Supporting Information

Palladium-catalyzed enantioselective domino ring-opening/Hiyama coupling of cyclobutanones: Development and application to the synthesis of (+)-herbertene-1,14-diol

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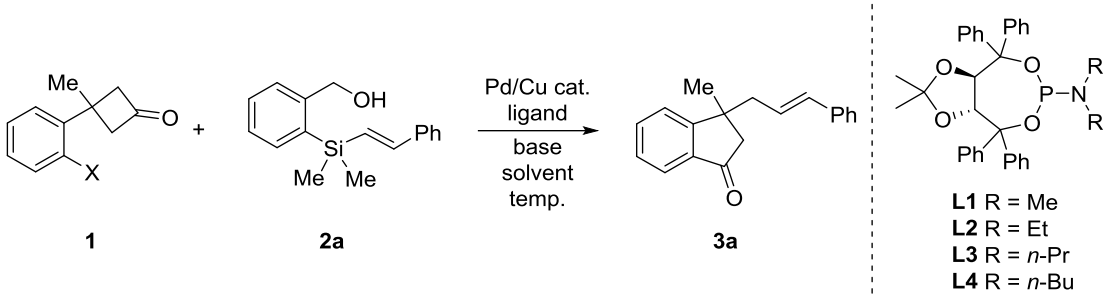
General information

Unless otherwise noted, all reactions were carried out under N_2 atmosphere. All reagents were from commercial sources and used as received without further purification. All solvents were dried by standard techniques and distilled prior to use. Column chromatography was performed on silica gel (300-400 meshes) using petroleum ether (bp. 60~90 °C) and ethyl acetate as eluent. ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded on a Bruker Avance (400 MHz) spectrometer, using $CDCl_3$ as the solvent and TMS as internal standard; chemical shifts were quoted in parts per million and *J* values were given in hertz. The following abbreviations were used to describe peak splitting patterns when appropriate: s = singlet, d = doublet, dd = double doublet, ddd = double doublet of doublets, t = triplet, dt = double triplet, q = quatriplet, m = multiplet, br = broad. High resolution mass spectrometry (HRMS) was performed on a Waters Micromass. HPLC was carried out on an Agilent 1260 infinity instrument. Cyclobutanones[1] and alkenyl[2-(hydroxymethyl)phenyl] dimethylsilanes[2] were prepared according to the reported methods.

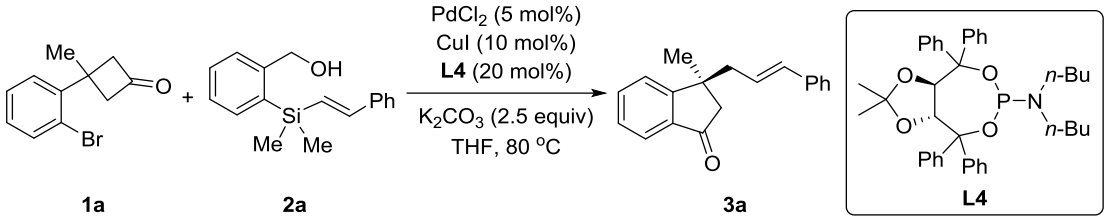
Table S1: Optimization of reaction conditions.[a]



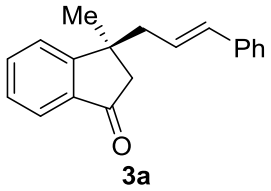
Entry	Catalyst	Ligand	Base	Temp (°C)	Solvent	X	Yield	er
1	$PdCl_2$/CuI	**L1**	K_2CO_3	80	THF	Br	35	59.5:40.5
2	$PdCl_2$/CuI	**L2**	K_2CO_3	80	THF	Br	48	94.5:5.5
3	$PdCl_2$/CuI	**L3**	K_2CO_3	80	THF	Br	77	95:5
4	***$PdCl_2$/CuI***	***L4***	***K_2CO_3***	***80***	***THF***	***Br***	***92***	***96.5:3.5***
5	$PdCl_2$/CuI	**L4**	K_2CO_3	80	THF	I	82	96.5:3.5
6	$PdCl_2$	**L4**	K_2CO_3	80	THF	Br	70	91.5:8.5
7	CuI	**L4**	K_2CO_3	80	THF	Br	trace	-
8	$PdCl_2$/CuI	**L4**	Na_2CO_3	80	THF	Br	trace	-
9	$PdCl_2$/CuI	**L4**	Cs_2CO_3	80	THF	Br	14	95.5:4.5
10	$PdCl_2$/CuI	**L4**	K_3PO_4	80	THF	Br	69	95.5:4.5
11	$PdCl_2$/CuI	**L4**	K_2CO_3	80	1,4-dioxane	Br	80	93.5:6.5
12	$PdCl_2$/CuI	**L4**	K_2CO_3	80	toluene	Br	62	95.5:4.5
13	$PdCl_2$/CuI	**L4**	K_2CO_3	80	DCE	Br	80	96:4
14	$PdCl_2$/CuI	**L4**	K_2CO_3	40	THF	Br	49	96:4
15	$PdCl_2$/CuI	**L4**	K_2CO_3	60	THF	Br	80	95.5:4.5
16	$PdBr_2$/CuI	**L4**	K_2CO_3	80	THF	Br	59	94.5:5.5
17	$Pd(OAc)_2$/CuI	**L4**	K_2CO_3	80	THF	Br	32	94:6

[a] Reaction conditions: **1** (0.2 mmol), **2a** (0.24 mmol), Pd catalyst (0.01 mmol), CuI (0.02 mmol), ligand (0.04 mmol), base (0.5 mmol), solvent (2 mL) for 24 h, isolated yield.

Typical procedure for the synthesis of 3a.

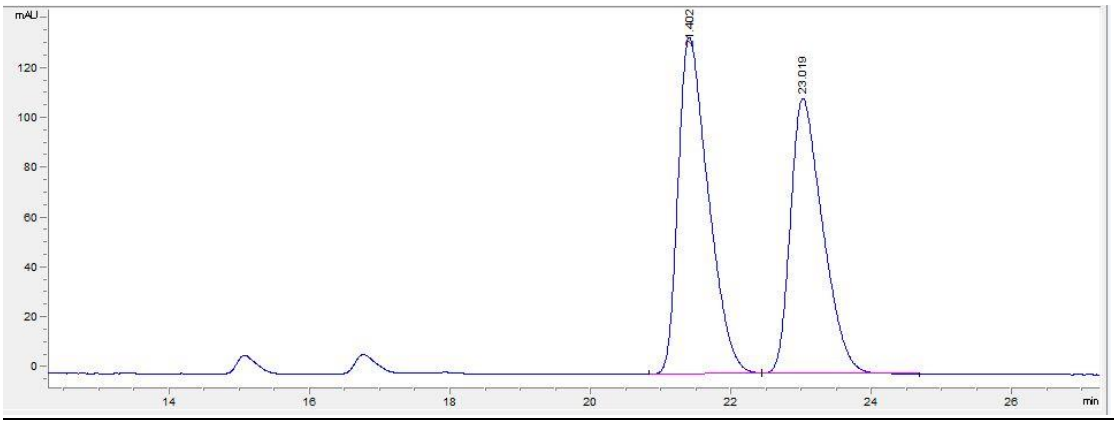


A vial was charged with $PdCl_2$ (1.8 mg, 0.01 mmol), CuI (3.8 mg, 0.02 mmol), **L4** (25.0 mg, 0.04 mmol), and K_2CO_3 (69.1 mg, 0.5 mmol), and evacuated under high vacuum and backfilled with N_2. THF (1 mL) was added via syringe and the mixture was stirred at room temperature for 20 min. A solution of **1a** (47.8 mg, 0.2 mmol) and **2a** (64.3 mg, 0.24 mmol) in THF (1 mL) was added via syringe and the mixture was stirred at 80 °C in an oil bath for 24 h, and then cooled to room temperature. The mixture was filtered over a plug of silica gel (washed with 50 mL EtOAc), and the filtrate was concentrated under reduced pressure and then purified by silica column to get the product **3a**.



3-cinnamyl-3-methyl-2,3-dihydro-1*H*-inden-1-one

The mobile phase for flash chromatography: hexane/ethyl acetate = 60:1. Colorless oil (48.4 mg, 92%). $[\alpha]_D^{25}$ = + 49.3 (c = 0.067, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$): δ 7.72 (d, *J* = 7.6 Hz, 1H), 7.66-7.62 (m, 1H), 7.53 (d, *J* = 7.6 Hz, 1H), 7.41-7.37 (m, 1H), 7.28-7.17 (m, 5H), 6.39 (d, *J* = 15.6 Hz, 1H), 5.95 (dt, *J* = 15.6, 7.6 Hz, 1H), 2.79 (d, *J* = 18.8 Hz, 1H), 2.62-2.54 (m, 2H), 2.47 (d, *J* = 18.8 Hz, 1H), 1.48 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 205.6, 162.2, 137.3, 136.3, 135.0, 134.1, 128.6, 127.8, 127.5, 126.3, 125.7, 124.1, 123.6, 50.0, 45.9, 42.5, 28.2. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{19}H_{18}NaO$, 285.1250; found 285.1241. Enantiomeric excess was determined by HPLC with double Chiralpak OD-H column (hexane:2-propanol = 95:5, 1.0 mL/min, 254 nm, 96.5:3.5 *er*); major enantiomer t_r = 21.1 min, minor enantiomer t_r = 22.9 min.



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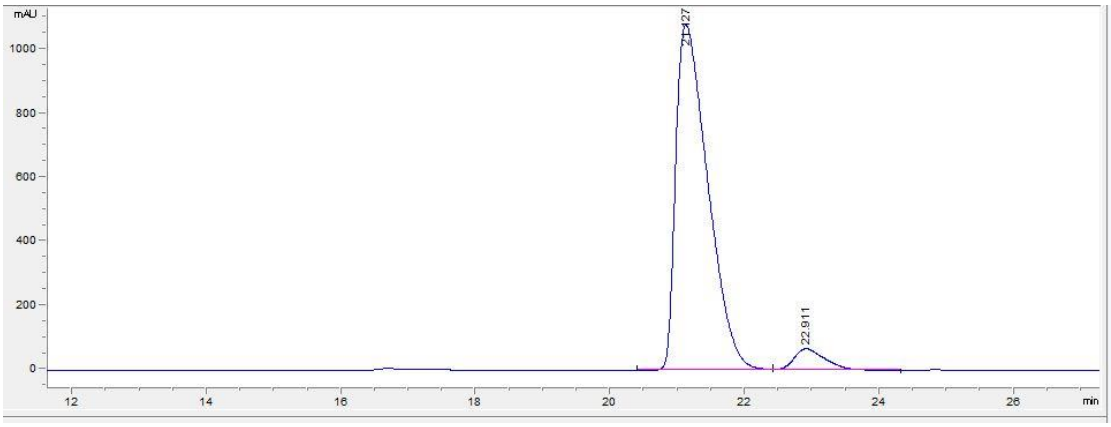
2	23.019	4183.9	120.3	48.649



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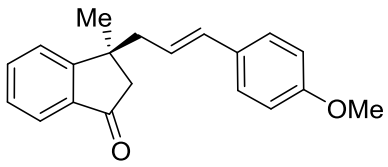
2	22. 91	4721.3	142.8	3.577

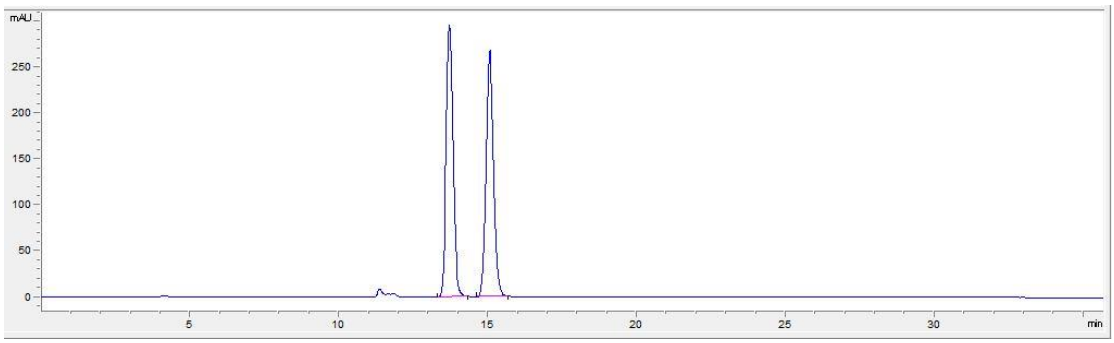


3b

(*E*)-3-(3-(4-methoxyphenyl)allyl)-3-methyl-2,3-dihydro-1*H*-inden-1-one

The mobile phase for flash chromatography: hexane/ethyl acetate = 20:1. Colorless oil. (31.0 mg, 53%). $[\alpha]_D^{25}$ = + 29.2 (c = 0.34, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 7.71 (d, *J* = 7.6 Hz, 1H), 7.63 (t, *J* = 7.6 Hz, 1H), 7.52 (d, *J* = 7.6 Hz, 1H), 7.39 (t, *J* = 8.4 Hz, 1H), 7.17 (d, *J* = 8.4 Hz, 2H), 6.80 (d, *J* = 8.4 Hz, 2H), 6.32 (d, *J* = 15.6 Hz, 1H),

5.80 (td, *J* = 15.6, 7.6 Hz, 1H), 3.78 (s, 3H), 2.79 (d, *J* = 18.8 Hz, 1H), 2.55 (d, *J* = 6.8 Hz, 2H), 2.46 (d, *J* = 18.8 Hz, 1H), 1.47 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.8, 162.3, 159.1, 136.2, 135.0, 133.4, 130.1, 127.7, 127.4, 124.1, 123.5, 123.4, 114.0, 55.4 , 50.0, 45.9, 42.6, 28.1. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{20}H_{20}NaO_2$, 315.1356; found 315.1349. Enantiomeric excess was determined by HPLC with a Chiralpak OX-H column (hexanes: 2-propanol = 90:10, 0.8 mL/min, 210 nm, 95:5 er); major enantiomer tr = 13.7 min, minor enantiomer tr = 15.0 min.



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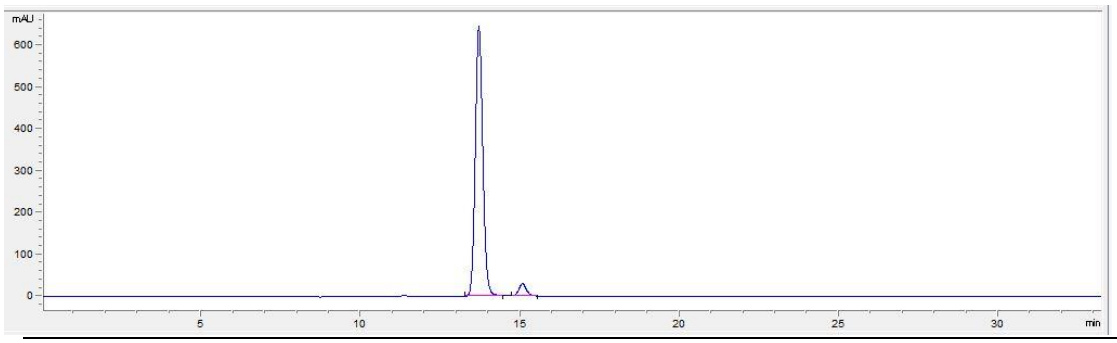

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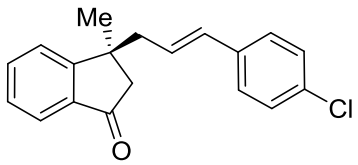
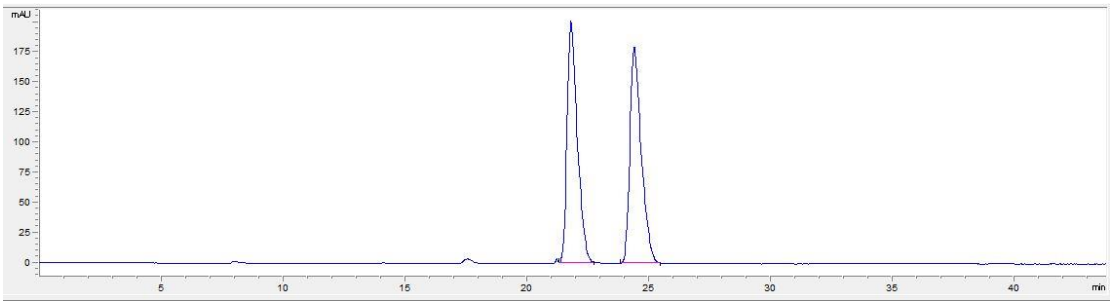
1	13.711	10346.3	644	95.0
2	15.07	529.4	30.5	5.0



3c

(*E*)-3-(3-(4-chlorophenyl)allyl)-3-methyl-2,3-dihydro-1*H*-inden-1-one

The mobile phase for flash chromatography: hexane/ethyl acetate = 60:1. Colorless oil. (38.0 mg, 65%). $[\alpha]_D^{25}$ = + 27.6 (c = 1.02, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 7.71 (d, *J* = 7.6 Hz, 1H), 7.64 (t, *J* = 7.6 Hz, 1H), 7.51 (d, *J* = 7.6 Hz, 1H), 7.39 (t, *J* = 7.6 Hz, 1H), 7.21 (d, *J* = 8.4 Hz, 2H), 7.14 (d, *J* = 8.4 Hz, 2H), 6.32 (d, *J* = 15.6 Hz, 1H), 5.90 (td, *J* = 15.6, 7.6 Hz, 1H), 2.77 (d, *J* = 18.8 Hz, 1H), 2.57 (d, *J* = 7.6 Hz, 2H), 2.47 (d, *J* = 18.8 Hz, 1H), 1.48 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.5, 162.0, 136.2, 135.6, 135.1, 133.0, 132.8, 128.7, 127.8, 127.4, 126.3, 124.0, 123.6, 50.0, 45.9, 42.5, 28.1. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{19}H_{17}ClNaO$, 319.0860; found 319.0851. Enantiomeric excess was determined by HPLC with double Chiralpak OX-H column(hexanes:2-propanol = 95:5, 0.8 mL/min, 210 nm, 95.5:4.5 er); major enantiomer tr = 21.9 min, minor enantiomer tr = 24.5 min.



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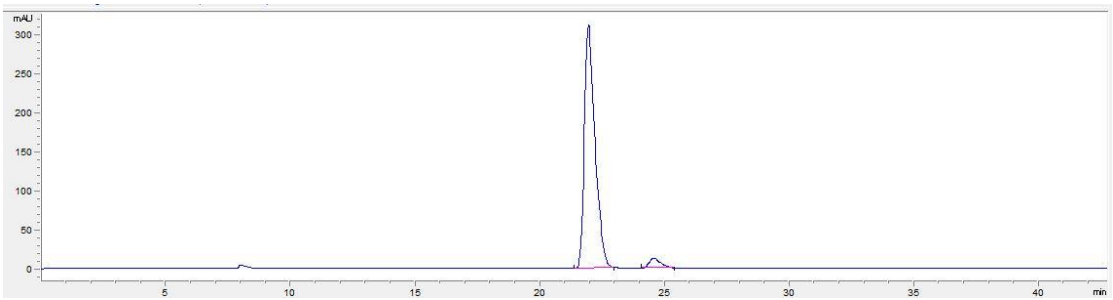
1	21.793	5835.9	201.1	50.0
2	24.387	5848.9	180.1	50.0



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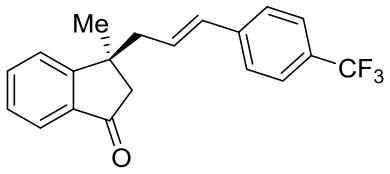
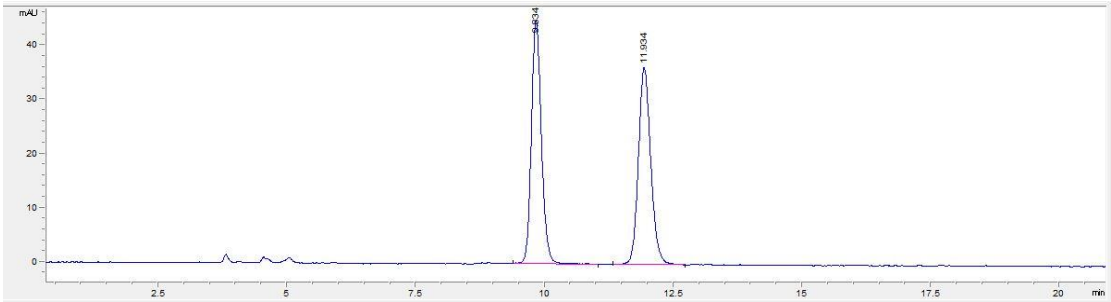
1	21.936	9164.6	311.2	95.5
2	24.549	413.1	12.8.	4.5



3d

(*S*,*E*)-3-methyl-3-(3-(4-(trifluoromethyl)phenyl)allyl)-2,3-dihydro-1*H*-inden-1-one

The mobile phase for flash chromatography: hexane/ethyl acetate = 50:1. Yellow oil (50 mg, 75%). $[\alpha]_D^{25}$ = + 12.3 (c = 2.83, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, J = 7.6 Hz, 1H), 7.65 (t, J = 7.6 Hz, 1H), 7.57-7.47 (m, 3H), 7.40 (t, J = 7.6 Hz, 1H), 7.31 (d, J = 8.4 Hz, 2H), 6.41 (d, J = 16.0 Hz, 1H), 6.02 (dt, J =16.0, 7.6 Hz, 1H), 2.77 (d, J = 18.8 Hz, 1H), 2.61 (d, J = 7.6 Hz, 2H), 2.49 (d, J = 18.8 Hz, 1H), 1.50 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.4, 161.8, 140.6, 136.2, 135.1, 132.8, 129.3 (q, J = 32.2 Hz), 128.5, 127.9, 126.4, 125.6 (q, J = 2.9 Hz), 124.3 (q, J = 250.5 Hz), 124.0, 123.6, 50.0, 45.9, 42.5, 28.2. HRMS (ESI-TOF) m/z: $[M + H]^+$ Calcd for $C_{20}H_{18}F_3O$, 331.1304; found 331.1328. Enantiomeric excess was determined by HPLC with a chiralcel OD column (hexanes: 2-propanol = 95:5, 0.8 mL/min, 230 nm, 94.5:5.5 er); major enantiomer tr = 9.8 min, minor enantiomer tr = 11.9 min.



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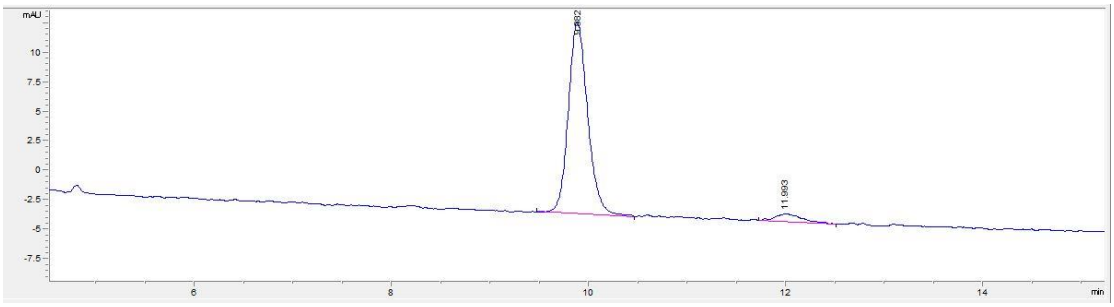

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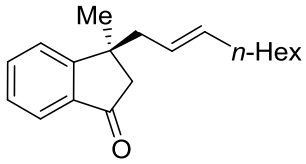
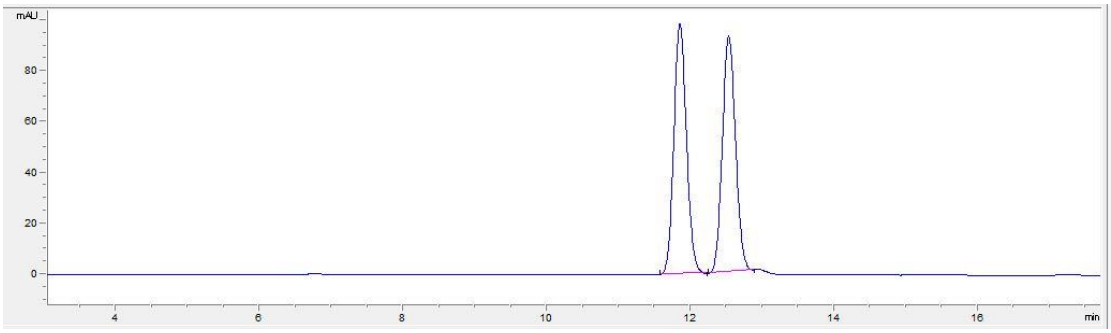
2	11.993	13.6	6.8E-1	5.5



3e

(*E*)-3-methyl-3-(non-2-en-1-yl)-2,3-dihydro-1*H*-inden-1-one

The mobile phase for flash chromatography: hexane/ethyl acetate = 60:1. Colorless oil (41.3 mg, 77%). $[\alpha]_D^{25}$ = - 9.1 (c = 1.01, $CHCl_3$). ^1H NMR (500 MHz, $CDCl_3$) δ 7.68 (d, *J* = 7.5 Hz, 1H), 7.60 (t, *J* = 7.5 Hz, 1H), 7.46 (d, *J* = 7.5 Hz, 1H), 7.36 (t, *J* = 7.5 Hz, 1H), 5.43-5.36 (m, 1H), 5.14-5.07 (m, 1H), 2.71 (d, *J* = 15.2 Hz, 1H), 2.39 (d, *J* = 15.2 Hz, 1H), 2.35 (d, *J* = 6.0 Hz, 2H), 1.93-1.87 (m, 2H), 1.40 (s, 3H), 1.27-1.16 (m, 8H), 0.86 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.0, 162.5, 136.3, 135.4, 134.9, 127.5, 125.1, 124.0, 123.4, 49.8, 45.5, 42.3, 32.6, 31.8, 29.4, 28.8, 28.2, 22.7, 14.2. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{19}H_{26}NaO$, 293.1876; found 293.1869. Enantiomeric excess was determined by HPLC with a Chiralpak OX-H column (hexane: 2-propanol = 98:2, 0.5 mL/min, 254 nm, 94:6 er); major enantiomer tr = 12.3 min, minor enantiomer tr = 11.8 min.



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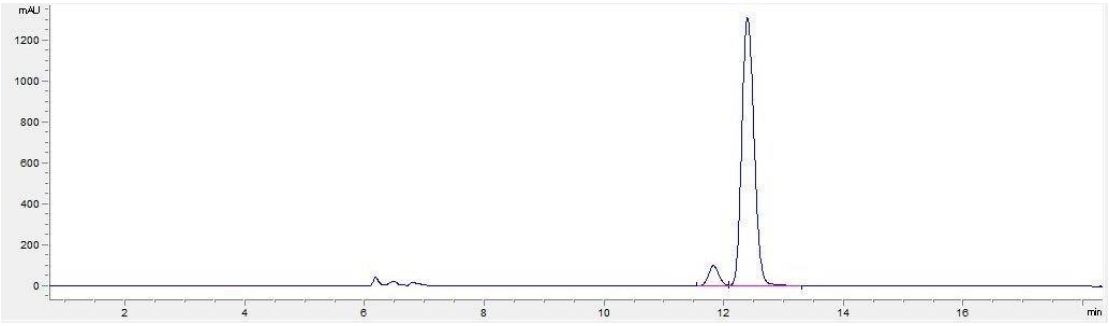

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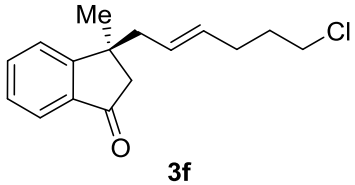
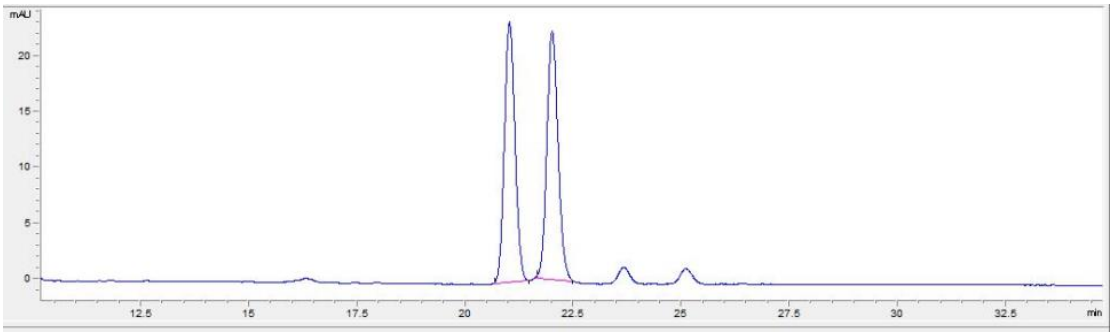
1	11.819	1223	99.8	6.116
2	12.389	18773.5	1308.3	93.884



3f

(*E*)-3-(6-chlorohex-2-en-1-yl)-3-methyl-2,3-dihydro-1*H*-inden-1-one

The mobile phase for flash chromatography: hexane/ethyl acetate = 60:1. Colorless oil (43.8 mg, 84%). $[\alpha]_D^{25}$ = + 19.3 (c = 0.34, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.6 Hz, 1H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.46 (d, *J* = 7.6 Hz, 1H), 7.37 (d, *J* = 7.6 Hz, 1H), 5.35 (dt, *J* = 15.2, 6.8 Hz, 1H), 5.17 (dt, *J* = 15.2, 6.8 Hz, 1H), 3.39 (t, *J* = 6.4 Hz, 1H), 2.69 (d, *J* = 18.8 Hz, 1H), 2.44-2.35 (m, 3H), 2.09-2.03 (m, 2H), 1.74-1.66 (m, 2H), 1.42 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.9, 162.3, 136.3, 135.0, 133.1, 127.7, 126.9, 124.0, 123.4, 49.8, 45.5, 44.3, 42.2, 32.1, 29.7, 28.2. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{16}H_{19}ClNaO$, 285.1017; found 285.1008. Enantiomeric excess was determined by HPLC with double Chiralpak OX-H column (hexane:2-propanol 95:5, 0.8 mL/min, 290 nm, 96:4 er); major enantiomer tr = 21.0 min, minor enantiomer tr = 22.0 min.



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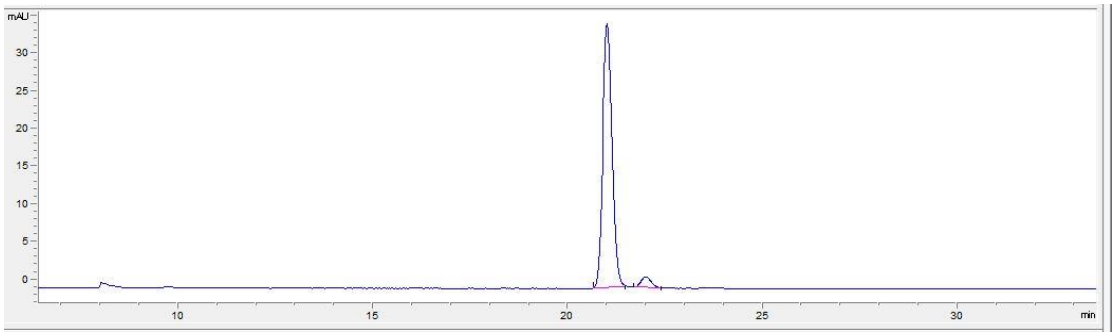

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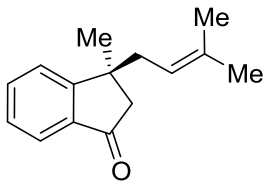
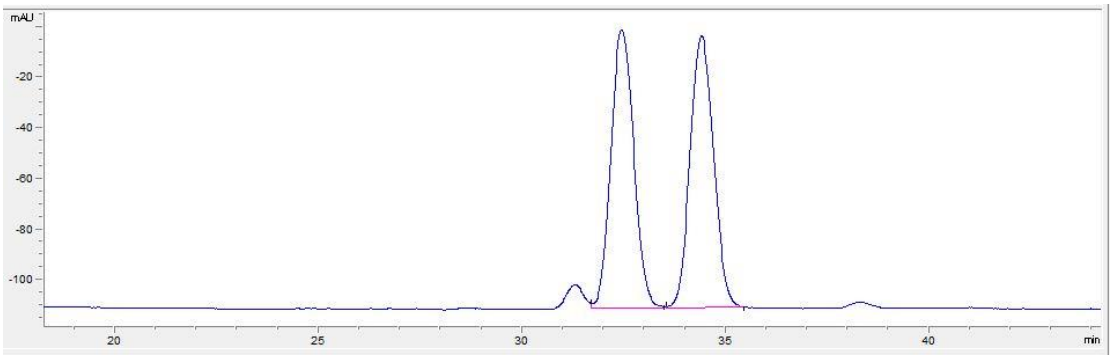
1	21.009	602	34.9	96.0
2	21.988	24	1.4	4.0



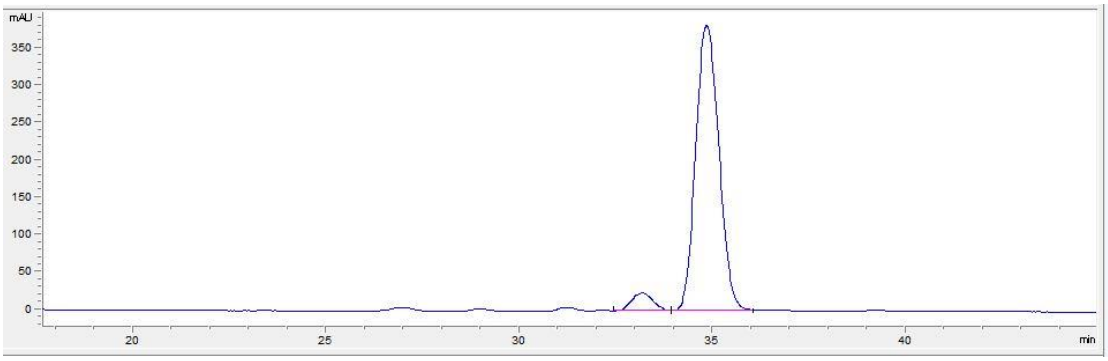
3g

3-methyl-3-(3-methylbut-2-en-1-yl)-2,3-dihydro-1*H*-inden-1-one

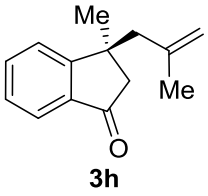
The mobile phase for flash chromatography: hexane/ethyl acetate = 60:1. Colorless oil. (22.0 mg, 51%). $[\alpha]_D^{25}$ = + 39.2 (c = 0.34, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 7.6 Hz, 1H), 7.60 (t, *J* = 7.6 Hz, 1H), 7.48 (d, *J* = 7.6 Hz, 1H), 7.36 (t, *J* = 7.6 Hz, 1H), 4.91-4.84 (m, 1H), 2.65 (d, *J* = 18.4 Hz, 1H), 2.47-2.28 (m, 3H), 1.61 (s, 3H), 1.52 (s, 3H), 1.42 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.3, 162.7, 136.2, 135.2, 134.9, 127.5, 124.0, 123.4, 119.8, 50.1, 42.7, 40.5, 28.2, 26.0, 18.0. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{15}H_{18}NaO$, 237.1250; found 237.1237. Enantiomeric excess was determined by HPLC with double Chiralpak OX-H column (hexanes:2-propanol = 95:5, 1 mL/min, 210 nm, 94.5:5.5 er); major enantiomer tr = 34.8 min, minor enantiomer tr = 33.1 min.



	Time/min	Area	Height	Area%
1	32.447	4317.5	110.2	50.0
2	34.403	4316.3	107.4	50.0



	Time/min	Area	Height	Area%
1	33.156	907.5	23.7	5.5
2	34.841	15902.5	381.2	94.5

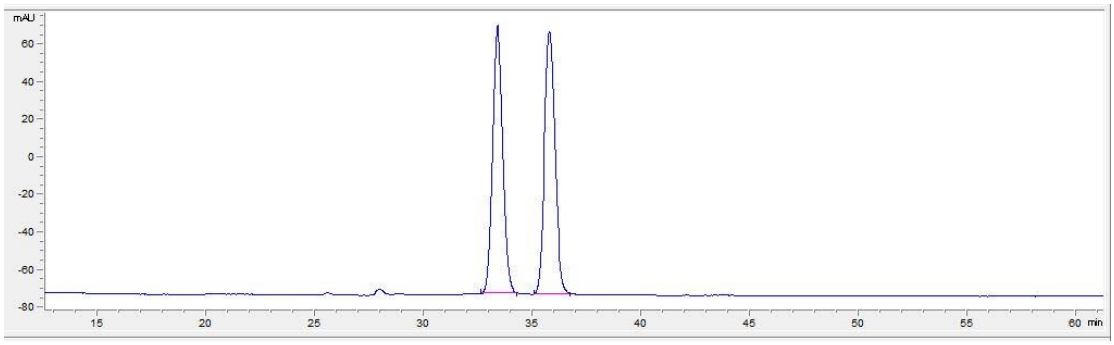


3h

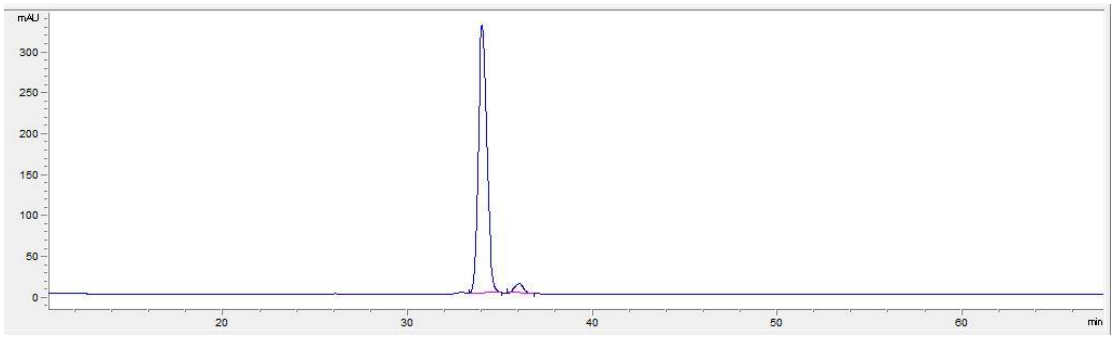
3-methyl-3-(2-methylallyl)-2,3-dihydro-1*H*-inden-1-one

The mobile phase for flash chromatography: hexane/ethyl acetate = 60:1. Colorless oil. (31.9 mg, 80%). $[\alpha]_D^{25}$ = + 20.8 (c = 0.99, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 8.0 Hz, 1H), 7.60 (t, *J* = 8.0 Hz, 1H), 7.53 (d, *J* = 8.0 Hz, 1H), 7.34 (t, *J* = 8.0 Hz, 1H), 4.80 (s, 1H), 4.63 (s, 1H), 2.83 (d, *J* = 18.8 Hz, 1H), 2.49-2.39 (m, 3H), 1.44 (s, 3H), 1.33 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.1, 162.7, 142.4, 136.1, 134.8, 127.7, 124.2, 123.5, 115.9, 50.0, 42.0, 29.5, 24.2. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{14}H_{16}NaO$, 223.1093; found 223.1086. Enantiomeric excess was determined

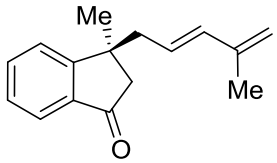
by HPLC with double Chiralpak OX-H column (hexanes: 2-propanol = 90:10, 0.8 mL/min, 254 nm, 96.5:3.5 er); major enantiomer tr = 34.0 min, minor enantiomer tr = 35.0 min.



	Time/min	Area	Height	Area%
1	33.378	4666.9	142.6	50.0
2	35.768	4709.9	139.7	50.0



	Time/min	Area	Height	Area%
1	34.013	10657.4	327	96.5
2	35.994	396.1	12	3.5

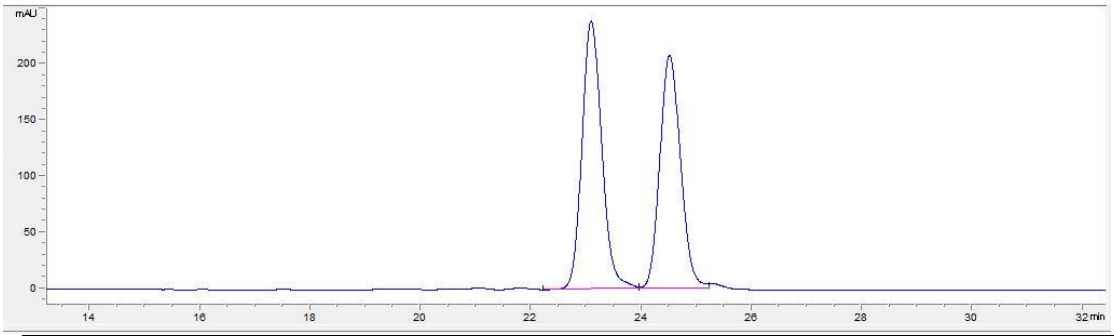


3i

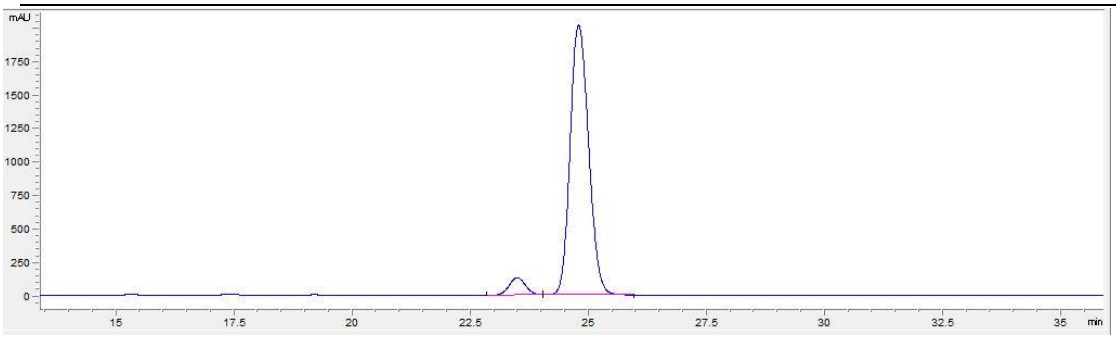
(*E*)-3-methyl-3-(4-methylpenta-2,4-dien-1-yl)-2,3-dihydro-1*H*-inden-1-one

The mobile phase for flash chromatography: hexane/ethyl acetate = 60:1. Colorless oil (37.9 mg, 84%). $[\alpha]_D^{25}$ = + 39.4 (c = 0.35, $CHCl_3$). ^1H NMR (500 MHz, $CDCl_3$) δ 7.70 (d, *J* = 7.5 Hz, 1H), 7.62 (t, *J* = 7.5 Hz, 1H), 7.48 (d, *J* = 7.5 Hz, 1H), 7.37 (t, *J* = 7.5

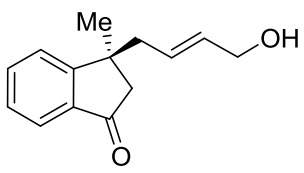
Hz, 1H), 6.11 (d, *J* = 15.5 Hz, 1H), 5.38-5.32 (m, 1H), 4.86 (d, *J* = 10.5 Hz, 2H), 2.72 (d, *J* = 18.5 Hz, 1H), 2.47 (d, *J* = 7.0 Hz, 2H), 2.42 (d, *J* = 18.5 Hz, 1H), 1.69 (s, 3H), 1.44 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.8, 162.3, 141.7, 137.0, 136.3, 135.0, 127.7, 125.3, 124.0, 123.5, 115.8, 50.0, 45.7, 42.5, 28.1, 18.7. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{16}H_{18}NaO$, 249.1250; found 249.1265. Enantiomeric excess was determined by HPLC with a Chiralpak OX-H column (hexane:2-propanol 90:10, 0.8 mL/min, 254 nm, 94.5:5.5 er); major enantiomer tr = 24.8 min, minor enantiomer tr = 23.5 min.



	Time/min	Area	Height	Area%
1	23.094	6014.1	239.2	52.349
2	24.514	5474.4	208.3	47.651



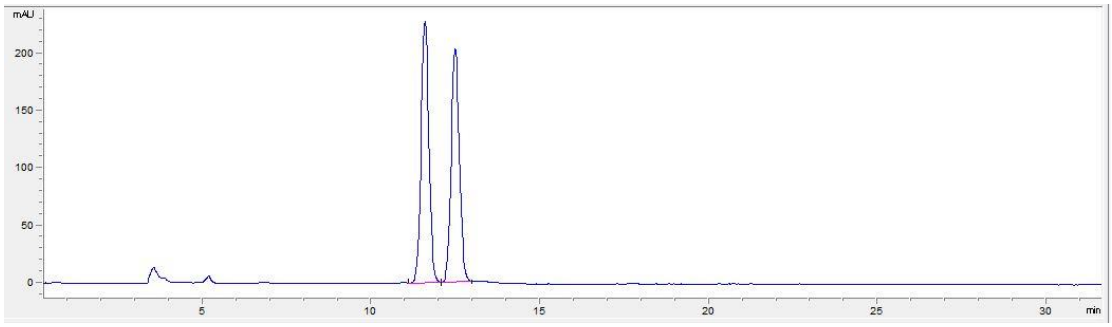
	Time/min	Area	Height	Area%
1	23.488	3287.5	131.5	5.625
2	24.788	55155.1	2015.3	94.375



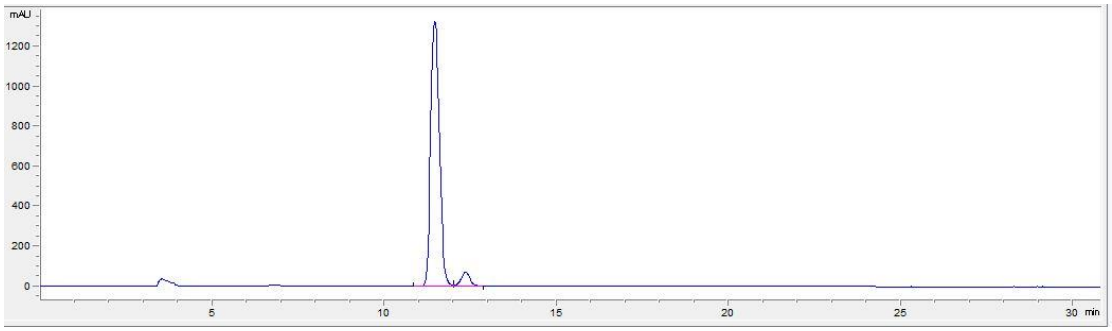
3j

(*E*)-3-(4-hydroxybut-2-en-1-yl)-3-methyl-2,3-dihydro-1*H*-inden-1-one

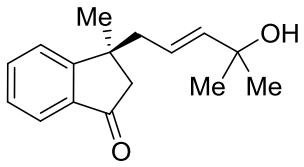
The mobile phase for flash chromatography: hexane/ethyl acetate = 20:1 to 5:1. Colorless oil. (34.0 mg, 79%). $[\alpha]_D^{25}$ = + 24.4 (c = 0.31, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.6 Hz, 1H), 7.62 (t, *J* = 7.6 Hz, 1H), 7.48 (d, *J* = 7.6 Hz, 1H), 7.37 (t, *J* = 7.6 Hz, 1H), 5.64 (td, *J* = 15.2, 5.6 Hz, 1H), 5.36 (td, *J* = 15.2, 7.2 Hz, 1H), 3.99 (d, *J* = 5.6 Hz, 2H), 2.70 (d, *J* = 18.8 Hz, 1H), 2.45-2.39 (m, 3H), 1.43 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.0, 162.1, 136.3, 135.1, 133.7, 127.8, 127.5, 123.9, 123.5, 63.3, 49.8, 45.1, 42.1, 28.2. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{14}H_{16}NaO_2$, 239.1043; found 239.1037. Enantiomeric excess was determined by HPLC with Chiralpak OX-H column (hexanes:2-propanol = 80:20, 0.9 mL/min, 210 nm, 95:5 er); major enantiomer tr = 11.4 min, minor enantiomer tr = 12.3 min.



	Time/min	Area	Height	Area%
1	11.584	3677.4	227.5	51.4
2	12.479	3475.6	202.7	48.6



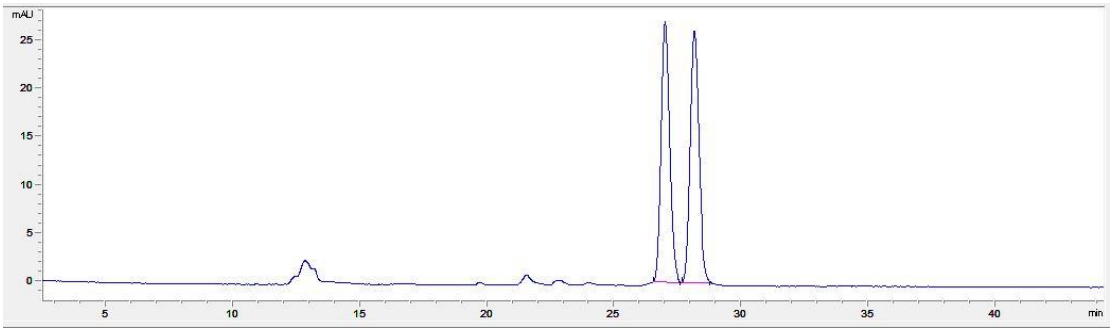
	Time/min	Area	Height	Area%
1	11.452	23532.2	1321.8	95.0
2	12.348	1291.2	72.4	5.0



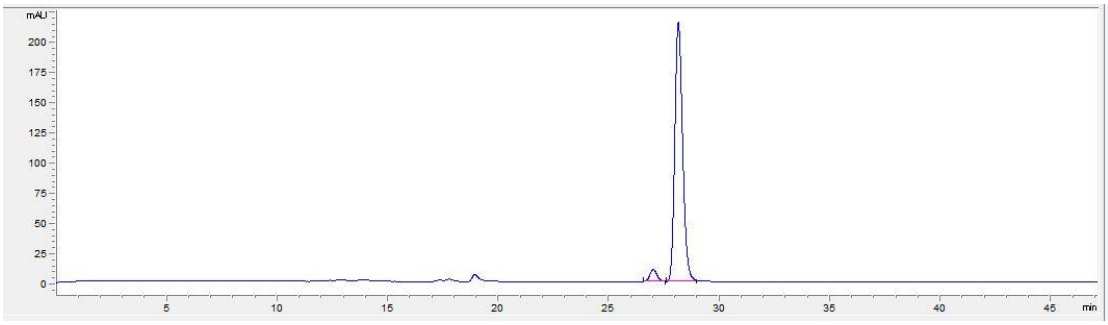
3k

(*E*)-3-(4-hydroxy-4-methylpent-2-en-1-yl)-3-methyl-2,3-dihydro-1*H*-inden-1-one

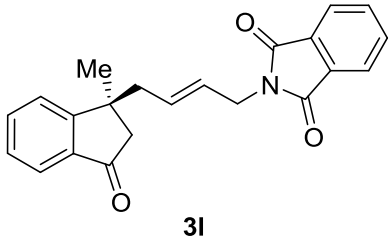
The mobile phase for flash chromatography: hexane/ethyl acetate = 5:1. Colorless oil (30.5 mg, 63%). $[\alpha]_D^{25}$ = + 106.6 (c = 1.02, $CHCl_3$). ^1H NMR (500 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.6 Hz, 1H), 7.64-7.59 (m, 1H), 7.47 (d, *J* = 8.0 Hz, 1H), 7.40-7.34 (m, 1H), 5.55 (dt, *J* = 15.2, 1.2 Hz, 1H), 5.29 (dt, *J* = 15.2, 7.2 Hz, 1H), 2.69 (d, *J* = 18.8 Hz, 1H), 2.42 (d, *J* = 18.8 Hz, 1H), 2.38 (dd, *J* = 7.2, 1.2 Hz, 2H), 1.44 (s, 3H), 1.18 (s, 3H), 1.17 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.9, 162.0, 142.8, 136.4, 135.0, 127.7, 124.0, 123.4, 122.0, 70.6, 50.0, 45.2, 42.4, 29.8, 29.7, 27.9. HRMS (ESI-TOF) m/z: [M + Na]$^+$ Calcd for $C_{16}H_{20}NaO_2$, 267.1356; found 267.1339. Enantiomeric excess was determined by HPLC with double Chiralpak OX-H column (hexane:2-propanol 80:20, 0.5 mL/min, 254 nm, 96:4 er); major enantiomer tr = 28.1 min, minor enantiomer tr = 27.0 min.



	Time/min	Area	Height	Area%
1	26.997	616.7	27	49.5
2	28.144	625.4	26.1	50.5



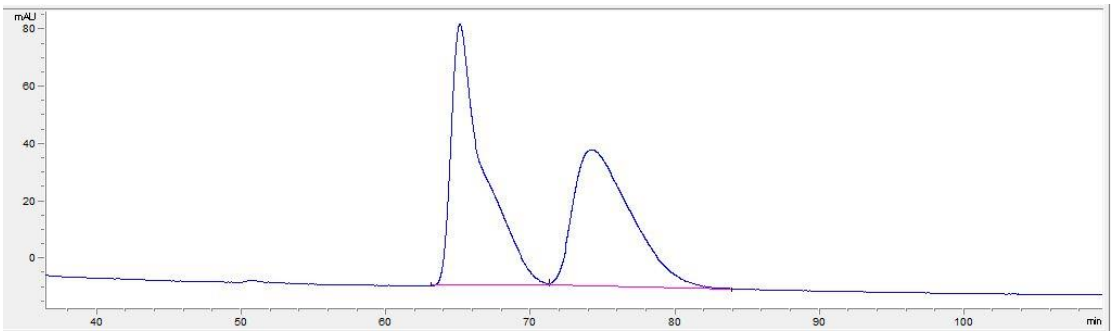
	Time/min	Area	Height	Area%
1	26.995	222.2	9.9	4.0
2	28.12	5219.1	214.2	96.0



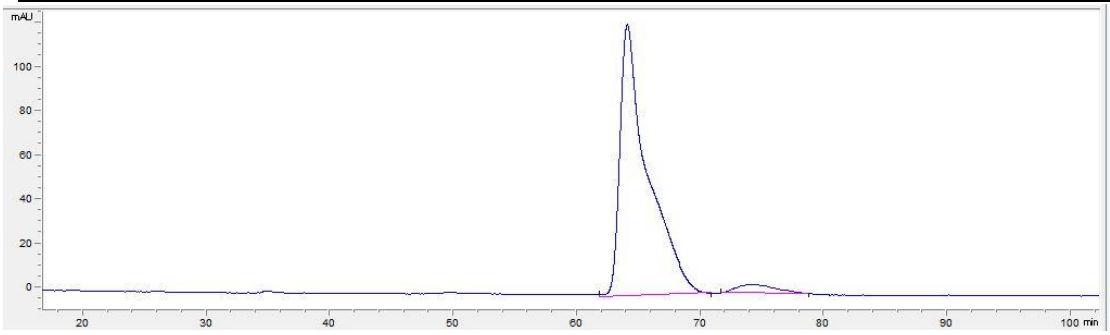
3l

(*E*)-2-(4-(1-methyl-3-oxo-2,3-dihydro-1*H*-inden-1-yl)but-2-en-1-yl)isoindoline-1,3-dione

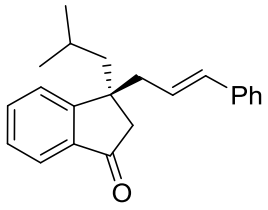
The mobile phase for flash chromatography: hexane/ethyl acetate = 20:1. Colorless oil. (40.8 mg, 59%). $[\alpha]_D^{25}$ = + 18.3 (c = 0.31, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 7.85-7.81 (m, 2H), 7.74-7.68 (m, 2H), 7.63 (d, *J* = 7.6 Hz, 1H), 7.54 (t, *J* = 7.6 Hz, 1H), 7.43 (d, *J* = 7.6 Hz 1H), 7.29 (t, *J* = 7.6 Hz, 1H), 5.53-5.49 (m, 2H), 4.21-4.09 (m, 2H), 2.65 (d, *J* = 18.8 Hz, 1H), 2.44-2.35 (m, 3H), 1.39 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.4, 167.9, 161.8, 136.2, 134.9, 134.0, 132.2, 130.5, 127.73, 127.68, 124.1, 123.5, 123.4, 50.0, 45.0, 42.1, 39.4, 27.9. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{22}H_{19}NNaO_3$, 368.1257; found 368.1231. Enantiomeric excess was determined by HPLC with double Chiralpak AS-H column (hexanes:2-propanol = 90:10, 1 mL/min, 210 nm, 95:5 er); major enantiomer tr = 64.1 min, minor enantiomer tr = 74.3 min.



	Time/min	Area	Height	Area%
1	65.069	14213.7	91.2	51.5
2	74.218	13281.6	47.9	48.5



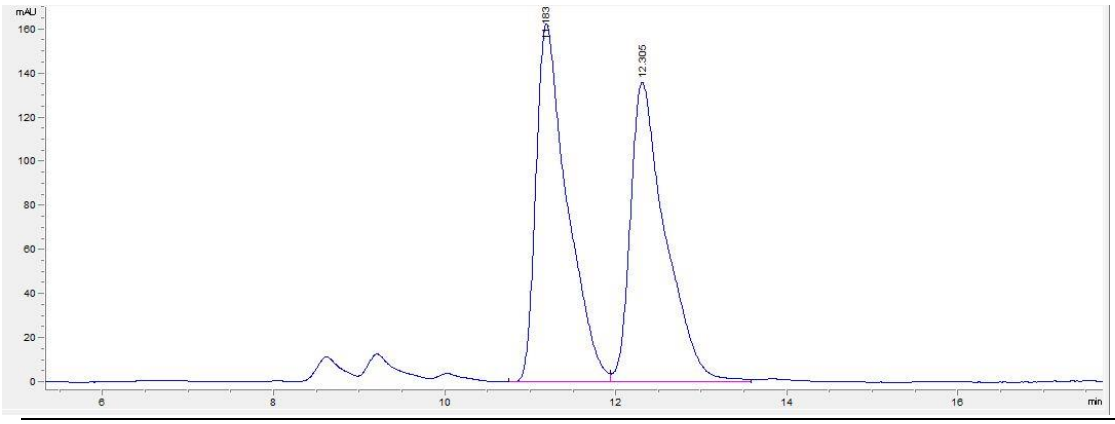
	Time/min	Area	Height	Area%
1	64.093	18933.1	122.8	95.0
2	74.37	1004.1	4.2	5.0



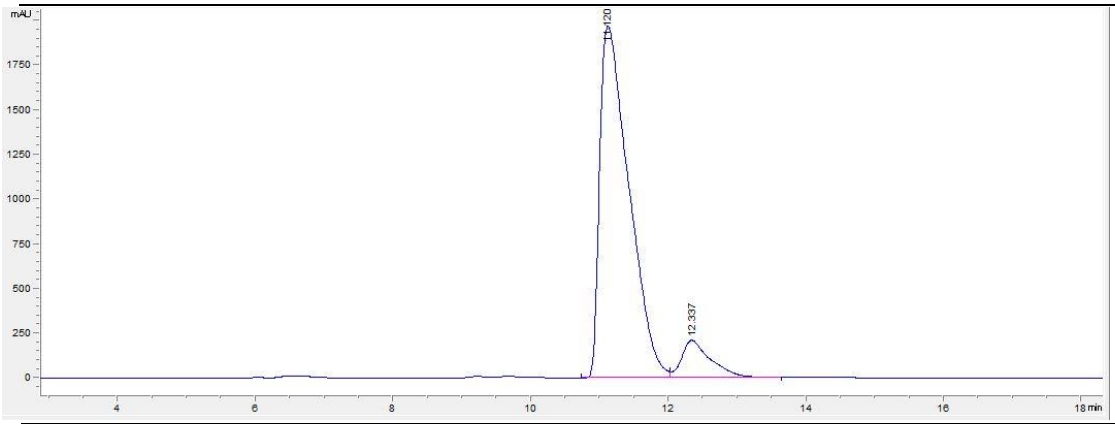
3m

3-cinnamyl-3-isobutyl-2,3-dihydro-1*H*-inden-1-one

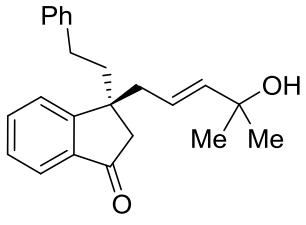
The mobile phase for flash chromatography: hexane/ethyl acetate = 60:1. Colorless oil (41.3 mg, 65%). $[\alpha]_D^{25}$ = + 26.1 (c = 0.34, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 7.6 Hz, 1H), 7.56 7.52 (m, 1H), 7.44 (d, *J* = 7.6 Hz, 1H), 7.31 (d, *J* = 7.6 Hz, 1H), 7.18-7.07 (m, 5H), 6.27 (d, *J* = 16.0 Hz, 1H), 5.79-5.71 (m, 1H), 2.61-2.47 (m, 4H), 1.78 (dd, *J* = 14.0, 4.4 Hz, 1H), 1.61 (dd, *J* = 14.0, 7.2 Hz, 1H), 1.50-1.41 (m, 1H), 0.81 (d, *J* = 6.4 Hz, 3H), 0.52 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.0, 161.1, 137.2, 137.1, 134.7, 134.1, 128.6, 127.8, 127.4, 126.2, 125.4, 124.7, 123.5, 49.1, 47.5, 46.2, 45.6, 25.3, 25.2, 24.2. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{22}H_{24}NaO$, 327.1719; found 327.1699. Enantiomeric excess was determined by HPLC with a Chiralpak OD-H column (hexane:2-propanol = 95:5, 0.6 mL/min, 254 nm, 90:10 er); major enantiomer tr = 11.1 min, minor enantiomer tr = 12.3 min.



	Time/min	Area	Height	Area%
1	11.183	4110.8	162.1	51.531
2	12.305	3866.6	136	48.469



	Time/min	Area	Height	Area%
1	11.12	58025.6	1967.7	90.214
2	12.337	6294.1	210.3	9.786

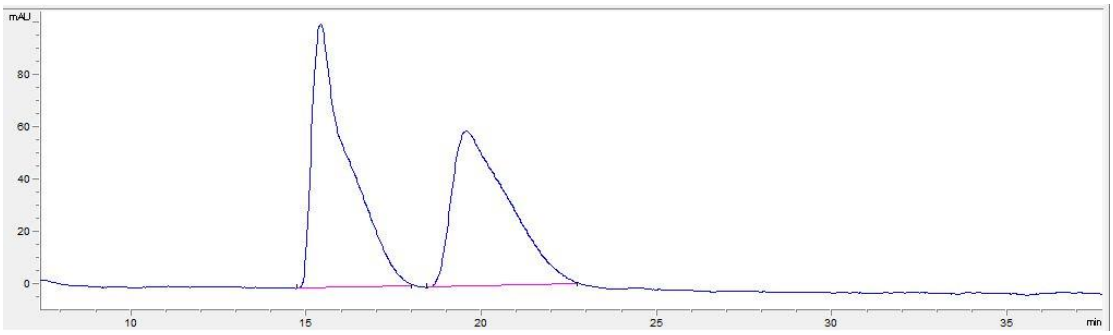


3n

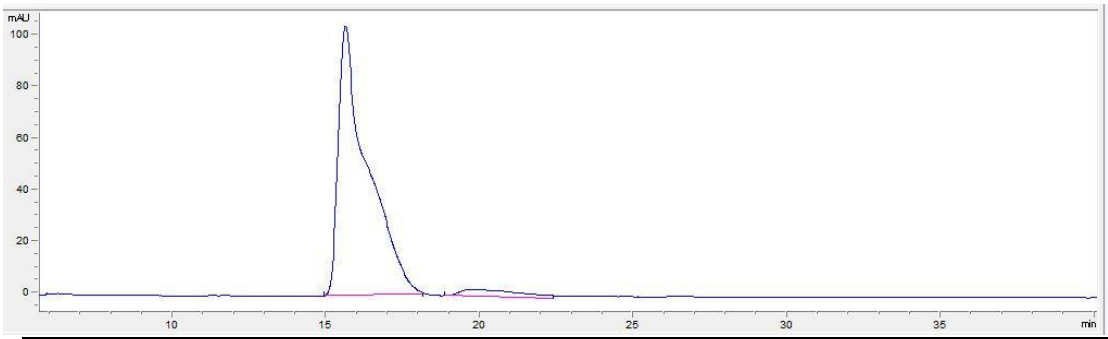
(*E*)-3-(4-hydroxy-4-methylpent-2-en-1-yl)-3-phenethyl-2,3-dihydro-1*H*-inden-1-one

The mobile phase for flash chromatography: hexane/ethyl acetate = 20:1 to 5:1. Colorless oil (30.6 mg, 46%). $[\alpha]_D^{25}$ = + 31.9 (c = 1.01, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, *J* = 7.6 Hz, 1H), 7.66-7.61 (m, 1H), 7.49 (d, *J* = 7.6 Hz, 1H), 7.42-7.37 (m, 1H), 7.27-7.21 (m, 2H), 7.19-7.13 (m, 1H), 7.07 (d, *J* = 7.2 Hz, 2H), 5.57 (d,

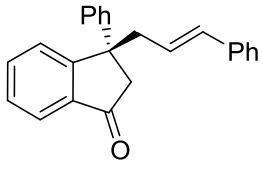
J = 15.2 Hz, 1H), 5.28 (dt, *J* = 15.2, 7.2 Hz, 1H), 2.64 (s, 2H), 2.57-2.45 (m, 3H), 2.26 (td, *J* = 12.4, 4.4 Hz, 1H), 2.17-2.00 (m, 2H), 1.17 (s, 3H), 1.16 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.7, 160.2, 143.0, 141.8, 137.3, 135.0, 128.6, 128.3, 127.9, 126.1, 124.3, 123.5, 121.5, 70.6, 47.1, 46.1, 43.9, 42.3, 31.2, 29.8, 29.7. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{23}H_{26}NaO_2$, 357.1825; found 357.1817. Enantiomeric excess was determined by HPLC with a Chiralpak AS-H column (hexane:2-propanol 90:10, 0.5 mL/min, 254 nm, 95:5 er); major enantiomer tr = 15.6 min, minor enantiomer tr = 19.8 min.



	Time/min	Area	Height	Area%
1	15.396	7208.5	100.4	52.0
2	19.543	6637	59.3	48.0



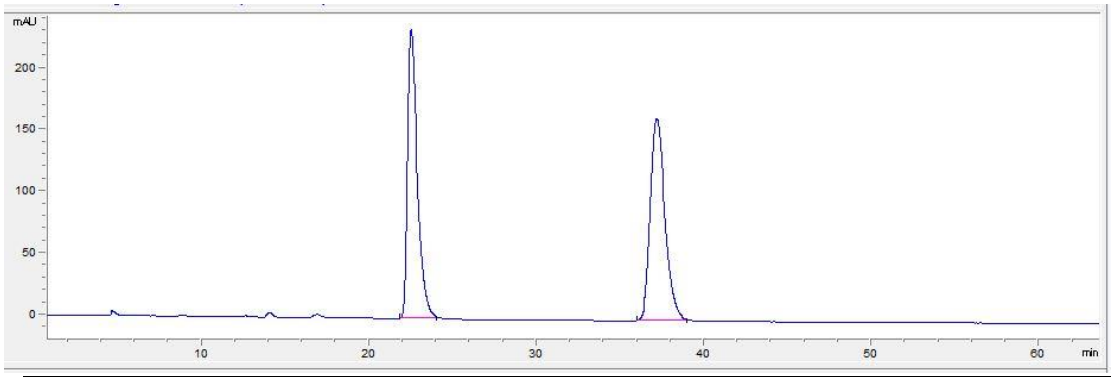
	Time/min	Area	Height	Area%
1	15.619	6874	104.6	95.0
2	19.823	362.2	2.6	5.0



3o

3-cinnamyl-3-phenyl-2,3-dihydro-1*H*-inden-1-one

The mobile phase for flash chromatography: hexane/ethyl acetate = 30:1. Light yellow oil (47.4 mg, 73%). $[\alpha]_D^{25}$ = - 25.7 (c = 0.36, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 7.71 (d, *J* = 7.6 Hz, 1H), 7.54 (td, *J* = 7.6, 1.6 Hz, 1H), 7.38-7.32 (m, 2H), 7.26-7.05 (m, 10H), 6.32 (d, *J* = 16.0 Hz, 1H), 5.72 (dt, *J* = 16.0, 7.2 Hz, 1H), 3.07-2.99 (m, 3H), 2.85 (d, *J* = 18.8 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.4, 160.1, 146.4, 137.1, 137.0, 135.1, 134.6, 128.8, 128.6, 128.2, 127.5, 126.7, 126.4, 126.2, 125.1, 123.6, 52.6, 50.2, 43.8. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{24}H_{20}NaO$, 347.1406; found 347.1399. Enantiomeric excess was determined by HPLC with Chiralpak OD-H column (hexanes: 2-propanol = 95:5, 0.8 mL/min, 270 nm, 91:9 er); major enantiomer tr = 22.9 min, minor enantiomer tr = 36.5 min.



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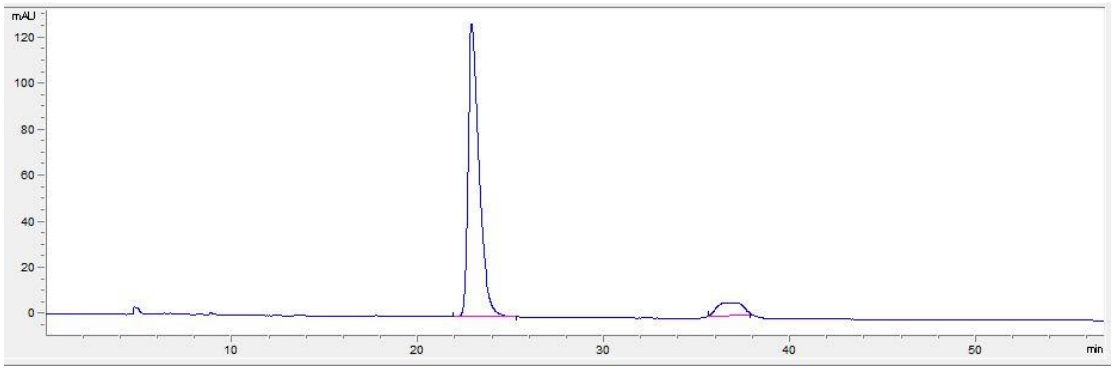
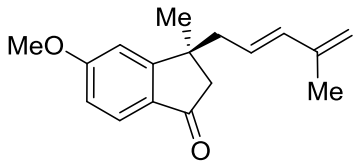
1	22.494	9541.5	233.6	49.0
2	37.163	9921.4	163.8	51.0



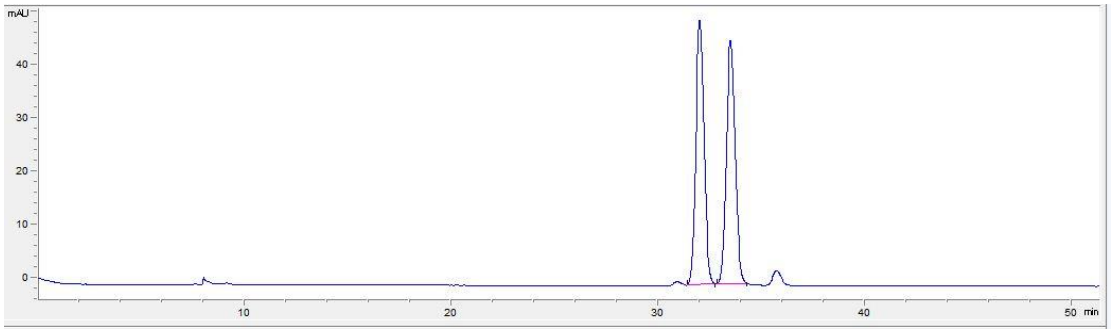
	Time/min	Area	Height	Area%
1	22.89	5370.5	127.2	91.0
2	36.497	535.1	5.6	9.0



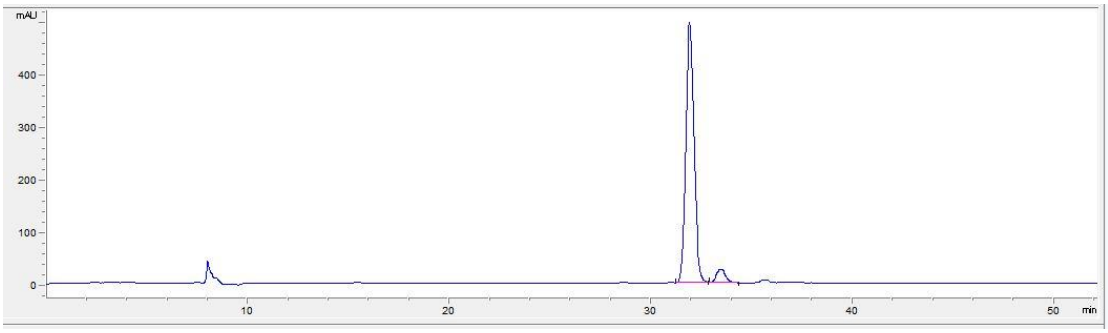
3p

(*E*)-5-methoxy-3-methyl-3-(4-methylpenta-2,4-dien-1-yl)-2,3-dihydro-1*H*-inden-1-one

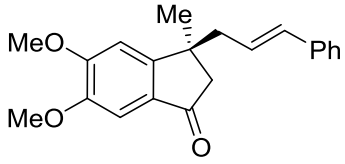
The mobile phase for flash chromatography: hexane/ethyl acetate = 30:1. Colorless oil (31.1 mg, 61%). $[\alpha]_D^{25}$ = + 17.8 (c = 0.28, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 8.4 Hz, 1H), 6.92-6.86 (m, 2H), 6.13 (d, *J* = 15.6 Hz, 1H), 5.36 (dt, *J* = 15.6, 7.6 Hz, 1H), 4.87 (s, 1H), 4.86 (s, 1H), 3.86 (s, 3H), 2.69 (d, *J* = 18.8 Hz, 1H), 2.46-2.37 (m, 3H), 1.71 (s, 3H), 1.41 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.9, 165.5, 165.2, 141.7, 136.9, 129.6, 125.4, 125.3, 115.8, 115.2, 107.6, 55.8, 50.2, 45.6, 42.2, 28.0, 18.7. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{17}H_{20}NaO_2$, 279.1356; found 279.1369. Enantiomeric excess was determined by HPLC with double Chiralpak OX-H column (hexane:2-propanol 95:5, 0.8 mL/min, 270 nm, 94.5:5.5 er); major enantiomer tr = 31.9 min, minor enantiomer tr = 33.5 min.



	Time/min	Area	Height	Area%
1	31.988	1377.1	49.4	50.5
2	33.478	1359.5	45.6	49.5



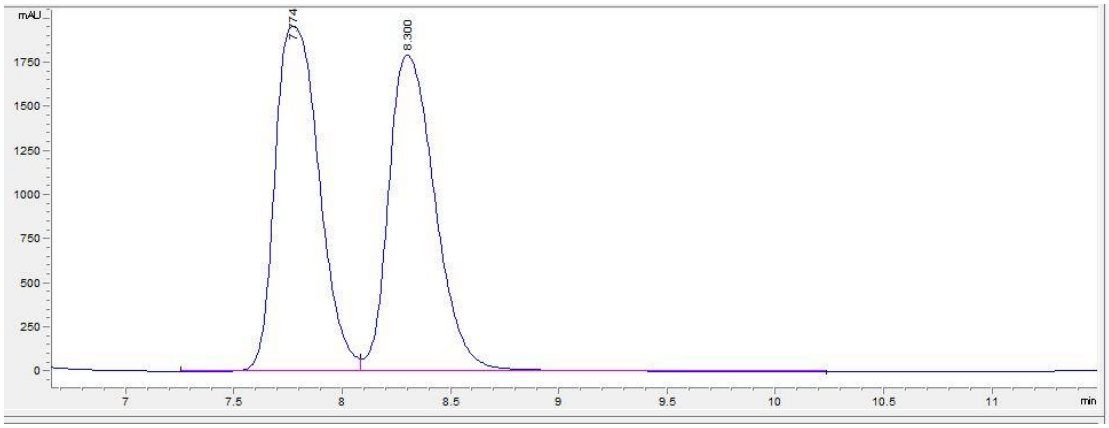
	Time/min	Area	Height	Area%
1	31.914	14072.2	494.9	94.5
2	33.463	827.4	26	5.5



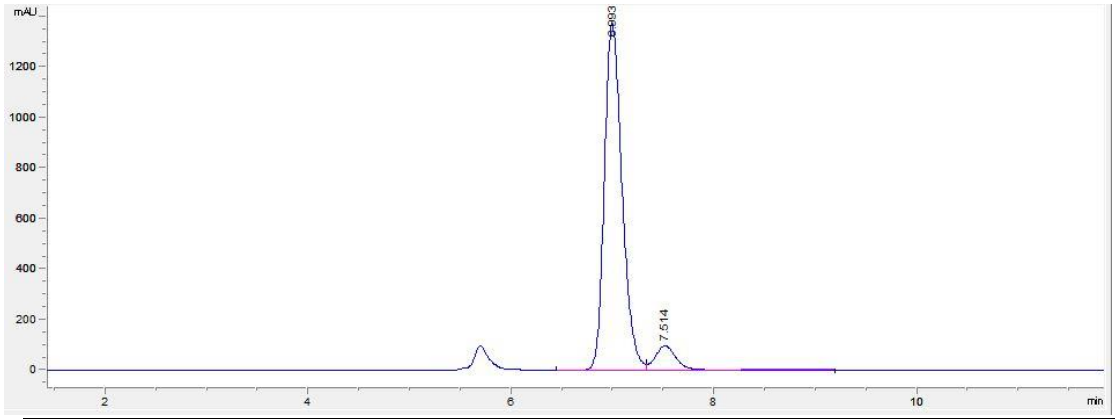
3q

3-cinnamyl-5,6-dimethoxy-3-methyl-2,3-dihydro-1*H*-inden-1-one

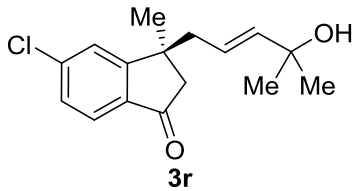
The mobile phase for flash chromatography: hexane/ethyl acetate = 20:1 to 6:1. Colorless oil (53.9 mg, 84%). $[\alpha]_D^{25}$ = + 6.3 (c = 0.34, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 7.24-7.10 (m, 5H), 7.07 (s, 1H), 6.81 (s, 1H), 6.32 (d, *J* = 15.6 Hz, 1H), 5.92-5.85 (m, 1H), 3.89 (s, 3H), 3.84 (s, 3H), 2.68 (d, *J* = 18.8 Hz, 1H), 2.52-2.44 (m, 2H), 2.38 (d, *J* = 18.8 Hz, 1H), 1.39 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.0, 157.3, 155.6, 149.6, 137.1, 133.9, 129.1, 128.6, 127.4, 126.2, 125.7, 105.0, 104.0, 56.3, 56.2, 50.1, 45.8, 42.1, 28.1. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{21}H_{22}NaO_3$, 345.1461; found 345.1457. Enantiomeric excess was determined by HPLC with a Chiralpak OX-H column (hexane:2-propanol = 60:40, 1.0 mL/min, 254 nm, 92:8 er); major enantiomer tr = 7.0 min, minor enantiomer tr = 7.5 min.



	Time/min	Area	Height	Area%
1	7.774	27725	1956.5	50.743
2	8.3	26912.5	1792.9	49.257



	Time/min	Area	Height	Area%
1	6.993	16626.6	1374.2	92.129
2	7.514	1420.5	97	7.871

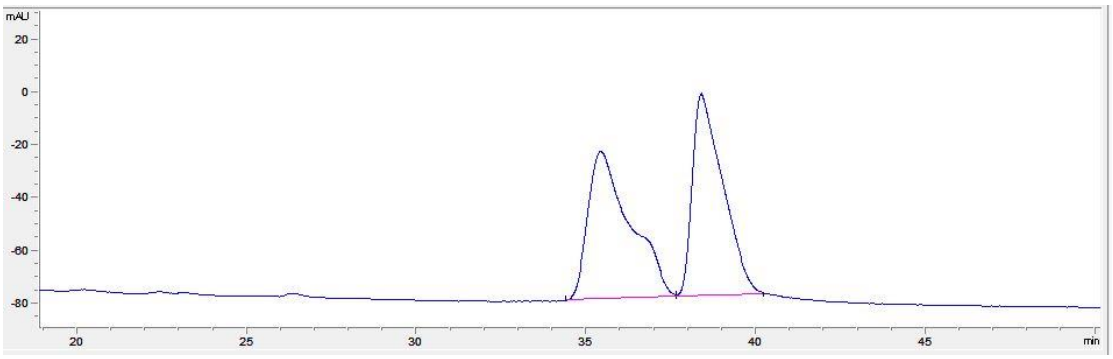


3r

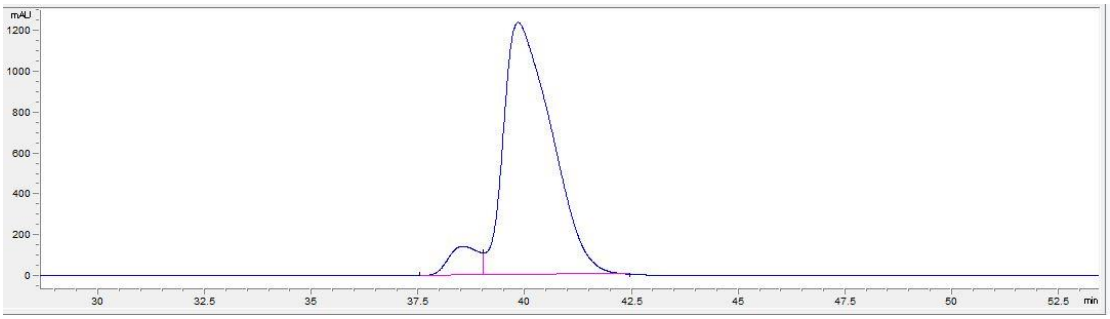
(*E*)-5-chloro-3-(4-hydroxy-4-methylpent-2-en-1-yl)-3-methyl-2,3-dihydro-1*H*-inden-1-one

The mobile phase for flash chromatography: hexane/ethyl acetate = 20:1. Colorless oil. (35.0 mg, 63%). $[\alpha]_D^{25}$ = + 8.7 (c = 0.36, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 7.64 (d, *J* = 2.0 Hz, 1H), 7.57 (dd, *J* = 8.0, 2.0 Hz, 1H), 7.41 (d, *J* = 8.0 Hz, 1H), 5.57 (d, *J* = 15.6 Hz, 1H), 5.30 (td, *J* = 15.6, 7.6 Hz, 1H), 2.70 (d, *J* = 18.8 Hz, 1H), 2.44 (d, *J* =

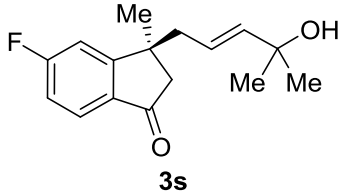
18.8 Hz, 1H), 2.37 (d, J = 7.6 Hz, 2H), 1.43 (s, 3H), 1.20 (s, 3H), 1.19 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.2, 160.0, 143.2, 137.9, 135.0, 134.2, 125.4, 123.2, 121.6, 70.7, 50.2, 45.0, 42.2, 29.9, 29.8, 27.9. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{16}H_{19}ClNaO_2$, 301.0966; found 301.0958. Enantiomeric excess was determined by HPLC with double Chiralpak AS-H column (hexanes:2-propanol = 90:10, 0.6 mL/min, 210 nm, 93.5:6.5 er); major enantiomer tr = 39.8 min, minor enantiomer tr = 38.5 min.



	Time/min	Area	Height	Area%
1	35.415	4840.4	56.2	50.0
2	38.386	4852	76.9	50.0



	Time/min	Area	Height	Area%
1	38.534	6999	141.9	6.5
2	39.833	96487.4	1236.2	93.5

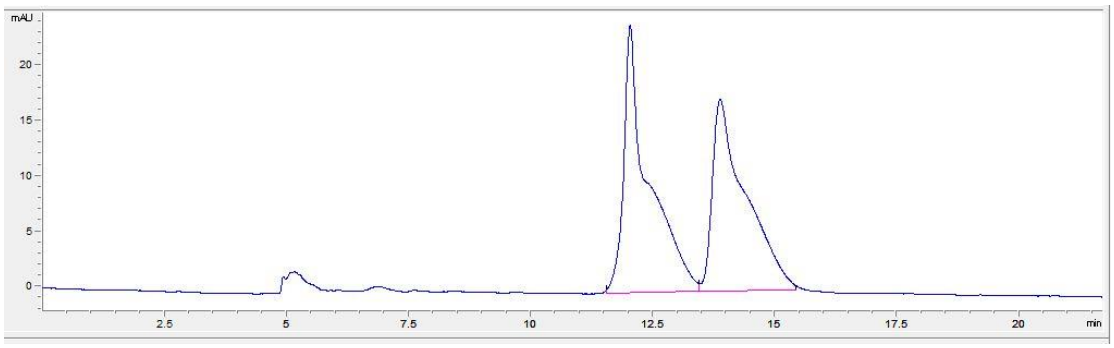


3s

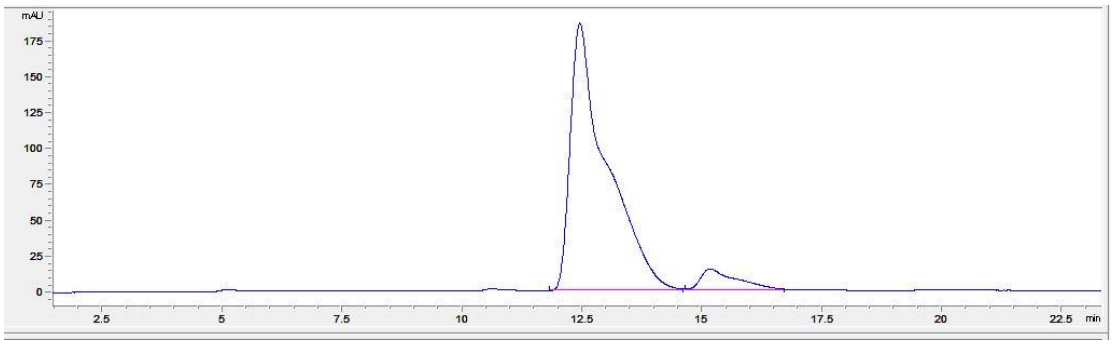
(*E*)-5-fluoro-3-(4-hydroxy-4-methylpent-2-en-1-yl)-3-methyl-2,3-dihydro-1*H*-inden-1-one

The mobile phase for flash chromatography: hexane/ethyl acetate = 20:1 to 5:1. Colorless oil (36.2 mg, 69%). $[\alpha]_D^{25}$ = + 6.9 (c = 0.34, $CHCl_3$). ^{1}H NMR (400 MHz,

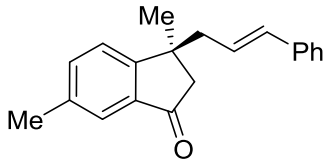
$CDCl_3$) δ 7.69 (dd, *J* = 8.4, 5.2 Hz, 1H), 7.10 (dd, *J* = 8.8, 2.0 Hz, 1H), 7.06 (ddd, *J* = 8.8, 8.4, 2.0 Hz, 1H), 5.57 (d, *J* = 15.2 Hz, 1H), 5.30 (dt, *J* = 15.2, 7.2 Hz, 1H), 2.70 (d, *J* = 18.4 Hz, 1H), 2.44 (d, *J* = 18.4 Hz, 1H), 2.36 (d, *J* = 7.2 Hz, 2H), 1.43 (s, 3H), 1.19 (s, 3H), 1.18 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.8, 167.4 (d, *J* = 255.5 Hz), 165.0 (d, *J* = 8.8 Hz), 143.2, 132.8 (d, *J* = 1.9 Hz), 125.8 (d, *J* = 10.3 Hz), 121.5, 116.0 (d, *J* = 23.7 Hz), 110.8 (d, *J* = 22.0 Hz), 70.6, 50.1, 45.0, 42.3 (d, *J* = 2.0 Hz), 29.9, 29.8, 27.8. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{16}H_{19}FNaO_2$, 285.1261; found 285.1248. Enantiomeric excess was determined by HPLC with a Chiralpak AS-H column (hexane:2-propanol 90:10, 06 mL/min, 254 nm, 92.5:7.5 er); major enantiomer tr = 12.4 min, minor enantiomer tr = 15.2 min.



	Time/min	Area	Height	Area%
1	12.035	854.9	24.2	50.5
2	13.879	831.4	17.4	49.5



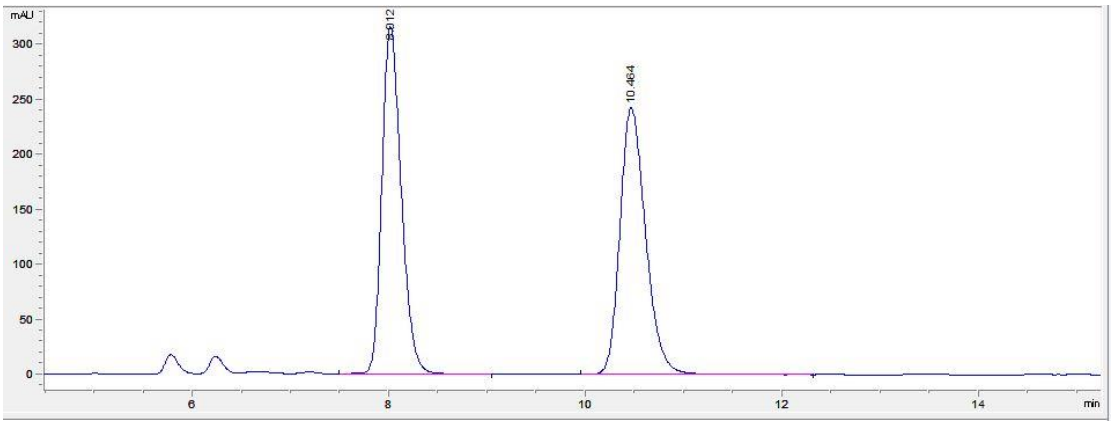
	Time/min	Area	Height	Area%
1	12.453	9795.4	186.1	92.5
2	15.165	777.6	14.9	7.5



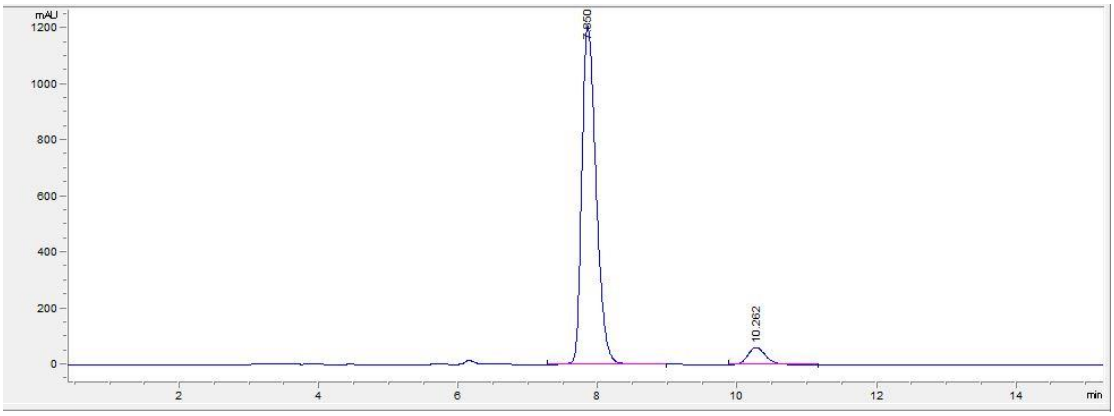
3t

3-cinnamyl-3,6-dimethyl-2,3-dihydro-1*H*-inden-1-one

The mobile phase for flash chromatography: hexane/ethyl acetate = 60:1. Colorless oil (40.3 mg, 73%). $[\alpha]_D^{25}$ = + 20.2 (c = 0.077, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 7.43 (s, 1H), 7.38-7.31 (m, 2H), 7.23-7.09 (m, 5H), 6.30 (d, *J* = 15.6 Hz, 1H), 5.87 (dt, *J* = 15.6, 7.6 Hz, 1H), 2.69 (d, *J* = 18.4 Hz, 1H), 2.49-2.46 (m, 2H), 2.37 (d, *J* = 18.4 Hz, 1H), 2.32 (s, 3H), 1.37 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.8, 159.7, 137.8, 137.3, 136.4, 136.3, 133.9, 128.6, 127.4, 126.3, 125.8, 123.8, 123.5, 50.4, 46.0, 42.2, 28.3, 21.2. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{20}H_{20}NaO$, 299.1406; found 299.1401. Enantiomeric excess was determined by HPLC with a Chiralpak OD-H column (hexane:2-propanol = 95:5, 1.0 mL/min, 254 nm, 94:6 er); major enantiomer tr = 7.9 min, minor enantiomer tr = 10.3 min.

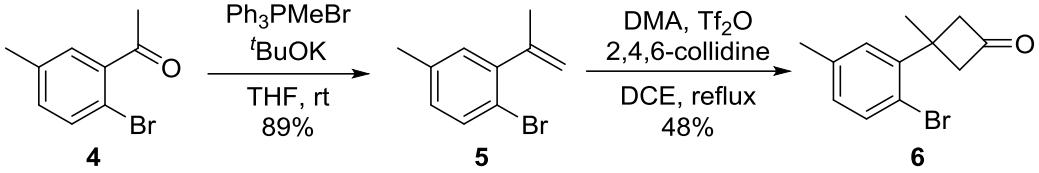


	Time/min	Area	Height	Area%
1	7.94	3094.6	227.4	49.627
2	10.377	3141.1	174.2	50.373



	Time/min	Area	Height	Area%
1	7.85	17194.9	1207.6	94.051
2	10.262	1087.7	61.3	5.949

Synthesis of (+)-herbertene-1,14-diol

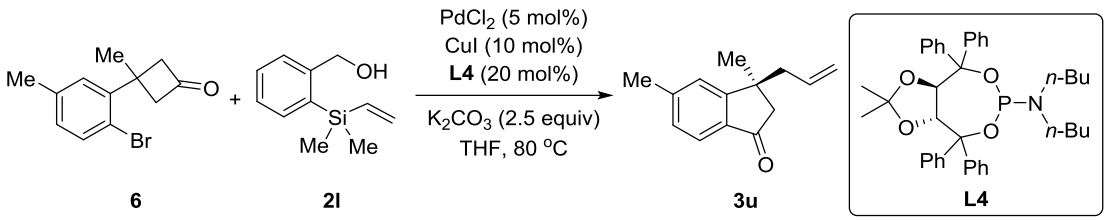


3-(2-bromo-5-methylphenyl)-3-methylcyclobutan-1-one 6

A vial was charged with Ph_3PMeBr (18.32 g, 51.31 mmol, 2.0 eq), KO^{t}Bu (7.20 g, 64.16 mmol, 2.5 eq) and evacuated under high vacuum and backfilled with N_2. THF (100 mL) was added via syringe and the mixture was stirred at room temperature for 1 h. A solution of 1-(2-bromo-5-methylphenyl)ethan-1-one **4** (5.40 g, 25.66 mmol) in THF (20 mL) was added dropwise and the mixture was stirred at room temperature. After complete consumption of starting material (12 h, TLC, eluent: hexane), the mixture was extracted with ethyl acetate and water. The organic layer was separated, dried over Na_2SO_4, concentrated under reduced pressure and then purified by silica column (eluent: hexane) to get the product **5** (4.80 g, 89% yield) as the colorless oil.

Triflic anhydride (9.03 g, 32 mmol, 1.4 equiv) was added dropwise to a solution of *N*,*N*-dimethylacetamide (2.39 g, 27.4 mmol, 1.2 equiv) in 30 mL of 1,2-dichloroethane under stirring at 5 ^{o}C. The mixture was stirred at 5 ^{o}C for 30 min, and then a mixture of **5** (4.8 g, 22.86 mmol, 1.0 equiv) and 2,4,6-collidine (4.59 g, 32 mmol, 1.4 equiv) in 5 mL of 1,2-dichloroethane was added dropwise. After the reaction mixture was refluxed for 18 h, 1,2-dichloromethane was removed in vacuum and the residue was treated with 8 mL H_2O and CCl_4 (1:1). The obtained mixture was refluxed for 18 h, and 30 mL of water was added. The mixture was extracted with CH_2Cl_2 (3 × 50 mL), and the combined organic layers was washed with 200 mL of saturated brine, dried over Na_2SO_4 and filtered. Concentration of the solution by rotary evaporation under reduced pressure gave a residue, which was purified by silica gel (petroleum ether: EtOAc= 25:1) to afford **6** as yellow oil (2.78 g, 48% yield). ^{1}HNMR (400 MHz, $CDCl_3$) δ 7.45 (d, J = 8.0 Hz, 1H), 7.09 (d, J = 2.0 Hz, 1H), 6.93 (dd, J = 8.0, 2.0 Hz, 1H), 3.56-3.49 (m, 2H), 3.23-3.17 (m, 2H), 2.32 (s, 3H), 1.61 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.9, 145.6, 137.7, 134.2, 129.3, 129.1, 118.9, 59.3, 36.2, 27.9, 21.1. HRMS (ESI-TOF) m/z:

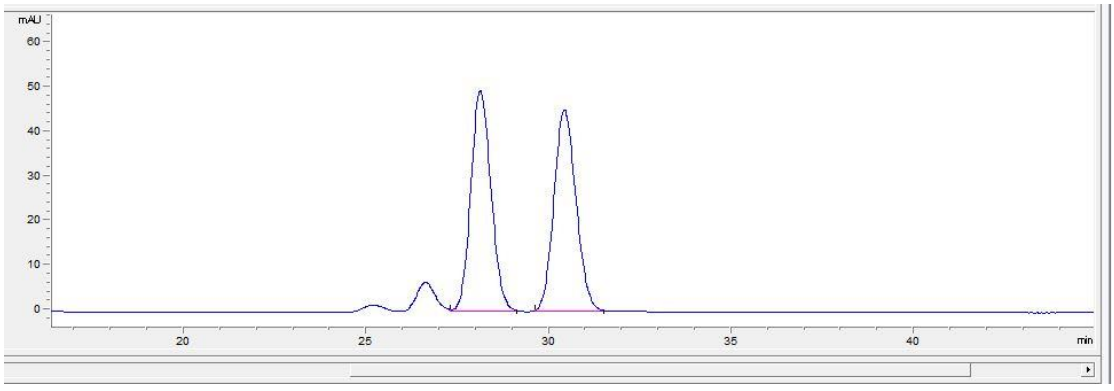
[M + Na]$^+$ Calcd for $C_{12}H_{13}BrNaO$, 275.0042; found 275.0057.



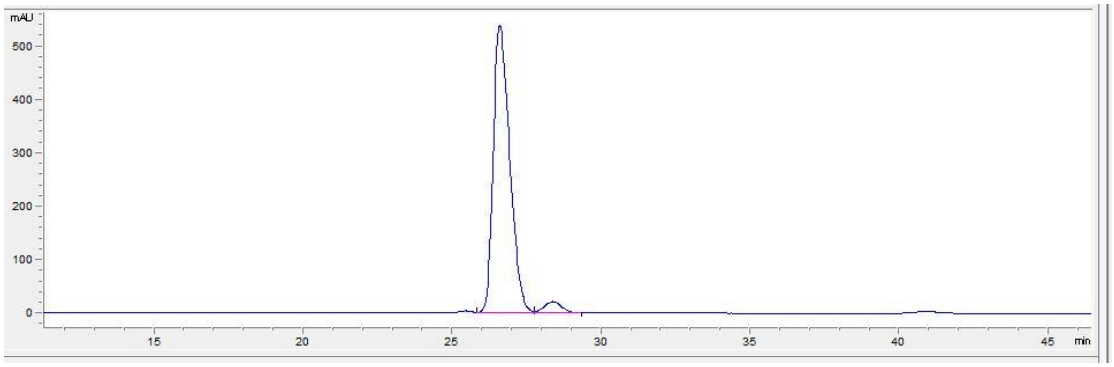
3-allyl-3,5-dimethyl-2,3-dihydro-1*H*-inden-1-one 3u

A vial was charged with $PdCl_2$ (88.0 mg, 0.50 mmol), CuI (190.4 mg, 1.0 mmol), **L4** (1.25 g, 2.0 mmol), and K_2CO_3 (3.45 g, 25.0 mmol), and evacuated under high vacuum and backfilled with N_2. THF (25 mL) was added via syringe and the mixture was stirred at room temperature for 20 min. A solution of **6** (2.5 g, 10.0 mmol) and **2l** (2.30 g, 12.0 mmol) in THF (25 mL) was added via syringe and the mixture was stirred at 80 °C in an oil bath for 24 h, and then cooled to room temperature. The mixture was filtered over a plug of silica gel (washed with 100 mL EtOAc), and the filtrate was concentrated under reduced pressure and then purified by silica column to get the product **3u**.

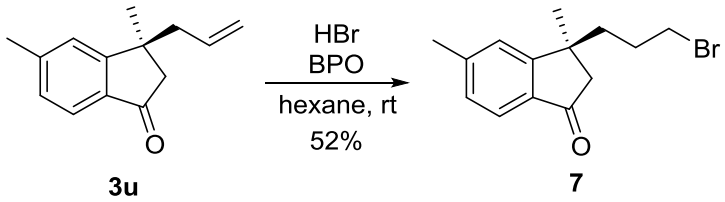
The mobile phase for flash chromatography: hexane/ethyl acetate = 50:1. Colorless oil (1.06 g, 52%). $[\alpha]_D^{25}$ = + 26.6 (c = 0.28, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (d, *J* = 8.0 Hz, 1H), 7.19 (s, 1H), 7.11 (d, *J* = 8.0 Hz, 1H), 5.50-5.38 (m, 1H), 5.00-4.93 (m, 2H), 2.64 (d, *J* = 18.4 Hz, 1H), 2.40-2.30 (m, 6H), 1.34 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.4, 162.8, 146.2, 134.1, 134.0, 129.0, 124.3, 123.3, 118.9, 49.9, 46.6, 41.7, 28.3, 22.4. HRMS (ESI-TOF) m/z: [M + Na]$^+$ Calcd for $C_{14}H_{16}NaO$, 223.1093; found 223.1086. Enantiomeric excess was determined by HPLC with a Chiralpak OX-H column (hexanes: 2-propanol = 95:5, 1.2 mL/min, 254 nm, 96:4 er); major enantiomer t_r = 27.1 min, minor enantiomer t_r = 29.1 min.



	Time/min	Area	Height	Area%
1	28.119	1912.7	49.7	50.014
2	30.423	1911.7	45.2	49.986



	Time/min	Area	Height	Area%
1	26.581	20725.4	537.2	96.060
2	28.361	850.1	21.1	3.940



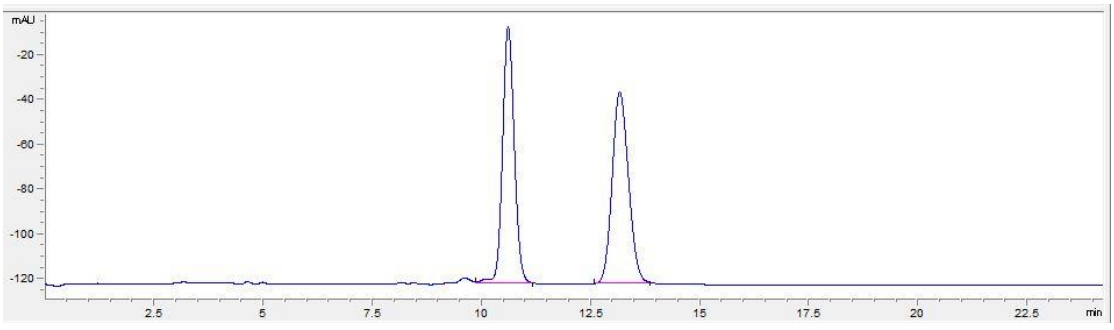
3u **7**

3-(3-bromopropyl)-3,5-dimethyl-2,3-dihydro-1*H*-inden-1-one 7

A vigorously stirred solution of compound **3u** (211.4 mg, 1.05 mmol) and dibenzoylperoxide (9.6 mg, 0.042 mmol) in dry *n*-hexane (10 mL) was treated with dry HBr gas for 2 h, produced by the addition of bromine (0.5 mL) to tetraline (15 mL), and the mixture was stirred overnight. Petroleum ether (10 mL), ethyl acetate (10 mL), and brine (10 mL) were added and separated. The aqueous layer was extracted by ethyl acetate (20 mL). The combined organic layers were dried over Na_2SO_4 and filtered, and the filtrate was concentrated under reduced pressure and then purified by silica column to get the product **7**.

The mobile phase for flash chromatography: hexane/ethyl acetate = 50:1. Colorless oil (150.4 mg, 51%). $[\alpha]_D^{25}$ = + 6.7 (c = 3.14, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, *J* = 8.0 Hz, 1H), 7.24 (s, 1H), 7.18 (d, *J* = 8.0 Hz, 1H), 3.31 (t, *J* = 6.0 Hz, 2H), 2.63 (d, *J* = 18.8 Hz, 1H), 2.46 (d, *J* = 18.8 Hz, 1H), 2.45 (s, 3H), 1.90-1.73 (m, 3H), 1.54-1.43 (m, 1H), 1.41 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.1, 162.6, 146.4, 133.9,

129.1, 124.2, 123.4, 50.3, 41.5, 40.6, 33.9, 28.63, 28.57, 22.4. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{14}H_{17}BrNaO$, 303.0355; found 303.0363. Enantiomeric excess was determined by HPLC with a Chiralpak AS-H column (hexanes: 2-propanol = 80:20, 1 mL/min, 260 nm, 96.5:3.5 *er*); major enantiomer tr = 10.6 min, minor enantiomer tr = 13.2 min.



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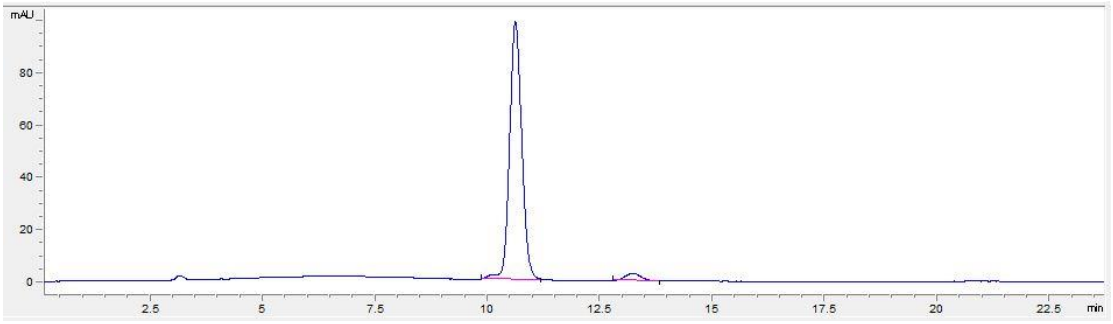

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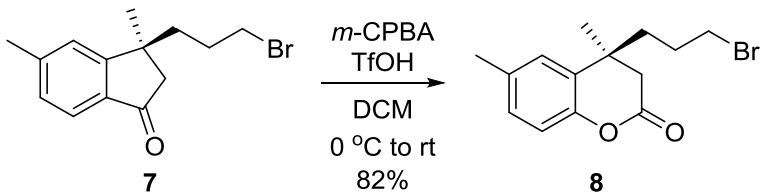


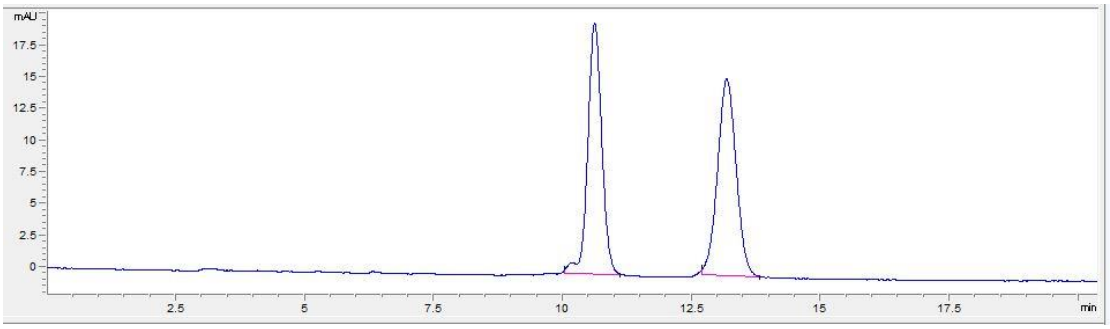
7 **8**

4-(3-bromopropyl)-4,6-dimethylchroman-2-one 8

To a solution of **7** (60.0 mg, 0.214 mmol) in CH_2Cl_2 (2 mL) was added TfOH (3.2 mg, 0.0214 mmol, 0.1 equiv) and *m*-CPBA (73.8 mg, 0.428 mmol, 2.0 equiv) at 0 °C. Then

the mixture was stirred at room temperature for 58 h. Water (5.0 mL) was then added and the aqueous layer was extracted with CH_2Cl_2 (3 × 5 mL). The combined organic phase was dried over anhydrous Na_2SO_4, filtered and concentrated under vacuum. The residue was then purified by flash column chromatography to give product **8**.

The mobile phase for flash chromatography: hexane/ethyl acetate = 50:1. Colorless oil (52.2 g, 82% yield). $[\alpha]_D^{25}$ = + 30.0 (c = 0.2, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 7.08-7.02 (m, 2H), 6.95 (d, *J* = 8.0 Hz, 1H), 3.32 (t, *J* = 6.4 Hz, 2H), 2.68 (d, *J* = 16.0 Hz, 1H), 2.57 (d, *J* = 16.0 Hz, 1H), 2.35 (s, 3H), 1.88-1.79 (m, 1H), 1.77-1.63 (m, 3H), 1.36 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.4, 148.9, 134.4, 129.4, 129.1, 125.7, 117.1, 42.0, 38.8, 36.1, 33.5, 27.7, 25.3, 21.2. HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{14}H_{17}BrNaO_2$, 319.0304; found 319.0315. Enantiomeric excess was determined by HPLC with a Chiralpak AS-H column (hexanes: 2-propanol = 80:20, 1 mL/min, 260 nm, 95.0:5.0 *er*); major enantiomer tr = 10.8 min, minor enantiomer tr = 12.2 min.



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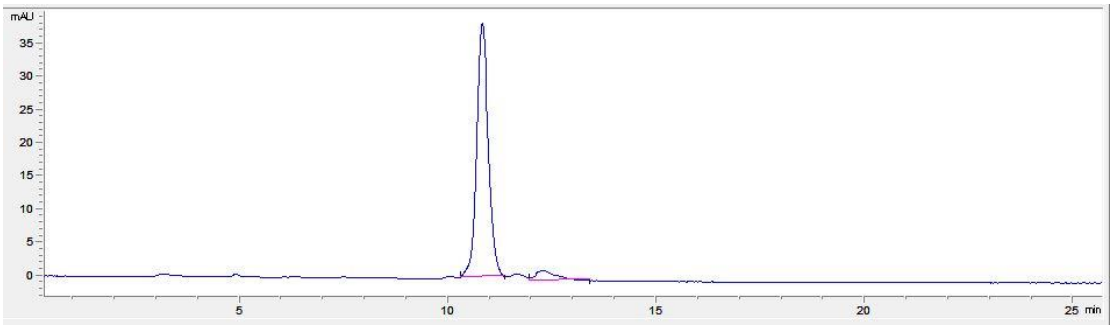

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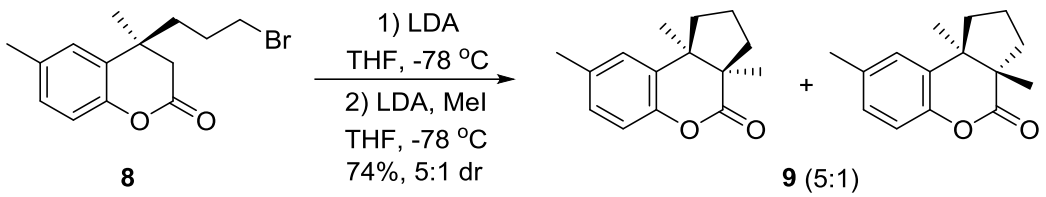
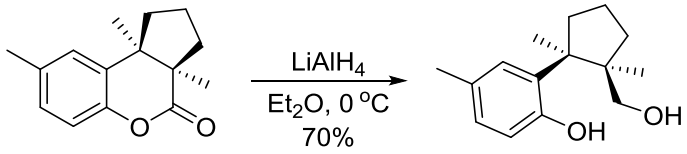


8 **9** (5:1)

(3a*R*,9b*R*)-3a,8,9b-trimethyl-2,3,3a,9b-tetrahydrocyclopenta[*c*]chromen-4(1*H*)-one 9

LDA (0.11 mL, 0.211 mmol, 2.0 M in THF) was added dropwise to a solution of compound **8** (52.2 mg, 0.176 mmol) in dry THF (2 mL) under N_2 at -78 °C. The reaction mixture was stirred for 2 h at -78 °C. Then LDA (0.14 mL, 0.282 mmol, 2.0 M in THF) and MeI (21.7 μL, 0.35 mmol) were added, and the reaction was stirred at -78 °C for 5 h and at room temperature for 30 min. Water (5.0 mL) was added to the reaction mixture, and the organic layer was separated and aqueous layer was extracted with ethyl acetate (3 × 5 mL). The combined organic layer was washed with brine (5 mL), dried over Na_2SO_4 and concentrated under vacuum. The residue was then purified by flash column chromatography to give product **9** as an inseparable diastereomers (5:1).

The mobile phase for flash chromatography: hexane/ethyl acetate = 50:1. Colorless oil (30.0 g, 74% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.11 (d, *J* = 2.0 Hz, 1H), 7.01 (dd, *J* = 8.0, 2.0 Hz, 1H), 6.89 (d, *J* = 8.0 Hz, 1H), 2.33 (s, 1H), 2.42-2.31 (m, 1H), 2.13-2.04 (m, 1H), 1.90-1.57 (m, 4H), 1.26 (s, 3H), 1.23 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.9, 147.6, 134.2, 128.7, 128.6, 127.0, 116.6, 51.1, 47.8, 39.0, 35.8, 21.8, 21.2, 20.7, 18.1; data matched that from the literature.[3-4] HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{15}H_{18}NaO_2$, 253.1199; found 253.1194.



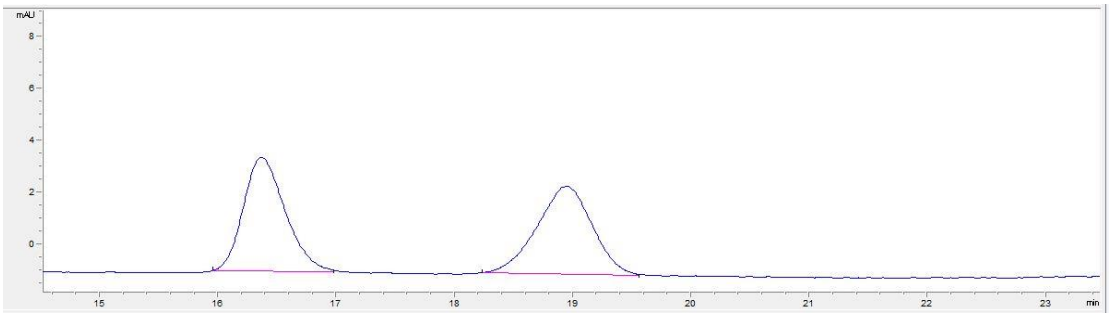
9 **10**

2-((1*S*,2*R*)-2-(hydroxymethyl)-1,2-dimethylcyclopentyl)-4-methylphenol 10

To a solution of **9** (19.1 mg, 0.083 mmol) in anhydrous Et_2O (2.0 mL) at 0 °C was treated with $LiAlH_4$ (10.7 mg, 0.282 mmol). The reaction mixture was stirred for 30 min at 0 °C and 3 h at room temperature. Then isopropanol (5 mL) was added to quench

the reaction. Water (5.0 mL) was added, and the organic layer was separated and aqueous layer was extracted with ethyl acetate (3 × 5 mL) and the combined organic phase was dried over Na_2SO_4, filtered and concentrated to give the crude compound. Purification of crude compound by flash chromatography afforded **10**.

The mobile phase for flash chromatography: hexane/ethyl acetate = 20:1. White solid, m.p. 106-108 ^oC (CH_2Cl_2). (13.0 mg, 70% yield). $[\alpha]_D^{25}$ = + 11.0 (c = 0.3, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 6.96 (d, *J* = 2.1 Hz, 1H), 6.91 (dd, *J* = 8.0, 2.1 Hz, 1H), 6.73 (d, *J* = 8.0 Hz, 1H), 3.34 (d, *J* = 11.1 Hz, 1H), 3.27 (d, *J* = 11.0 Hz, 1H), 2.52-2.38 (m, 1H), 2.27 (s, 3H), 1.98-1.77 (m, 3H), 1.56 (s, 3H), 1.50-1.41 (m, 1H), 1.32-1.25 (m, 1H), 1.24 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.2, 133.0, 130.0, 129.3, 128.1, 117.9, 70.8, 51.0, 49.1, 42.5, 36.1, 24.1, 21.3, 21.1, 20.6; data matched that from the literature.[3] HRMS (ESI-TOF) m/z: $[M + Na]^+$ Calcd for $C_{15}H_{22}NaO_2$, 257.1512; found 257.1508. Enantiomeric excess was determined by HPLC with a Chiralpak As-H column (hexanes: 2-propanol = 70.0:30.0, 1 mL/min, 260 nm, 3.5:96.5 *er*); major enantiomer tr = 18.8 min, minor enantiomer tr = 15.1 min.



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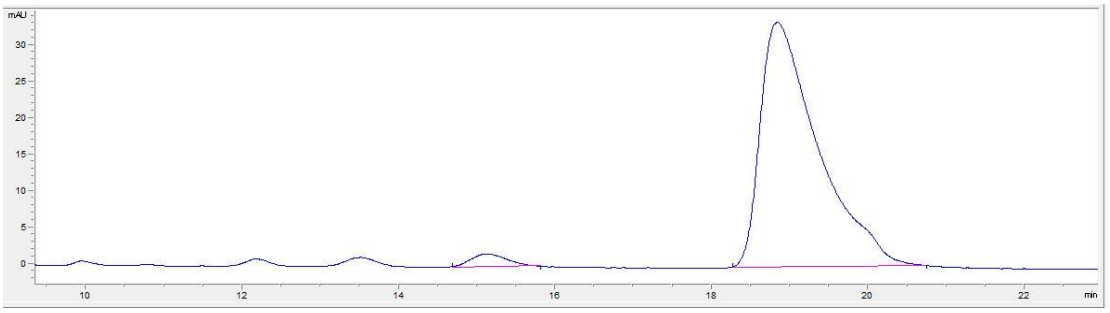

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1	15.139	60.1	1.8	3.5
2	18.841	1732.6	33.6	96.5

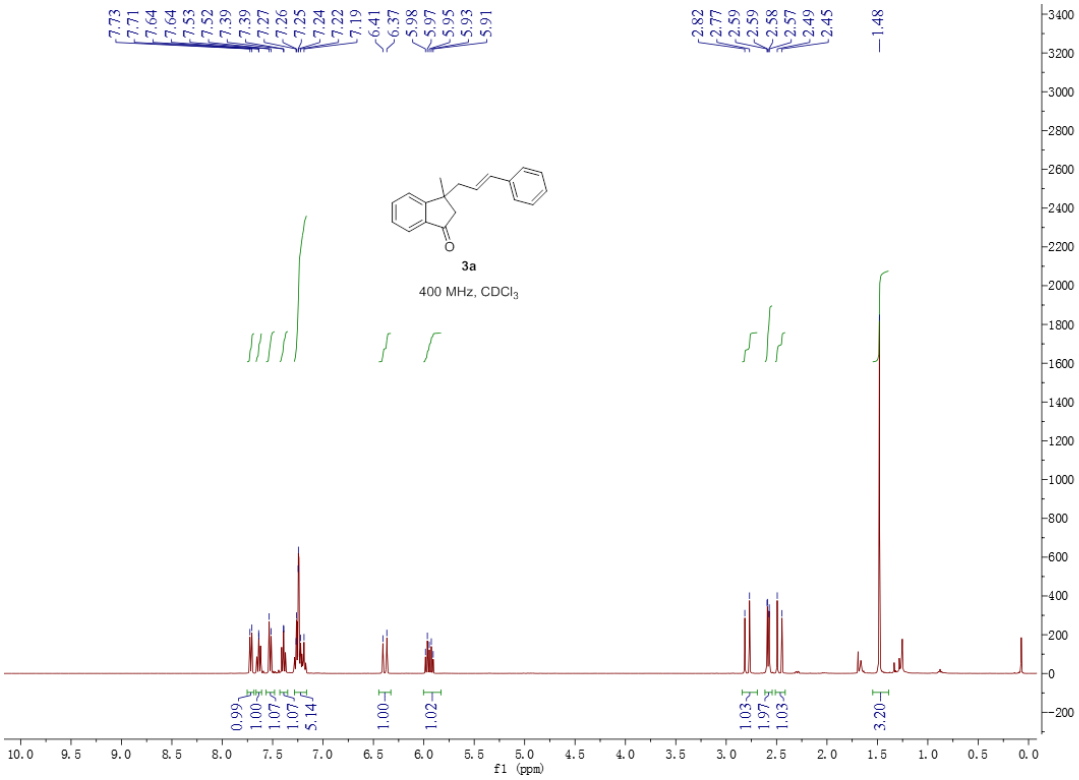
References

1 (a) J. Cao, L. Chen, F.-N. Sun, Y.-L. Sun, K.-Z. Jiang, K.-F. Yang, Z. Xu and L.-W. Xu, *Angew. Chem., Int. Ed*., 2019, **58**, 897–901; (b) Y.-L. Sun, X.-B. Wang, F.-N. Sun, Q.-Q. Chen, J. Cao, Z. Xu and L.-W. Xu, *Angew. Chem., Int. Ed.,* 2019, **58**, 6747–6751.

2 (a) Y. Nakao, H. Imanaka, A. K. Sahoo, A. Yada and T. Hiyama, *J. Am. Chem. Soc.*, 2005, **127**, 6952–6953; (b) Y. Nakao, J. Chen, H. Imanaka, T. Hiyama, Y. Ichikawa, W.-L. Duan, R. Shintani and T. Hayashi, *J. Am. Chem. Soc.*, 2007, **129**, 9137–9143; (c) Y. Nakao, H. Imanaka, J. Chen, A. Yada and T. Hiyama, *J. Organomet. Chem.*, 2007, **692**, 585–603.

3 S. Acherar, G. Audran, F. Fotiadu and H. Monti, *Eur. J. Org. Chem.*, 2004, 5092–5099.

4 T. Eicher, F. Servet and A. Speicher, *Synthesis*, 1996, 863–870.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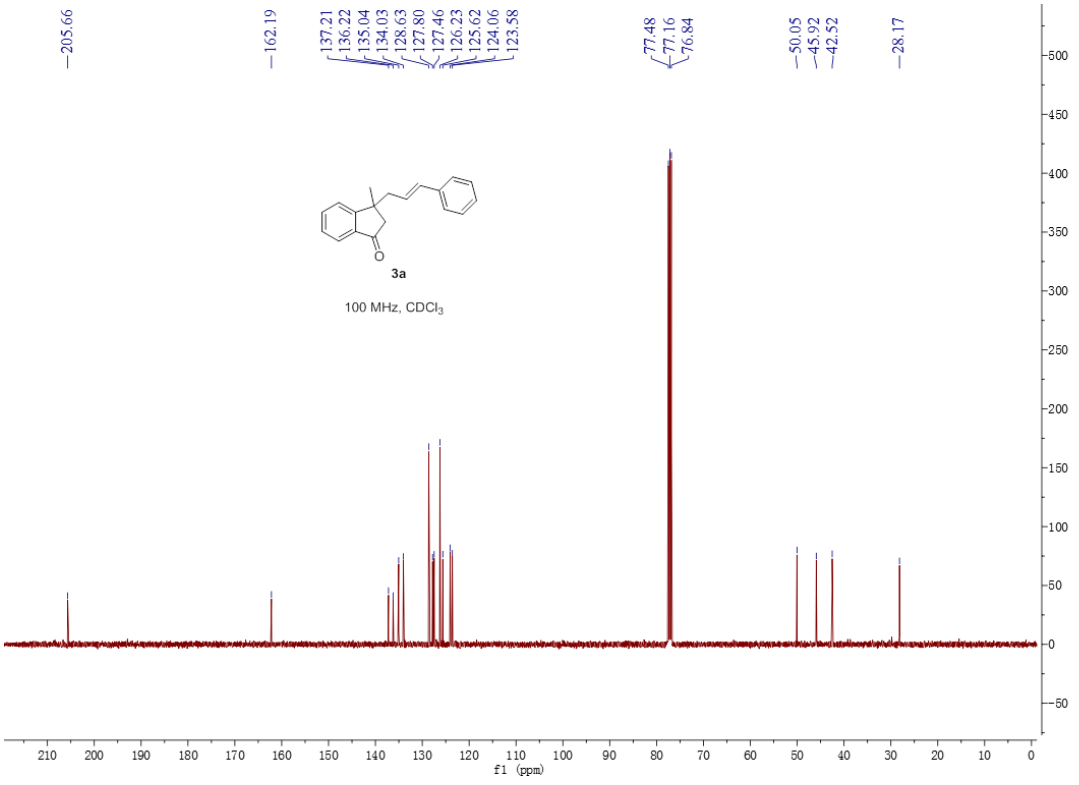
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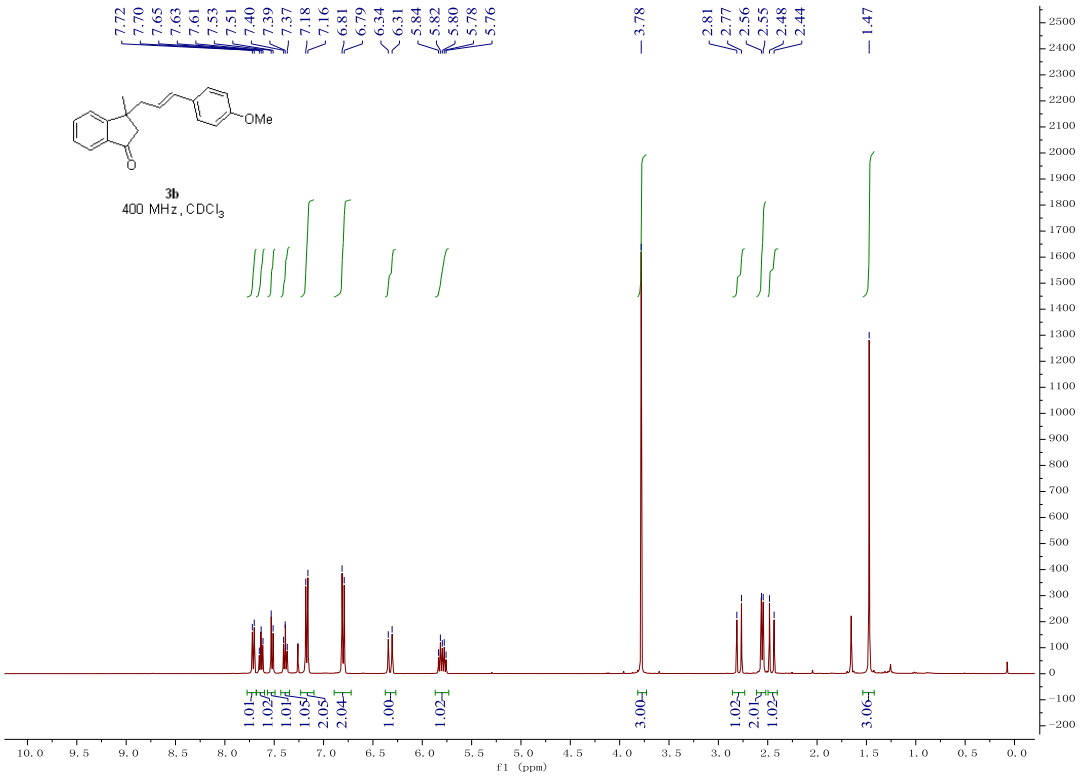
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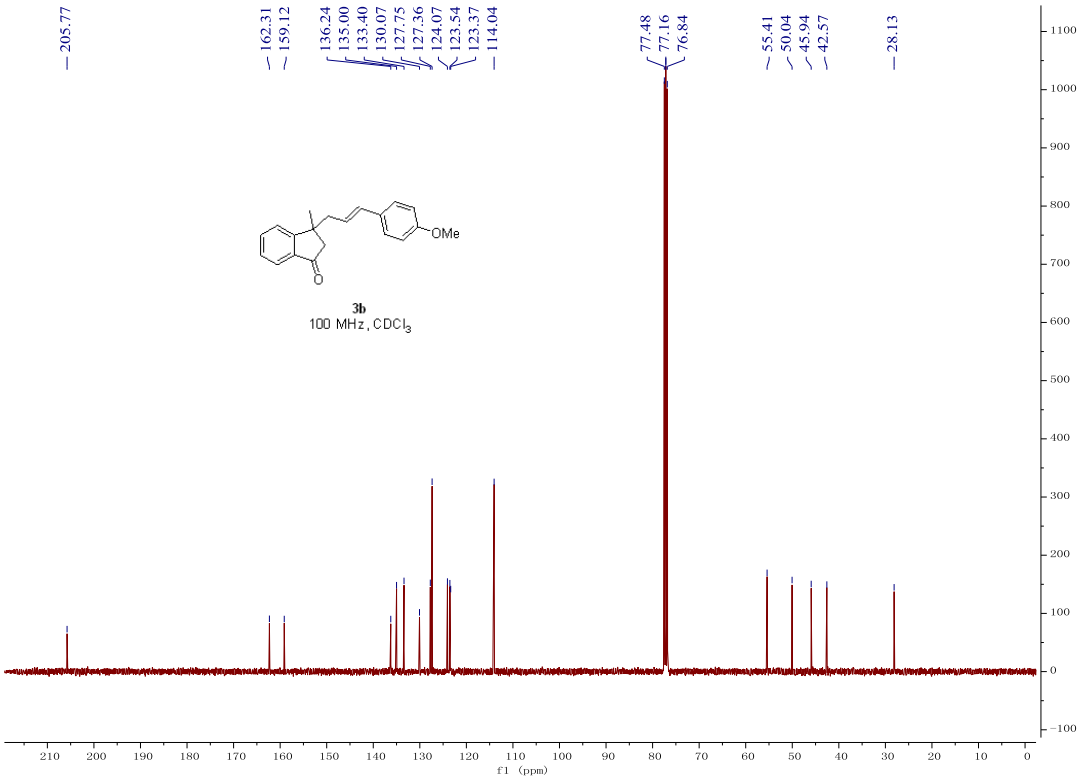
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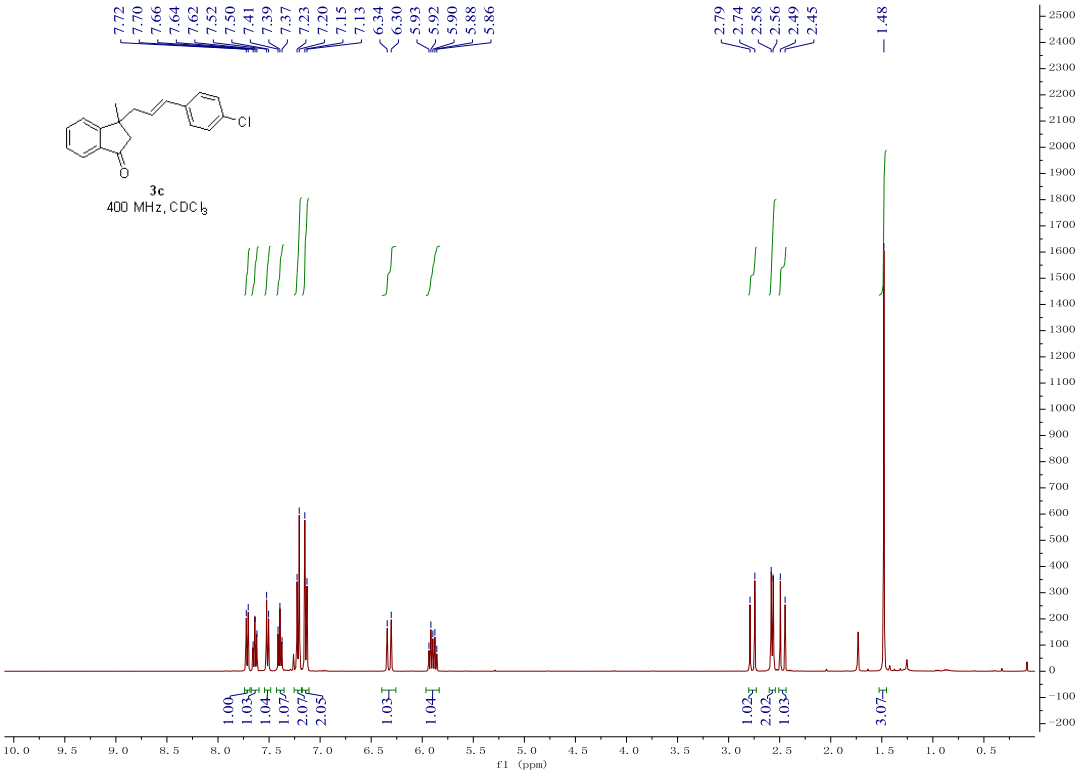
f1 (ppm)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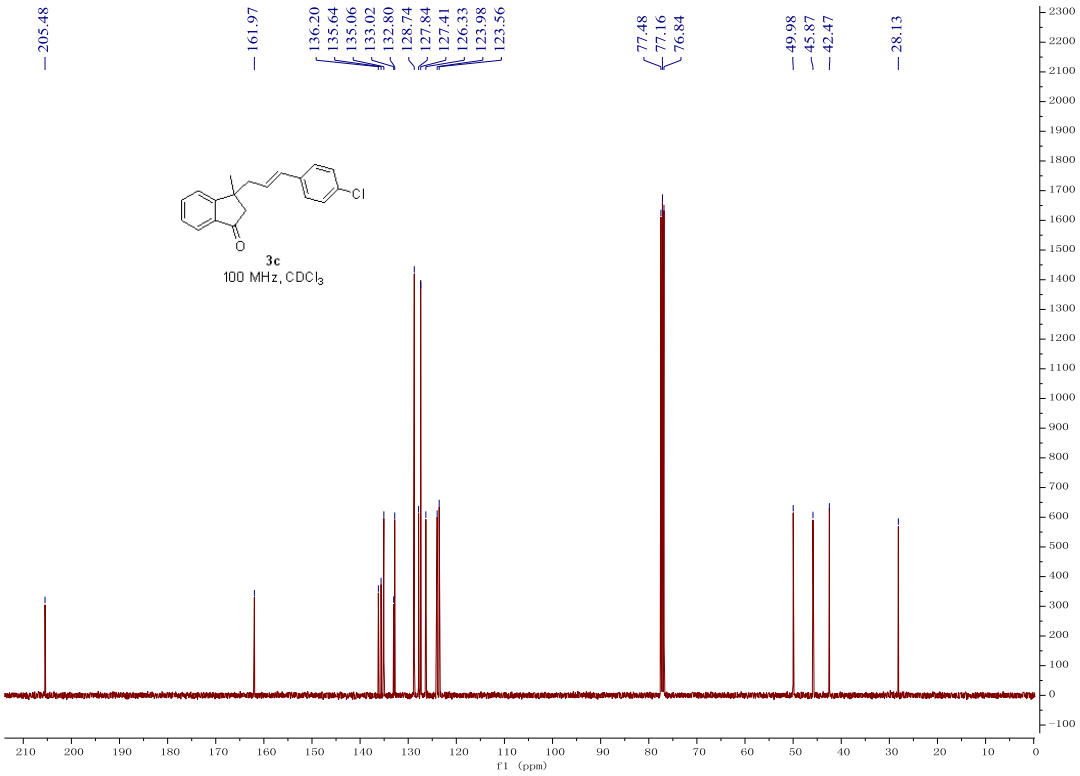
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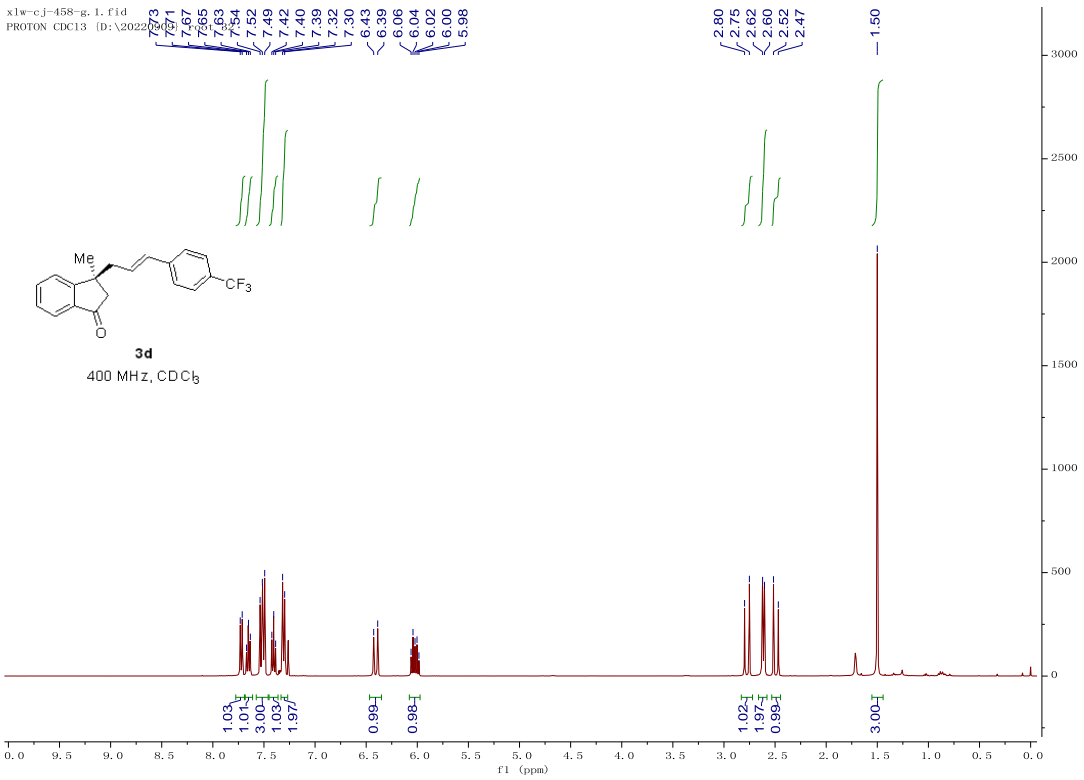
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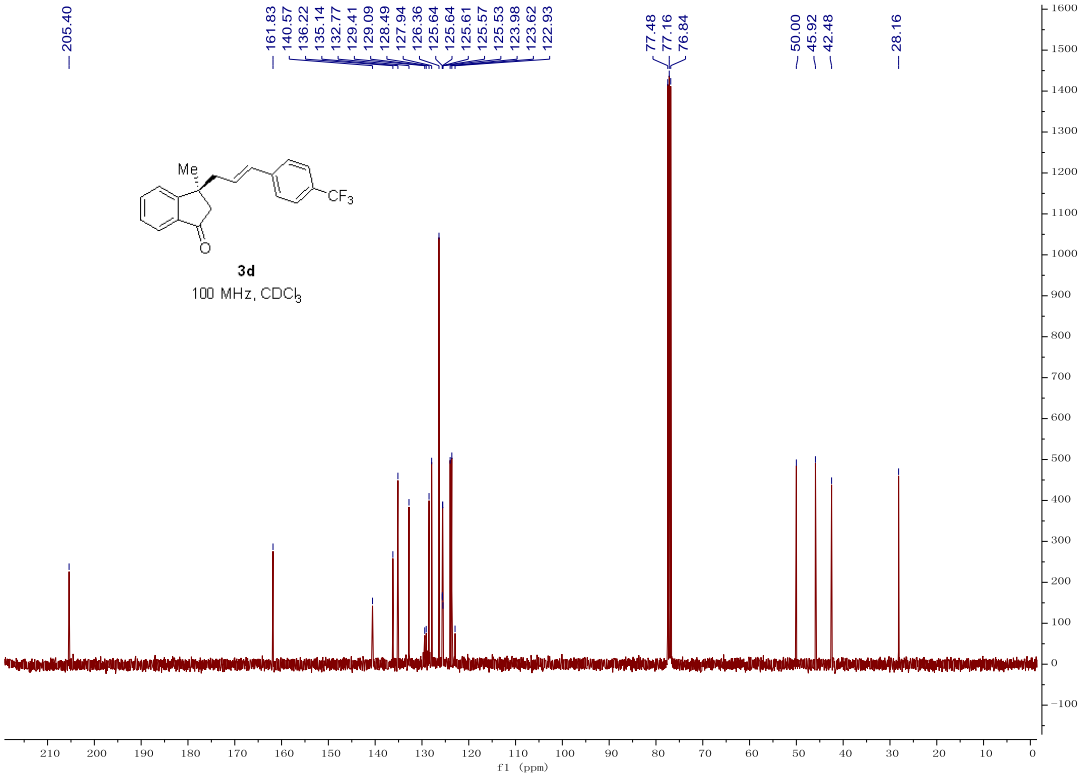
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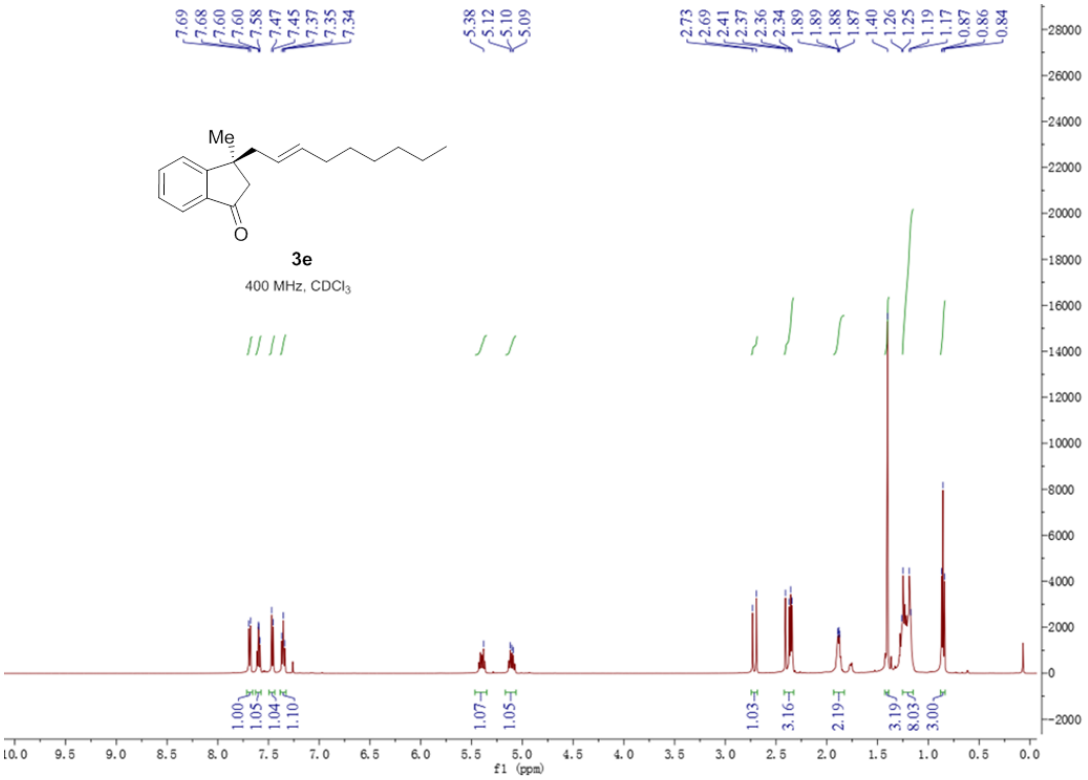
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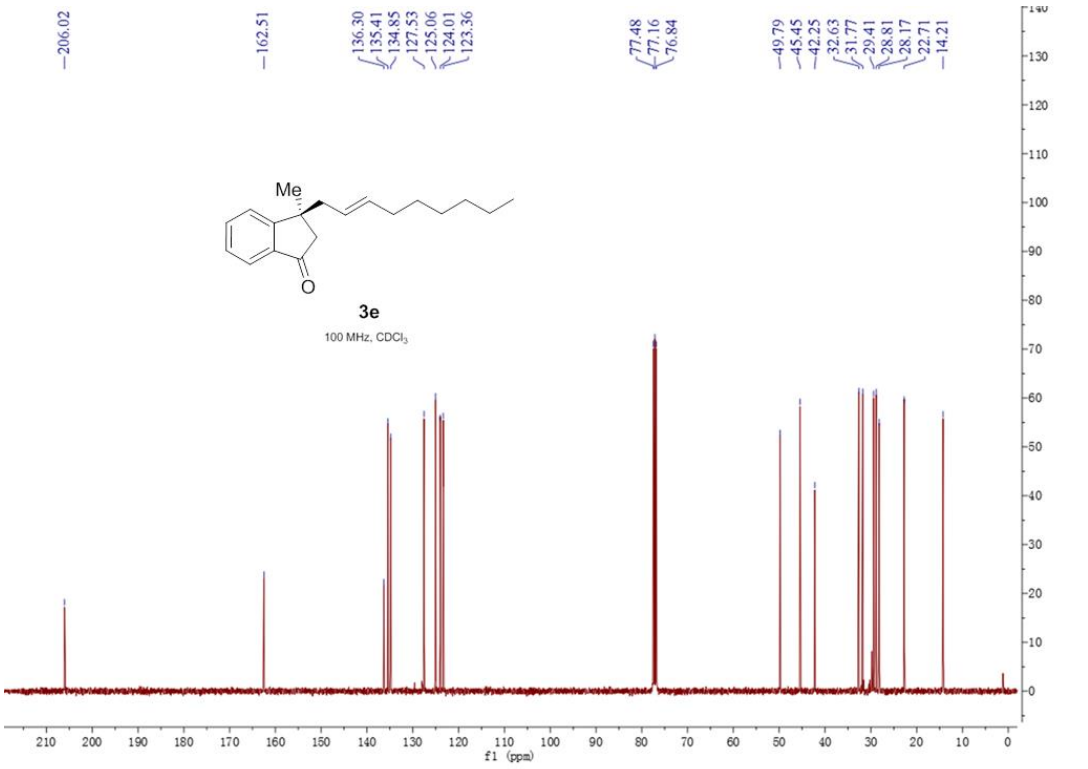
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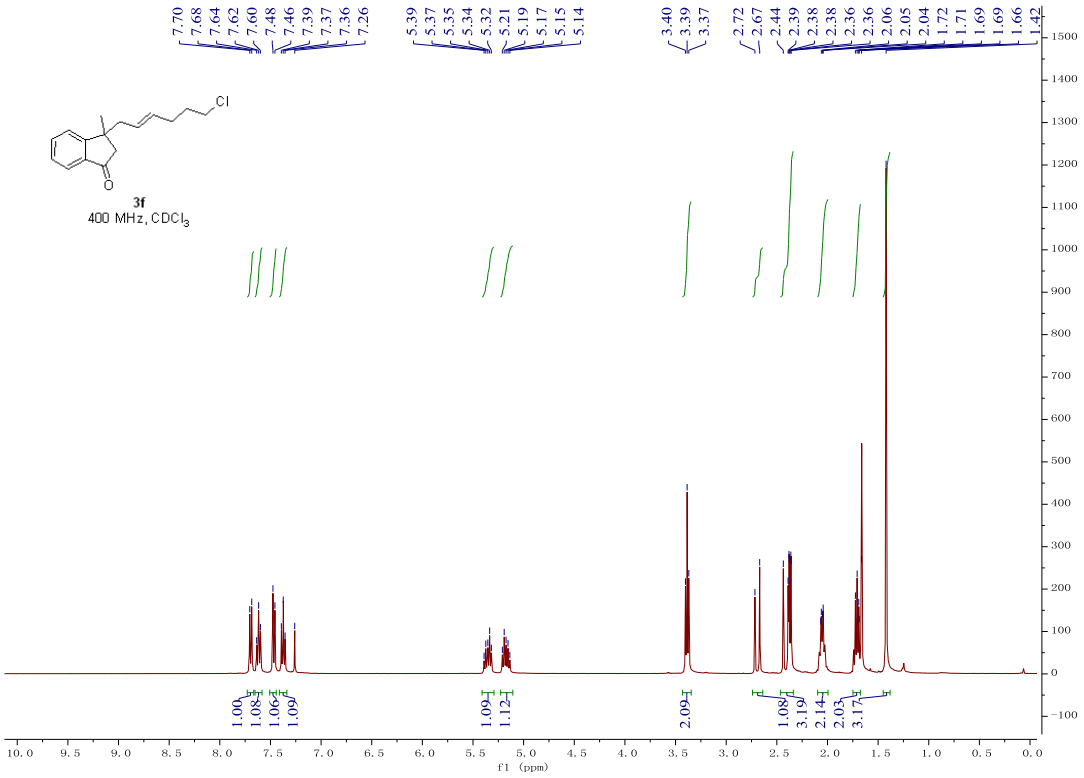
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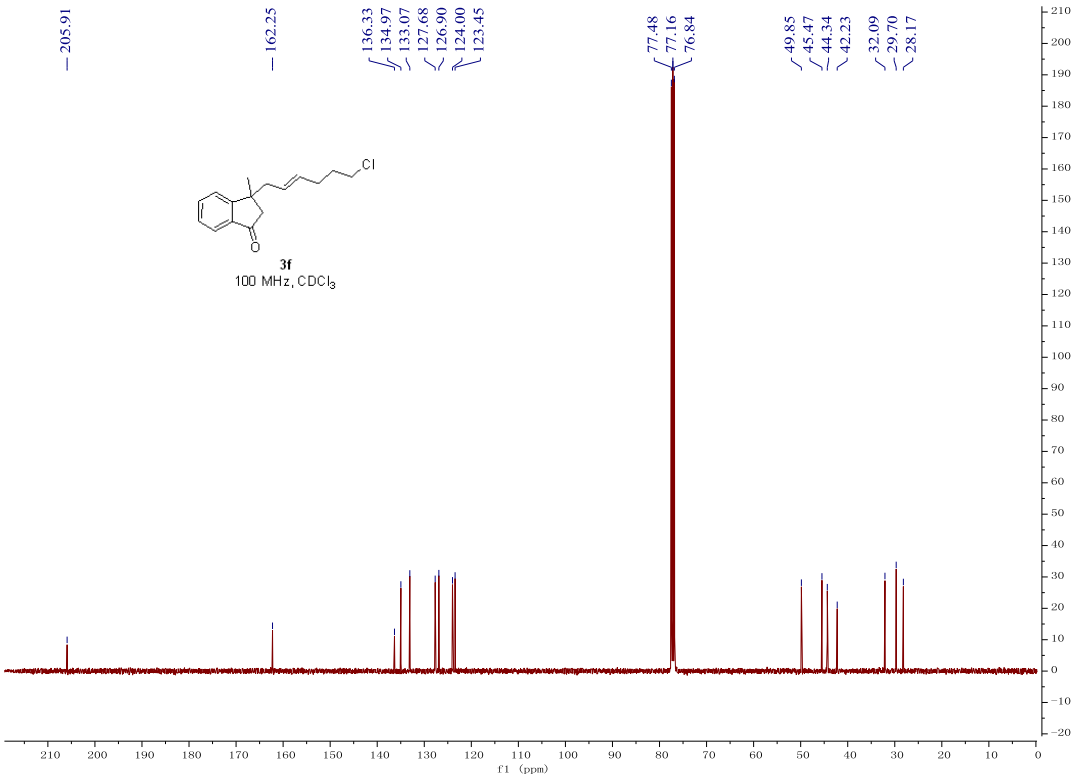
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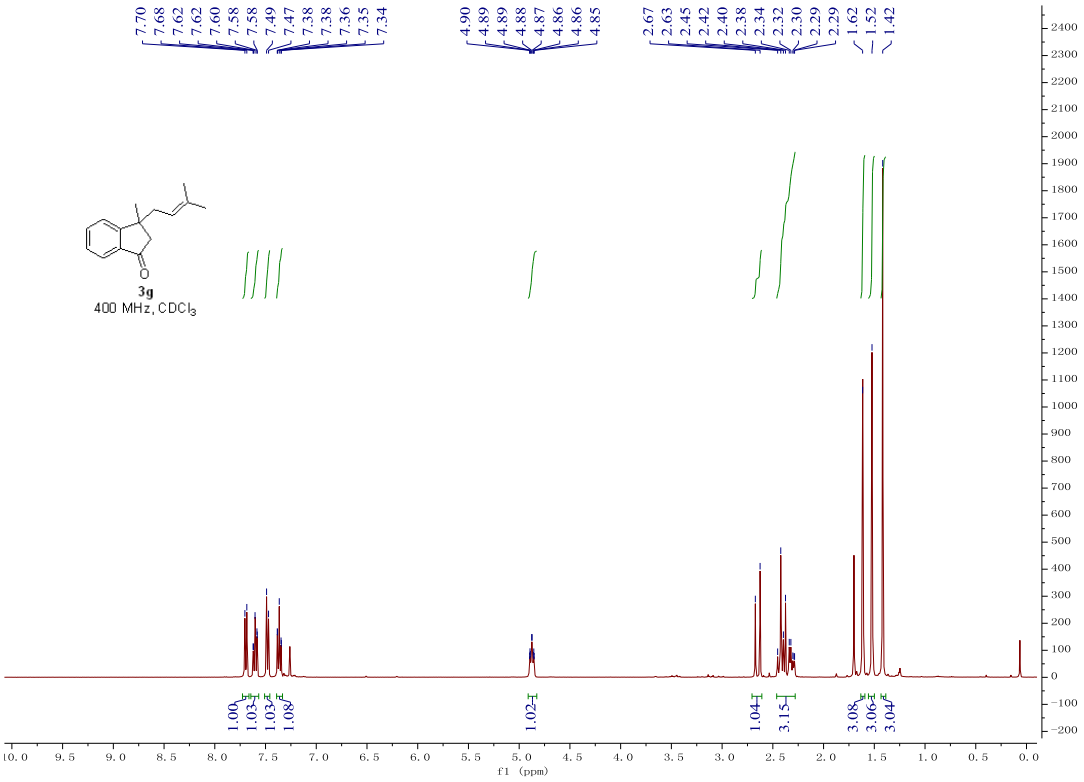
100 MHz, CDCl3
f1 (ppm)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7.62
7.62
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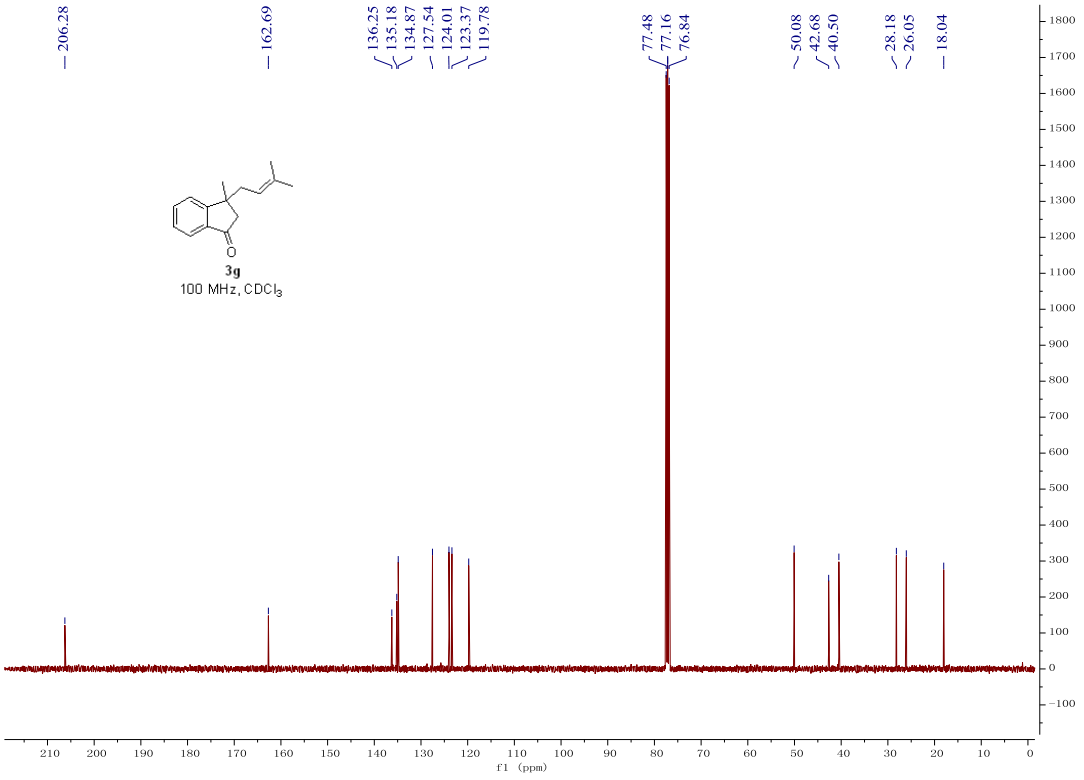
7.58
7.58
7.49
7.47
7.38
7.38
7.36
7.35
7.34
4.90
4.89
4.89
4.88
4.87
4.86
4.86
4.85
2.67
2.63
2.45
2.42
2.40
2.38
2.34
2.32
2.30
2.29
2.29
1.62
1.52
1.42
3g
400 MHz, CDCl3
1.00
1.03
1.03
1.08
1.02
1.04
3.15
3.08
3.06
3.04
f1 (ppm)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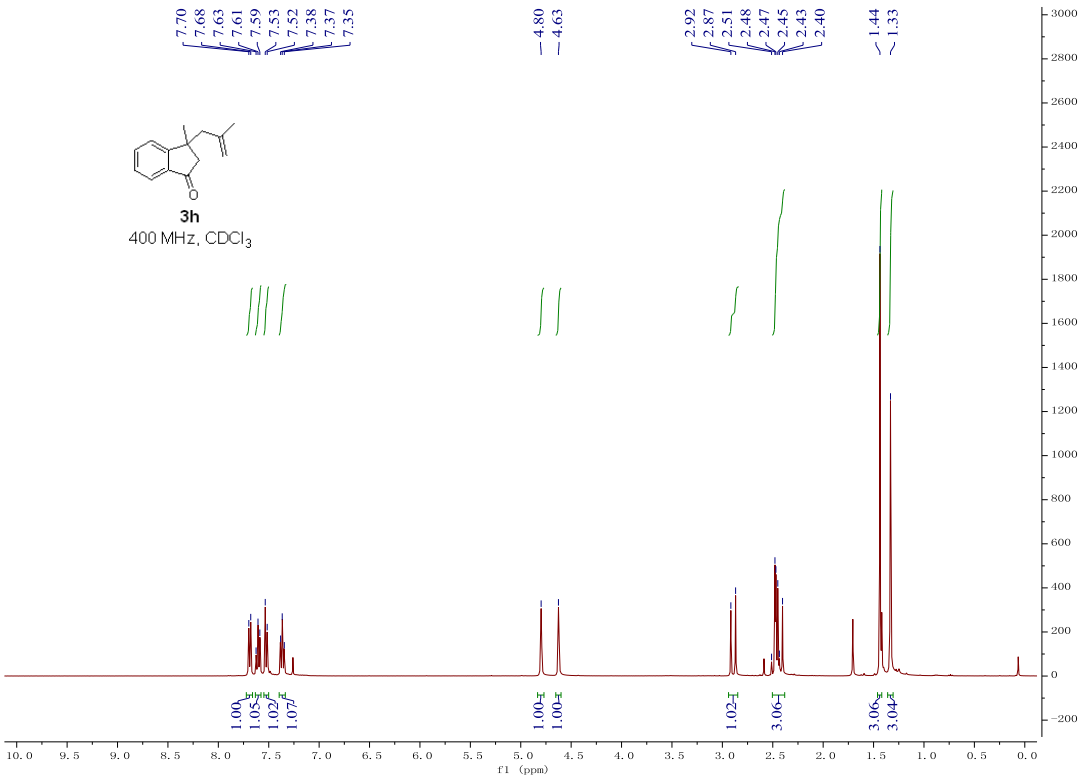
135.18
134.87
127.54
124.01
123.37
119.78
77.48
77.16
76.84
50.08
42.68
40.50
28.18
26.05
18.04
3g
100 MHz, CDCl3
f1 (ppm)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7.63
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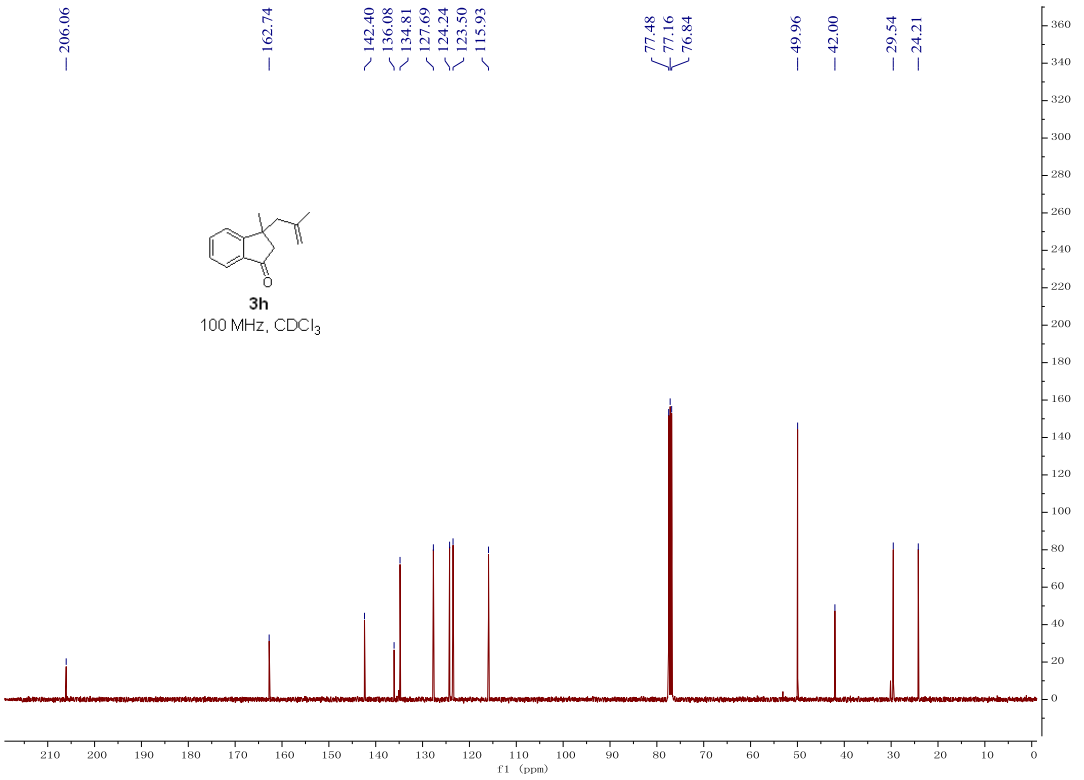
7.53
7.52
7.38
7.37
7.35
4.80
4.63
2.92
2.87
2.51
2.48
2.47
2.45
2.43
2.40
1.44
1.33
3h
400 MHz, CDCl3
1.00
1.05
1.02
1.07
1.00
1.00
1.02
3.06
3.06
3.04
f1 (ppm)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123.50
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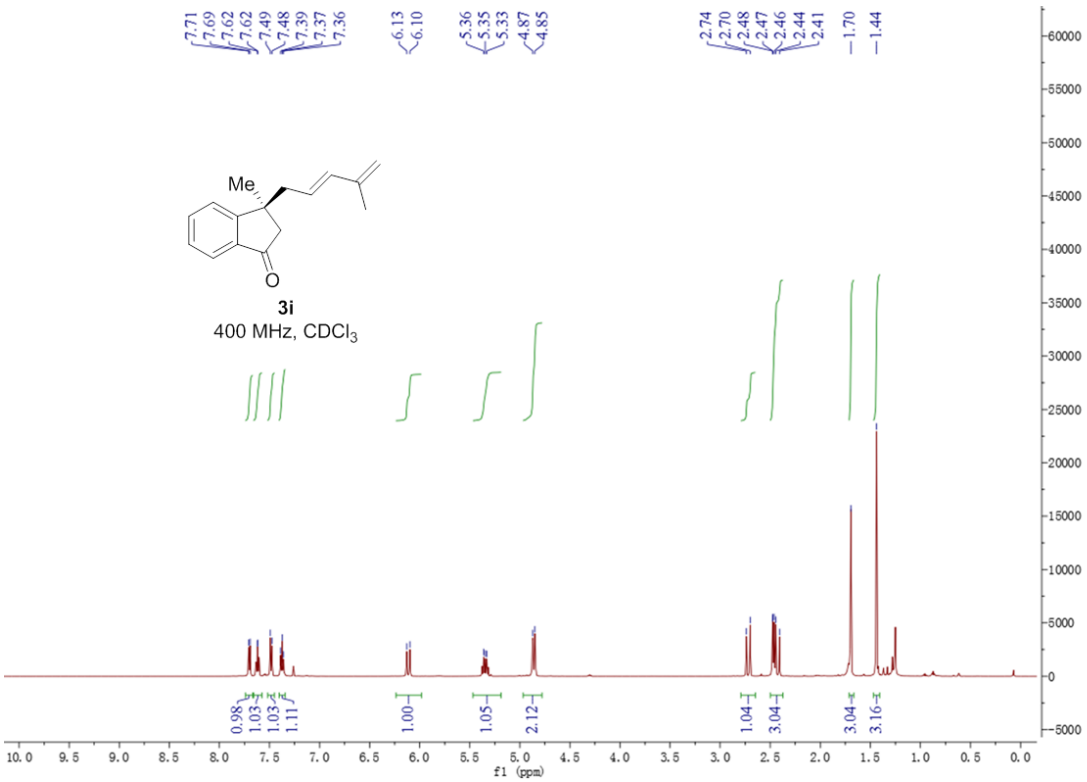
76.84
49.96
42.00
29.54
24.21
3h
100 MHz, CDCl3
f1 (ppm)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7.62
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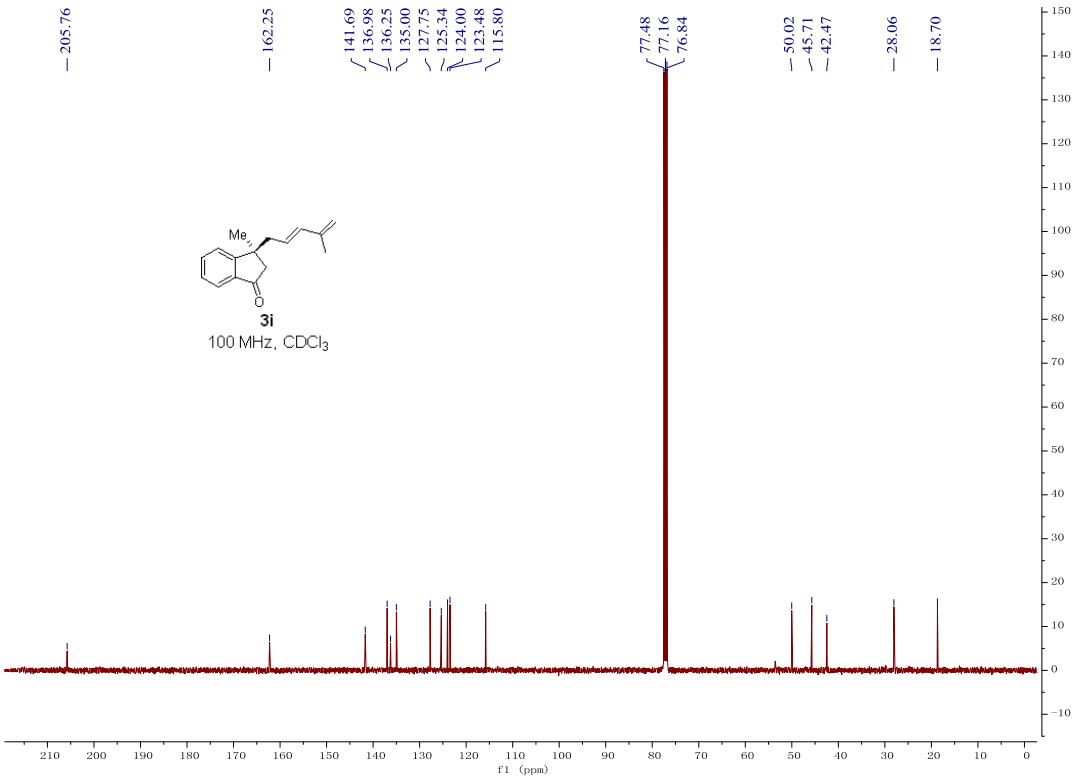
7.48
7.39
7.37
7.36
6.13
6.10
5.36
5.35
5.33
4.87
4.85
2.74
2.70
2.48
2.47
2.46
2.44
2.41
1.70
1.44
Me
O
3i
400 MHz, CDCl3
0.98
1.03
1.03
1.11
1.00
1.05
2.12
1.04
3.04
3.04
3.16
f1 (ppm)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123.48
115.80
77.48
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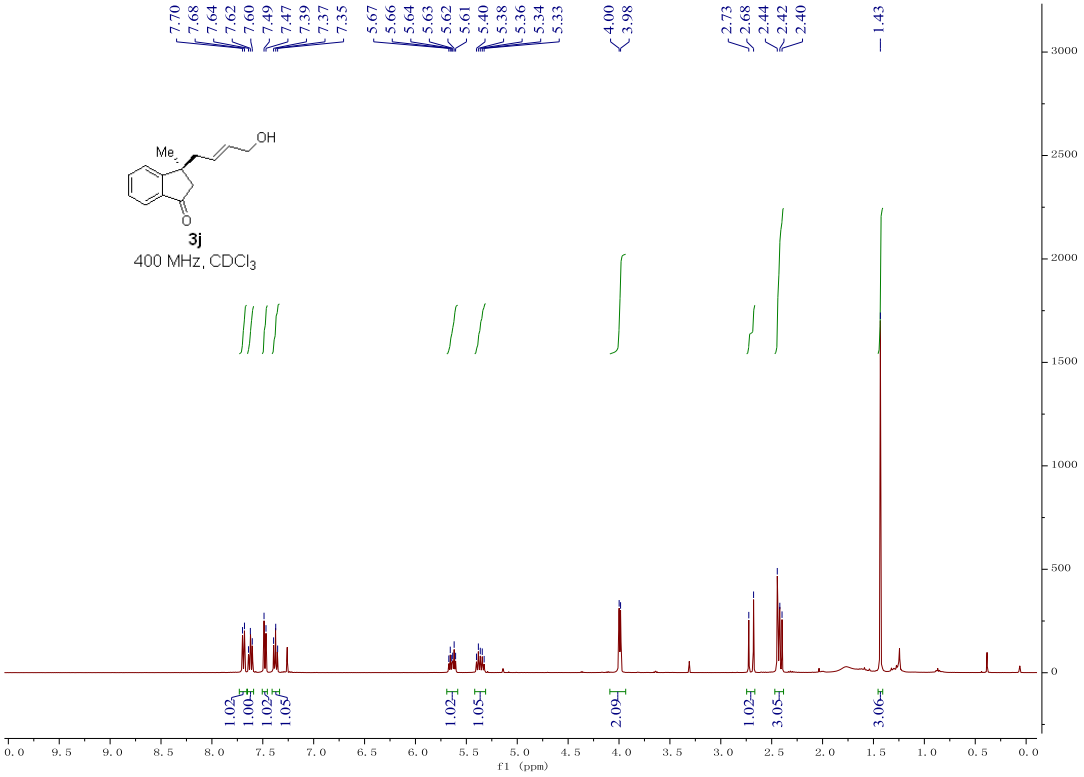
76.84
50.02
45.71
42.47
28.06
18.70
Me
O
3i
100 MHz, CDCl3
f1 (ppm)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7.35
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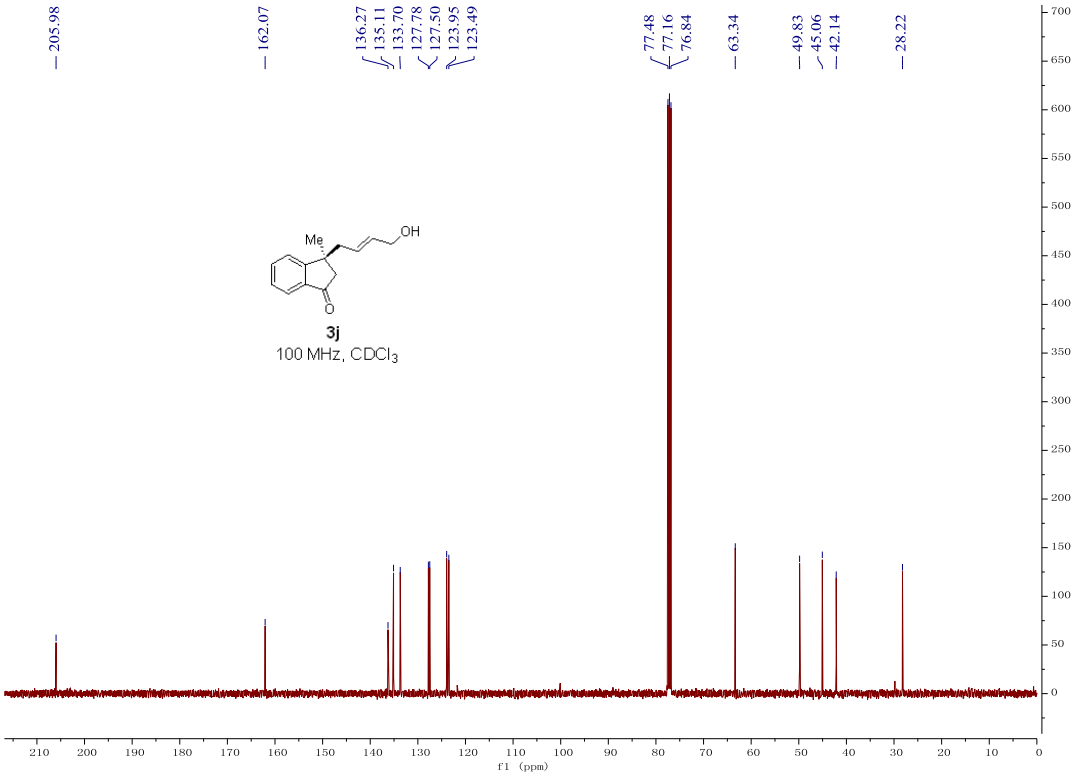
5.66
5.64
5.63
5.62
5.61
5.40
5.38
5.36
5.34
5.33
4.00
3.98
2.73
2.68
2.44
2.42
2.40
1.43
Me
OH
O
3j
400 MHz, CDCl3
1.02
1.00
1.02
1.05
1.02
1.05
2.09
1.02
3.05
3.06
f1 (ppm)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135.11
133.70
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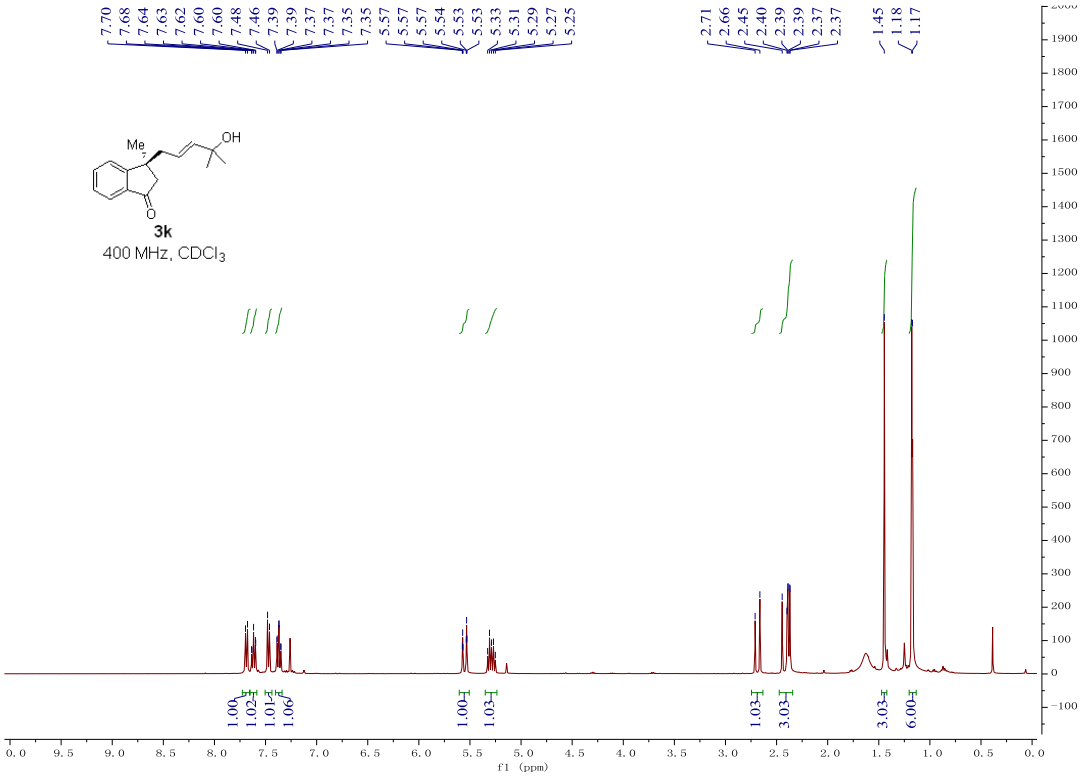
127.50
123.95
123.49
77.48
77.16
76.84
63.34
49.83
45.06
42.14
28.22
Me
OH
O
3j
100 MHz, CDCl3
f1 (ppm)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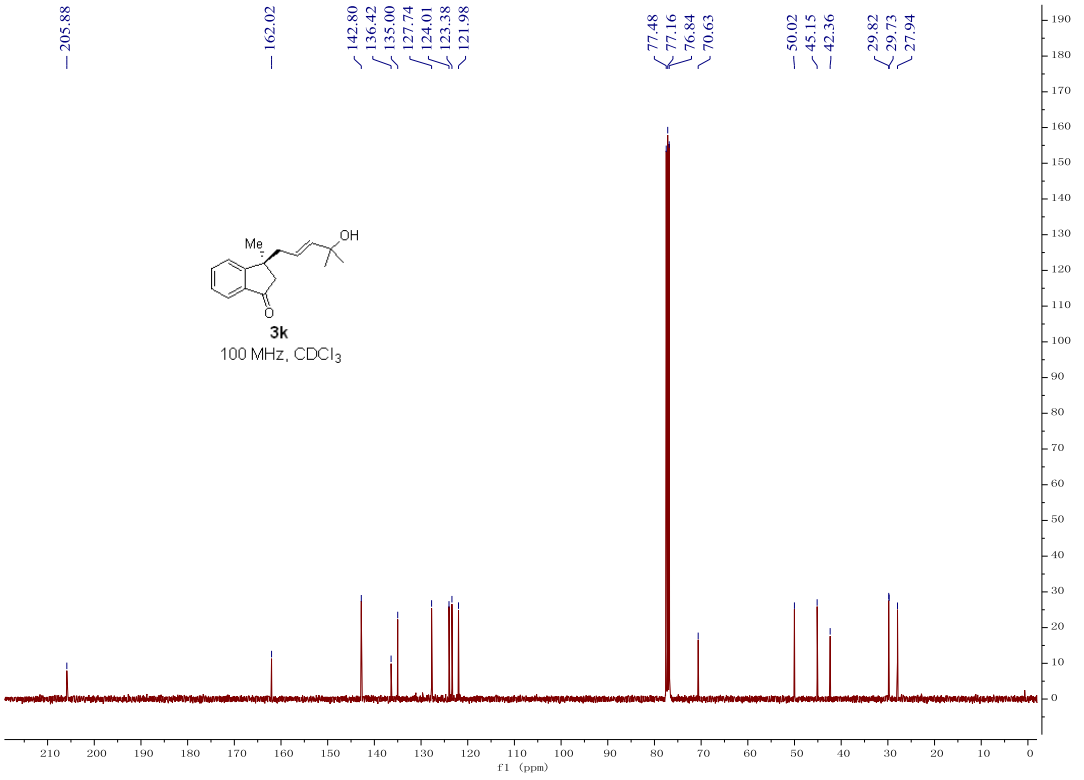
7.60
7.48
7.46
7.39
7.39
7.37
7.37
7.35
7.35
5.57
5.57
5.57
5.54
5.53
5.53
5.33
5.31
5.29
5.27
5.25
2.71
2.66
2.45
2.40
2.39
2.39
2.37
2.37
1.45
1.18
1.17
Me
OH
O
3k
400 MHz, CDCl3
1.00
1.02
1.01
1.06
1.00
1.03
1.03
3.03
3.03
6.00
f1 (ppm)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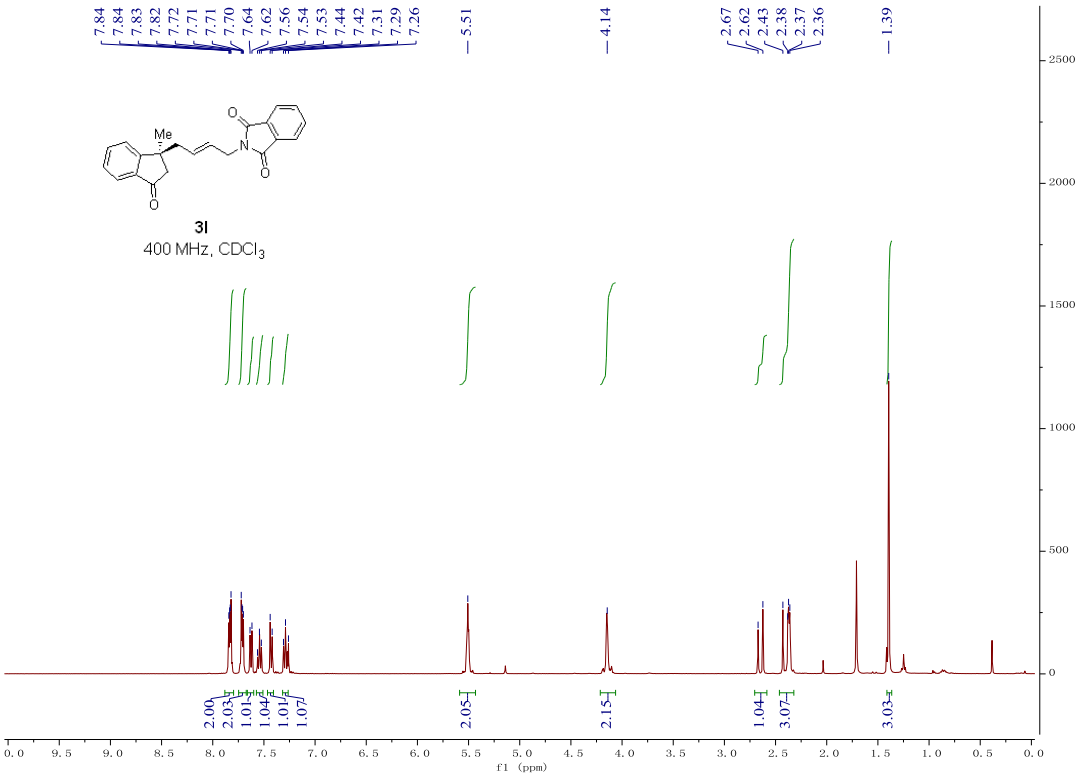
142.80
136.42
135.00
127.74
124.01
123.38
121.98
77.48
77.16
76.84
70.63
50.02
45.15
42.36
29.82
29.73
27.94
Me
OH
O
3k
100 MHz, CDCl3
f1 (ppm)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7.84
7.83
7.82
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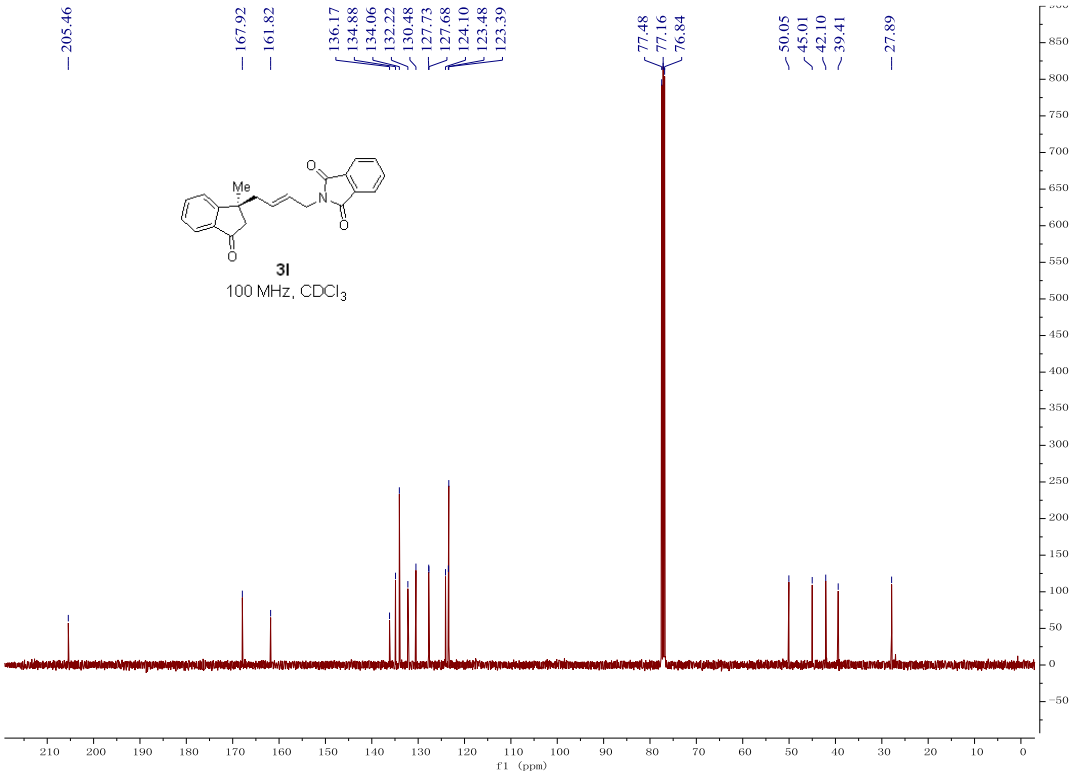
7.71
7.71
7.70
7.64
7.62
7.56
7.54
7.53
7.44
7.42
7.31
7.29
7.26
5.51
4.14
2.67
2.62
2.43
2.38
2.37
2.36
1.39
Me
O
N
O
O
3l
400 MHz, CDCl3
2.00
2.03
1.01
1.04
1.01
1.07
2.05
2.15
1.04
3.07
3.03
f1 (ppm)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136.17
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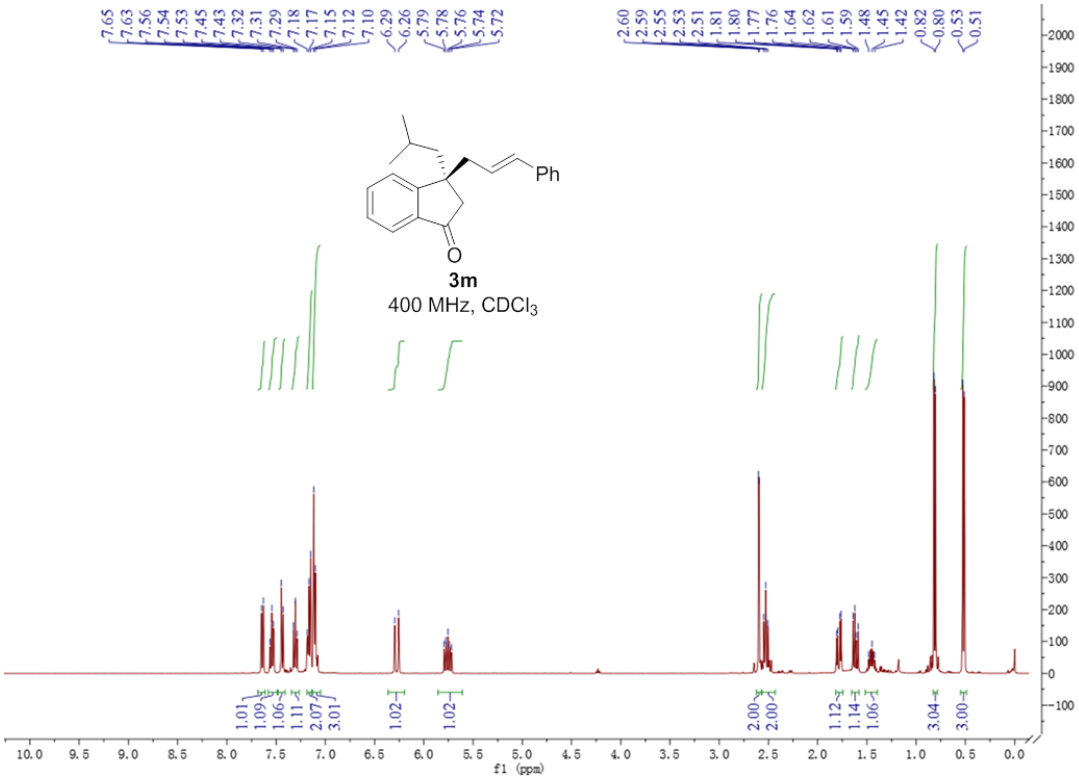
134.06
132.22
130.48
127.73
127.68
124.10
123.48
123.39
77.48
77.16
76.84
50.05
45.01
42.10
39.41
27.89
Me
O
N
O
O
3l
100 MHz, CDCl3
f1 (ppm)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7.45
7.43
7.32
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7.29
7.18
7.17
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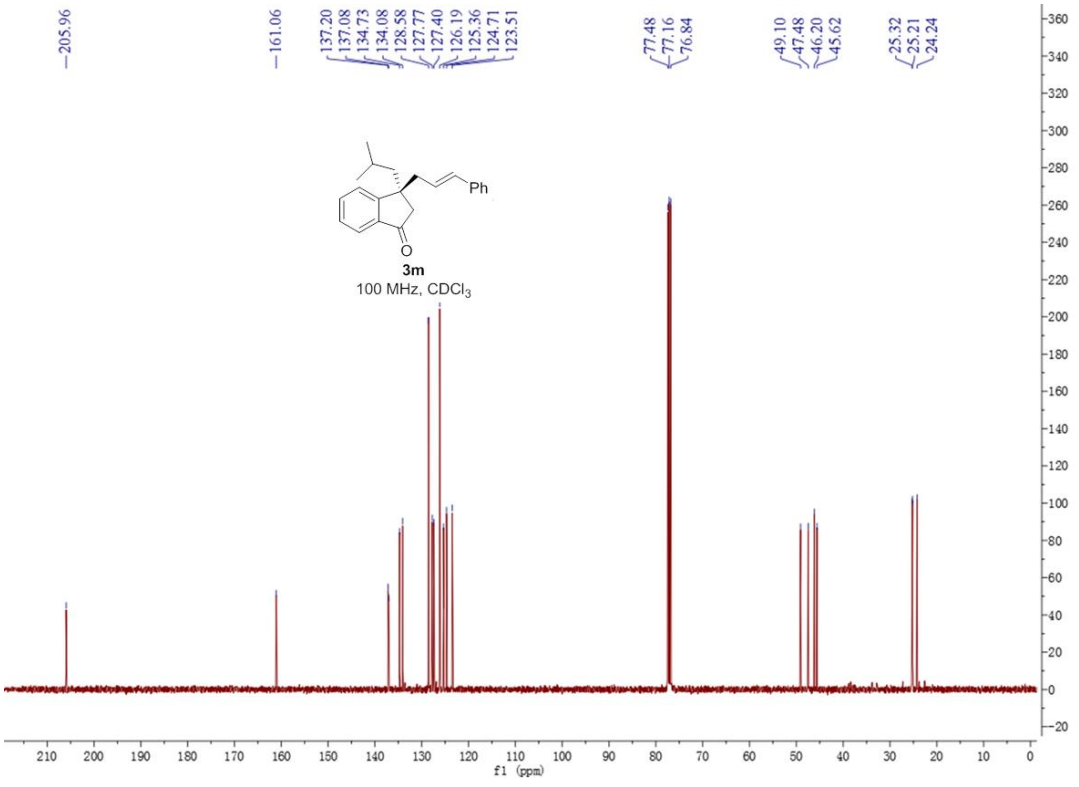
7.12
7.10
6.29
6.26
5.79
5.78
5.76
5.74
5.72
2.60
2.59
2.55
2.53
2.51
1.81
1.80
1.77
1.76
1.64
1.62
1.61
1.59
1.48
1.45
1.42
0.82
0.80
0.53
0.51
Ph
O
3m
400 MHz, CDCl3
1.01
1.09
1.06
1.11
2.07
3.01
1.02
1.02
2.00
2.00
1.12
1.14
1.06
3.04
3.00
f1 (ppm)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134.08
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127.77
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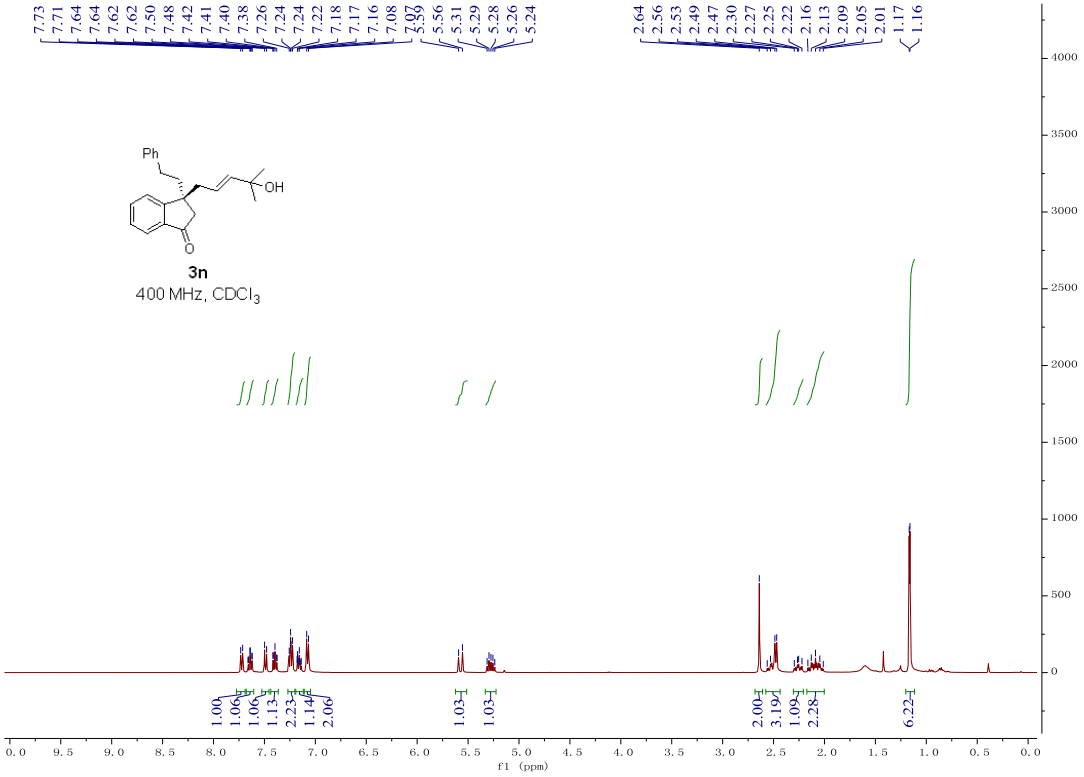
126.19
125.36
124.71
123.51
77.48
77.16
76.84
49.10
47.48
46.20
45.62
25.32
25.21
24.24
Ph
O
3m
100 MHz, CDCl3
f1 (ppm)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7.62
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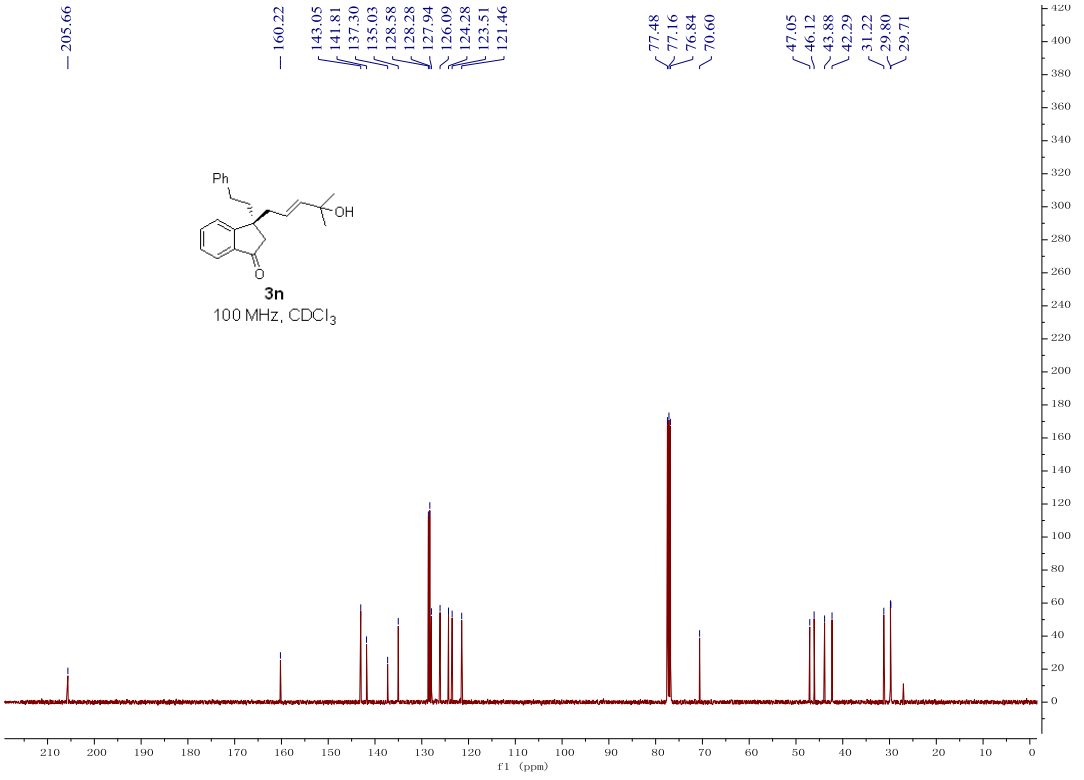
7.48
7.42
7.41
7.40
7.38
7.26
7.24
7.24
7.22
7.18
7.17
7.16
7.08
5.56
5.31
5.29
5.28
5.26
5.24
2.64
2.56
2.53
2.49
2.47
2.30
2.27
2.25
2.22
2.16
2.13
2.09
2.05
2.01
1.17
1.16
Ph
OH
O
3n
400 MHz, CDCl3
1.00
1.06
1.06
1.13
2.23
1.14
2.06
1.03
1.03
2.00
3.19
1.09
2.28
6.22
f1 (ppm)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141.81
137.30
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128.58
128.28
127.94
126.09
124.28
123.51
121.46
77.48
77.16
76.84
70.60
47.05
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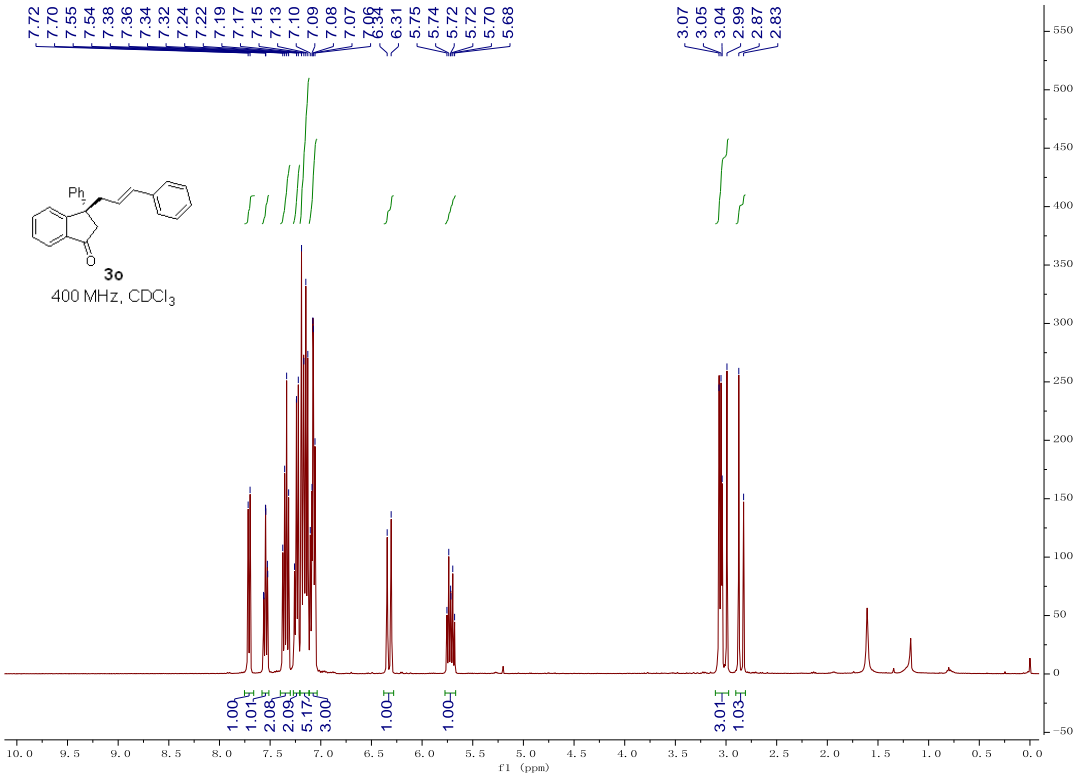
42.29
31.22
29.80
29.71
Ph
OH
O
3n
100 MHz, CDCl3
f1 (ppm)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7.54
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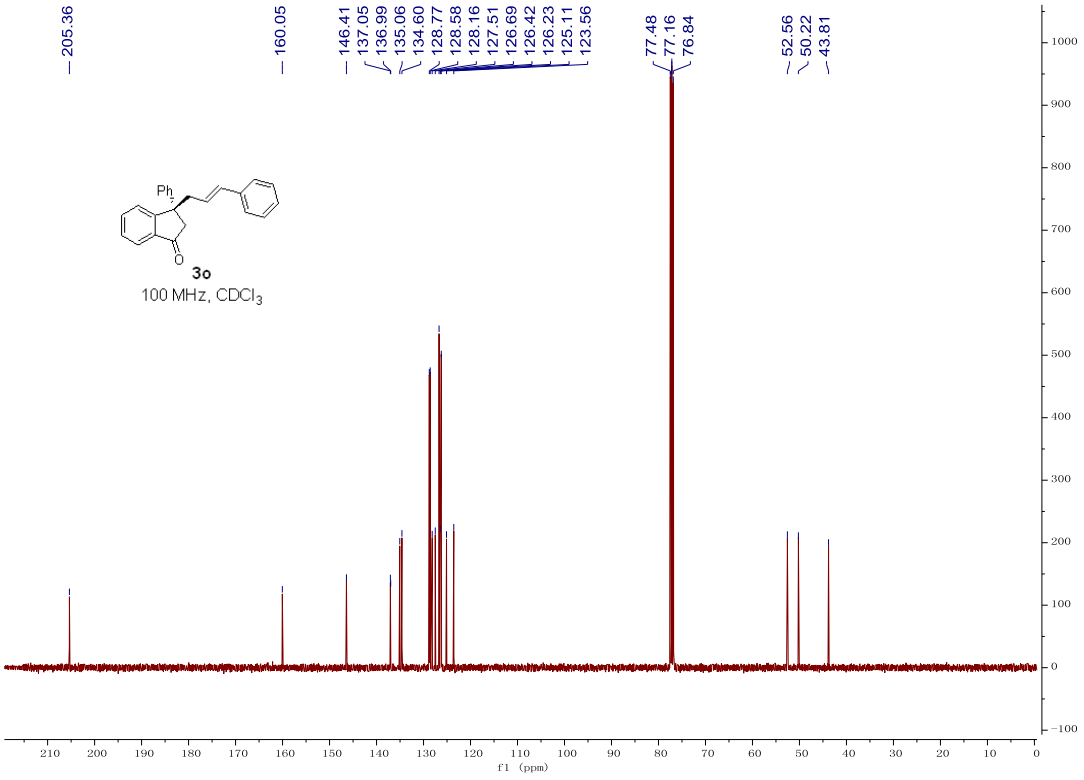
7.36
7.34
7.32
7.24
7.22
7.19
7.17
7.15
7.13
7.10
7.09
7.08
7.07
6.34
6.31
5.75
5.74
5.72
5.72
5.70
5.68
3.07
3.05
3.04
2.99
2.87
2.83
Ph
O
3o
400 MHz, CDCl3
1.00
1.01
2.08
2.09
5.17
3.00
1.00
1.00
3.01
1.03
f1 (ppm)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136.99
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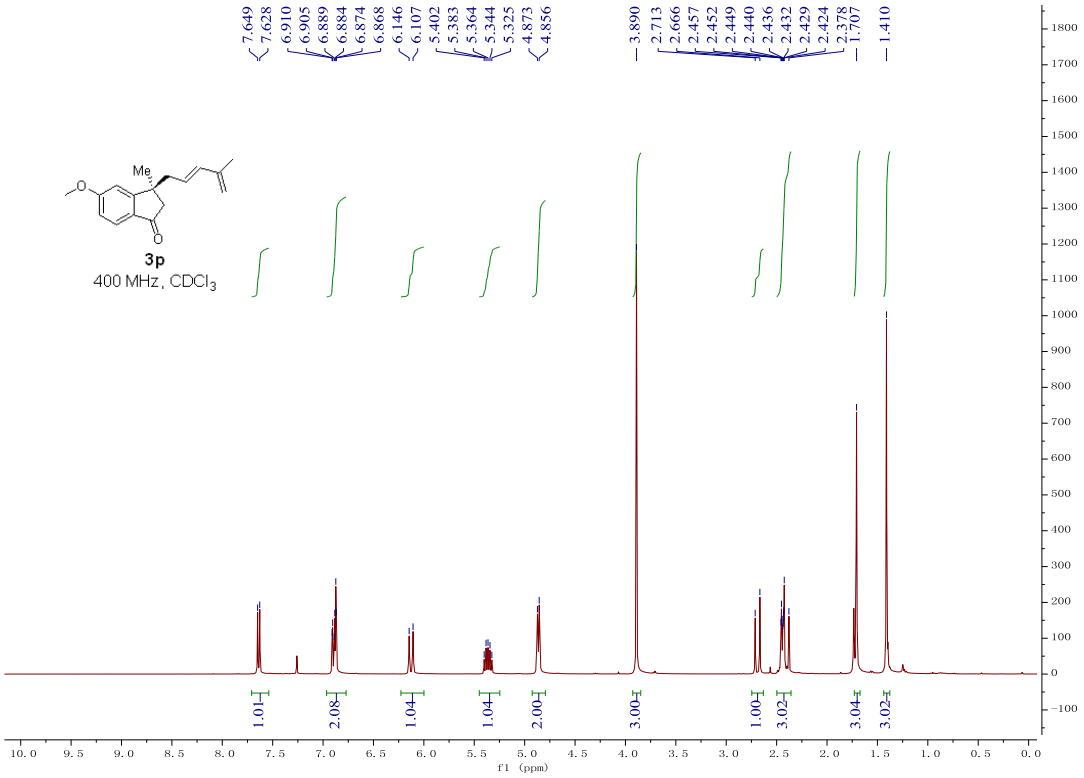
128.77
128.58
128.16
127.51
126.69
126.42
126.23
125.11
123.56
77.48
77.16
76.84
52.56
50.22
43.81
Ph
O
3o
100 MHz, CDCl3
f1 (ppm)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6.884
6.874
6.868
6.146
6.107
5.402
5.383
5.364
5.344
5.325
4.873
4.856
3.890
2.713
2.666
2.457
2.452
2.449
2.440
2.436
2.432
2.429
2.424
2.378
1.707
1.410
Me
O
O
3p
400 MHz, CDCl3
1.01
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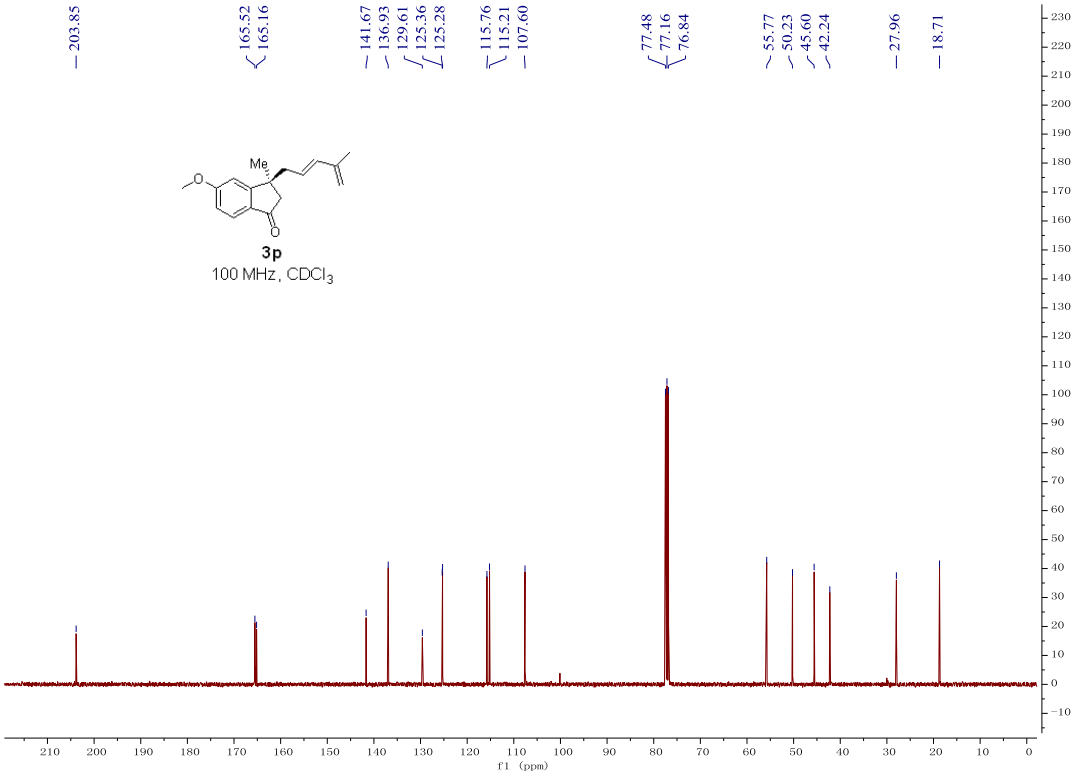
1.04
1.04
2.00
3.00
1.00
3.02
3.04
3.02
f1 (ppm)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125.28
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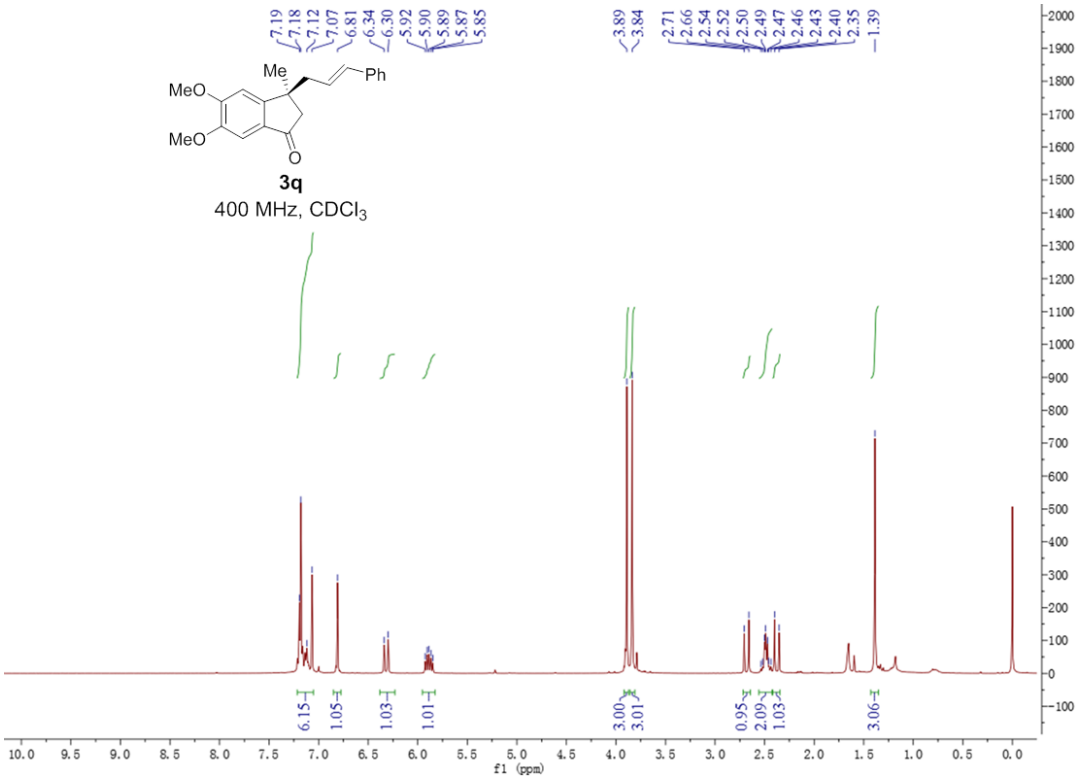
115.21
107.60
77.48
77.16
76.84
55.77
50.23
45.60
42.24
27.96
18.71
Me
O
O
3p
100 MHz, CDCl3
f1 (ppm)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5.89
5.87
5.85
3.89
3.84
2.71
2.66
2.54
2.52
2.50
2.49
2.47
2.46
2.43
2.40
2.35
1.39
Me
Ph
MeO
MeO
O
3q
400 MHz, CDCl3
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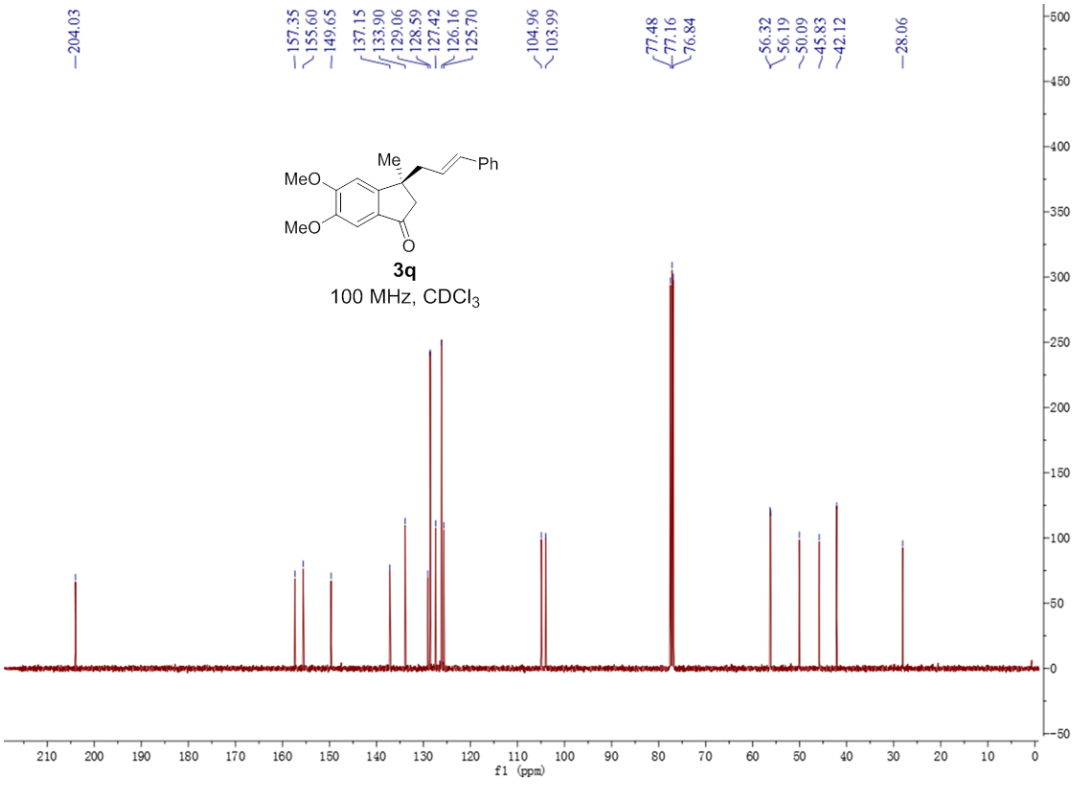
1.01
3.00
3.01
0.95
2.09
1.03
3.06
f1 (ppm)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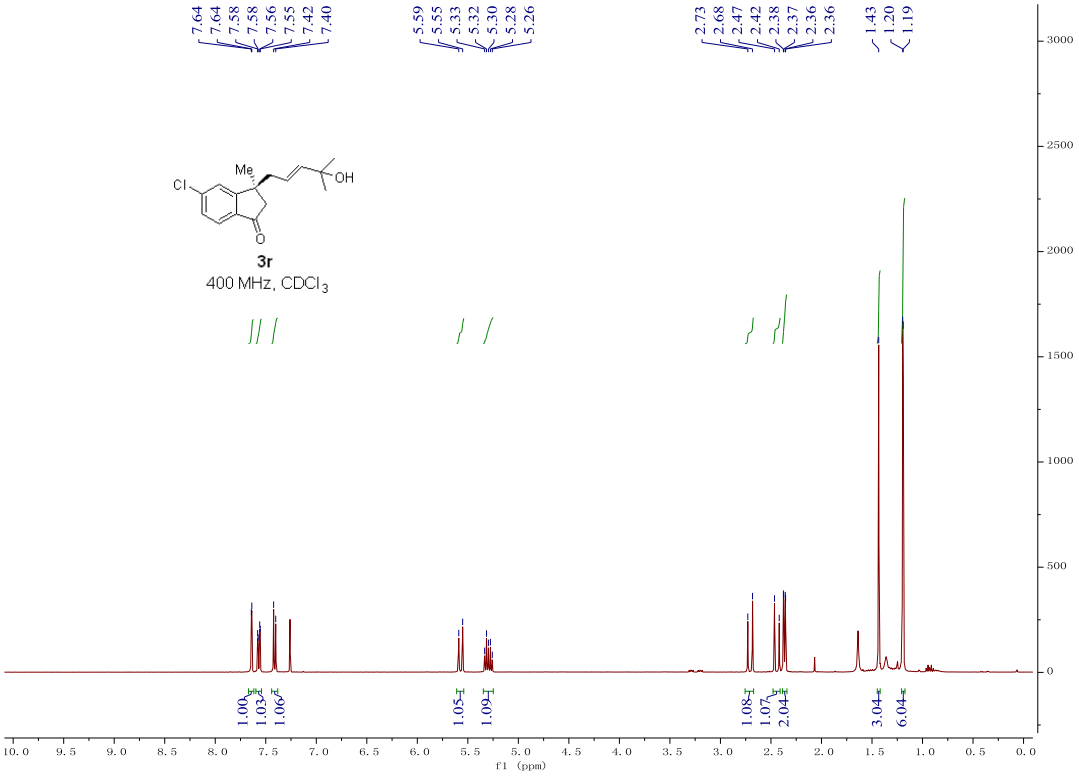
129.06
128.59
127.42
126.16
125.70
104.96
103.99
77.48
77.16
76.84
56.32
56.19
50.09
45.83
42.12
28.06
Me
Ph
MeO
MeO
O
3q
100 MHz, CDCl3
f1 (ppm)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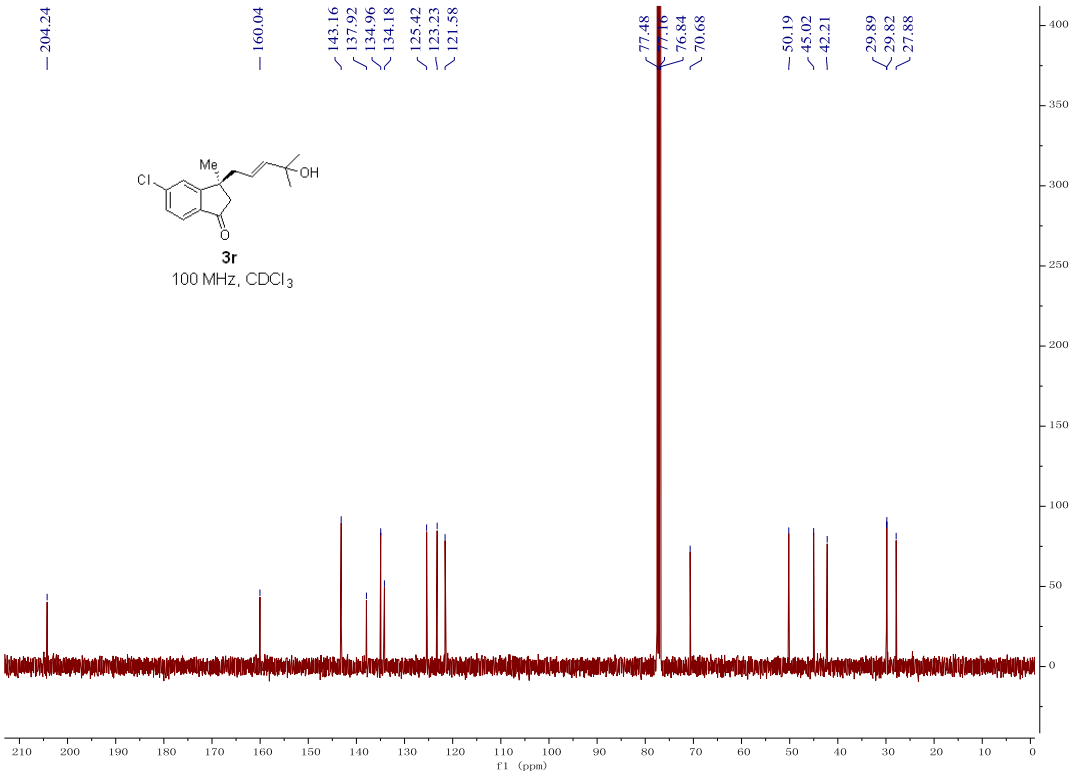
5.55
5.33
5.32
5.30
5.28
5.26
2.73
2.68
2.47
2.42
2.38
2.37
2.36
2.36
1.43
1.20
1.19
Me
Cl
OH
O
3r
400 MHz, CDCl3
1.00
1.03
1.06
1.05
1.09
1.08
1.07
2.04
3.04
6.04
f1 (ppm)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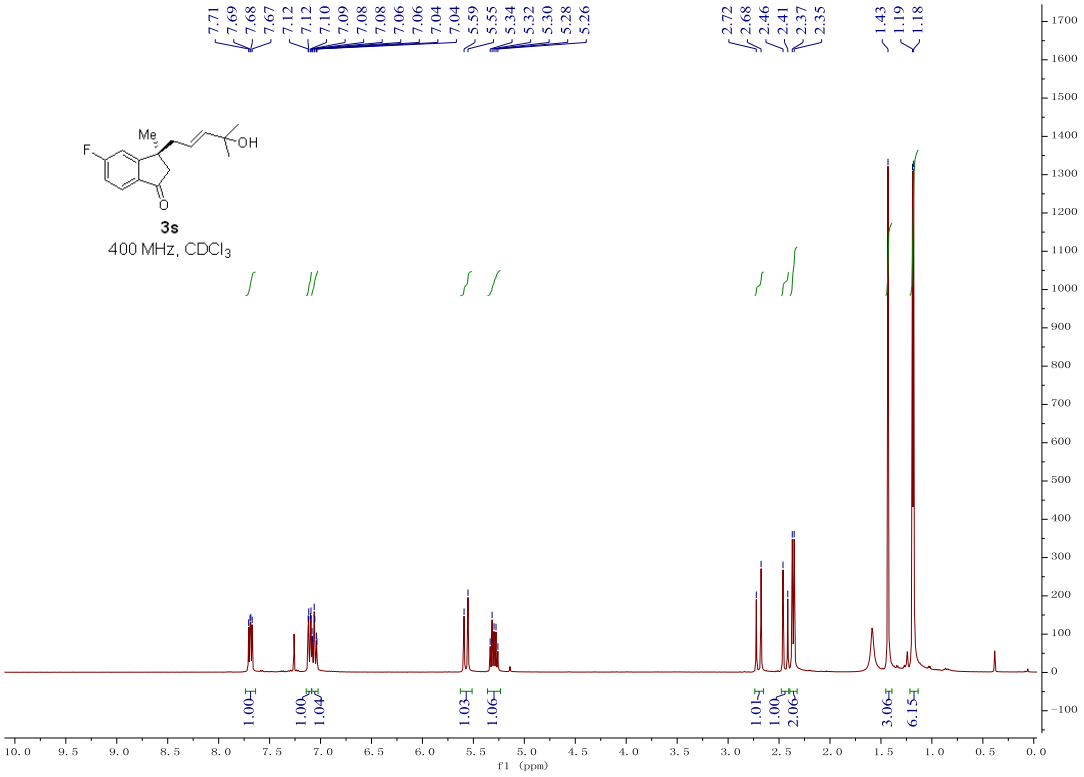
134.96
134.18
125.42
123.23
121.58
77.48
77.16
76.84
70.68
50.19
45.02
42.21
29.89
29.82
27.88
Me
Cl
OH
O
3r
100 MHz, CDCl3
f1 (ppm)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7.67
7.12
7.12
7.10
7.09
7.08
7.08
7.06
7.06
7.04
7.04
5.59
5.55
5.34
5.32
5.30
5.28
5.26
2.72
2.68
2.46
2.41
2.37
2.35
1.43
1.19
1.18
Me
F
OH
O
3s
400 MHz, CDCl3
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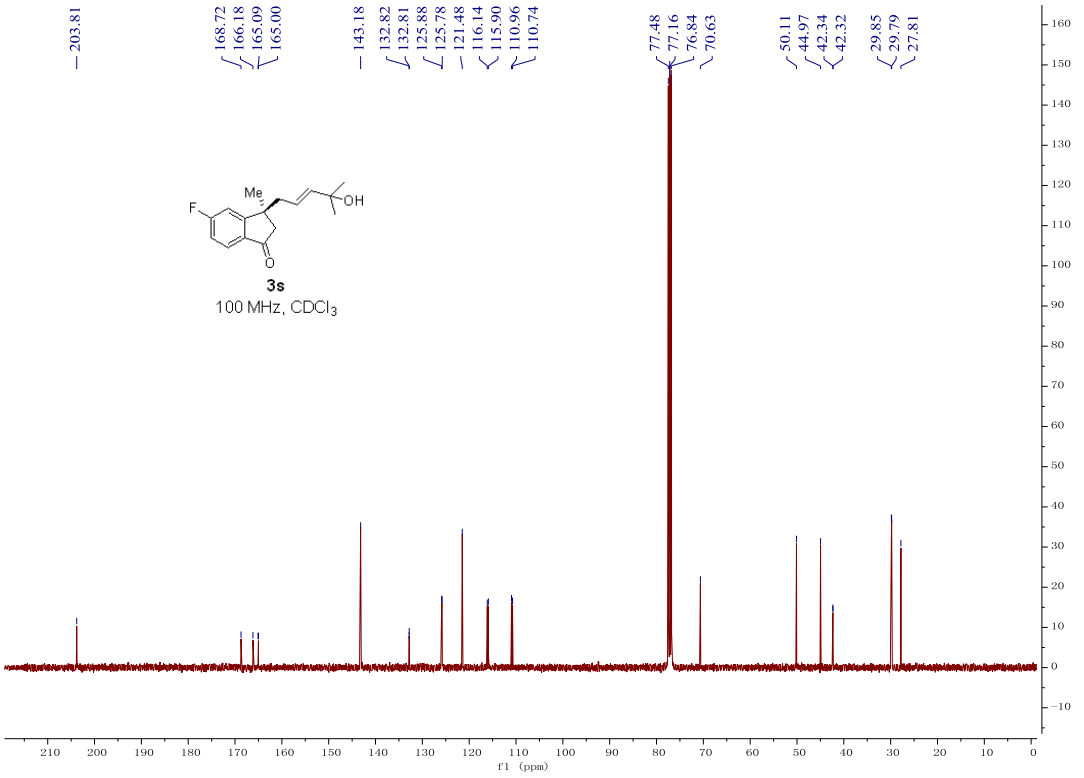
1.04
1.03
1.06
1.01
1.00
2.06
3.06
6.15
f1 (ppm)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132.81
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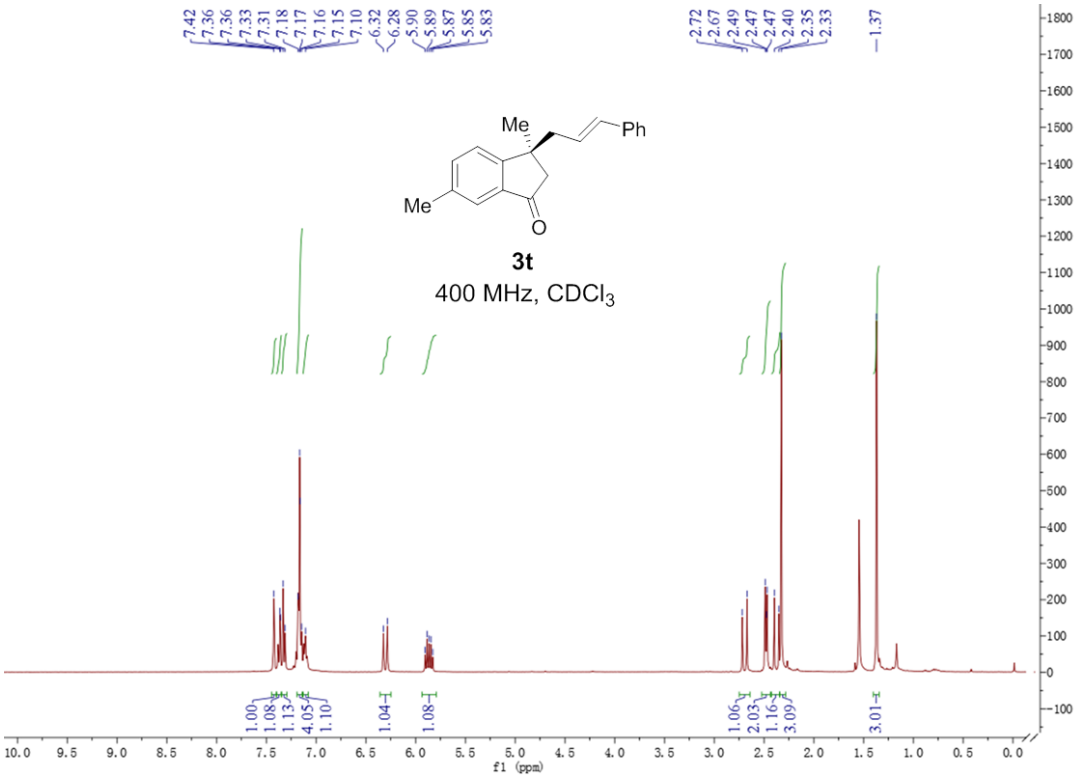
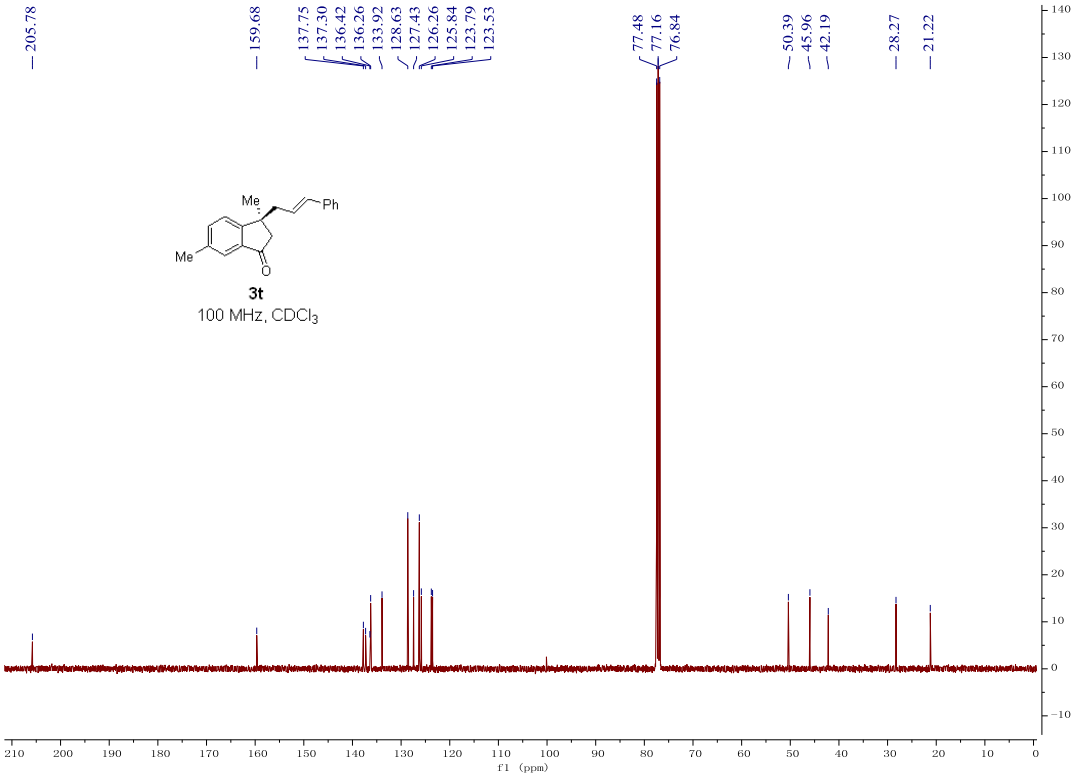
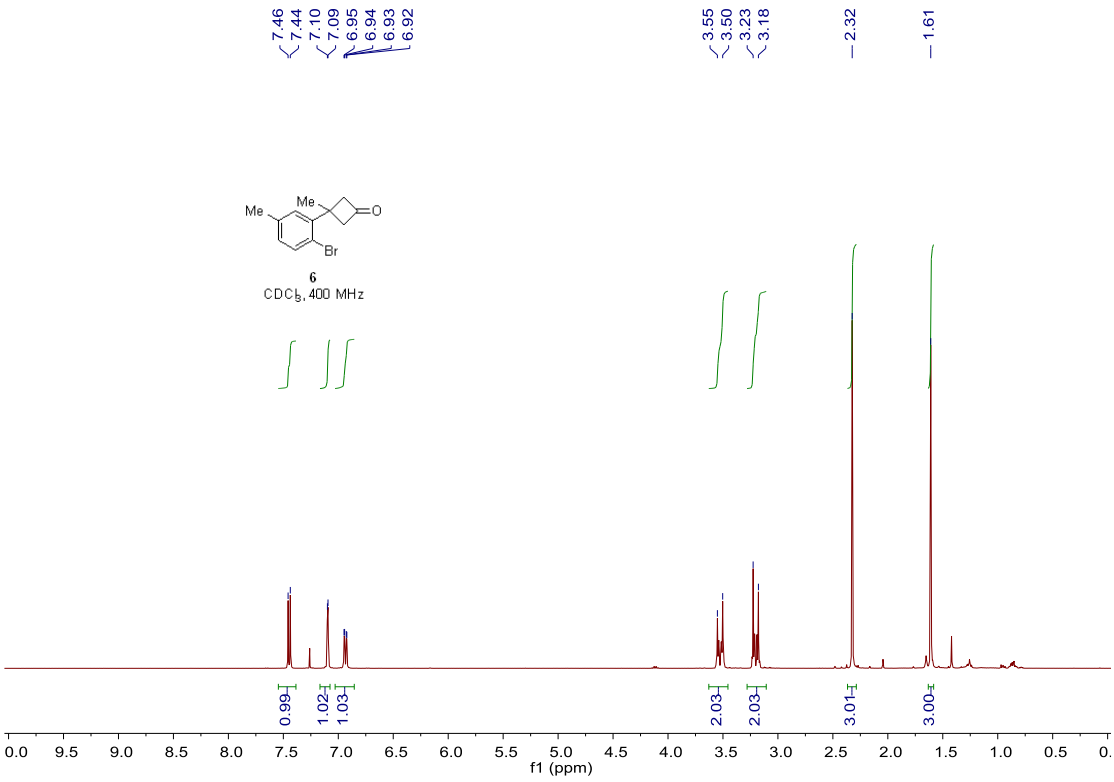
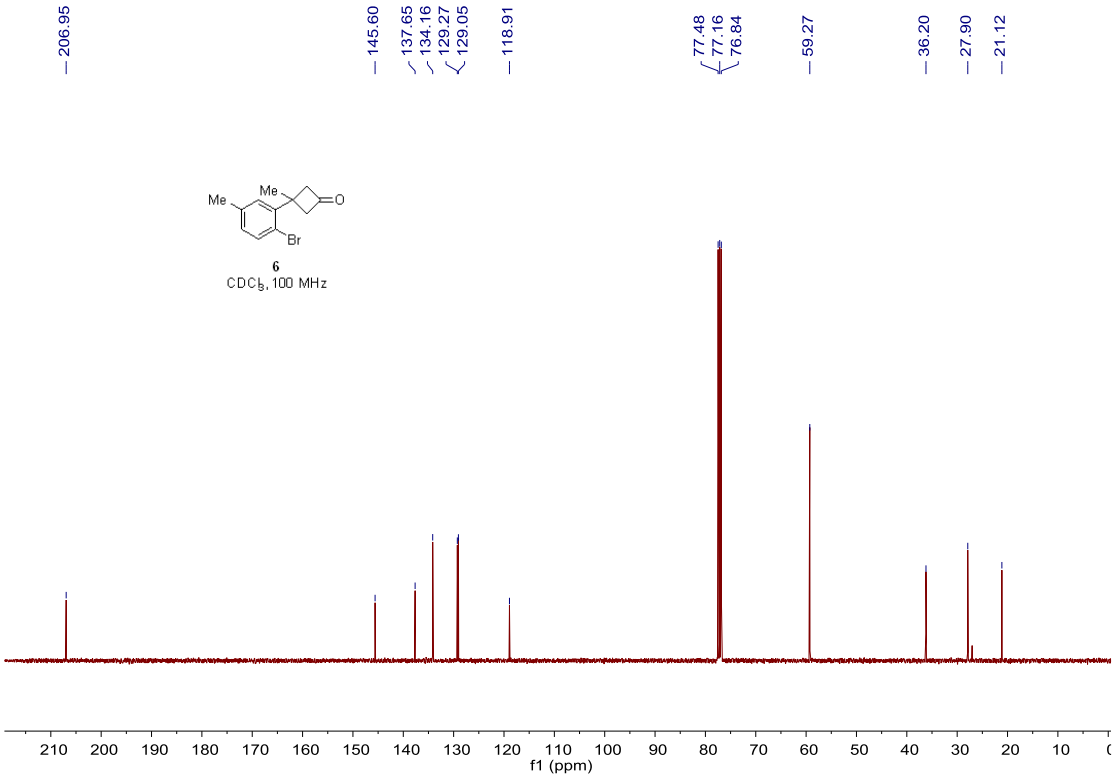
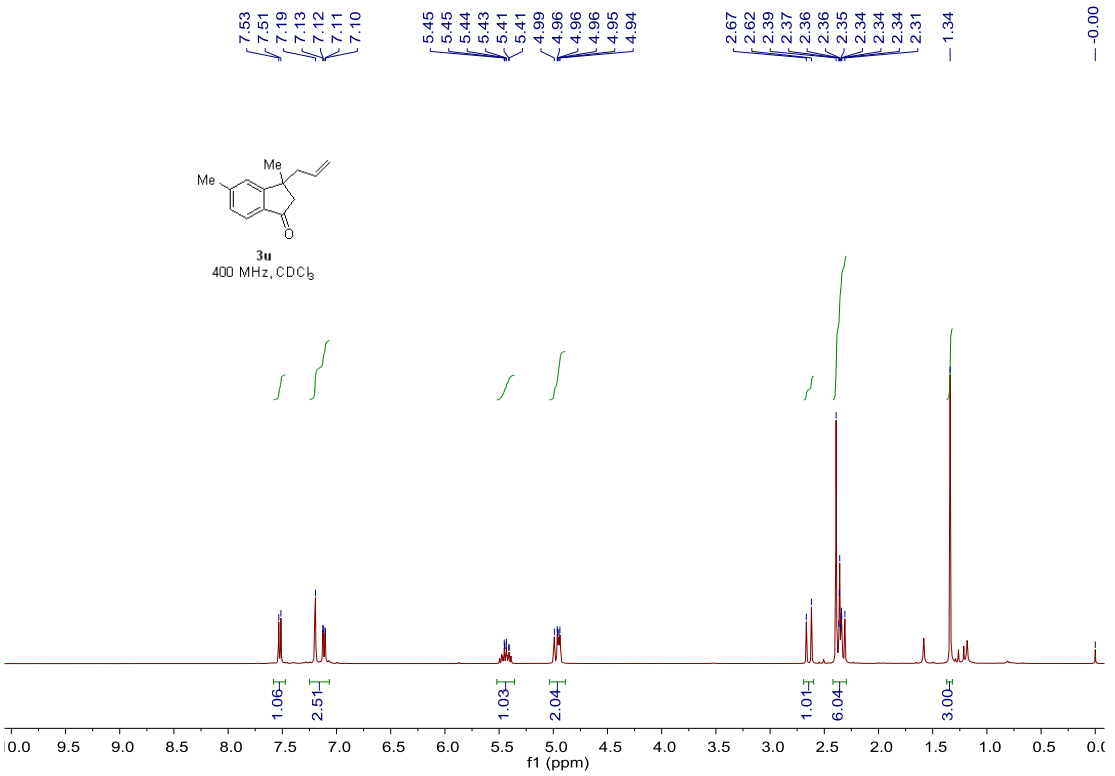
125.78
121.48
116.14
115.90
110.96
110.74
77.48
77.16
76.84
70.63
50.11
44.97
42.34
42.32
29.85
29.79
27.81
Me
F
OH
O
3s
100 MHz, CDCl3
f1 (ppm)

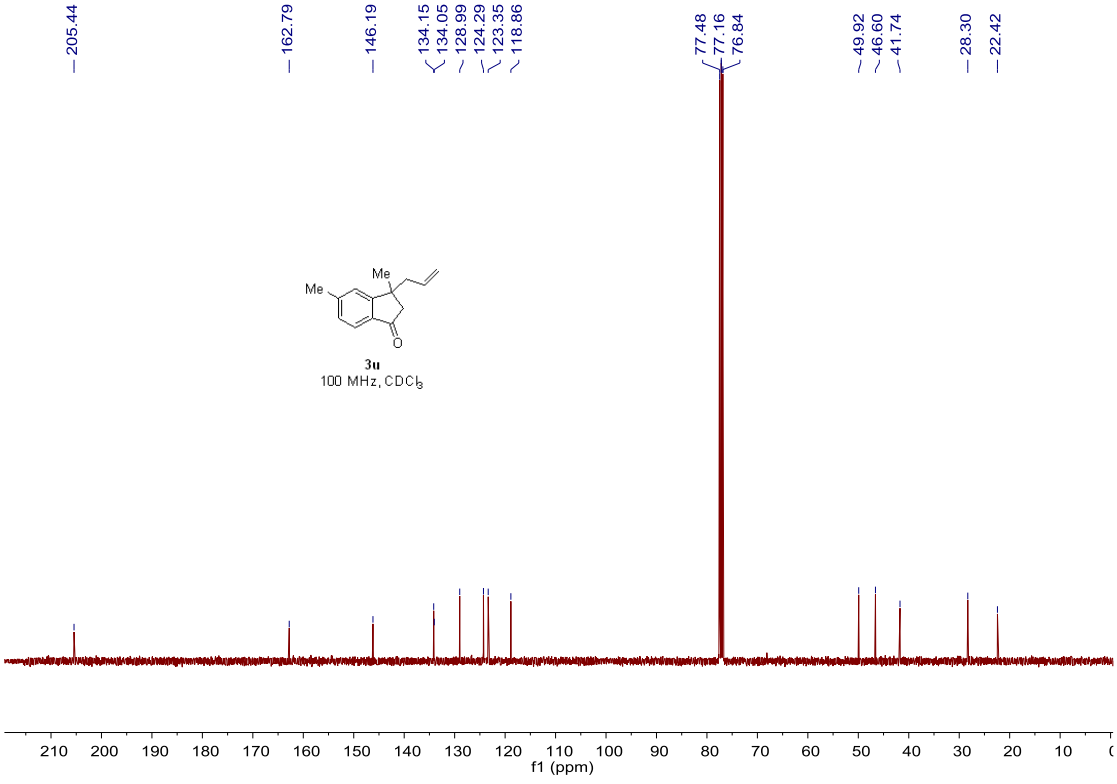

7.42
7.36
7.36
7.33
7.31
7.18
7.17
7.16
7.15
7.10
6.32
6.28
5.90
5.89
5.87
5.85
5.83
2.72
2.67
2.49
2.47
2.47
2.40
2.35
2.33
1.37
Me
Ph
Me
O
3t
400 MHz, CDCl3
1.00
1.08
1.13
4.05
1.10
1.04
1.08
1.06
2.03
1.16
3.09
3.01
f1 (ppm)

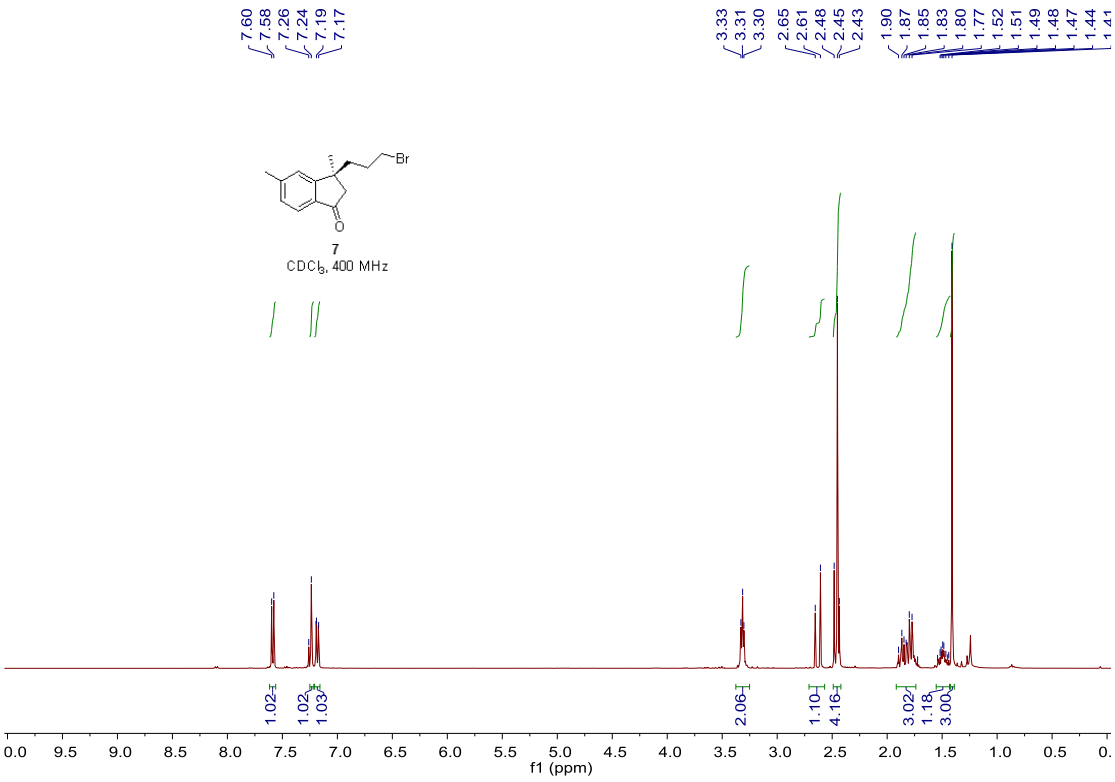

205.78
159.68
137.75
137.30
136.42
136.26
133.92
128.63
127.43
126.26
125.84
123.79
123.53
77.48
77.16
76.84
50.39
45.96
42.19
28.27
21.22
Me
Ph
Me
O
3t
100 MHz, CDCl3
f1 (ppm)

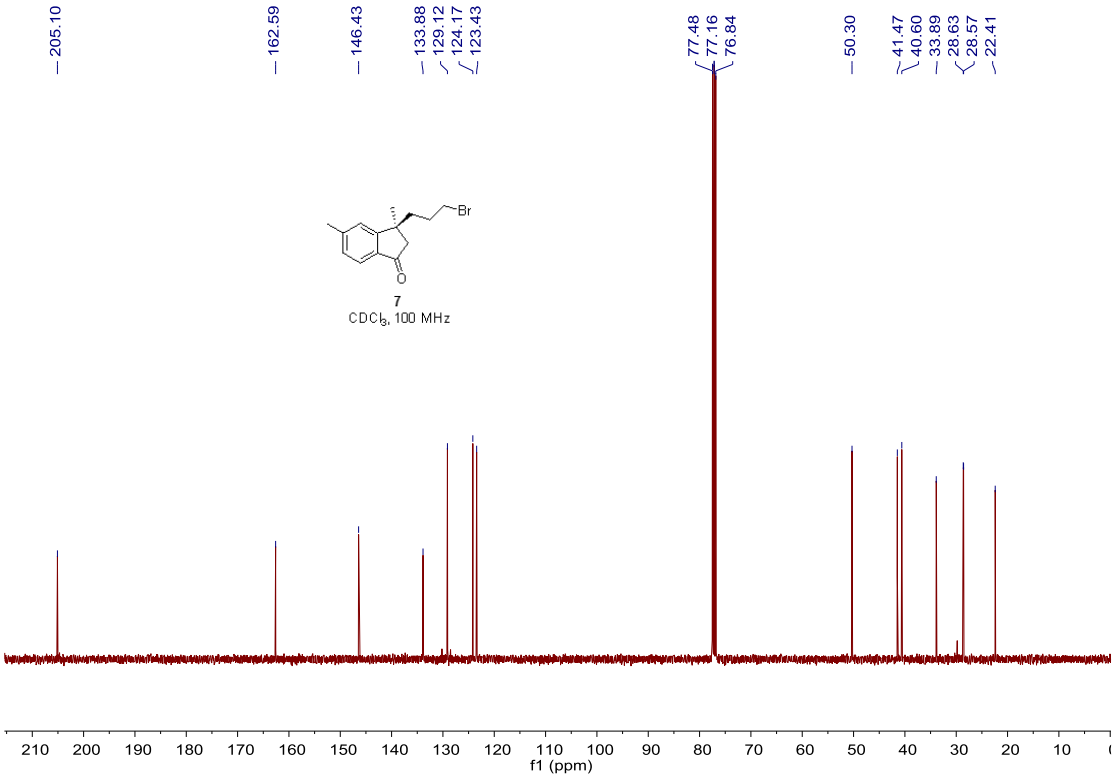

7.46
7.44
7.10
7.09
6.95
6.94
6.93
6.92
3.55
3.50
3.23
3.18
2.32
1.61
Me
Me
O
Br
6
CDCl3, 400 MHz
0.99
1.02
1.03
2.03
2.03
3.01
3.00
f1 (ppm)

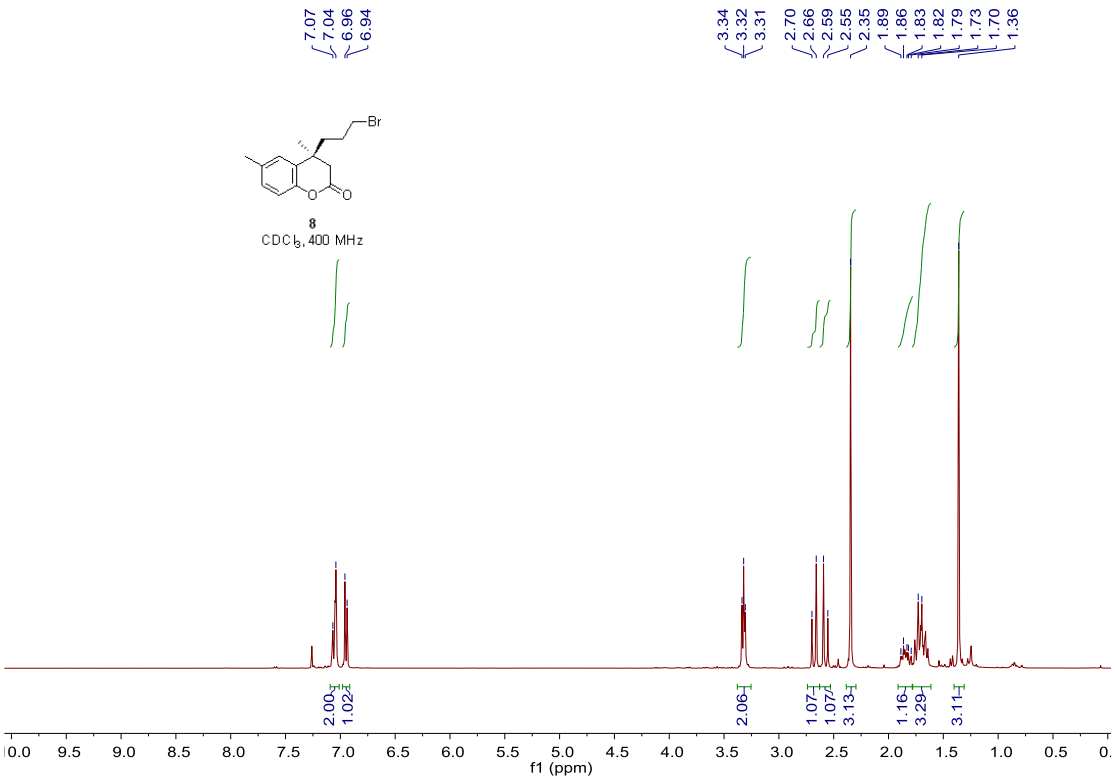

206.95
145.60
137.65
134.16
129.27
129.05
118.91
77.48
77.16
76.84
59.27
36.20
27.90
21.12
Me
Me
O
Br
6
CDCl3, 100 MHz
f1 (ppm)

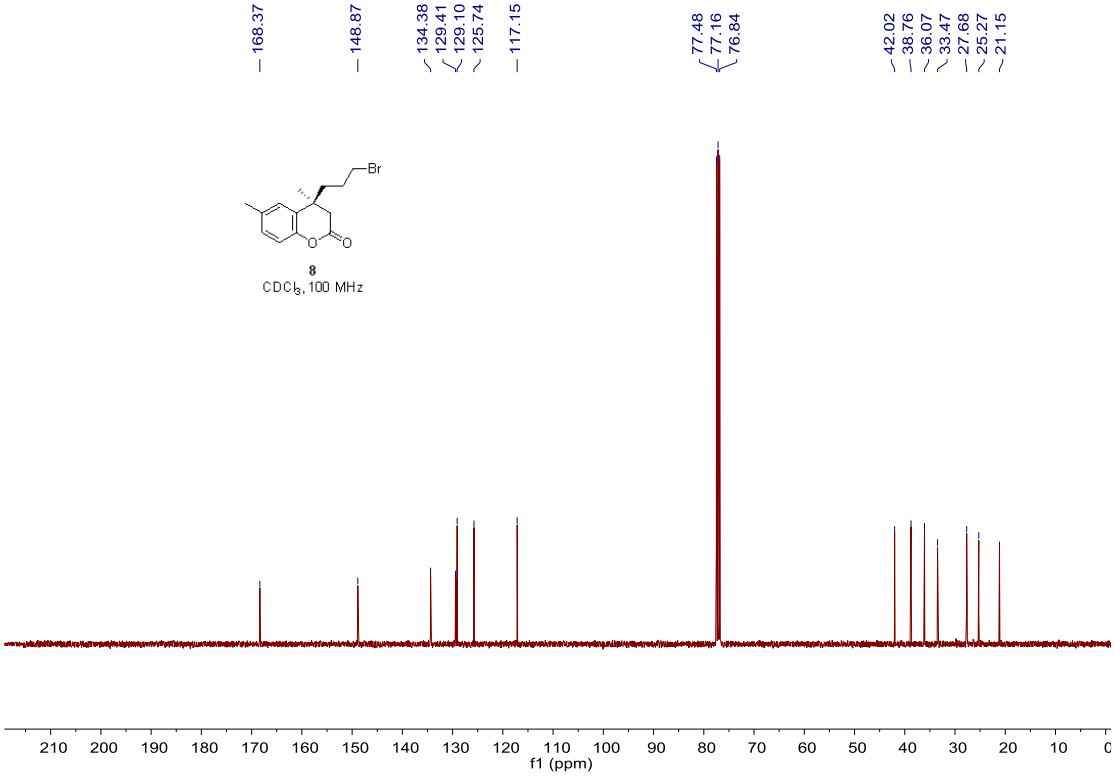

7.53
7.51
7.19
7.13
7.12
7.11
7.10
5.45
5.45
5.44
5.43
5.41
5.41
4.99
4.96
4.96
4.96
4.95
4.94
2.67
2.62
2.39
2.37
2.36
2.36
2.35
2.34
2.34
2.34
2.31
1.34
0.00
Me
Me
O
3u
400 MHz, CDCl3
1.06
2.51
1.03
2.04
1.01
6.04
3.00
f1 (ppm)

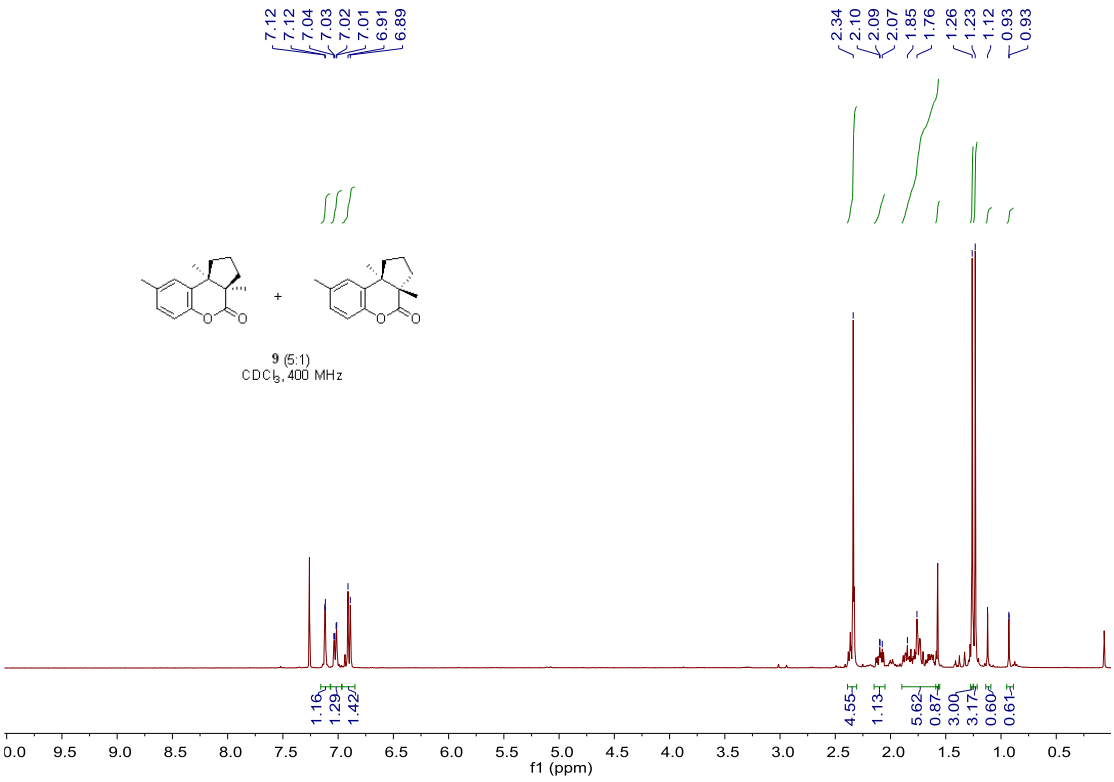

205.44
162.79
146.19
134.15
134.05
128.99
124.29
123.35
118.86
77.48
77.16
76.84
49.92
46.60
41.74
28.30
22.42
Me
Me
O
3u
100 MHz, CDCl3
f1 (ppm)

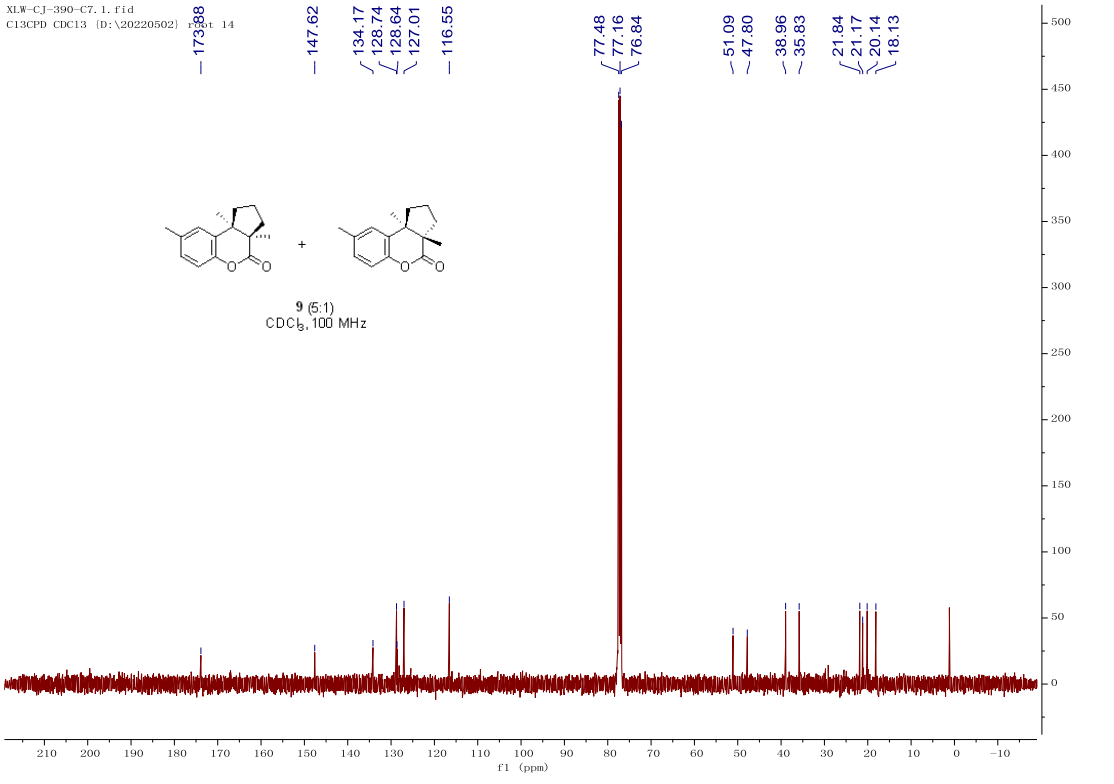

7.60
7.58
7.26
7.24
7.19
7.17
3.33
3.31
3.30
2.65
2.61
2.48
2.45
2.43
1.90
1.87
1.85
1.83
1.80
1.77
1.52
1.51
1.49
1.48
1.47
1.44
1.41
Br
O
7
CDCl3, 400 MHz
1.02
1.02
1.03
2.06
1.10
4.16
3.02
1.18
3.00
f1 (ppm)

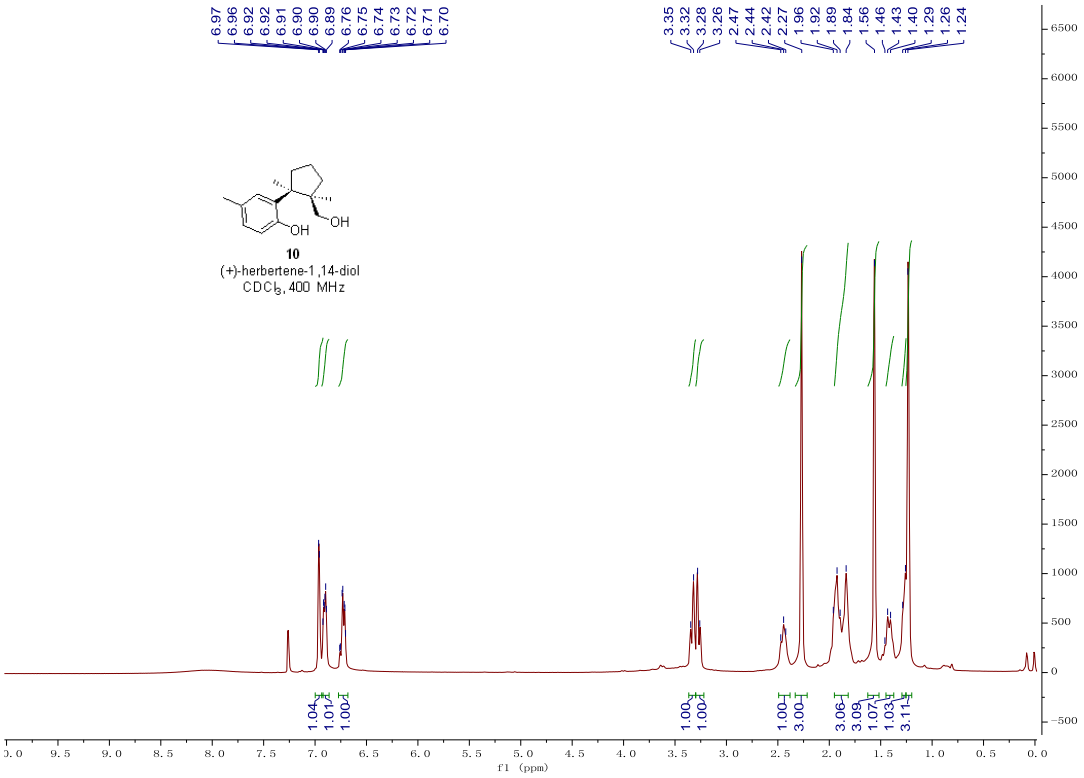

205.10
162.59
146.43
133.88
129.12
124.17
123.43
77.48
77.16
76.84
50.30
41.47
40.60
33.89
28.63
28.57
22.41
Br
O
7
CDCl3, 100 MHz
f1 (ppm)

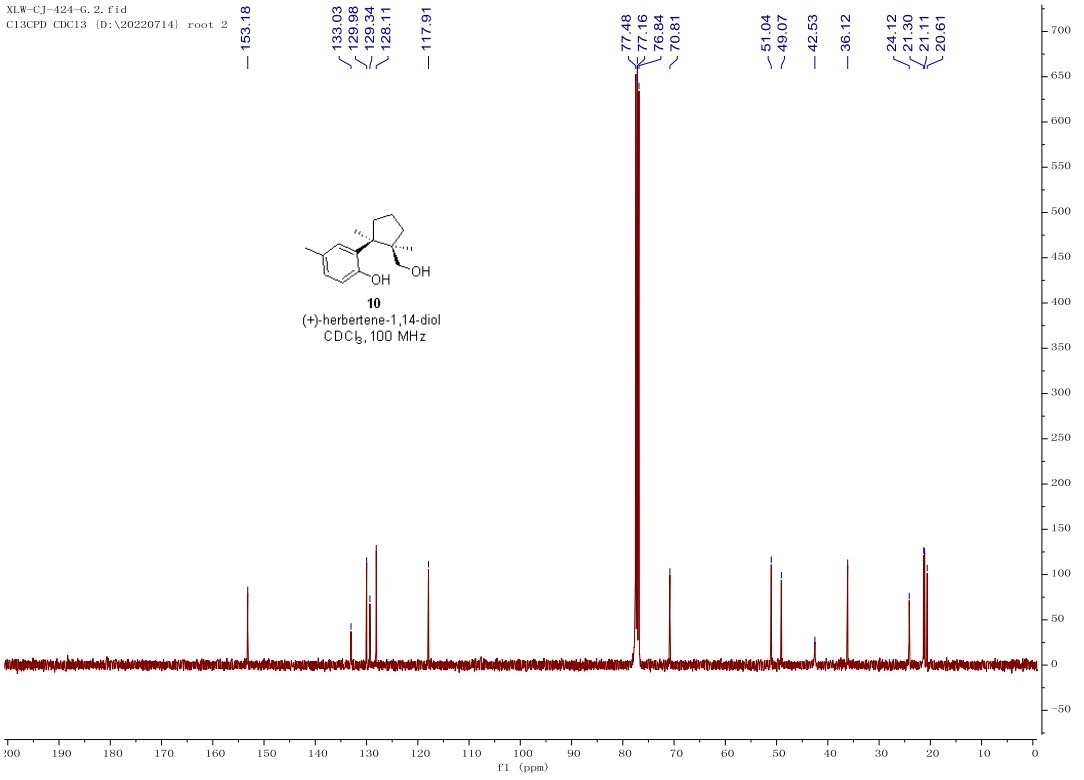

7.07
7.04
6.96
6.94
3.34
3.32
3.31
2.70
2.66
2.59
2.55
2.35
1.89
1.86
1.83
1.82
1.79
1.73
1.70
1.36
Br
O
O
8
CDCl3, 400 MHz
2.00
1.02
2.06
1.07
1.07
3.13
1.16
3.29
3.11
f1 (ppm)


168.37
148.87
134.38
129.41
129.10
125.74
117.15
77.48
77.16
76.84
42.02
38.76
36.07
33.47
27.68
25.27
21.15
Br
O
O
8
CDCl3, 100 MHz
f1 (ppm)


7.12
7.12
7.04
7.03
7.02
7.01
6.91
6.89
2.34
2.10
2.09
2.07
1.85
1.76
1.26
1.23
1.12
0.93
0.93
9 (5:1)
CDCl3, 400 MHz
1.16
1.29
1.42
4.55
1.13
5.62
0.87
3.00
3.17
0.60
0.61
f1 (ppm)


XLW-CJ-390-C7.1.fid
C13CPD CDC13 (D:\20220502)
173.88
147.62
134.17
128.74
128.64
127.01
116.55
77.48
77.16
76.84
51.09
47.80
38.96
35.83
21.84
21.17
20.14
18.13
9 (5:1)
CDCl3, 100 MHz
f1 (ppm)


6.97
6.96
6.92
6.92
6.91
6.90
6.90
6.89
6.76
6.75
6.74
6.73
6.72
6.71
6.70
3.35
3.32
3.28
3.26
2.47
2.44
2.42
2.27
1.96
1.92
1.89
1.84
1.56
1.46
1.43
1.40
1.29
1.26
1.24
OH
OH
10
(+)-herbertene-1,14-diol
CDCl3, 400 MHz
1.04
1.01
1.00
1.00
1.00
1.00
3.00
3.06
3.09
1.07
1.03
3.11
f1 (ppm)


XLW-CJ-424-G.2.fid
C13CPD CDC13 (D:\20220714) root 2
153.18
133.03
129.98
129.34
128.11
117.91
77.48
77.16
76.84
70.81
51.04
49.07
42.53
36.12
24.12
21.30
21.11
20.61
OH
OH
10
(+)-herbertene-1,14-diol
CDCl3, 100 MHz
f1 (ppm)