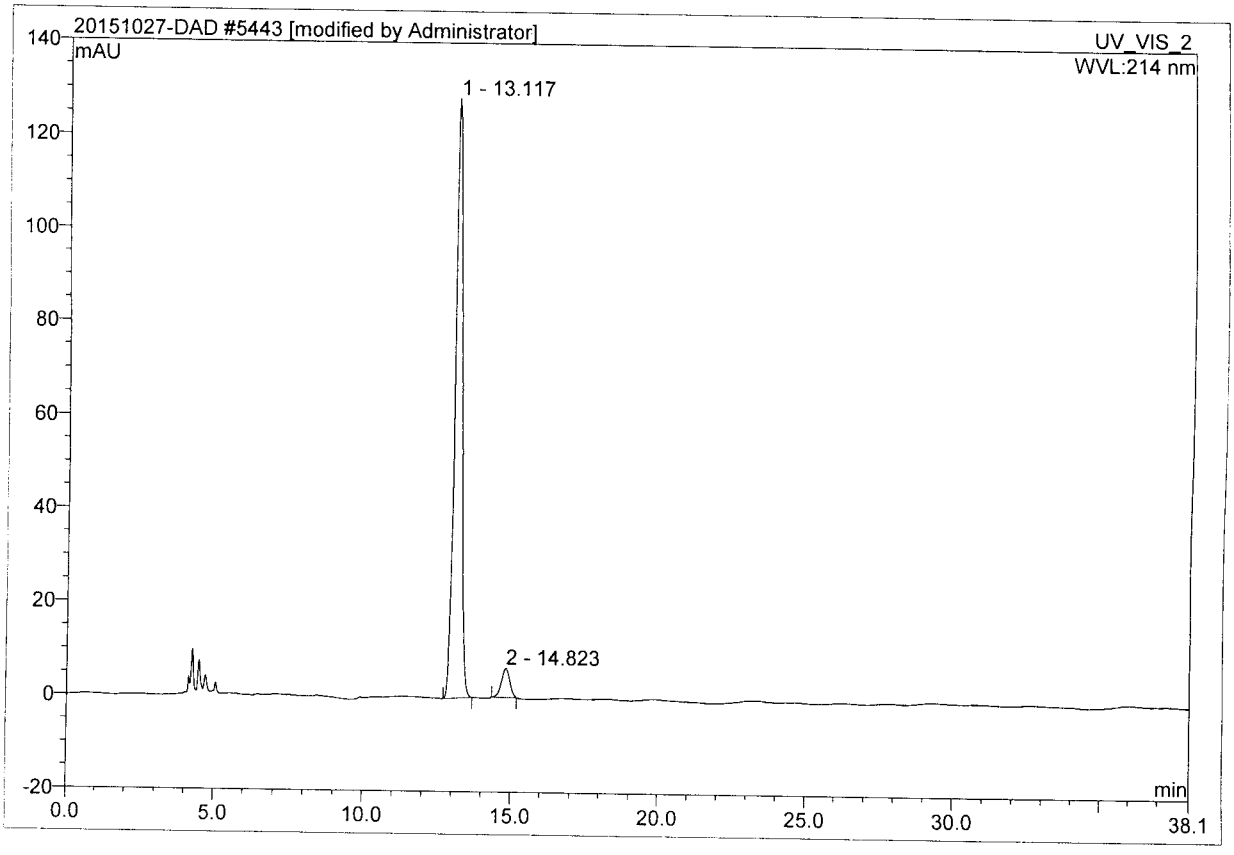
No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	12.57	n.a.	289.356	78.989	50.38	n.a.	BMB
2	14.03	n.a.	252.287	77.808	49.62	n.a.	BMB
Total:			541.642	156.797	100.00	0.000	

5443 JYJ-7-8 IG 91 214 0.7

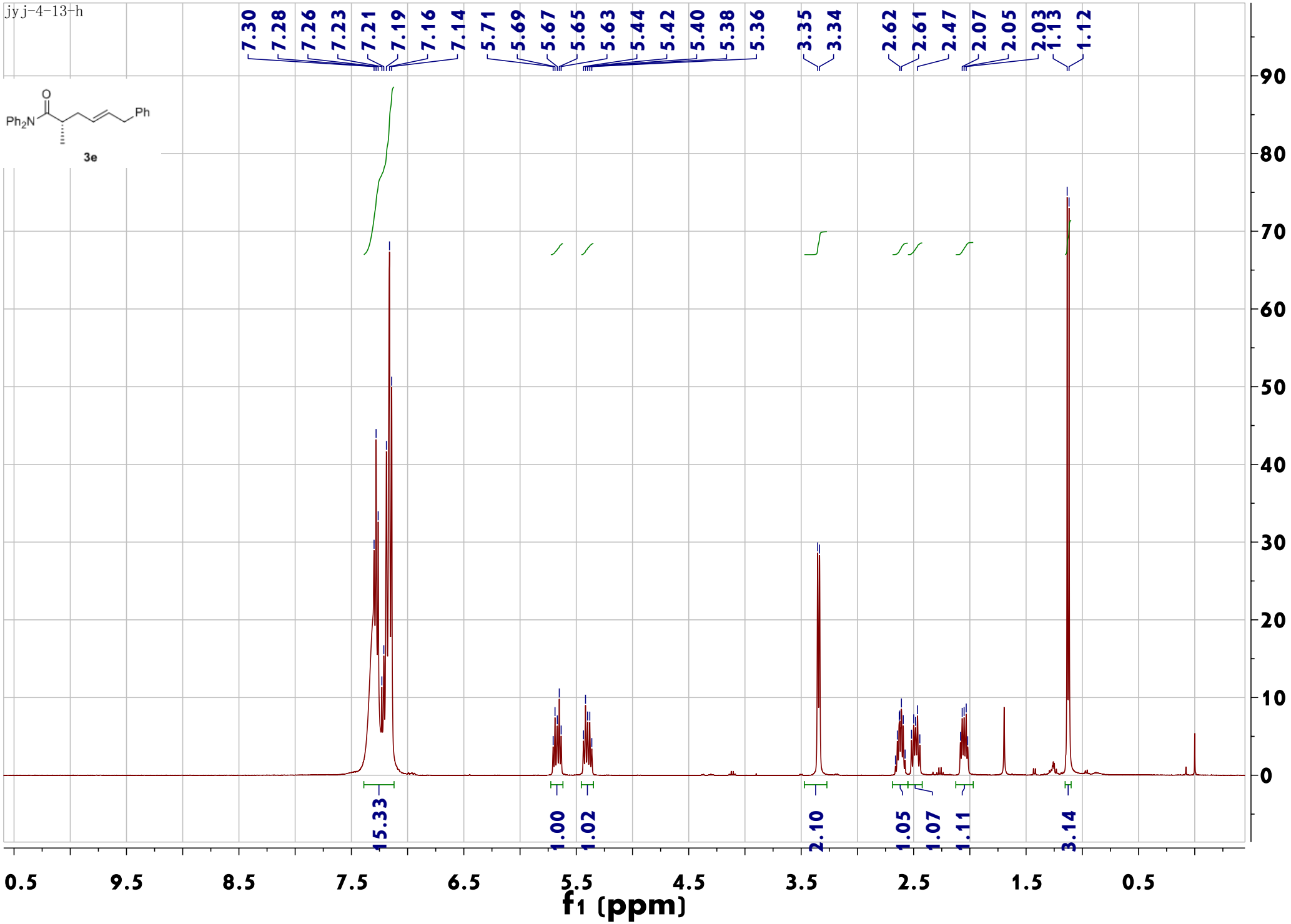


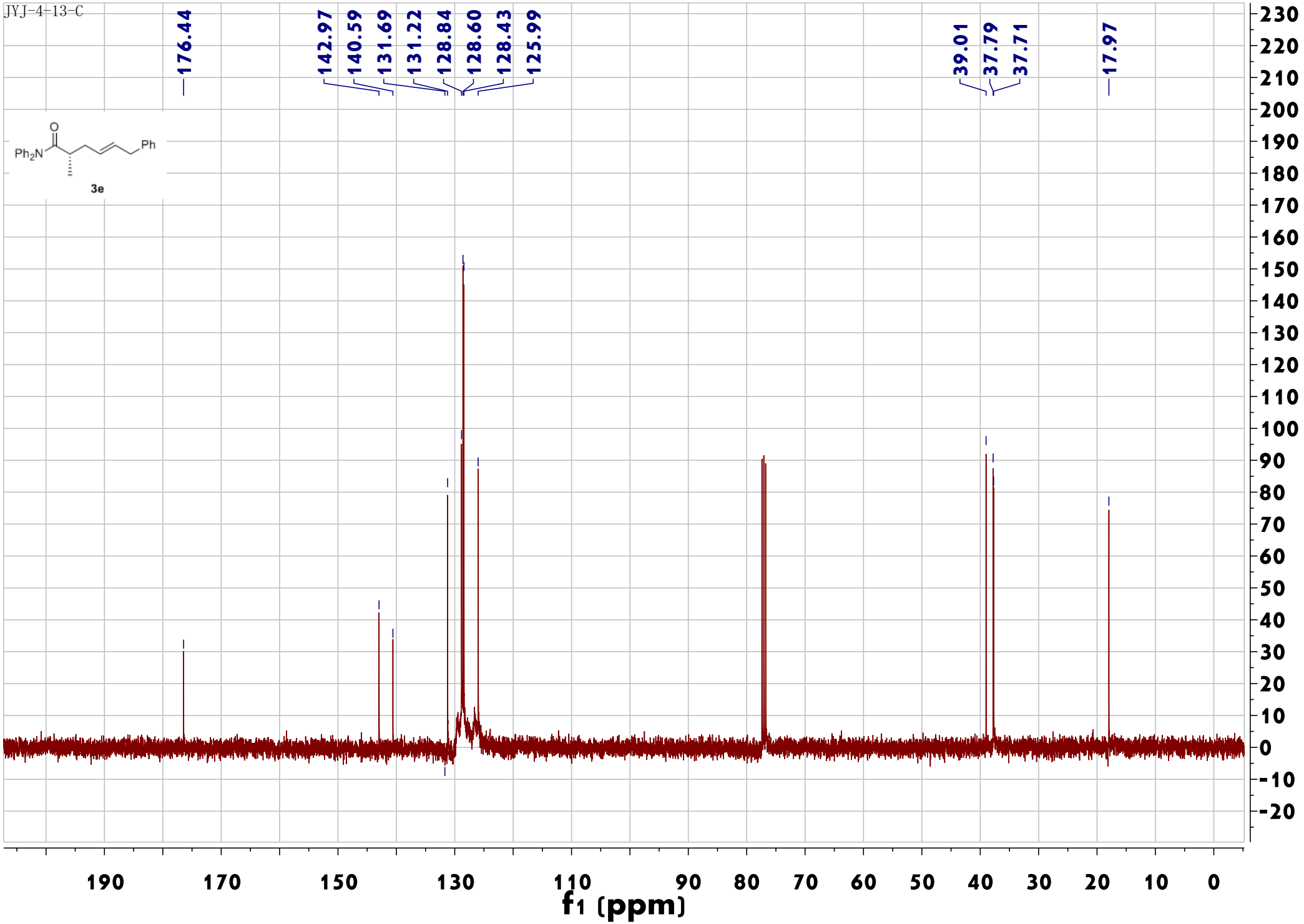
3d

Sample Name:	**JYJ-7-8 IG 91 214 0.7**	*Injection Volume:*	**2.0**
Vial Number:	**RD4**	*Channel:*	**UV_VIS_2**
Sample Type:	**unknown**	*Wavelength:*	**214.0**
Control Program:	**test-dad5**	*Bandwidth:*	**4**
Quantif. Method:	**WXL**	*Dilution Factor:*	**1.0000**
Recording Time:	**2017-2-24 10:35**	*Sample Weight:*	**1.0000**
Run Time (min):	**38.09**	*Sample Amount:*	**1.0000**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	13.12	n.a.	128.146	36.392	94.61	n.a.	BMB
2	14.82	n.a.	6.244	2.071	5.39	n.a.	BMB
Total:			134.390	38.463	100.00	0.000	

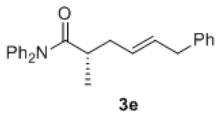

jyj-4-13-h
7.30
7.28
7.26
7.23
7.21
7.19
7.16
7.14
5.71
5.69
5.67
5.65
5.63
5.44
5.42
5.40
5.38
5.36
3.35
3.34
2.62
2.61
2.47
2.07
2.05
2.03
1.13
1.12
Ph2N
Ph
O
3e
15.33
1.00
1.02
2.10
1.05
1.07
1.11
3.14
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
f1 (ppm)


JYJ-4-13-C
176.44
142.97
140.59
131.69
131.22
128.84
128.60
128.43
125.99
39.01
37.79
37.71
17.97
Ph2N
O
Ph
3e
f1 (ppm)

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\5\JYJ-5-56-RAC.lcd

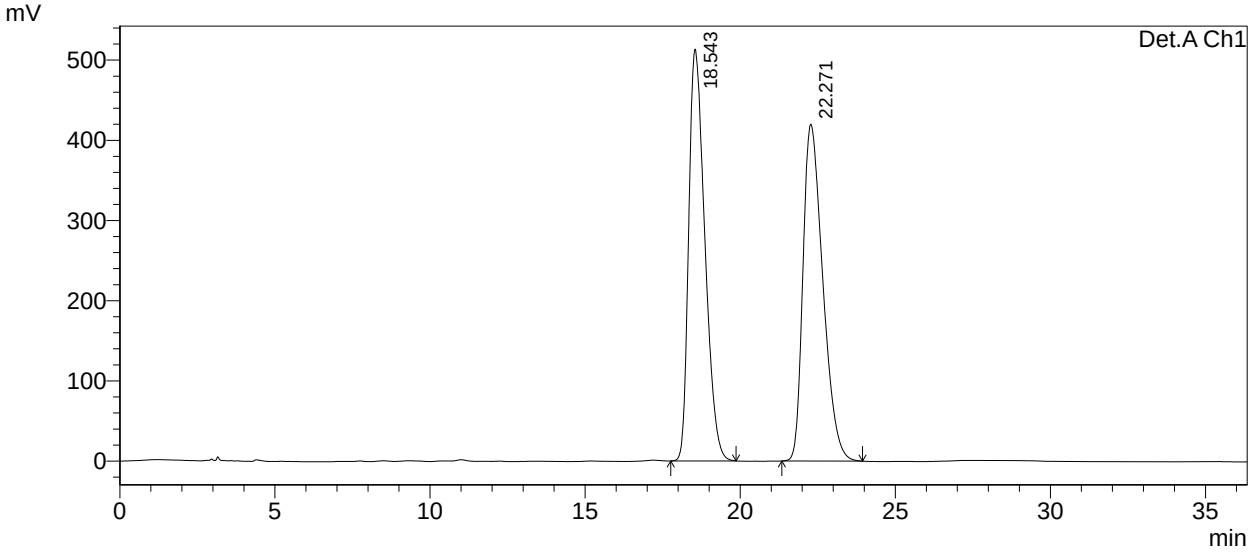
Acquired by	: Admin
Sample Name	: JYJ-5-56-RAC
Sample ID	: OD-H,99/1,1,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: JYJ-5-56-RAC.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2016-9-19 10:02:38
Data Processed	: 2016-9-19 11:07:55



3e

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\5\JYJ-5-56-RAC.lcd



1 Det.A Ch1/214nm

PeakTable

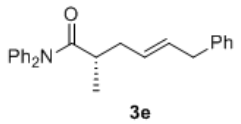
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.543	18578616	513453	49.820	55.007
2	22.271	18712804	419981	50.180	44.993
Total		37291420	933434	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\5\JYJ-5-56.lcd

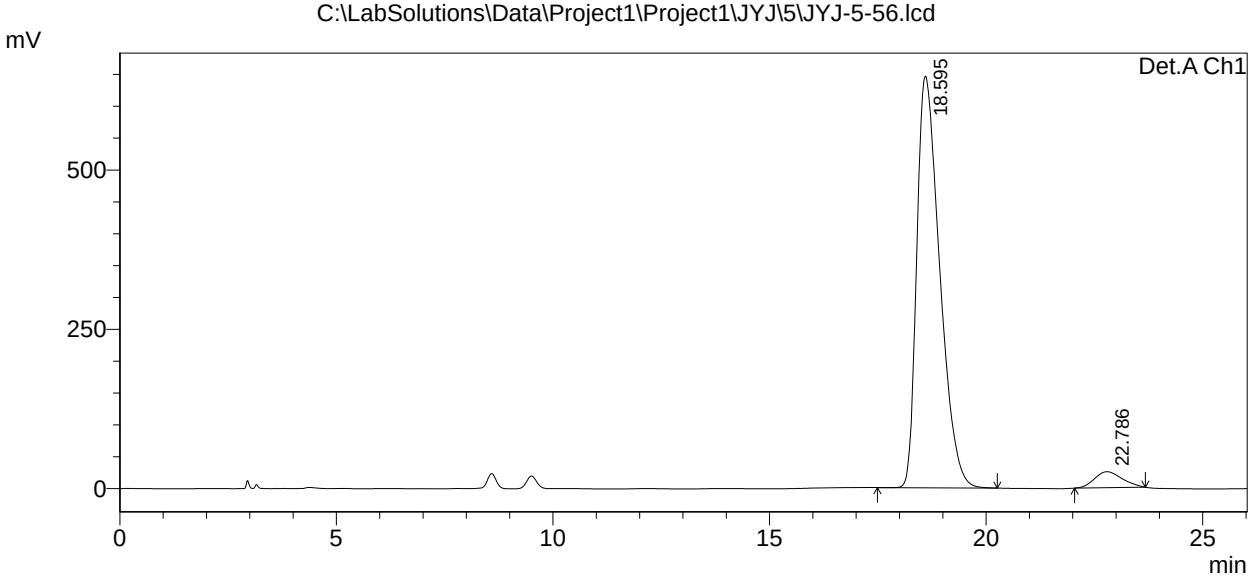
Acquired by	: Admin
Sample Name	: JYJ-5-56
Sample ID	: OD-H,99/1,1,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: JYJ-5-56.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2016-9-19 10:41:42
Data Processed	: 2016-9-19 11:11:00



3e

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\5\JYJ-5-56.lcd

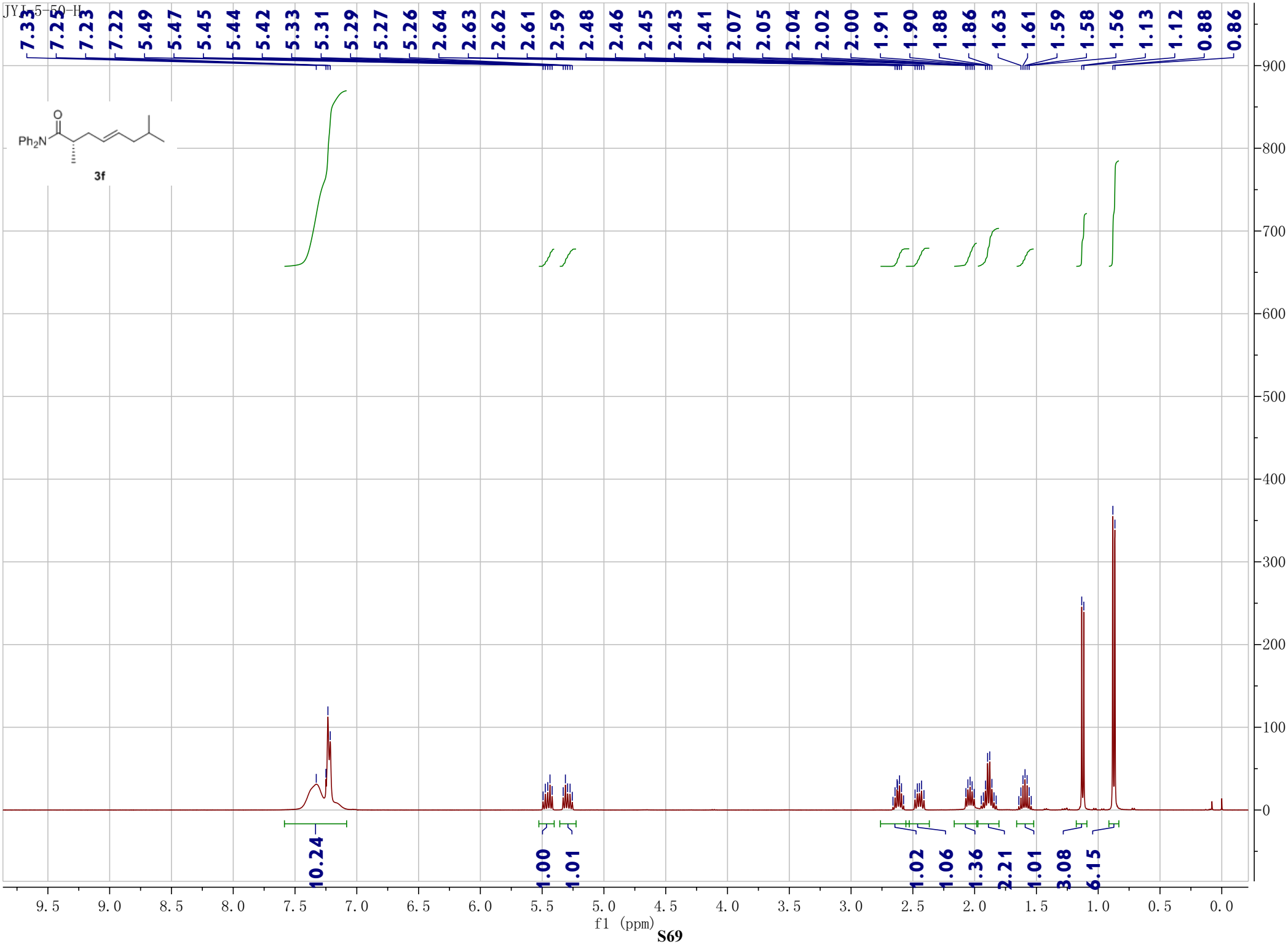


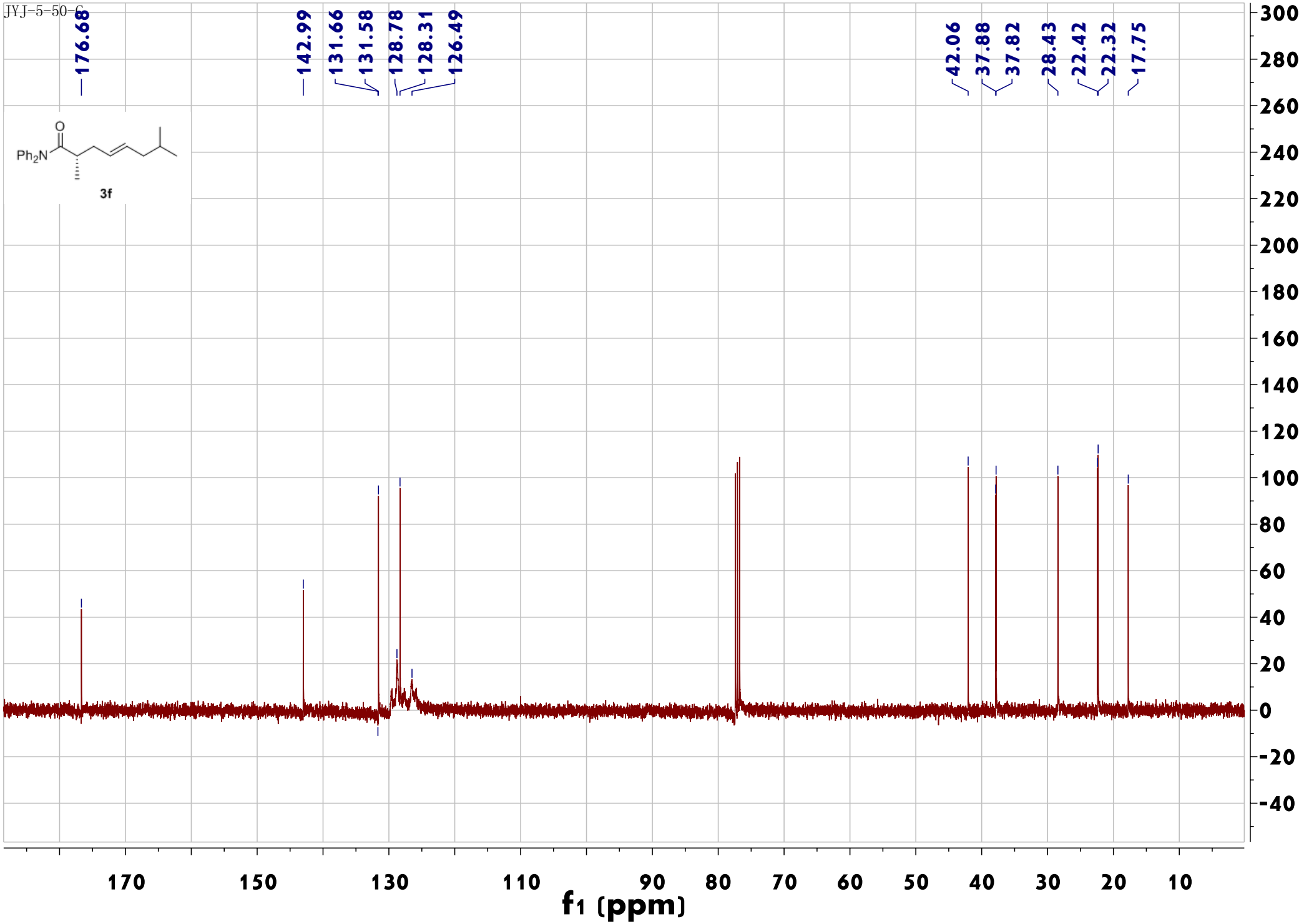
1 Det.A Ch1/214nm

PeakTable

Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.595	23764689	646177	95.501	96.255
2	22.786	1119647	25143	4.499	3.745
Total		24884337	671320	100.000	100.000

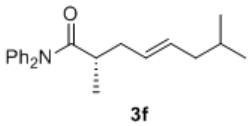

JYL-5-50-H
7.33
7.25
7.23
7.22
5.49
5.47
5.45
5.44
5.42
5.33
5.31
5.29
5.27
5.26
2.64
2.63
2.62
2.61
2.59
2.48
2.46
2.45
2.43
2.41
2.07
2.05
2.04
2.02
2.00
1.91
1.90
1.88
1.86
1.63
1.61
1.59
1.58
1.56
1.13
1.12
0.88
0.86
Ph2N
3f
10.24
1.00
1.01
1.02
1.06
1.36
2.21
1.01
3.08
6.15
f1 (ppm)


JYJ-5-50-6
176.68
142.99
131.66
131.58
128.78
128.31
126.49
42.06
37.88
37.82
28.43
22.42
22.32
17.75
Ph2N
O
3f
f1 (ppm)

==== Shimadzu LCsolution Analysis Report ====

C:\Users\hou-418\Desktop\JYJ\JYJ-5-68-RAC.lcd

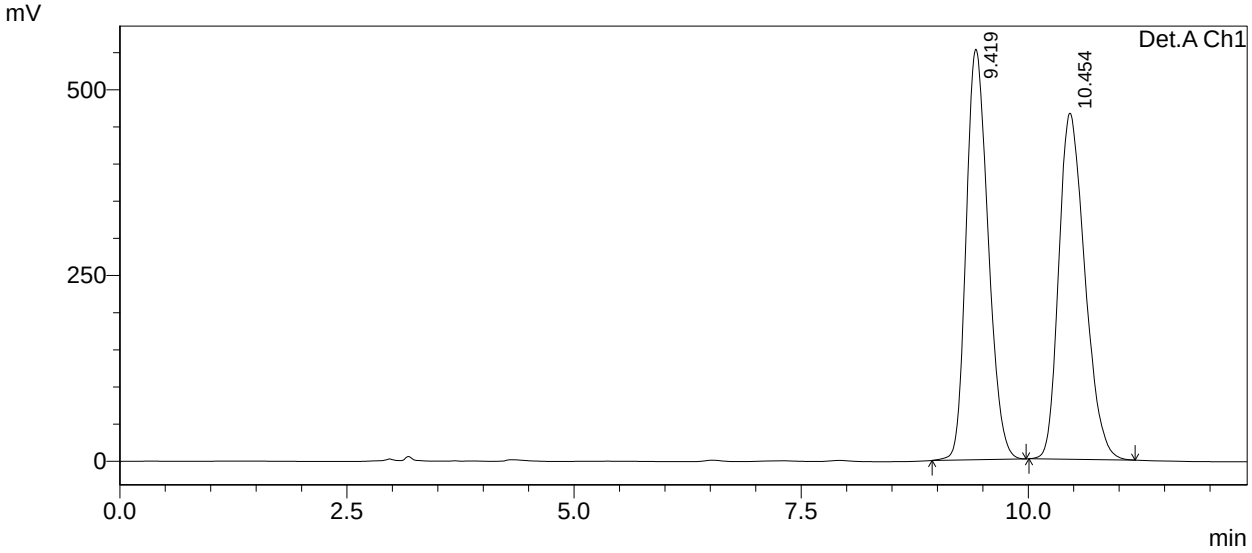
Acquired by	: Admin
Sample Name	: JYJ-5-68-RAC
Sample ID	: OD-H,99/1,1,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: JYJ-5-68-RAC.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2016-10-11 21:03:39
Data Processed	: 2016-10-11 21:33:29



3f

<Chromatogram>

C:\Users\hou-418\Desktop\JYJ\JYJ-5-68-RAC.lcd



1 Det.A Ch1/214nm

PeakTable

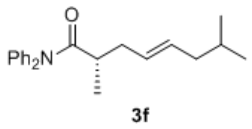
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.419	9308541	552707	49.943	54.257
2	10.454	9329847	465970	50.057	45.743
Total		18638389	1018677	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\Users\hou-418\Desktop\JYJ\JYJ-5-68.lcd

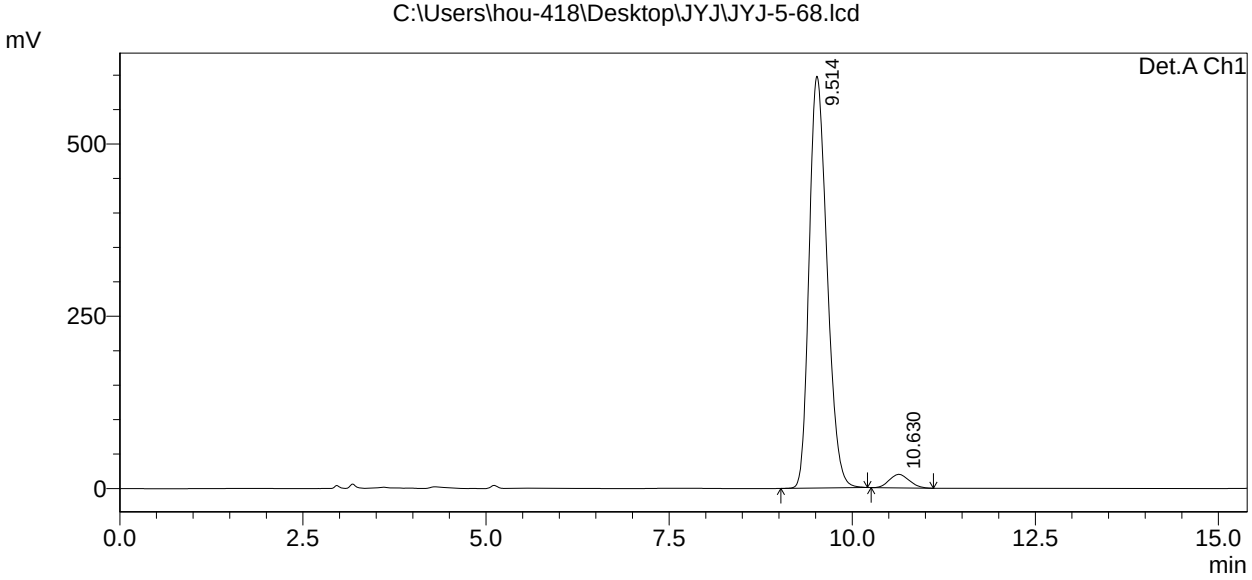
Acquired by	: Admin
Sample Name	: JYJ-5-68
Sample ID	: OD-H,99/1,1,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: JYJ-5-68.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2016-10-11 21:17:15
Data Processed	: 2016-10-12 8:43:59



3f

<Chromatogram>

C:\Users\hou-418\Desktop\JYJ\JYJ-5-68.lcd

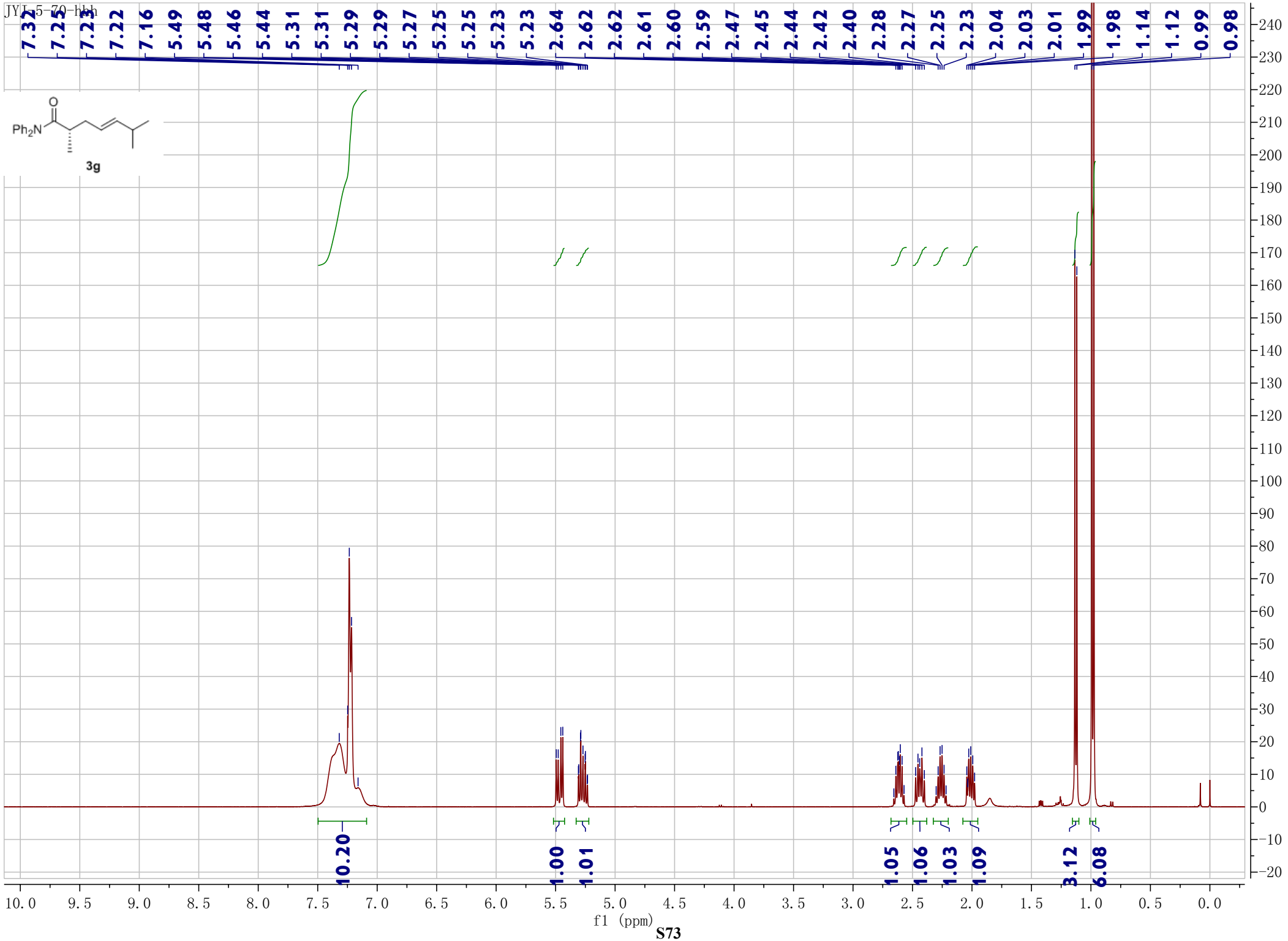


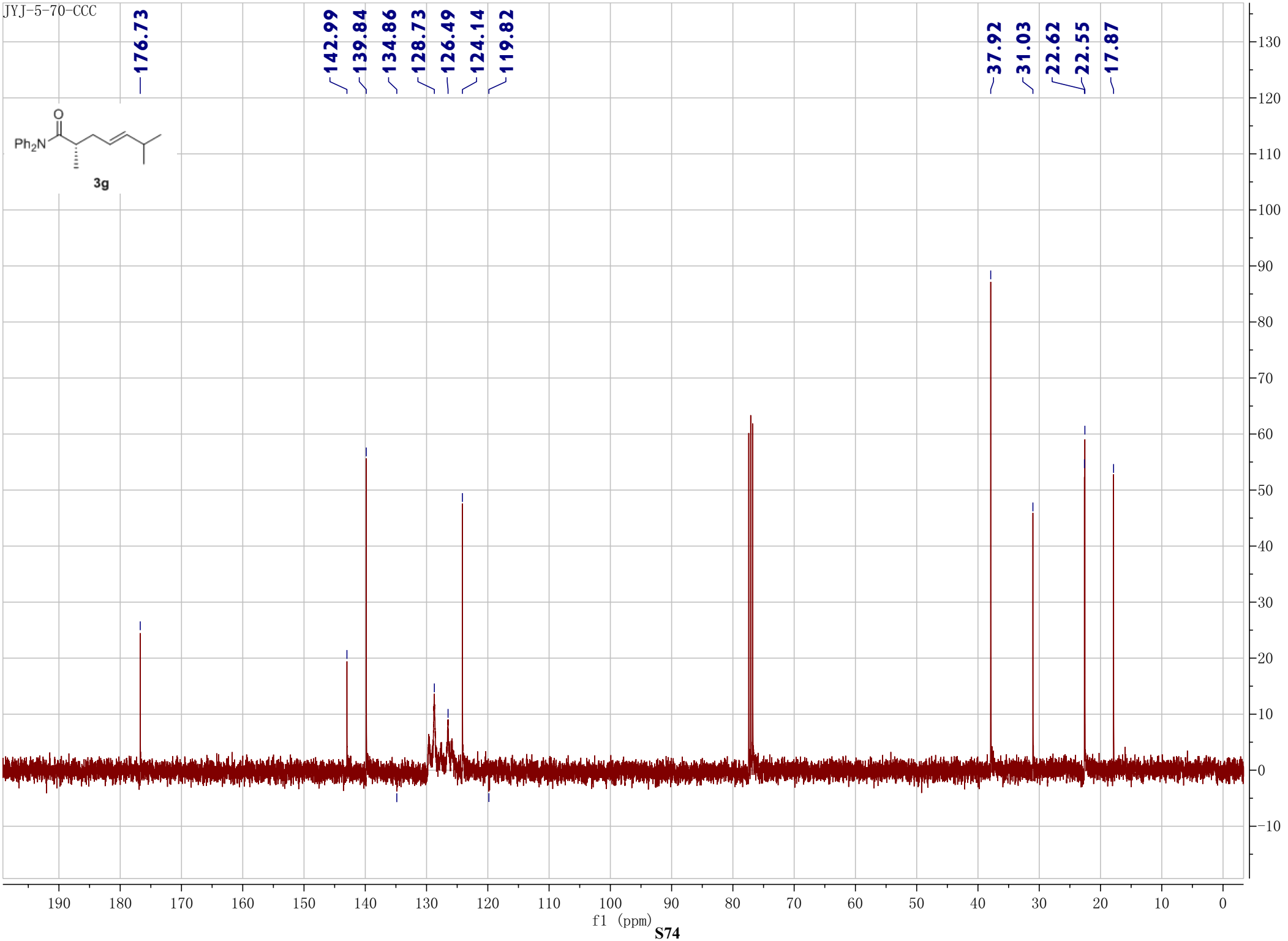
1 Det.A Ch1/214nm

PeakTable

Detector A Ch1 214nm

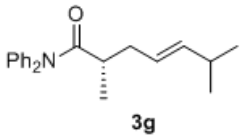
Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.514	10170178	597868	96.445	96.822
2	10.630	374859	19624	3.555	3.178
Total		10545036	617492	100.000	100.000


JYL-5-70-hhh
7.32
7.25
7.23
7.22
7.16
5.49
5.48
5.46
5.44
5.31
5.31
5.29
5.29
5.27
5.25
5.25
5.23
5.23
2.64
2.62
2.62
2.61
2.60
2.59
2.47
2.45
2.44
2.42
2.40
2.28
2.27
2.25
2.23
2.04
2.03
2.01
1.99
1.98
1.14
1.12
0.99
0.98
Ph2N
O
3g
10.20
1.00
1.01
1.05
1.06
1.03
1.09
3.12
6.08
f1 (ppm)


JYJ-5-70-CCC
176.73
142.99
139.84
134.86
128.73
126.49
124.14
119.82
37.92
31.03
22.62
22.55
17.87
Ph2N
O
3g
f1 (ppm)

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\5\JYJ-5-69-RAC.lcd

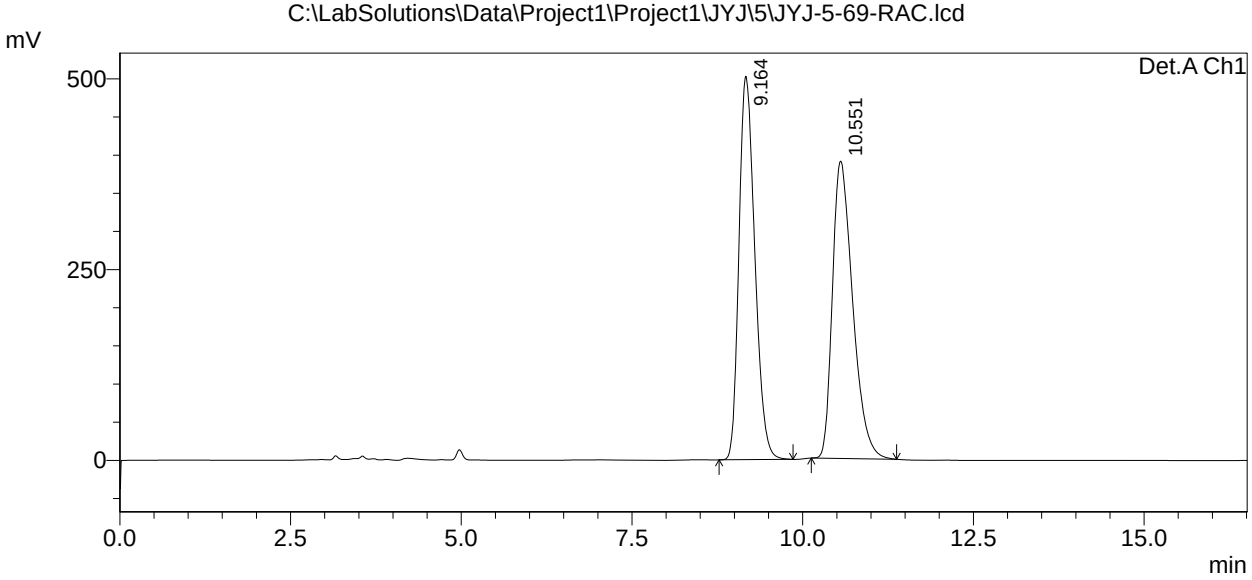


3g

Acquired by	: Admin
Sample Name	: JYJ-5-69-RAC
Sample ID	: OD-H,99/1,1,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: JYJ-5-69-RAC.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2016-10-11 20:12:05
Data Processed	: 2016-10-11 20:46:58

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\5\JYJ-5-69-RAC.lcd



1 Det.A Ch1/214nm

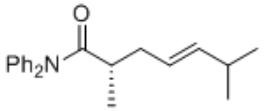
PeakTable

Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.164	8130042	502691	50.219	56.330
2	10.551	8059217	389718	49.781	43.670
Total		16189258	892409	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\5\JYJ-5-70.lcd

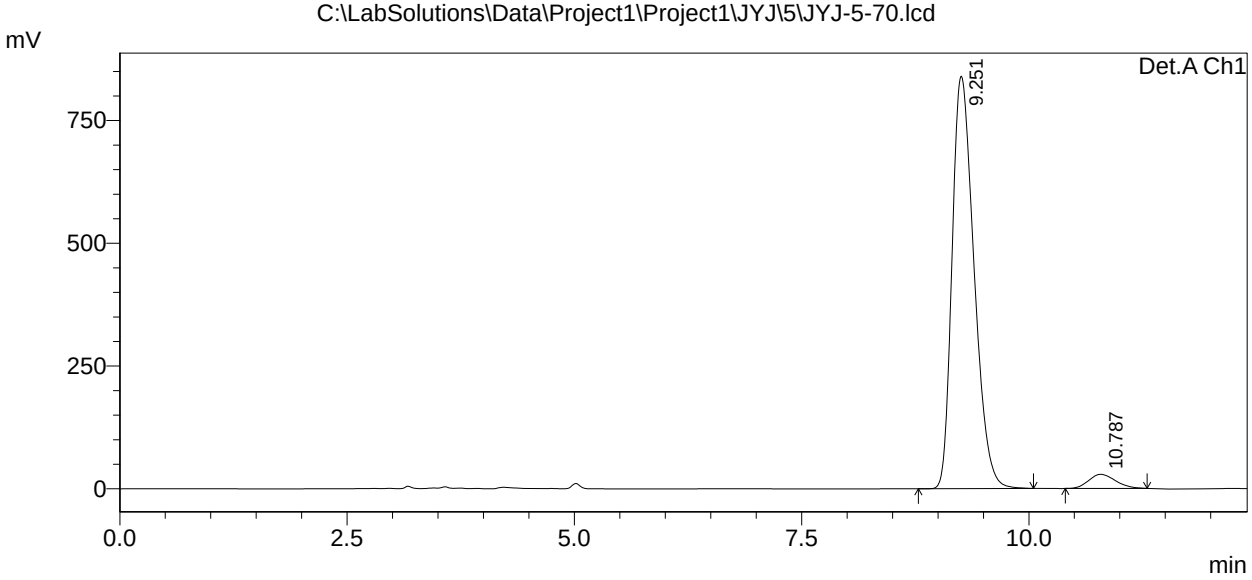


3g

Acquired by	: Admin
Sample Name	: JYJ-5-70
Sample ID	: OD-H,99/1,1,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: JYJ-5-70.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2016-10-11 20:34:17
Data Processed	: 2016-10-11 20:47:42

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\5\JYJ-5-70.lcd

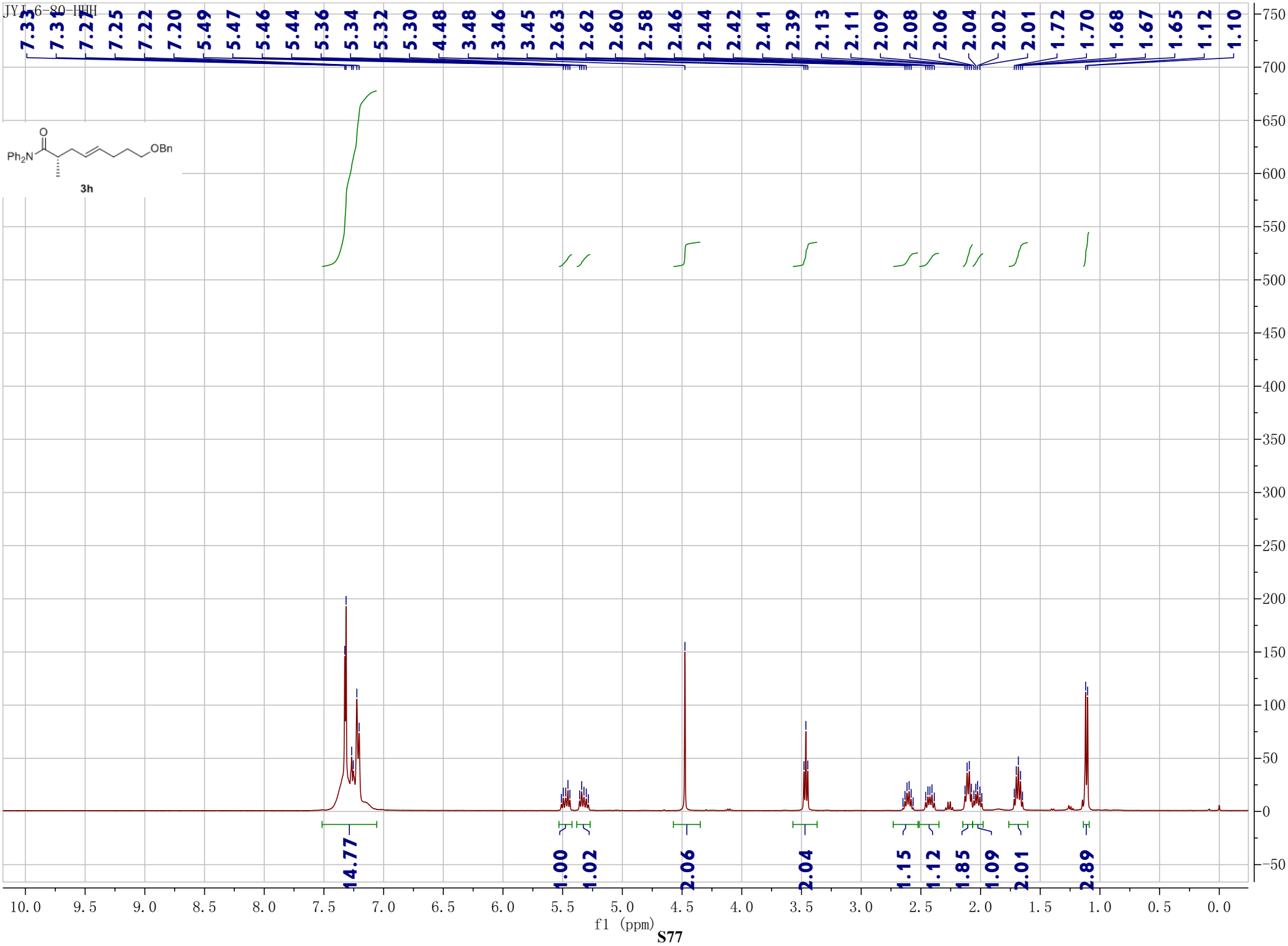


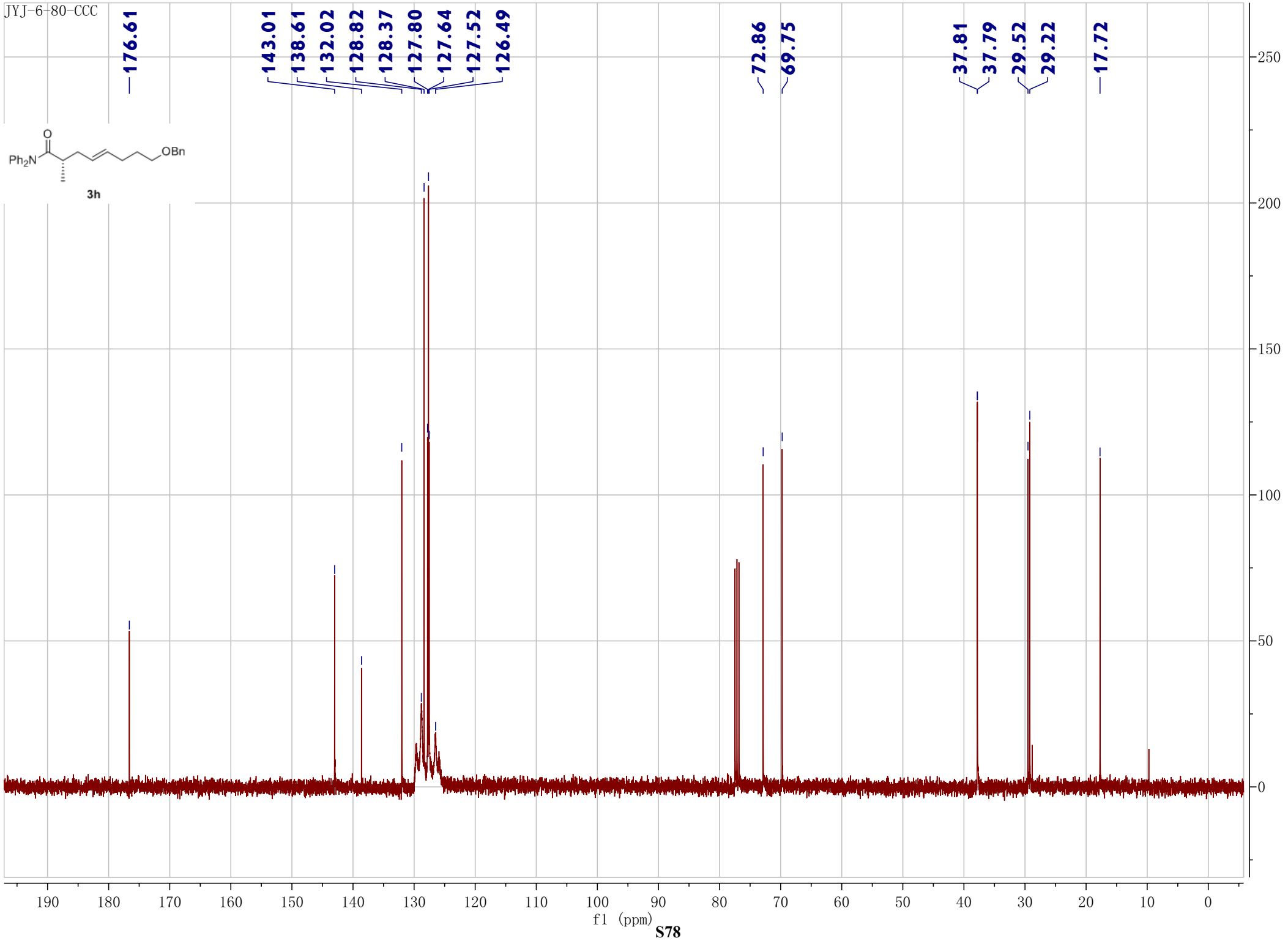
1 Det.A Ch1/214nm

PeakTable

Detector A Ch1 214nm

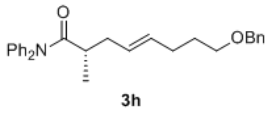
Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.251	14105938	840023	95.943	96.670
2	10.787	596475	28933	4.057	3.330
Total		14702413	868956	100.000	100.000


JYL-6-80-HHH
7.33
7.31
7.27
7.25
7.22
7.20
5.49
5.47
5.46
5.44
5.36
5.34
5.32
5.30
4.48
3.48
3.46
3.45
2.63
2.62
2.60
2.58
2.46
2.44
2.42
2.41
2.39
2.13
2.11
2.09
2.08
2.06
2.04
2.02
2.01
1.72
1.70
1.68
1.67
1.65
1.12
1.10
Ph2N
OBn
3h
14.77
1.00
1.02
2.06
2.04
1.15
1.12
1.85
1.09
2.01
2.89
f1 (ppm)


JYJ-6-80-CCC
176.61
143.01
138.61
132.02
128.82
128.37
127.80
127.64
127.52
126.49
72.86
69.75
37.81
37.79
29.52
29.22
17.72
Ph2N
O
OBn
3h
250
200
150
100
50
0
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\6\JYJ-6-80-RAC-2.lcd

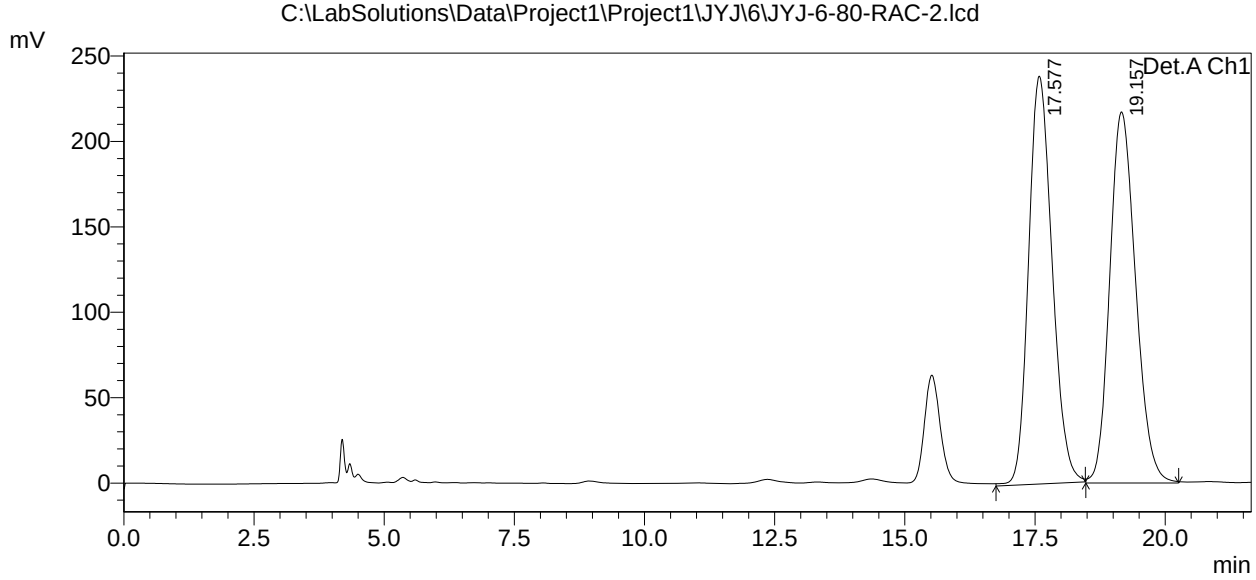


3h

Acquired by	: Admin
Sample Name	: JYJ-6-80-RAC-2
Sample ID	: OD-H,95/5,0.7,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: JYJ-6-80-RAC-2.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2017-1-7 19:06:03
Data Processed	: 2017-1-7 19:52:37

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\6\JYJ-6-80-RAC-2.lcd



1 Det.A Ch1/214nm

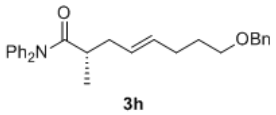
PeakTable

Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.577	7520350	238806	50.013	52.368
2	19.157	7516554	217205	49.987	47.632
Total		15036903	456011	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\6\JYJ-6-81-1.lcd

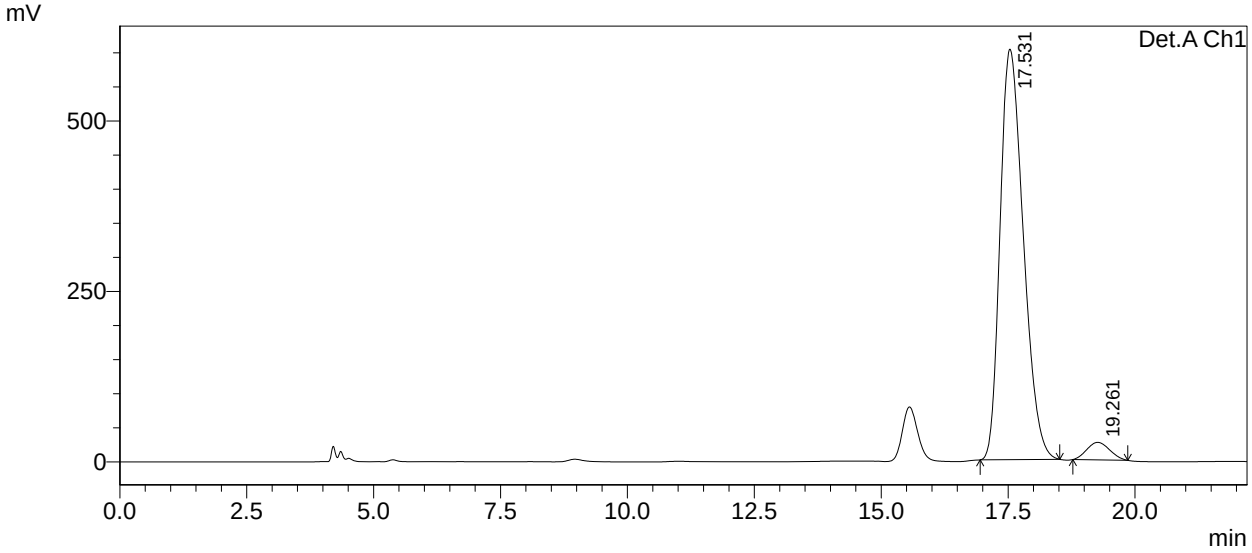


3h

Acquired by	: Admin
Sample Name	: JYJ-6-81-1
Sample ID	: OD-H,95/5,0.7,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: JYJ-6-81-1.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2017-1-7 19:28:46
Data Processed	: 2017-7-3 11:13:04

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\6\JYJ-6-81-1.lcd

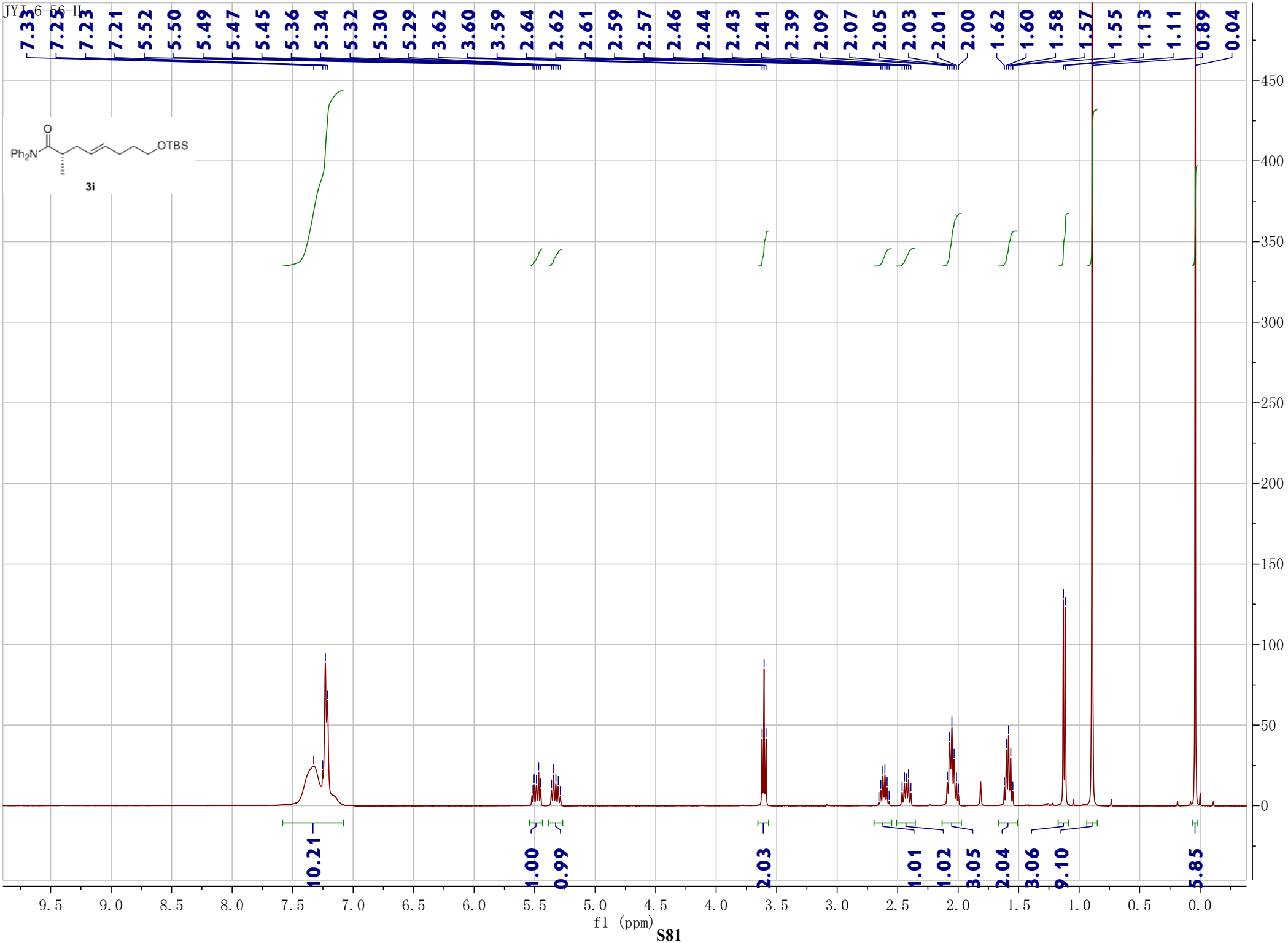


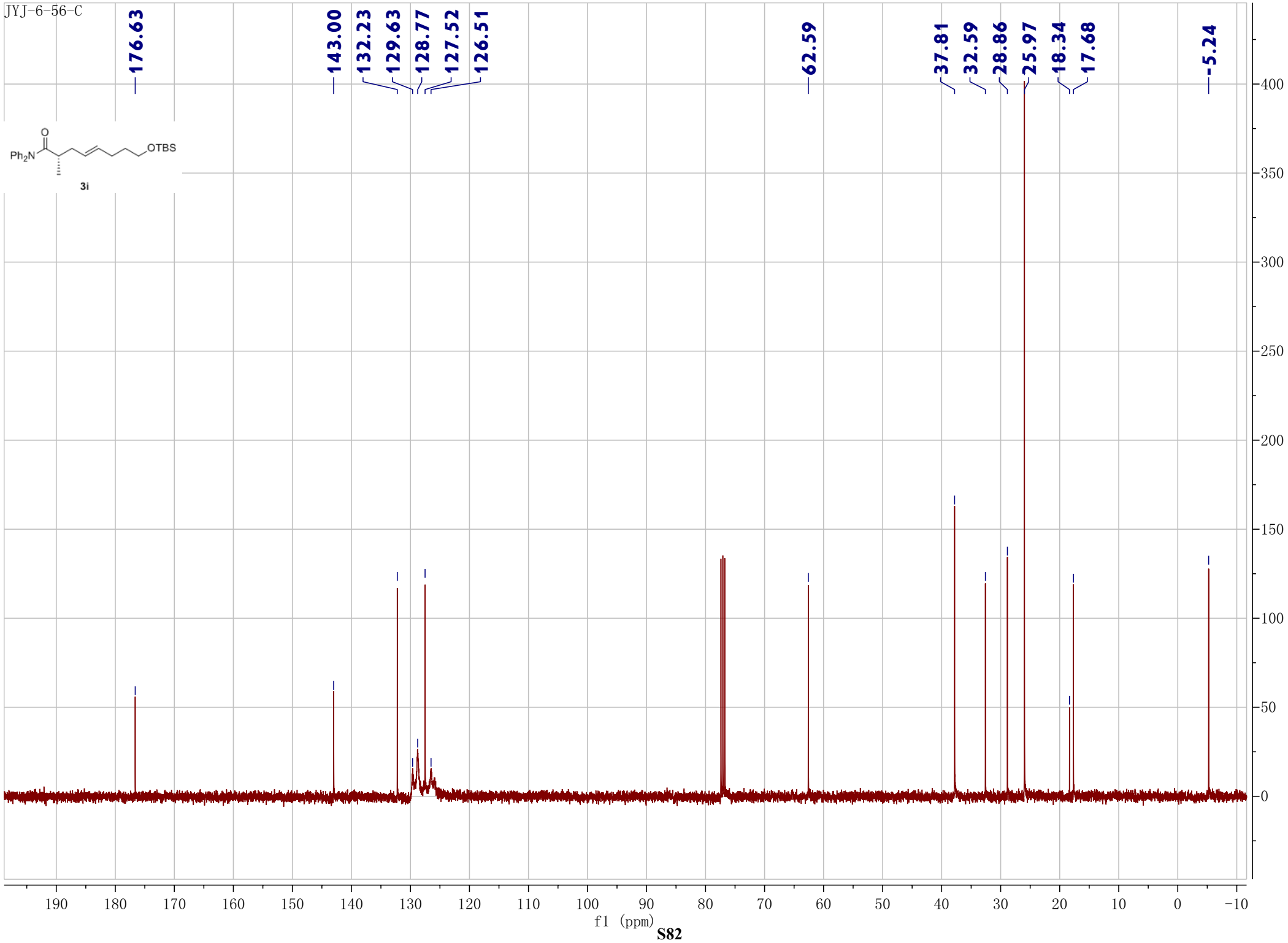
1 Det.A Ch1/214nm

PeakTable

Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.531	19196460	602139	95.964	95.902
2	19.261	807257	25728	4.036	4.098
Total		20003716	627867	100.000	100.000

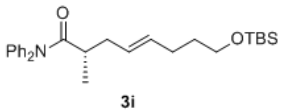

JYL-6-56-H
7.33 7.25 7.23 7.21 5.52 5.50 5.49 5.47 5.45 5.36 5.34 5.32 5.30 5.29 3.62 3.60 3.59 2.64 2.62 2.61 2.59 2.57 2.46 2.44 2.43 2.41 2.39 2.09 2.07 2.05 2.03 2.01 2.00 1.62 1.60 1.58 1.57 1.55 1.13 1.11 0.89 0.04
Ph2N
OTBS
3i
10.21 1.00 0.99 2.03 1.01 1.02 3.05 2.04 3.06 9.10 5.85
f1 (ppm)


JYJ-6-56-C
176.63
143.00
132.23
129.63
128.77
127.52
126.51
62.59
37.81
32.59
28.86
25.97
18.34
17.68
-5.24
Ph2N
O
OTBS
3i
f1 (ppm)

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\6\JYJ-6-69-RAC.lcd

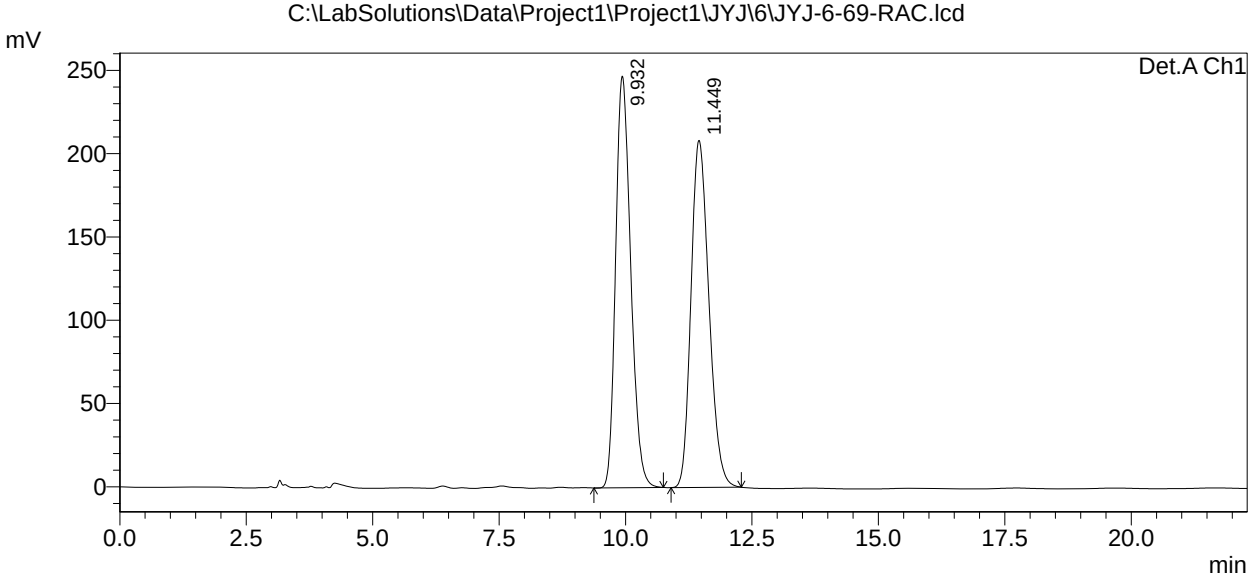
Acquired by	: Admin
Sample Name	: JYJ-6-69-RAC
Sample ID	: OD-H,99/1,1,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: JYJ-6-69-RAC.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2016-12-30 9:42:02
Data Processed	: 2016-12-30 10:05:05



3i

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\6\JYJ-6-69-RAC.lcd



1 Det.A Ch1/214nm

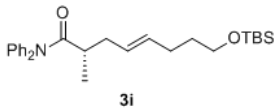
PeakTable

Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.932	5134543	247163	49.991	54.248
2	11.449	5136435	208457	50.009	45.752
Total		10270978	455621	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\6\JYJ-6-69.lcd

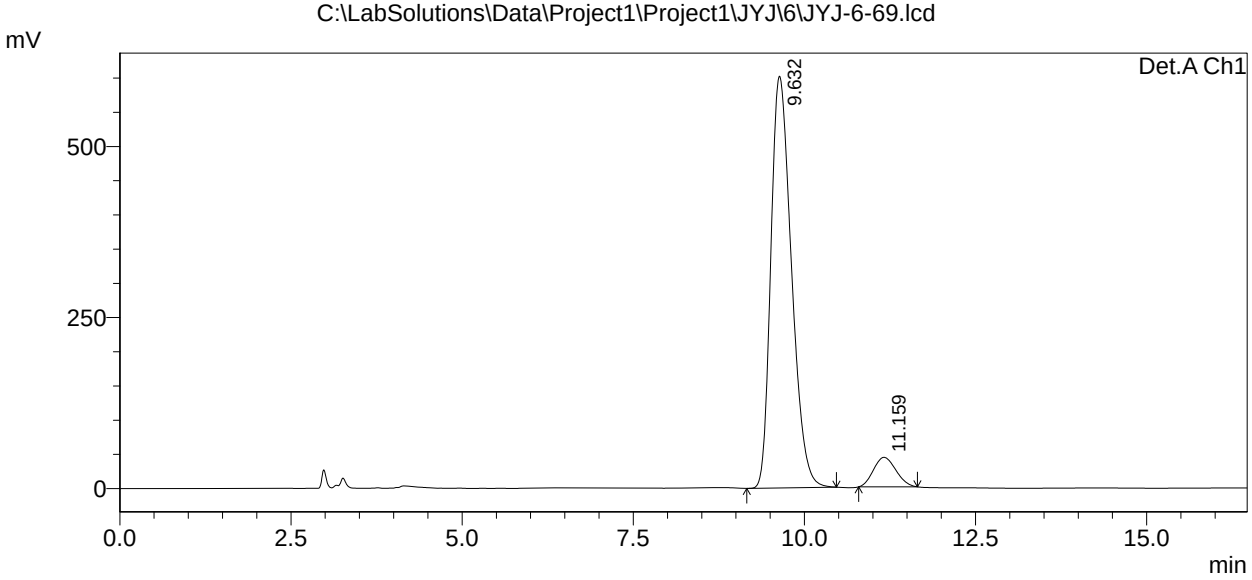


3i

Acquired by	: Admin
Sample Name	: JYJ-6-69-1
Sample ID	: OD-H,99/1,1,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: JYJ-6-69.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2016-12-30 10:07:18
Data Processed	: 2016-12-30 10:24:45

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\6\JYJ-6-69.lcd

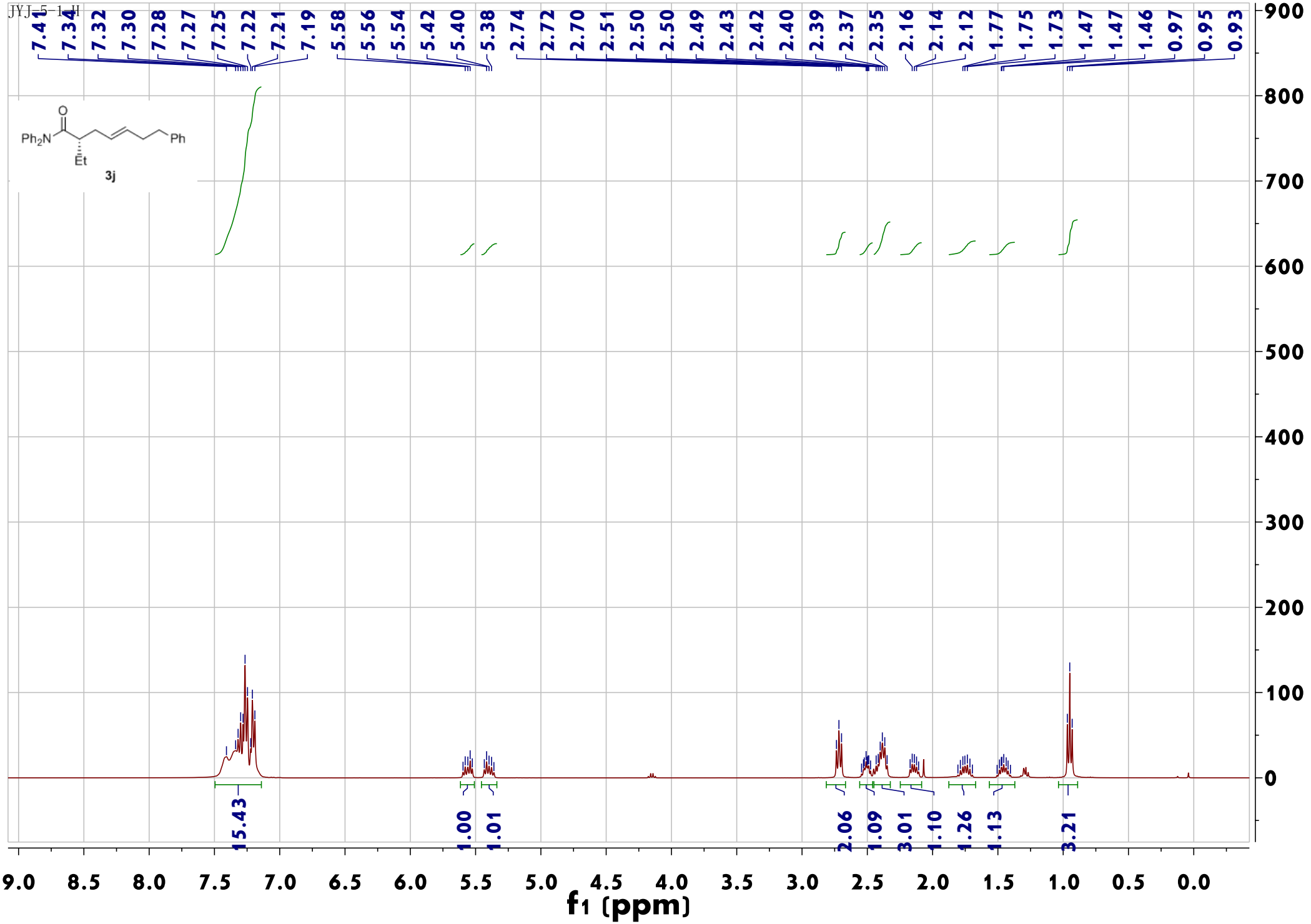


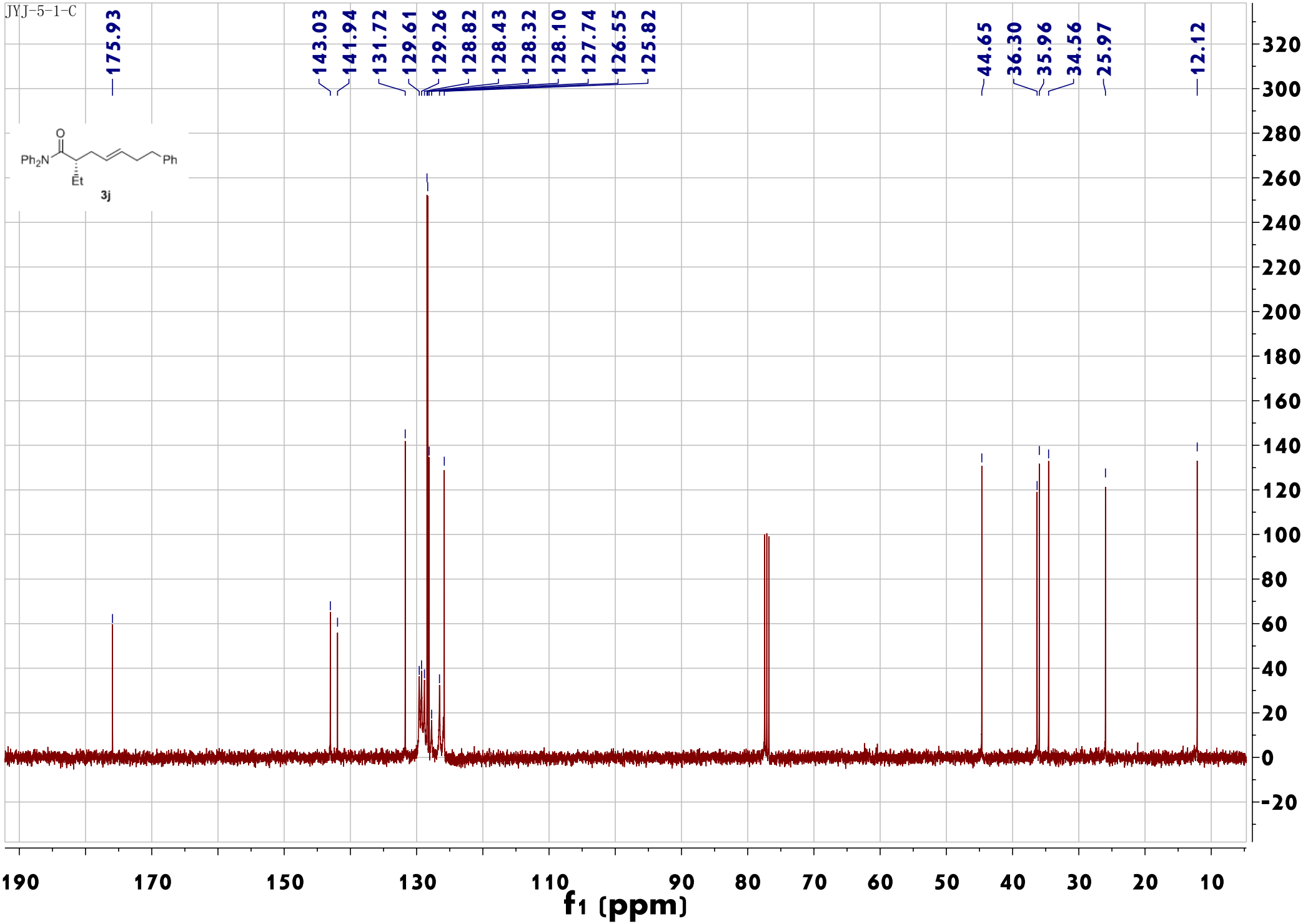
1 Det.A Ch1/214nm

PeakTable

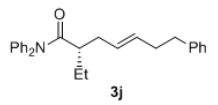
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.632	12604649	601958	92.758	93.317
2	11.159	984139	43109	7.242	6.683
Total		13588787	645067	100.000	100.000


JYJ-5-1-H
7.41
7.34
7.32
7.30
7.28
7.27
7.25
7.22
7.21
7.19
5.58
5.56
5.54
5.42
5.40
5.38
2.74
2.72
2.70
2.51
2.50
2.50
2.49
2.43
2.42
2.40
2.39
2.37
2.35
2.16
2.14
2.12
1.77
1.75
1.73
1.47
1.47
1.46
0.97
0.95
0.93
Ph2N
O
Et
Ph
3j
15.43
1.00
1.01
2.06
1.09
3.01
1.10
1.26
1.13
3.21
f1 (ppm)

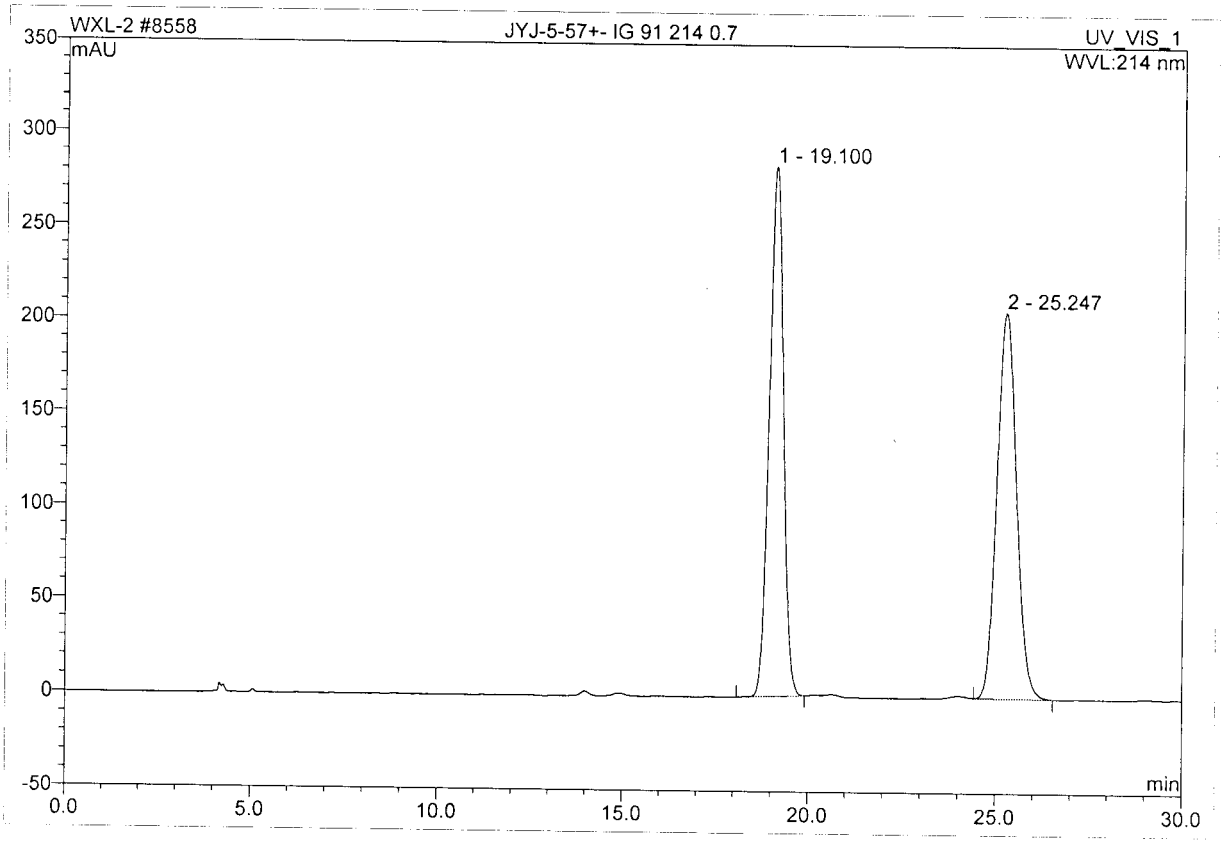

JYJ-5-1-C
175.93
143.03
141.94
131.72
129.61
129.26
128.82
128.43
128.32
128.10
127.74
126.55
125.82
44.65
36.30
35.96
34.56
25.97
12.12
Ph2N
O
Et
Ph
3j
320
300
280
260
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
190
170
150
130
110
90
80
70
60
50
40
30
20
10
f1 (ppm)

8558 JYJ-5-57+- IG 91 214 0.7



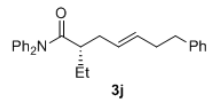
3j

Sample Name:	**JYJ-5-57+- IG 91 214 0.7**	*Injection Volume:*	**3.0**
Vial Number:	**RC5**	*Channel:*	**UV_VIS_1**
Sample Type:	**unknown**	*Wavelength:*	**214**
Control Program:	**WXL-2014**	*Bandwidth:*	**n.a.**
Quantif. Method:	**WXL**	*Dilution Factor:*	**1.0000**
Recording Time:	**2016/9/21 14:42**	*Sample Weight:*	**1.0000**
Run Time (min):	**30.00**	*Sample Amount:*	**1.0000**



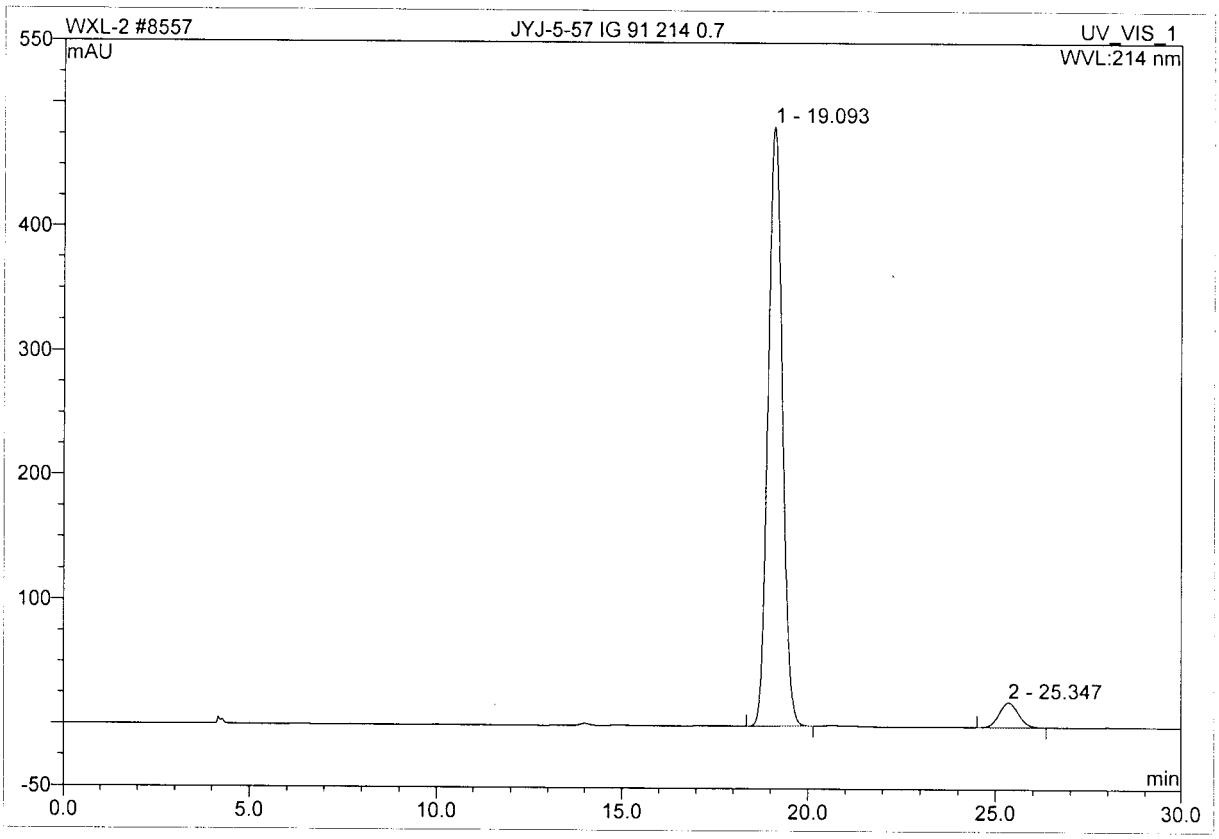
No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	19.10	n.a.	282.777	123.700	49.80	n.a.	BMB
2	25.25	n.a.	206.131	124.710	50.20	n.a.	BMB
Total:			488.908	248.410	100.00	0.000	

8557 JYJ-5-57 IG 91 214 0.7

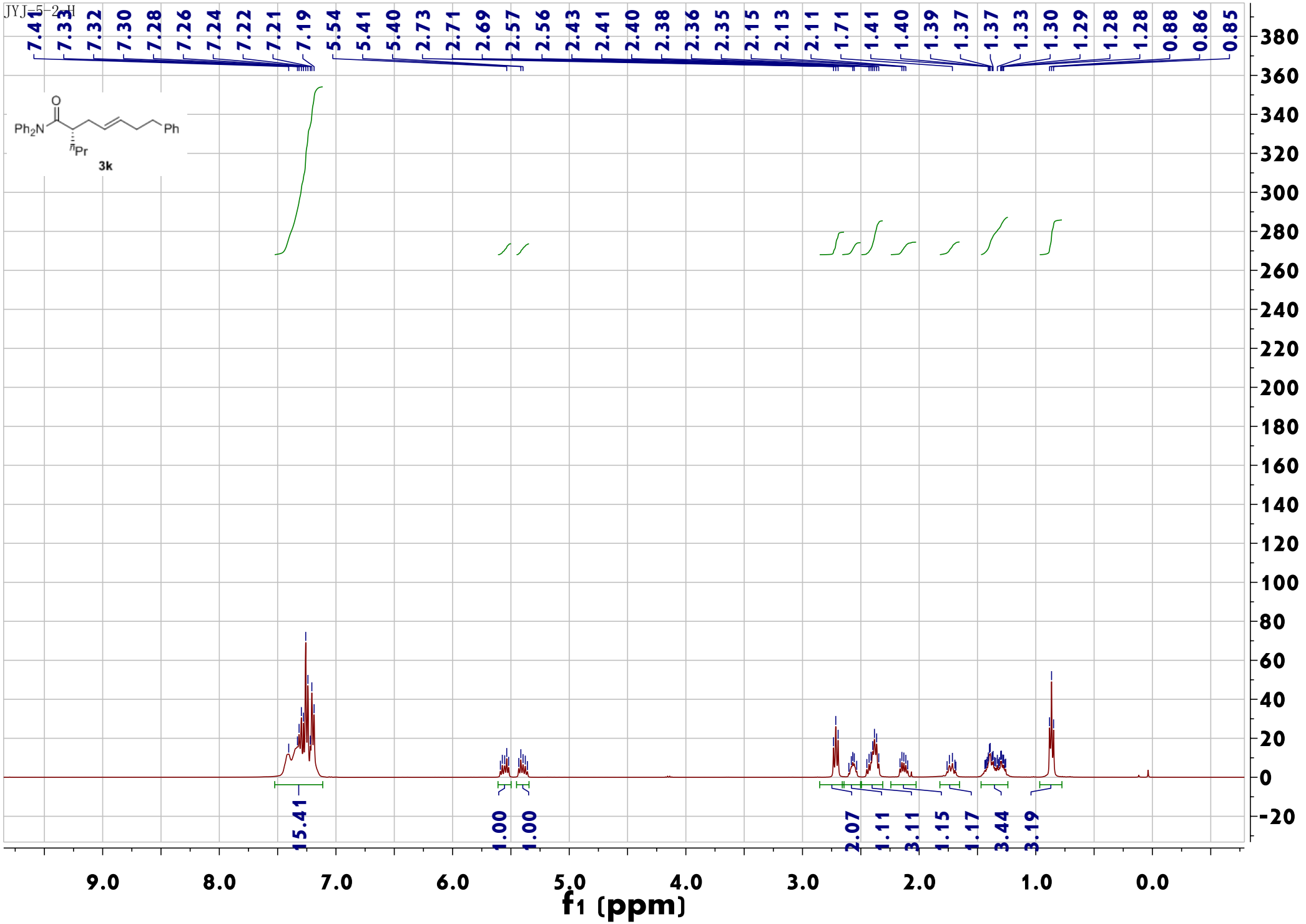


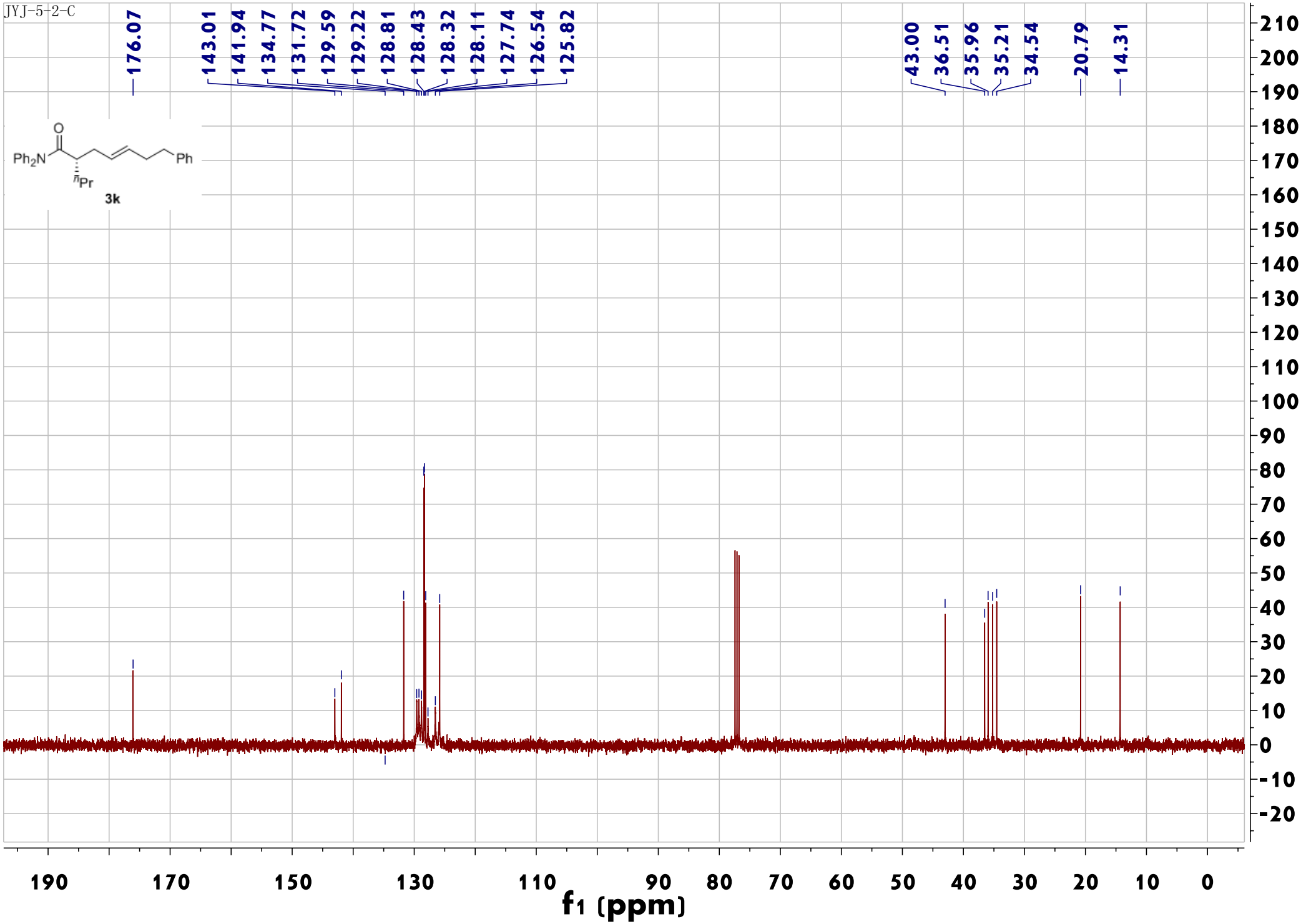
3j

Sample Name:	**JYJ-5-57 IG 91 214 0.7**	*Injection Volume:*	**3.0**
Vial Number:	**RD7**	*Channel:*	**UV_VIS_1**
Sample Type:	**unknown**	*Wavelength:*	**214**
Control Program:	**WXL-2014**	*Bandwidth:*	**n.a.**
Quantif. Method:	**WXL**	*Dilution Factor:*	**1.0000**
Recording Time:	**2016/9/21 14:10**	*Sample Weight:*	**1.0000**
Run Time (min):	**29.96**	*Sample Amount:*	**1.0000**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	19.09	n.a.	482.582	214.439	94.71	n.a.	BMB
2	25.35	n.a.	20.162	11.975	5.29	n.a.	BMB
Total:			502.744	226.414	100.00	0.000	

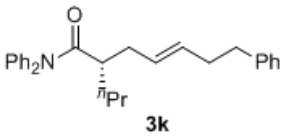

JYJ-5-2-H
7.41 7.33 7.32 7.30 7.28 7.26 7.24 7.22 7.21 7.19 5.54 5.41 5.40 2.73 2.71 2.69 2.57 2.56 2.43 2.41 2.40 2.38 2.36 2.35 2.15 2.13 2.11 1.71 1.41 1.40 1.39 1.37 1.37 1.33 1.30 1.29 1.28 1.28 0.88 0.86 0.85
Ph2N
Ph
nPr
3k
15.41
1.00
1.00
2.07
1.11
3.11
1.15
1.17
3.44
3.19
f1 (ppm)


JYJ-5-2-C
176.07
143.01
141.94
134.77
131.72
129.59
129.22
128.81
128.43
128.32
128.11
127.74
126.54
125.82
43.00
36.51
35.96
35.21
34.54
20.79
14.31
Ph2N
O
nPr
Ph
3k
f1 (ppm)

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\5\JYJ-5-33-RAC-2.lcd

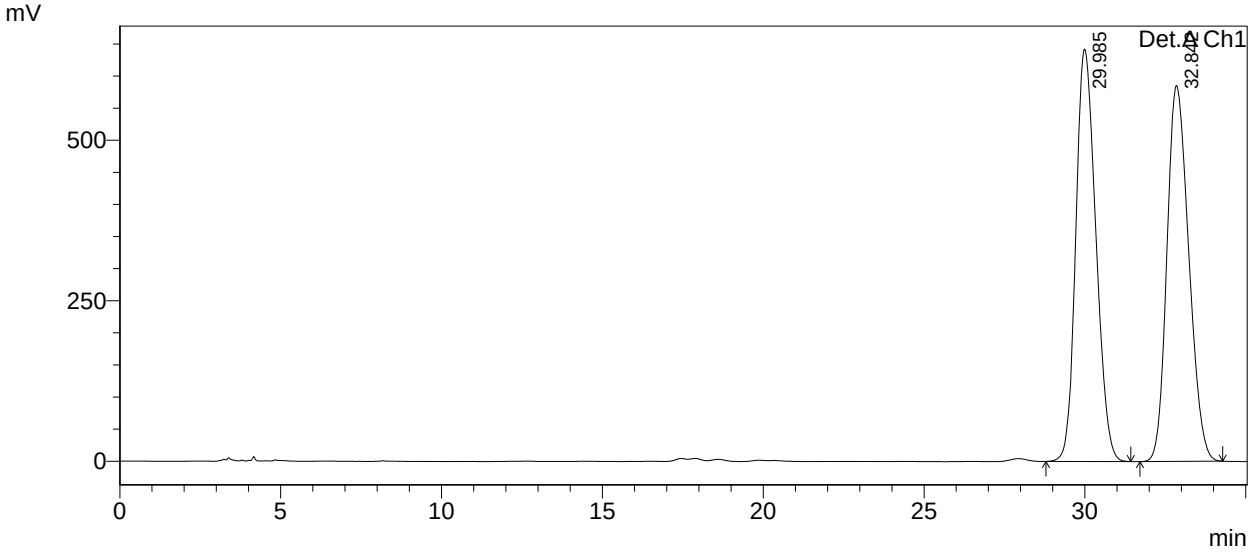
Acquired by	: Admin
Sample Name	: JYJ-5-33-RAC-2
Sample ID	: IC,99/1,1,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: JYJ-5-33-RAC-2.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2016-9-7 17:34:29
Data Processed	: 2016-9-7 18:10:16



3k

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\5\JYJ-5-33-RAC-2.lcd



1 Det.A Ch1/214nm

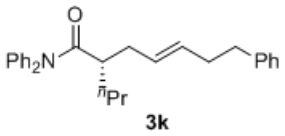
PeakTable

Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	29.985	27837361	642449	50.148	52.312
2	32.842	27673266	585658	49.852	47.688
Total		55510626	1228107	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\5\JYJ-5-33-1.lcd

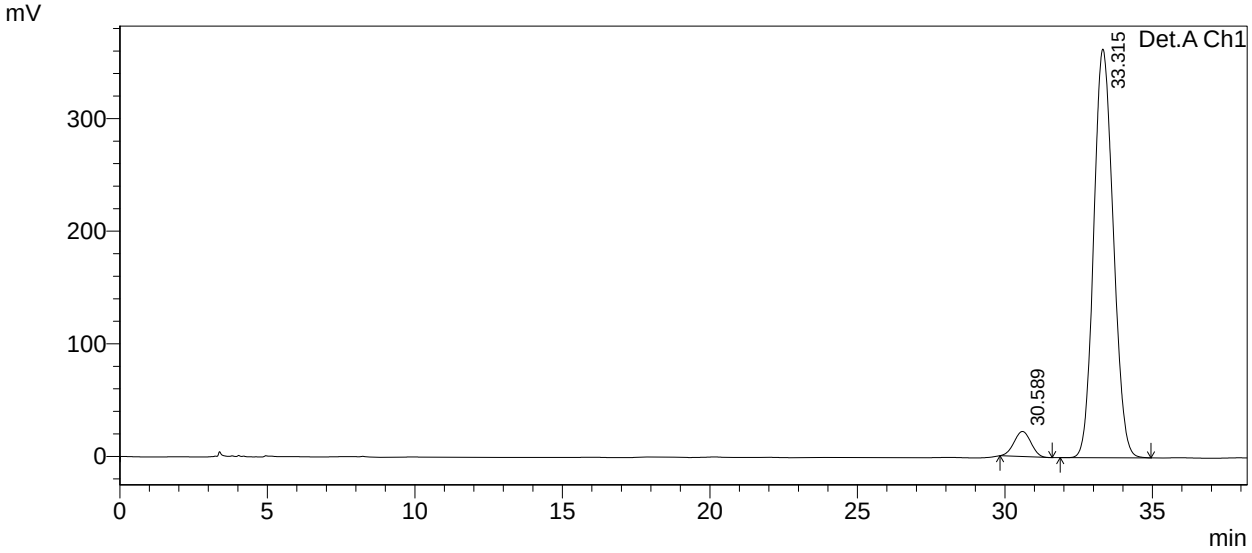


3k

Acquired by	: Admin
Sample Name	: JYJ-5-33-1
Sample ID	: IC,99/1,1,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: JYJ-5-33-1.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2016-9-7 16:53:01
Data Processed	: 2016-9-7 17:32:40

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\5\JYJ-5-33-1.lcd

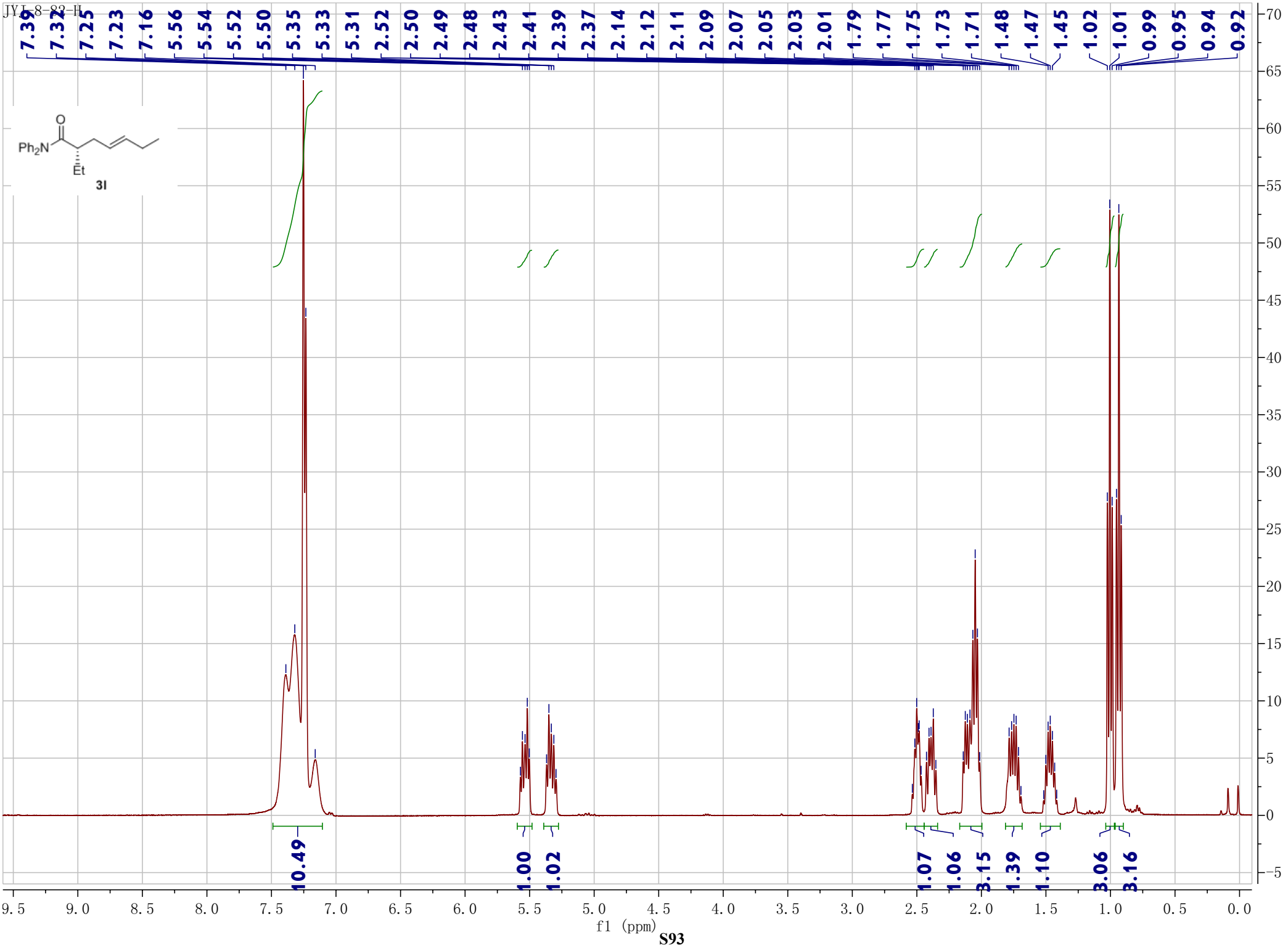


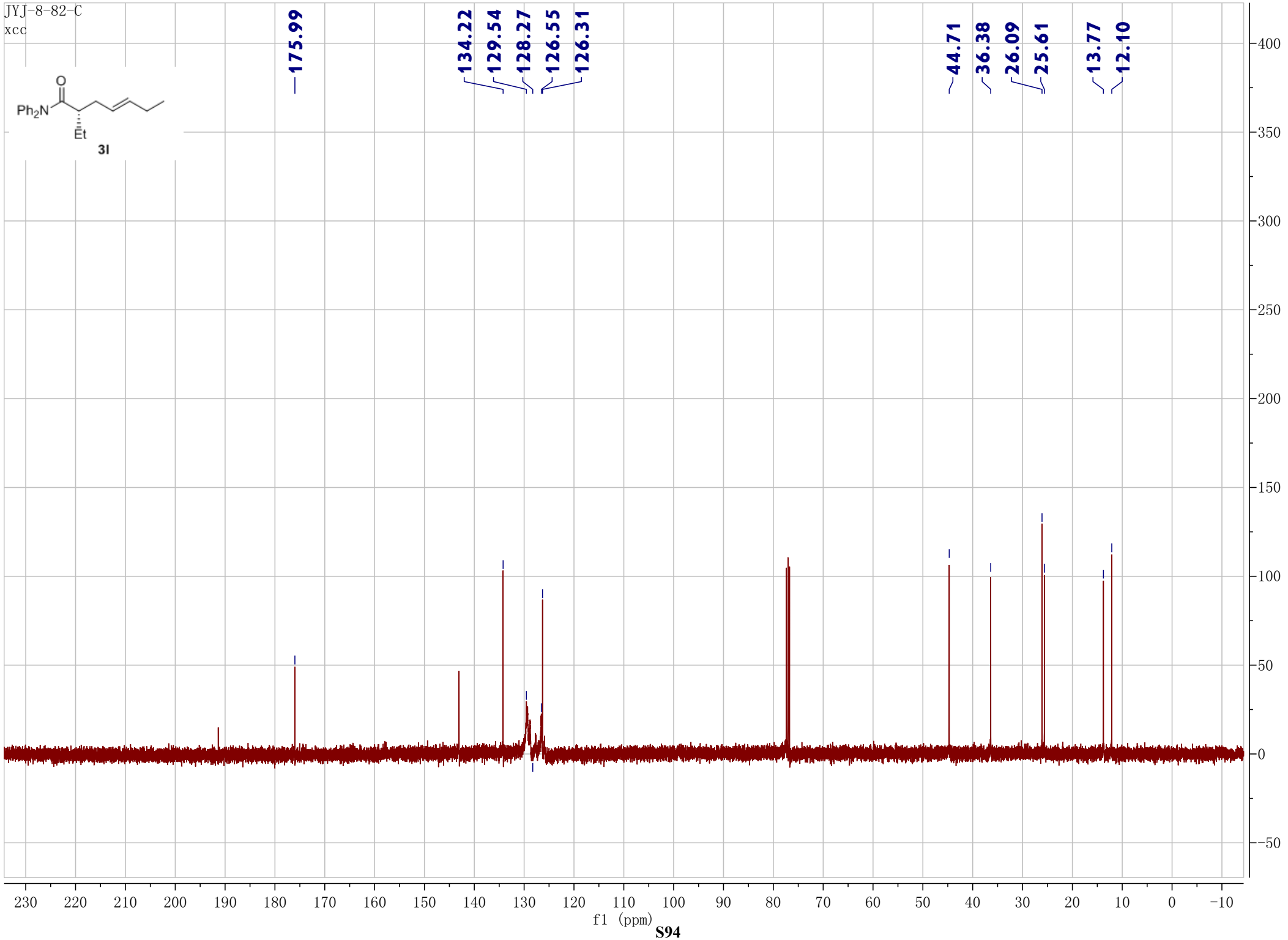
1 Det.A Ch1/214nm

PeakTable

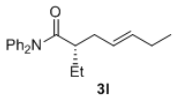
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	30.589	885298	22249	5.042	5.777
2	33.315	16671612	362855	94.958	94.223
Total		17556910	385103	100.000	100.000


JYL-8-82-H
7.39
7.32
7.25
7.23
7.16
5.56
5.54
5.52
5.50
5.35
5.33
5.31
2.52
2.50
2.49
2.48
2.43
2.41
2.39
2.37
2.14
2.12
2.11
2.09
2.07
2.05
2.03
2.01
1.79
1.77
1.75
1.73
1.71
1.48
1.47
1.45
1.02
1.01
0.99
0.95
0.94
0.92
Ph2N
Et
3l
10.49
1.00
1.02
1.07
1.06
3.15
1.39
1.10
3.06
3.16
f1 (ppm)

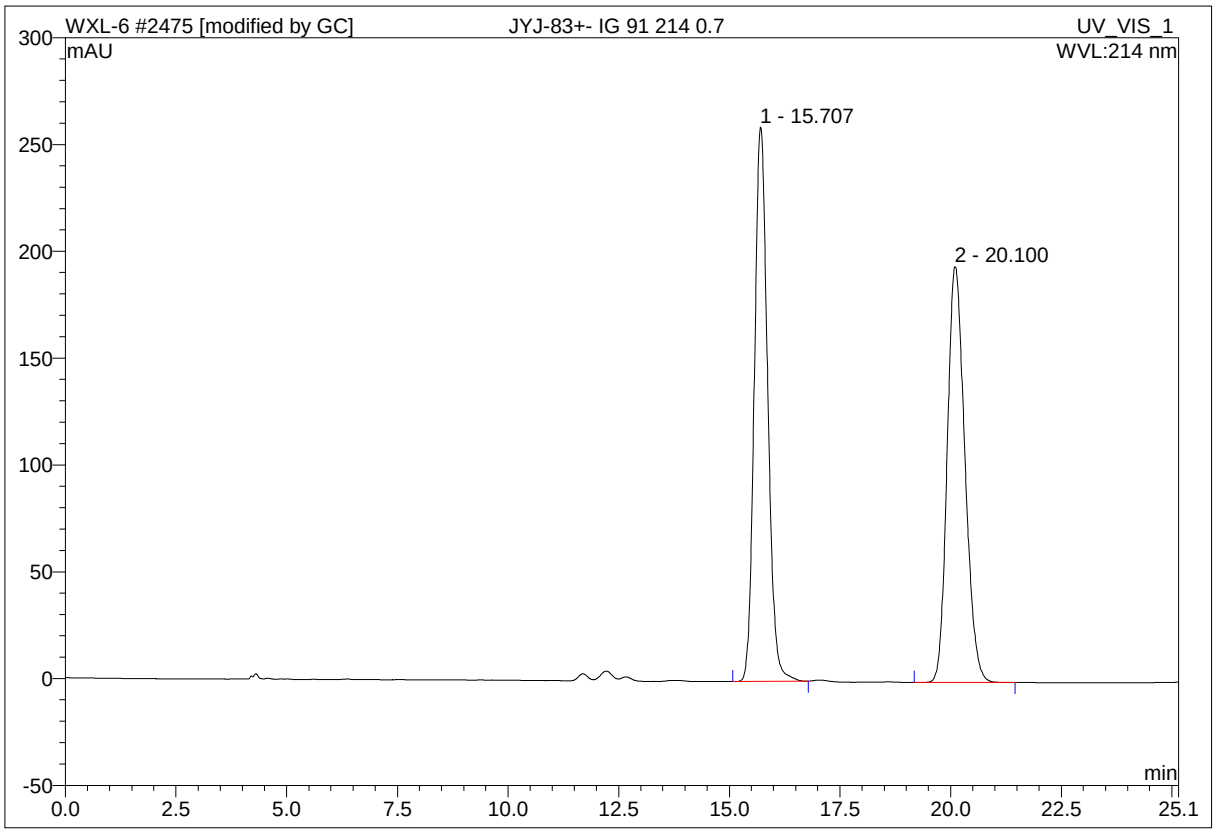

JYJ-8-82-C
xcc
O
Ph2N
Et
3l
175.99
134.22
129.54
128.27
126.55
126.31
44.71
36.38
26.09
25.61
13.77
12.10
400
350
300
250
200
150
100
50
0
−50
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

2475 JYJ-83+- IG 91 214 0.7



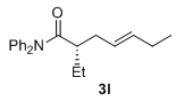
3l

Sample Name:	**JYJ-83+- IG 91 214 0.7**	*Injection Volume:*	**2.0**
Vial Number:	**RC1**	*Channel:*	**UV_VIS_1**
Sample Type:	**unknown**	*Wavelength:*	**214**
Control Program:	**201701-4**	*Bandwidth:*	**n.a.**
Quantif. Method:	**201701**	*Dilution Factor:*	**1.0000**
Recording Time:	**2017/7/14 11:04**	*Sample Weight:*	**1.0000**
Run Time (min):	**25.15**	*Sample Amount:*	**1.0000**



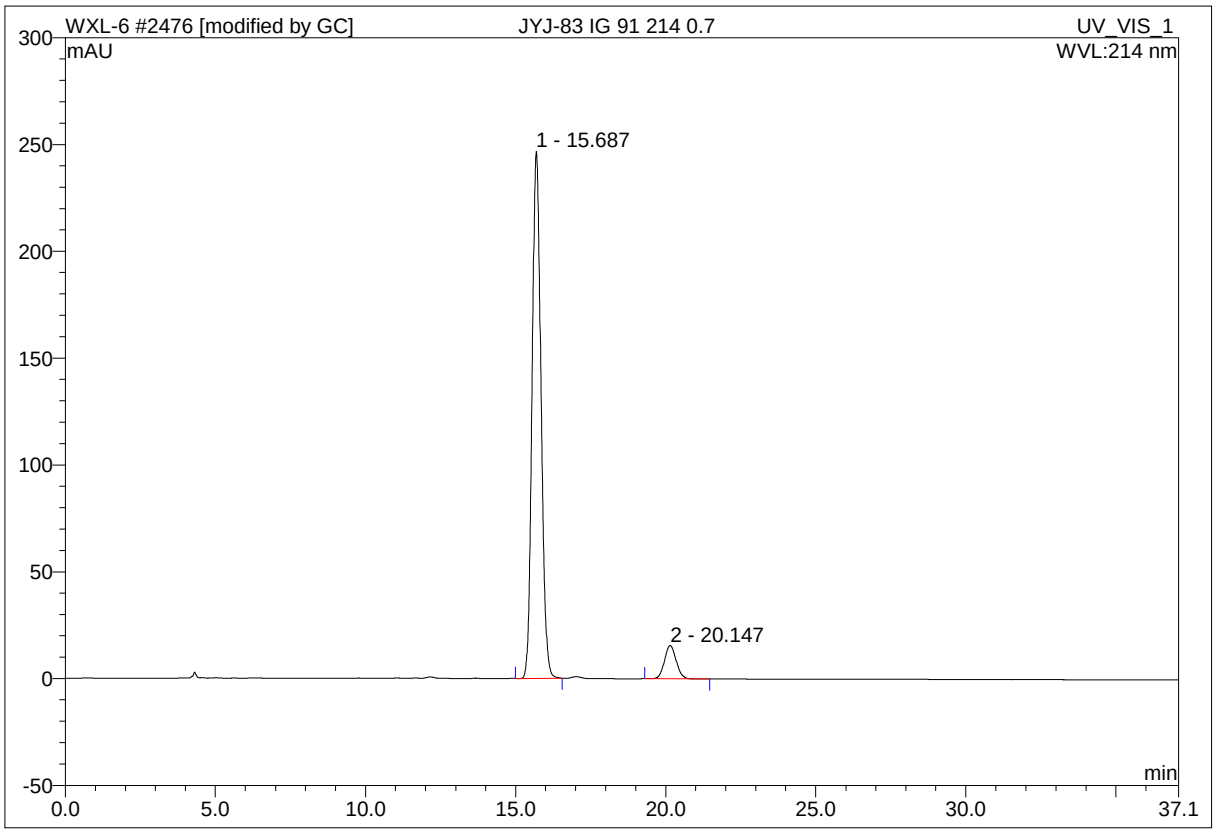
No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	15.71	n.a.	259.490	90.611	50.15	n.a.	BMB*
2	20.10	n.a.	194.522	90.082	49.85	n.a.	BMB*
Total:			454.011	180.693	100.00	0.000	

2476 JYJ-83 IG 91 214 0.7

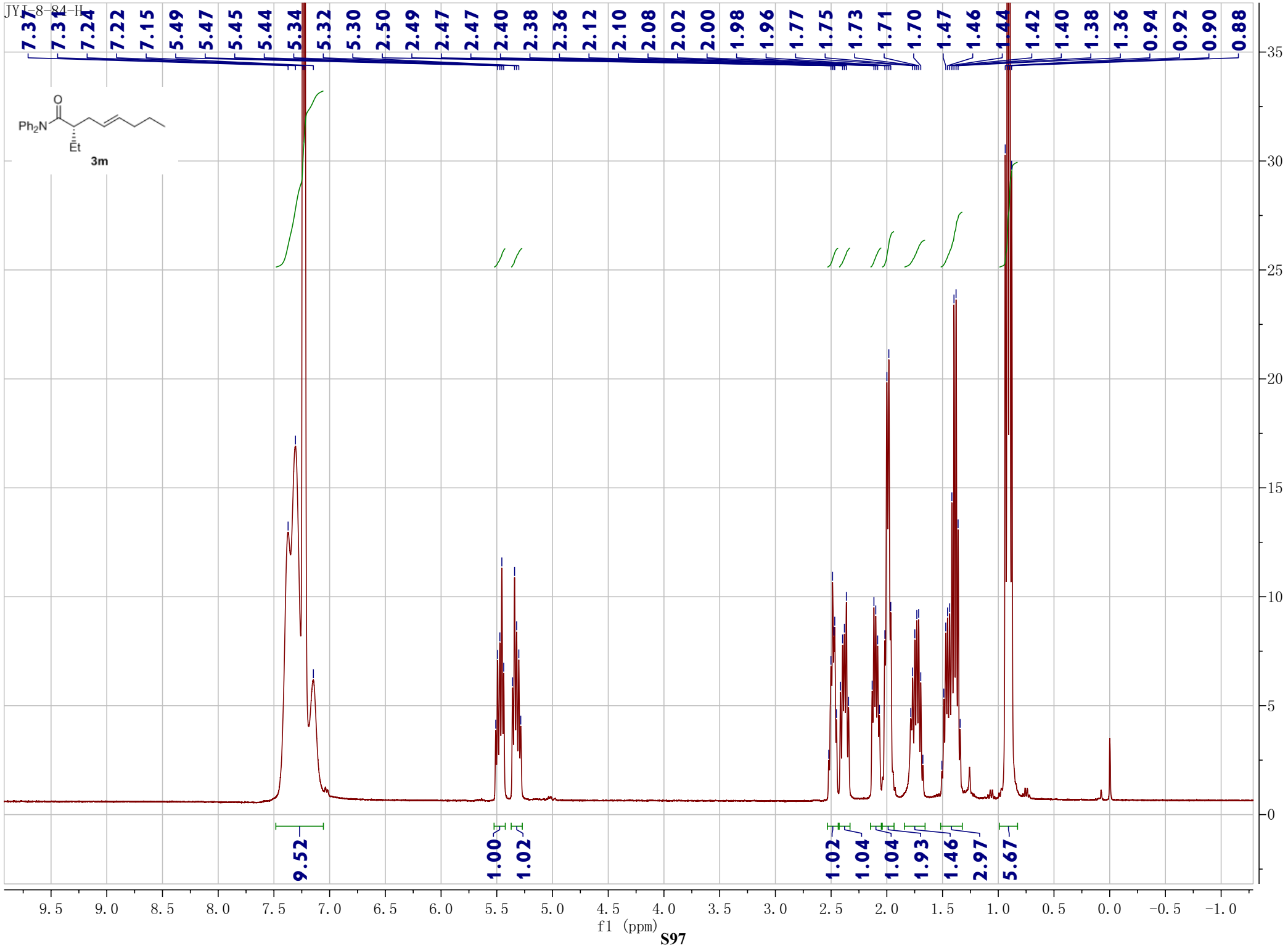


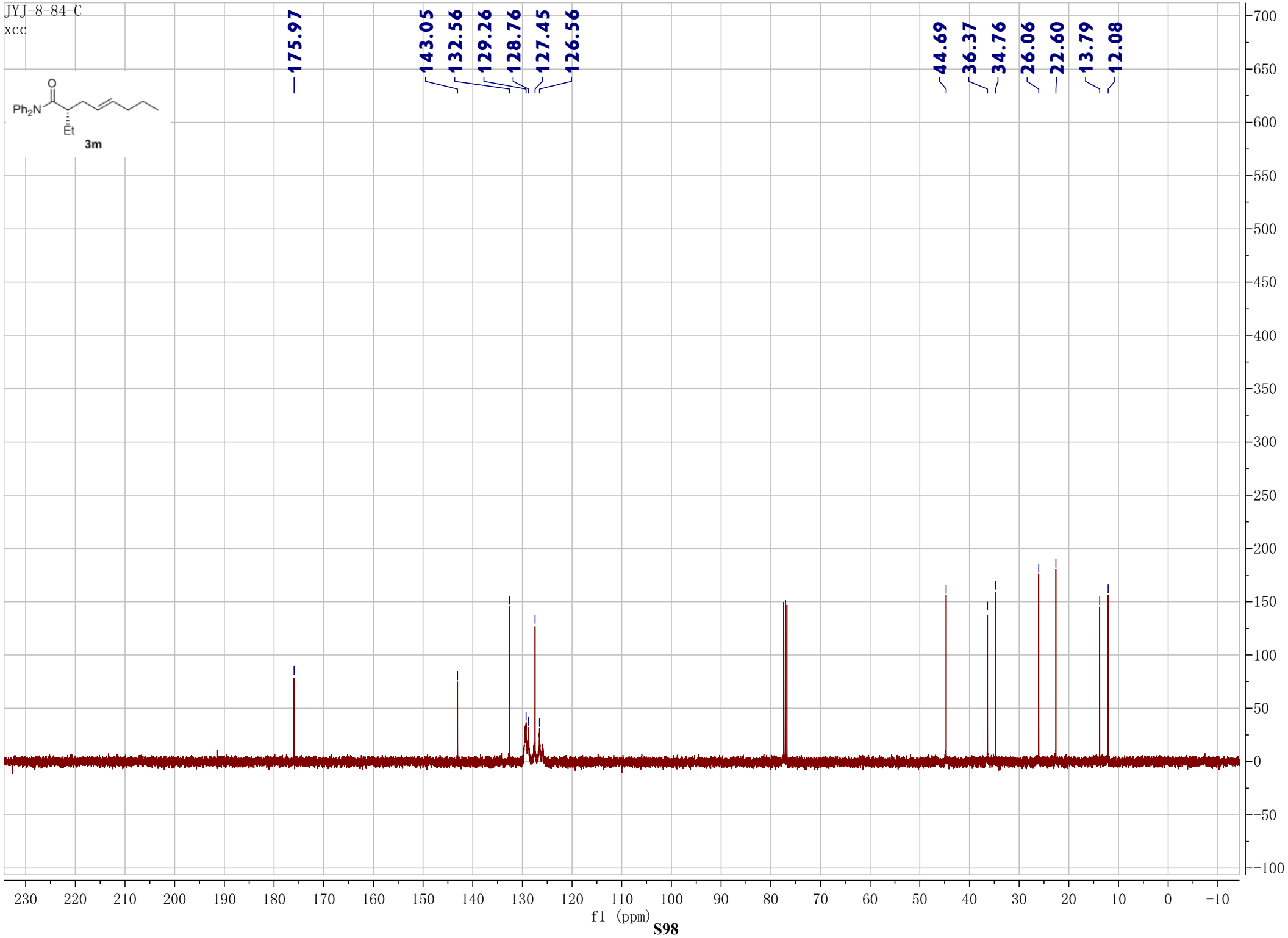
3l

Sample Name:	**JYJ-83 IG 91 214 0.7**	*Injection Volume:*	**2.0**
Vial Number:	**RC2**	*Channel:*	**UV_VIS_1**
Sample Type:	**unknown**	*Wavelength:*	**214**
Control Program:	**201701-4**	*Bandwidth:*	**n.a.**
Quantif. Method:	**201701**	*Dilution Factor:*	**1.0000**
Recording Time:	**2017/7/14 11:32**	*Sample Weight:*	**1.0000**
Run Time (min):	**37.08**	*Sample Amount:*	**1.0000**



No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %	Amount	Type
1	15.69	n.a.	246.675	85.171	92.41	n.a.	BMB*
2	20.15	n.a.	15.478	6.993	7.59	n.a.	BMB*
Total:			262.153	92.164	100.00	0.000	

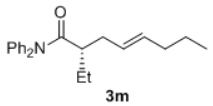

JYL-8-84-H
7.37
7.31
7.24
7.22
7.15
5.49
5.47
5.45
5.44
5.34
5.32
5.30
2.50
2.49
2.47
2.47
2.40
2.38
2.36
2.12
2.10
2.08
2.02
2.00
1.98
1.96
1.77
1.75
1.73
1.71
1.70
1.47
1.46
1.44
1.42
1.40
1.38
1.36
0.94
0.92
0.90
0.88
Ph2N
Et
3m
9.52
1.00
1.02
1.02
1.04
1.04
1.93
1.46
2.97
5.67
f1 (ppm)


JYJ-8-84-C
xcc
3m
175.97
143.05
132.56
129.26
128.76
127.45
126.56
44.69
36.37
34.76
26.06
22.60
13.79
12.08
f1 (ppm)

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\8\jyj-8-84-RAC-IE.lcd

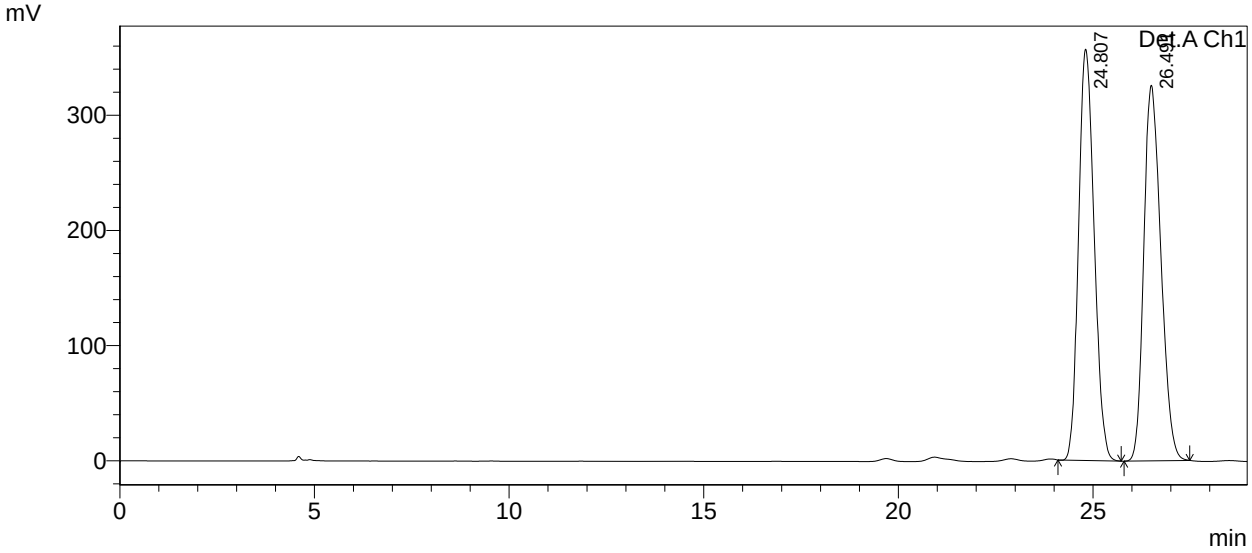
Acquired by	: Admin
Sample Name	: jyj-8-84-RAC-IE
Sample ID	: IE,95/5,0.7,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: jyj-8-84-RAC-IE.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2017-7-14 11:21:08
Data Processed	: 2017-7-14 12:20:30



3m

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\8\jyj-8-84-RAC-IE.lcd



1 Det.A Ch1/214nm

PeakTable

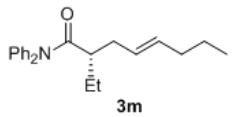
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	24.807	9947744	357162	49.903	52.276
2	26.491	9986598	326064	50.097	47.724
Total		19934342	683226	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\8\jyj-8-85-IE.lcd

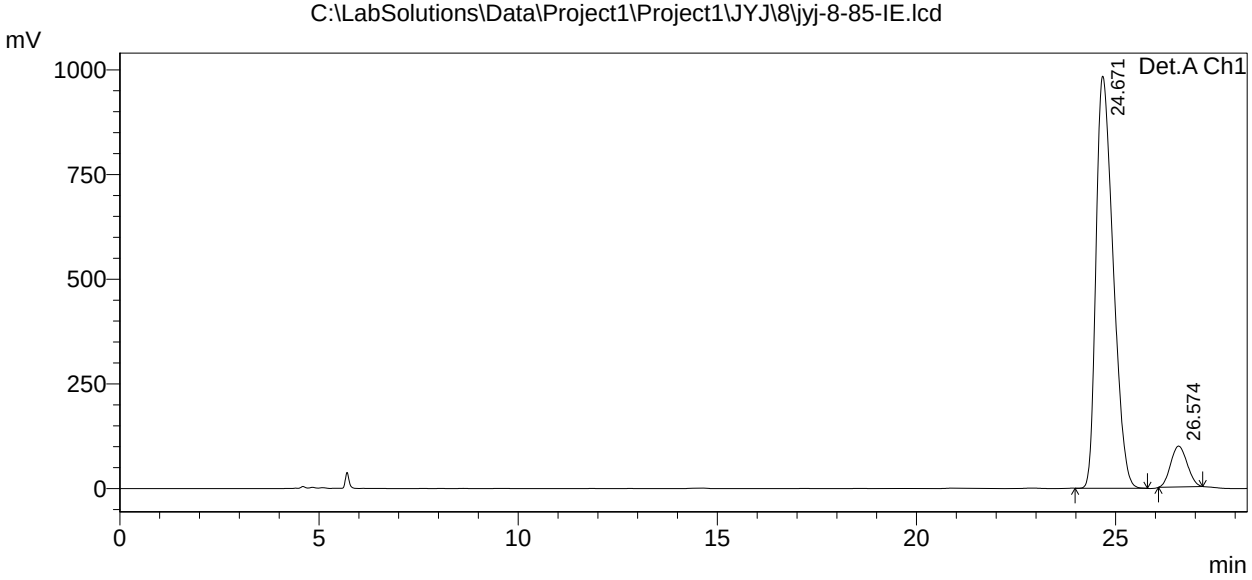
Acquired by	: Admin
Sample Name	: jyj-8-85-IE
Sample ID	: IE,95/5,0.7,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: jyj-8-85-IE.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2017-7-14 11:50:42
Data Processed	: 2017-7-14 13:14:20



3m

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\8\jyj-8-85-IE.lcd

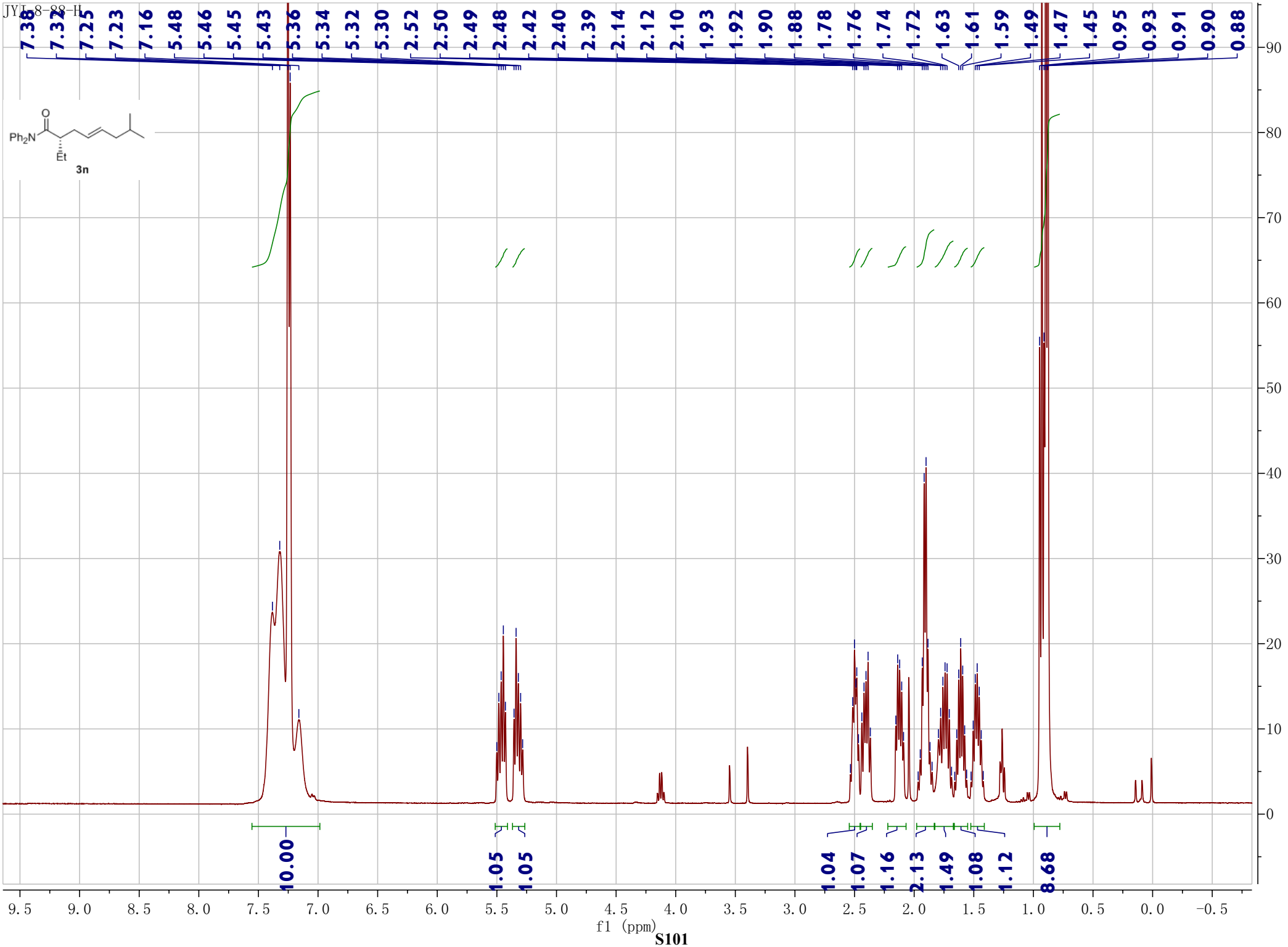


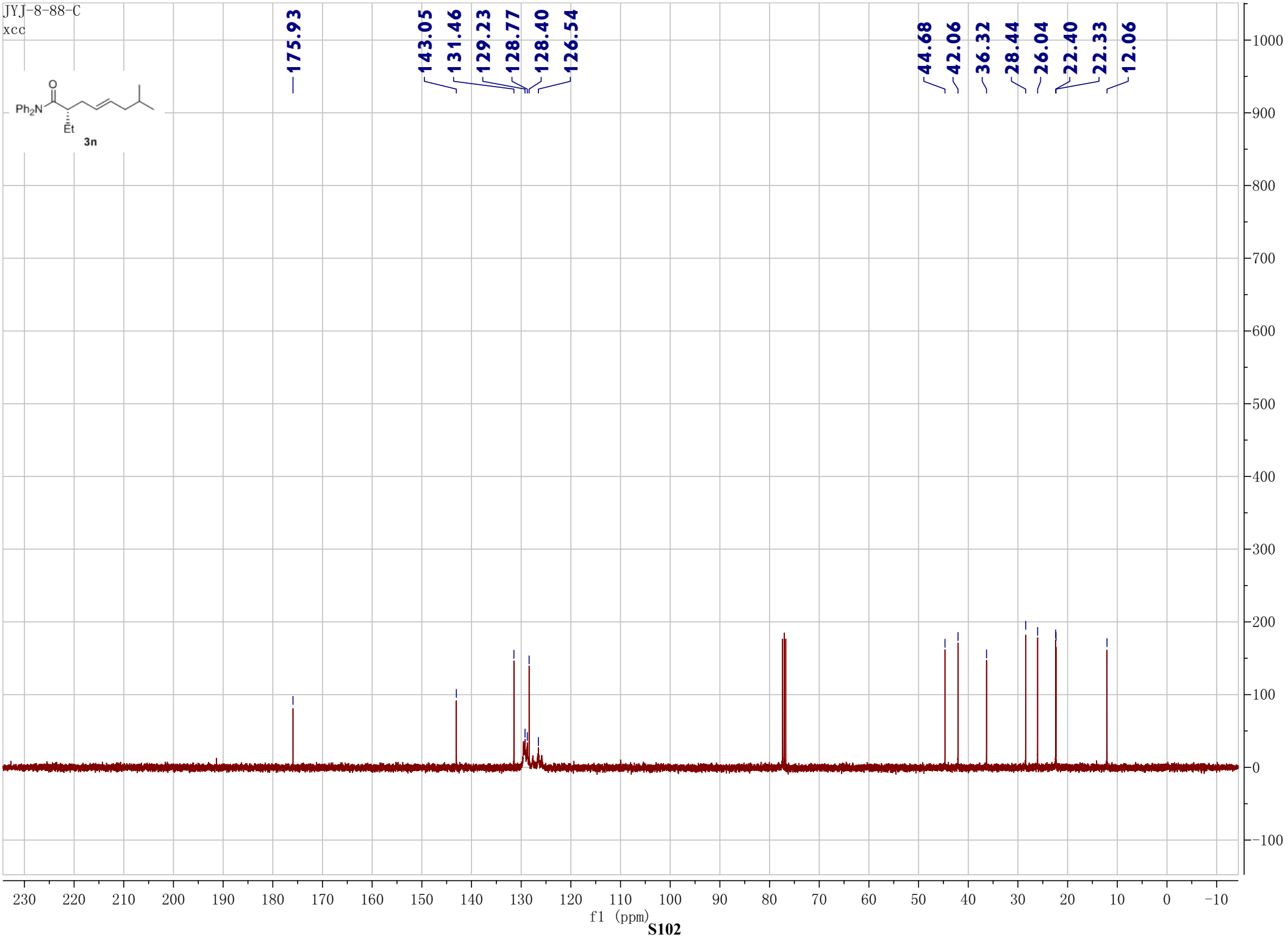
1 Det.A Ch1/214nm

PeakTable

Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	24.671	29416798	984227	91.269	90.974
2	26.574	2813971	97653	8.731	9.026
Total		32230769	1081880	100.000	100.000

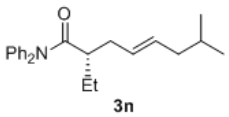

JYL-8-88-H
7.38
7.32
7.25
7.23
7.16
5.48
5.46
5.45
5.43
5.36
5.34
5.32
5.30
2.52
2.50
2.49
2.48
2.42
2.40
2.39
2.14
2.12
2.10
1.93
1.92
1.90
1.88
1.78
1.76
1.74
1.72
1.63
1.61
1.59
1.49
1.47
1.45
0.95
0.93
0.91
0.90
0.88
Ph2N
Et
3n
10.00
1.05
1.05
1.04
1.07
1.16
2.13
1.49
1.08
1.12
8.68
f1 (ppm)


JYJ-8-88-C
xcc
3n
175.93
143.05
131.46
129.23
128.77
128.40
126.54
44.68
42.06
36.32
28.44
26.04
22.40
22.33
12.06
f1 (ppm)

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\8\jyj-8-88-RAC-IE.lcd

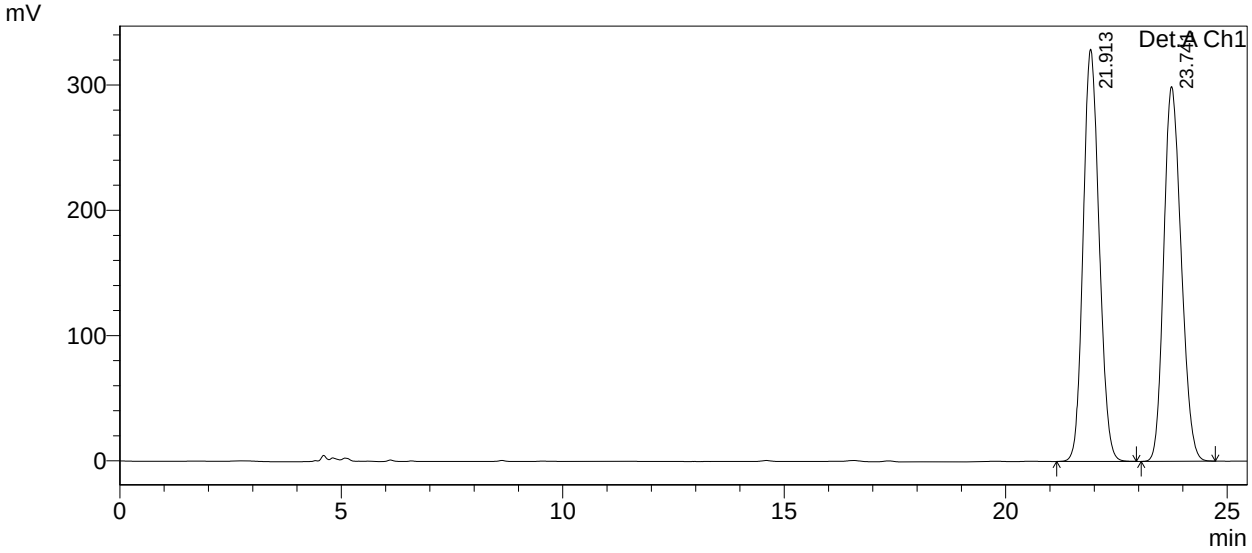
Acquired by	: Admin
Sample Name	: jyj-8-88-RAC-IE
Sample ID	: IE,95/5,0.7,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: jyj-8-88-RAC-IE.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2017-7-14 12:50:52
Data Processed	: 2017-7-14 13:49:40



3n

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\8\jyj-8-88-RAC-IE.lcd



1 Det.A Ch1/214nm

PeakTable

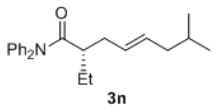
Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.913	8276698	329138	50.249	52.387
2	23.741	8194664	299145	49.751	47.613
Total		16471362	628284	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\8\jyj-8-89-IE.lcd

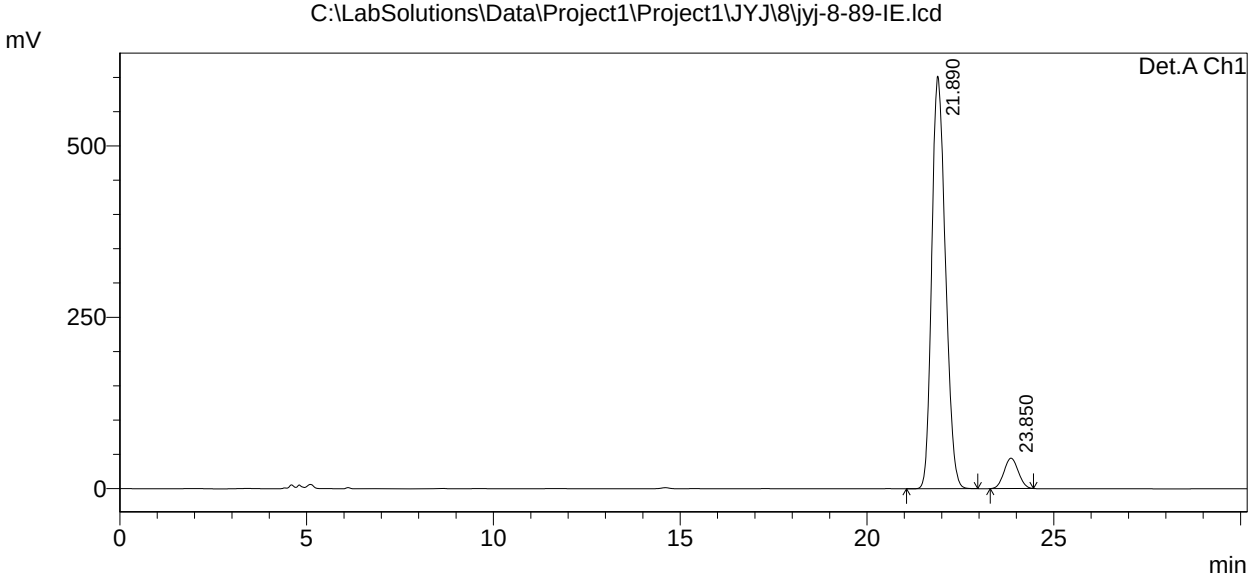
Acquired by	: Admin
Sample Name	: jyj-8-89-IE
Sample ID	: IE,95/5,0.7,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: jyj-8-89-IE.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2017-7-14 13:17:31
Data Processed	: 2017-7-14 13:48:40



3n

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\8\jyj-8-89-IE.lcd

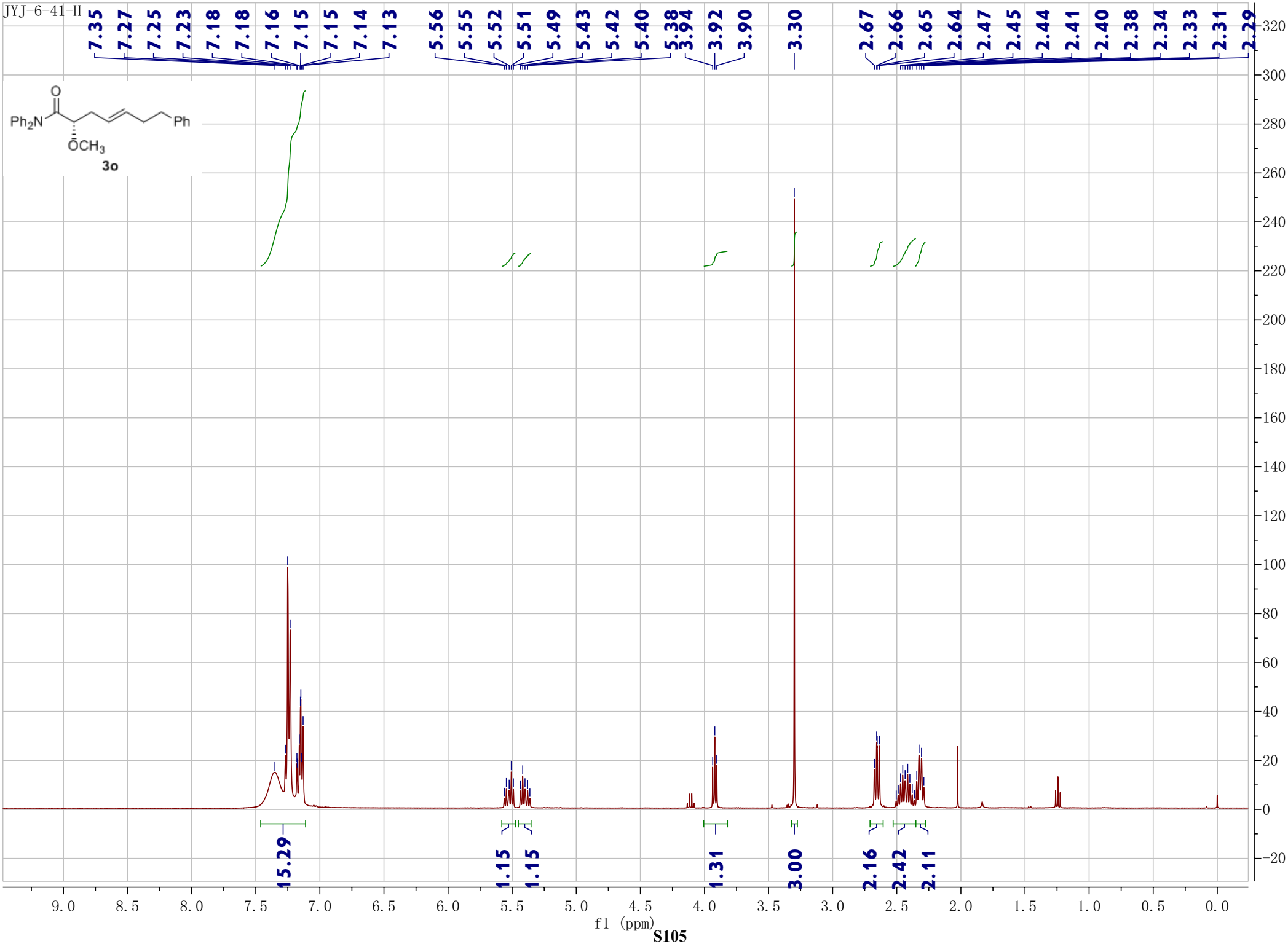


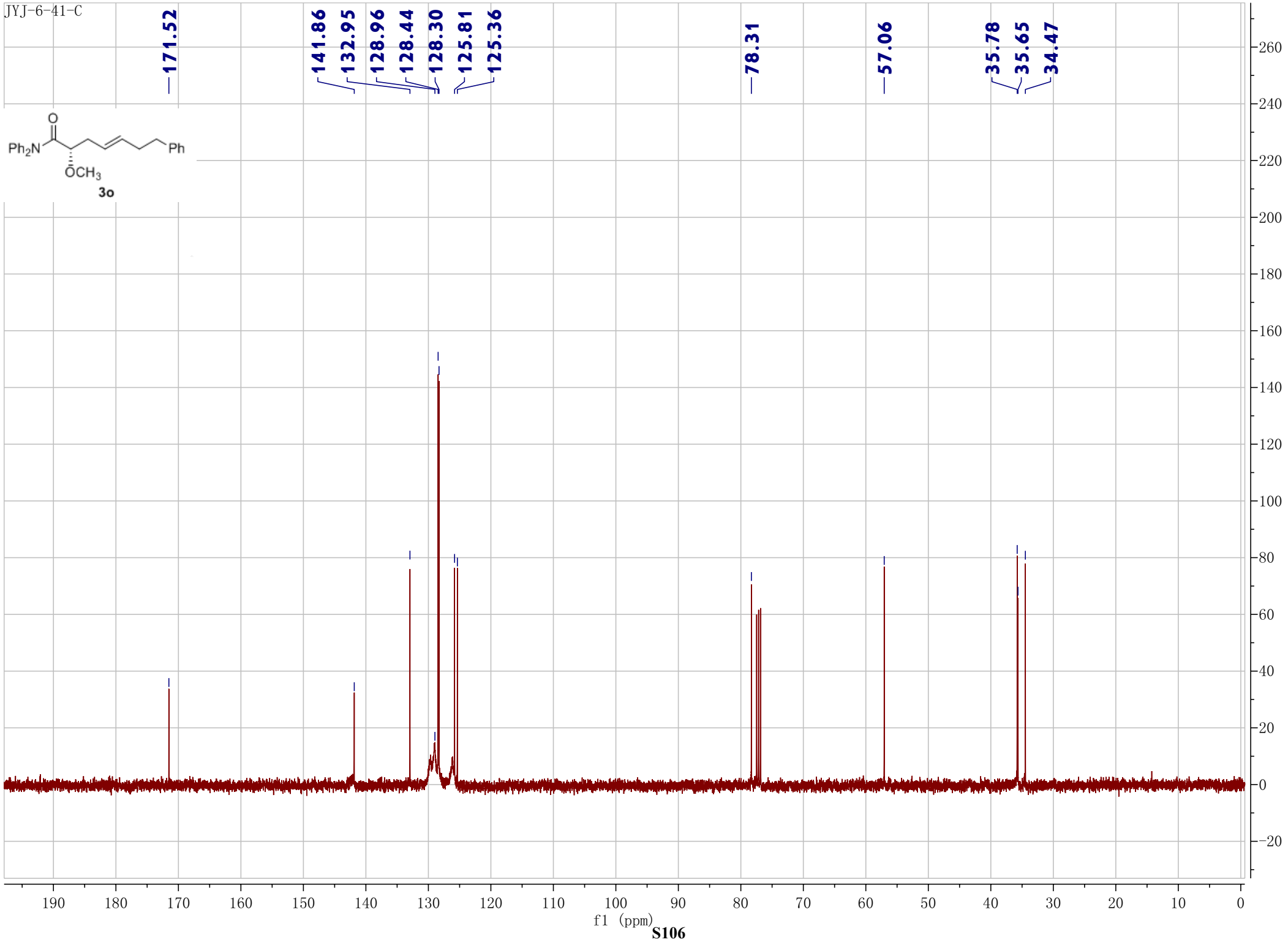
1 Det.A Ch1/214nm

PeakTable

Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.890	15393868	602031	92.876	93.141
2	23.850	1180796	44331	7.124	6.859
Total		16574664	646362	100.000	100.000

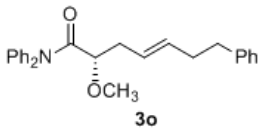

JYJ-6-41-H
7.35 7.27 7.25 7.23 7.18 7.18 7.16 7.15 7.15 7.14 7.13
5.56 5.55 5.52 5.51 5.49 5.43 5.42 5.40 5.38 3.94 3.92 3.90
3.30
2.67 2.66 2.65 2.64 2.47 2.45 2.44 2.41 2.40 2.38 2.34 2.33 2.31 2.29
Ph2N
O
OCH3
Ph
3o
15.29
1.15
1.15
1.31
3.00
2.16
2.42
2.11
f1 (ppm)


JYJ-6-41-C
171.52
141.86
132.95
128.96
128.44
128.30
125.81
125.36
78.31
57.06
35.78
35.65
34.47
Ph2N
O
OCH3
Ph
3o
f1 (ppm)

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\6\JYJ-6-42-RAC.lcd

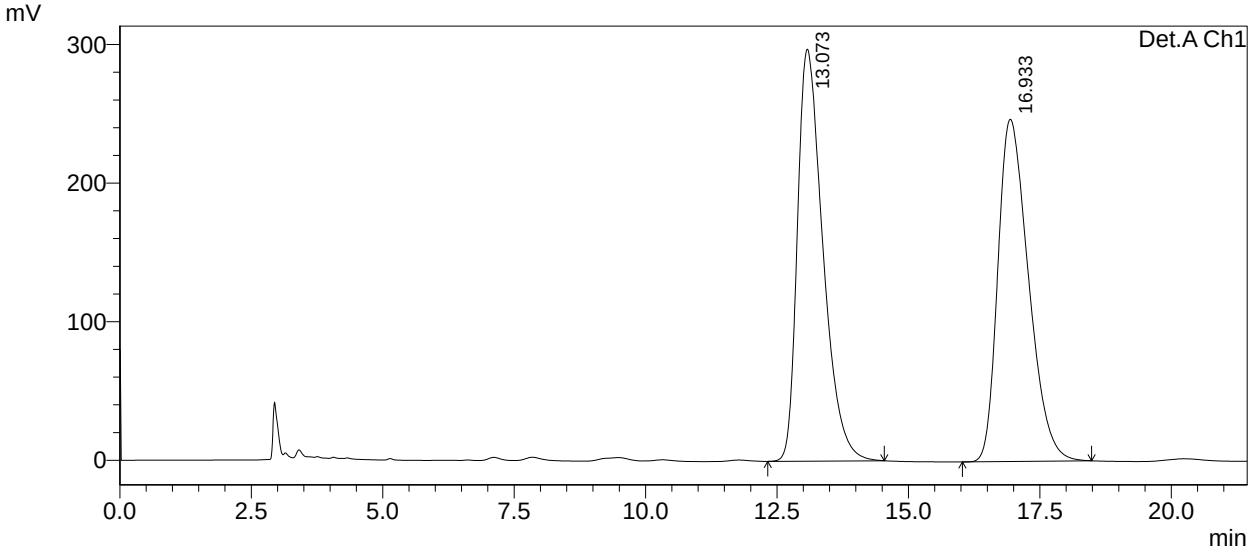
Acquired by	: Admin
Sample Name	: JYJ-6-42-RAC
Sample ID	: OD-H,99/1,1,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: JYJ-6-42-RAC.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2016-12-10 16:25:58
Data Processed	: 2016-12-10 17:09:00



3o

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\6\JYJ-6-42-RAC.lcd



1 Det.A Ch1/214nm

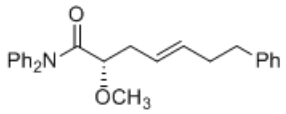
PeakTable

Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.073	10036249	297346	50.048	54.625
2	16.933	10017083	246999	49.952	45.375
Total		20053332	544345	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\6\JYJ-6-42.lcd

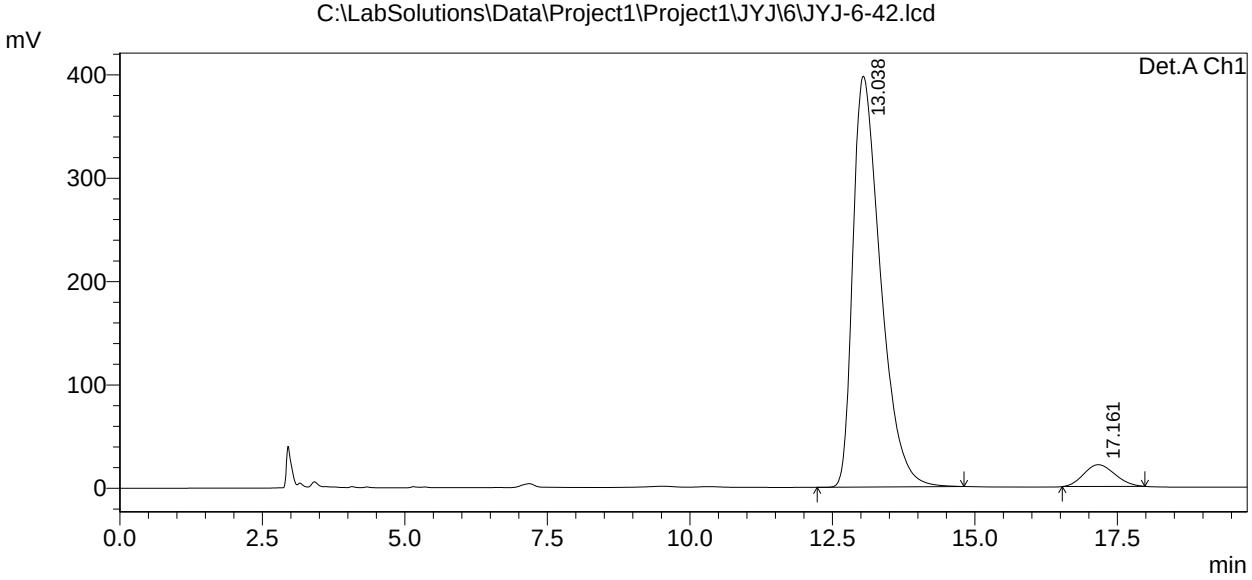


3o

Acquired by	: Admin
Sample Name	: JYJ-6-42
Sample ID	: OD-H,90/10,1,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: JYJ-6-42.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2016-12-10 16:49:03
Data Processed	: 2016-12-10 17:11:26

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\6\JYJ-6-42.lcd

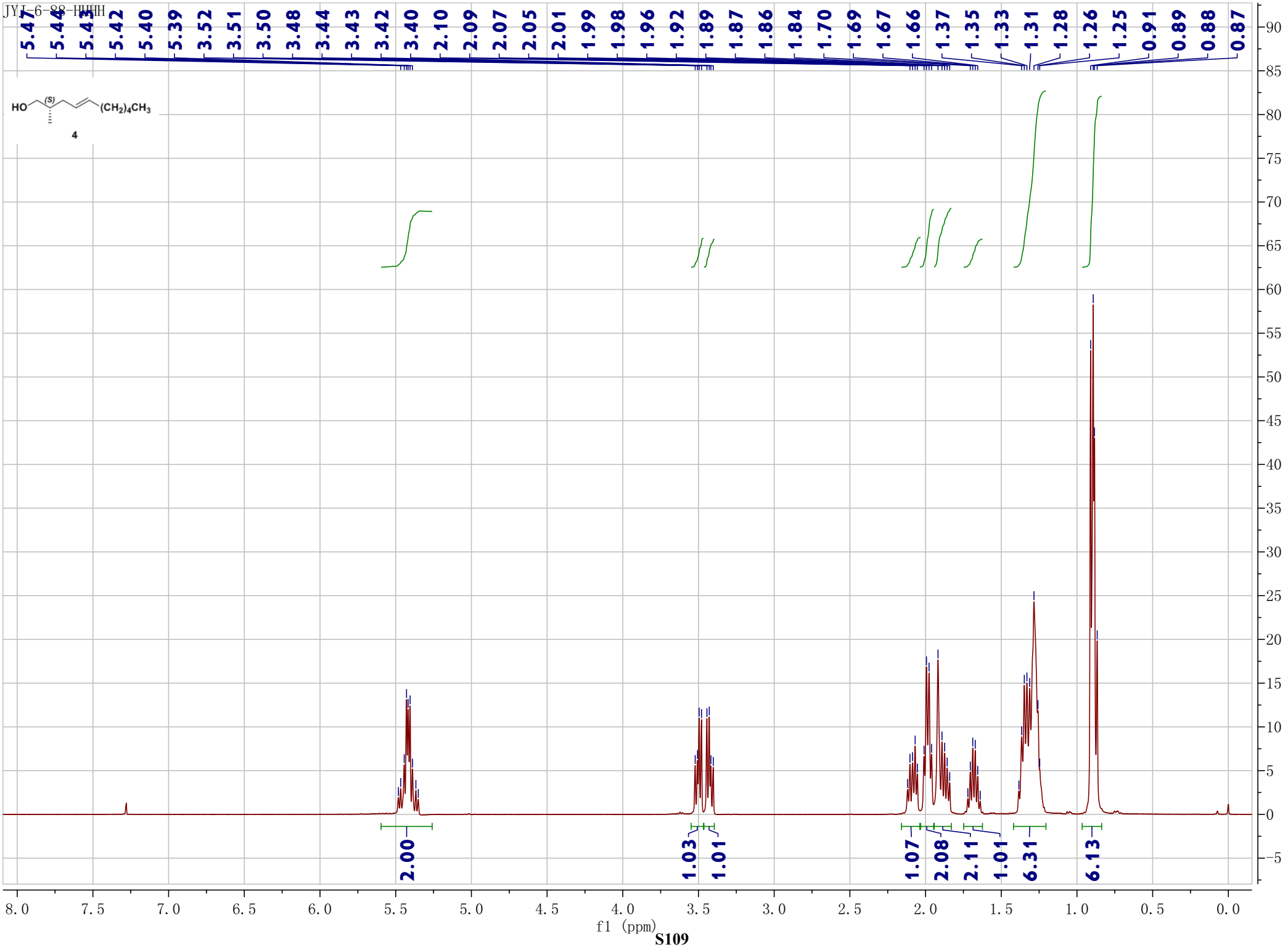


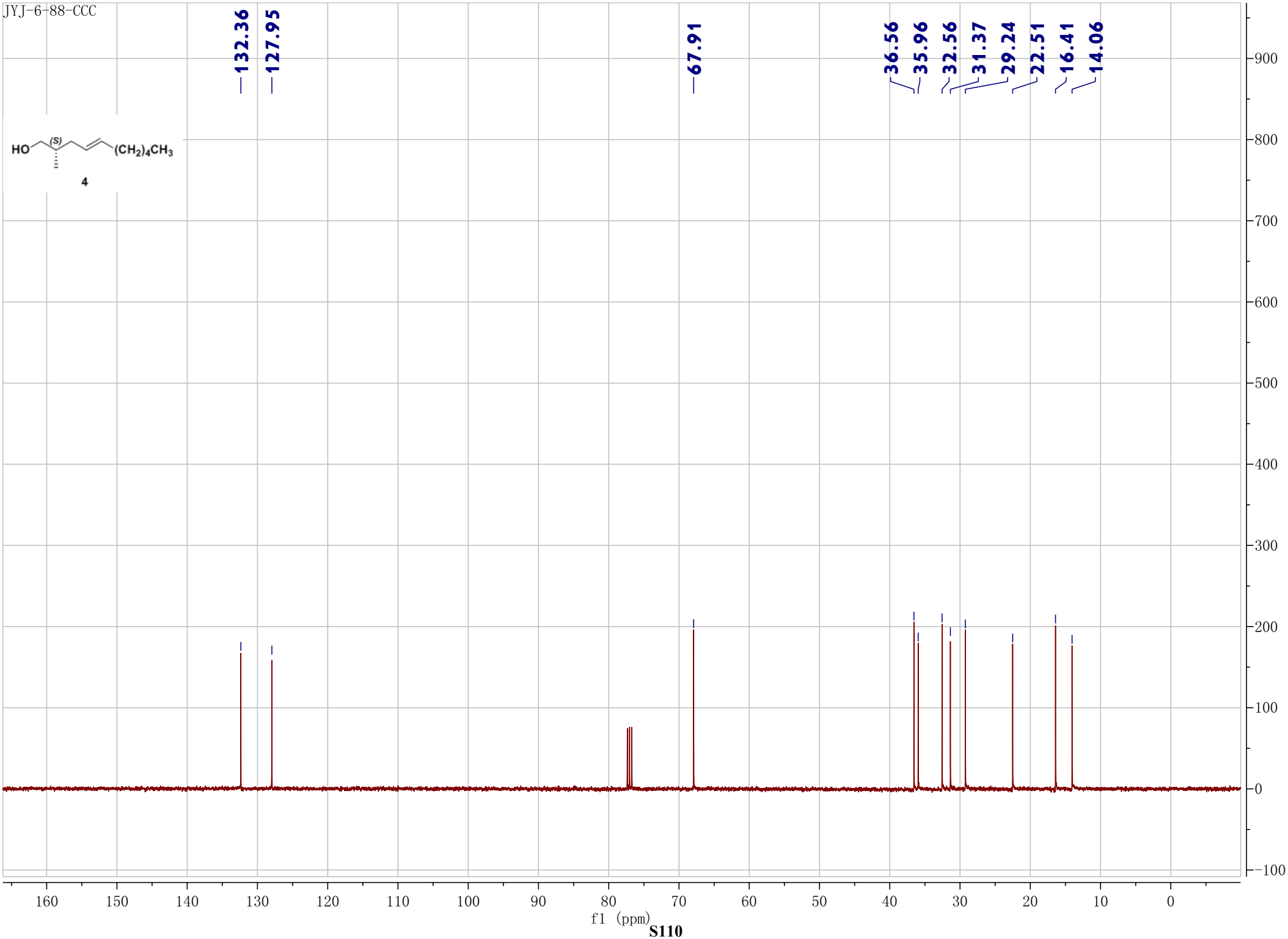
1 Det.A Ch1/214nm

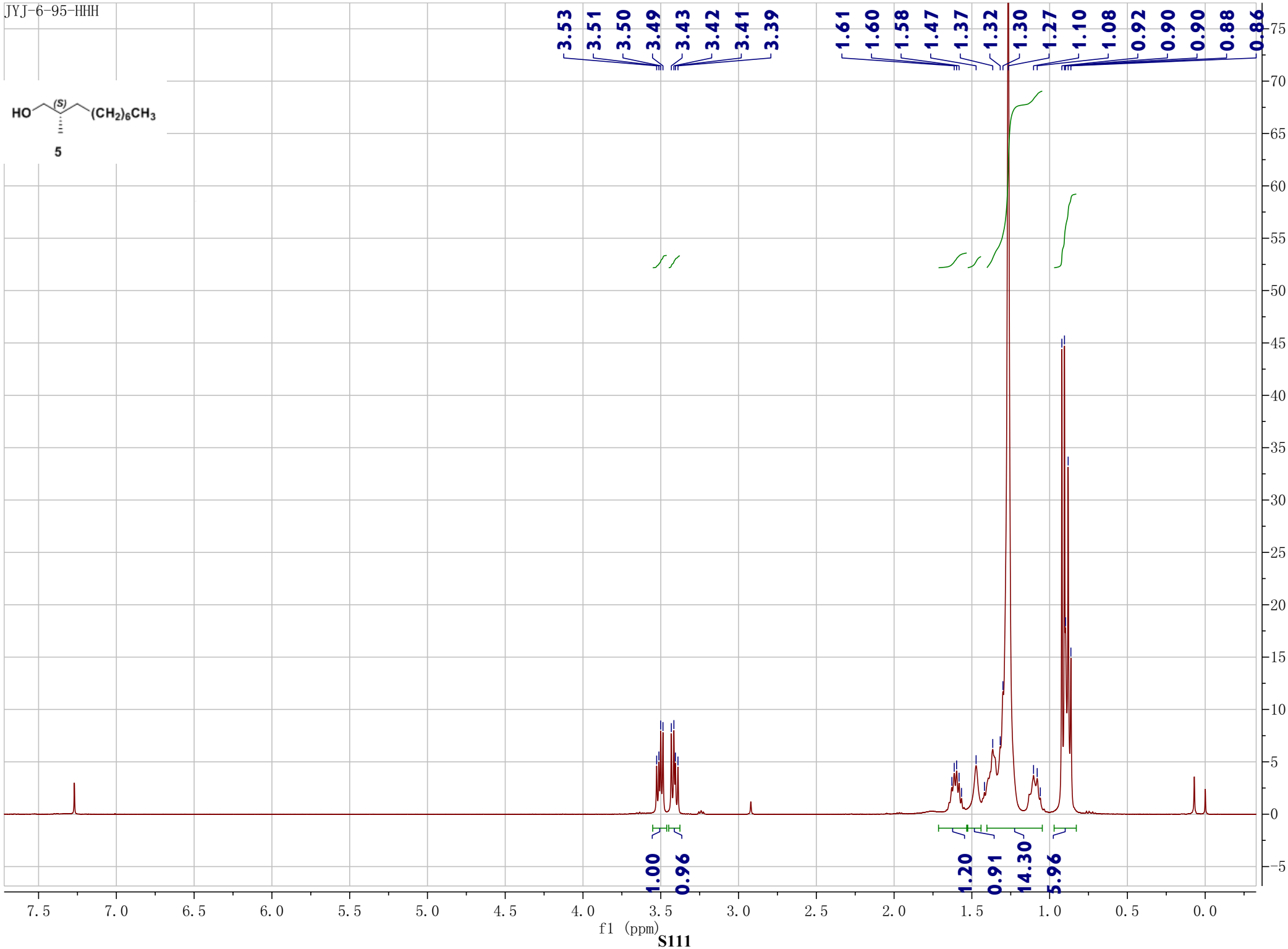
PeakTable

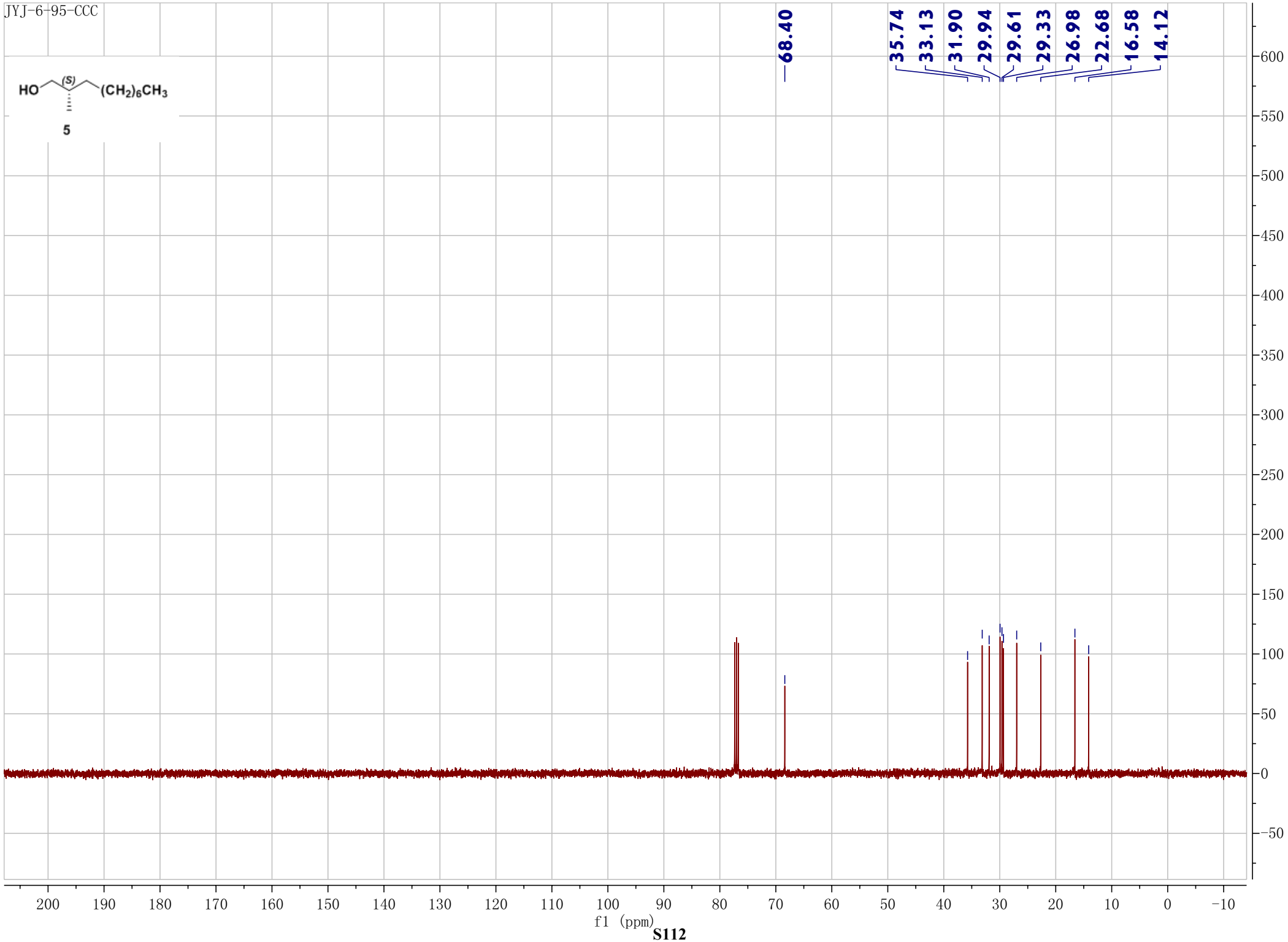
Detector A Ch1 214nm

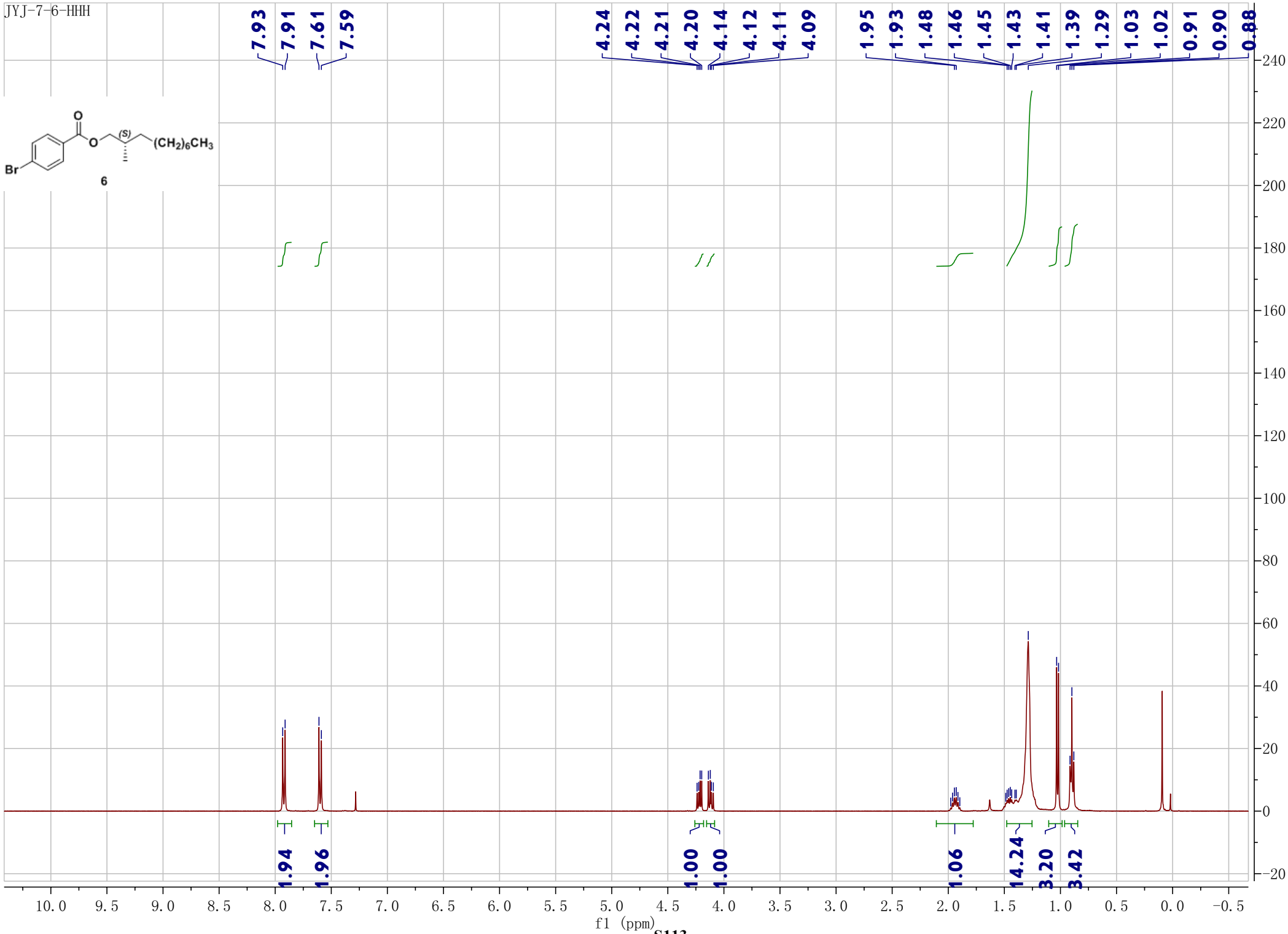
Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.038	13422479	397594	94.367	94.958
2	17.161	801170	21111	5.633	5.042
Total		14223649	418705	100.000	100.000

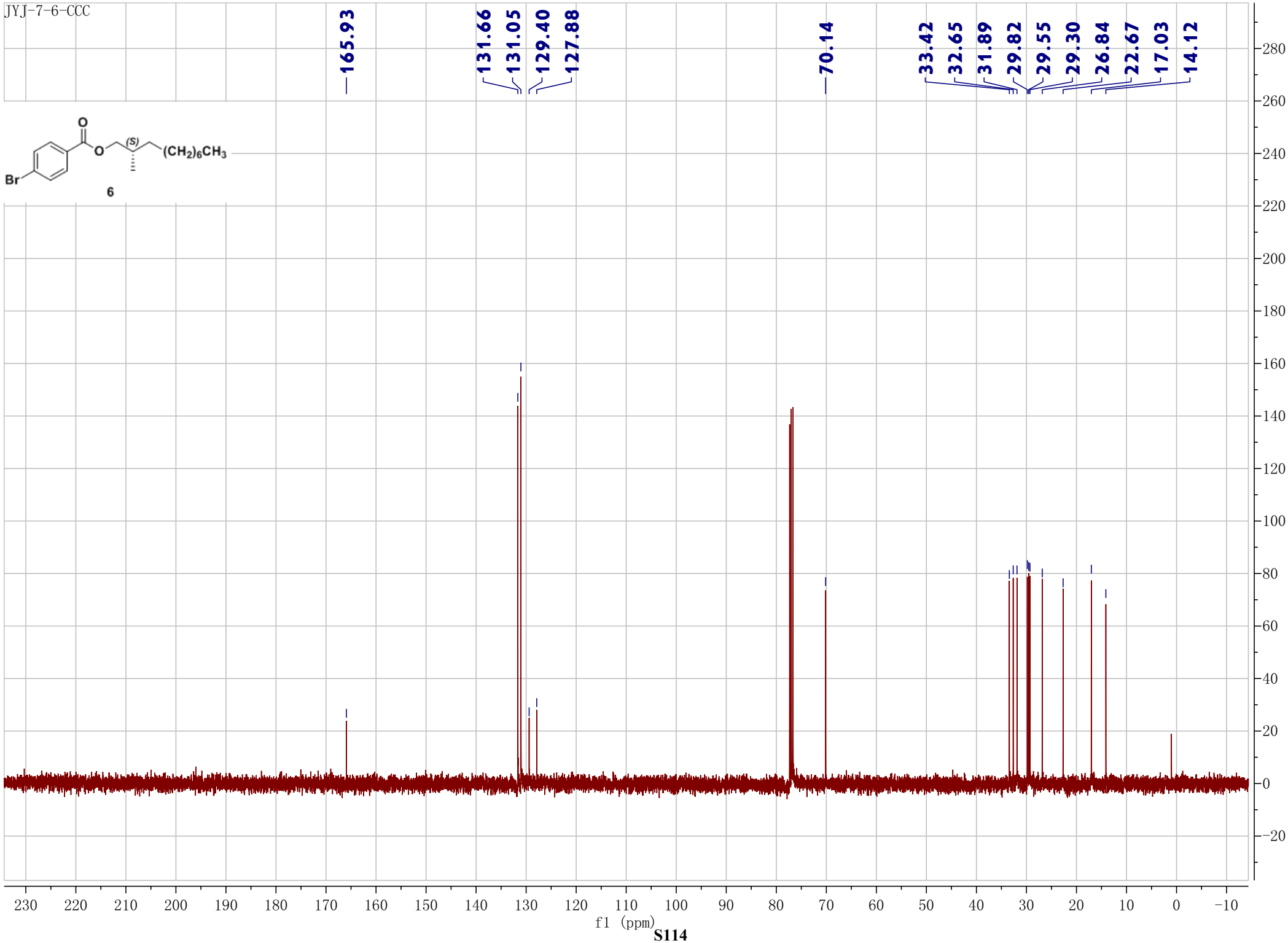

JYL-6-88-HHH
5.47
5.44
5.43
5.42
5.40
5.39
3.52
3.51
3.50
3.48
3.44
3.43
3.42
3.40
2.10
2.09
2.07
2.05
2.01
1.99
1.98
1.96
1.92
1.89
1.87
1.86
1.84
1.70
1.69
1.67
1.66
1.37
1.35
1.33
1.31
1.28
1.26
1.25
0.91
0.89
0.88
0.87
HO
(S)
(CH2)4CH3
4
2.00
1.03
1.01
1.07
2.08
2.11
1.01
6.31
6.13
f1 (ppm)


JYJ-6-88-CCC
132.36
127.95
67.91
36.56
35.96
32.56
31.37
29.24
22.51
16.41
14.06
HO
(S)
(CH2)4CH3
4
f1 (ppm)


JYJ-6-95-HHH
3.53
3.51
3.50
3.49
3.43
3.42
3.41
3.39
1.61
1.60
1.58
1.47
1.37
1.32
1.30
1.27
1.10
1.08
0.92
0.90
0.90
0.88
0.86
HO
(S)
(CH2)6CH3
5
1.00
0.96
1.20
0.91
14.30
5.96
f1 (ppm)

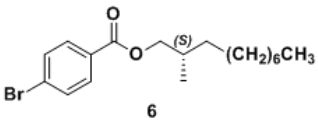

JYJ-6-95-CCC
HO
(S)
(CH2)6CH3
5
68.40
35.74
33.13
31.90
29.94
29.61
29.33
26.98
22.68
16.58
14.12
600
550
500
450
400
350
300
250
200
150
100
50
0
−50
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)


JYJ-7-6-HHH
7.93
7.91
7.61
7.59
4.24
4.22
4.21
4.20
4.14
4.12
4.11
4.09
1.95
1.93
1.48
1.46
1.45
1.43
1.41
1.39
1.29
1.03
1.02
0.91
0.90
0.88
Br
(S)
(CH2)6CH3
6
1.94
1.96
1.00
1.00
1.06
14.24
3.20
3.42
f1 (ppm)

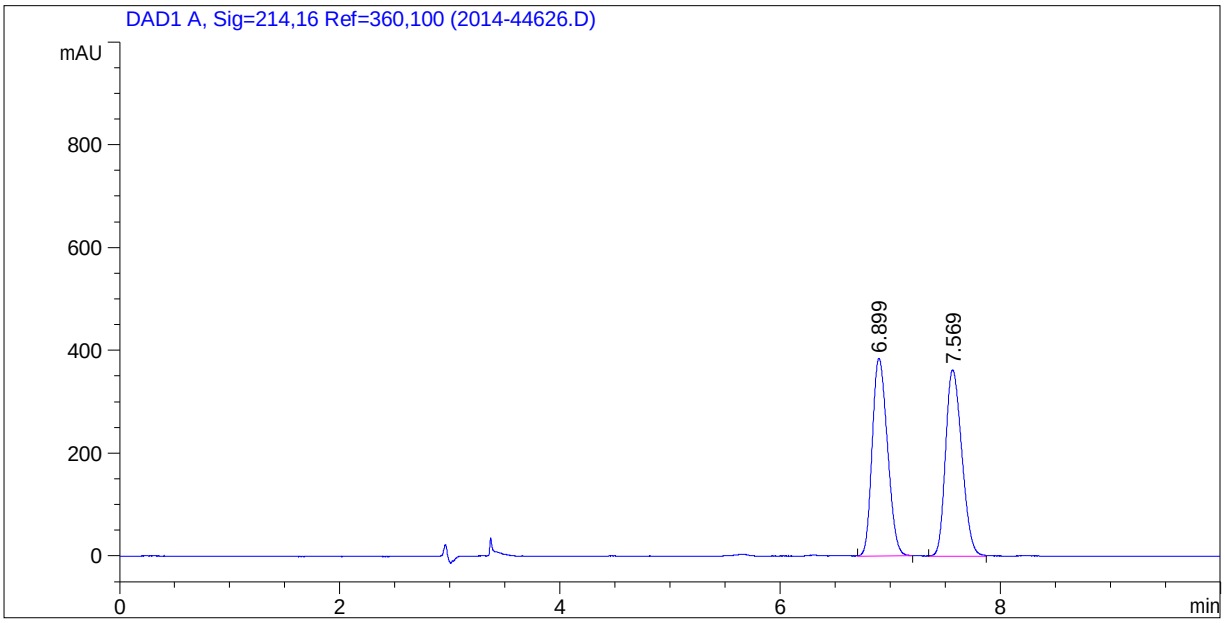

JYJ-7-6-CCC
165.93
131.66
131.05
129.40
127.88
70.14
33.42
32.65
31.89
29.82
29.55
29.30
26.84
22.67
17.03
14.12
O
O
(S)
(CH2)6CH3
Br
6
f1 (ppm)

Data File C:\CHEM32\1\DATA\2014-44626.D
Sample Name: jyj-7-6-rac-oj-h-98-2

```
==========================================================================
Acq. Operator   : 系统
Sample Operator : 系统
Acq. Instrument : SFC                              Location : Vial 2
Injection Date  : 07/03/2017 15:59:36
                                                   Inj Volume : 3.000 µl
Acq. Method     : C:\CHEM32\1\METHODS\DEF_LC-TEST-2016.M
Last changed    : 07/03/2017 15:51:55 by 系统
                  (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC-TEST-2016.M
Last changed    : 13/03/2017 16:49:53 by 系统
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

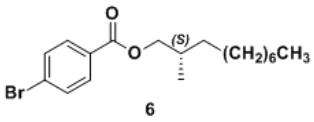


6




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=214,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.899 VV    0.1528 3700.52661  384.53714  49.9081
   2   7.569 VV    0.1593 3714.14746  361.81332  50.0919

Totals :                  7414.67407  746.35046


==========================================================================
                        *** End of Report ***
```

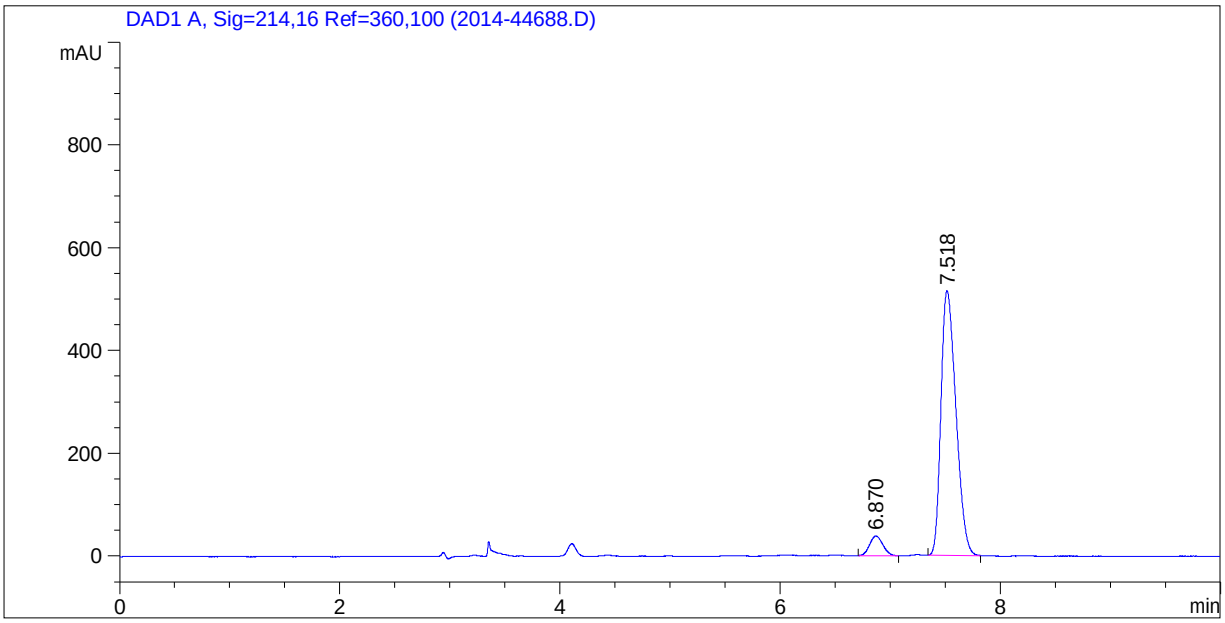
Data File C:\CHEM32\1\DATA\2014-44688.D
Sample Name: JYJ-7-22



6

==========

Acq. Operator : 系统
Sample Operator : 系统
Acq. Instrument : SFC Location : Vial 11
Injection Date : 16/03/2017 14:31:05
Inj Volume : 1.000 µl
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC-TEST-2016.M
Last changed : 16/03/2017 14:19:22 by 系统
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC-TEST-2016.M
Last changed : 27/02/2017 16:26:37 by 系统
Additional Info : Peak(s) manually integrated



==========

Area Percent Report

==========

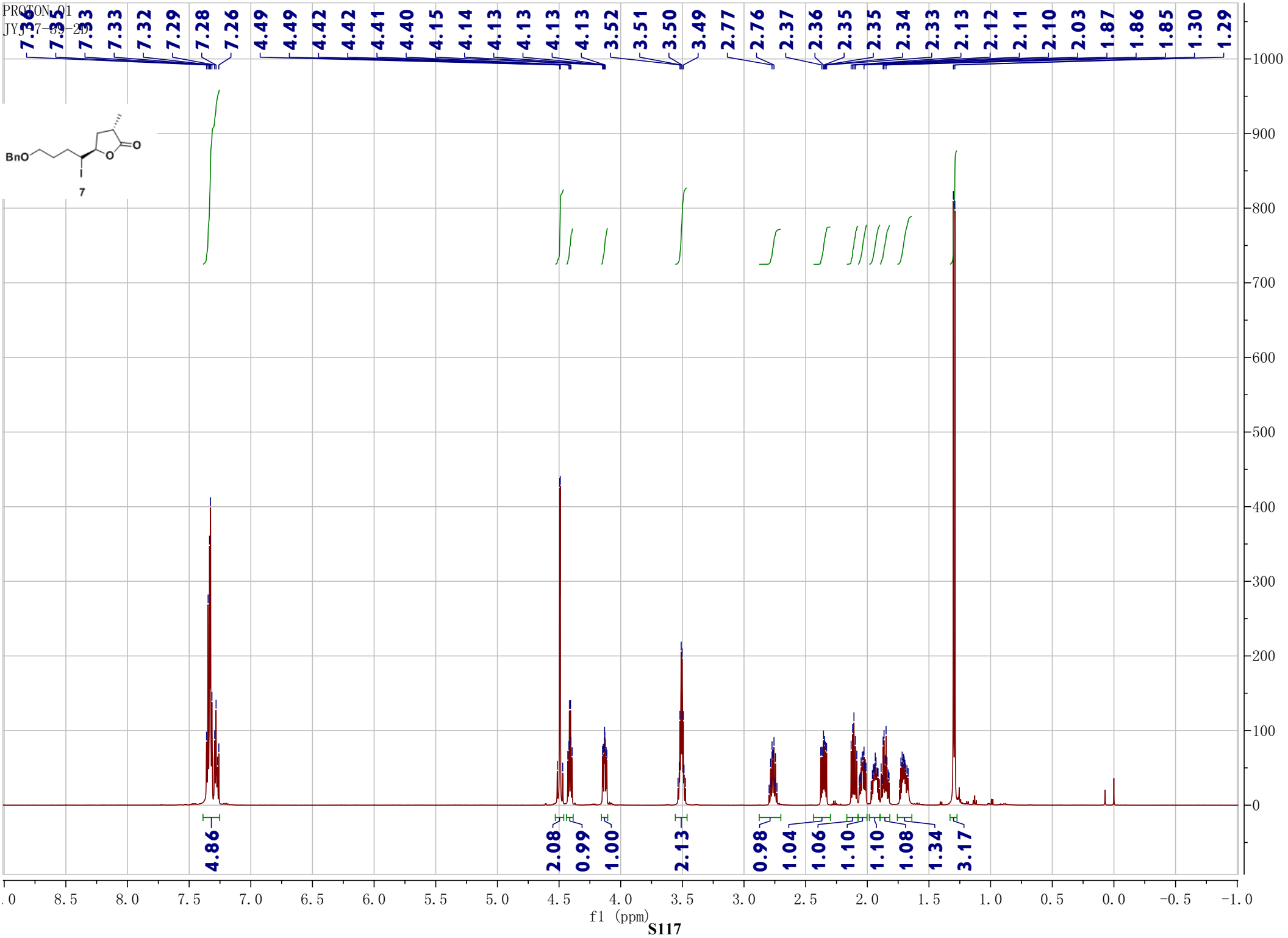
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

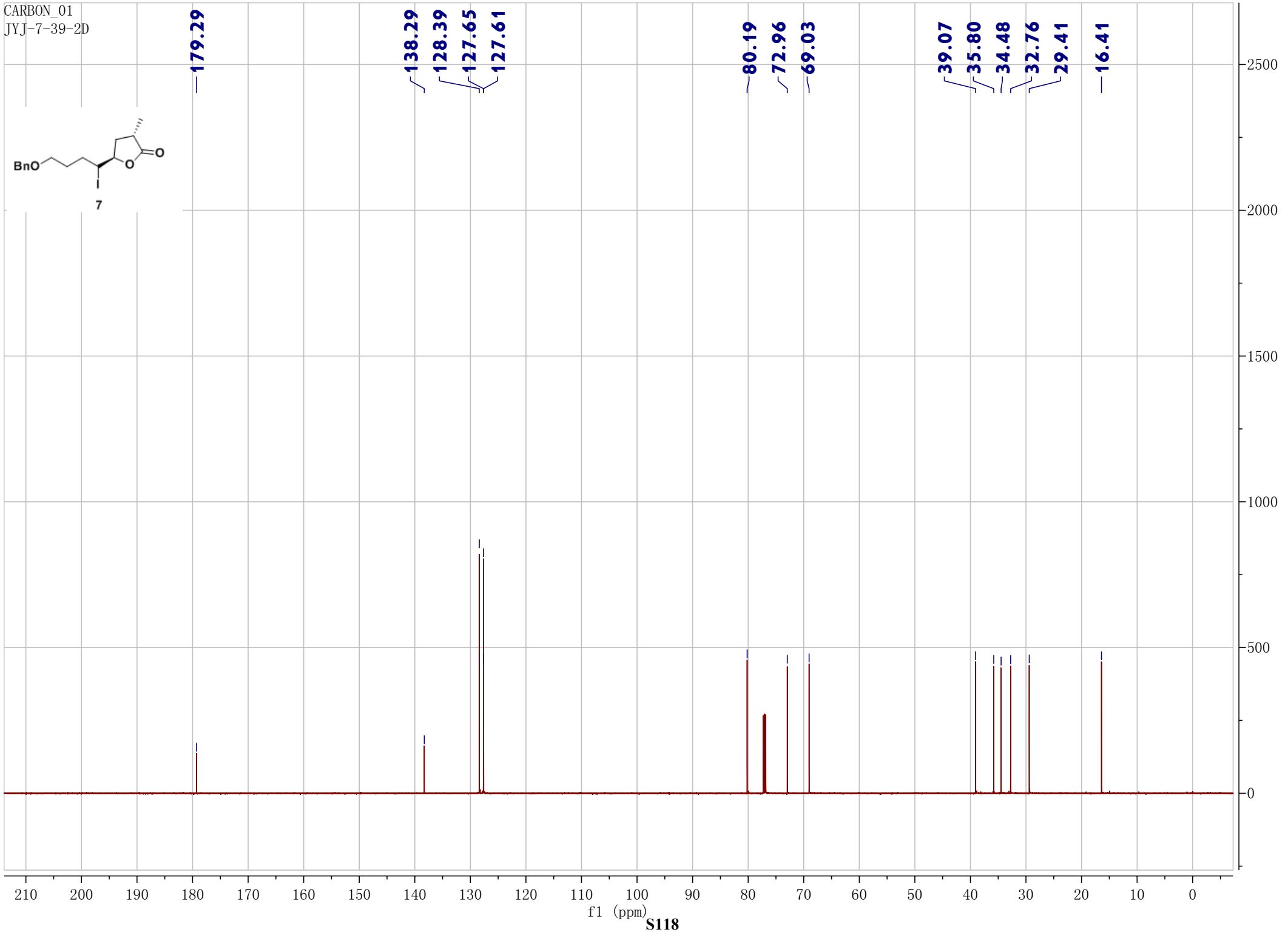
Signal 1: DAD1 A, Sig=214,16 Ref=360,100

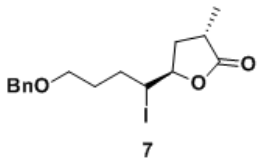
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.870	VV	0.1209	329.39648	38.67027	6.3413
2	7.518	VV	0.1426	4865.03174	515.31256	93.6587
Totals :				5194.42822	553.98283	

==========

*** End of Report ***


PROTON-01
JYJ-7-39-2D
7.36
7.35
7.33
7.33
7.32
7.29
7.28
7.26
4.49
4.49
4.42
4.42
4.41
4.40
4.15
4.14
4.13
4.13
4.13
4.13
3.52
3.51
3.50
3.49
2.77
2.76
2.37
2.36
2.35
2.35
2.34
2.33
2.13
2.12
2.11
2.10
2.03
1.87
1.86
1.85
1.30
1.29
BnO
O
O
I
7
4.86
2.08
0.99
1.00
2.13
0.98
1.04
1.06
1.10
1.10
1.08
1.34
3.17
f1 (ppm)


CARBON_01
JYJ-7-39-2D
179.29
138.29
128.39
127.65
127.61
80.19
72.96
69.03
39.07
35.80
34.48
32.76
29.41
16.41
BnO
O
O
I
7
2500
2000
1500
1000
500
0
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



7

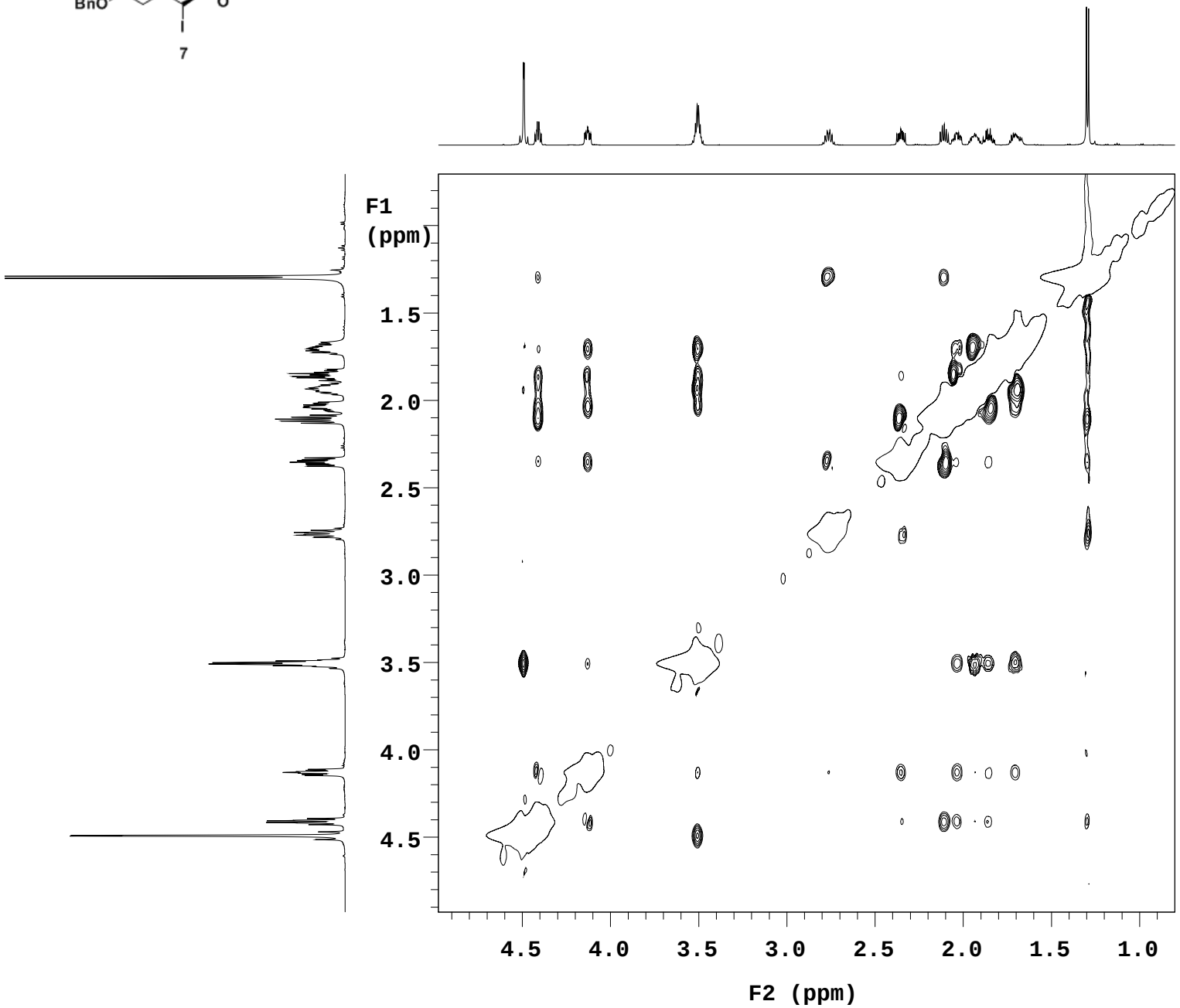
JYJ-7-39-2D

Sample Name:
JYJ-7-39-2D
Data Collected on:
OMC-NMR600-vnmrs600
Archive directory:
/home/omc/vnmrsys/data
Sample directory:
JYJ-7-39-2D_20170320_01
FidFile: NOESY_01

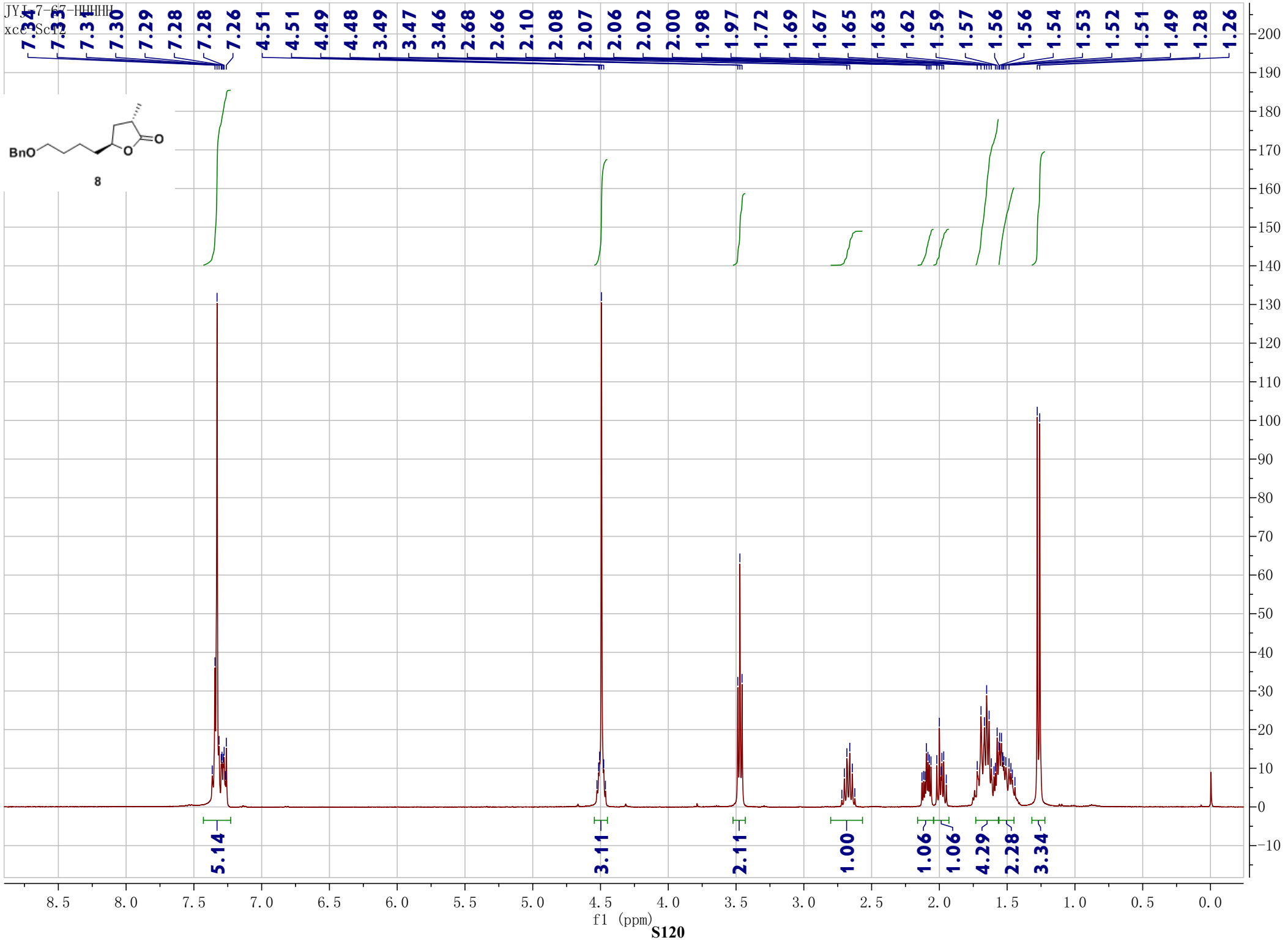
Pulse Sequence: NOESY
Solvent: cdcl3
Data collected on: Mar 20 2017

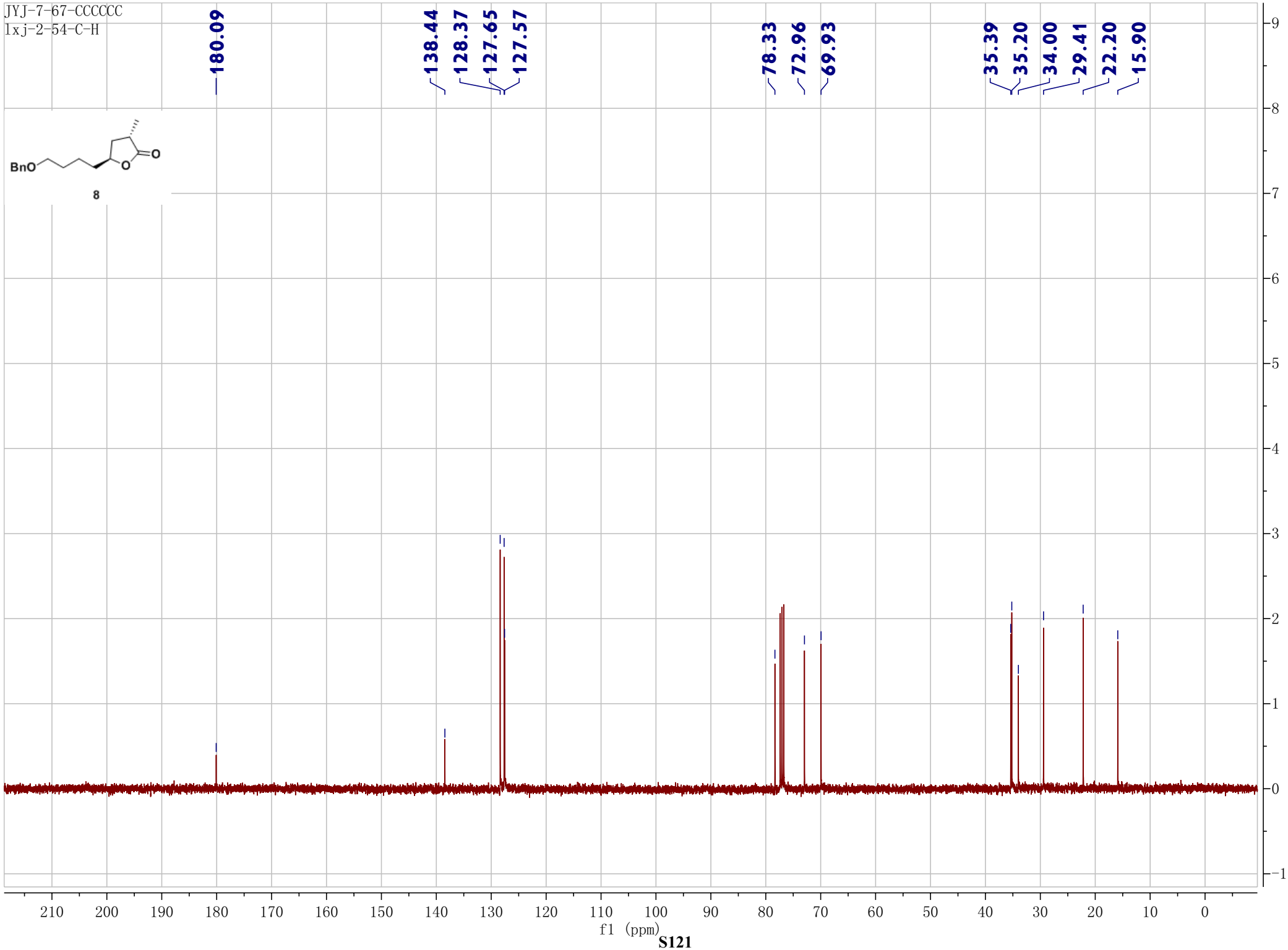
Temp. 25.0 C / 298.1 K
Operator: omc

Relax. delay 1.500 sec
Acq. time 0.382 sec
Width 5787.0 Hz
2D Width 5787.0 Hz
8 repetitions
2 x 128 increments
OBSERVE H1, 599.7751422 MHz
DATA PROCESSING
Line broadening 3.0 Hz
Gauss apodization 0.056 sec
F1 DATA PROCESSING
Gauss apodization 0.012 sec
FT size 4096 x 2048
Total time 1 hr, 20 min



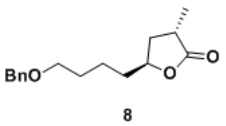
Plotname: --Not assigned--


JYL-7-67-HHHH
xcc1Sc12
7.34 7.33 7.31 7.30 7.29 7.28 7.28 7.26 4.51 4.51 4.49 4.48 3.49 3.47 3.46 2.68 2.66 2.10 2.08 2.07 2.06 2.02 2.00 1.98 1.97 1.72 1.69 1.67 1.65 1.63 1.62 1.59 1.57 1.56 1.56 1.54 1.53 1.52 1.51 1.49 1.28 1.26
BnO
O
O
8
5.14
3.11
2.11
1.00
1.06
1.06
4.29
2.28
3.34
f1 (ppm)


JYJ-7-67-CCCCCC
lxj-2-54-C-H
180.09
138.44
128.37
127.65
127.57
78.33
72.96
69.93
35.39
35.20
34.00
29.41
22.20
15.90
BnO
O
O
8
f1 (ppm)

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\7\JYJ-7-81-RAC.lcd

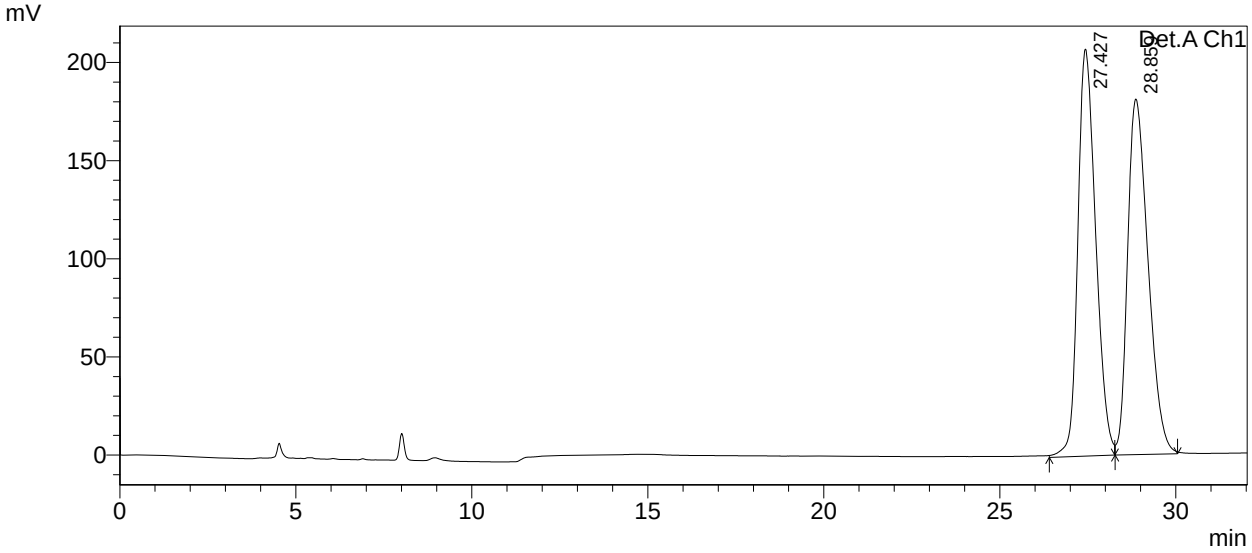


8

Acquired by	: Admin
Sample Name	: JYJ-7-81-RAC
Sample ID	: OD-H,95/5,0.7,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: JYJ-7-81-RAC.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2017-4-18 21:39:40
Data Processed	: 2017-4-18 22:51:23

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\7\JYJ-7-81-RAC.lcd



1 Det.A Ch1/214nm

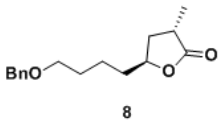
PeakTable

Detector A Ch1 214nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	27.427	7344401	207373	50.039	53.380
2	28.859	7333018	181108	49.961	46.620
Total		14677420	388481	100.000	100.000

==== Shimadzu LCsolution Analysis Report ====

C:\LabSolutions\Data\Project1\Project1\JYJ\7\JYJ-7-82.lcd

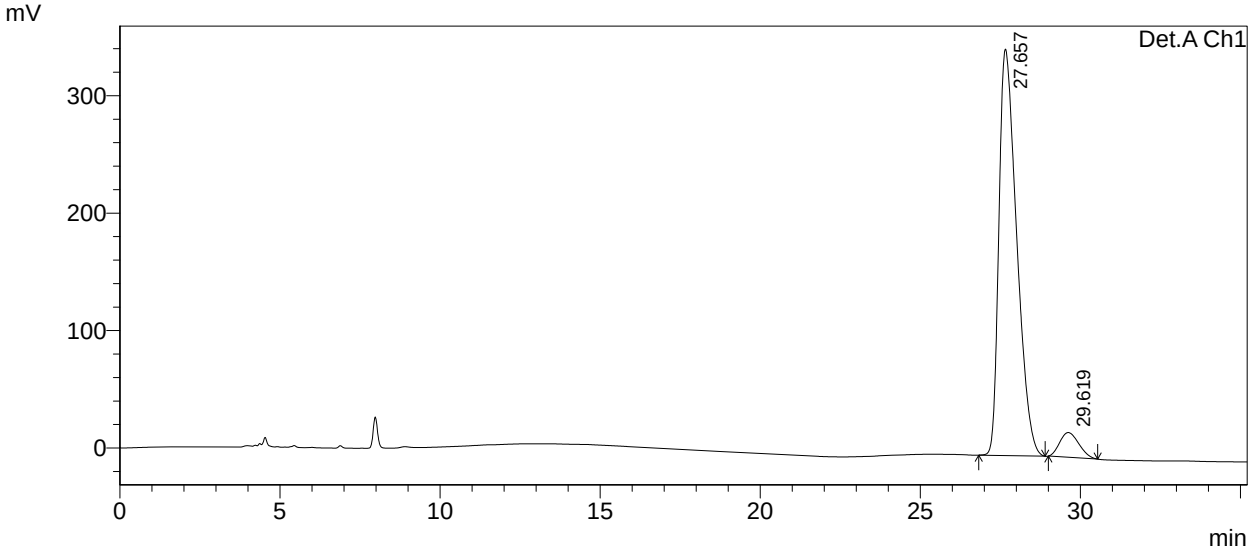


8

Acquired by	: Admin
Sample Name	: JYJ-7-82
Sample ID	: OD-H,95/5,0.7,214
Vail #	:
Injection Volume	: 2 uL
Data File Name	: JYJ-7-82.lcd
Method File Name	: 123.lcm
Batch File Name	:
Report File Name	: Default.lcr
Data Acquired	: 2017-4-18 22:15:55
Data Processed	: 2017-4-20 10:57:47

<Chromatogram>

C:\LabSolutions\Data\Project1\Project1\JYJ\7\JYJ-7-82.lcd

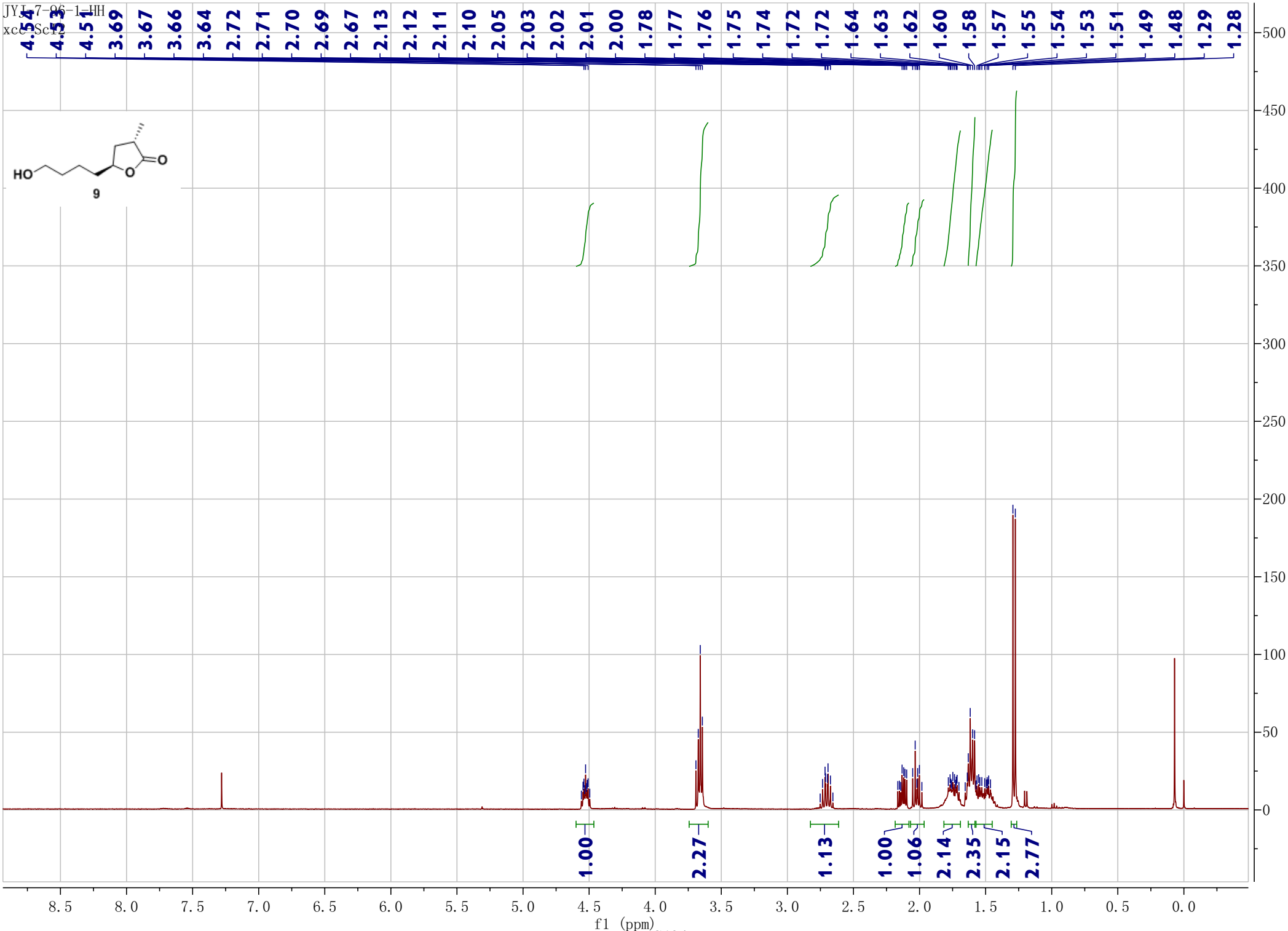


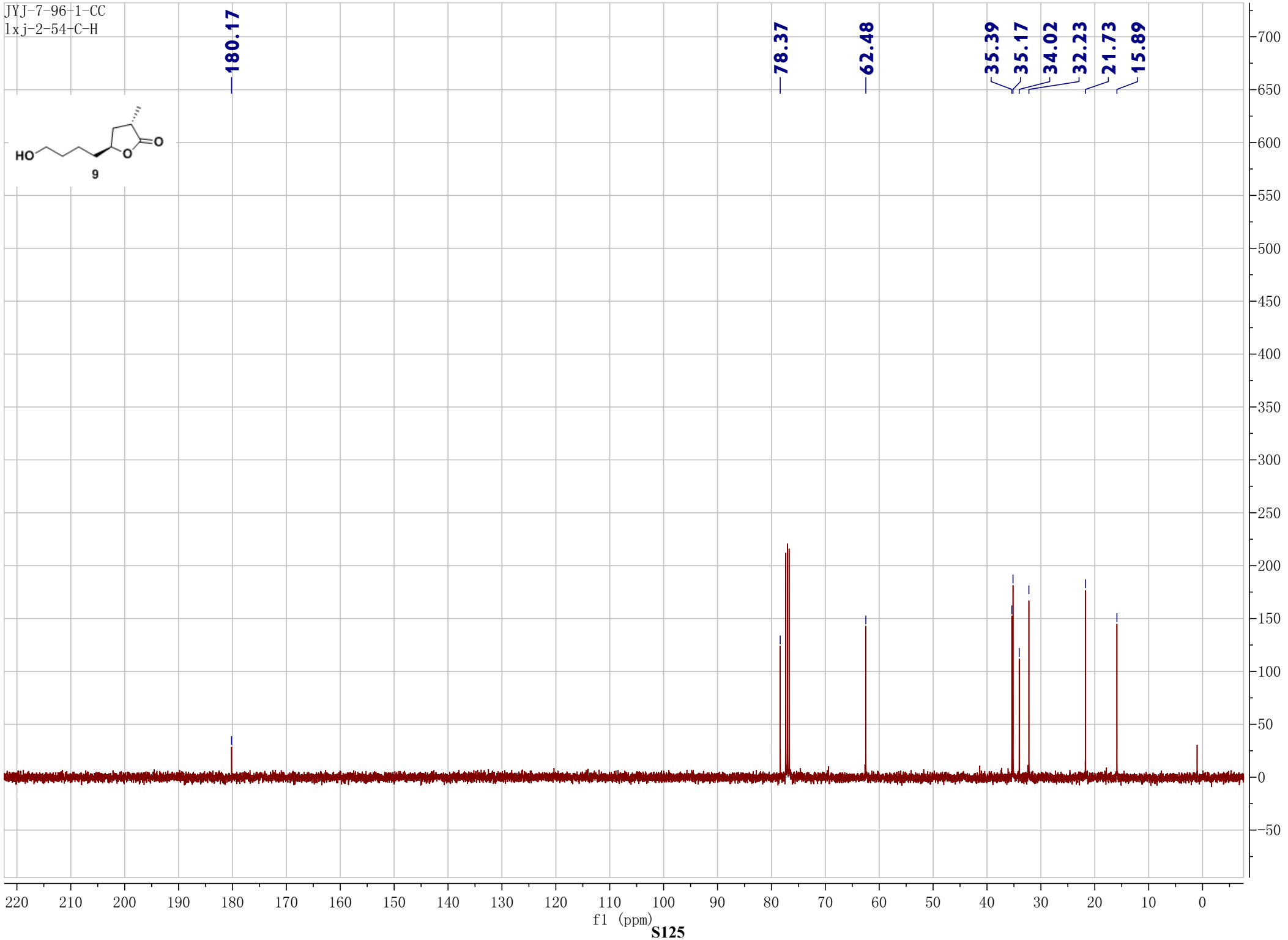
1 Det.A Ch1/214nm

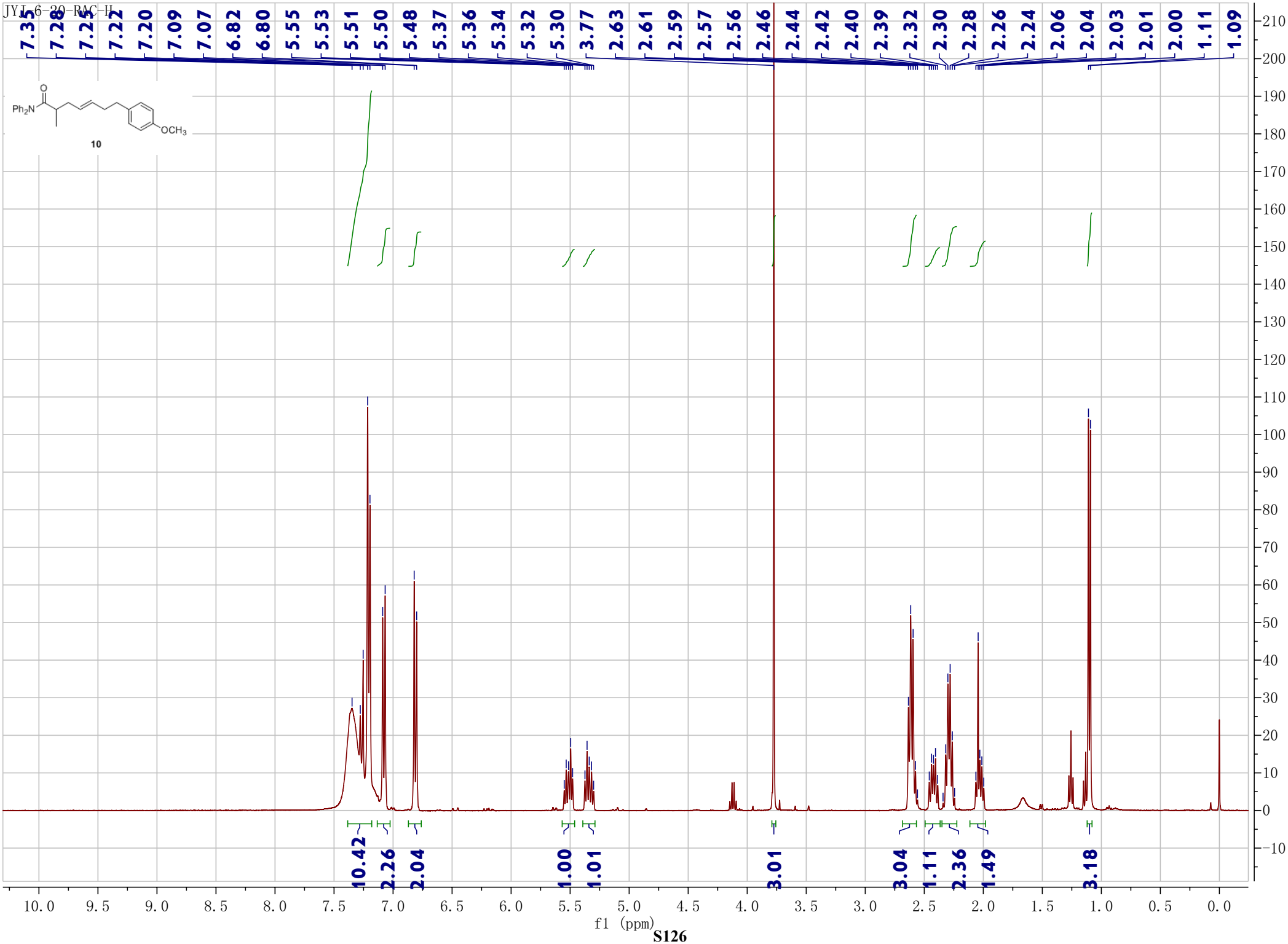
PeakTable

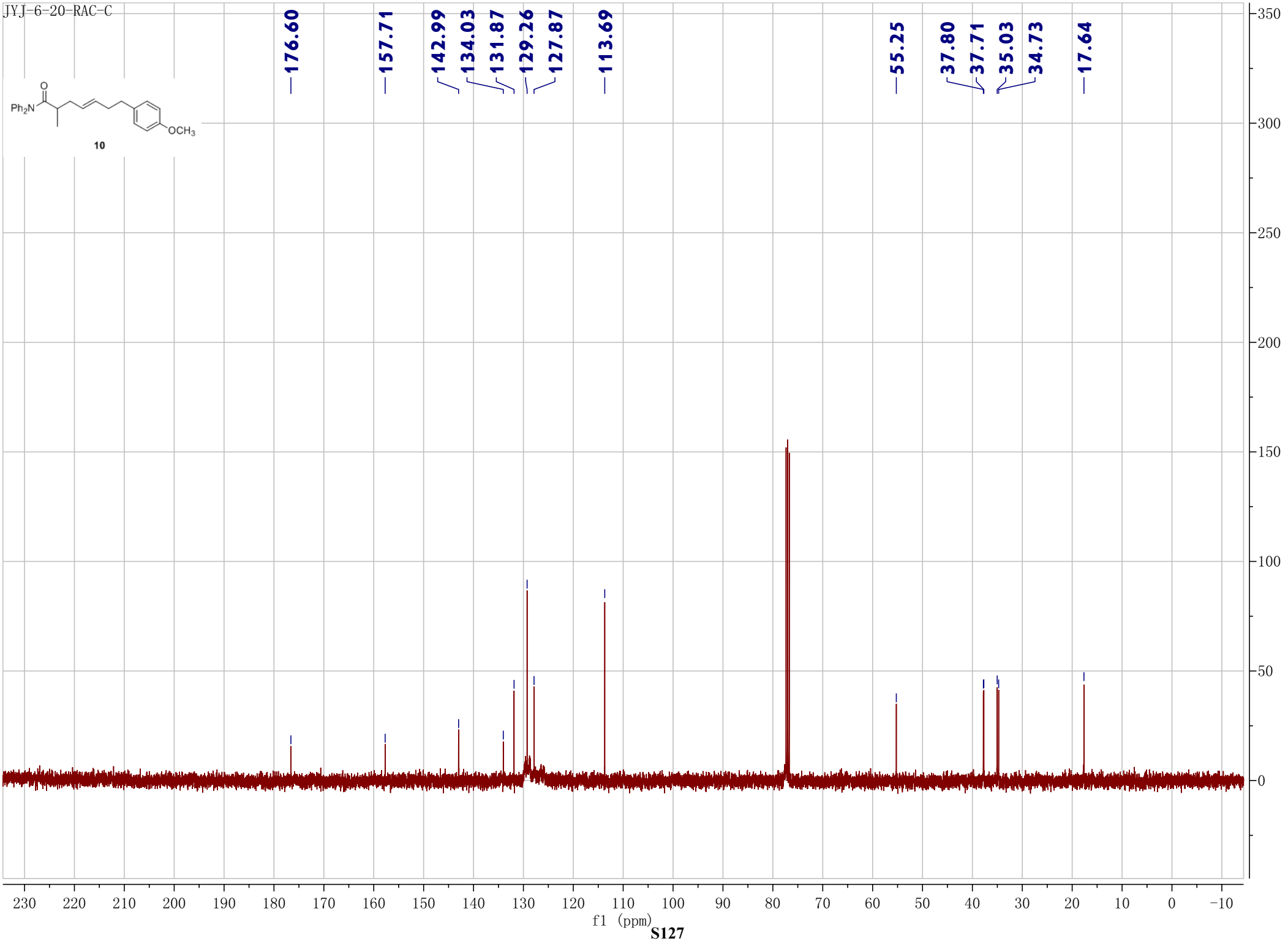
Detector A Ch1 214nm

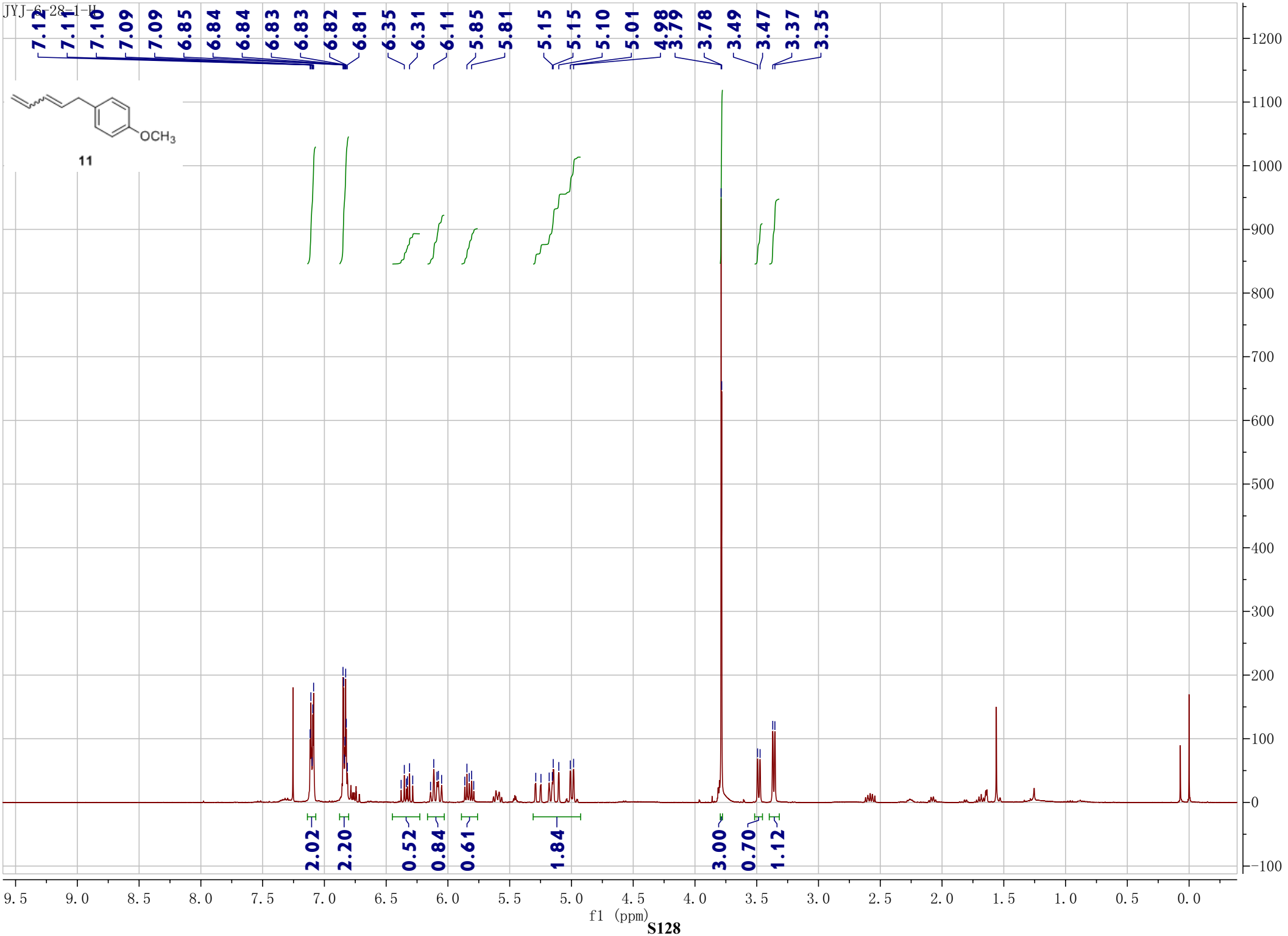
Peak#	Ret. Time	Area	Height	Area %	Height %
1	27.657	13357004	346109	93.821	94.258
2	29.619	879748	21083	6.179	5.742
Total		14236752	367192	100.000	100.000

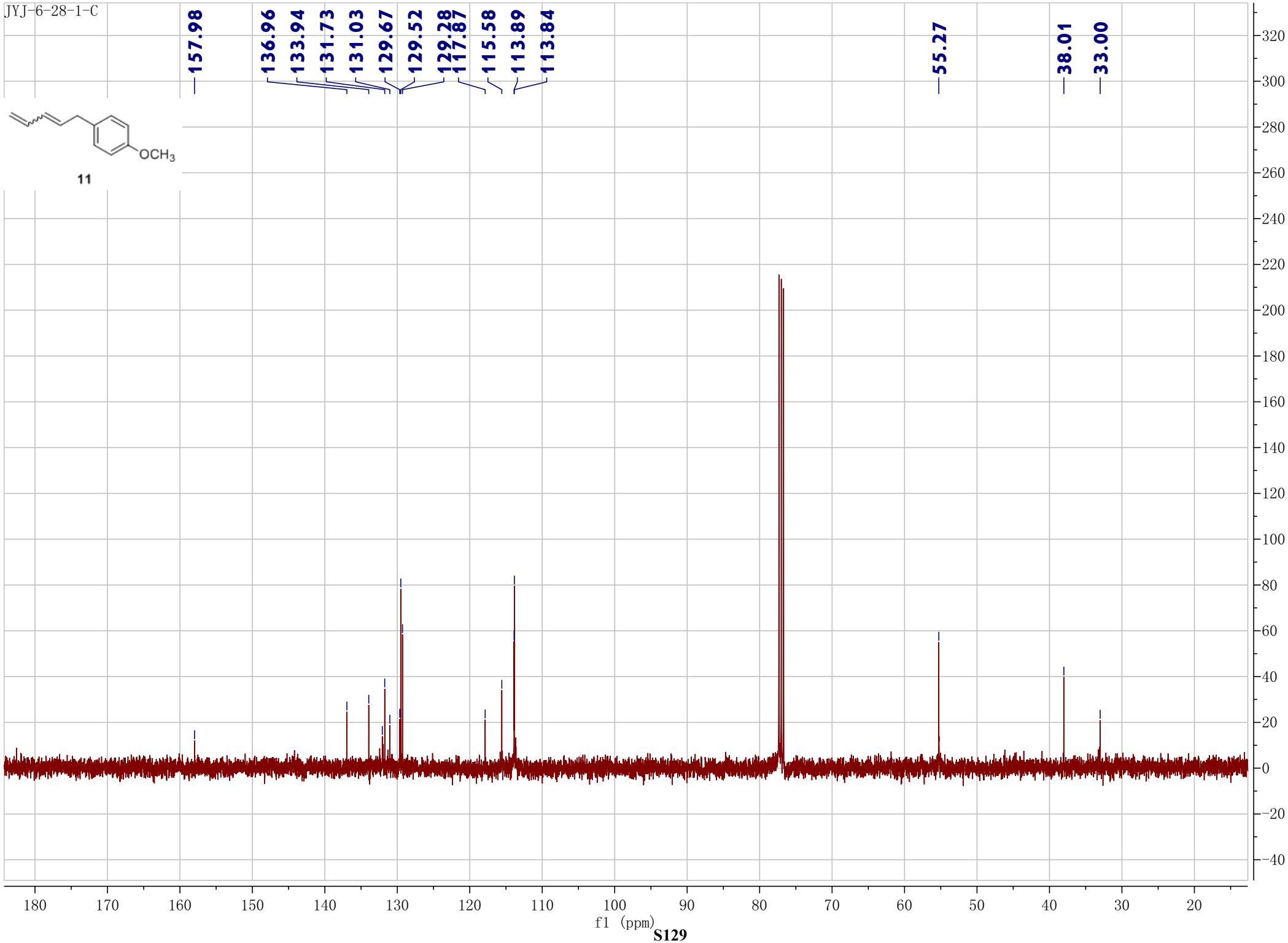

JYL-7-06-1-HH
xcc.1Sc1r
4.54
4.53
4.51
3.69
3.67
3.66
3.64
2.72
2.71
2.70
2.69
2.67
2.13
2.12
2.11
2.10
2.05
2.03
2.02
2.01
2.00
1.78
1.77
1.76
1.75
1.74
1.72
1.72
1.64
1.63
1.62
1.60
1.58
1.57
1.55
1.54
1.53
1.51
1.49
1.48
1.29
1.28
HO
O
O
9
1.00
2.27
1.13
1.00
1.06
2.14
2.35
2.15
2.77
f1 (ppm)

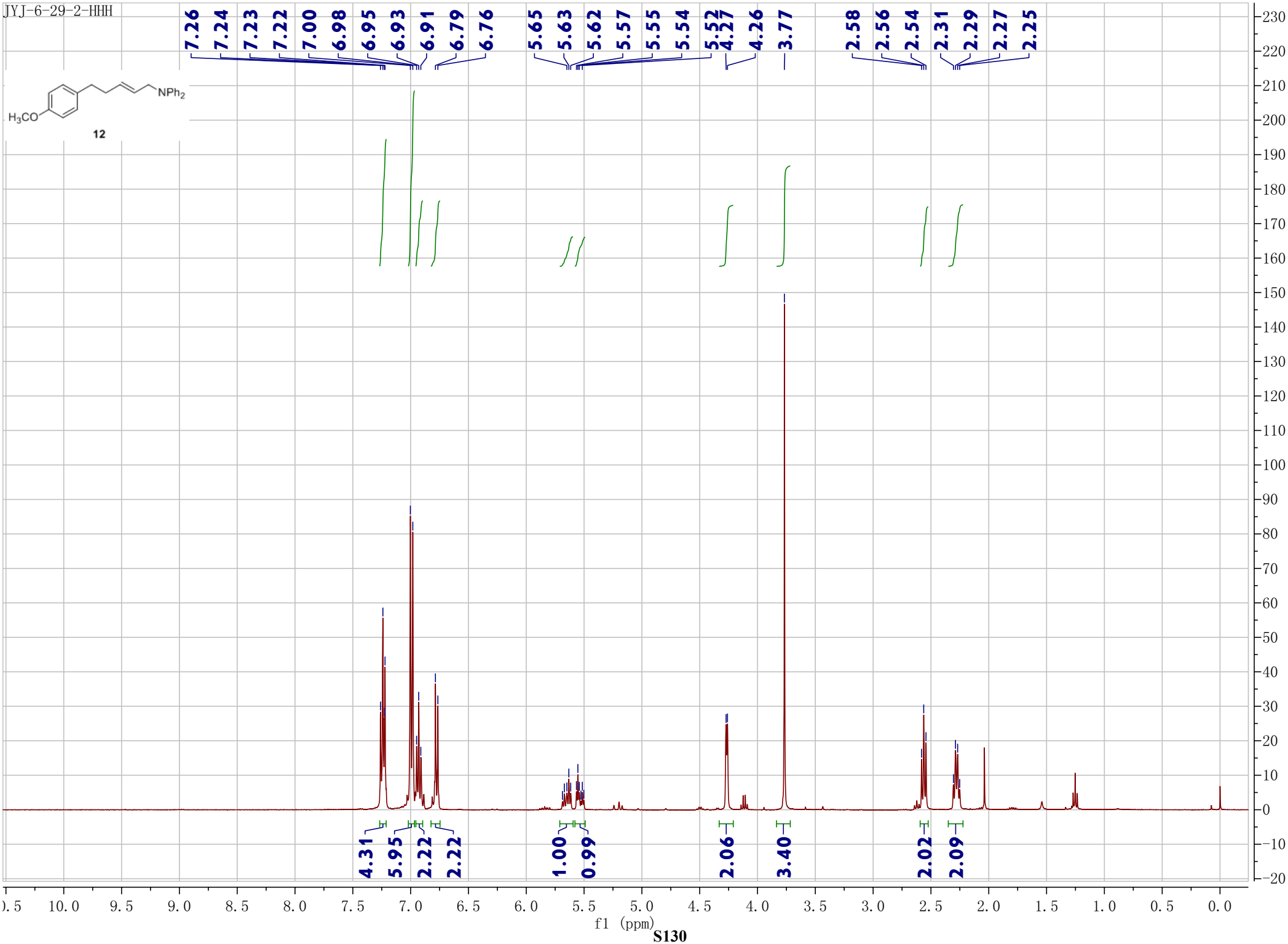

JYJ-7-96-1-CC
lxj-2-54-C-H
180.17
78.37
62.48
35.39
35.17
34.02
32.23
21.73
15.89
HO
O
O
9
f1 (ppm)

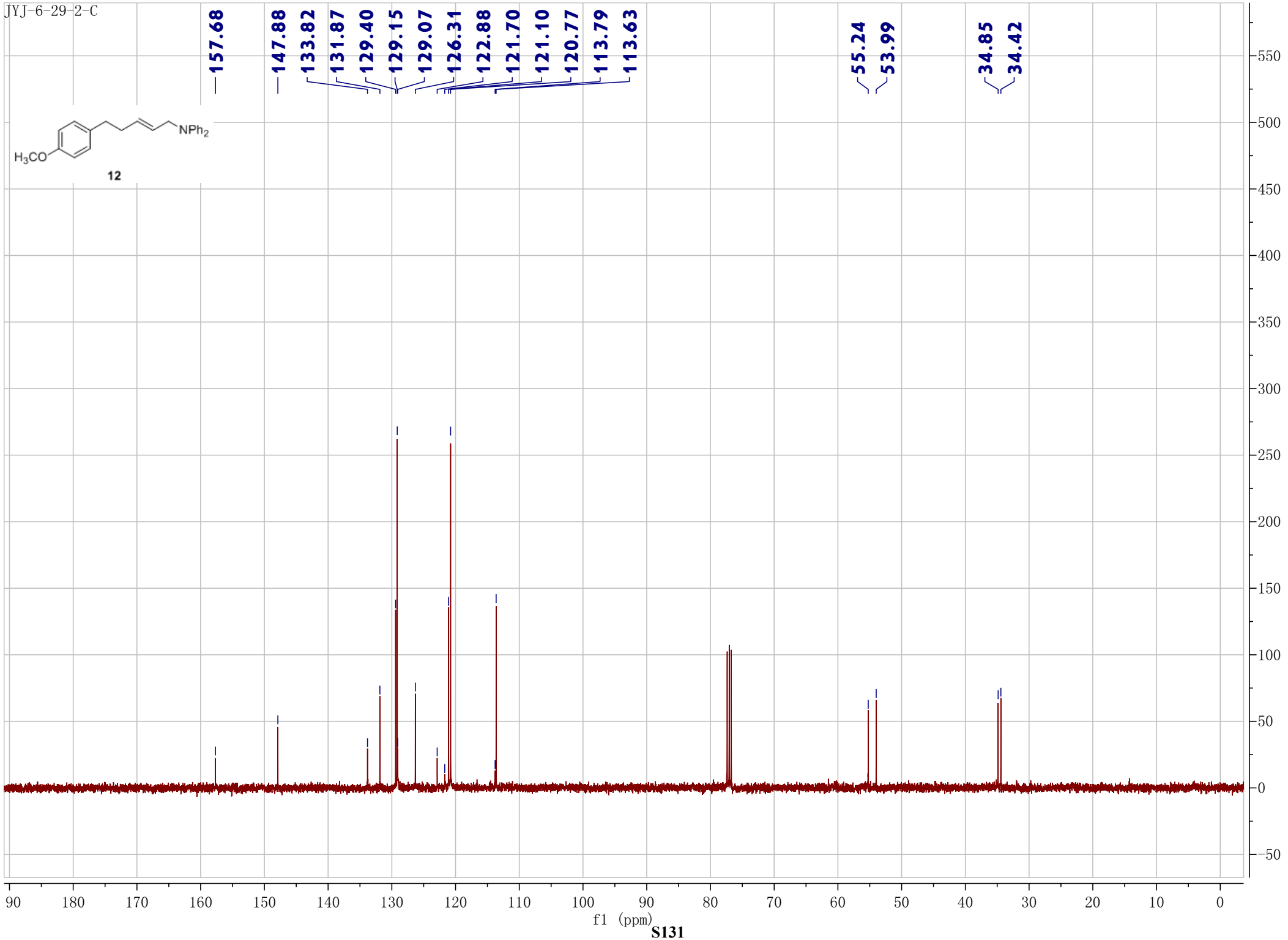

JYL-6-20-RAC-H
7.35
7.28
7.25
7.22
7.20
7.09
7.07
6.82
6.80
5.55
5.53
5.51
5.50
5.48
5.37
5.36
5.34
5.32
5.30
3.77
2.63
2.61
2.59
2.57
2.56
2.46
2.44
2.42
2.40
2.39
2.32
2.30
2.28
2.26
2.24
2.06
2.04
2.03
2.01
2.00
1.11
1.09
Ph2N
OCH3
10
10.42
2.26
2.04
1.00
1.01
3.01
3.04
1.11
2.36
1.49
3.18
f1 (ppm)


JYJ-6-20-RAC-C
176.60
157.71
142.99
134.03
131.87
129.26
127.87
113.69
55.25
37.80
37.71
35.03
34.73
17.64
Ph2N
OCH3
10
f1 (ppm)


JYJ-6-28-1-H
7.12
7.11
7.10
7.09
7.09
6.85
6.84
6.84
6.83
6.83
6.82
6.81
6.35
6.31
6.11
5.85
5.81
5.15
5.15
5.10
5.01
4.98
3.79
3.78
3.49
3.47
3.37
3.35
OCH3
11
2.02
2.20
0.52
0.84
0.61
1.84
3.00
0.70
1.12
f1 (ppm)


JYJ-6-28-1-C
157.98
136.96
133.94
131.73
131.03
129.67
129.52
129.28
117.87
115.58
113.89
113.84
55.27
38.01
33.00
OCH3
11
f1 (ppm)


JYJ-6-29-2-HHH
7.26
7.24
7.23
7.22
7.00
6.98
6.95
6.93
6.91
6.79
6.76
5.65
5.63
5.62
5.57
5.55
5.54
5.52
4.27
4.26
3.77
2.58
2.56
2.54
2.31
2.29
2.27
2.25
NPh2
H3CO
12
4.31
5.95
2.22
2.22
1.00
0.99
2.06
3.40
2.02
2.09
f1 (ppm)


JYJ-6-29-2-C
157.68
147.88
133.82
131.87
129.40
129.15
129.07
126.31
122.88
121.70
121.10
120.77
113.79
113.63
55.24
53.99
34.85
34.42
NPh2
H3CO
12
f1 (ppm)