Supporting Information

Mono-carboxylation and Intramolecular Coupling of Butenylated Arenes via Palladium-CatalyzedC-H Activation Process

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1. General Information

All commercial reagents and solvents were used without further purificaton. Synthesis of butenylated arenes **1** were undertaken as previously described.[1] The products were purified by column chromatography over silica gel. All ^{1}H NMR spectra were recorded on a Bruker spectrometer at 400 MHz. The ^{19}F NMR spectra were recorded on a Bruker spectrometer at 376 MHz. The ^{13}C NMR spectra were recorded on a Bruker spectrometer at 100 MHz. Chemical shifts (δ value) were reported in ppm down field from internal tetramethylsilane(TMS). IR spectran (Film) were recorded on a Nicolet 6700 spectrophotometer in the range of 400~4000 cm^{-1}. HRMS (ESI) Mass Spectra were recorded on a Waters LCT Premier mass spectrometerwith electrospray ionization.HRMS (EI) Mass Spectra were recorded on a Waters GCT Premier mass spectrometerwith electron impact.

2. Typical procedures

General procedure for the preparation of allylmagnesium bromide[2] :

Magnesium turnings (2.12g, 88.25 mmol) and spiked with I_2 (trace) were added to a flame dried three-neck 250 mL round-bottom flask assembled with a constant pressure funnel, which had been previously flame dried and allowed to cool under a stream of nitrogen and then, anhydrous THF (50 mL) was added in using syringe. Freshly distilled THF (50 mL) mixed with allyl bromide (4.73g, 35.3 mmol) in the constant pressure funnel were slowly added to the flask. After several minutes, the reaction mixture turned from yellow to turbid gray. Then, the round-bottom flask was cooled to 0 °C with an ice bath. After dropping was finished, the reaction was stirred for 3 hours at the room temperature.

General procedure for the preparation of butenylated arenes 1a-j [1] (1a as an example):

Benzyl bromide (1.0 equiv) and 20 mL anhydrous THF were added to a flame dried 100 mL round-bottom flash assembled with a constant pressure funnel which had been previously flame dried. Allylmagnesium bromide (2.0 equiv) in the constant pressure funnel were added dropwise. The reaction was stirred for 4h at room temperature and then quenched with saturated aqueous NH_4Cl. The aqueous layer was extracted with CH_2Cl_2 and the combined organics were dried over $MgSO_4$, filtered through celite and concentrated under reduced pressure. The crude product was purified by flash chromatography(eluent:hexane), affording the desired product as a clear,

colorless oil.

General procedure for the synthesis of 2-tetralyl carboxylate esters (3a as an example):`

A round bottom flask was charged with the corresponding acetic acid (60 mmol, 3.6g) and dissolved with MeCN 4mL. Then, $Pd(OAc)_2$ (5 mol %), Selectfluor (2.8 equiv, 0.99g) and 4-phenyl-1-butene(1 mmol, 0.132g) were subsequently added to the reaction vessel at room temperature. The reaction was stirred overnight at room temperature and then 2mL water was add to the solution.The aqueous layer was extracted with CH_2Cl_2 (3mL×3) and the combined organics were concentrated under reduced pressure, the resulting crude product was purified by conventional flash chromatography (petroleum ether/AcOEt 50/1).

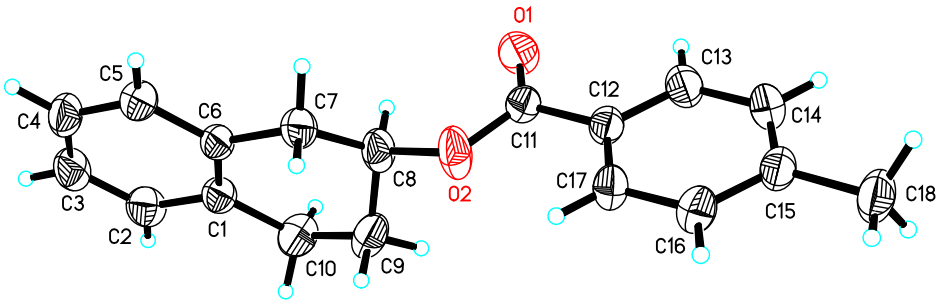
General procedure for screening the optimized conditions

A round bottom flask was charged with a certain amount of acetic acid and dissolved with solvent 4 mL. Then, $Pd(OAc)_2$, Selectfluor and 4-phenyl-1-butene (1 mmol, 0.132 g) were subsequently added to the reaction vessel and stirred overnight. Entry 1, 2, 5 generate no product which were determined by ^1H NMR. The yields of 3, 4, 7, 8 were determined by ^1H NMR analysis with mesitylene as internal standard. The reactions of entry 6, 9-25 were stirred overnight and then 2 mL water was add to the solution. The aqueous layer was extracted with CH_2Cl_2 (3 mL×3) and the combined organics were concentrated under reduced pressure, the resulting crude product was purified by conventional flash chromatography (petroleum ether/AcOEt 50/1).

Entry	**2a**（equiv）	Selectfluor（equiv）	$Pd(OAc)_2$（mol %）	Solvent	Temp (^oC)	Yield (%)
1	70	2.5	10	EA	rt	n.r
2	70	2.5	10	DCM	rt	n.r
3	70	2.5	10	acetone	rt	4.6
4	70	2.5	10	toluene	rt	5.3
5	70	2.5	10	MeOH	rt	n.r
6	70	2.5	10	MeCN	rt	68
7	2	2.5	10	MeCN	rt	1.2
8	10	2.5	10	MeCN	rt	9.5
9	40	2.5	10	MeCN	rt	48
10	60	2.5	10	MeCN	rt	69
11	90	2.5	10	MeCN	rt	51
12	60	2.5	1	MeCN	rt	41
13	60	2.5	5	MeCN	rt	70
14	60	2.5	15	MeCN	rt	66
15	60	2.5	5	MeCN	0	53

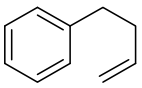
16	60	2.5	5	MeCN	40	40
17	60	2.5	5	MeCN	50	56
18	60	2.5	5	MeCN	60	28
19	60	1.5	5	MeCN	r.t	44
20	60	2.0	5	MeCN	r.t	62
21	60	2.4	5	MeCN	r.t	65
22	60	2.7	5	MeCN	r.t	80
23	60	2.8	5	MeCN	r.t	83
24	60	2.9	5	MeCN	r.t	82
25	60	3.0	5	MeCN	r.t	79

3. X-ray diffraction parameters and data for 3h (cd: 214686)



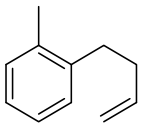
Identification code	cd214686	
Empirical formula	C18 H18 O2	
Formula weight	266.32	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21/c	
Unit cell dimensions	a = 14.0260(15) Å	a= 90°.
	b = 8.7035(9) Å	b= 104.008(2)°.
	c = 12.0599(12) Å	g = 90°.
Volume	1428.4(3) $Å^3$	
Z	4	
Density (calculated)	1.238 Mg/m^3	
Absorption coefficient	0.079 mm^{-1}	
F(000)	568	
Crystal size	0.211 x 0.165 x 0.123 mm^3	
Theta range for data collection	2.778 to 25.999°.	
Index ranges	-15<=h<=17, -9<=k<=10, -14<=l<=14	
Reflections collected	8430	
Independent reflections	2807 [R(int) = 0.0302]	
Completeness to theta = 25.242°	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7457 and 0.6495	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2807 / 0 / 182	
Goodness-of-fit on F^2	1.065	
Final R indices [I>2sigma(I)]	R1 = 0.0500, wR2 = 0.1330	
R indices (all data)	R1 = 0.0777, wR2 = 0.1469	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.145 and -0.141 $e.Å^{-3}$	

4. Analytical Data for the Products



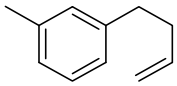
(1a)

4-Phenyl-1-butene 1a (2.3 g, 97% yield): A colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 7.29-7.25 (m, 2H), 7.19-7.15 (m, 3H), 5.89-5.80 (m, 1H), 5.06-4.96 (m, 2H), 2.70 (t, J = 7.6 Hz, 2H), 2.40-2.33 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 141.98, 138.21, 128.56, 128.42, 125.94, 115.04, 35.67, 35.52.



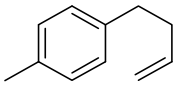
(1b)

4-(2-Methylphenyl)-1-butene 1b(2.4 g, 94% yield): A colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 7.13-7.07 (m, 4H), 5.91-5.83 (m, 1H), 5.08-4.97 (m, 2H), 2.71-2.64 (m, 2H), 2.34-2.28 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 140.09, 138.33, 135.91, 130.23, 128.86, 126.06, 126.01, 114.91, 24.44, 32.83, 19.38.



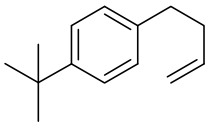
(1c)

4-(3-Methylphenyl)-1-butene 1c(2.3 g, 92% yield): A colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 7.15-7.13 (m, 1H), 6.99 (s, 3H), 5.89-5.80 (m, 1H), 5.06-4.95 (m, 2H), 2.66-2.63 (m, 2H), 2.35-2.30 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 141.90, 138.27, 137.86, 129.35, 128.30, 126.67, 125.54, 114.91, 35.71, 35.46, 21.50.



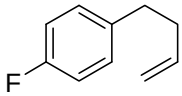
(1d)

4-(4-Methylphenyl)-1-butene 1d(2.3 g, 90% yield): A colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 7.18-7.12 (m, 4H), 5.96-5.87 (m, 1H), 5.13-5.02 (m, 2H), 2.74 (t, J = 7.6 Hz, 2H), 2.45-2.39 (m, 2H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 138.91, 138.33, 135.32, 129.11, 128.43, 114.94, 35.79, 35.08, 21.13.



(1e)

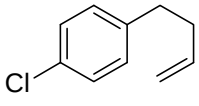
4-(4- tert-Butylphenyl)-1-butene 1e (3.3 g, 94% yield): A colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 7.30 (d, J = 8.4 Hz, 2H), 7.12 (d, J = 8.0 Hz, 2H), 5.90-5.81 (m, 1H), 5.08-4.95 (m, 2H), 2.67 (t, J = 7.2 Hz, 2H), 2.39-2.33 (m, 2H), 1.30 (s, 9 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 148.68, 138.94, 138.43, 128.20, 125.32, 114.91, 35.65, 34.99, 34.48, 31.58.



(1f)

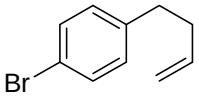
4-(4-Fluorophenyl)-1-butene 1f(2.5 g, 93% yield): A colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 7.20-7.15 (m, 2H), 7.04-6.98 (m, 2H), 5.93-5.85 (m, 1H), 5.12-5.02 (m, 2H), 2.73 (t, J = 7.2 Hz,

2H), 2.43-2.37 (m, 2H); ^{19}F NMR (367 MHz, $CDCl_3$) δ : -117.71 (s, 1F);^{13}C NMR (100 MHz, $CDCl_3$) δ : 161.41 (d, J = 241.0 Hz), 137.89, 137.54 (d, J = 2.7 Hz), 129.87 (d, J = 7.2 Hz), 115.24 (d, J = 4.2 Hz), 115.01, 35.75, 34.66.



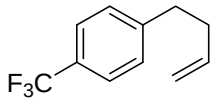
(1g)

4-(4-Chlorophenyl)-1-butene 1g (2.5 g, 85% yield): A colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 7.21 (d, J = 8.4 Hz, 2H), 7.07 (d, J = 8.4 Hz, 2H), 5.84-5.76 (m, 1H), 5.04-4.95 (m, 2H), 2.65 (t, J = 7.6 Hz, 2H), 2.35-2.29 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 140.30, 137.66, 131.61, 129.87, 128.45, 115.35, 35.43, 34.76.



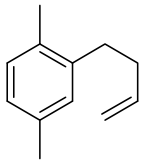
(1h)

4-(4-Bromophenyl)-1-butene 1h (3.3 g, 88% yield): A colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 7.43 (d, J = 8.4 Hz, 2H), 7.09 (d, J = 8.4 Hz, 2H), 5.90-5.83 (m, 1H), 5.11-5.02 (m, 2H), 2.70 (t, J = 7.6 Hz, 2H), 2.45-2.39 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 140.80, 137.64, 131.40, 130.29, 119.64, 115.39, 35.36, 34.81.



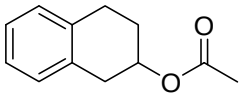
(1i)

4-(4-Trifluoromethyphenyl)-1-butene 1i (3.1 g, 87% yield): A colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 7.57 (d, J = 8.4 Hz, 2H), 7.32 (d, J = 8.0 Hz, 2H), 5.92-5.81 (m, 1H), 5.11-5.02 (m, 2H), 2.80 (t, J = 7.6 Hz, 2H), 2.45-2.39 (m, 2H); ^{19}F NMR (367 MHz, $CDCl_3$) δ : -62.84 (s, 1F); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 146.09, 137.50, 128.90, 128.38 (q, J = 32.1, 64.2 Hz),125.36 (q, J = 3.8, 7.6 Hz), 115.59, 35.30, 35.25.



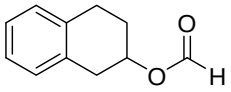
(1j)

2-(but-3-enyl)-1,4-dimethylbenzene 1j (2.3 g, 82% yield): A colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 7.19-7.07 (m, 3H), 6.10-6.02 (m, 1H), 5.25-5.13 (m, 2H), 2.82-2.77 (m, 2H), 2.45-2.41 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 139.96, 138.46, 135.32, 132.75, 130.17, 129.70, 126.74, 114.81, 34.59, 32.89, 21.08, 18.91.



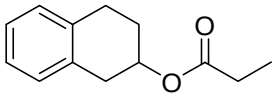
(3a)

1,2,3,4-tetrahydronaphthalen-2-yl acetate 3a(157.8 mg, 83% yield): A lightyellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 7.12-7.03 (m, 4H), 5.22-5.16 (m, 1H), 3.09 (dd, J = 4.8, 16.8 Hz, 1H), 2.95-2.80 (m, 3H), 2.03 (s, 3H), 2.07-1.86 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 170.69, 135.50, 133.62, 129.31, 128.59, 126.03, 125.92, 69.70, 34.52, 27.76, 26.41, 21.36; IR (Film, cm^{-1}): 3062, 3021, 2960, 2849, 1736, 1604, 1582, 1495, 1453, 1438, 1247, 1109, 1034, 802, 748; HRMS-ESI (m/z) (M+Na) *calcd.* for ($C_{12}H_{14}NaO_2$): 213.0891, *found* 213.0893.



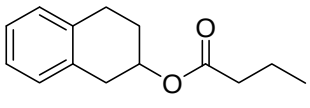
(3b)

1,2,3,4-tetrahydronaphthalen-2-yl formate 3b(149.7 mg, 85% yield): A lightyellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 8.03 (s, 1H), 7.13-7.03 (m, 4H), 5.36-5.30 (m, 1H), 3.10 (dd, J = 5.2, 16.8 Hz, 1H), 2.96-2.81 (m, 3H), 2.09-1.93 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 160.72, 135.28, 133.17, 129.31, 128.65, 126.21, 126.05, 69.60, 34.42, 27.63, 26.14; IR (Film, cm^{-1}): 3062, 3021, 2930, 2848, 1722, 1604, 1582, 1495, 1453, 1438, 1177, 1111, 747; HRMS-EI (m/z) *calcd.* for ($C_{11}H_{12}O_2$-CH_2O_2): 130.0783, *found* 130.0775.



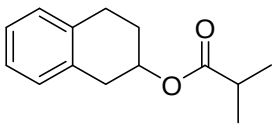
(3c)

1,2,3,4-tetrahydronaphthalen-2-yl propionate 3c (159.2 mg, 78% yield): A light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 7.11-7.02 (m, 4H), 5.23-5.16 (m, 1H), 3.09 (dd, J = 5.2, 16.4 Hz, 1H), 2.94-2.79 (m, 3H), 2.30 (q, J = 7.6, 15.2 Hz, 2H), 2.06-1.88 (m, 2H), 1.12 (t, J = 7.6, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 174.04, 135.51, 133.67, 129.27, 128.56, 125.99, 125.89, 69.47, 34.56, 27.82, 26.44, 9.15; IR (Film, cm^{-1}): 3021, 2940, 2850, 1735, 1496, 1458, 1438, 1379, 1188, , 1110, 748; HRMS-EI (m/z) *calcd.* for ($C_{13}H_{16}O_2$): 204.115, *found* 204.1151.



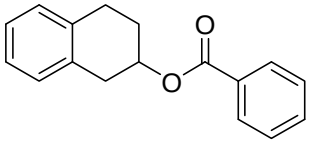
(3d)

1,2,3,4-tetrahydronaphthalen-2-yl butyrate 3d (141.8 mg, 65% yield): A light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 7.13-7.03 (m, 4H), 5.25-5.18 (m, 1H), 3.10 (dd, J = 5.2, 16.4 Hz, 1H), 2.96-2.77 (m, 3H), 2.27 (t, J = 7.2, 14.8 Hz, 2H), 2.07-1.89 (m, 2H), 1.64 (m, 2H), 0.93 (t, J = 7.6, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 173.38, 135.62, 133.78, 129.36, 128.64, 126.07, 125.97, 69.47, 36.56, 34.66, 27.92, 26.53, 18.59, 13.70; IR (Film, cm^{-1}): 3062, 3020, 2963, 2875, 2848, 1731, 1605, 1582, 1495, 1455, 1437, 1382, 1258, 1108, 1089, 747; HRMS-EI (m/z) *calcd.* for ($C_{14}H_{18}O_2$-$C_4H_8O_2$): 130.0783, *found* 130.0775.



(3e)

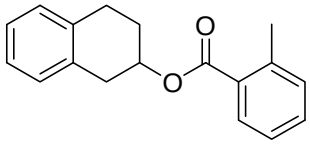
1,2,3,4-tetrahydronaphthalen-2-yl isobutyrate 3e (130.9 mg, 60% yield): A light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 7.12-7.03 (m, 4H), 5.22-5.16 (m, 1H), 3.09 (dd, J = 5.2, 16.8 Hz, 1H), 2.95-2.81 (m, 3H), 2.57-2.46 (m, 1H), 2.06-1.88 (m, 2H), 1.15 (d, J = 6.8, 3H), 1.14 (d, J = 6.8, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 176.78, 135.58, 133.75, 129.32, 128.60, 126.02, 125.93, 69.33, 34.56, 34.15, 27.83, 26.49, 19.07, 18.98; IR (Film, cm^{-1}): 3062, 3020, 2972, 2875, 2848, 1731, 1495, 1468, 1455, 1387, 1193, 1158, 747; HRMS-ESI (m/z) (M+Na) *calcd.* for ($C_{14}H_{18}NaO_2$): 241.1204, *found* 241.1207.



(3f)

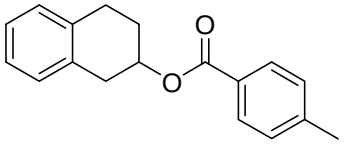
1,2,3,4-tetrahydronaphthalen-2-yl benzoate 3f (121.0 mg, 48% yield): A light yellow oil; ^{1}H

NMR (400 MHz, $CDCl_3$) δ : 8.04 (d, J = 7.6 Hz, 2H), 7.55 (t, J = 7.6 Hz, 1H), 7.43 (t, J = 7.6 Hz, 2H), 7.18-7.11 (m, 4H), 5.53-5.46 (m, 1H), 3.26 (dd, J = 5.2, 16.8 Hz, 1H), 3.09-3.01 (m, 2H), 2.97-2.89 (m, 1H), 2.23-2.05 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 166.28, 135.70, 133.78, 132.99, 130.73, 129.72, 129.49, 128.77, 128.43, 126.18, 126.10, 70.42, 34.74, 28.04, 26.57; IR (Film, cm^{-1}): 3062, 3022, 2926, 2892, 2845, 1708, 1600, 1582, 1492, 1449, 1272, 1114, 751, 709; HRMS-ESI (m/z) (M+Na) *calcd.* for ($C_{17}H_{16}NaO_2$): 275.1048, *found* 275.1049.



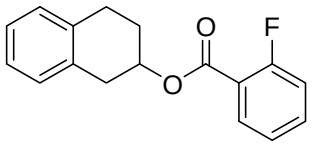
(3g)

1,2,3,4-tetrahydronaphthalen-2-yl 2-methylbenzoate 3g (125.1 mg, 47% yield): A light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 7.88-7.85 (m, 1H), 7.39-7.35 (m, 1H), 7.24-7.19 (m, 2H), 7.16-7.08 (m, 4H), 5.49-5.43 (m, 1H), 3.23 (dd, J = 5.2, 16.8 Hz, 1H), 3.06-2.98 (m, 2H), 2.96-2.87 (m, 1H), 2.56 (s, 3H), 2.22-2.04 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 167.42, 140.11, 135.68, 133.82, 131.99, 131.77, 130.72, 130.18, 129.48, 128.76, 126.18, 126.08, 125.81, 70.28, 34.80, 28.03, 26.62, 21.87; IR (Film, cm^{-1}): 3062, 3021, 2928, 2848, 1716, 1602, 1576, 1493, 1456, 1438, 1380, 1259, 1131, 1082, 787, 740; HRMS-ESI (m/z) (M+Na) *calcd.* for ($C_{18}H_{18}NaO_2$): 289.1204, *found* 289.1205.



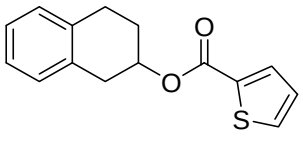
(3h)

1,2,3,4-tetrahydronaphthalen-2-yl 4-methylbenzoate 3h (133.1 mg, 50% yield): A white solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 7.91 (d, J = 8.0 Hz, 2H), 7.21 (d, J = 8.0 Hz, 2H), 7.16-7.09 (m, 4H), 5.49-5.42 (m, 1H), 3.23 (dd, J = 4.8, 16.4 Hz, 1H), 3.07-3.00 (m, 2H), 2.95-2.87 (m, 1H), 2.40 (s, 3H), 2.20-2.04 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 166.40, 143.66, 135.78, 133.90, 129.77, 129.52, 129.16, 128.78, 128.02, 126.16, 126.09, 70.21, 34.79, 28.08, 26.63, 21.80; IR (Film, cm^{-1}): 3063, 3017, 2926, 2852, 1704, 1608, 1574, 1486, 1452, 1370, 1272, 1105, 844, 754, 691; HRMS-ESI (m/z) (M+Na) *calcd.* for ($C_{18}H_{18}NaO_2$): 289.1204, *found* 289.1204.



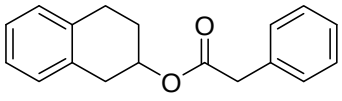
(3i)

1,2,3,4-tetrahydronaphthalen-2-yl 2-fluorobenzoate 3i (124.2 mg, 46% yield): A light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 7.81 (d, J = 8.0 Hz, 1H), 7.71-7.67 (m, 1H), 7.42-7.36 (m, 1H), 7.27-7.21 (m, 1H), 7.17-7.08 (m, 4H), 5.51-5.44 (m, 1H), 3.24 (dd, J = 4.8, 16.4 Hz, 1H), 3.07-2.99 (m, 2H), 2.96-2.87 (m, 1H), 2.21-2.04 (m, 2H); ^{19}F NMR (367 MHz, $CDCl_3$) δ: -112.44 (s, 1F); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 165.14, 162.65 (d, J = 245.5 Hz), 135.59, 133.58, 132.90 (d, J = 7.3 Hz), 130.08 (d, J = 7.6 Hz), 129.49, 128.80, 126.27, 126.16, 125.47 (d, J = 2.9 Hz), 120.07 (d, J = 21.1 Hz), 116.61 (d, J = 22.8 Hz), 70.97, 34.68, 28.00, 26.54; IR (Film, cm^{-1}): 3077, 3021, 2928, 2850, 1720, 1592, 1486, 1446, 1282, 1204, 1096, 755; HRMS-EI (m/z) *calcd.* for ($C_{17}H_{15}FO_2$-$C_7H_5FO_2$): 130.0783, *found* 130.0779.



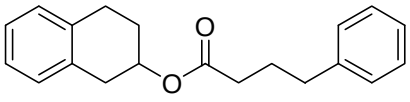
(3j)

1,2,3,4-tetrahydronaphthalen-2-yl thiophene-2-carboxylate 3j (183.2 mg, 71% yield): A light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 7.79 (d, J = 3.6 Hz, 1H), 7.54 (d, J = 4.8 Hz, 1H), 7.17-7.07 (m, 5H), 5.47-5.40 (m, 1H), 3.24 (dd, J = 5.2, 16.8 Hz, 1H), 3.07-3.00 (m, 2H), 2.96-2.87 (m, 1H), 2.10-2.04 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 161.99, 135.64, 134.41, 133.67, 133.44, 132.42, 129.46, 128.73, 127.80, 126.19, 126.09, 70.85, 34.71, 28.08, 26.63; IR (Film, cm^{-1}): 3061, 3020, 2859, 2925, 2854, 1699, 1525, 1492, 1423, 1415, 1261, 1093, 750, 729; HRMS-EI (m/z) *calcd.* for ($C_{15}H_{14}O_2S$): 258.0715, *found* 258.0708



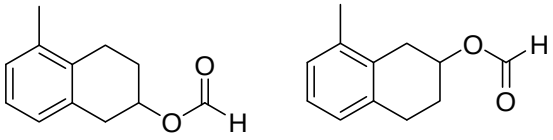
(3k)

1,2,3,4-tetrahydronaphthalen-2-yl 2-phenylacetate 3k (218.2 mg, 82% yield): A light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 7.31-7.22 (m, 5H), 7.13-7.02 (m, 4H), 5.25-5.18 (m, 1H), 3.60 (s, 2H), 3.09 (dd, J = 4.8, 16.4 Hz, 1H), 2.92-2.76 (m, 3H), 2.05-1.88 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 171.35, 135.61, 134.26, 133.67, 129.41, 129.30, 128.69, 128.62, 127.11, 126.14, 126.02, 70.28, 41.75, 34.55, 27.78, 26.41; IR (Film, cm^{-1}): 3062, 3028, 2930, 2847, 1731, 1603, 1583, 1495, 1454, 1436, 1259, 1158, 1049, 747, 709; HRMS-ESI (m/z) (M+Na) *calcd.* for ($C_{18}H_{18}NaO_2$): 289.1204, *found* 289.1205.



(3l)

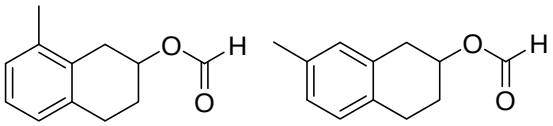
1,2,3,4-tetrahydronaphthalen-2-yl 4-phenylbutanoate 3l (120.6 mg, 41% yield): A light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 7.25 (t, J = 7.6 Hz, 2H), 7.18-7.03 (m, 7H), 5.24-5.18 (m, 1H), 3.09 (dd, J = 4.8, 16.8 Hz, 1H), 2.94-2.80 (m, 3H), 2.61 (t, J = 7.6 Hz, 2H), 2.30 (t, J = 7.6 Hz, 2H), 2.06-1.89 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 173.15, 141.45, 135.57, 133.68, 129.38, 128.66, 128.54, 128.42, 126.09, 126.00, 125.99, 69.60, 35.16, 34.61, 33.96, 27.85, 26.69, 26.43; IR (Film, cm^{-1}): 3061, 3024, 2935, 2861, 1730, 1603, 1582, 1495, 1454, 1438, 1381, 1248, 1127, 747, 700; HRMS-ESI (m/z) (M+Na) *calcd.* for ($C_{20}H_{22}NaO_2$): 317.1517, *found* 317.1518.



(4b& 5b)

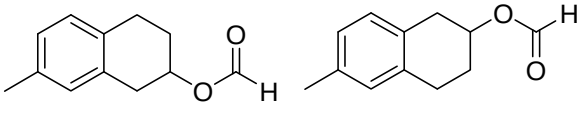
4b& 5b (**4b** + **5b** : 152.0 mg, 80% yield): A light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : Higher proton peak group for one isomer: 8.12 (s, 1H), 7.14-6.98 (m, 3H), 5.48-5.37 (m, 1H), 3.18 (dd, J = 5.2, 17.2 Hz, 1H), 3.03-2.73 (m, 3H), 2.29 (s, 3H), 2.20-1.97 (m, 2H); Lower proton peak group for the other isomer: 8.14 (s, 1H), 7.13-6.96 (m, 3H), 5.41-5.35 (m, 1H), 3.08 (dd, J = 4.4, 16.4 Hz, 1H), 3.01-2.72 (m, 3H), 2.28 (s, 3H), 2.19-1.98 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ : Higher ^{13}C peak group for one isomer: 160.95, 136.31, 133.78, 133.04, 127.80, 127.19, 125.91, 69.56, 34.90, 27.65, 23.85, 19.59; Lower ^{13}C peak group for the other isomer: 160.95, 136.78, 135.22, 131.88, 127.58, 126.45, 125.92,70.20, 32.15, 27.45, 26.68, 19.56; IR (Film, cm^{-1}): 3068, 3021, 2931, 1722, 1589, 1466, 1439, 1378, 1179, 1098, 770; HRMS-ESI (m/z) (M+Na) *calcd.* for

($C_{12}H_{14}NaO_2$): 213.0891, *found* 213.0893.



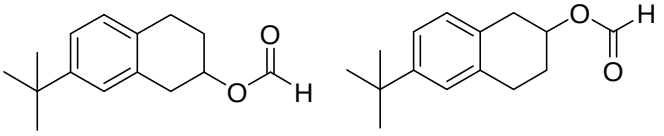
(4c & 5c)

4c or 5c (4c + 5c : 153.9 mg, 81% yield)**:** A light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : Higher proton peak group for one isomer: 8.14 (s, 1H), 7.15-6.95 (m, 3H), 5.49-5.42 (m, 1H), 3.08 (dd, J = 5.6, 17.2 Hz, 1H), 3.04-2.76 (m, 3H), 2.28 (s, 3H), 2.16-1.99 (m, 2H); Lower proton peak group for the other isomer: 8.12 (s, 1H), 7.15-6.95 (m, 3H), 5.44-5.37 (m, 1H), 3.15 (dd, J = 4.8, 16.4 Hz, 1H), 3.04-2.76 (m, 3H), 2.36 (s, 3H), 2.16-1.99 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : Higher ^{13}C peak group for one isomer: 160.76, 136.74, 135.06, 131.88, 127.56, 126.43, 125.89,70.09, 32.14, 27.45, 26.13, 19.53; Lower ^{13}C peak group for the other isomer: 160.76, 135.71, 135.20, 130.06, 129.21, 126.95, 69.82, 34.10, 27.73, 26.13, 20.98; IR (Film, cm^{-1}): 3003, 2927, 2850, 1722, 1616, 1580, 1506, 1439, 1375, 1180, 1122, 808; HRMS-ESI (m/z) (M+Na) *calcd.* for ($C_{12}H_{14}NaO_2$): 213.0891, *found* 213.0890.



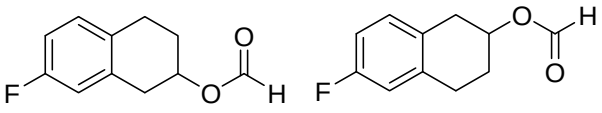
(4d& 5d)

4d or 5d (4d + 5d : 165.3 mg, 87% yield): A light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : Higher proton peak group for one isomer: 8.10 (s, 1H), 7.05-6.92 (m, 3H), 5.41-5.35 (m, 1H), 3.12 (dd, J = 5.2, 16.8 Hz, 1H), 3.00-2.81 (m, 3H), 2.33 (s, 3H), 2.13-1.97 (m, 2H); Lower proton peak group for the other isomer: 8.10 (s, 1H), 7.05-6.93 (m, 3H), 5.41-5.35 (m, 1H), 3.12 (dd, J = 5.2, 16.8 Hz, 1H), 3.00-2.81 (m, 3H), 2.33 (s, 3H), 2.13-1.97 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : Higher ^{13}C peak group for one isomer: 160.83, 135.82, 135.14, 130.12, 129.28, 127.02, 69.90, 34.17, 27.79, 26.18, 21.04; Lower ^{13}C peak group for the other isomer: 160.83, 135.62, 133.05, 132.27, 129.90, 128.61, 127.19, 69.85, 34.49, 27.86, 25.81, 21.02; IR (Film, cm^{-1}): 3003, 2927, 2850, 1722, 1616, 1580, 1506, 1439, 1375, 1180, 1122, 808; HRMS-ESI (m/z) (M+Na) *calcd.* for ($C_{12}H_{14}NaO_2$): 213.0891, *found* 213.0890.



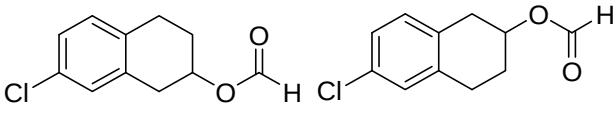
(4e& 5e)

4e or 5e (4e + 5e : 194.9 mg, 84% yield): A light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : Higher proton peak group for one isomer: 8.05 (s, 1H), 7.19-6.99 (m, 3H), 5.37-5.31 (m, 1H), 3.09 (dd, J = 4.4, 16.8 Hz, 1H), 2.99-2.77 (m, 3H), 2.10-1.94(m, 2H), 1.30 (s, 9H); Lower proton peak group for the other isomer: 8.05 (s, 1H), 7.19-6.99 (m, 3H), 5.37-5.31 (m, 1H), 3.13 (dd, J = 4.8, 12 Hz, 1H), 2.99-2.77 (m, 3H), 2.10-1.94(m, 2H), 1.29 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : Higher ^{13}C peak group for one isomer: 160.79, 149.20, 134.77, 130.30, 129.08, 125.47, 123.34, 69.90, 34.79, 34.10, 31.46, 27.86, 26.53; Lower ^{13}C peak group for the other isomer: 160.80, 149.03, 132.71, 132.37, 128.43, 126.13, 123.50, 69.90, 34.39, 34.37, 31.46, 27.82, 25.75; IR (Film, cm^{-1}): 3052, 2960, 2868, 1723, 1613, 1575, 1505, 1477, 1393, 1364, 1179, 1138, 838, 817; HRMS-ESI (m/z) (M+Na) *calcd.* for ($C_{15}H_{20}NaO_2$): 255.1361, *found* 255.1363.



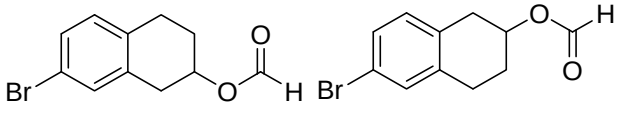
(4f& 5f)

4f or 5f (4f + 5f : 108.6 mg, 56% yield): A light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : Higher proton peak group for one isomer: 8.07 (s, 1H), 7.08-7.00 (m, 1H), 6.87-6.75 (m, 2H), 5.39-5.32 (m, 1H), 3.09 (dd, *J* = 5.2, 16.8 Hz, 1H), 2.99-2.77 (m, 3H), 2.10-1.96(m, 2H); Lower proton peak group for the other isomer: 8.07 (s, 1H), 7.08-7.00 (m, 1H), 6.87-6.75 (m, 2H), 5.39-5.32 (m, 1H), 3.11 (dd, *J* = 5.2, 16.8 Hz, 1H), 2.99-2.77 (m, 3H), 2.10-1.96(m, 2H); ^{19}F NMR (367 MHz, $CDCl_3$) δ : -117.45 (d, *J* = 77.8 Hz, 1F);^{13}C NMR (100 MHz, $CDCl_3$) δ : Higher ^{13}C peak group for one isomer: 161.34 (d, *J* = 242.7 Hz), 160.76, 137.38 (d, *J* = 7.3 Hz), 130.35 (d, *J* = 8 Hz), 128.73 (d, *J* = 2.9 Hz), 114.96 (d, *J* = 20.4 Hz), 113.32 (d, *J* = 21.4 Hz), 69.46, 33.85, 27.33, 26.26 (d, *J* = 1.2 Hz); Lower ^{13}C peak group for the other isomer: 161.23 (d, *J* = 242.1) 160.76, 135.20 (d, *J* = 7.3 Hz), 130.93 (d, *J* = 2.9 Hz), 130.09 (d, *J* = 8 Hz), 115.41 (d, *J* = 20.6 Hz), 113.45 (d, *J* = 21.1 Hz), 69.16, 34.56 (d, *J* = 1.2 Hz), 27.65, 25.41; IR (Film, cm^{-1}): 3026, 2933, 2850, 1726, 1616, 1501, 1438, 1261, 1177, 838, 807; HRMS-EI (m/z) *calcd.* for ($C_{11}H_{11}FO_2$): 194.0743, *found* 194.0744.



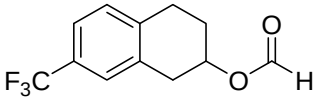
(4g& 5g)

4g or 5g (4g + 5g : 113.4 mg, 54% yield): A light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : Higher proton peak group for one isomer: 8.05 (s, 1H), 7.09-6.97 (m, 3H), 5.36-5.30 (m, 1H), 3.07 (dd, *J* = 4.8, 16.8 Hz, 1H), 2.96-2.75 (m, 3H), 2.08-1.94 (m, 2H); Lower proton peak group for the other isomer: 8.05 (s, 1H), 7.09-6.97 (m, 3H), 5.36-5.30 (m, 1H), 3.07 (dd, *J* = 4.8, 16.8 Hz, 1H), 2.96-2.75 (m, 3H), 2.08-1.94 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : Higher ^{13}C peak group for one isomer: 160.63, 137.17, 133.80, 131.62, 130.58, 128.44, 126.24, 69.14, 33.88, 27.22, 25.89; Lower ^{13}C peak group for the other isomer: 160.63, 135.06, 131.73, 131.51, 129.98, 128.98, 126.38, 68.96, 34.21, 27.37, 25.46; IR (Film, cm^{-1}): 3050, 3020, 2932, 2848, 1722, 1598, 1487, 1460, 1437, 1177, 1121, 808; HRMS-EI (m/z) *calcd.* for ($C_{11}H_{11}ClO_2$): 210.0448, *found* 210.0447.



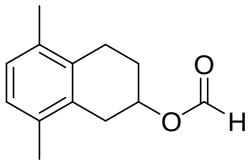
(4h& 5h)

4h or 5h (4h + 5h : 127.0 mg, 50% yield): A light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : Higher proton peak group for one isomer: 8.05 (s, 1H), 7.25-7.20 (m, 2H), 6.98-6.91 (m, 1H), 5.37-5.30 (m, 1H), 3.05 (dd, *J* = 5.2, 16.4 Hz, 1H), 2.97-2.74 (m, 3H), 2.07-1.94 (m, 2H); Lower proton peak group for the other isomer: 8.05 (s, 1H), 7.25-7.20 (m, 2H), 6.98-6.91 (m, 1H), 5.37-5.30 (m, 1H), 3.08 (dd, *J* = 4.8, 16.8 Hz, 1H), 2.97-2.74 (m, 3H), 2.07-1.94 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : Higher ^{13}C peak group for one isomer: 160.65, 137.62, 131.97, 131.43, 130.94, 129.16, 119.84, 69.08, 33.97, 27.23, 25.84; Lower ^{13}C peak group for the other isomer: 160.64, 135.51, 134.35, 132.17, 130.34, 129.31, 119.55, 68.94, 34.16, 27.33, 25.54; IR (Film, cm^{-1}): 3048, 3015, 2931, 2847, 1722, 1592, 1484, 1460, 1436, 1176, 1077, 860, 806; HRMS-EI (m/z) *calcd.* for ($C_{11}H_{11}BrO_2$): 253.9942, *found* 253.9946, 255.9936.



(4i)

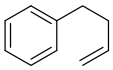
6-(trifluoromethyl)-1,2,3,4-tetrahydronaphthalen-3-yl formate 4i (90.3 mg, 37% yield): A light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 8.07 (s, 1H), 7.38 (d, J = 4.0 Hz, 1H), 7.33 (s, 1H), 7.22 (d, J = 4 Hz, 1H), 5.43-5.37 (m, 1H), 3.17 (dd, J = 4.8 Hz, 16.8 Hz, 1H), 3.06-2.85 (m, 3H), 2.10-2.04 (m, 2H); ^{19}F NMR (367 MHz, $CDCl_3$) δ : -62.39 (s, 1F);^{13}C NMR (100 MHz, $CDCl_3$) δ : 160.70, 139.60, 134.04, 129.33,128.58 (q, J = 32.0, 64.1 Hz), 126.35 (q, J = 3.8, 7.7 Hz), 123.06 (q, J = 3.7, 7.4 Hz), 68.91, 34.41, 27.26, 25.99; IR (Film, cm^{-1}): 2935, 2851, 1725, 1621, 1586, 1503, 1429, 1332, 1299, 1180, 1122, 1074, 868, 841; HRMS-EI (m/z) *calcd.* for ($C_{12}H_{11}F_3O_2$-CH_2O_2): 198.0656, *found* 198.0656.



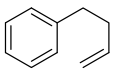
(4j)

1,2,3,4-tetrahydro-5,8-dimethylnaphthalen-2-yl formate 4j (91.8 mg, 45% yield): A light yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 8.13 (s, 1H), 6.99 (s, 2H), 5.45-5.38 (m, 1H), 3.05 (dd, J = 5.2, 17.2 Hz, 1H), 2.92-2.71 (m, 3H), 2.26 (s, 3H), 2.24 (s, 3H), 2.18-2.01 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ : 160.88, 134.28, 133.74, 131.79, 127.44, 127.39, 69.89, 32.56, 27.34, 24.28, 19.56, 19.53; IR (Film, cm^{-1}): 3066, 3013, 2931, 2867, 1722, 1600, 1502, 1477, 1462, 1379, 1181, 1055, 834, 810; HRMS-ESI (m/z) (M+Na) *calcd.* for ($C_{13}H_{16}NaO_2$): 227.1048, *found* 227.1046.

5. ^{1}H and ^{13}C NMR spectral data



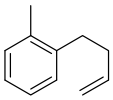
1 a



1 a

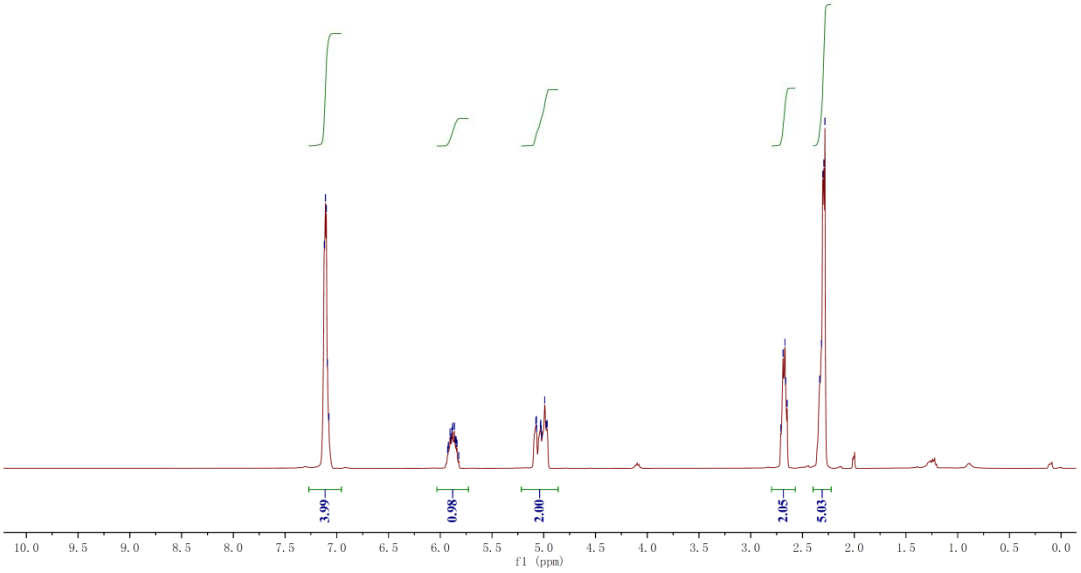
1.

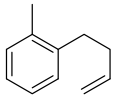
2.



1 b

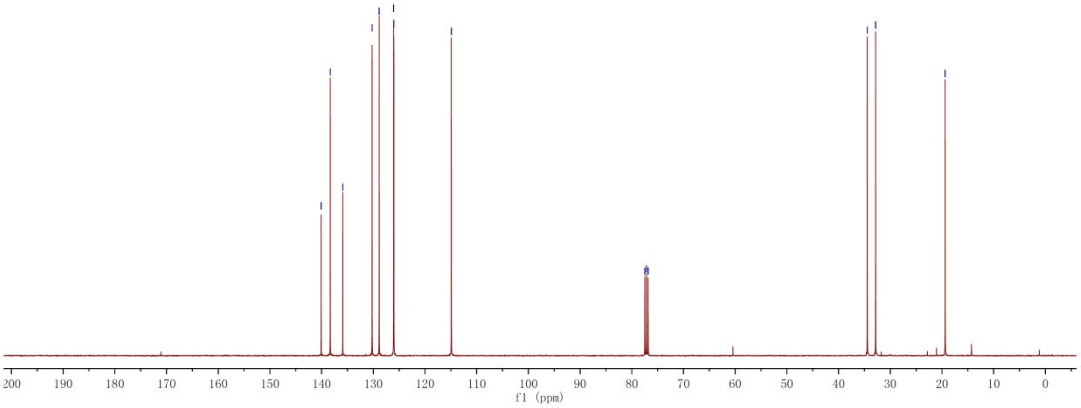

7.121
7.109
7.102
7.090
7.077
5.906
5.897
5.889
5.880
5.863
5.855
5.847
5.838
5.078
5.072
5.068
5.033
5.029
5.025
5.020
4.991
4.977
4.969
4.966
2.709
2.687
2.669
2.663
2.648
2.333
2.317
2.304
2.293
2.283

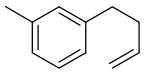

3.99
0.98
2.00
2.05
5.03
f1 (ppm)



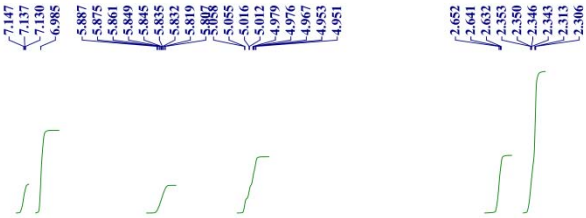
1 b


140.088
138.325
135.906
130.230
128.862
126.064
126.016
114.905
77.478
77.160
76.843
34.439
32.827
19.383


f1 (ppm)



1 c


7.147
7.137
7.130
6.985
5.887
5.875
5.861
5.849
5.845
5.835
5.832
5.819
5.055
5.016
5.012
4.979
4.976
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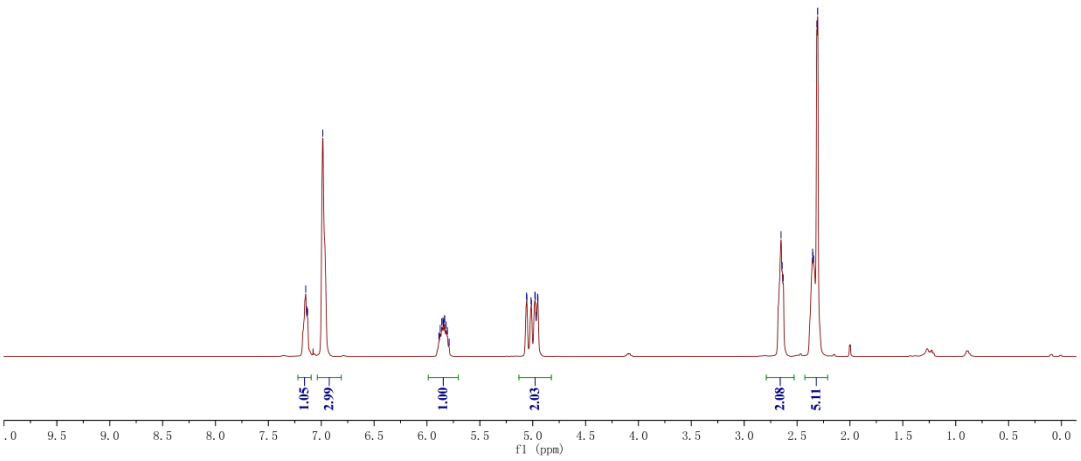
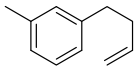
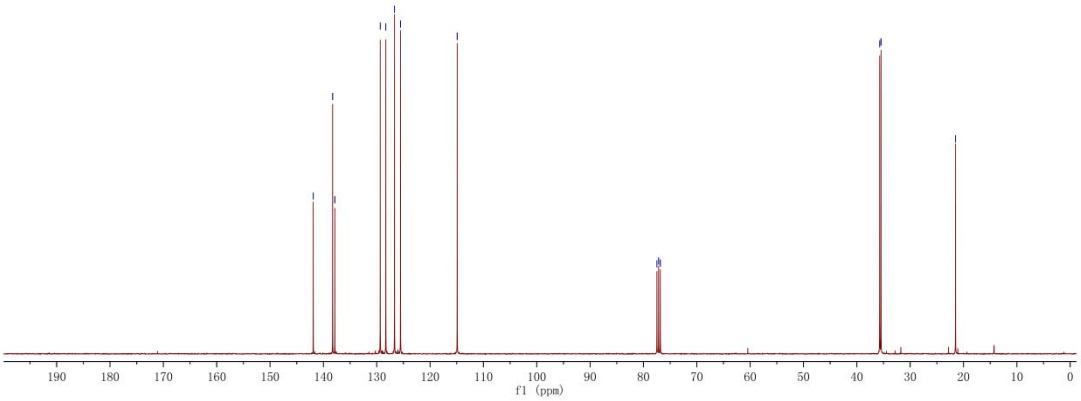
2.353
2.350
2.346
2.343
2.313
2.306

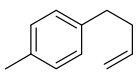

1.05
2.99
1.00
2.03
2.08
5.11
f1 (ppm)



1 c

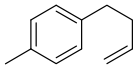

141.900
138.267
137.858
129.346
128.303
126.667
125.537
114.913
77.477
77.160
76.842
35.714
35.462
21.498


f1 (ppm)



1 d

7.172
7.150
7.128
5.957
5.948
5.941
5.932
5.915
5.905
5.898
5.889
5.873
5.130
5.126
5.087
5.083
5.053
5.050
5.027
5.025
2.755
2.736
2.715
2.447
2.430
2.427
2.410
2.390
2.380
4.07
1.00
2.04
2.10
5.21
f1 (ppm)



1 d

138.909
138.333
135.320
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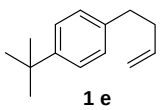
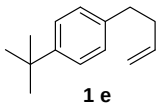
128.426
114.940
77.477
77.160
76.842
35.790
35.075
21.125
f1 (ppm)



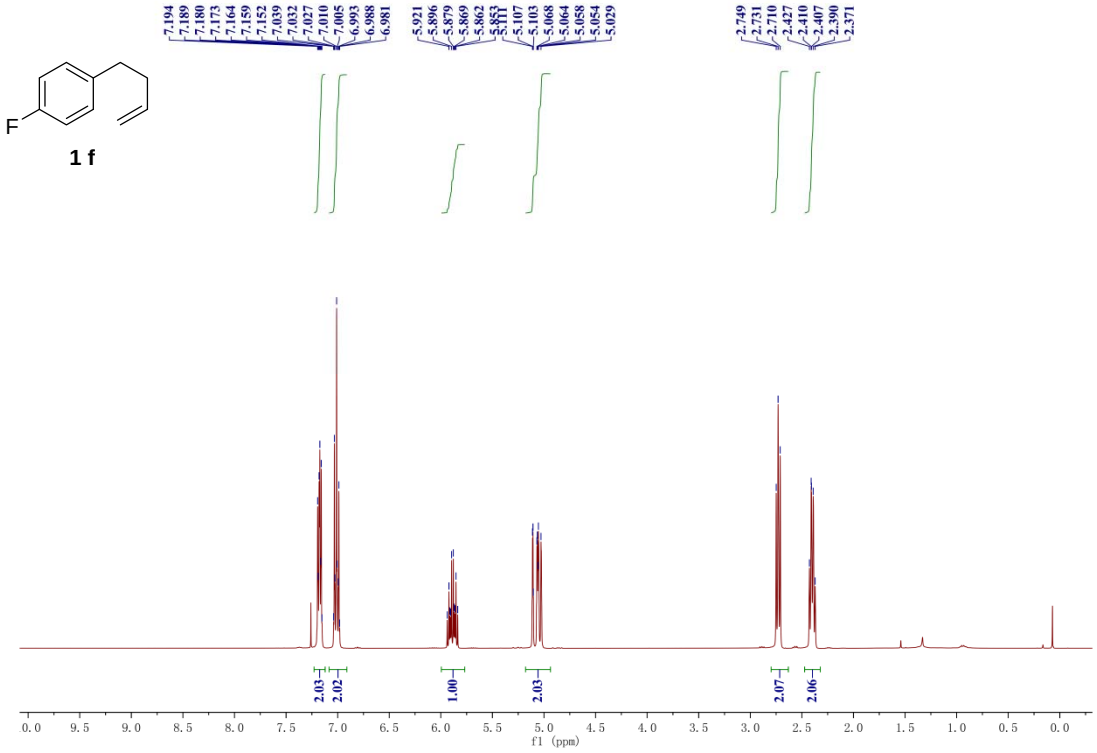
1 e

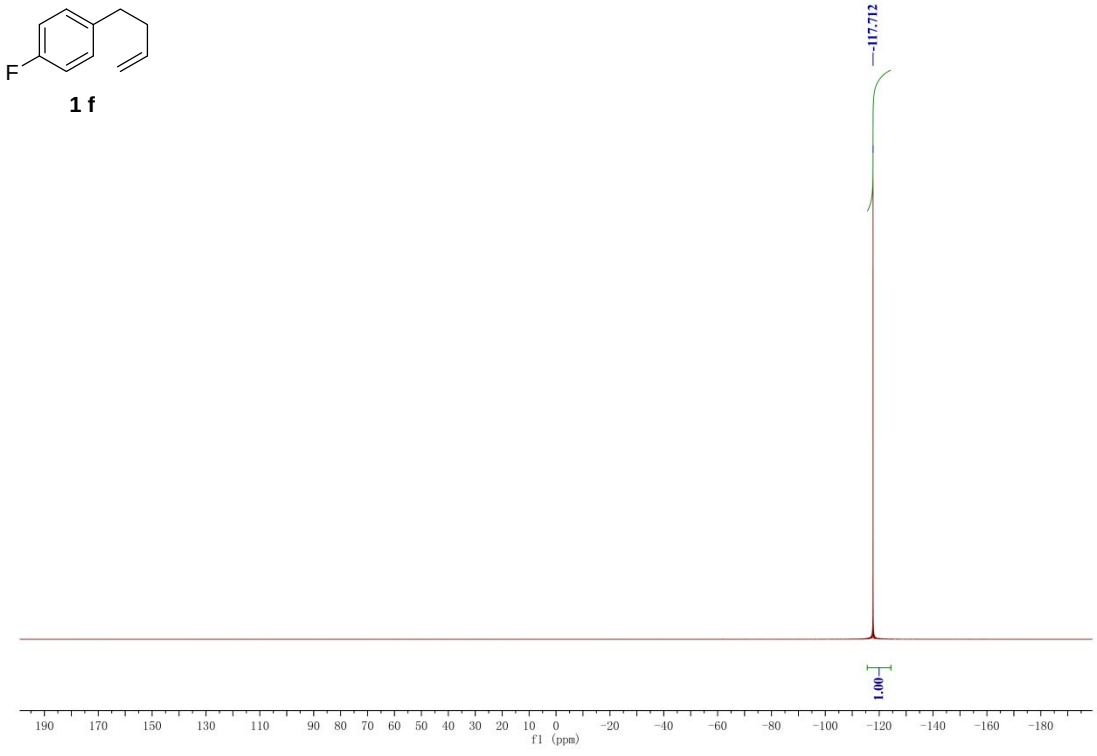
7.306
7.286
7.127
7.107
5.897
5.888
5.881
5.871
5.855
5.845
5.838
5.829
5.071
5.067
5.062
5.032
5.028
5.024
5.020
4.984
4.982
4.980
4.962
4.959
4.956
4.954
2.690
2.671
2.650
2.387
2.370
2.367
2.350
2.347
2.331
1.302
2.01
2.10
1.00
2.04
2.06
2.09
9.04
f1 (ppm)



1 e

148.677
138.942
138.427
128.195
125.315
114.907
77.478
77.160
76.843
35.645
34.993
34.484
31.578
f1 (ppm)


F
1 f
7.194
7.189
7.180
7.173
7.164
7.159
7.152
7.039
7.032
7.027
7.010
7.005
6.993
6.988
6.981
5.921
5.896
5.879
5.869
5.862
5.853
5.111
5.107
5.103
5.068
5.064
5.058
5.054
5.029
2.749
2.731
2.710
2.427
2.410
2.407
2.390
2.371
2.03
2.02
1.00
2.03
2.07
2.06
f1 (ppm)


F
1 f
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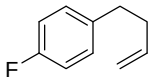
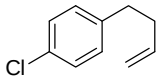
1.00
f1 (ppm)


F

1 f

162.615
160.199
137.887
137.553
137.523
129.905
129.830
115.262
115.220
115.010
77.479
77.160
76.843
35.748
34.664
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)



1 g


7.224
7.203
7.083
7.062
5.834
5.812
5.808
5.796
5.792
5.782
5.775
5.770
5.034
5.029
4.990
4.986
4.982
4.956
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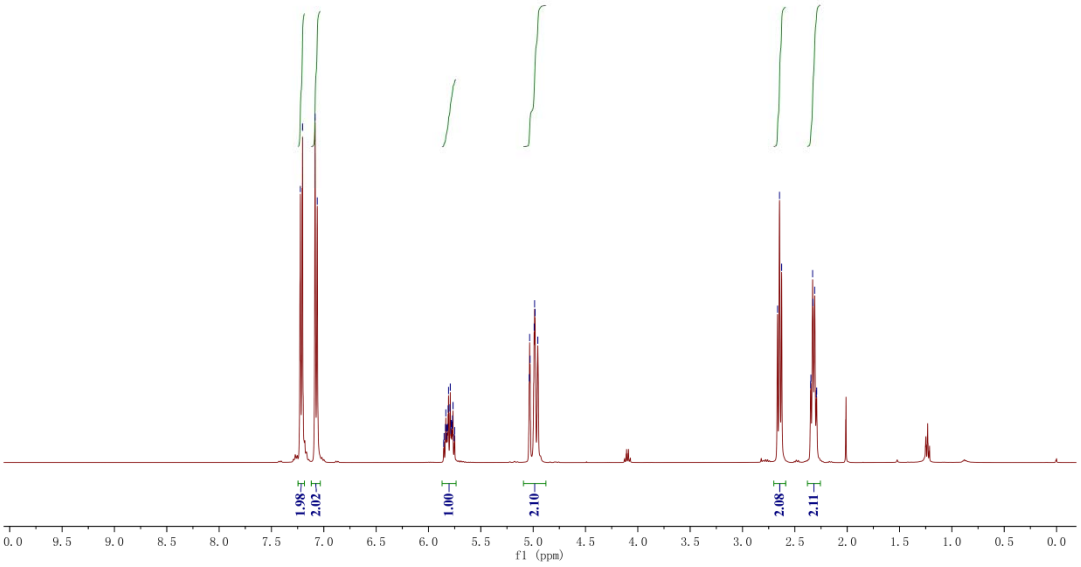
2.647
2.626
2.350
2.347
2.330
2.327
2.311
2.295
2.292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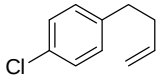
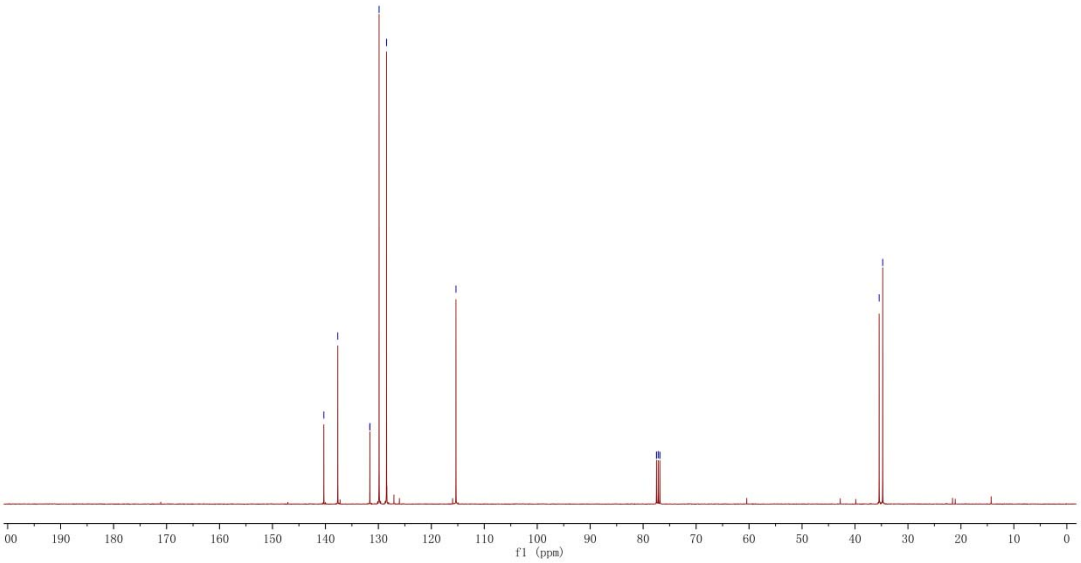
1.00
2.10
2.08
2.11
f1 (ppm)



1 g


140.298
137.663
131.609
129.867
128.451
115.348
77.478
77.160
76.843
35.434
34.759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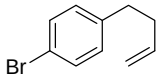
Br

1 h


7.443
7.422
7.100
7.080
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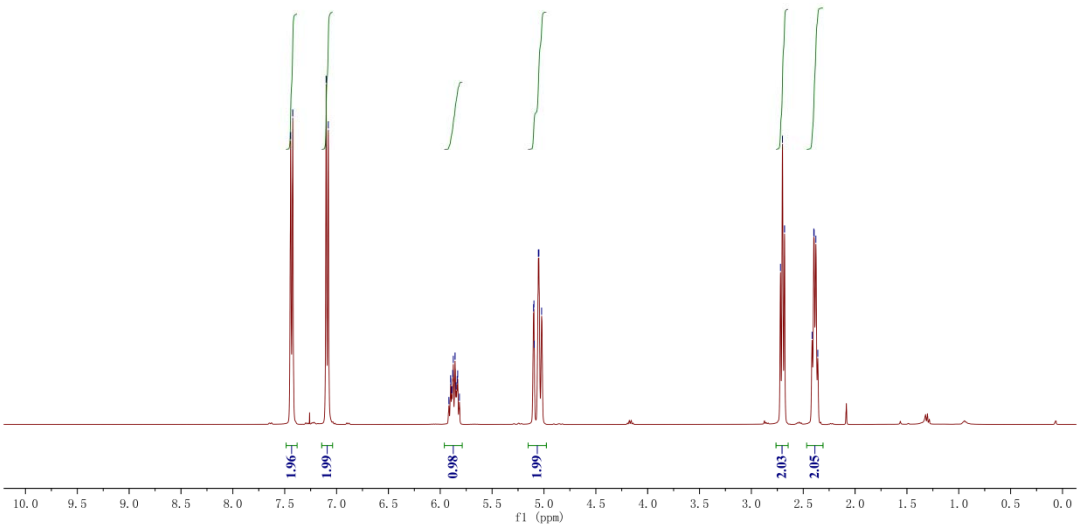
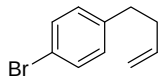
5.888
5.879
5.874
5.857
5.848
5.837
5.101
5.097
5.093
5.054
5.049
5.022
2.719
2.700
2.680
2.415
2.398
2.395
2.378
2.360


1.96
1.99
0.98
1.99
2.03
2.05
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


Br

1 h


140.799
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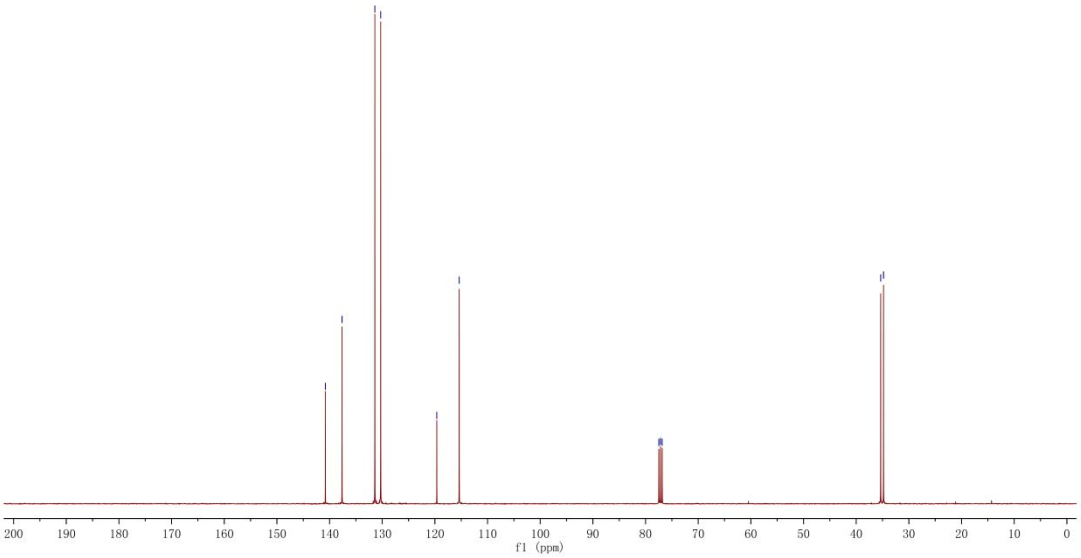
77.477
77.160
76.842
35.355
34.814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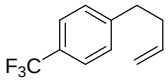
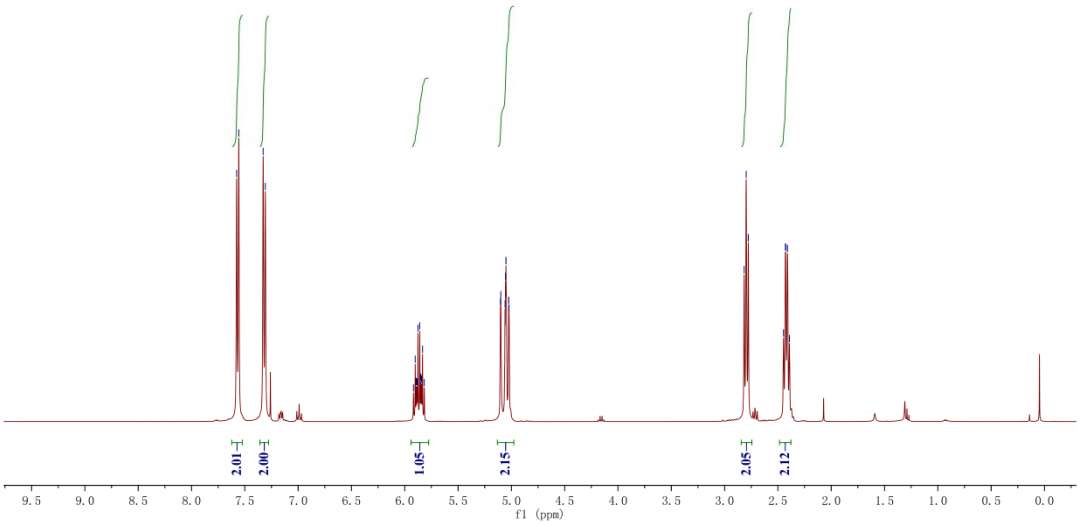
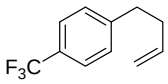
f1 (ppm)



1 i

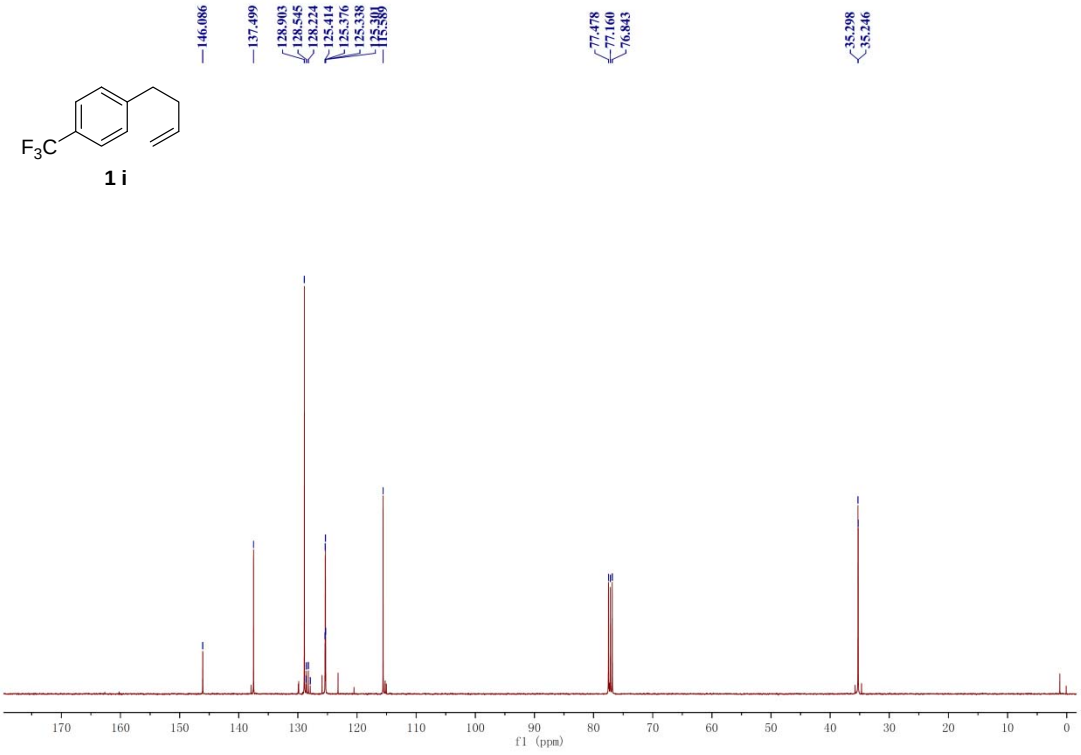

7.577
7.557
7.328
7.308
5.918
5.902
5.893
5.885
5.876
5.859
5.850
5.842
5.833
5.817
5.102
5.098
5.060
5.055
5.051
5.025
5.022
2.816
2.798
2.778
2.447
2.430
2.428
2.411
2.392

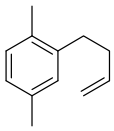

2.01
2.00
1.05
2.15
2.05
2.12
f1 (ppm)



1 i

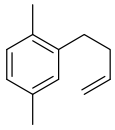
-62.841
1.00
f1 (ppm)


146.086
137.499
128.903
128.545
128.224
125.414
125.376
125.338
77.478
77.160
76.843
35.298
35.246
F3C
1 i
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



1 j

7.183
7.165
7.159
7.112
7.078
7.071
6.077
6.067
6.060
6.051
6.042
6.035
6.025
5.247
5.243
5.205
5.200
5.165
5.160
5.139
5.135
2.817
2.806
2.799
2.792
2.778
2.448
2.417
2.410
3.04
1.00
2.05
2.11
8.26
f1 (ppm)



1 j

139.960
138.456
135.323
132.749
130.167
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77.160
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21.076
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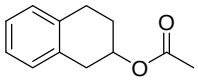
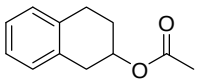
f1 (ppm)



3 a

7.117
7.105
7.094
7.082
7.072
7.052
7.039
7.030
5.218
5.209
5.205
5.197
5.188
5.179
5.172
5.167
5.159
3.118
3.105
3.076
3.063
2.942
2.916
2.900
2.885
2.868
2.856
2.842
2.824
2.706
2.061
2.053
2.028
2.013
2.005
1.990
1.974
1.959
1.954
1.940
1.934
1.928
1.920
1.907
1.901
1.887
1.865
3.96
1.00
1.02
3.07
f1 (ppm)



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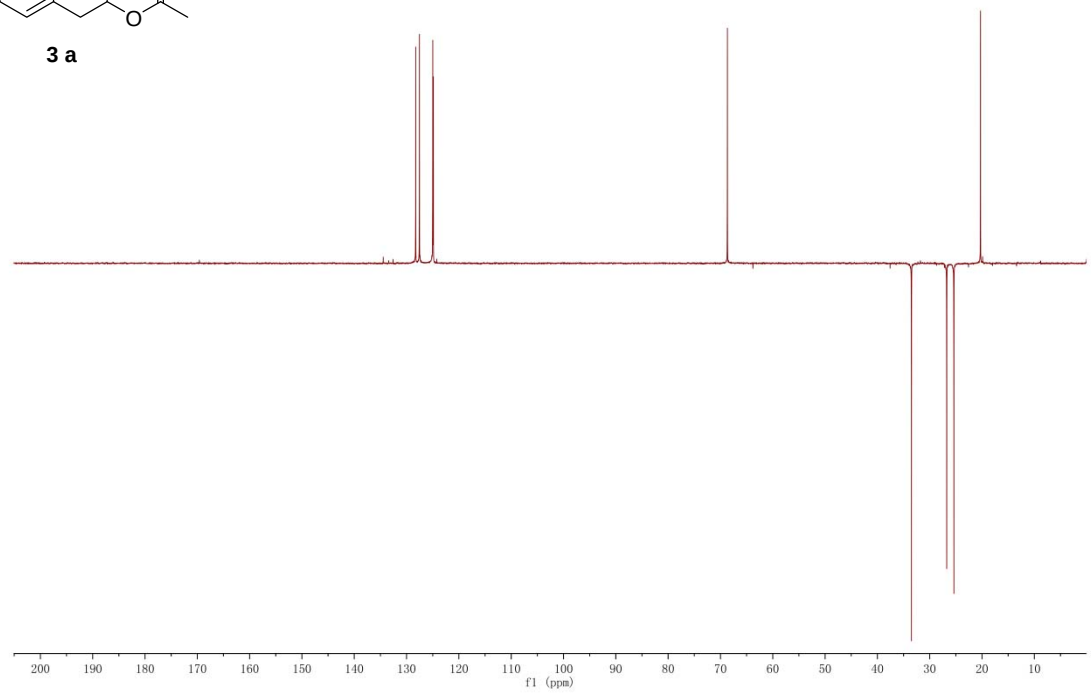
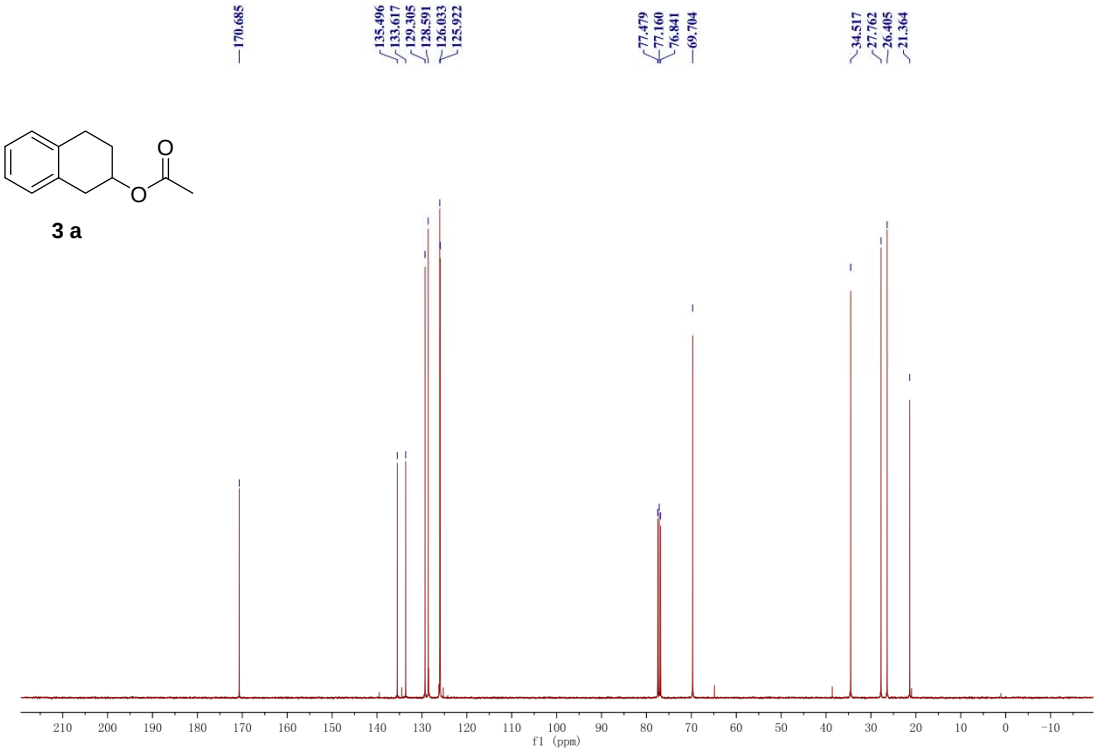

f1 (ppm)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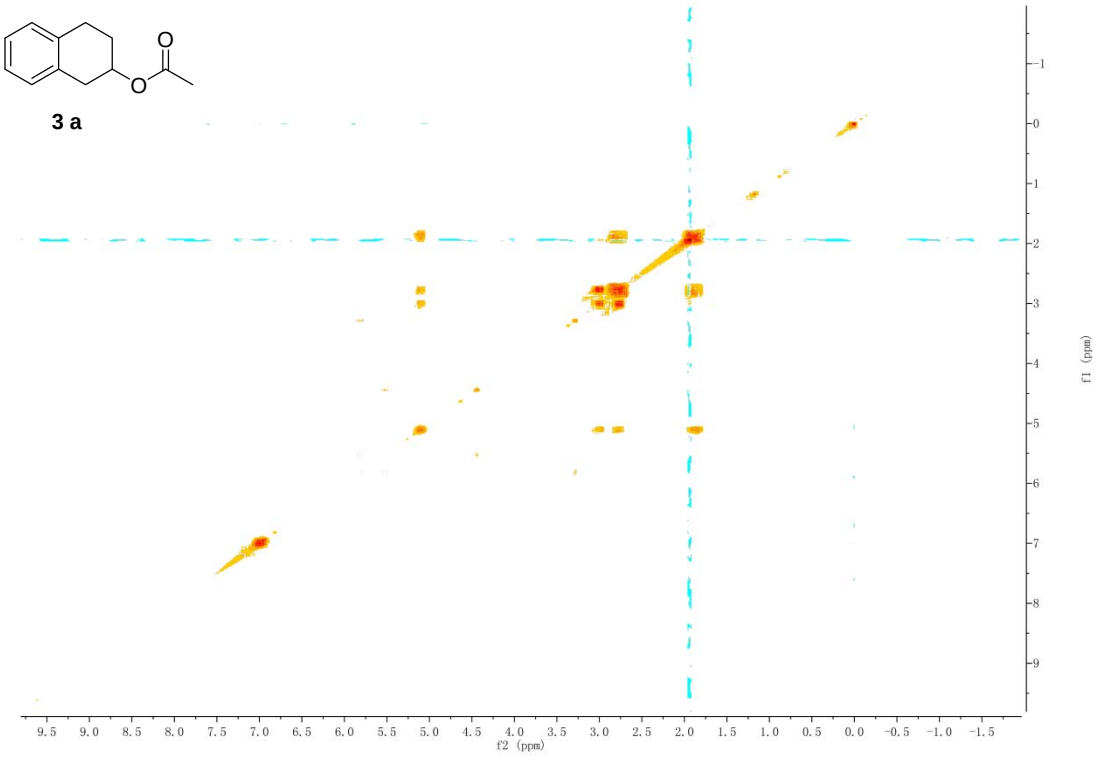
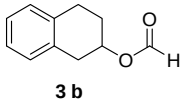
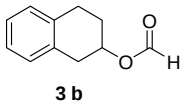
135.496
133.617
129.305
128.591
126.033
125.922
77.479
77.160
76.841
69.704
34.517
27.762
26.405
21.364
O
O
3 a
f1 (ppm)


O
O
3 a
f2 (ppm)
f1 (ppm)



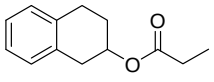
3 b

8.032
7.124
7.119
7.113
7.101
7.089
7.079
7.065
7.054
7.041
7.032
5.359
5.348
5.340
5.330
5.321
5.302
3.131
3.118
3.089
3.077
2.958
2.941
2.924
2.915
2.908
2.899
2.882
2.866
2.849
2.832
2.825
2.076
2.067
2.059
2.051
2.043
2.035
2.025
2.008
2.005
1.989
1.971
1.957
1.937
1.02
4.01
1.00
1.03
3.10
2.07
f1 (ppm)



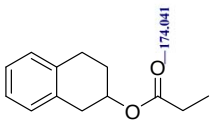
3 b

160.717
135.284
133.173
129.312
128.646
126.213
126.047
77.479
77.160
76.842
69.597
34.424
27.633
26.139
f1 (ppm)



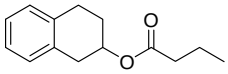
3 c

7.109
7.097
7.086
7.076
7.065
7.045
7.033
7.023
5.227
5.219
5.214
5.207
5.202
5.198
5.194
5.189
5.181
5.176
5.168
3.118
3.105
3.077
3.064
2.936
2.910
2.894
2.877
2.857
2.834
2.817
2.791
2.325
2.306
2.287
2.268
2.011
1.948
1.934
1.118
1.099
3.93
1.00
1.00
3.06
2.06
2.11
3.14
f1 (ppm)



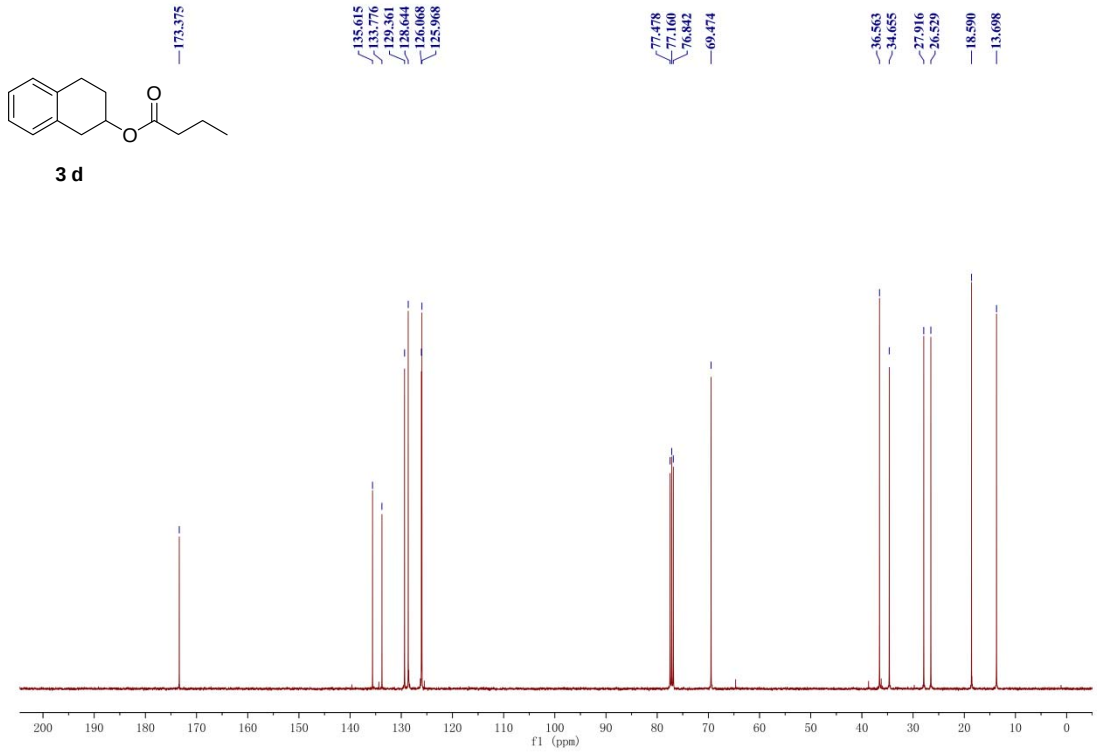
3 c

174.041
135.512
133.673
129.276
128.556
125.989
125.888
77.479
77.160
76.842
69.469
34.556
27.818
26.441
9.146
f1 (ppm)



3 d

7.123
7.111
7.101
7.090
7.080
7.066
7.060
7.047
7.038
5.243
5.234
5.229
5.222
5.217
5.213
5.209
5.204
5.196
5.191
5.183
3.128
3.116
3.087
3.074
2.964
2.886
2.868
2.827
2.792
2.288
2.269
2.251
1.958
1.944
1.670
1.652
1.633
0.934
0.915
3.96
1.00
1.02
3.07
2.11
2.11
2.16
3.17
f1 (ppm)


173.375
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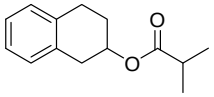
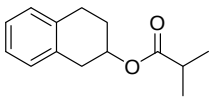
133.776
129.361
128.644
126.068
125.968
77.478
77.160
76.842
69.474
36.563
34.655
27.916
26.529
18.590
13.698
f1 (ppm)

3 d



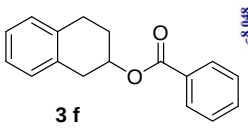
3 e

7.116
7.104
7.094
7.084
7.074
7.060
7.054
7.041
7.032
5.219
5.211
5.206
5.198
5.193
5.189
5.185
5.181
5.173
5.168
5.160
3.121
3.108
3.080
3.067
2.901
2.870
2.861
2.853
2.828
2.811
2.531
2.514
2.496
2.007
1.999
1.953
1.938
1.148
1.141
1.131
3.93
1.00
1.01
3.06
1.03
2.07
2.95
2.95
f1 (ppm)



3 e

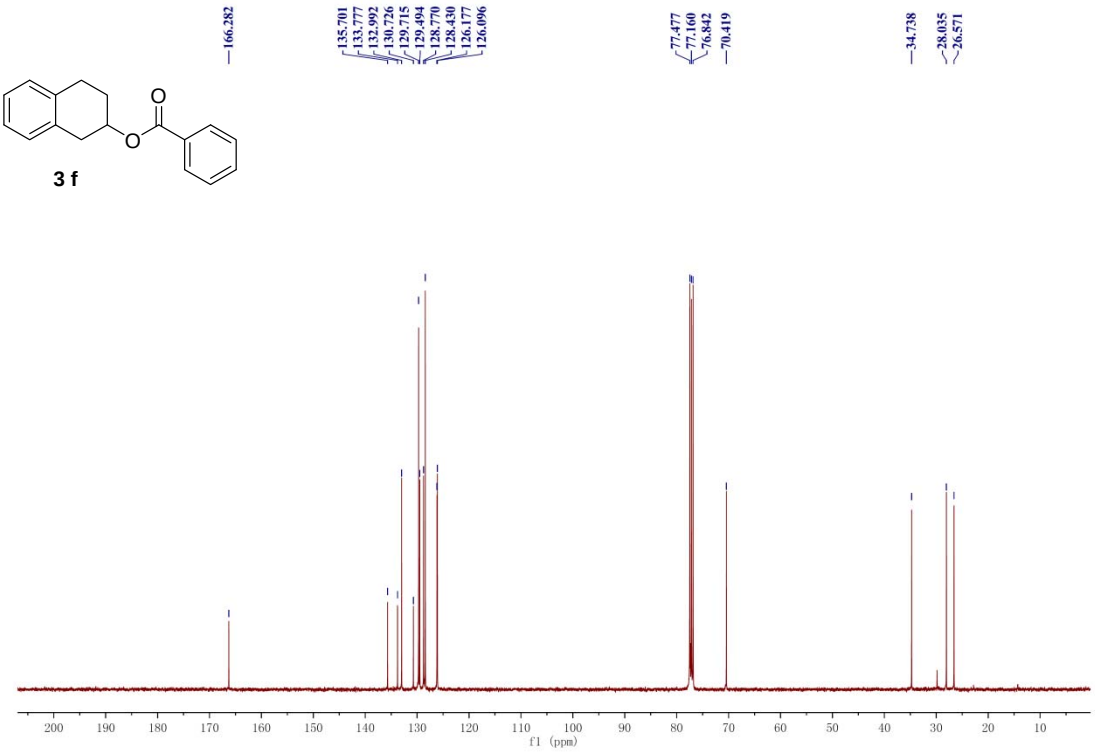
176.781
135.580
133.751
129.316
128.596
126.016
125.928
77.478
77.160
76.842
69.334
34.563
34.145
27.828
26.490
19.074
18.979
f1 (ppm)

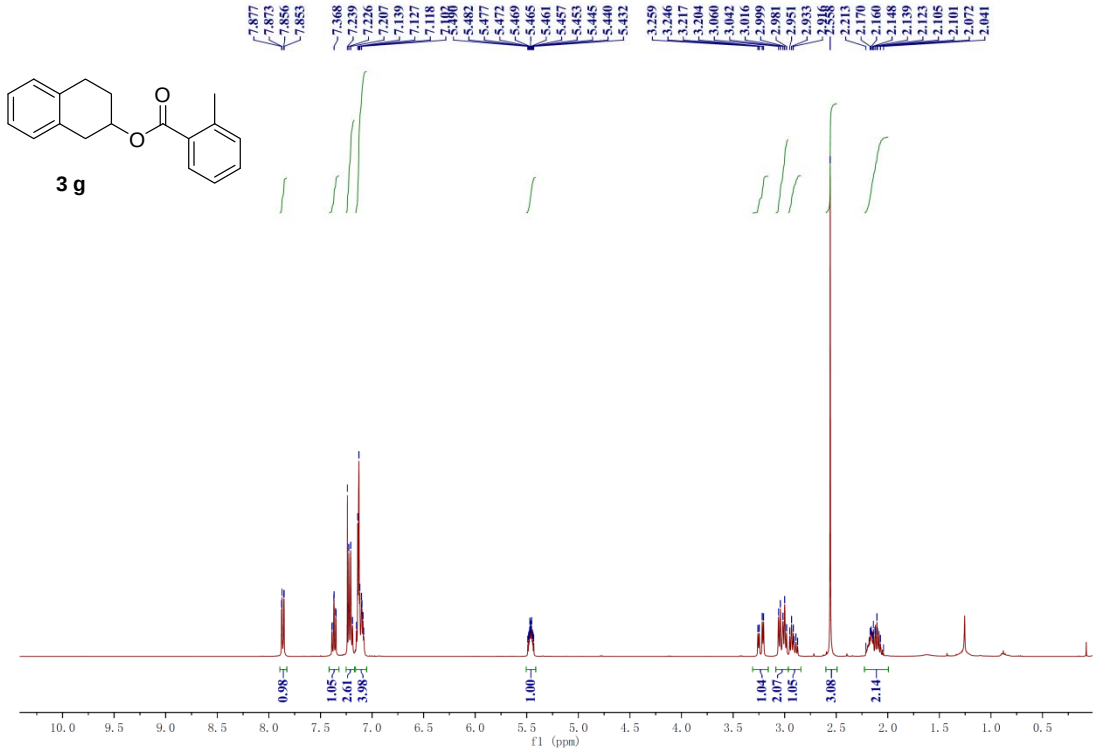


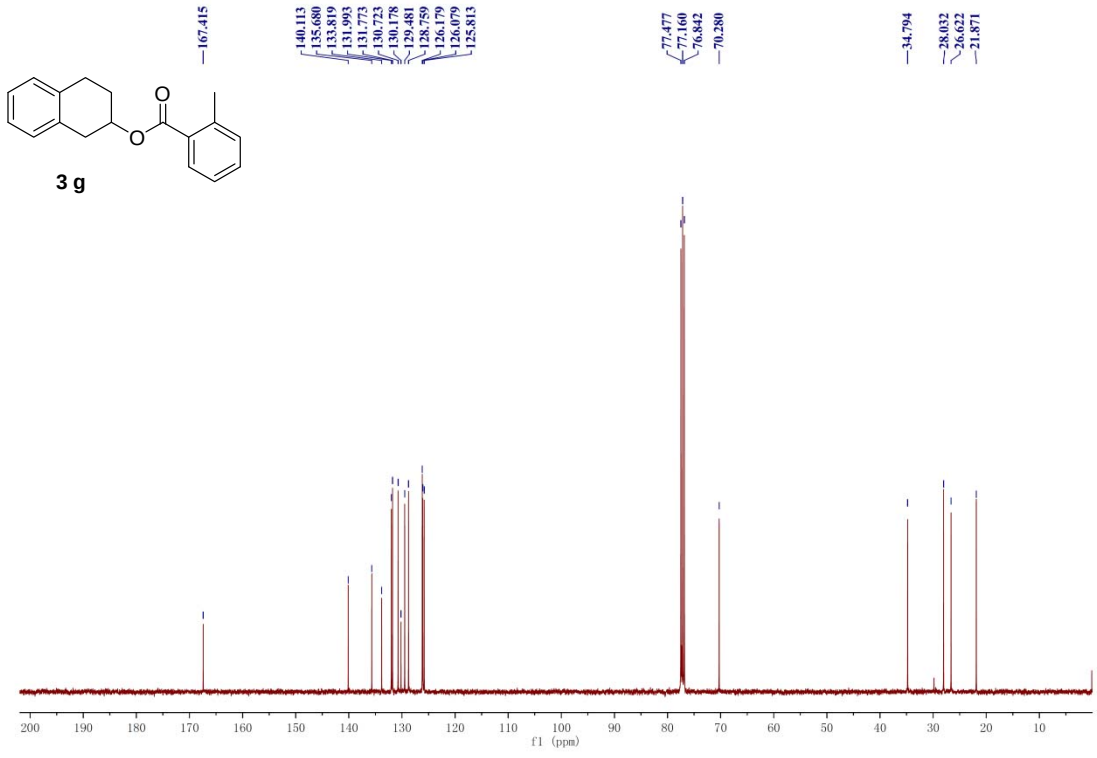
3 f

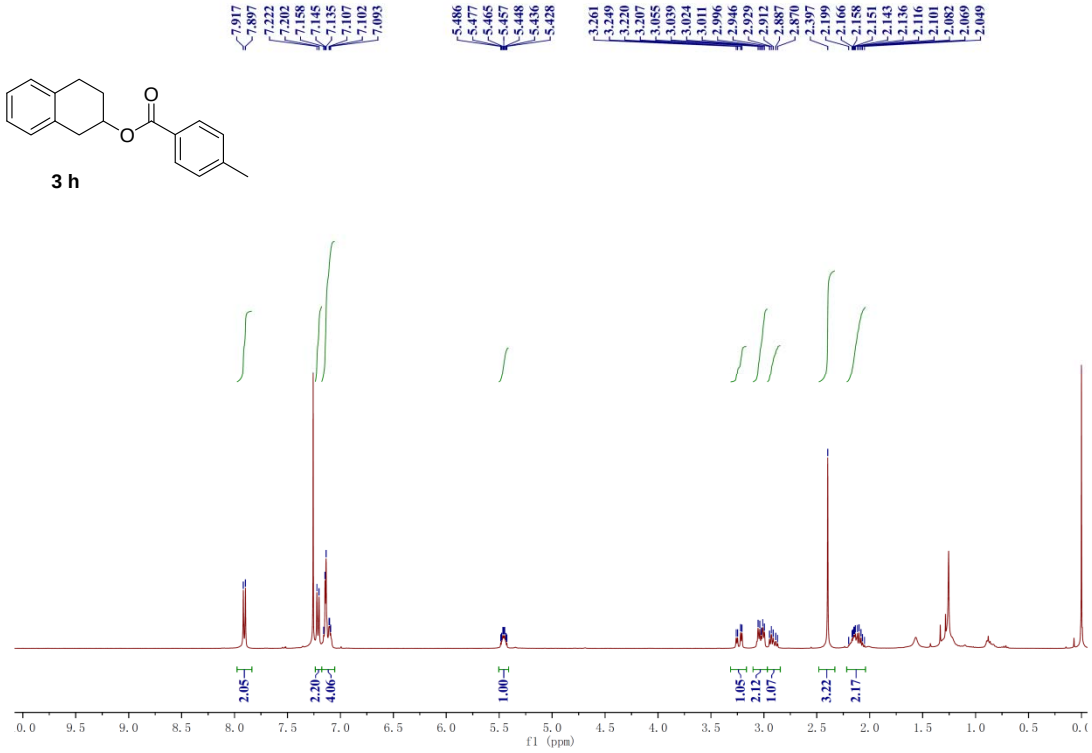
8.048
8.030
7.553
7.535
7.449
7.430
7.411
7.176
7.163
7.153
7.124
7.120
5.515
5.510
5.503
5.494
5.486
5.479
5.474
5.466
3.284
3.272
3.242
3.230
3.083
3.067
3.058
3.042
3.031
3.016
2.967
2.950
2.933
2.907
2.891
2.223
2.207
2.199
2.190
2.182
2.175
2.164
2.149
2.144
2.130
2.111
2.097
2.077
2.058
2.02
1.07
2.05
4.02
1.00
1.04
2.05
1.07
2.15
f1 (ppm)

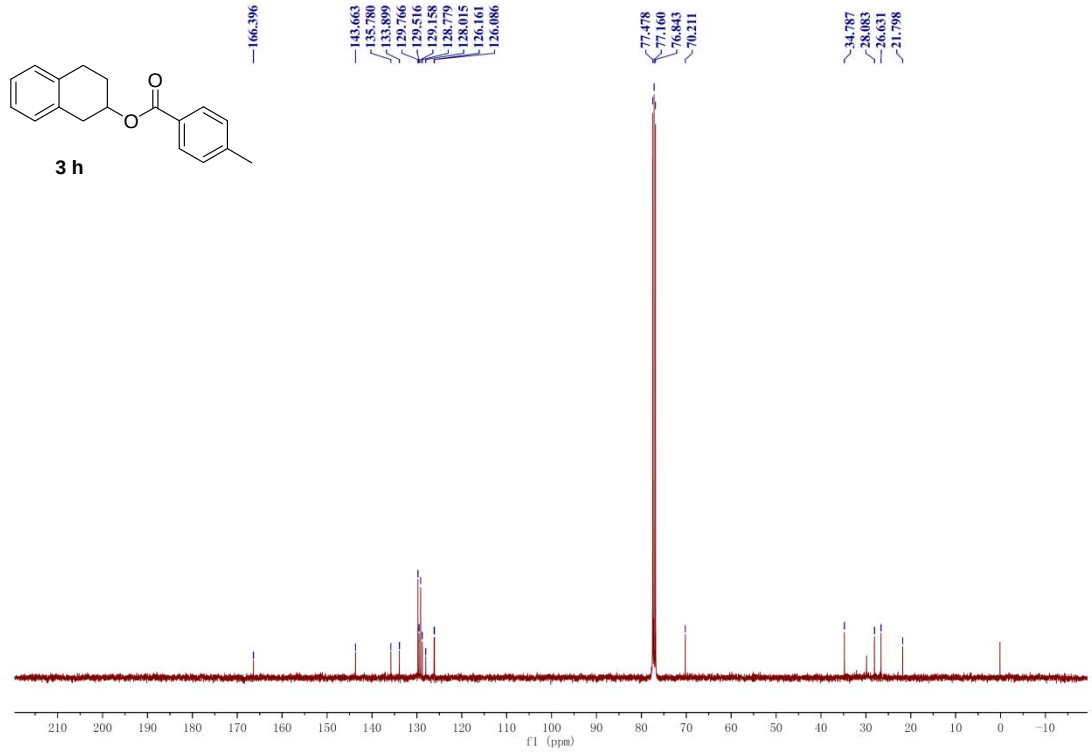
3 f

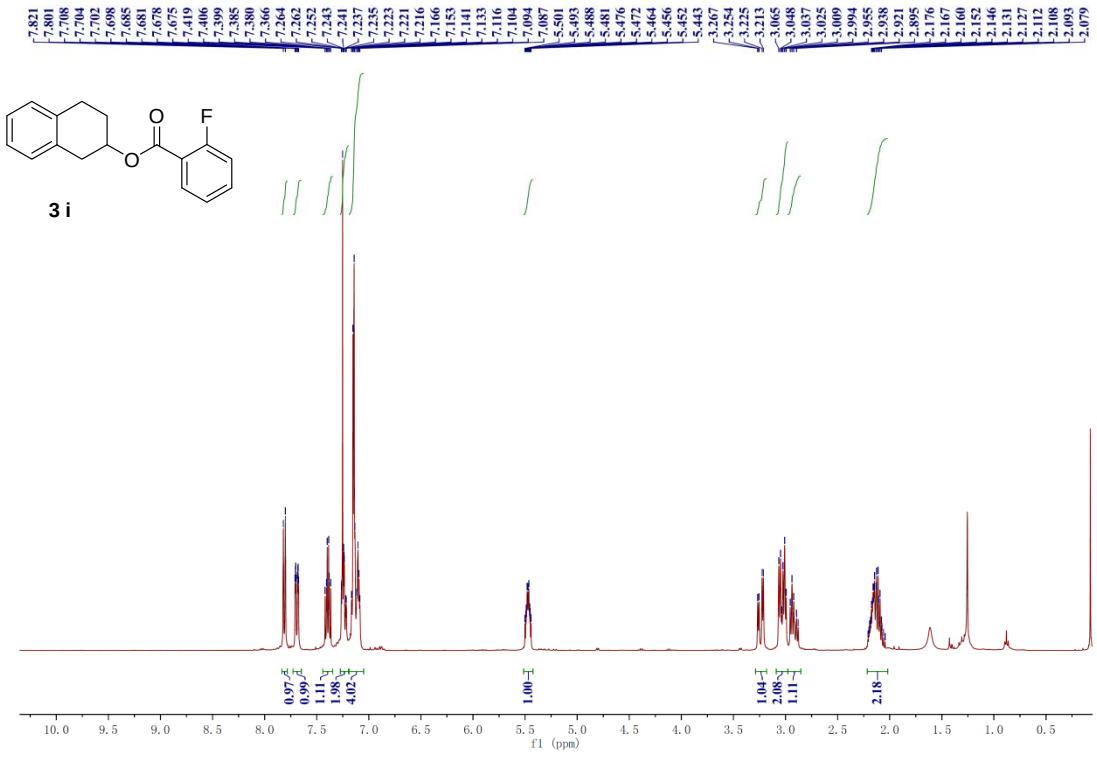

166.282
135.701
133.777
132.992
130.726
129.715
129.494
128.770
128.430
126.177
126.096
77.477
77.160
76.842
70.419
34.738
28.035
26.571
f1 (ppm)

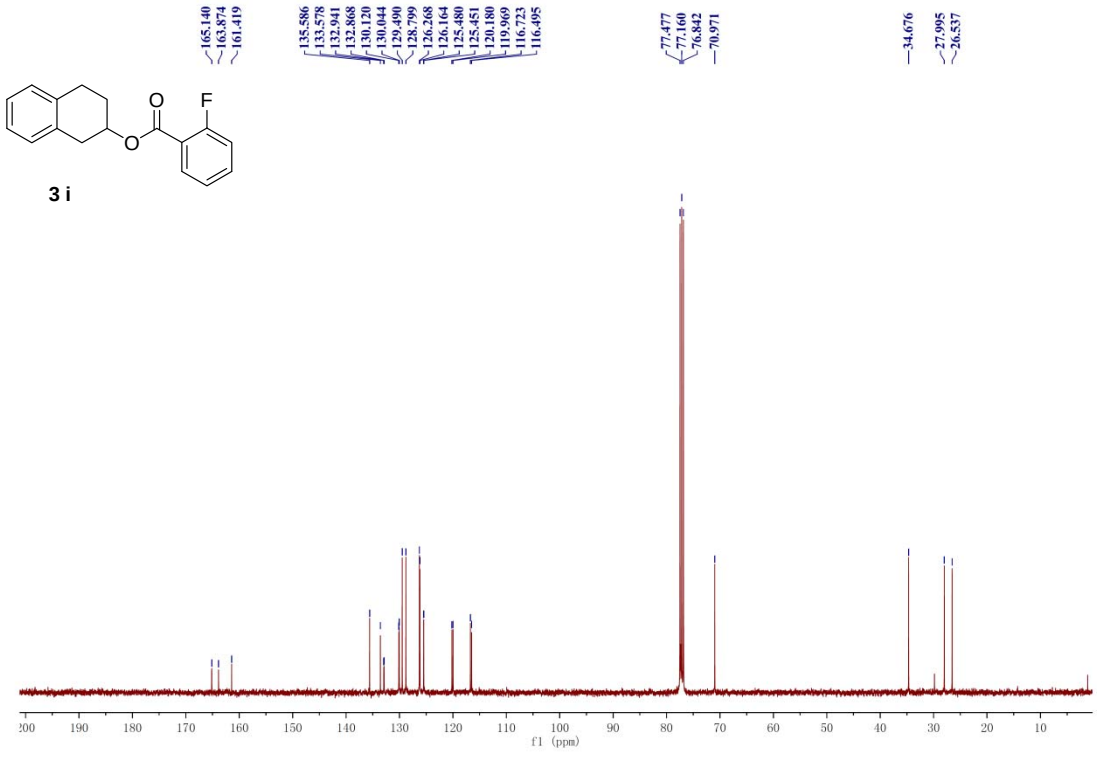

3 g
7.877
7.873
7.856
7.853
7.368
7.239
7.226
7.207
7.139
7.127
7.118
5.482
5.477
5.472
5.469
5.465
5.461
5.457
5.453
5.445
5.440
5.432
3.259
3.246
3.217
3.204
3.060
3.042
3.016
2.999
2.981
2.951
2.933
2.916
2.558
2.213
2.170
2.160
2.148
2.139
2.123
2.105
2.101
2.072
2.041
0.98
1.05
2.61
3.98
1.00
1.04
2.07
1.05
3.08
2.14
f1 (ppm)


3 g
167.415
140.113
135.680
133.819
131.993
131.773
130.723
130.178
129.481
128.759
126.179
126.079
125.813
77.477
77.160
76.842
70.280
34.794
28.032
26.622
21.871
f1 (ppm)


7.917
7.897
7.222
7.202
7.158
7.145
7.135
7.107
7.102
7.093
5.486
5.477
5.465
5.457
5.448
5.436
5.428
3.261
3.249
3.220
3.207
3.055
3.039
3.024
3.011
2.996
2.946
2.929
2.912
2.887
2.870
2.397
2.199
2.166
2.158
2.151
2.143
2.136
2.116
2.101
2.082
2.069
2.049
O
O
3 h
2.05
2.20
4.06
1.00
1.05
2.12
1.07
3.22
2.17
f1 (ppm)


166.396
143.663
135.780
133.899
129.766
129.516
129.158
128.779
128.015
126.161
126.086
77.478
77.160
76.843
70.211
34.787
28.083
26.631
21.798
O
O
3 h
f1 (ppm)


7.821
7.801
7.708
7.704
7.702
7.698
7.685
7.681
7.678
7.675
7.419
7.406
7.399
7.385
7.380
7.366
7.264
7.262
7.252
7.243
7.241
7.237
7.235
7.223
7.221
7.216
7.166
7.153
7.141
7.133
7.116
7.104
7.094
7.087
5.501
5.493
5.488
5.481
5.476
5.472
5.464
5.456
5.452
5.443
3.267
3.254
3.225
3.213
3.065
3.048
3.037
3.025
3.009
2.994
2.955
2.938
2.921
2.895
2.176
2.167
2.160
2.152
2.146
2.131
2.127
2.112
2.108
2.093
2.079
O
F
O
3 i
0.97
0.99
1.11
1.98
4.02
1.00
1.04
2.08
1.11
2.18
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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161.419
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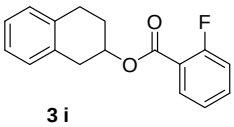
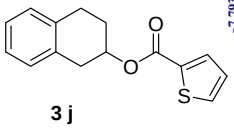
133.578
132.941
132.868
130.120
130.044
129.490
128.799
126.268
126.164
125.480
125.451
120.180
119.969
116.723
116.495
77.477
77.160
76.842
70.971
34.676
27.995
26.537
O
F
O
3 i
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

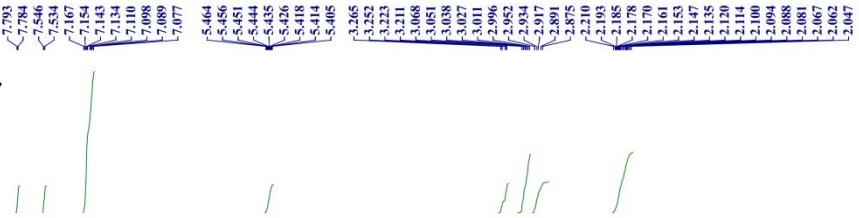

O
F
O

3 i

-112.437
1.00
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -20 -40 -60 -80 -100 -120 -140 -160 -180
f1 (ppm)



3 j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7.154
7.143
7.134
7.110
7.098
7.089
7.077
5.464
5.456
5.451
5.444
5.435
5.426
5.418
5.414
5.405
3.265
3.252
3.223
3.211
3.068
3.051
3.038
3.027
3.011
2.996
2.952
2.934
2.917
2.891
2.875
2.210
2.193
2.185
2.178
2.170
2.161
2.153
2.147
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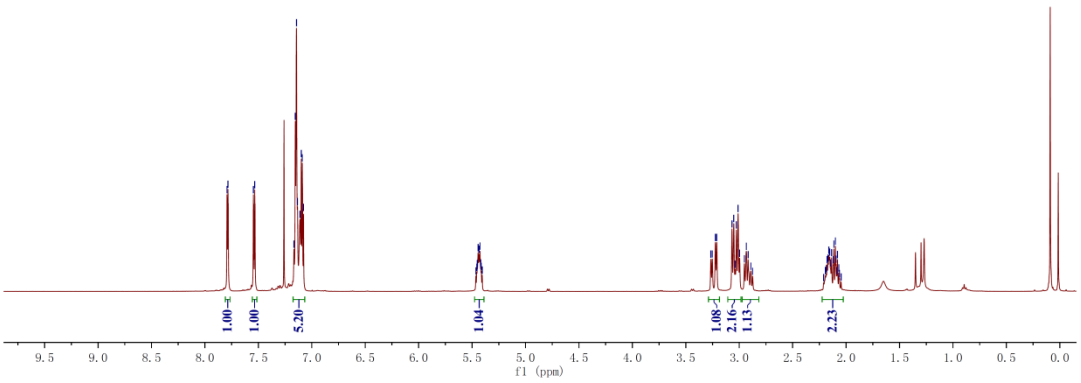
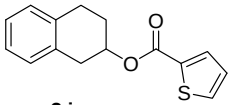
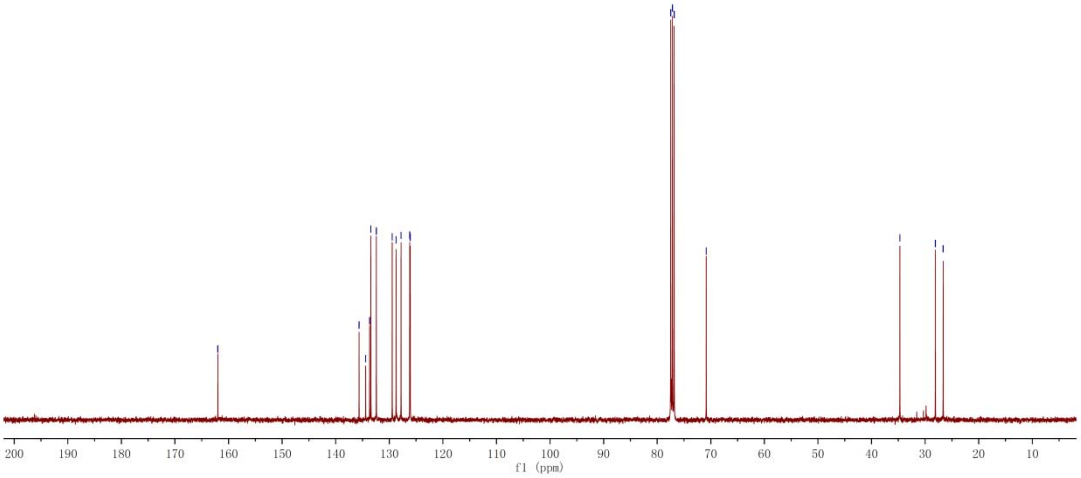
2.088
2.081
2.067
2.062
2.047

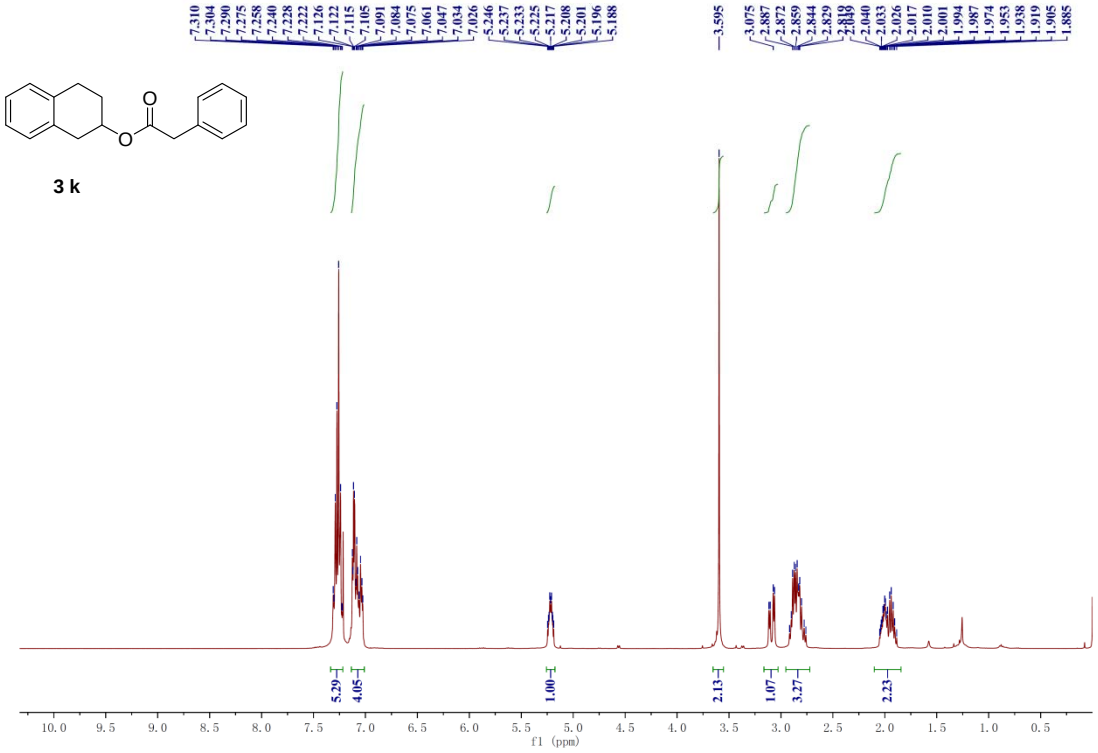

1.00
1.00
5.20
1.04
1.08
2.16
1.13
2.23
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

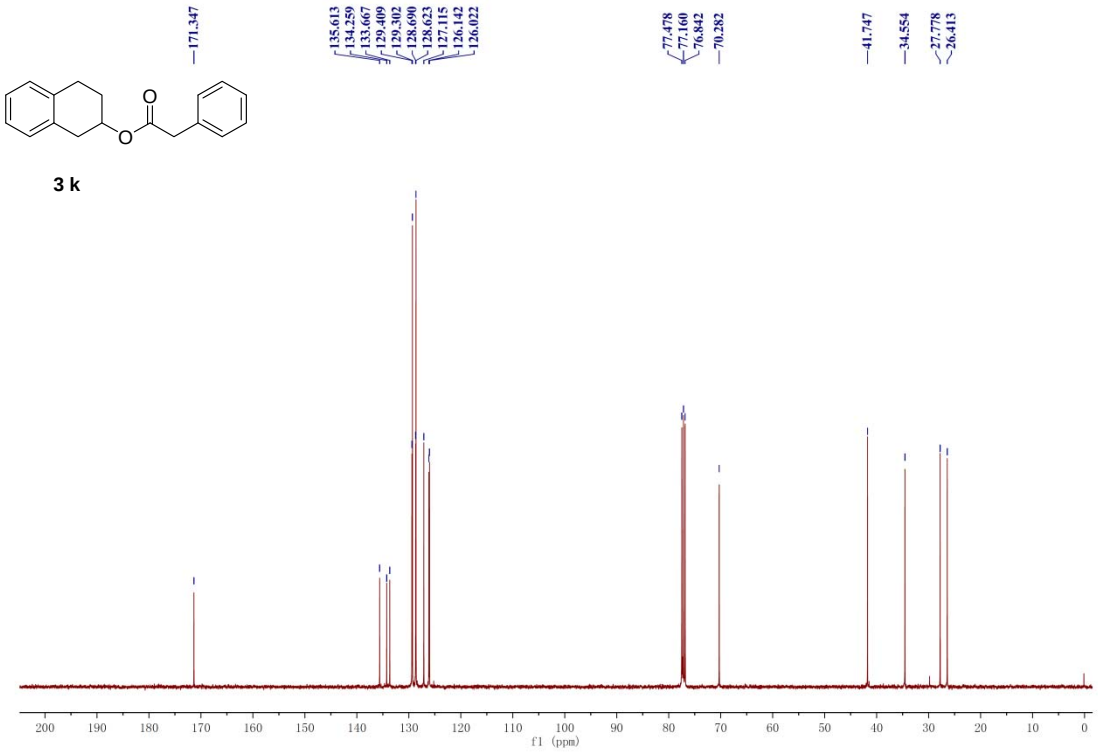

161.993
135.640
134.407
133.673
133.439
132.420
129.458
128.732
127.805
126.186
126.088
77.478
77.160
76.843
70.851
34.711
28.082
26.627



3 j


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


7.310
7.304
7.290
7.275
7.258
7.240
7.228
7.222
7.126
7.122
7.115
7.105
7.091
7.084
7.075
7.061
7.047
7.034
7.026
5.246
5.237
5.233
5.225
5.217
5.208
5.201
5.196
5.188
3.595
3.075
2.887
2.872
2.859
2.844
2.829
2.040
2.033
2.026
2.017
2.010
2.001
1.994
1.987
1.974
1.953
1.938
1.919
1.905
1.885
O
O
3 k
5.29
4.05
1.00
2.13
1.07
3.27
2.23
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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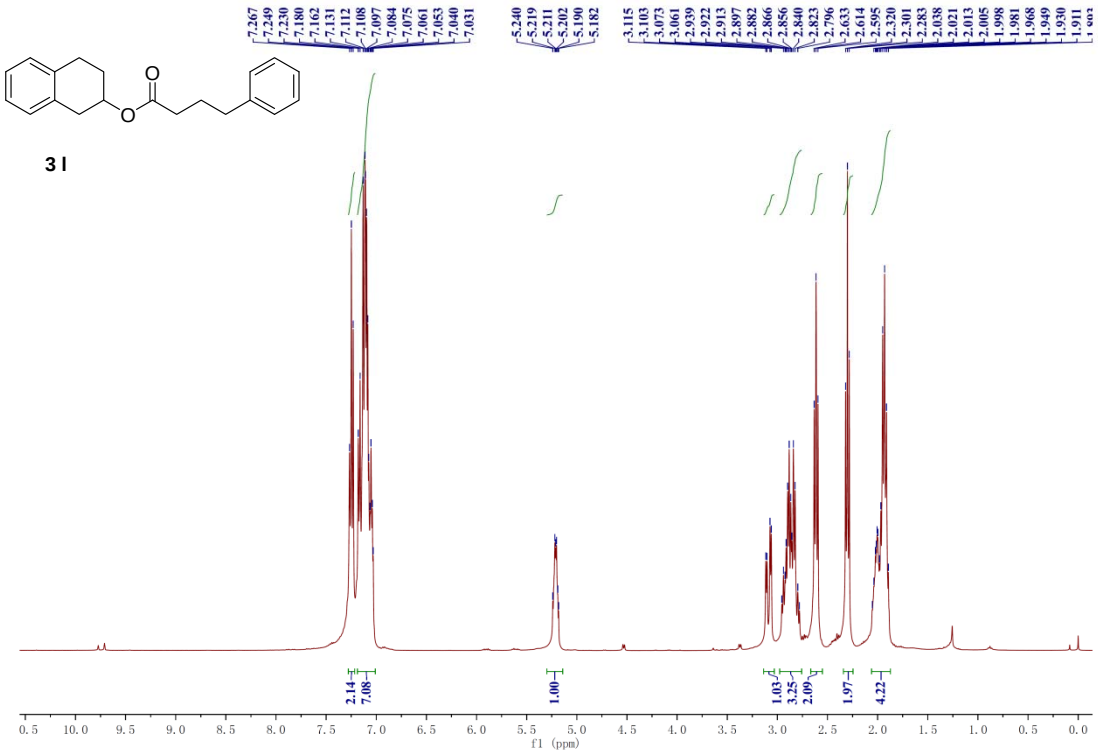
126.022
77.478
77.160
76.842
70.282
41.747
34.554
27.778
26.413
O
O
3 k
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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7.249
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7.108
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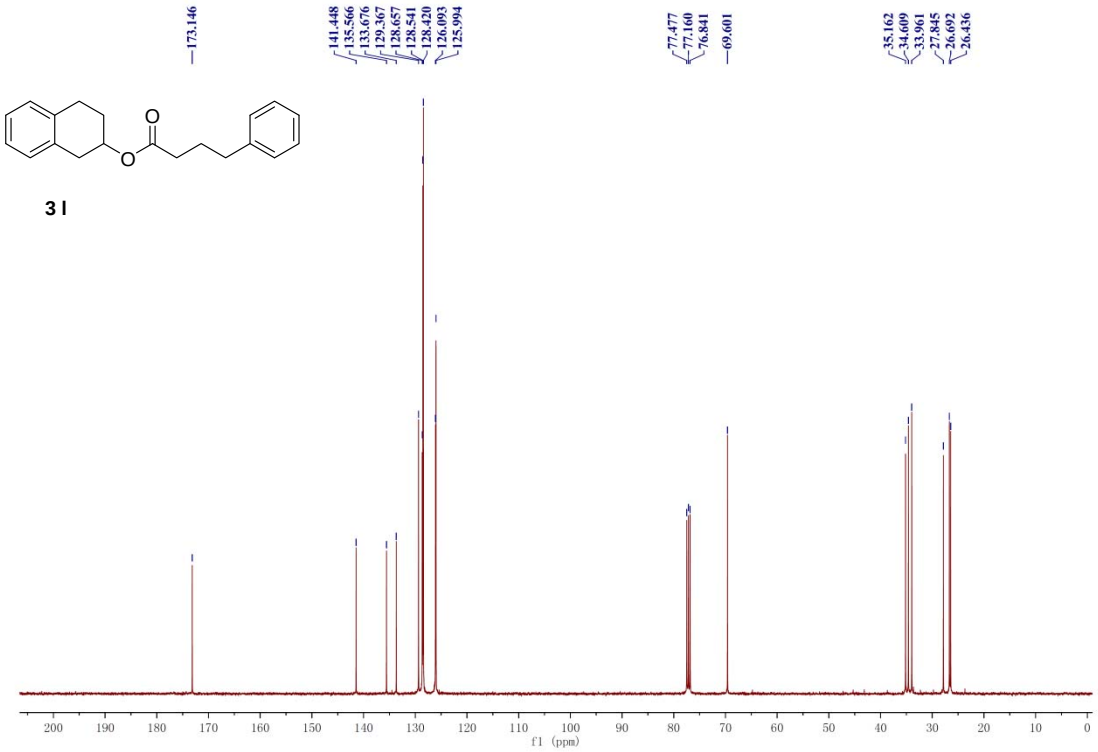
7.075
7.061
7.053
7.040
7.031
5.240
5.219
5.211
5.202
5.190
5.182
3.115
3.103
3.073
3.061
2.939
2.922
2.913
2.897
2.882
2.866
2.856
2.840
2.823
2.796
2.633
2.614
2.595
2.320
2.301
2.283
2.038
2.021
2.013
2.005
1.998
1.981
1.968
1.949
1.930
1.911
2.14
7.08
1.00
1.03
3.25
2.09
1.97
4.22
f1 (ppm)

3 l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125.994
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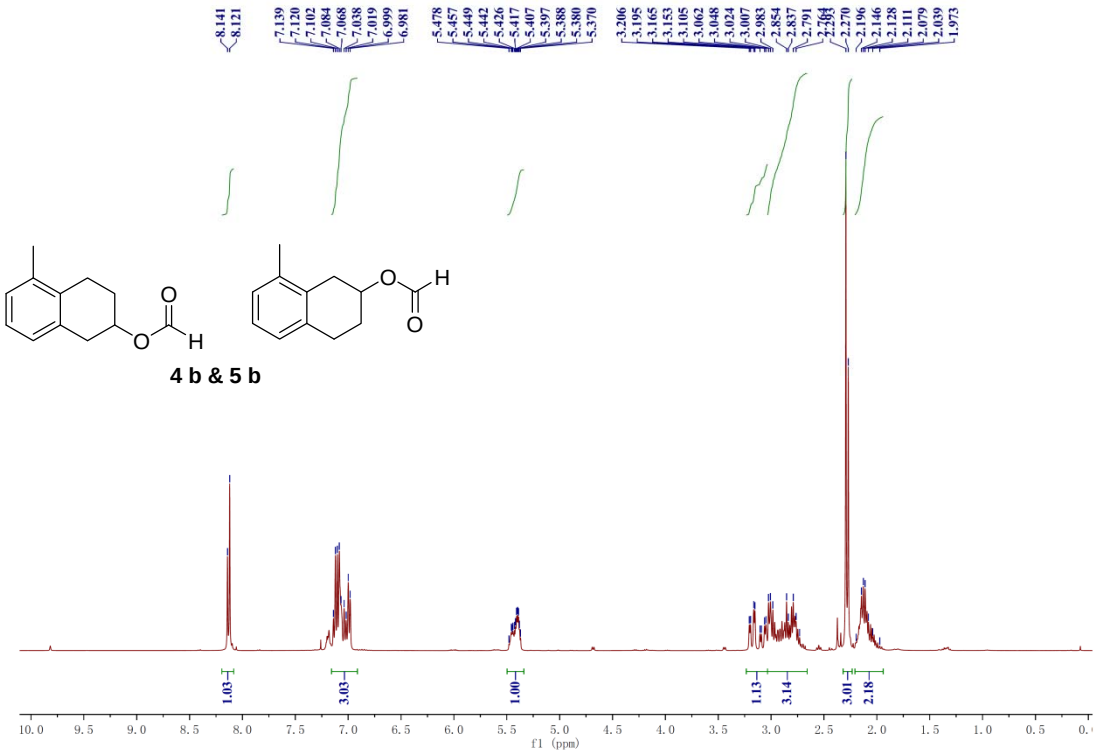
77.160
76.841
69.601
35.162
34.609
33.961
27.845
26.692
26.436
f1 (ppm)

3 l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7.068
7.038
7.019
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6.981
5.478
5.457
5.449
5.442
5.426
5.417
5.407
5.397
5.388
5.380
5.370
3.206
3.195
3.165
3.153
3.105
3.062
3.048
3.024
3.007
2.983
2.854
2.837
2.791
2.270
2.196
2.146
2.128
2.111
2.079
2.039
1.973
O
H
O
O
H
O
4 b & 5 b
1.03
3.03
1.00
1.13
3.14
3.01
2.18
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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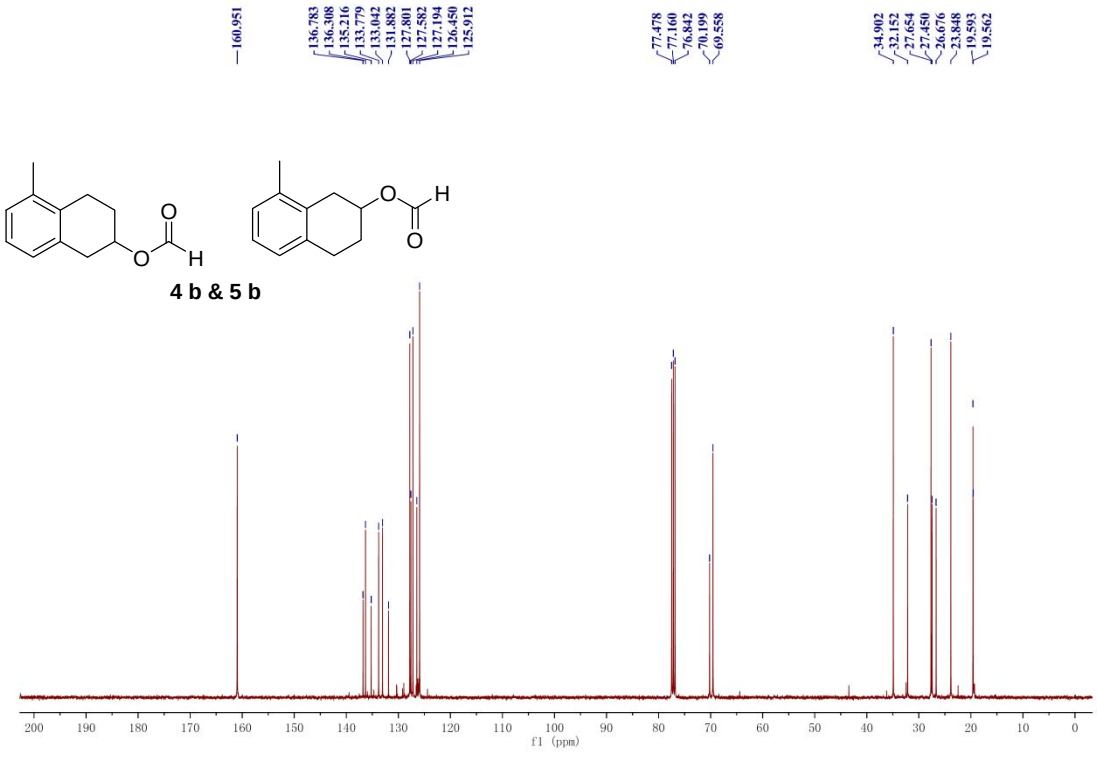
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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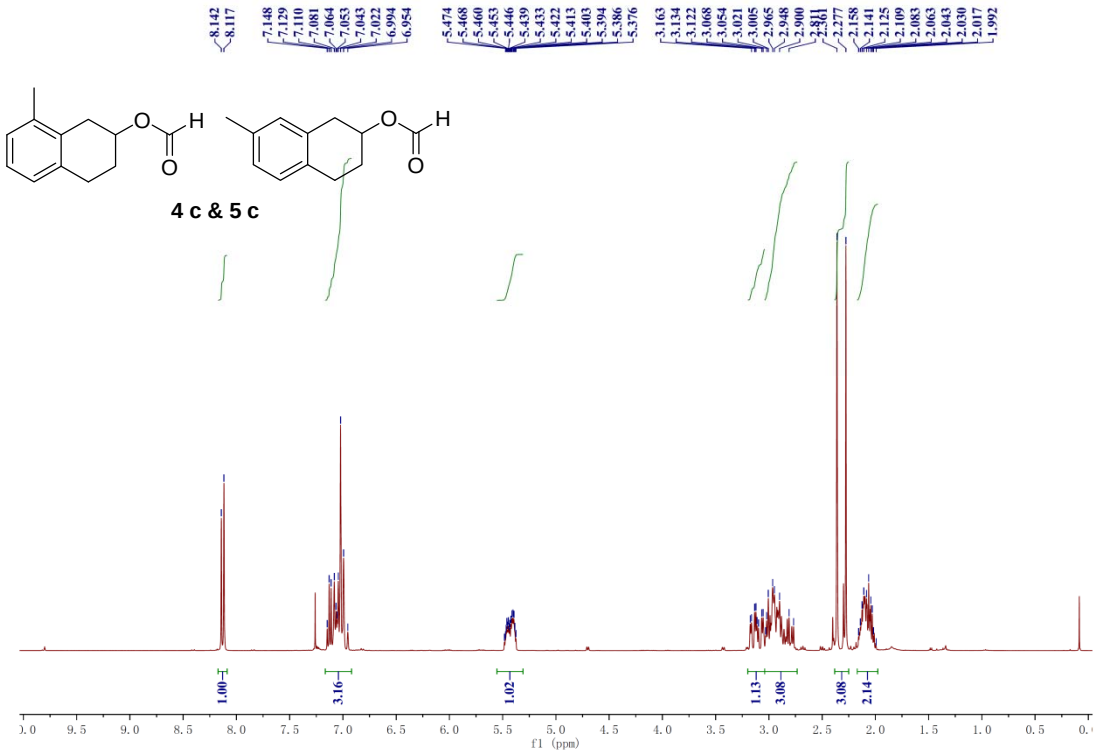
77.160
76.842
70.199
69.558
34.902
32.152
27.654
27.450
26.676
23.848
19.593
19.562
O
H
O
O
H
O
4 b & 5 b
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.053
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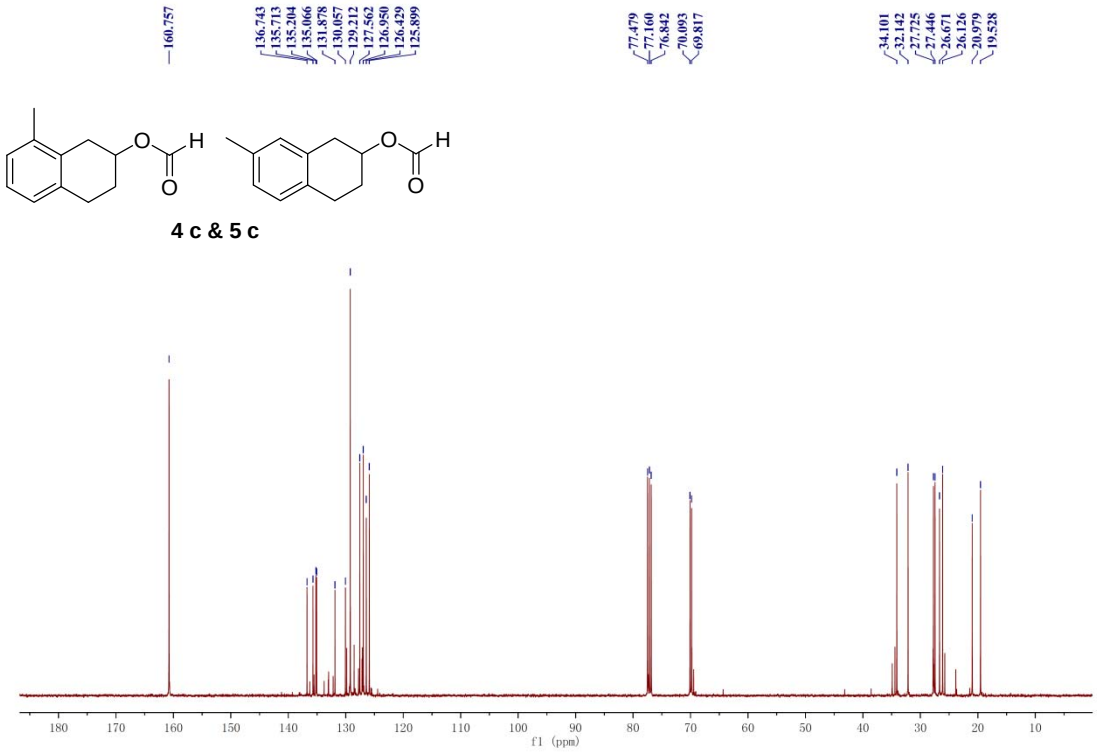
6.954
5.474
5.468
5.460
5.453
5.446
5.439
5.433
5.422
5.413
5.403
5.394
5.386
5.376
3.163
3.134
3.122
3.068
3.054
3.021
3.005
2.965
2.948
2.900
2.277
2.158
2.141
2.125
2.109
2.083
2.063
2.043
2.030
2.017
1.992
O
H
O
O
H
O
4 c & 5 c
1.00
3.16
1.02
1.13
3.08
3.08
2.14
f1 (ppm)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129.212
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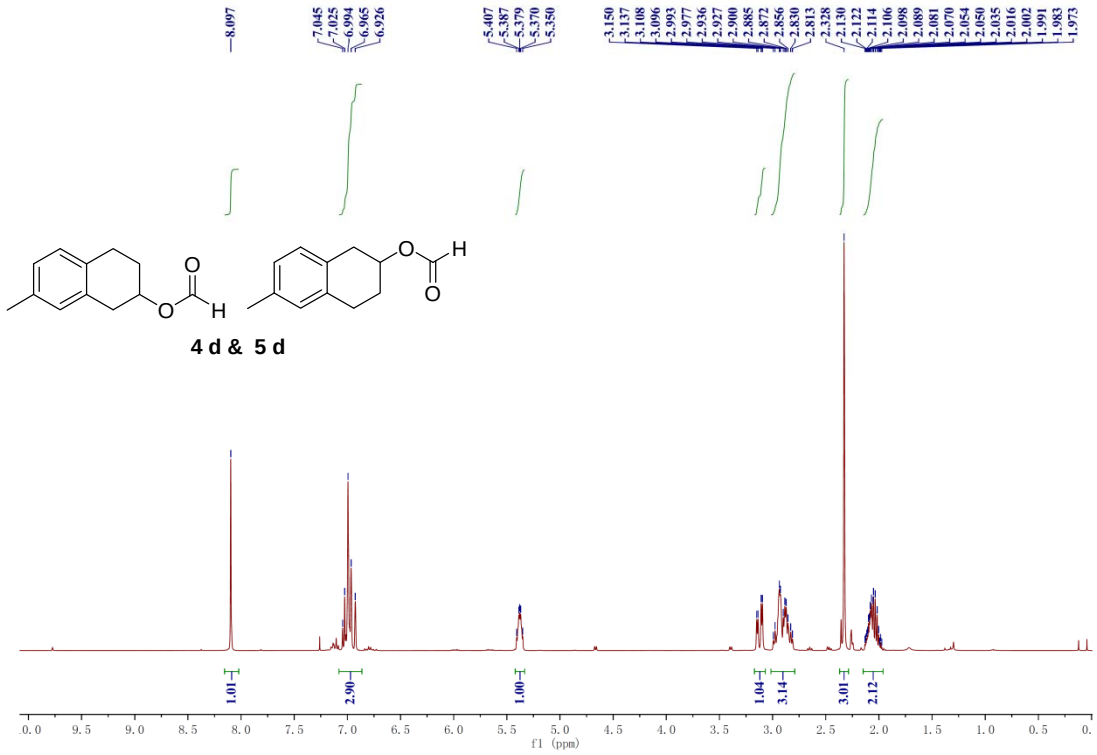
126.950
126.429
125.899
77.479
77.160
76.842
70.093
69.817
34.101
32.142
27.725
27.446
26.671
26.126
20.979
19.528
O
H
O
O
H
O
4 c & 5 c
f1 (ppm)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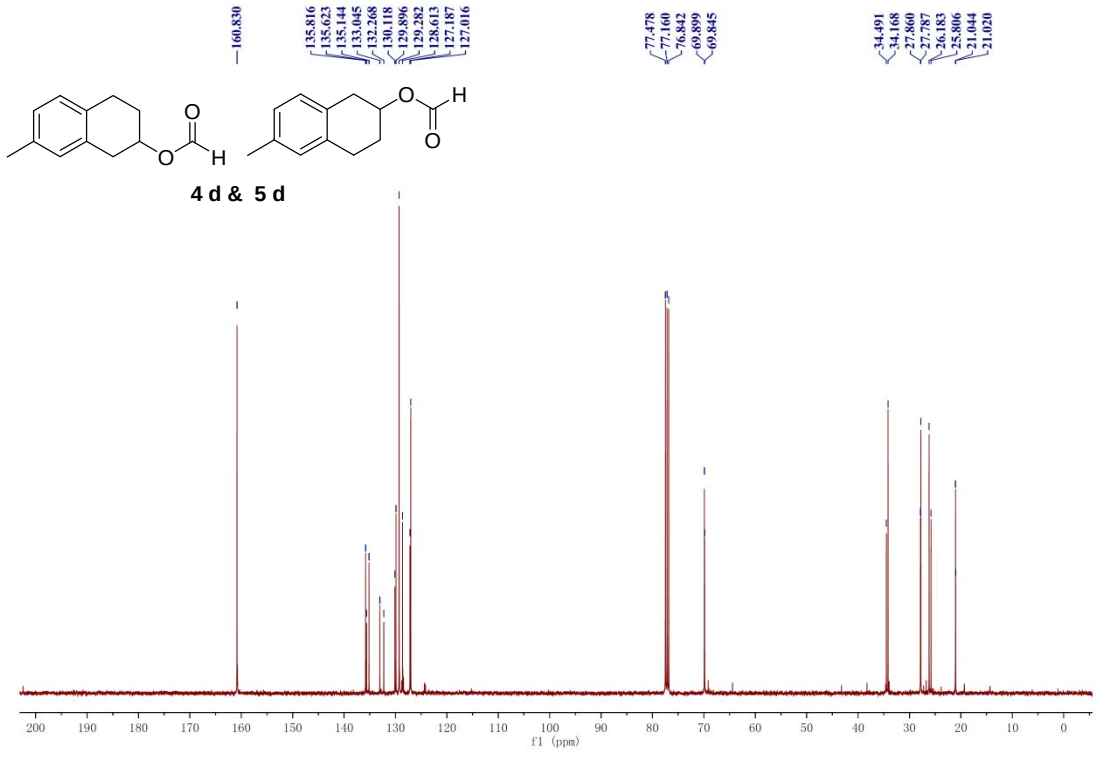
5.387
5.379
5.370
5.350
3.150
3.137
3.108
3.096
2.993
2.977
2.936
2.927
2.900
2.885
2.872
2.856
2.830
2.813
2.328
2.130
2.122
2.114
2.106
2.098
2.089
2.081
2.070
2.054
2.050
2.035
2.016
2.002
1.991
1.983
1.973
O
H
O
O
H
O
1.01
2.90
1.00
1.04
3.14
3.01
2.12
f1 (ppm)

4 d & 5 d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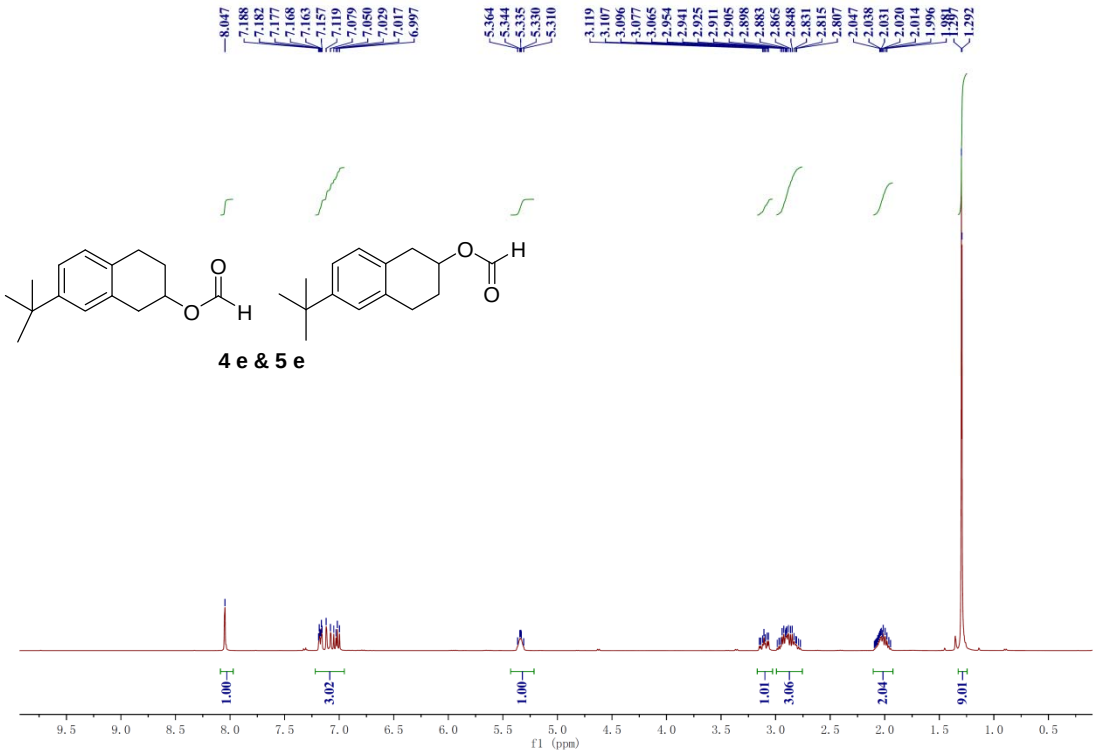
135.144
133.045
132.268
130.118
129.896
129.282
128.613
127.187
127.016
77.478
77.160
76.842
69.899
69.845
34.491
34.168
27.860
27.787
26.183
25.806
21.044
21.020
O
H
O
O
H
O
f1 (ppm)

4 d & 5 d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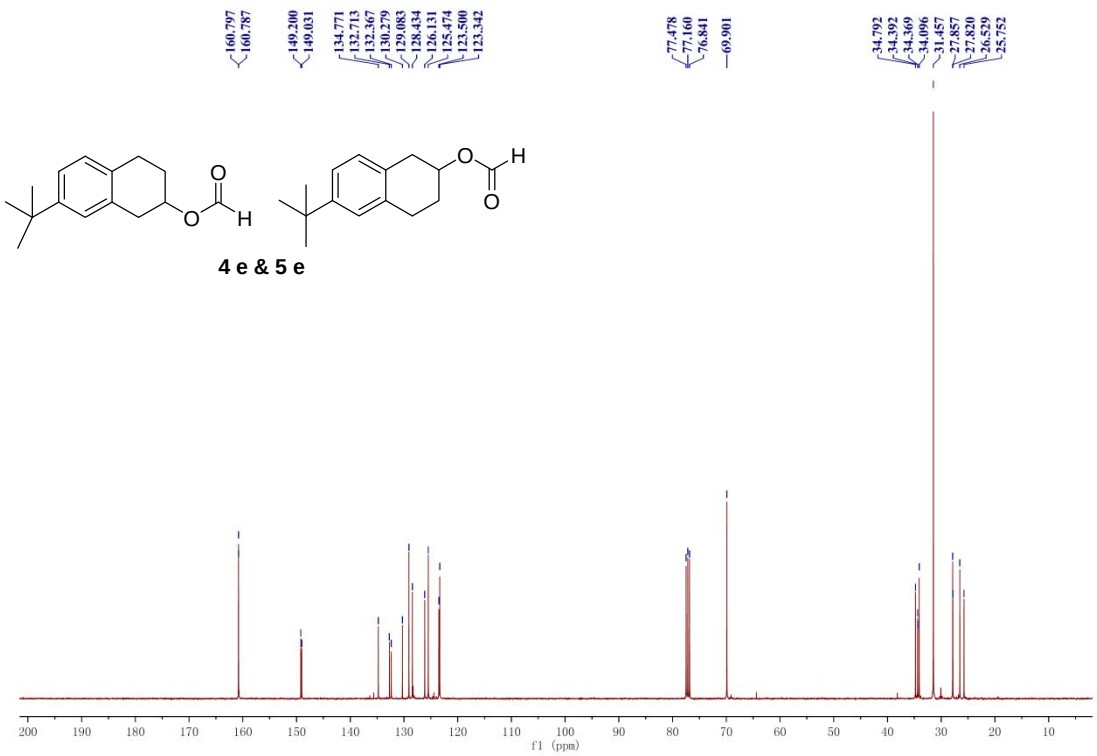
7.188
7.182
7.177
7.168
7.163
7.157
7.119
7.079
7.050
7.029
7.017
6.997
5.364
5.344
5.335
5.330
5.310
3.119
3.107
3.096
3.077
3.065
2.954
2.941
2.925
2.911
2.905
2.898
2.883
2.865
2.848
2.831
2.815
2.807
2.047
2.038
2.031
2.020
2.014
1.996
1.292
1.00
3.02
1.00
1.01
3.06
2.04
9.01
f1 (ppm)

4 e & 5 e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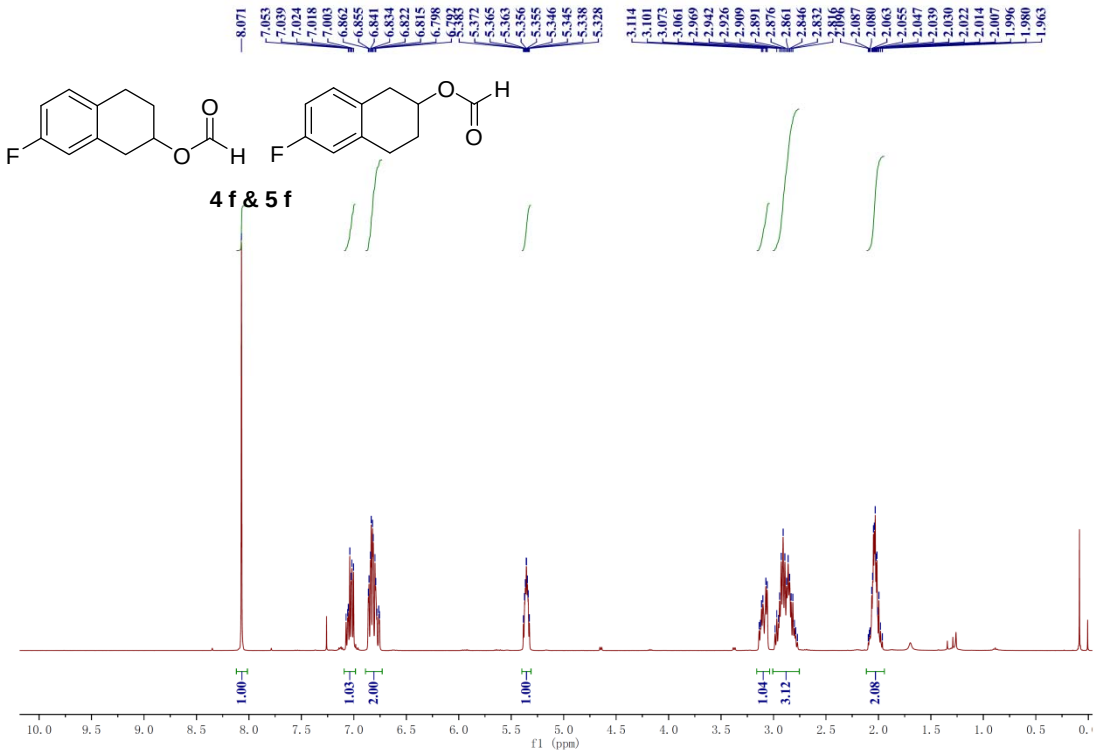
149.200
149.031
134.771
132.713
132.367
130.279
129.083
128.434
126.131
125.474
123.500
123.342
77.478
77.160
76.841
69.901
34.792
34.392
34.369
34.096
31.457
27.857
27.820
26.529
25.752
f1 (ppm)

4 e & 5 e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7.039
7.024
7.018
7.003
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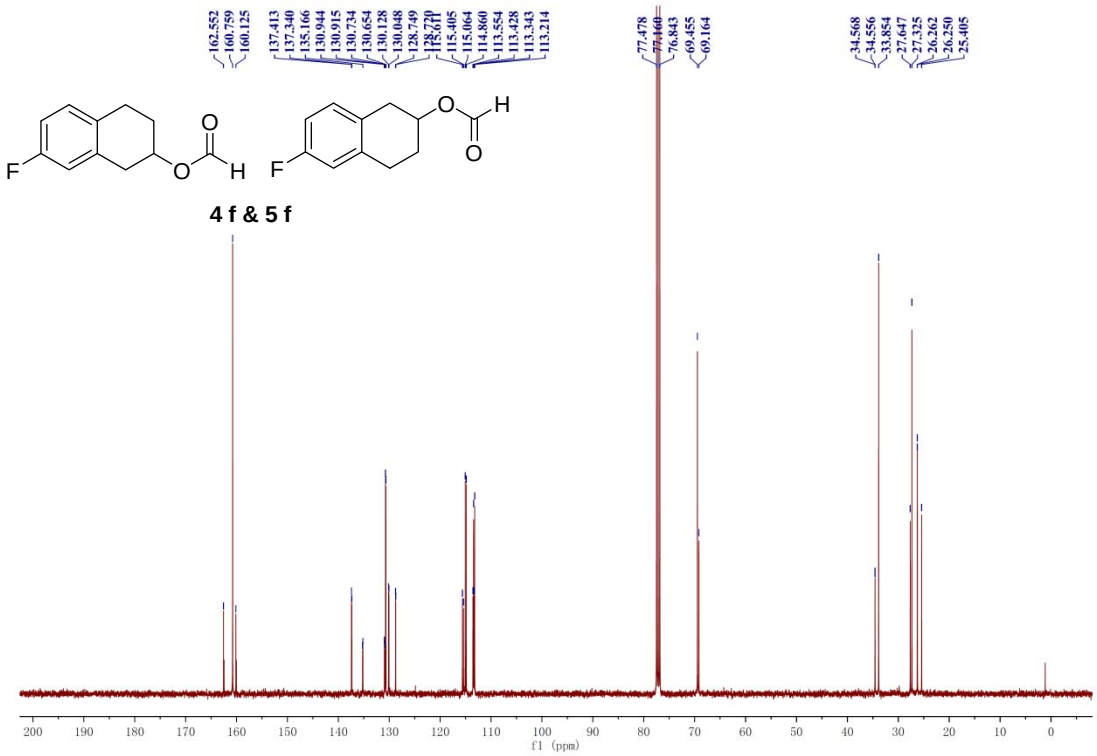
6.855
6.841
6.834
6.822
6.815
6.798
5.372
5.365
5.363
5.356
5.355
5.346
5.345
5.338
5.328
3.114
3.101
3.073
3.061
2.969
2.942
2.926
2.909
2.891
2.876
2.861
2.846
2.832
2.087
2.080
2.063
2.055
2.047
2.039
2.030
2.022
2.014
2.007
1.996
1.980
1.963
F
O
O
H
F
O
O
H
4 f & 5 f
1.00
1.03
2.00
1.00
1.04
3.12
2.08
f1 (ppm)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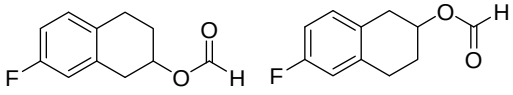
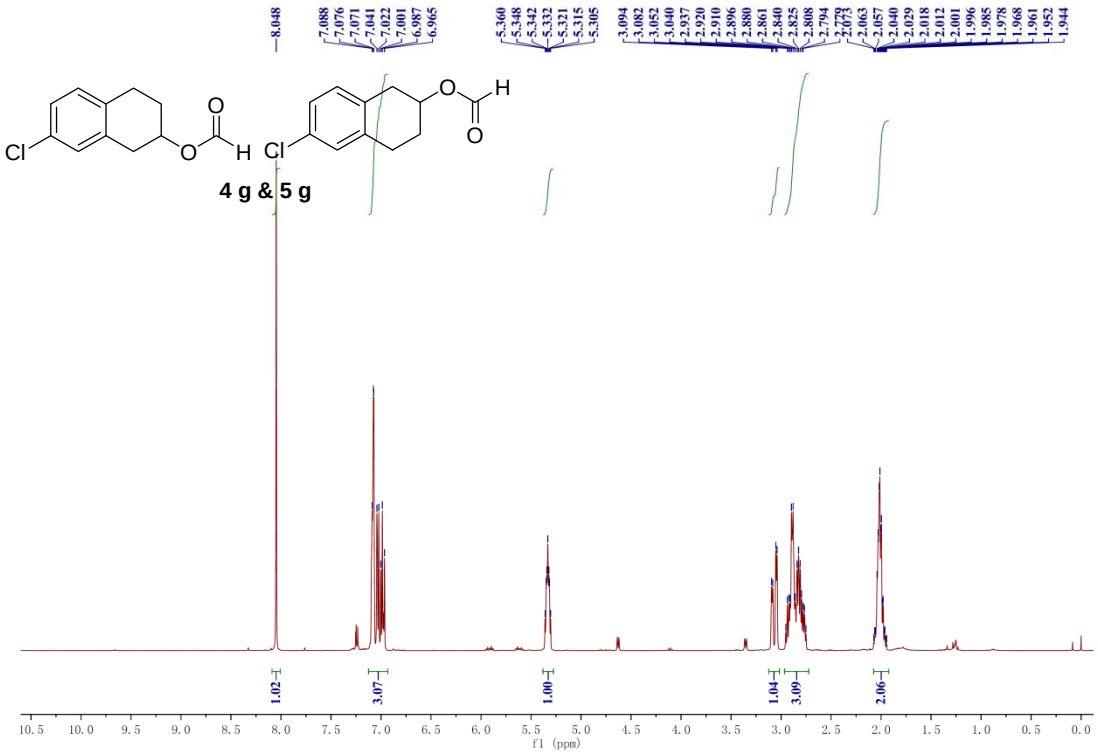
137.413
137.340
135.166
130.944
130.915
130.734
130.654
130.128
130.048
128.749
128.720
115.611
115.405
115.064
114.860
113.554
113.428
113.343
113.214
77.478
77.160
76.843
69.455
69.164
34.568
34.556
33.854
27.647
27.325
26.262
26.250
25.405
F
O
O
H
F
O
O
H
4 f & 5 f
f1 (ppm)



4 f & 5 f

-117.347
-117.554
5.47
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -20 -40 -60 -80 -100 -120 -140 -160 -180
f1 (ppm)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7.041
7.022
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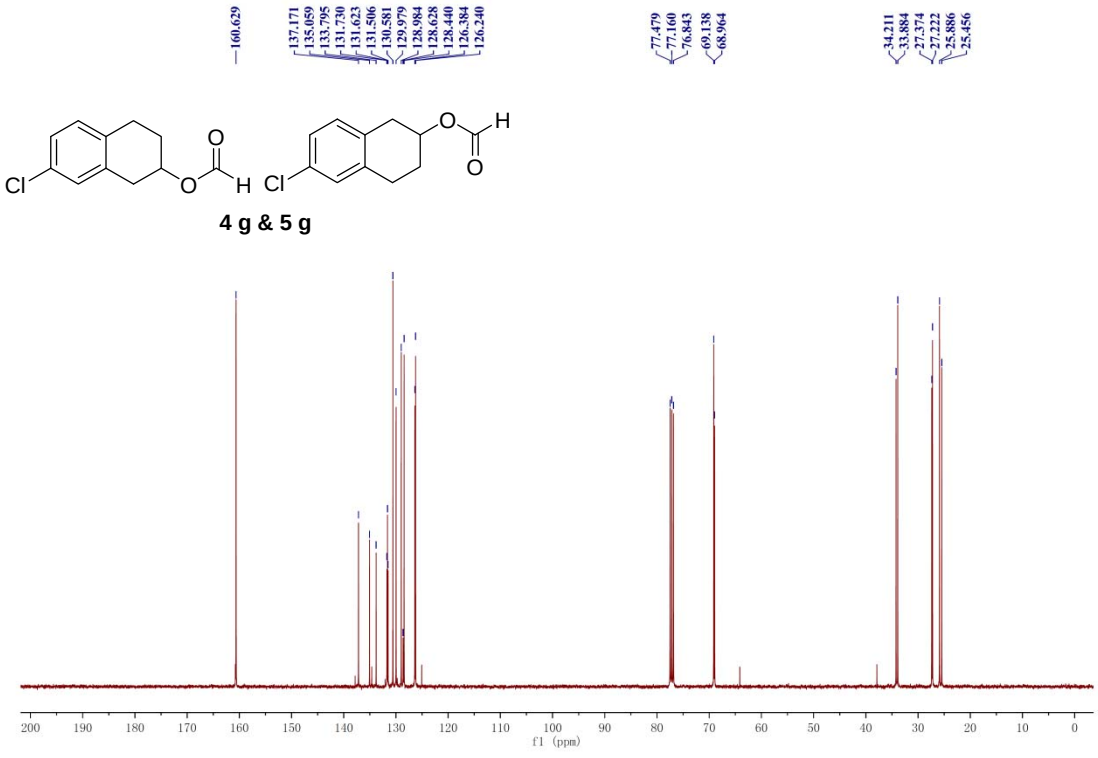
5.342
5.332
5.321
5.315
5.305
3.094
3.082
3.052
3.040
2.937
2.920
2.910
2.896
2.880
2.861
2.840
2.825
2.808
2.794
2.779
2.073
2.063
2.057
2.040
2.029
2.018
2.012
2.001
1.996
1.985
1.978
1.968
1.961
1.952
1.944
Cl
O
O
H
Cl
O
O
H
4 g & 5 g
1.02
3.07
1.00
1.04
3.09
2.06
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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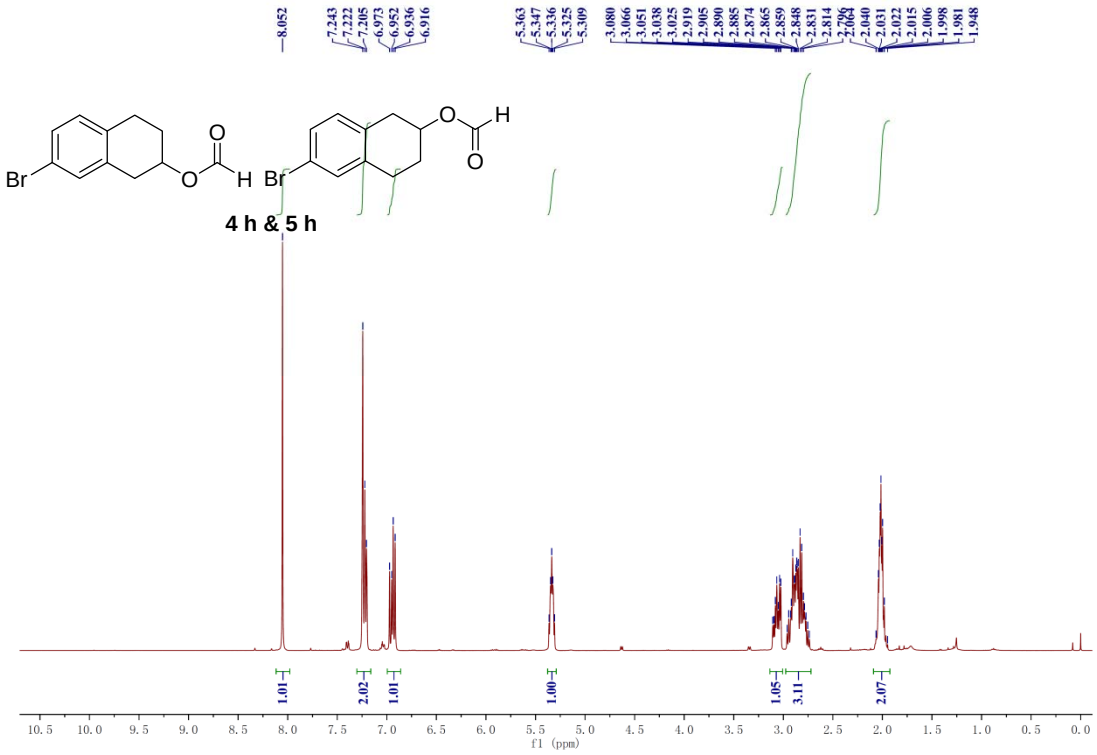
129.979
128.984
128.628
128.440
126.384
126.240
77.479
77.160
76.843
69.138
68.964
34.211
33.884
27.374
27.222
25.886
25.456
Cl
O
O
H
Cl
O
O
H
4 g & 5 g
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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6.916
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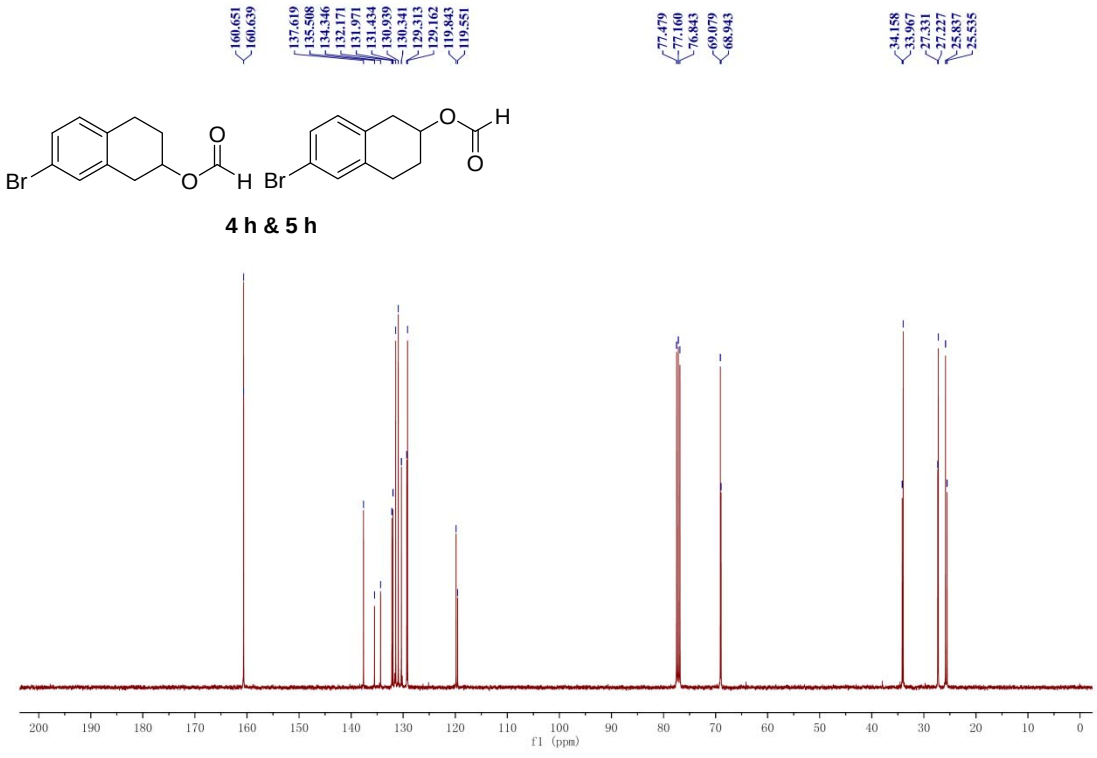
5.325
5.309
3.080
3.066
3.051
3.038
3.025
2.919
2.905
2.890
2.885
2.874
2.865
2.859
2.848
2.831
2.814
2.040
2.031
2.022
2.015
2.006
1.998
1.981
1.948
Br
O
H
Br
O
O
H
1.01
2.02
1.01
1.00
1.05
3.11
2.07
f1 (ppm)

4 h & 5 h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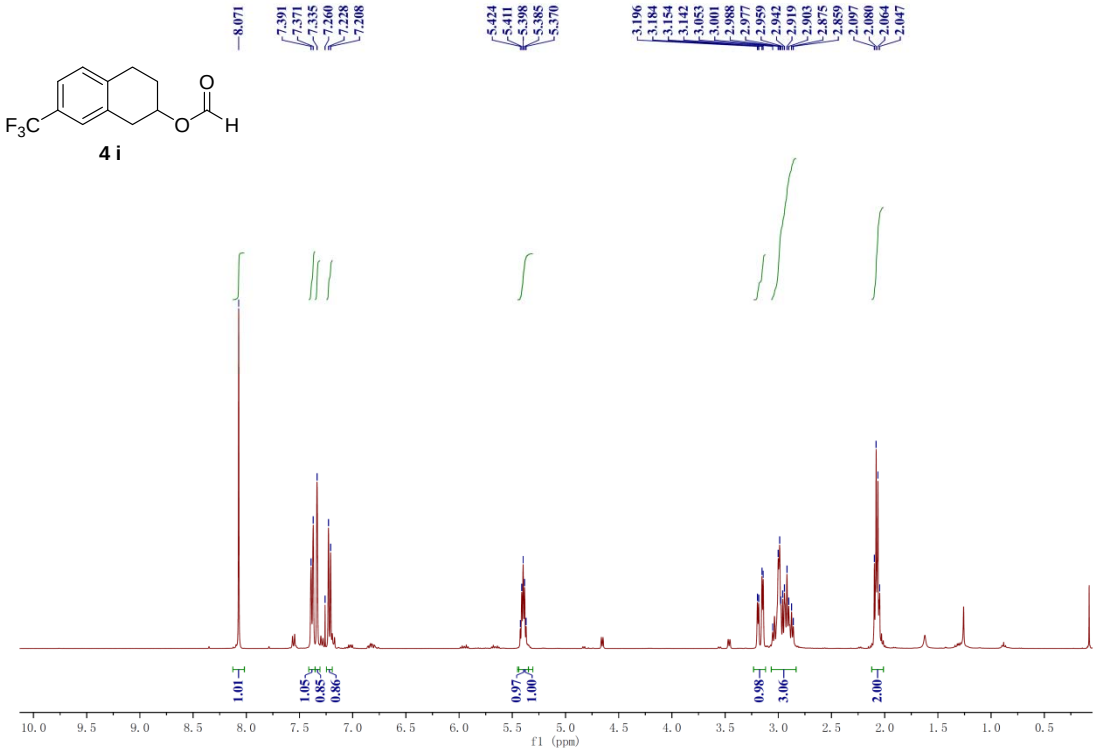
129.313
129.162
119.843
119.551
77.479
77.160
76.843
69.079
68.943
34.158
33.967
27.331
27.227
25.837
25.535
Br
O
H
Br
O
O
H
f1 (ppm)

4 h & 5 h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7.208
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5.411
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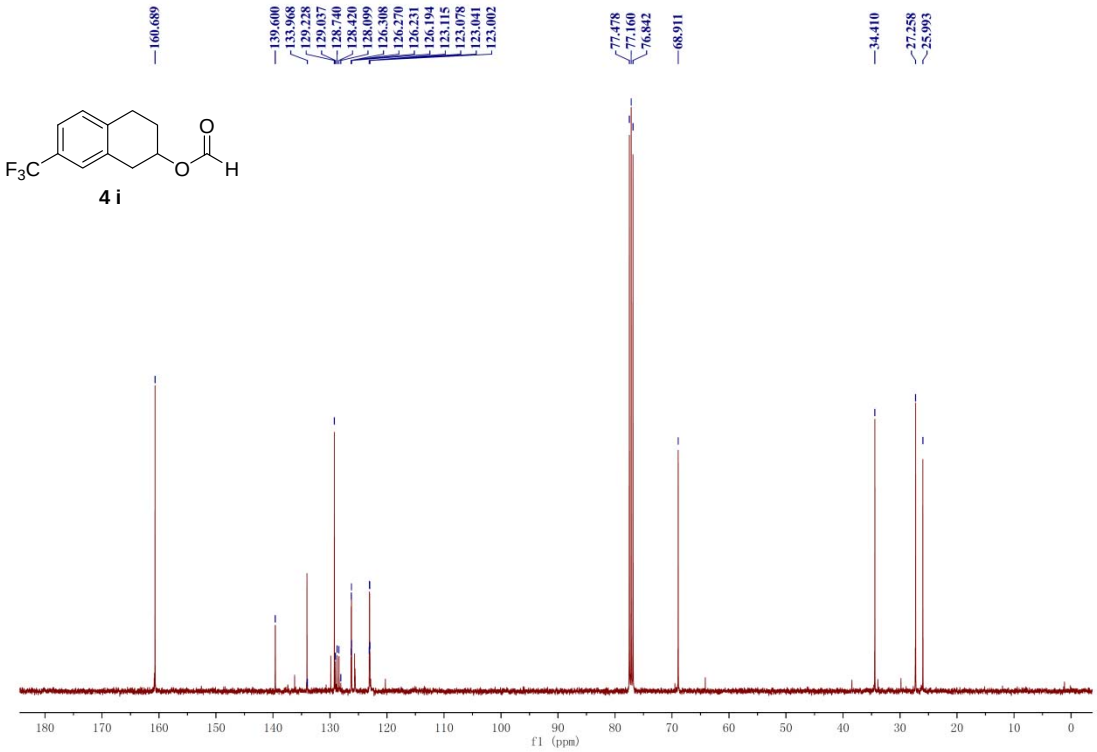
5.385
5.370
3.196
3.184
3.154
3.142
3.053
3.001
2.988
2.977
2.959
2.942
2.919
2.903
2.875
2.859
2.097
2.080
2.064
2.047
F3C
O
O
H
4 i
1.01
1.05
0.85
0.86
0.97
1.00
0.98
3.06
2.00
f1 (ppm)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128.420
128.099
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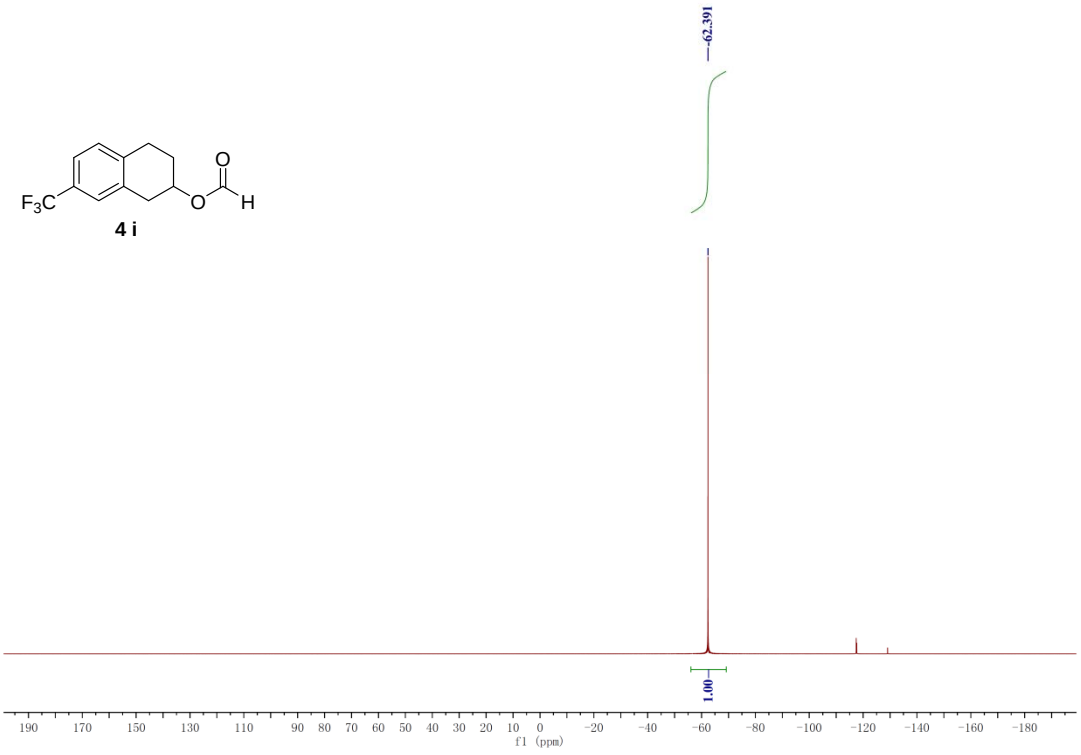
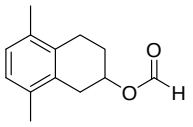
126.231
126.194
123.115
123.078
123.041
123.002
77.478
77.160
76.842
68.911
34.410
27.258
25.993
F3C
O
O
H
4 i
f1 (ppm)


F3C
O
O
H
4 i
62.391
1.00
190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -20 -40 -60 -80 -100 -120 -140 -160 -180
f1 (ppm)



4 j

8.132
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3.067
3.037
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2.896
2.880
2.869
2.853
2.837
2.828
2.810
2.785
2.771
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2.732
2.715
2.236
2.176
2.159
2.126
2.120
2.100
2.079
2.064
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2.011
0.96
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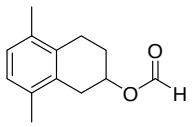
1.00
1.02
3.14
6.06
2.27
f1 (ppm)



4 j

160.881
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127.442
127.390
77.478
77.160
76.842
69.893
32.562
27.341
24.278
19.560
19.525
f1 (ppm)

6. Notes and References

1. Wang, Y.-F.; Gao, Y.-R.; Mao, S.; Zhang, Y.-L.; Guo, D.-D.; Yan, Z.-L.; Guo, S.-H.; Wang, Y.-Q. *Org. Lett*. **2014**, *7*, 1610.
2. Pan, J.-F.; Zhang, M.; Zhang, S.-L. *Org. Biomol. Chem*. **2012**, *10*, 1060.