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Rh-catalyzed Cascade C–H Activation/C–C Cleavage/Cyclization of Carboxylic Acids with Cyclopropanols

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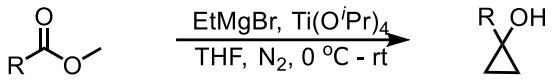
Contents:

General Remark

All chemicals were obtained from commercial suppliers and were used as received unless otherwise noted. ^{1}H and ^{13}C NMR spectra were recorded using $CDCl_3$ as a solvent on a Bruker 500 or 600 MHz NMR spectrometer. The chemical shift is given in dimensionless δ values and is referenced relative to TMS in ^{1}H and ^{13}C NMR spectroscopy. High resolution mass spectra were obtained on a Waters Xevo G2-XS QTOF or an Agilent 6230 LC-TOF MS spectrometer. The compounds were analyzed using a Waters C18 column (ACQUITY UPLC BEH C18, 1.7 m, 2.1 x 50 mm) and eluted with 70% methanol containing 0.1% formic acid. Mass spectra of small molecule were recorded in the mass range of 200-3000 or 600-2000 under high resolution mass-spec mode (HRMS, standard 3200 m/z, 4 GHz). Key source parameters: drying nitrogen gas flow of 11 L/min; nebulizer pressure of 40 psi; gas temperature of 35 ^{o}C; fragmenter voltage of 175 V; skimmer voltage of 65 V; and capillary voltage of 4000 V. Column chromatography was performed on silica gel (200-300 mesh) with freshly distilled ethyl acetate (EA) and petroleum ether (PE).

General procedure to the preparation of cyclopropanols

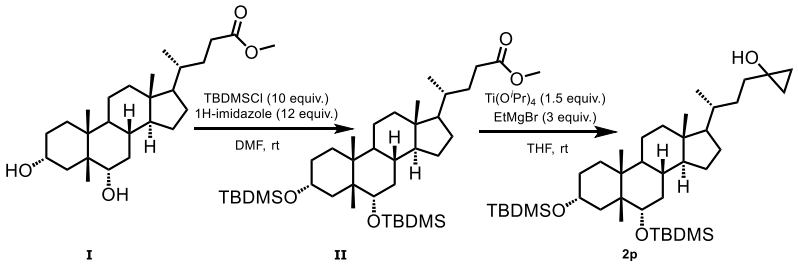


2a-o

Cyclopropanols were synthesized according to literature procedure[1]. Under the protection of nitrogen, EtMgBr (2.8 equiv., 1 M in THF, 28 mL) was slowly added to a solution of the ester (10.0 mmol) and Ti(O^{i}Pr)$_4$ (14.0 mmol, 4.3 mL) in 6 mL of anhydrous THF at 0 ^{o}C over 30 min. The dark mixture was warmed to room temperature and allowed to stir overnight. Then 5 mL of water was slowly added to quench the reaction. After the precipitate was removed by filtration, the filtrate was extracted by Et_2O (20 mL × 3) and the combined organic phase was dried over anhydrous Na_2SO_4. The crude products were purified by column chromatography (PE/EA = 1/1 to 20/1) to afford the pure cyclopropanols.

Synthesis of several starting materials

Preparation of 2p



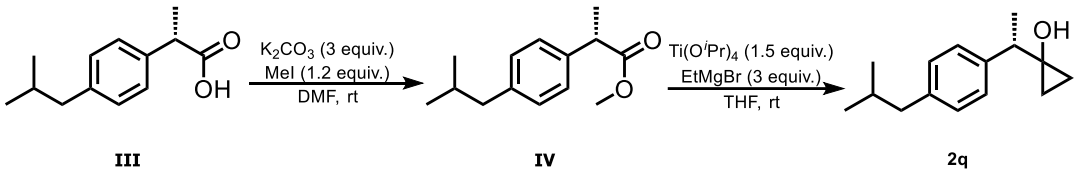
I II 2p

Preparation of **II**: 1H-imidazole (2.5 g, 36 mmol) and *t*-butyldimethylsilyl chloride (TBDMSCl) (4.5 g, 30 mmol) were added to a solution of methyl hyodeoxycholate (1.2 g, 3 mmol) in 3mL of anhydrous DMF. The mixture was allowed to stir overnight at room temperature. Aqueous NaCl was added and the mixture was extracted with ethyl acetate (5 mL × 3). Combined organic phase was dried over anhydrous Na_2SO_4. The

crude products were purified by column chromatography (PE/EA = 80/1) to afford the pure product **II** (1.22 g, 63%).

Preparation of **2p**: Under the protection of nitrogen, EtMgBr (9 mmol, 1M in THF, 9mL) was dropwise added to a solution of **II** (2 mmol, 1.2 g) and $Ti(O^iPr)_4$ (3 mmol, 1.5 mL) in 40 mL of anhydrous THF at 0 °C over 30 min. The dark mixture was warmed to room temperature and allowed to stir overnight. Then 10 mL of water was slowly added to quench the reaction. After the precipitate was removed by filtration, the filtrate was extracted by Et_2O (20 mL × 3) and the combined organic phase was dried over anhydrous Na_2SO_4. The crude products were purified by column chromatography (PE/EA = 20/1) to afford the product **2p** (0.816 g, 67%).

Preparation of 2q

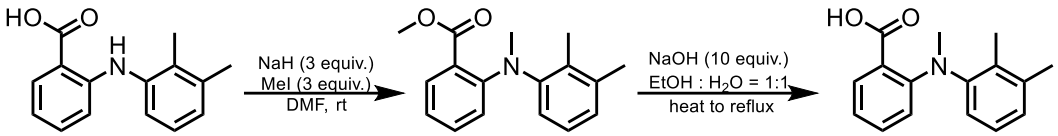


III IV 2q

Preparation of **IV**: MeI (1.2 equiv.) was added to a solution of *S*(+)-Ibuprofen (1.03 g, 5 mmol) and K_2CO_3 (3 equiv.) in DMF (10 mL) at room temperature. The mixture was allowed to stir overnight at room temperature. After pouring 10 mL of water, the mixture was extracted by EtOAc (10 mL × 3). The combined organic layer was washed by brine and dried over anhydrous Na_2SO_4. The residue was purified by silica gel chromatography using PE/EA (10/1) to obtain pure compound **IV** (95%, 1.05 g).

Preparation of **2q**: According to the general procedure to the preparation of cyclopropanes, the pure **2q** was obtained in 90% yield (933 mg).

Preparation of 1s

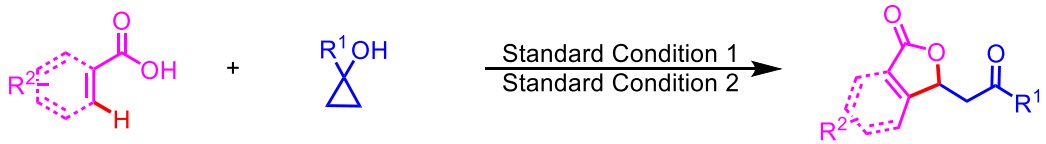


V VI 1s

Preparation of **VI**: NaH (600 mg, 60% in mineral oil, 15 mmol) was added to a solution of mefenamic acid (1.21 g, 5 mmol) in DMF (10 mL) in an ice-water bath. The mixture was stirred for 30 minutes followed by the addition of MeI (933μL, 15 mmol). Then the resulting mixture was stirred at room temperature overnight. After pouring 10 mL of water, the mixture was extracted by EtOAc (10 mL × 3). The combined organic layer was washed by brine and dried over anhydrous Na_2SO_4. The residue was purified by silica gel chromatography using PE/EA (10/1) to obtain pure compound **VI** (1.30 g, 96%).

Preparation of **1s**: The pure compound **VI** was hydrolyzed with sodium hydroxide (10 equiv.) in a solution of 1:1 ethanol/water heated to reflux for 24 h. Then the mixture was cooled down to room temperature and was acidified with 1 M HCl to pH = 1, followed by extracting with EtOAc. The combined organic layer was washed with brine and dried over anhydrous Na_2SO_4. The residue was purified by silica gel chromatography using PE/EA (from 10/1 to 1/1) to obtain pure compound (1.20 g, 98%).

General procedure for C-H activation



1 2 3

Standard Condition 1

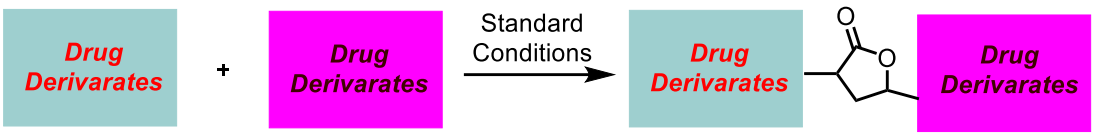
A teflon-capped vial was charged with the benzoic acids (0.1 mmol, 1.0 equiv.), cyclopropanols (0.25 mmol, 2.5 equiv.), Cs_2CO_3 (0.1 mmol, 1 equiv.), $[Cp^*RhCl_2]_2$ (4 mol%), AgOAc (2 equiv.), 4Å Ms (10 mg) under an air atmosphere. The reaction

mixture was stirred at 80 °C for 24 h. After that, the mixture was cooled down and was extracted with EtOAc. The organic layers were combined, filtered through a pad of Celite and concentrated *in vacuo*. The residue was purified by silica gel chromatography using EA/PE to afford compounds.

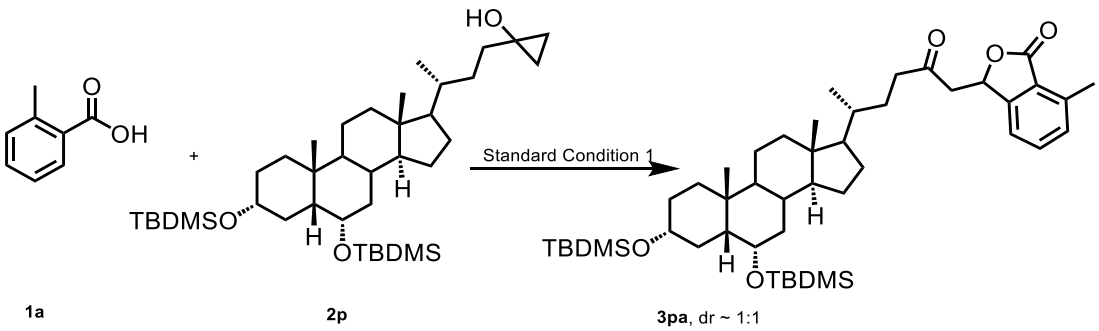
Standard Condition 2

A teflon-capped vial was charged with the benzoic acids (0.1 mmol, 1.0 equiv.), cyclopropanols (0.25 mmol, 2.5 equiv.), K_3PO_4 (0.1 mmol, 1 equiv.), $[Cp^*RhCl_2]_2$ (4 mol%), AgOAc (2 equiv.), 4Å Ms (10 mg) under an air atmosphere. The reaction mixture was stirred at 80 °C for 24 h. After that, the mixture was cooled down and was extracted with EtOAc. The organic layers were combined, filtered through a pad of Celite and concentrated *in vacuo*. The residue was purified by silica gel chromatography using EA/PE to afford compounds.

The potential utility of the reaction



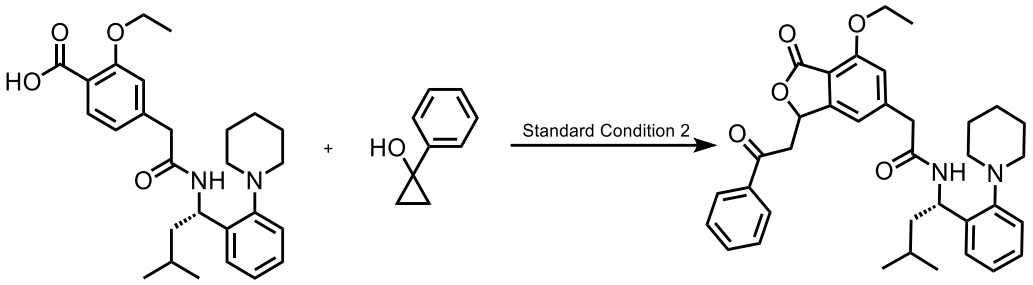
Preparation of 3pa



A teflon-capped vial was charged with the **1a** (13.6 mg, 0.1 mmol, 1.0 equiv.), **2p** (25μL, 0.25 mmol, 2.5 equiv.), Cs_2CO_3 (33mg, 0.1 mmol, 1 equiv.), $[Cp^*RhCl_2]_2$ (2.5

mg, 4 mol%), AgOAc (34 mg, 2 equiv.), 4Å Ms (10 mg) under an air atmosphere. The reaction mixture was stirred at 80 ^{o}C for 48 h. After that, the mixture was cooled down and was extracted with EtOAc. The organic layers were combined, filtered through a pad of Celite and concentrated *in vacuo*. The residue was purified by silica gel chromatography using EA/PE to afford pure compound **3pa** (30 mg) in 40% isolated yield and the value of dr is 1:1.

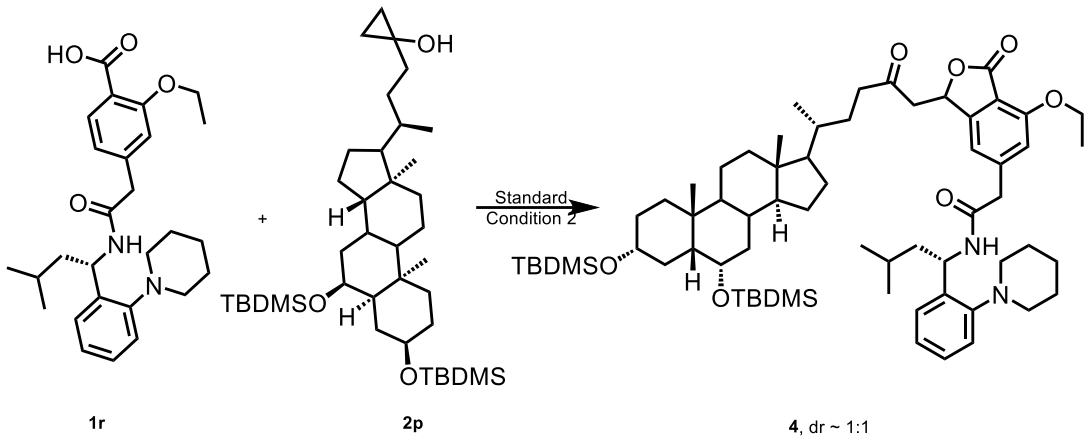
Preparation of 3ar



1r **2a** **3ar**, dr ~ 1:1

A teflon-capped vial was charged with the **1r** (45.2 mg, 0.1 mmol, 1.0 equiv.), **2a** (25 μL, 0.25 mmol, 2.5 equiv.), K_3PO_4 (21.2 mg, 0.1 mmol, 1 equiv.), $[Cp^*RhCl_2]_2$ (2.5 mg, 4 mol%), AgOAc (34 mg, 2 equiv.), 4Å Ms (10 mg) under an air atmosphere. The reaction mixture was stirred at 80 ^{o}C for 24 h. After that, the mixture was cooled down and was extracted with EtOAc. The organic layers were combined, filtered through a pad of Celite and concentrated *in vacuo*. The residue was purified by silica gel chromatography using EA/PE to afford pure compound **3ar** (23 mg) in 40% isolated yield and the value of dr is 1:1.

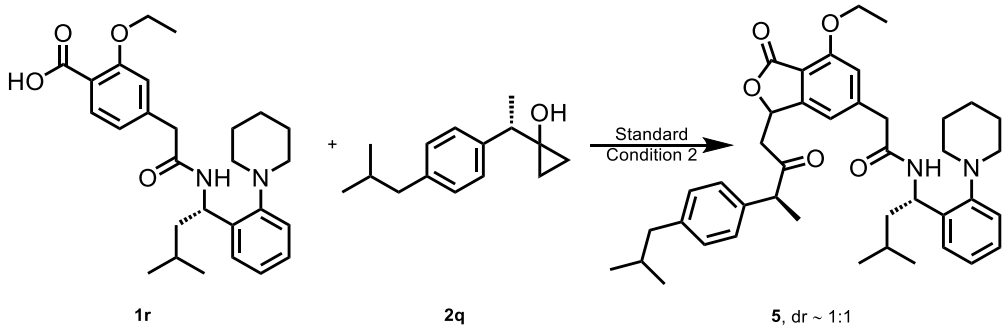
Preparation of 4



1r **2p** **4**, dr ~ 1:1

A teflon-capped vial was charged with the **1r** (45.2 mg, 0.1 mmol, 1.0 equiv.), **2q** (158 mg, 0.25 mmol, 2.5 equiv.), K_3PO_4 (21.2 mg, 0.1 mmol, 1 equiv.), $[Cp^*RhCl_2]_2$ (2.5 mg, 4 mol%), AgOAc (34 mg, 2 equiv.), 4Å Ms (10 mg) under an air atmosphere. The reaction mixture was stirred at 80 ^oC for 24 h. After that, the mixture was cooled down and was extracted with EtOAc. The organic layers were combined, filtered through a pad of Celite and concentrated *in vacuo*. The residue was purified by silica gel chromatography using EA/PE to afford pure compound **4** (40 mg) in 37% isolated yield and the value of dr is 1:1.

Preparation of 5

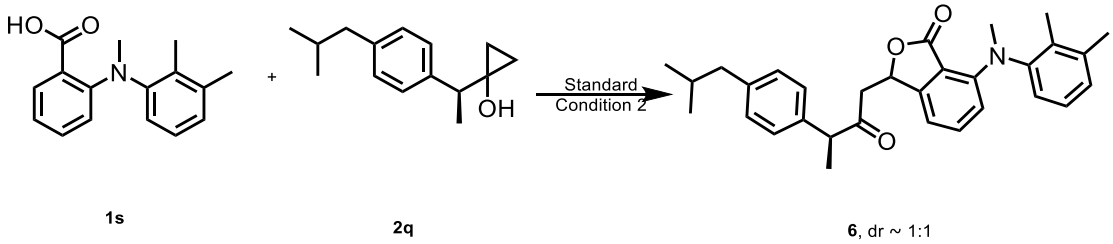


1r **2q** **5**, dr ~ 1:1

A teflon-capped vial was charged with the **1r** (45.2 mg, 0.1 mmol, 1.0 equiv.), **2q** (54.6 mg, 0.25 mmol, 2.5 equiv.), K_3PO_4 (21.2 mg, 0.1 mmol, 1 equiv.), $[Cp^*RhCl_2]_2$ (2.5 mg, 4 mol%), AgOAc (34 mg, 2 equiv.), 4Å Ms (10 mg) under an air atmosphere. The reaction mixture was stirred at 80 ^oC for 24 h. After that, the mixture was cooled down and was extracted with EtOAc. The organic layers were combined, filtered

through a pad of Celite and concentrated *in vacuo*. The residue was purified by silica gel chromatography using EA/PE to afford pure compound **5** (51 mg) in 77% isolated yield and 1:1 dr value.

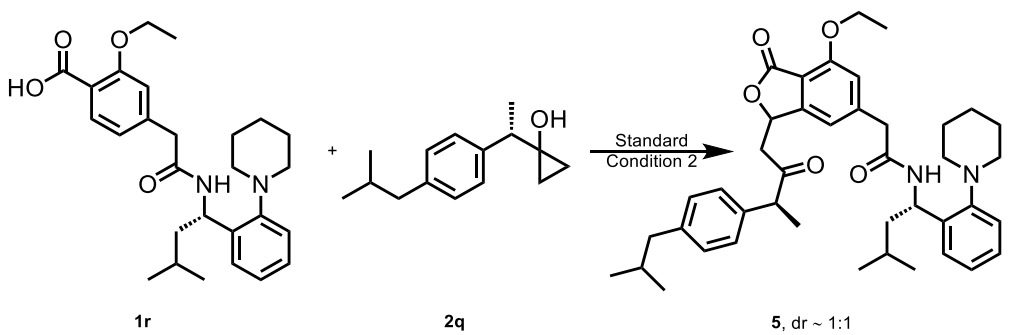
Preparation of 6



1s **2q** **6**, dr ~ 1:1

A teflon-capped vial was charged with the **1s** (25.5 mg, 0.1 mmol, 1.0 equiv.), **2q** (54.6 mg, 0.25 mmol, 2.5 equiv.), K_3PO_4 (21.2 mg, 0.1 mmol, 1 equiv.), $[Cp^*RhCl_2]_2$ (2.5 mg, 4 mol%), AgOAc (34 mg, 2 equiv.), 4Å Ms (10 mg) under an air atmosphere. The reaction mixture was stirred at 80 ^oC for 24 h. After that, the mixture was cooled down and was extracted with EtOAc. The organic layers were combined, filtered through a pad of Celite and concentrated *in vacuo*. The residue was purified by silica gel chromatography using EA/PE to afford pure compound **6** (42 mg) in 90% isolated yield and the value of dr is 1:1.

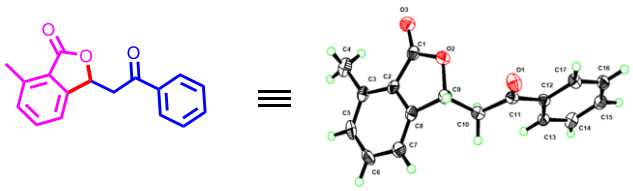
Reaction in gram scale of 5



1r **2q** **5**, dr ~ 1:1

To the solution of the **1r** (2.26 g, 5 mmol, 1 equiv.), **2q** (2.73 g, 12.5 mmol, 2.5 equiv.), $[Cp^*RhCl_2]_2$ (125 mg, 4 mol %), AgOAc (1.7 g, 2 equiv.), K_3PO_4 (1.06 g, 1 equiv.) was added in MeCN (50 mL). The solution was stirred at 80 ºC for 24 hours. After that, the mixture was cooled down and the mixture was extracted with EtOAc. The organic layers were combined, filtered through a pad of Celite and concentrated *in vacuo*. The residue was purified by silica gel chromatography using EA/PE to afford compound **5** (2.43 g) in 73% isolated yield and the value of dr is 1:1.

X-ray molecular structure and crystallographic data



3aa CCDC 2058379

Table 2. Crystal data for 3aa	
Empirical formula	$C_{17}H_{14}O_3$
Formula weight	266.28
Temperature/K	150.0
Crystal system	monoclinic
Space group	$P2_1/c$(no. 14)
a/Å	16.843(11)
b/Å	5.480(2)
c/Å	15.129(6)
β/°	111.15(2)
Volume/Å^3	1302.2(11)
Z	4
ρ_{calcg}/cm^3	1.358
μ/mm^{-1}	0.093
2Θ range for data collection/°	4.524 to 50.052
Independent reflections	6128 [R_{int} = 0.0666, R_{sigma} = 0.0836]
Final R indexes [I>=2σ (I)]	R_1 = 0.0997
Final R indexes [all data]	wR_2 = 0.2709

Table 3. Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3). U_{eq} is defined as 1/3 of of the trace of the

orthogonalised U_{IJ} tensor				
Atom	***x***	***y***	***z***	**U(eq)**
O2	2273(3)	1985(8)	5478(3)	30.3(10)
O1	3599(3)	-2165(7)	5572(3)	32.8(11)
O3	1491(3)	4615(9)	5964(3)	40.7(12)
C2	1514(3)	4779(11)	4358(4)	25.0(13)
C8	1958(3)	3394(10)	6841(4)	24.1(13)
C17	5263(4)	-1528(10)	3610.6(10)	24.5(13)
C9	2482(4)	1539(11)	4634(4)	25.8(13)
C1	1728(4)	3902(11)	5335(4)	27.1(13)
C11	3930(4)	-194(10)	5552(4)	21.0(12)
C12	4846(3)	226(10)	6159(4)	20.7(12)
C16	6107(4)	-1165(11)	7434(4)	28.7(14)
C3	984(4)	6715(11)	3906(4)	27.1(13)
C7	1898(4)	3819(12)	3015(4)	30.2(14)
C6	1387(4)	5756(12)	2538(4)	32.8(15)
C14	6136(4)	2647(11)	6654(4)	30.7(14)
C13	5289(3)	2324(11)	6074(4)	24.0(13)
C4	502(4)	8227(13)	4353(5)	39.5(17)
C5	944(4)	7194(12)	2974(4)	35.1(16)
C10	3441(4)	1862(10)	4920(4)	23.5(13)
C15	6536(4)	912(11)	7339(4)	29.3(14)

Table 4. Anisotropic Displacement Parameters (Å2×103). The Anisotropic displacement factor exponent takes the form: -2π2[h2a*2U11+2hka*b*U12+…].						
Atom	**U_{11}**	**U_{22}**	**U_{33}**	**U_{23}**	**U_{13}**	**U_{12}**
O2	30(2)	35(2)	26(2)	6.7(19)	10.8(18)	-0.1(19)
O1	34(2)	22(2)	37(2)	5.0(19)	6.2(19)	-2.8(19)
O3	34(3)	62(3)	27(2)	-2(2)	12(2)	0(2)
C2	13(3)	33(3)	27(3)	-3(3)	6(2)	-8(2)
C8	17(3)	25(3)	27(3)	-7(3)	4(2)	-4(2)
C17	31(3)	19(3)	22(3)	-1(2)	6(2)	0(2)
C9	26(3)	25(3)	24(3)	-1(2)	7(2)	-2(2)
C1	20(3)	32(3)	29(3)	-1(3)	7(2)	-4(3)
C11	28(3)	16(3)	20(3)	-4(2)	11(2)	1(2)
C12	25(3)	18(3)	20(3)	1(2)	9(2)	1(2)
C16	32(3)	30(3)	24(3)	1(3)	9(3)	10(3)
C3	19(3)	27(3)	33(3)	-4(3)	5(2)	-2(2)
C7	25(3)	36(4)	27(3)	-6(3)	6(3)	-5(3)
C6	28(3)	43(4)	19(3)	6(3)	0(2)	-5(3)
C14	27(3)	25(3)	39(4)	-4(3)	11(3)	-3(3)

C13	24(3)	25(3)	23(3)	0(2)	9(2)	3(2)
C4	28(3)	39(4)	47(4)	-11(3)	8(3)	3(3)
C5	23(3)	35(4)	34(3)	5(3)	-6(3)	1(3)
C10	29(3)	18(3)	23(3)	-2(2)	10(2)	-3(2)
C15	25(3)	34(3)	28(3)	-4(3)	9(3)	3(3)

Table 5. Bond Lengths

Atom	Atom	Length/Å		Atom	Atom	Length/Å
O2	C9	1.464(7)		C9	C10	1.524(8)
O2	C1	1.360(7)		C11	C12	1.501(8)
O1	C11	1.221(7)		C11	C10	1.514(8)
O3	C1	1.220(7)		C12	C13	1.401(8)
C2	C8	1.373(8)		C16	C15	1.384(9)
C2	C1	1.471(8)		C3	C4	1.483(9)
C2	C3	1.396(8)		C3	C5	1.412(9)
C8	C9	1.500(8)		C7	C6	1.392(9)
C8	C7	1.383(8)		C6	C5	1.403(9)
C17	C12	1.399(8)		C14	C13	1.390(8)
C17	C16	1.395(8)		C14	C15	1.388(9)
O2	C9	1.464(7)		C9	C10	1.524(8)
O2	C1	1.360(7)		C11	C12	1.501(8)

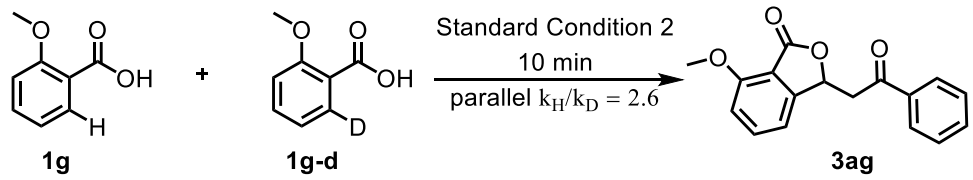
Table 6. Bond Angles

Atom	Atom	Atom	Angle/°		Atom	Atom	Atom	Angle/°
C1	O2	C9	110.64		C12	C11	C10	118.5(5)
C8	C2	C1	107.9(5)		C17	C12	C11	118.5(5)
C8	C2	C3	123.6(5)		C17	C12	C13	119.3(5)
C3	C2	C1	128.5(6)		C13	C12	C11	122.2(5)
C2	C8	C9	109.3(5)		C15	C16	C17	120.0(6)
C2	C8	C7	121.4(5)		C2	C3	C4	124.0(6)
C7	C8	C9	129.3(5)		C2	C3	C5	114.9(5)
C16	C17	C12	120.0(5)		C5	C3	C4	121.0(6)
O2	C9	C8	103.5(5)		C8	C7	C6	117.3(6)
O2	C9	C10	107.3(4)		C7	C6	C5	121.2(6)
C8	C9	C10	114.5(5)		C15	C14	C13	119.6(6)
O2	C1	C2	108.6(5)		C14	C13	C12	120.4(5)
O3	C1	O2	121.0(5)		C6	C5	C3	121.6(6)
O3	C1	C2	130.4(6)		C11	C10	C9	111.9(5)
O1	C11	C12	119.9(5)		C16	C15	C14	120.7(6)
O1	C11	C10	121.6(5)					

Table 7. Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3)				
Atom	***x***	***y***	***z***	**U(eq)**
H17	4970.47	-2965.82	6899.27	29
H9	2310	-150.01	4388.8	31
H16	6387.02	-2344.38	7903.29	34
H7	2192.07	2829.58	2717.86	36
H6	1338.04	6110.01	1905.59	39
H14	6439.77	4046.62	6581.09	37
H13	5008.72	3532.95	5618.56	29
H4A	-4.98	7334.97	4344.93	59
H4B	329.05	9756.45	4000.15	59
H4C	863.52	8588.07	5009.81	59
H5	609.69	8522.21	2633.69	42
H10A	3588.04	1922.62	4343.11	28
H10B	3612.63	3432.82	5258.59	28
H15	7110.22	1154.63	7746.59	35

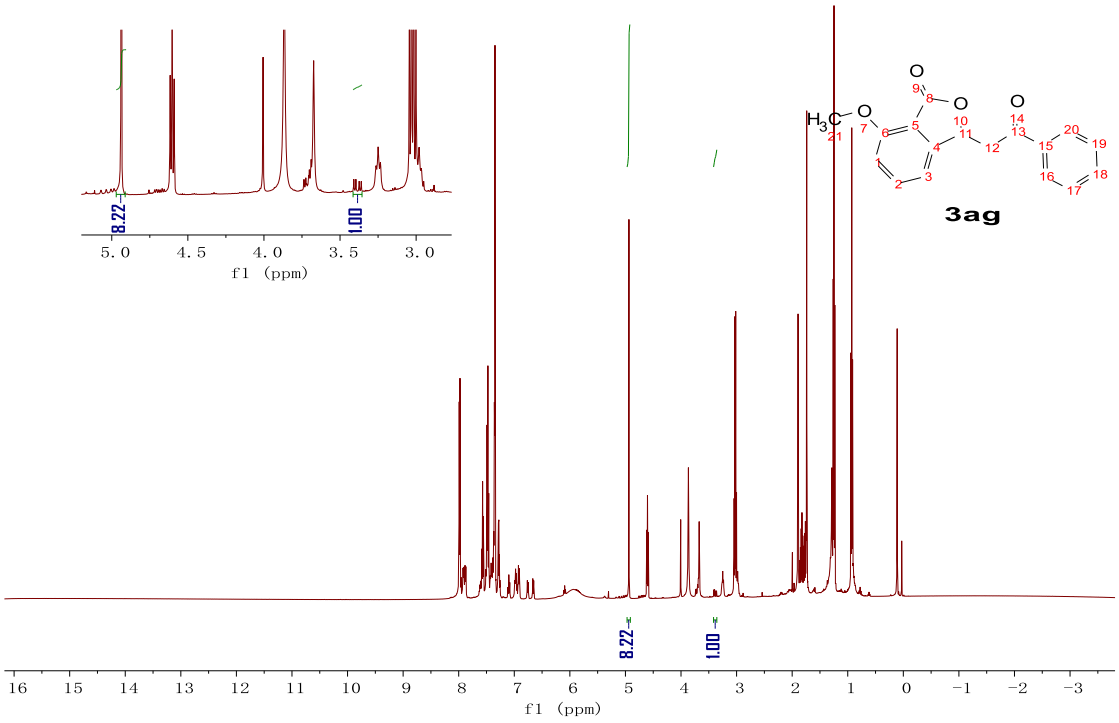
Mechanistic Studies

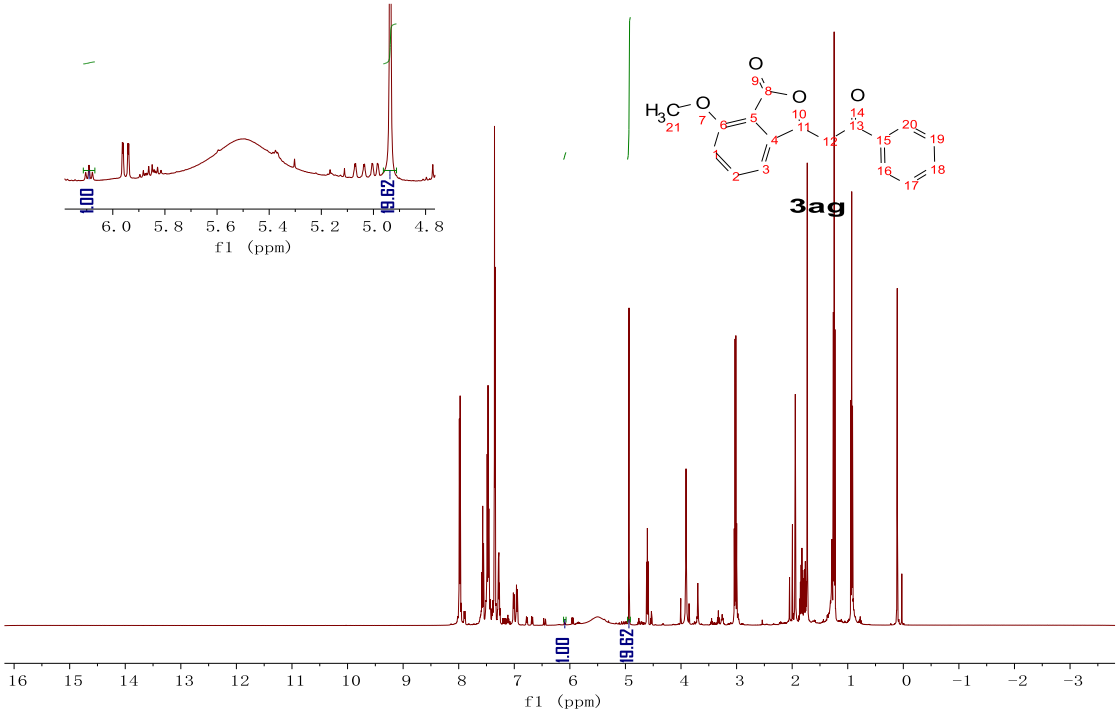
Independent KIE study



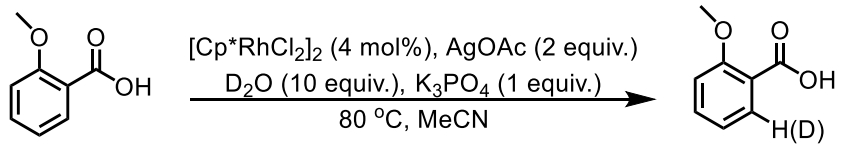
A teflon-capped vial was charged with the 2-methoxyl benzoic acid （**1g**） or mono-deuterated 2-methoxyl benzoic acid (**1g-d**) (0.1 mmol, 1.0 equiv.), **2a** (0.25 mmol, 2.5 equiv.), K_3PO_4 (0.1 mmol, 1equiv.), $[Cp^*RhCl_2]_2$ (4 mol%), AgOAc (2 mol, 2 equiv.) and 4Å Ms (10 mg) in MeCN (1 mL) under an air atmosphere. The reaction mixture was allowed to stir at 80 ºC for 10 min. After that, the mixture was cooled down and was extracted with EtOAc. The organic layers were combined, filtered through a pad of Celite and concentrated *in vacuo*. The crude reaction mixture was diluted with $CDCl_3$, CH_2Br_2 as internal standard was added and the mixture was analyzed via ^1H NMR spectroscopy. The yields of **3ag** were obtained in 13.6 % and 5.2 % respectively

(determined as an average of 2 runs), resulting in an k_H/k_D of 2.6.



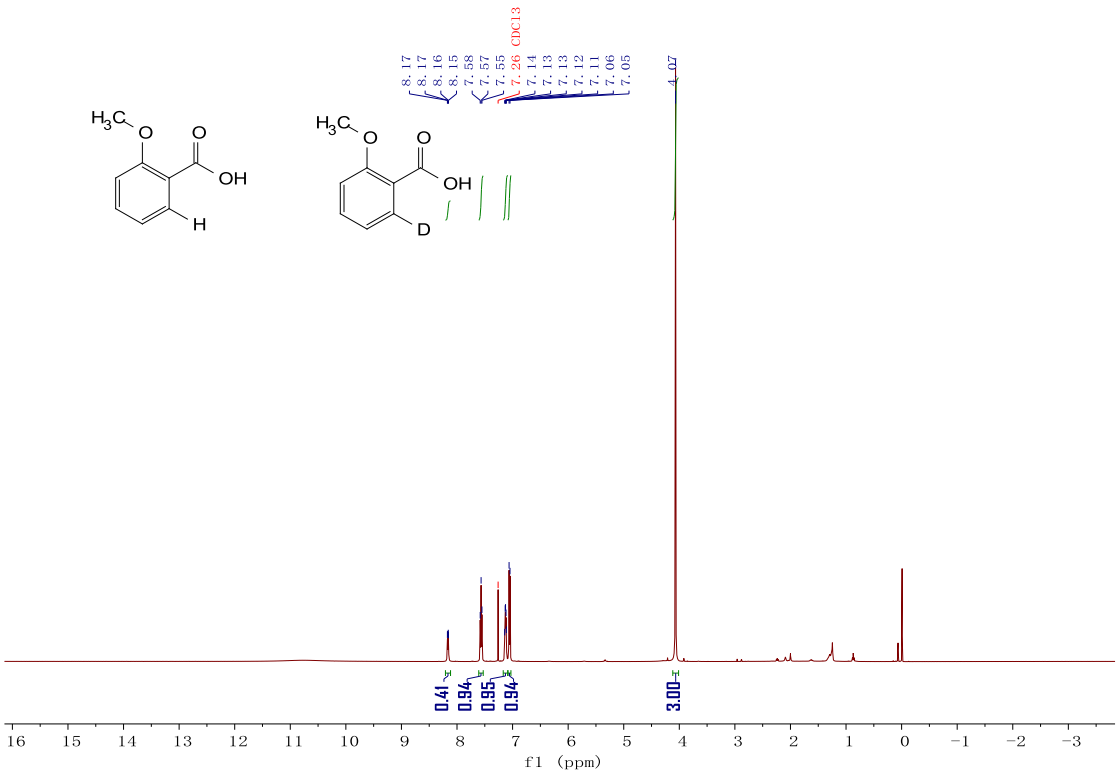


H/D exchange

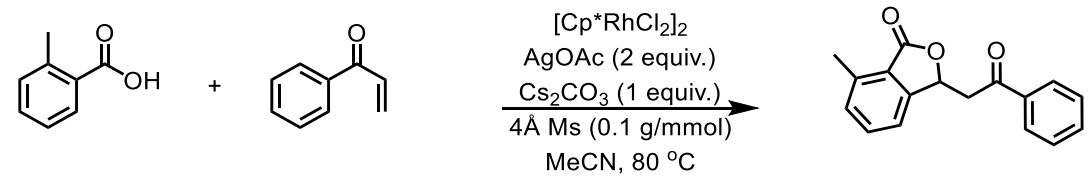


1g **1g-d**: 59%(D)

A teflon-capped vial was charged with 2-methoxyl benzoic acid (**1g**) (0.1 mmol, 1 equiv.), D_2O (1.0 mmol, 10 equiv.), K_3PO_4 (0.1 mmol, 1 equiv.), $[Cp^*RhCl_2]_2$ (4 mol%), AgOAc (0.2 mmol) at 1 mL of MeCN under an air atmosphere. The reaction mixture was allowed to stir at 80 °C for 2 h. The reaction mixture was cooled down to room temperature and acidified with 1M HCl to pH = 1. Then poured into water, the mixture was extracted by EtOAc, followed by dried with anhydrous Na_2SO_4. Then the solvent was removed under reduced pressure and the residue was purified by silica gel chromatography using EA/PE to afford compounds. The ratio was identified by ^{1}HNMR (59% D).



Control experiment

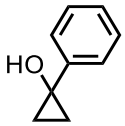


1a, 1 equiv. **7**, 2.5 equiv. **3aa**, 44%

A teflon-capped vial was charged with the benzoic acids (0.1 mmol, 1.0 equiv.), 1-phenylprop-2-enone **7** (0.25 mmol, 2.5 equiv.), Cs_2CO_3 (0.1 mmol, 1 equiv.), $[Cp^*RhCl_2]_2$ (5 mol%), AgOAc (2 equiv.), 4Å Ms (10 mg) under an air atmosphere. The reaction mixture was stirred at 80 °C for 24 h. After that, the mixture was cooled down and the mixture was extracted with EtOAc. The organic layers were combined, filtered through a pad of Celite and concentrated *in vacuo*. The residue was purified by silica gel chromatography using EA/PE to afford pure compound **3aa** in 44% isolated yield.

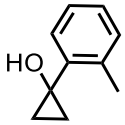
Characterization data of compounds

1- phenylcyclopropan-1-ol (2a)



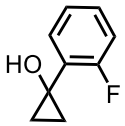
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.37 - 7.17 (m, 5H), 1.30 - 1.23 (m, 2H), 1.07 - 1.03 (m, 2H).

1-(2-methylphenyl)cyclopropan-1-ol (2b)



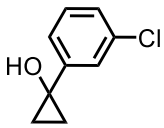
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.33 (d, *J* = 7.1Hz 1H), 7.24 - 7.10 (m, 3H), 2.33 (s, 3H), 1.18 - 1.11 (m, 2H), 0.94 - 0.88 (m, 2H).

1-(2-fluorophenyl)cyclopropan-1-ol (2c)



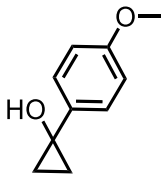
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.38 (td, *J* = 7.6, 1.8 Hz, 1H), 7.29 - 7.25(m, 1H), 7.10 (td, *J* = 7.6, 1.8 Hz, 1H), 7.05 (ddd, *J* = 11.0, 7.6, 1.8Hz, 1H), 1.43 - 1.37 (m, 2H), 1.18-1.11(m, 2H).

1-(3-chlorophenyl)cyclopropan-1-ol (2d)



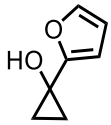
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.29 (t, *J* = 2Hz, 1H), 7.26 - 7.21 (m, 1H), 7.18 (t, *J* = 7.8 Hz, 1H), 7.12 (dt, *J* = 7.8, 2 Hz, 1H), 1.30 - 1.25 (m, 2H), 1.06 - 1.00 (m, 2H).

1-(4-methoxyphenyl)cyclopropan-1-ol (2e)



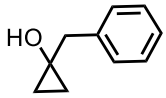
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.36 - 7.16 (m, 2H), 6.96 - 6.81 (m, 2H), 3.8 (s, 3H), 1.23 - 1.14 (m, 2H), 1.01 - 0.92 (m, 2H).

1-(2-furanyl)cyclopropan-1-ol (2f)



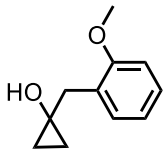
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.32 - 7.29 (m, 1H), 6.32 (dd, *J* = 3.2, 1.8, 1H), 6.21 (d, *J* =3.2, 1H), 1.18 - 1.13 (m, 2H), 1.09 - 1.05 (m, 2H).

1-benzylcyclopropan-1-ol (2g)



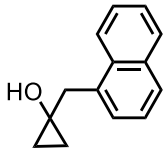
^{1}H NMR (500MHz, $CDCl_3$): δ 7.36 - 7.29 (m, 4H), 7.26 (tt, *J* = 7, 1.5 Hz, 1H), 2.88 (s, 2H), 0.85 - 0.78 (m, 2H), 0.67-0.61 (m, 2H).

1-(2-methoxybenzyl)cyclopropan-1-ol (2h)



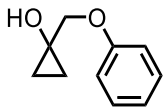
^{1}H NMR (500MHz, $CDCl_3$): δ 7.28 - 7.21 (m, 2H), 6.94 (td, *J* = 7.5, 1 Hz, 1H), 6.91 (d, *J* = 7.5 Hz, 1H), 3.85 (s, 3H), 2.96 (s, 2H), 0.79 - 0.74 (m, 2H), 0.64 - 0.59 (m, 2H).

1-(naphthalenylmethyl)cyclopropan-1-ol (2i)



^{1}H NMR (500 MHz, $CDCl_3$): δ 8.17 (d, *J* = 8.5 1H), 7.90 (dd, *J* = 8.0, 1 Hz, 1H), 7.81 (dt, *J* = 8.0, 1 Hz, 1H), 7.59 - 7.43 (m, 4H), 3.47 (s, 2H), 0.91 - 0.84 (m, 2H), 0.75 - 0.67 (m, 2H).

1-(phenoxymethyl)cyclopropan-1-ol (2j)



^{1}H NMR (500MHz, $CDCl_3$): δ 7.33 - 7.27 (m, 2H), 6.98 (tt, *J* = 7.3, 1 Hz, 1H), 6.96 - 6.91 (m, 2H), 4.01 (s, 2H), 1.12 - 0.84 (m, 2H), 0.86 - 0.56 (m, 2H).

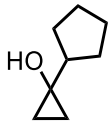
[1,1'-bi(cyclopropan)]-1-ol (2k)

^{1}H NMR (500 MHz, $CDCl_3$): δ 1.37 (tt, *J* = 8, 5 Hz, 1H), 0.74 - 0.67 (m, 2H), 0.54 - 0.48 (m, 2H), 0.45 - 0.41 (m, 2H), 0.23 - 0.18 (m, 2H).

1-cyclobutylcyclopropan-1-ol (2l)

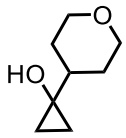
^{1}H NMR (600 MHz, $CDCl_3$): δ 2.5 (q, *J* = 8.4 Hz, 1H), 2.00 - 1.93 (m, 2H), 1.85 - 1.69 (m, 4H), 0.72 - 0.67 (m, 2H), 0.52 - 0.47 (m, 2H).

1-cyclopentylcyclopropan-1-ol (2m)



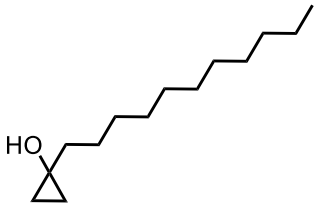
^{1}H NMR (600 MHz, $CDCl_3$): δ 1.92 - 1.82 (m, 1H), 1.77 - 1.69 (m, 2H), 1.68 - 1.60 (m, 2H), 1.58 - 1.50 (m, 2H), 1.41 - 1.30 (m, 2H), 0.72 - 0.66 (m, 2H), 0.49 - 0.45 (m, 2H).

1-(tetrahydropyran-4-yl)cyclopropan-1-ol (2n)



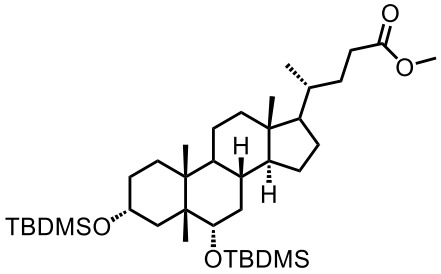
^{1}H NMR (600 MHz, $CDCl_3$): δ 4.03 (dt, *J* = 10.8, 3 Hz, 2H), 3.46 - 3.29 (m, 2H), 1.68 - 1.52 (m, 4H), 1.36 - 1.17 (m, 1H), 0.76 - 0.61 (m, 2H), 0.58 - 0.37 (m, 2H).

1-undecylcyclopropan-1-ol (2o)



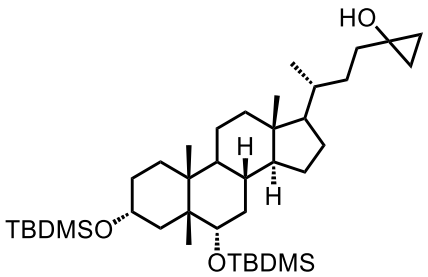
^{1}H NMR (600 MHz, $CDCl_3$): δ 1.59-1.54 (m, 2H), 1.54 - 1.49 (m, 2H), 1.38 - 1.22 (m, 16H), 0.9 (t, J= 6.9 Hz, 3H), 0.77 - 0.71 (m, 2H), 0.48 - 0.42 (m, 2H).

(4*R*)-4-((3*R*,5*R*,6*S*,8*S*,10*R*,13*R*,14*S*)-3,6-bis((tert-butyldimethylsilyl)oxy)-5,10,13-trimethylhexadecahydro-1H-cyclopenta[a]phenanthren-17-yl)pentanoate (II)



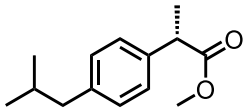
^{1}H NMR (500 MHz, $CDCl_3$): δ 4.00 (dt, *J* = 11.7, 4.3 Hz, 1H), 3.69 (s, 3H), 3.59 - 3.51 (m, 1H), 2.38 (ddd, *J* = 15.5, 10.0, 5.0 Hz, 1H), 2.24 (ddd, *J* = 15.5, 9.5, 6.0 Hz, 1H), 2.00 - 1.72 (m, 5H), 1.65 - 1.54 (m, 2H), 1.52 - 1.26 (m, 11H), 1.25 – 0.97 (m, 8H), 0.95 - 0.85 (m, 24H), 0.66 (s, 3H), 0.09 - 0.04 (m, 12H).

1-((3*R*)-3-((3*R*,5*R*,6*S*,8*S*,10*R*,13*R*,14*S*)-3,6-bis((tert-butyldimethylsilyl)oxy)-5,10,13-trimethylhexadecahydro-1H-cyclopenta[a]phenanthren-17-yl)butyl)cyclopropan-1-ol (2p)



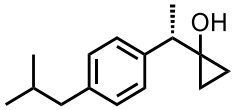
^{1}H NMR (500 MHz, $CDCl_3$): δ 4.20 - 3.86 (m, 1H), 3.57 - 3.49 (m, 1H), 1.97 - 1.91 (m, 1H), 1.91 -1.79 (m, 2H), 1.78 - 1.69 (m, 1H), 1.67 - 1.62 (m, 2H), 1.60 - 1.52 (m, 3H), 1.49 - 1.35 (m, 9H), 1.27 - 1.22 (m, 2H), 1.16 -1.05 (m, 4H), 1.02 – 0.94 (m, 2H), 0.93 - 0.82 (m, 24H), 0.78 - 0.67 (m, 2H), 0.63 (s, 3H), 0.48 - 0.37 (m, 2H), 0.08 - 0.01 (m, 12H).

methyl (*S*)-2-(4-isobutylphenyl)propanoate (IV)



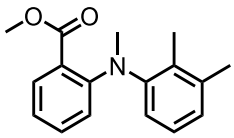
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.23 - 7.17 (m, 2H), 7.13 - 7.08 (m, 2H), 3.70 (d, *J* = 7.5 Hz, 1H), 3.66 (s, 3H), 2.45 (d, *J* = 7.0 Hz, 2H), 1.90 - 1.80 (m, 1H), 1.49 (d, *J* = 7.5 Hz, 3H), 0.90 (d, *J* = 7.0 Hz, 6H).

(*S*)-1-(1-(4-isobutylphenyl)ethyl)cyclopropan-1-ol (2q)



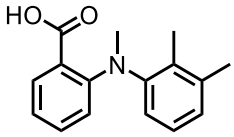
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.25 - 7.21 (m, 2H), 7.13 - 7.10 (m, 2H), 2.54 (q, *J* = 7.2 Hz, 1H), 2.46 (d, *J* = 7.5 Hz, 2H), 1.91 - 1.81 (m, 1H), 1.68 (s, 1H), 1.39 (d, *J* = 7.2 Hz, 3H), 0.92 (d, *J* = 6.5 Hz, 6H), 0.86 - 0.80 (m, 1H), 0.75 - 0.65 (m, 2H), 0.64 - 0.58 (m, 1H).

methyl 2-((2,3-dimethylphenyl)(methyl)amino)benzoate (VI)



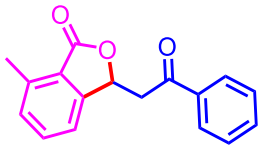
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.43 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.37 - 7.33 (m, 1H), 7.03 (t, *J* = 7.5 Hz, 1H), 6.97 (d, *J* = 7.2 Hz, 1H), 6.95 (d, *J* = 8.4 Hz, 1H), 6.87 (t, *J* = 7.5 Hz, 1H), 6.81 (d, *J* = 7.8 Hz, 1H), 3.41 (s, 3H), 3.16 (s, 3H), 2.29 (s, 3H), 2.18 (s, 3H).

2-((2,3-dimethylphenyl)(methyl)amino)benzoic acid (1s)



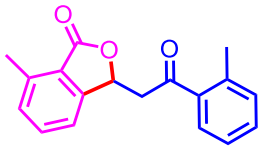
^{1}H NMR (500 MHz, $CDCl_3$): δ 8.29 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.48 - 7.40 (m, 2H), 7.31 (dd, *J* = 7.5, 1.0 Hz, 1H), 7.22 - 7.17 (m, 2H), 7.09 - 7.05 (m, 1H), 6.90 (dd, *J* = 8.0, 1.0 Hz, 1H), 3.18 (s, 3H), 2.24 (s, 3H), 1.89 (s, 3H).

7-methyl-3-(2-oxo-2-phenylethyl)isobenzofuran-1(3*H*)-one (3aa)



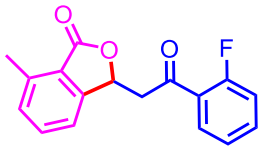
The product was synthesized by standard condition 1 in 75% yield.
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.98 - 7.93 (m, 2H), 7.62 - 7.56 (m, 1H), 7.53 - 7.44 (m, 3H), 7.34 (d, *J* = 8.0 Hz, 1H), 7.28 (d, *J* = 7.5 Hz, 1H), 6.11 (t, *J* = 6.5 Hz, 1H), 3.71 (dd, *J* = 17.5, 6.5 Hz, 1H), 3.38 (dd, *J* = 17.5, 6.5 Hz, 1H), 2.70 (s, 3H).
^{13}C NMR (151 MHz, $CDCl_3$): δ 196.26, 170.47, 150.38, 139.98, 136.39, 134.12, 133.96, 131.17, 128.96, 128.32, 123.48, 120.06, 76.30, 44.01, 17.53.
ESI-MS calcd. for $C_{17}H_{14}O_3Na$ [M+Na^+]: 289.0840, found: 289.0837.

7-methyl-3-(2-oxo-2-(o-tolyl)ethyl)isobenzofuran-1(3*H*)-one (3ba)



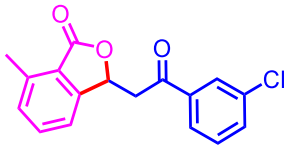
The product was synthesized by standard condition 1 in 75% yield.
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.65 (dd, *J* = 7.5, 1 Hz, 1H), 7.55 (t, *J* = 7.6 Hz, 1H), 7.43 (td, *J* = 7.5, 1 Hz, 1H), 7.36 (d, *J* = 7.5, 1H), 7.33 - 7.30 (m, 2H), 7.28 (dd, *J* = 7.5, 1 Hz, 1H), 6.11 (t, *J* = 6.5 Hz, 1H), 3.66 (dd, *J* = 17, 6.5 Hz, 1H), 3.37 (dd, *J* = 17, 6.5 Hz, 1H), 2.72 (s, 3H), 2.61 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 199.51, 170.43, 150.45, 140.01, 139.07, 136.72, 134.08, 132.42, 132.26, 131.13, 129.08, 126.01, 123.53, 119.83, 76.50, 46.50, 21.74, 17.50.
ESI-MS calcd. for $C_{18}H_{16}O_3Na$ [M+Na^+]: 303.0996, found: 303.0998.

3-(2-(2-fluorophenyl)-2-oxoethyl)-7-methylisobenzofuran-1(3H)-one (3ca)



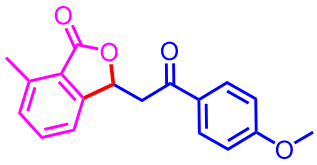
The product was synthesized by standard condition 1 in 61% yield.
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.96 (dt, *J* = 7.5, 2 Hz, 1H), 7.56 (dddd, *J* = 8, 7.5, 5.5, 2 Hz, 1H), 7.52 (t, *J* = 7.5, 1H), 7.33 (d, *J* = 7.5, 1H), 7.30 - 7.23 (m, 2H), 7.14 (ddd, *J* = 8, 6, 1 Hz, 1H), 6.09 (t, *J* = 6.5, 1H), 3.64 (ddd, *J* = 18, 6.5, 3 Hz, 1H), 3.45 (ddd, *J* = 18, 6.5, 3 Hz, 1H), 2.69 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 194.16, 170.45, 163.31, 161.28, 150.25, 139.99, 135.57 (d, *J* = 9.3 Hz), 134.08, 131.13, 130.84 (d, *J* = 1.5 Hz), 124.88, 123.54, 119.85, 116.93 (d, *J* = 23.8 Hz), 75.92, 48.78 (d, *J* = 8.2 Hz), 17.50.
ESI-MS calcd. for $C_{17}H_{13}FO_3Na$ [M+Na^+]: 307.0746, found: 307.0750.
^{19}F NMR (500 MHz, $CDCl_3$): δ -108.87.

3-(2-(3-chlorophenyl)-2-oxoethyl)-7-methylisobenzofuran-1(3H)-one (3da)



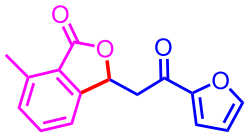
The product was synthesized by standard condition 1 in 63% yield.
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.94 (t, *J* = 1.8 Hz, 1H), 7.83 (d, *J* = 7.8 Hz, 1H), 7.58 (ddd, *J* = 7.8, 1.8, 1.2 Hz, 1H), 7.53 (t, *J* = 7.8, 1H), 7.44 (t, *J* = 7.8 Hz, 1H), 7.33 (d, *J* = 7.8 Hz, 1H), 7.30 (d, *J* = 7.8 Hz, 1H), 6.10 (t, *J* = 6.3 Hz, 1H), 3.68 (dd, *J* = 17.4, 6.3 Hz, 1H), 3.38 (dd, *J* = 17.4, 6.3 Hz, 1H), 2.71 (s, 3H).
^{13}C NMR (151 MHz, $CDCl_3$): δ 195.00, 170.31, 150.09, 140.08, 137.87, 135.36, 134.17, 133.86, 131.26, 130.30, 128.40, 126.42, 123.46, 119.91, 75.99, 44.10, 17.50.
ESI-MS calcd. for $C_{17}H_{13}ClO_3Na$ [M+Na^+]: 323.0450, found: 323.0447.

3-(2-(4-methoxyphenyl)-2-oxoethyl)-7-methylisobenzofuran-1(3*H*)-one (3ea)



The product was synthesized by standard condition 1 in 44% yield.
^{1}H NMR (500 MHz, CDCl3): δ 7.96 - 7.89 (m, 2H), 7.50 (t, *J* = 7.6 Hz, 1H), 7.35 - 7.30 (m, 1H), 7.30 - 7.24 (m, 1H), 6.97-6.91 (m, 2H), 6.09 (t, *J* = 6.5 Hz, 1H), 3.87 (s, 3H), 3.66 (dd, *J* = 17.5, 6.5 Hz, 1H), 3.31 (dd, *J* = 17.5, 6.5 Hz, 1H), 2.69 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 194.68, 170.51, 164.18, 150.55, 139.91, 134.07, 131.09, 130.68, 129.59, 123.50, 120.13, 114.11, 76.56, 55.68, 43.65, 17.50.
ESI-MS calcd. for $C_{18}H_{16}O_4Na$ [M+Na^+]: 319.0946, found: 319.0950.

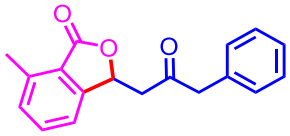
3-(2-(furan-2-yl)-2-oxoethyl)-7-methylisobenzofuran-1(3*H*)-one (3fa)



The product was synthesized by standard condition 1 in 51% yield.
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.61 - 7.59 (m, 1H), 7.51 (t, *J* = 7.5 Hz, 1H), 7.31 (d, *J* = 7.5 Hz, 1H), 7.28 (d, *J* = 7.5 Hz, 1H), 7.25 (d, *J* = 3.6 Hz, 1H), 6.57 (dd, *J* = 3.6, 1.8 Hz, 1H), 6.04 (t, *J* = 6.6 Hz, 1H), 3.53 (dd, *J* = 17.5, 6.6 Hz, 1H), 3.24 (dd, *J* = 17.5, 6.6 Hz, 1H), 2.69 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 184.91, 170.35, 152.40, 150.06, 147.22, 140.03, 134.12, 131.20,

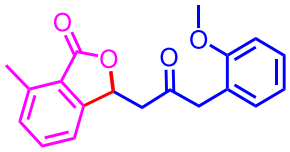
123.46, 119.83, 118.34, 112.80, 75.93, 43.71, 17.50.
ESI-MS calcd. for $C_{15}H_{12}O_4H$ [M+H$^+$]: 257.0814, found: 257.0819.

7-methyl-3-(2-oxo-3-phenylpropyl)isobenzofuran-1(3*H*)-one (3ga)



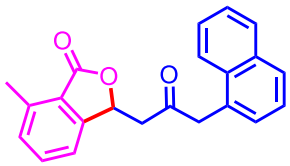
The product was synthsized by standard condition 2 in 40% yield.
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.47 (t, *J* = 7.6 Hz, 1H), 7.36 - 7.31 (m, 2H), 7.30 - 7.27 (m, 1H), 7.26 - 7.24 (m, 1H), 7.22 - 7.19 (m, 2H), 7.20 - 7.15 (m, 1H), 5.84 (t, *J* = 6.5 Hz, 1H), 3.80 (d, *J* = 15.5 Hz, 1H), 3.76 (d, *J* = 15.5 Hz, 1H), 3.08 (dd, *J* = 17.5, 6.5 Hz, 1H), 2.86 (dd, *J* = 17.5, 6.5 Hz, 1H), 2.67 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 204.54, 170.29, 149.95, 140.00, 134.07, 133.30, 131.13, 129.08 129.61, 127.54, 123.37, 119.61, 75.92, 50.92, 46.78, 17.47.
ESI-MS calcd. for $C_{18}H_{16}O_3Na$ [M+Na$^+$]: 303.0996, found: 303.0991.

3-(3-(2-methoxyphenyl)-2-oxopropyl)-7-methylisobenzofuran-1(3*H*)-one (3ha)



The product was synthesized by standard condition 2 in 64% yield.
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.47 (t, *J* = 7.6 Hz, 1H), 7.26 - 7.24 (m, 1H), 7.24 - 7.21 (m, 2H), 7.14 (dd, *J* = 7.4, 1.6 Hz, 1H), 6.92 (td, *J* = 7.4, 1.0 Hz, 1H), 6.86 (dd, *J* = 8.5, 1.0 Hz, 1H), 5.87 (t, *J* = 6.5 Hz, 1H), 3.77 (s, 3H), 3.74 - 3.72 (m, 2H), 3.11 (dd, *J* = 17.5, 6.5 Hz, 1H), 2.84 (dd, *J* = 17.5, 6.5 Hz, 1H), 2.67 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 205.23, 170.44, 157.34, 150.31, 139.84, 133.96, 131.38, 131.01, 129.05, 123.38, 122.78, 120.95, 119.84, 110.63, 76.04, 55.44, 46.67, 45.60, 17.46.
ESI-MS calcd. for $C_{19}H_{18}O_4H$ [M+H$^+$]: 311.1283, found: 311.1287.

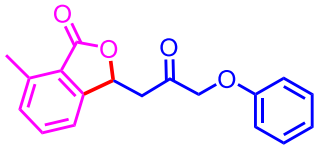
7-methyl-3-(3-(naphthalen-1-yl)-2-oxopropyl)isobenzofuran-1(3*H*)-one (3ia)



The product was synthesized by standard condition 2 in 76% yield.

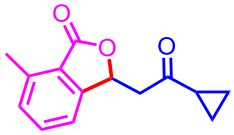
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.91 - 7.84 (m, 2H), 7.80 (d, *J* = 8.0 Hz, 1H), 7.58 - 7.48 (m, 2H), 7.45 - 7.35 (m, 3H), 7.22 (d, *J* = 7.5 Hz, 1H), 7.09 (d, *J* = 7.6 Hz, 1H), 5.82 (t, *J* = 6.5 Hz, 1H), 4.23 (d, *J* = 16.0 Hz, 1H), 4.18 (d, *J* = 16.0 Hz, 1H), 3.05 (dd, *J* = 17.5, 7.0 Hz, 1H), 2.79 (dd, *J* = 17.5, 7.0 Hz, 1H), 2.65 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 205.02, 170.23, 149.87, 139.91, 134.06, 133.98, 132.20, 131.06, 130.02, 129.02, 128.63, 128.55, 126.89, 126.19, 125.73, 123.74, 123.31, 119.57, 75.95, 49.15, 46.32, 17.42.
ESI-MS calcd. for $C_{22}H_{18}O_3Na$ [M+Na$^+$]: 353.1153, found: 353.1158.

7-methyl-3-(2-oxo-3-phenoxypropyl)isobenzofuran-1(3*H*)-one (3ja)



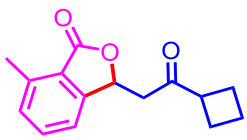
The product was synthesized by standard condition 2 in 78% yield.
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.52 (t, *J* = 7.6 Hz, 1H), 7.35 - 7.21 (m, 4H), 7.00 (dd, *J* = 7.5, 1.0 Hz, 1H), 6.89 - 6.85 (m, 2H), 5.92 (t, *J* = 6.5 Hz, 1H), 4.65 (d, *J* = 16.5 Hz, 1H), 4.61 (d, *J* = 16.5 Hz, 1H), 3.27 (dd, *J* = 18.0, 7.5 Hz, 1H), 3.09 (dd, *J* = 18.0, 6.0 Hz, 1H), 2.68 (s, 3H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 204.15, 170.07, 157.42, 149.65, 140.02, 134.06, 131.15, 129.81, 123.29, 122.05, 119.45, 114.50, 75.25, 73.01, 44.41, 17.37.
ESI-MS calcd. for $C_{18}H_{16}O_4Na$ [M+Na$^+$]: 319.0946, found: 319.0951.

3-(2-cyclopropyl-2-oxoethyl)-7-methylisobenzofuran-1(3*H*)-one (3ka)



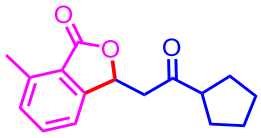
The product was synthesized by standard condition 1 for 48h in 52% yield.
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.50 (t, *J* = 7.5 Hz, 1H), 7.27 (d, *J* = 7.5 Hz, 1H), 7.24 (d, *J* = 7.5 Hz, 1H), 5.88 (t, *J* = 6.5 Hz, 1H), 3.22 (dd, *J* = 17.0, 6.5 Hz, 1H), 3.02 (dd, *J* = 17.0, 6.0 Hz, 1H), 2.68 (s, 3H), 1.97 (dd, *J* = 8.0, 4.5 Hz, 1H), 1.18 - 1.08 (m, 2H), 1.02 - 0.91 (m, 2H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 206.72, 170.31, 150.07, 139.82, 133.93, 130.96, 123.32, 119.62, 75.88, 48.05, 21.27, 17.35, 11.56, 11.48.
ESI-MS calcd. for $C_{14}H_{14}O_3Na$ [M+Na$^+$]: 253.0840, found: 253.0847.

3-(2-cyclobutyl-2-oxoethyl)-7-methylisobenzofuran-1(3*H*)-one (3la)



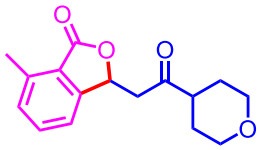
The product was synthesized by standard condition 1 for 48h in 71% yield.
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.50 (t, *J* = 7.5 Hz, 1H), 7.27 (d, *J* = 7.5 Hz, 1H), 7.24 (d, *J* = 7.5 Hz, 1H), 5.88 (t, *J* = 6.5 Hz, 1H), 3.30 (p, *J* = 8.6 Hz, 1H), 2.99 (dd, *J* = 17.5, 7.0 Hz, 1H), 2.79 (dd, *J* = 17.5, 6.0 Hz, 1H), 2.67 (s, 3H), 2.36 - 2.10 (m, 4H), 2.02 - 1.93 (m, 1H), 1.89 - 1.80 (m, 1H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 207.18, 169.78, 149.63, 139.38, 133.46, 130.49, 122.82, 118.99, 75.44, 45.35, 44.39, 23.59, 17.17, 16.87.
ESI-MS calcd. for $C_{15}H_{16}O_3Na$ [M+Na$^+$]: 267.0996, found: 267.1001.

3-(2-cyclopentyl-2-oxoethyl)-7-methylisobenzofuran-1(3*H*)-one (3ma)



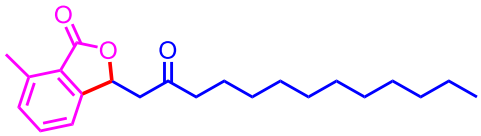
The product was synthesized by standard condition 1 for 48h in 72% yield.
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.50 (t, *J* = 7.5 Hz, 1H), 7.26 (d, *J* = 7.5 Hz, 1H), 7.23 (d, *J* = 7.5 Hz, 1H), 5.90 (t, *J* = 6.5 Hz, 1H), 3.10 (dd, *J* = 17.5, 7.0 Hz, 1H), 2.95 - 2.82 (m, 2H), 2.68 (s, 3H), 1.87 - 1.54 (m, 8H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 209.15, 170.41, 150.32, 139.96, 134.05, 131.06, 123.45, 119.61, 76.18, 52.06, 46.68, 28.76, 28.59, 26.12, 26.08, 17.47.
ESI-MS calcd. for $C_{16}H_{18}O_3Na$ [M+Na$^+$]: 281.1153, found: 281.1149.

7-methyl-3-(2-oxo-2-(tetrahydro-2*H*-pyran-4-yl)ethyl)isobenzofuran-1(3*H*)-one (3na)



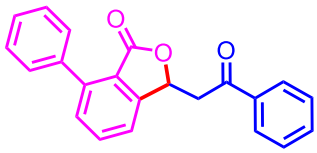
The product was synthesized by standard condition 1 for 48h in 77% yield.
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.50 (t, *J* = 7.5 Hz, 1H), 7.27 (d, *J* = 7.5 Hz, 1H), 7.22 (d, *J* = 7.5 Hz, 1H), 5.88 (t, *J* = 6.5 Hz, 1H), 4.03 - 3.95 (m, 2H), 3.44 - 3.38 (m, 2H), 3.09 (dd, *J* = 17.5, 7.0 Hz, 1H), 2.87 (dd, *J* = 17.5, 6.0 Hz, 1H), 2.67 (s, 3H), 2.60 (tt, *J* = 11.2, 4.2 Hz, 1H), 1.83 - 1.63 (m, 4H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 208.02, 170.26, 150.06, 140.05, 134.12, 131.17, 123.39, 119.52, 76.01, 67.17, 48.16, 45.39, 27.98, 17.45.
ESI-MS calcd. for $C_{16}H_{18}O_4H$ [M+H$^+$]: 275.1283, found: 275.1290.

7-methyl-3-(2-oxotridecyl)isobenzofuran-1(3*H*)-one (3oa)



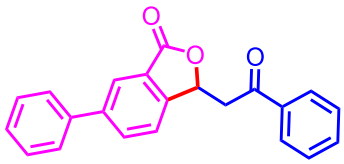
The product was synthesized by standard condition 1 for 48h in 63% yield.
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.50 (t, *J* = 7.5 Hz, 1H), 7.27 (d, *J* = 7.5 Hz, 2H), 7.23 (d, *J* = 7.5 Hz, 1H), 5.87 (t, *J* = 6.5 Hz, 1H), 3.04 (dd, *J* = 17.5, 7.0 Hz, 1H), 2.84 (dd, *J* = 17.5, 6.0 Hz, 1H), 2.68 (s, 3H), 2.56 - 2.39 (m, 2H), 1.63 - 1.57 (m, 2H), 1.31 - 1.22 (m, 16H), 0.87 (t, *J* = 6.9 Hz, 3H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 207.12, 170.23, 150.06, 139.88, 133.94, 130.98, 123.30, 119.49, 75.93, 47.46, 43.70, 31.90, 29.59, 29.44, 29.37, 29.33, 29.11, 23.52, 22.68, 17.34, 14.11.
ESI-MS calcd. for $C_{22}H_{32}O_3Na$ [M+Na^+]: 367.2248, found: 367.2241.

3-(2-oxo-2-phenylethyl)-7-phenylisobenzofuran-1(3*H*)-one (3ab)



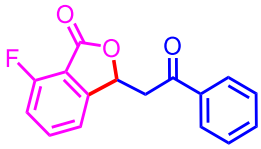
The product was synthesized by standard condition 2 in 69% yield.
^{1}H NMR (600 MHz, $CDCl_3$): δ 8.01 - 7.95 (m, 2H), 7.68 (t, *J* = 7.5 Hz, 1H), 7.64 - 7.58 (m, 1H), 7.57 - 7.52 (m, 3H), 7.52 - 7.40 (m, 6H), 6.17 (t, *J* = 6.6 Hz, 1H), 3.78 (dd, *J* = 17.4, 6.0 Hz, 1H), 3.45 (dd, *J* = 17.4, 6.6 Hz, 1H).
^{13}C NMR (151 MHz, $CDCl_3$): δ 196.28, 169.03, 151.20, 142.94, 136.48, 136.34, 134.21, 133.99, 131.35, 129.66, 128.96, 128.52, 128.32, 128.12, 121.97, 121.55, 75.77, 44.06.
ESI-MS calcd. for $C_{22}H_{16}O_3Na$ [M+Na^+]: 351.0996, found: 351.1001.

3-(2-oxo-2-phenylethyl)-6-phenylisobenzofuran-1(3*H*)-one (3ac)



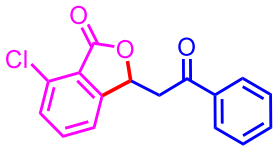
The product was synthesized by standard condition 2 in 40% yield.
^{1}H NMR (500 MHz, $CDCl_3$): δ 8.12 (d, *J* = 1.6 Hz, 1H), 8.00 – 7.96 (m, 2H), 7.88 (dd, *J* = 8.0, 1.7 Hz, 1H), 7.66 – 7.59 (m, 4H), 7.54 – 7.45 (m, 4H), 7.44 – 7.38 (m, 1H), 6.27 – 6.19 (m, 1H), 3.84 (dd, *J* = 17.7, 5.6 Hz, 1H), 3.44 (dd, *J* = 17.6, 7.6 Hz, 1H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 196.26, 170.30, 148.64, 143.21, 139.49, 136.27, 134.08, 133.55, 129.25, 129.01, 128.33, 127.39, 126.79, 124.11, 123.44, 77.35, 43.91.
ESI-MS calcd. for $C_{22}H_{16}O_3Na$ [M+Na^+]: 351.0996, found: 351.1001.

7-fluoro-3-(2-oxo-2-phenylethyl)isobenzofuran-1(3*H*)-one (3ad)



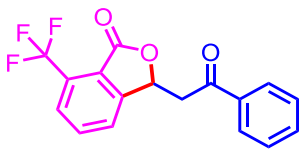
The product was synthesized by standard condition 2 in 52% yield.
^{1}H NMR (600 MHz, $CDCl_3$): δ 8.00 - 7.94 (m, 2H), 7.72 - 7.59 (m, 2H), 7.53 - 7.49 (m, 2H), 7.38 (d, *J* = 7.8 Hz, 1H), 7.19 (t, *J* = 8.5 Hz, 1H), 6.18 (d, *J* = 7.2 Hz, 1H), 3.81 (dd, *J* = 17.4, 6.0 Hz, 1H), 3.43 (dd, *J* = 17.4, 7.0 Hz, 1H).
^{13}C NMR (151 MHz, $CDCl_3$): δ 195.92, 166.32, 159.77 (d, *J* = 264.8 Hz), 152.45, 137.01 (d, *J* = 7.8 Hz), 136.13, 134.15, 129.03, 128.30, 118.95 (d, *J* = 4.4 Hz), 116.59 (d, *J* = 18.7 Hz), 113.91 (d, *J* = 14.4 Hz), 76.98, 43.70.
J**ESI-MS** calcd. for $C_{16}H_{11}FO_3H$ [M+H$^+$]: 271.0770, found: 271.0764.
^{19}F NMR (500 MHz, $CDCl_3$): δ -113.95.

7-chloro-3-(2-oxo-2-phenylethyl)isobenzofuran-1(3*H*)-one (3ae)



The product was synthesized by standard condition 2 in 77% yield.
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.97 - 7.93 (m, 2H), 7.64 - 7.54 (m, 2H), 7.52 - 7.45 (m, 4H), 6.14 - 6.10 (m, 1H), 3.78 (dd, *J* = 17.5, 5.5 Hz, 1H), 3.39 (dd, *J* = 17.5, 7.0 Hz, 1H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 195.98, 167.17, 152.25, 136.17, 135.37, 134.14, 133.55, 131.00, 129.03, 128.31, 122.76, 121.42, 75.94, 43.75.
ESI-MS calcd. for $C_{16}H_{11}ClO_3Na$ [M+Na$^+$]: 309.0294, found: 309.0291.

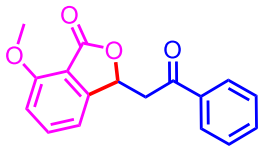
3-(2-oxo-2-phenylethyl)-7-(trifluoromethyl)isobenzofuran-1(3*H*)-one (3af)



The product was synthesized by method 2 in 68% yield.
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.98 - 7.93 (m, 2H), 7.86 - 7.81 (m, 2H), 7.80 – 7.75 (m, 1H), 7.64 - 7.60 (m, 1H), 7.52 - 7.46 (m, 2H), 6.19 (dd, *J* = 7.5, 5.0 Hz, 1H), 3.84 (dd, *J* = 17.8, 5.5 Hz, 1H), 3.42 (dd, *J* = 17.8, 7 Hz, 1H).

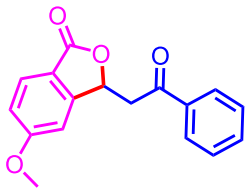
^{13}C NMR (126 MHz, $CDCl_3$): δ 195.99, 166.13, 152.28, 136.09, 134.34, 134.22, 129.05, 128.84 (q, *J* = 35.3 Hz), 128.31, 127.16 (q, *J* = 5.4 Hz), 126.86, 123.40, 122.31 (q, *J* = 274.5 Hz), 76.59, 43.64.
ESI-MS calcd. for $C_{17}H_{11}F_3O_3Na$ [M+Na$^+$]: 343.0557, found: 343.0551.
^{19}F NMR (500 MHz, $CDCl_3$): δ -60.97.

7-methoxy-3-(2-oxo-2-phenylethyl)isobenzofuran-1(3*H*)-one (3ag)



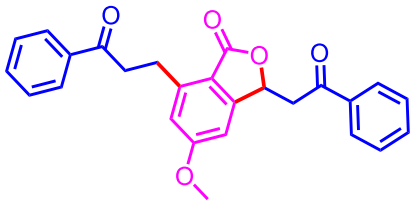
The product was synthesized by standard condition 2 in 60% yield.
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.96 - 7.92 (m, 2H), 7.62 - 7.56 (m, 2H), 7.51 - 7.43 (m, 2H), 7.07 (d, *J* = 8.0 Hz, 1H), 6.94 (d, *J* = 8.0 Hz, 1H), 6.08 (t, *J* = 6.5 Hz, 1H), 3.99 (s, 3H), 3.71 (dd, *J* = 17.5, 6.0 Hz, 1H), 3.37 (dd, *J* = 17.5, 7.0 Hz, 1H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 196.16, 168.26, 158.79, 152.68, 136.63, 136.40, 133.93, 128.95, 128.31, 114.40, 113.46, 111.15, 76.15, 56.17, 43.91.
ESI-MS calcd. for $C_{17}H_{14}O_4H$ [M+H$^+$]: 283.0970, found: 283.0966.

5-methoxy-3-(2-oxo-2-phenylethyl)isobenzofuran-1(3*H*)-one (3ah)



The product was synthesized by standard condition 2 in 20% yield.
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.98 – 7.94 (m, 2H), 7.81 (d, *J* = 8.4 Hz, 1H), 7.64 – 7.58 (m, 1H), 7.50 (t, *J* = 7.8 Hz, 2H), 7.08 – 6.97 (m, 2H), 6.14 – 6.03 (m, 1H), 3.87 (s, 3H), 3.80 (dd, *J* = 17.7, 5.5 Hz, 1H), 3.38 (dd, *J* = 17.7, 7.6 Hz, 1H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 196.43, 170.07, 164.98, 152.76, 136.29, 134.05, 129.00, 128.31, 127.38, 118.20, 117.07, 106.89, 76.65, 56.00, 43.98.
ESI-MS calcd. for $C_{17}H_{14}O_4H$ [M+H$^+$]: 283.0970, found: 283.0966.

5-methoxy-3-(2-oxo-2-phenylethyl)-7-(3-oxo-3-phenylpropyl)isobenzofuran-1(3*H*)-one (3ah')



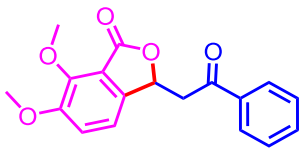
The product was synthesized by standard condition 2 in 31% yield.

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.01 – 7.93 (m, 4H), 7.63 – 7.58 (m, 1H), 7.57 – 7.52 (m, 1H), 7.51 – 7.40 (m, 4H), 6.95 (d, *J* = 2.1 Hz, 1H), 6.86 – 6.81 (m, 1H), 6.09 – 6.02 (m, 1H), 3.85 (s, 3H), 3.75 (dd, *J* = 17.6, 5.9 Hz, 1H), 3.48 – 3.32 (m, 5H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 199.19, 196.39, 169.91, 164.73, 153.67, 144.79, 136.84, 136.35, 134.00, 133.24, 128.98, 128.72, 128.33, 128.32, 118.00, 115.61, 105.12, 76.05, 55.96, 44.08, 39.39, 26.63.

ESI-MS calcd. for $C_{26}H_{22}O_5H$ [M+H$^+$]: ,found: 415.1545, found: 415.1551.

6,7-dimethoxy-3-(2-oxo-2-phenylethyl)isobenzofuran-1(3*H*)-one (3ai)



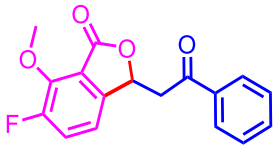
The product was synthesized by standard condition 2 in 55% yield.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.88 - 7.83 (m, 2H), 7.53 - 7.45 (m, 1H), 7.41 - 7.34 (m, 2H), 7.12 - 7.01 (m, 2H), 5.94 (d, *J* = 6.5 Hz, 1H), 4.00 (s, 3H), 3.80 (s, 3H), 3.62 (dd, *J* = 17.5, 5.5 Hz, 1H), 3.25 (dd, *J* = 17.5, 7.5 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 196.28, 167.62, 152.79, 148.37, 142.71, 136.28, 133.83, 128.83, 128.17, 119.53, 118.14, 117.31, 75.90, 62.38, 56.88, 44.15.

ESI-MS calcd. for $C_{18}H_{16}O_5Na$ [M+Na$^+$]: 335.0895, found: 335.0890.

6-fluoro-7-methoxy-3-(2-oxo-2-phenylethyl)isobenzofuran-1(3*H*)-one (3aj)



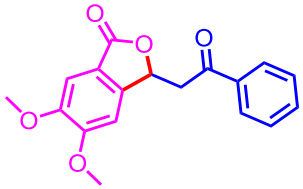
The product was synthesized by standard condition 2 in 86% yield.

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.00 - 7.94 (m, 2H), 7.63 (t, *J* = 7.4 Hz, 1H), 7.51 (t, *J* = 7.8 Hz, 2H), 7.38 (dd, *J* = 12.0, 8.5 Hz, 1H), 7.16 (dd, *J* = 8.5, 3.4 Hz, 1H), 6.08 (d, *J* = 6.5 Hz, 1H), 4.22 (d, *J* = 2.5 Hz, 3H), 3.77 (dd, *J* = 17.5, 5.5 Hz, 1H), 3.40 (dd, *J* = 17.5, 7.5 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 196.11, 167.02, 155.24, 153.27, 146.87, 136.20, 134.06, 128.98, 128.27, 123.89 (d, *J* = 21.9 Hz), 118.43 (d, *J* = 3.9 Hz), 116.77 (d, *J* = 7.7 Hz), 75.99, 62.47 (d, *J* = 5.4 Hz), 43.91.

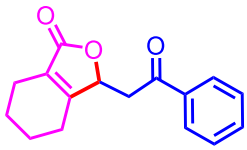
ESI-MS calcd. for $C_{17}H_{13}FO_4H$ [M+H$^+$]: 301.0876, found: 301.0880.
^{19}F NMR (500 MHz, $CDCl_3$): -131.14.

5,6-dimethoxy-3-(2-oxo-2-phenylethyl)isobenzofuran-1(3*H*)-one (3ak)



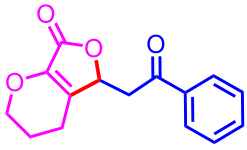
The product was synthesized by standard condition 2 in 50% yield.
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.99 – 7.95 (m, 2H), 7.64 – 7.59 (m, 1H), 7.50 (t, *J* = 7.8 Hz, 2H), 7.29 (s, 1H), 7.01 (s, 1H), 6.10 – 6.01 (m, 1H), 3.94 (s, 6H), 3.82 (dd, *J* = 17.7, 5.2 Hz, 1H), 3.35 (dd, *J* = 17.7, 8.0 Hz, 1H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 196.77, 170.65, 154.96, 150.77, 144.57, 136.28, 134.08, 129.01, 128.29, 117.84, 106.12, 104.57, 76.83, 56.55, 56.44, 44.10.
ESI-MS calcd. for $C_{18}H_{16}O_5Na$ [M+Na$^+$]: 335.0895, found: 335.0890.

3-(2-oxo-2-phenylethyl)-4,5,6,7-tetrahydroisobenzofuran-1(3*H*)-one (3al)



The product was synthesized by standard condition 2 in 66% yield.
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.95 - 7.90 (m, 2H), 7.62 - 7.57 (m, 1H), 7.51 - 7.44 (m, 2H), 5.53 - 5.48 (m, 1H), 3.41 (dd, *J* = 17.1, 6.7 Hz, 1H), 3.22 (dd, *J* = 17.1, 6.0 Hz, 1H), 2.38 - 2.30 (m, 1H), 2.27 - 2.18 (m, 3H), 1.79 - 1.62 (m, 4H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 196.05, 173.19, 163.69, 136.50, 133.91, 128.95, 128.35, 127.19, 78.96, 41.40, 23.72, 21.74, 21.61, 20.12.
ESI-MS calcd. for $C_{16}H_{16}O_3Na$ [M+Na$^+$]: 279.0996, found: 279.1001.

5-(2-oxo-2-phenylethyl)-3,4-dihydro-2H-furo[3,4-b]pyran-7(5*H*)-one (3am)



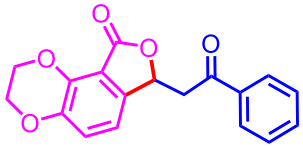
The product was synthesized by standard condition 2 in 66% yield.
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.97 - 7.92 (m, 2H), 7.64 - 7.58 (m, 1H), 7.51 - 7.46 (m, 2H), 5.56 - 5.52 (m, 1H), 4.27 - 4.20 (m, 1H), 4.16 - 4.09 (m, 1H), 3.54 (dd, *J* = 17.2, 5.9 Hz, 1H), 3.24 (dd,

J = 17.2, 7.0 Hz, 1H), 2.43 (dt, *J* = 18.4, 5.5 Hz, 1H), 2.23 (dt, *J* = 18.4, 6.6 Hz, 1H), 2.02 - 1.95 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 196.12, 166.72, 141.49, 136.36, 134.02, 133.86, 129.00, 128.33, 76.77, 67.88, 42.29, 21.38, 20.25.Z

ESI-MS calcd. for $C_{15}H_{14}O_4Na$ [M+Na$^+$]: 281.0789, found: 281.0795.

7-(2-oxo-2-phenylethyl)-2,3-dihydro-[1,4]dioxino[2,3-e]isobenzofuran-9(7*H*)-one (3an)



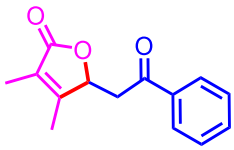
The product was synthesized by standard condition 2 in 32% yield.

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.99 - 7.89 (m, 2H), 7.63 - 7.56 (m, 1H), 7.52 - 7.46 (m, 2H), 7.13 (d, *J* = 8.0 Hz, 1H), 6.94 (dd, *J* = 8.0, 0.9 Hz, 1H), 6.06 - 6.01 (m, 1H), 4.47 - 4.42 (m, 2H), 4.34 - 4.29 (m, 2H), 3.71 (dd, *J* = 17.5, 5.5 Hz, 1H), 3.34 (dd, *J* = 17.5, 7.0 Hz, 1H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 196.31, 168.00, 144.10, 143.98, 142.60, 136.43, 133.95, 128.96, 128.31, 124.32, 114.71, 114.06, 76.41, 65.19, 64.26, 44.14.

ESI-MS calcd. for $C_{18}H_{14}O_5Na$ [M+Na$^+$]: 333.0738, found: 333.0733.

3,4-dimethyl-5-(2-oxo-2-phenylethyl)furan-2(5H)-one (3ao)



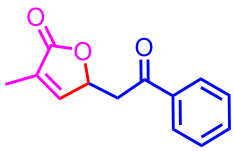
The product was synthesized by standard condition 2 in 72% yield.

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.99 - 7.82 (m, 2H), 7.67 - 7.56 (m, 1H), 7.54 - 7.42 (m, 2H), 5.45 (t, *J* = 6.1 Hz, 1H), 3.33 (dd, *J* = 17.4, 7.2 Hz, 1H), 3.25 (dd, *J* = 17.4, 5.2 Hz, 1H), 1.99 (s, 3H), 1.83 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 195.99, 174.15, 159.03, 136.47, 133.89, 128.92, 128.35, 124.19, 79.22, 41.10, 12.37, 8.69.

ESI-MS calcd. for $C_{14}H_{14}O_3Na$ [M+Na$^+$]: 253.0840, found: 253.0833.

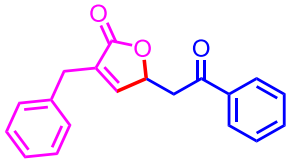
3-methyl-5-(2-oxo-2-phenylethyl)furan-2(5*H*)-one (3ap)



The product was synthesized by standard condition 2 in 46% yield.

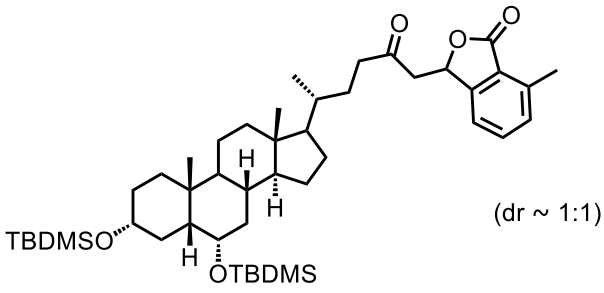
^{1}H NMR (500 MHz, $CDCl_3$): δ 8.00 - 7.89 (m, 2H), 7.67 - 7.55 (m, 1H), 7.52 - 7.43 (m, 2H), 7.30 (p, *J* = 1.6 Hz, 1H), 5.54 - 5.42 (m, 1H), 3.66 (dd, *J* = 17.5, 6.0 Hz, 1H), 3.11 (dd, *J* = 17.5, 8.3 Hz, 1H), 1.93 (t, *J* = 1.8 Hz, 3H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 196.20, 173.90, 148.91, 136.22, 134.02, 130.42, 128.99, 128.23, 77.33, 42.28, 10.79.
ESI-MS calcd. for $C_{13}H_{12}O_3Na$ [M+Na$^+$]: 239.0683, found: 239.0670.

3-benzyl-5-(2-oxo-2-phenylethyl)furan-2(5*H*)-one (3aq)



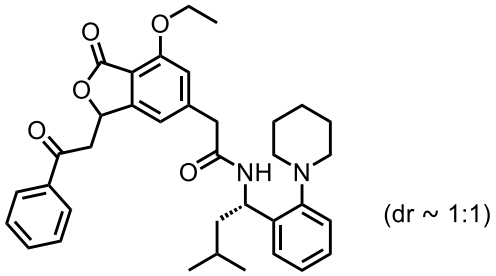
The product was synthesized by standard condition 2 in 27% yield.
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.93 - 7.87 (m, 2H), 7.65 - 7.56 (m, 1H), 7.51 - 7.44 (m, 2H), 7.35 - 7.30 (m, 2H), 7.28 - 7.25 (m, 1H), 7.25 - 7.21 (m, 2H), 7.10 (q, *J* = 1.5 Hz, 1H), 5.54 - 5.45 (m, 1H), 3.69 - 3.57 (m, 3H), 3.09 (dd, *J* = 17.5, 8.5 Hz, 1H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 196.01, 173.04, 149.30, 137.29, 136.10, 134.79, 134.05, 129.08, 128.98, 128.94, 128.23, 127.02, 77.62, 42.14, 31.96.
ESI-MS calcd. for $C_{19}H_{16}O_3Na$ [M+Na$^+$]: 315.0996, found:315.0992.

3-((5*R*)-5-((3*R*,5*R*,6*S*,8*S*,10*R*,13*R*,14*S*)-3,6-bis((tert-butyldimethylsilyl)oxy)-10,13-dimethylhexadecahydro-1H-cyclopenta[a]phenanthren-17-yl)-2-oxohexyl)-7-methylisobenzofuran-1(3*H*)-one (3pa)



^{1}H NMR (500 MHz, $CDCl_3$): δ 7.50 (t, *J* = 7.5 Hz, 1H), 7.27 (d, *J* = 7.5 Hz, 1H), 7.23 (d, *J* = 7.5 Hz, 1H), 5.87 (t, *J* = 6.5 Hz, 1H), 4.07 – 3.92 (m, 1H), 3.57 - 3.47 (m, 1H), 3.11 - 3.01 (m, 1H), 2.90 - 2.80 (m, 1H), 2.68 (s, 3H), 2.59 - 2.33 (m, 2H), 1.97 - 1.68 (m, 4H), 1.65 - 1.51 (m, 2H), 1.53 – 1.46 (m, 1H), 1.46-1.26 (m, 10H), 1.21 - 0.99 (m, 8H), 0.90 - 0.86 (m, 24H), 0.62 (s, 3H), 0.07 - 0.01 (m, 12H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 207.55, 170.35, 150.19, 140.00, 134.07, 131.11, 123.43, 119.64, 76.03, 73.08, 68.76, 56.26, 56.07, 49.70, 47.63, 47.59, 42.99, 40.70, 40.10, 39.73, 36.10, 36.06, 35.56, 35.36, 35.33, 35.02, 31.18, 29.95, 29.63, 29.57, 28.29, 26.14, 26.02, 24.33, 23.66, 20.91, 18.55, 18.25, 17.48, 12.19, -4.36, -4.49, -4.62, -4.64.
ESI-MS calcd. for $C_{46}H_{77}O_5Si_2$ [M+H$^+$]: 765.5309, found: 765.5314.

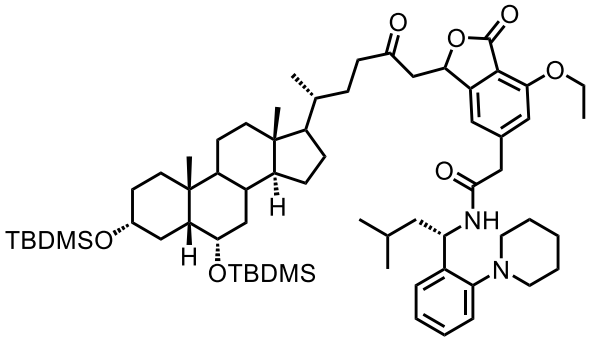
2-(7-ethoxy-1-oxo-3-(2-oxo-2-phenylethyl)-1,3-dihydroisobenzofuran-5-yl)-*N*-((*S*)-3-methyl-1-(2-(piperidin-1-yl)phenyl)butyl)acetamide (3ar)



(dr ~ 1:1)

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.96 - 7.90 (m, 2H), 7.64 - 7.56 (m, 1H), 7.52 - 7.43 (m, 2H), 7.23 - 7.10 (m, 3H), 7.09 - 7.02 (m, 1H), 6.94 - 6.80 (m, 2H), 6.00 (t, *J* = 6.5 Hz, 1H), 5.36 - 5.26 (m, 1H), 4.22 - 4.04 (m, 2H), 3.70 - 3.63 (m, 1H), 3.62 - 3.52 (m, 2H), 3.35 - 3.26 (m, 1H), 2.92 (s, 2H), 2.65 (s, 2H), 1.78 - 1.69 (m, 2H), 1.65 – 1.58 (m, 4H), 1.57 - 1.48 (m, 2H), 1.48 - 1.38 (m, 4H), 0.98 - 0.86 (m, 6H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 196.14, 168.50, 167.83, 158.12, 153.10, 145.00, 138.53, 136.33, 133.96, 128.95, 128.32, 128.23, 125.53, 123.06, 114.81, 112.98, 112.45, 75.78, 64.74, 55.30, 50.28, 46.63, 44.56, 43.92, 29.84, 26.83, 25.48, 24.15, 22.88, 22.65, 14.56, 14.53.
ESI-MS calcd. for $C_{36}H_{43}N_2O_5$ [M+H$^+$]: 583.3172, found: 583.3167.

2-(3-((5*R*)-5-((3*R*,5*R*,6S,10*R*,13*R*,14*S*)-3,6-bis((tert-butyldimethylsilyl)oxy)-10,13-dimethylhexadecahydro-1*H*-cyclopenta[a]phenanthren-17-yl)-2-oxohexyl)-7-ethoxy-1-oxo-1,3-dihydroisobenzofuran-5-yl)-*N*-((*S*)-3-methyl-1-(2-(piperidin-1-yl)phenyl)butyl)acetamide (4)



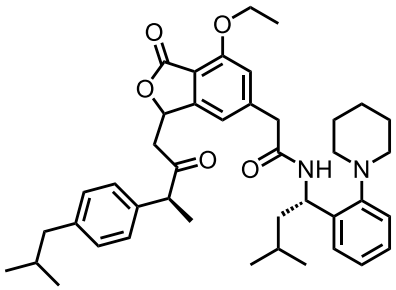
dr ~ 1:1

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.26 - 7.19 (m, 2H), 7.16 - 7.05 (m, 2H), 7.05 - 6.97 (m, 1H), 6.89 - 6.76 (m, 2H), 5.81 - 5.74 (m, 1H), 5.42 - 5.33 (m, 1H), 4.20 - 4.04 (m, 2H), 3.99 (dt, *J* = 11.6, 4.2 Hz, 1H), 3.63 - 3.49 (m, 3H), 3.03 - 2.91 (m, 3H), 2.85 - 2.72 (m, 1H), 2.72 - 2.59 (m, 2H), 2.55 - 2.32 (m, 2H), 1.97 - 1.81 (m, 3H), 1.81 - 1.52 (m, 15H), 1.51 - 0.95 (m, 29H), 0.96 - 0.81 (m, 38H), 0.66 - 0.62 (m, 3H), 0.08 - 0.03 (m, 12H).
^{13}C NMR (126 MHz, $CDCl_3$): δ 206.75, 167.63, 167.08, 157.48, 152.28, 151.97, 144.32, 138.04, 138.00, 127.57, 127.43, 124.76, 124.72, 122.57, 113.78, 113.74, 112.38, 112.26, 111.76, 74.92,

72.45, 68.13, 64.13, 55.63, 55.45, 54.34, 49.74, 49.08, 46.88, 46.80, 46.27, 46.22, 43.93, 42.36, 40.03, 39.47, 39.10, 35.47, 35.43, 34.93, 34.74, 34.39, 30.55, 29.31, 29.19, 28.98, 28.92, 27.67, 26.32, 25.51, 25.39, 24.87, 23.70, 23.62, 23.03, 22.27, 22.07, 20.28, 17.92, 17.61, 13.92, 13.88, 11.56, -4.99, -5.12, -5.25, -5.27.

ESI-MS calcd. for $C_{65}H_{104}N_2O_7Si_2Na$ [M+Na$^+$]: 1103.7279, found:1103.7283.

2-(7-ethoxy-3-((*S*)-3-(4-isobutylphenyl)-2-oxobutyl)-1-oxo-1,3-dihydroisobenzofuran-5-yl)-*N*-((*S*)-3-methyl-1-(2-(piperidin-1-yl)phenyl)butyl)acetamide (5)



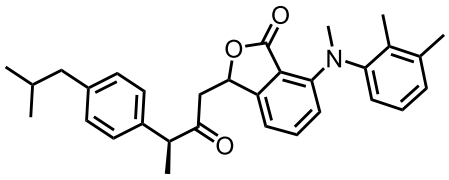
dr ~ 1:1

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.26 - 7.19 (m, 2H), 7.17 - 6.96 (m, 7H), 6.86 - 6.60 (m, 2H), 5.78 - 5.66 (m, 1H), 5.45 - 5.28 (m, 1H), 4.18 - 4.00 (m, 2H), 3.82 - 3.67 (m, 1H), 3.63 - 3.43 (m, 2H), 3.06 - 2.79 (m, 3H), 2.79 - 2.57 (m, 3H), 2.51 - 2.37 (m, 2H), 1.90 - 1.69 (m, 4H), 1.68 - 1.50 (m, 6H), 1.48 - 1.37 (m, 8H), 0.97 - 0.86 (m, 13H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 206.98, 168.26, 167.63, 157.99, 152.85, 144.87, 141.16, 138.70, 136.82, 129.97, 128.12, 127.66, 125.26, 123.07, 114.46, 114.18, 112.82, 112.21, 76.06, 75.41, 64.63, 55.33, 53.70, 53.11, 50.22, 46.82, 45.88, 45.42, 45.09, 44.45, 30.21, 26.89, 25.45, 24.21, 22.87, 22.49, 17.15, 14.49.

ESI-MS calcd. for $C_{42}H_{54}N_2O_5$ [M+Na$^+$]: 689.3930, found: 689.3923.

7-((2,3-dimethylphenyl)(methyl)amino)-3-((*S*)-3-(4-isobutylphenyl)-2-oxobutyl)isobenzofuran-1(3*H*)-one (6)

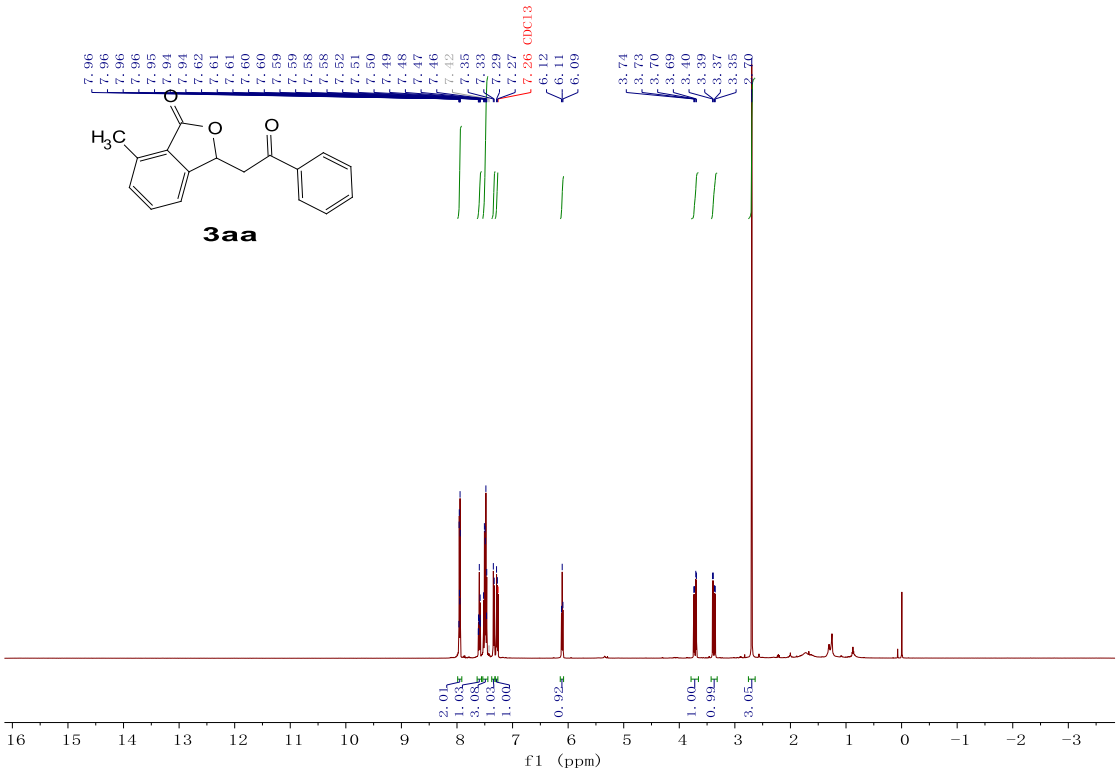


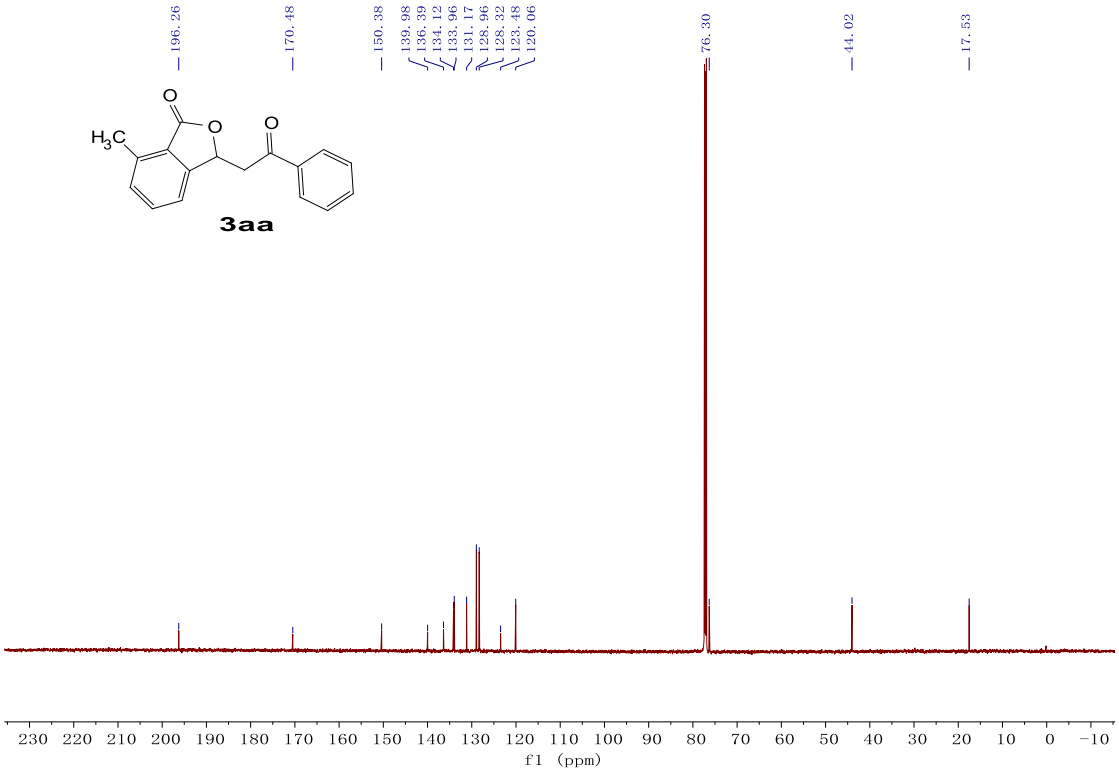
dr ~ 1:1

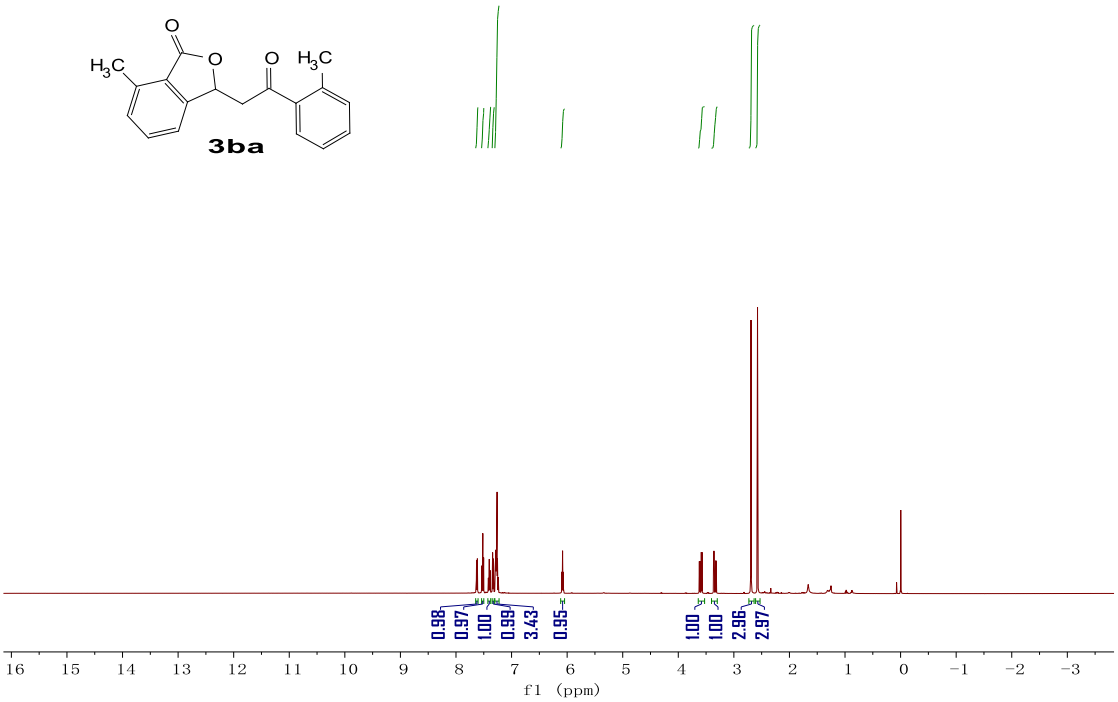
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.25 - 7.16 (m, 1H), 7.14 - 7.07 (m, 5H), 7.07 - 7.03 (m, 1H), 6.70 - 6.55 (m, 1H), 6.49 - 6.34 (m, 1H), 5.82 - 5.67 (m, 1H), 3.85 - 3.71 (m, 1H), 3.51 - 3.40 (m, 3H), 3.07 - 2.87 (m, 1H), 2.85 - 2.64 (m, 1H), 2.51 - 2.39 (m, 2H), 2.29 (s, 3H), 2.04 (s, 3H), 1.91 - 1.79 (m, 1H), 1.46 - 1.37 (m, 3H), 0.91 - 0.86 (m, 6H).

^{13}C NMR (126 MHz, CDCl3): δ 207.29, 168.25, 152.74, 150.79, 147.46, 141.11, 138.79, 136.96, 134.66, 134.13, 129.95, 127.88, 127.75, 126.55, 123.45, 118.29, 112.24, 111.62, 75.66, 75.13, 53.68, 53.23, 46.45, 45.94, 45.12, 43.76, 30.26, 22.50, 20.64, 17.22, 14.40.

ESI-MS calcd. for $C_{31}H_{35}NO_3Na$ [M+Na^+]: 492.2514, found: 492.2520.


7.96 7.96 7.96 7.96 7.95 7.94 7.94 7.62 7.61 7.61 7.60 7.60 7.59 7.59 7.58 7.58 7.52 7.51 7.50 7.49 7.48 7.47 7.46 7.42 7.35 7.33 7.29 7.27 7.26 CDCl3 6.12 6.11 6.09 3.74 3.73 3.70 3.69 3.40 3.39 3.37 3.35 2.10
O
O
O
H3C
3aa
2.01 1.03 3.08 1.03 1.00 0.92 1.00 0.99 3.05
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3
f1 (ppm)


196.26 170.48 150.38 139.98 136.39 134.12 133.96 131.17 128.96 128.32 123.48 120.06 76.30 44.02 17.53
O
O
O
H3C
3aa
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)


3ba
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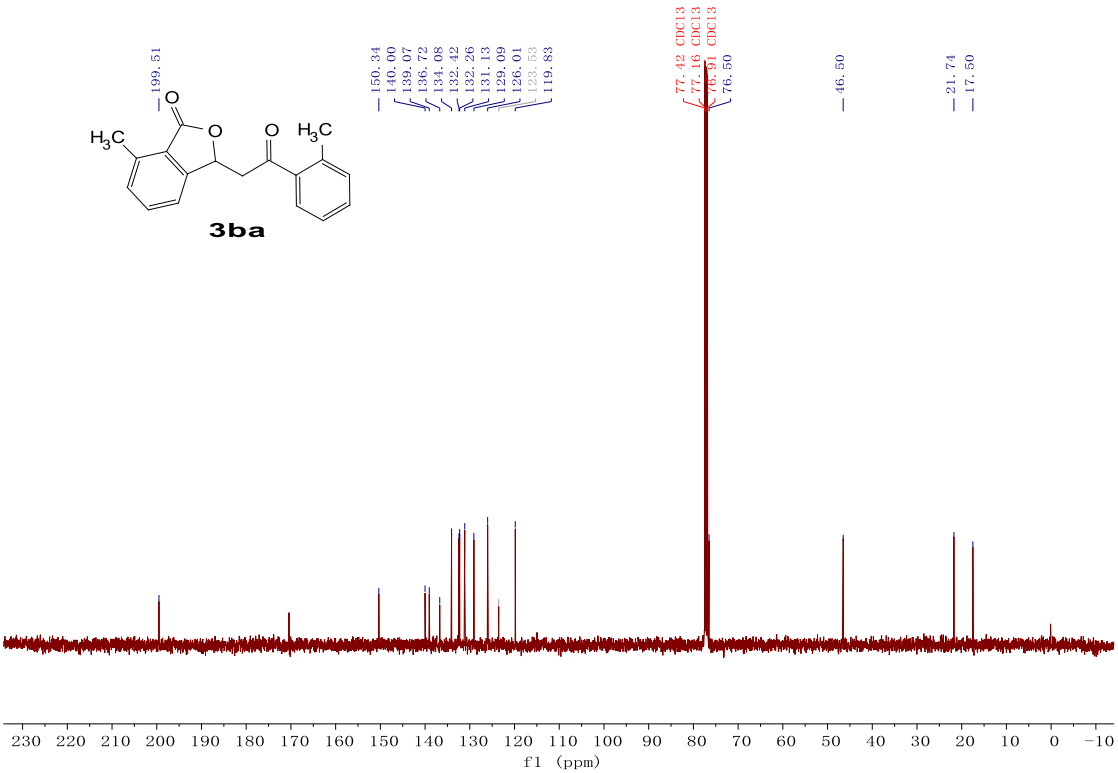
1.00
0.99
3.43
0.95
1.00
1.00
2.96
2.97
f1 (ppm)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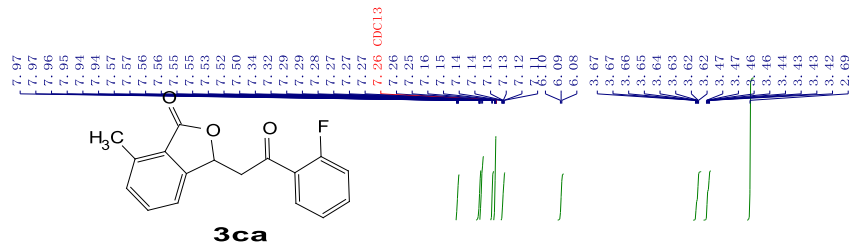
150.34
140.00
139.07
136.72
134.08
132.42
132.26
131.13
129.09
126.01
123.53
119.83
77.42 CDCl3
77.16 CDCl3
76.91 CDCl3
76.50
46.50
21.74
17.50
3ba
f1 (ppm)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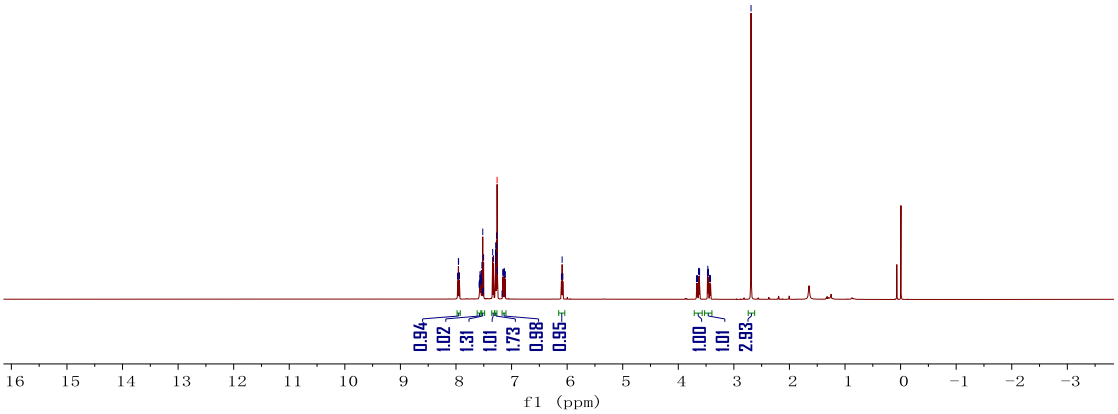
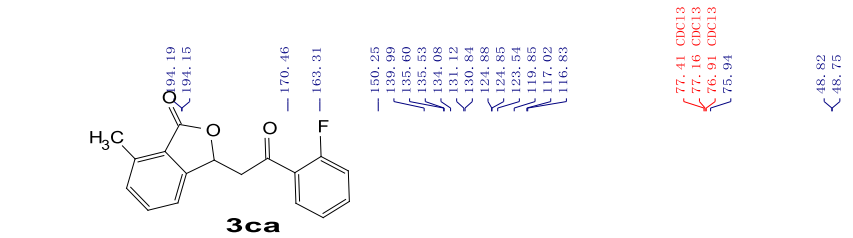
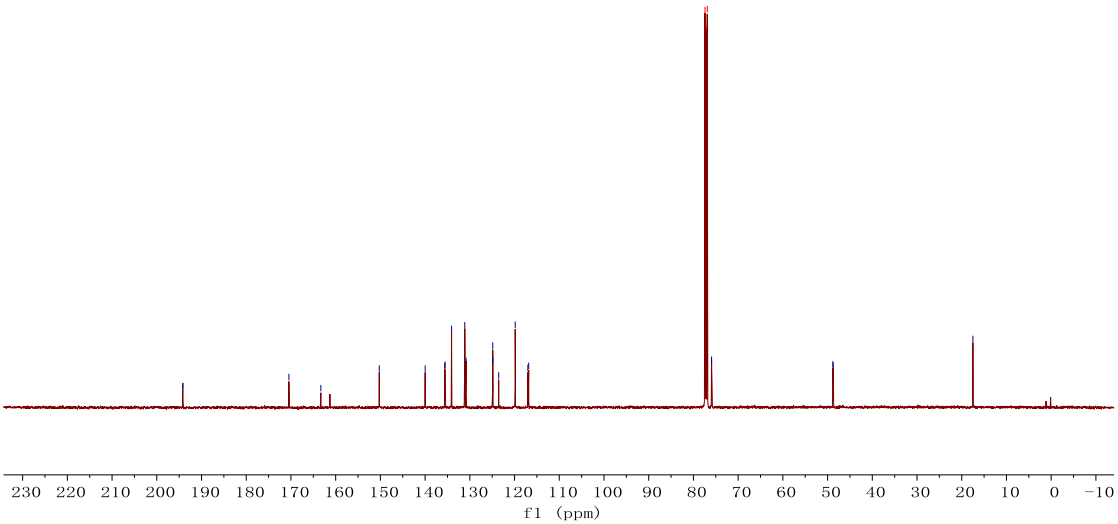
7.97
7.96
7.95
7.94
7.94
7.57
7.57
7.56
7.56
7.55
7.55
7.53
7.52
7.50
7.34
7.32
7.29
7.29
7.28
7.27
7.27
7.27
7.26 CDCl3
7.25
7.16
7.15
7.14
7.14
7.13
7.13
7.12
6.10
6.09
6.08
3.67
3.67
3.66
3.65
3.64
3.63
3.62
3.62
3.47
3.47
3.46
3.46
3.44
3.43
3.43
3.42
2.69
O
O
O
F
H3C

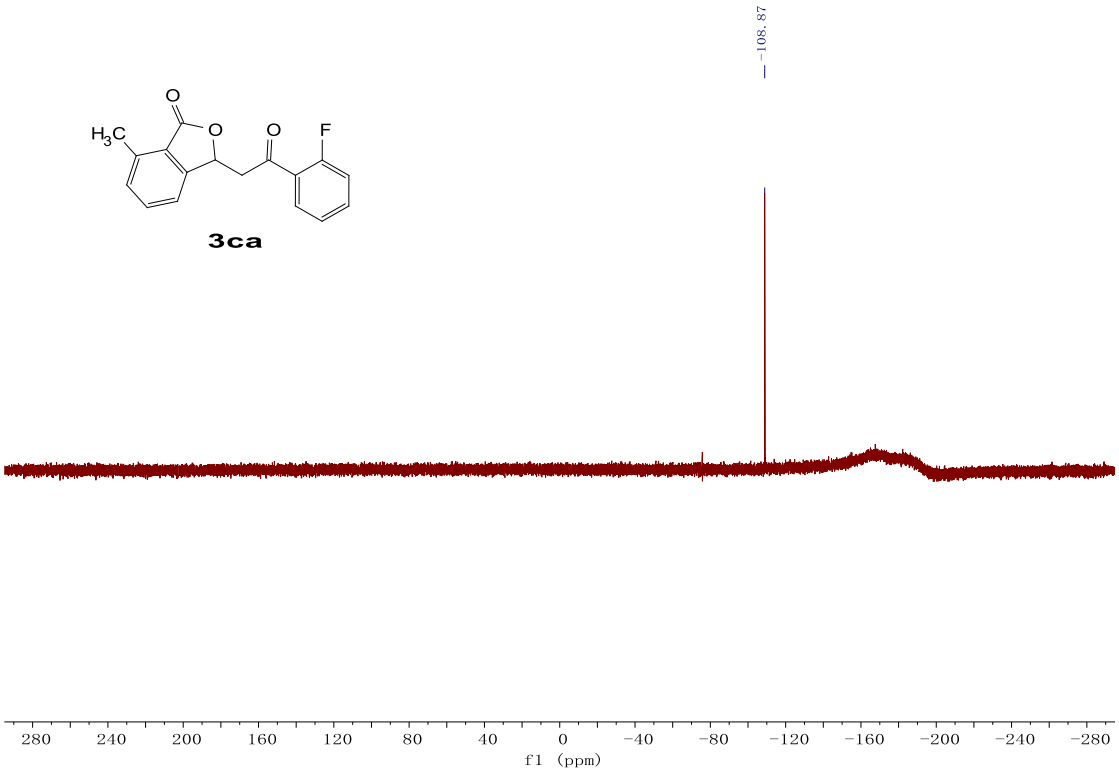
3ca

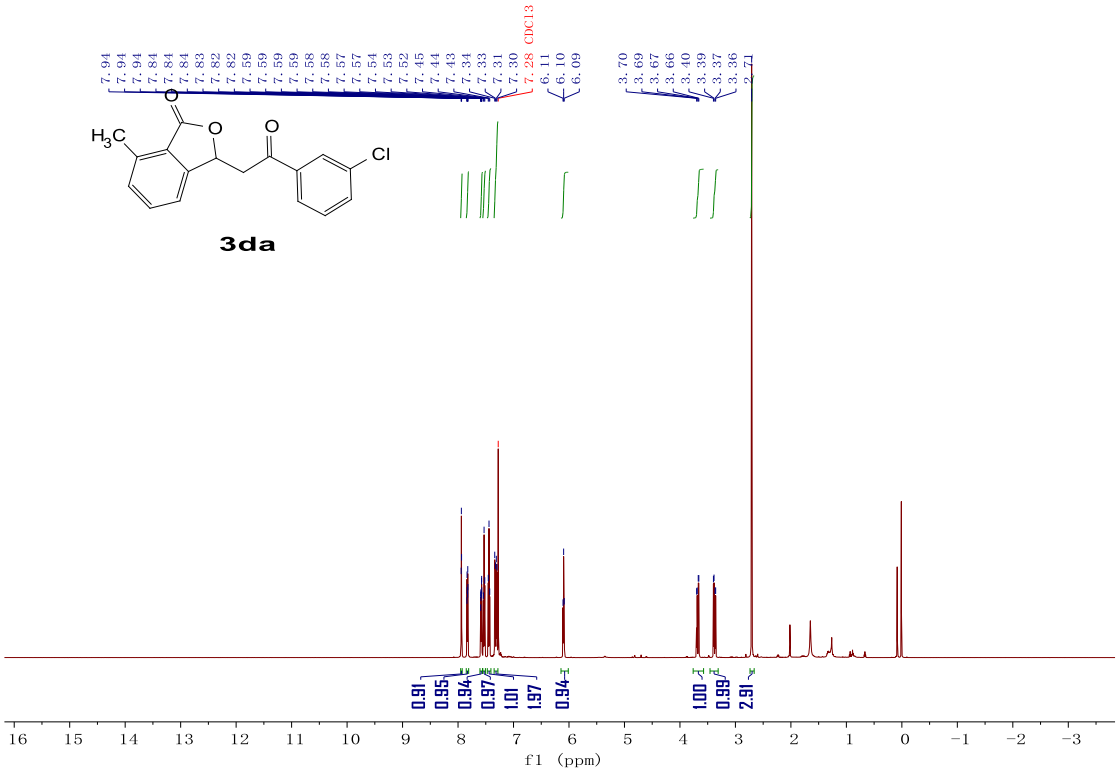

0.94
1.02
1.31
1.01
1.01
1.73
0.98
0.95
1.00
1.01
2.93
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3
f1 (ppm)


194.19
194.15
170.46
163.31
150.25
139.99
135.60
135.53
134.08
131.12
130.84
124.88
124.85
123.54
119.85
117.02
116.83
77.41 CDCl3
77.16 CDCl3
76.91 CDCl3
75.94
48.82
48.75
17.50
O
O
O
F
H3C

3ca


230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)


O
O
O
F
H3C
3ca
-108.87
280
240
200
160
120
80
40
0
-40
-80
-120
-160
-200
-240
-280
f1 (ppm)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7.94
7.84
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7.84
7.83
7.82
7.82
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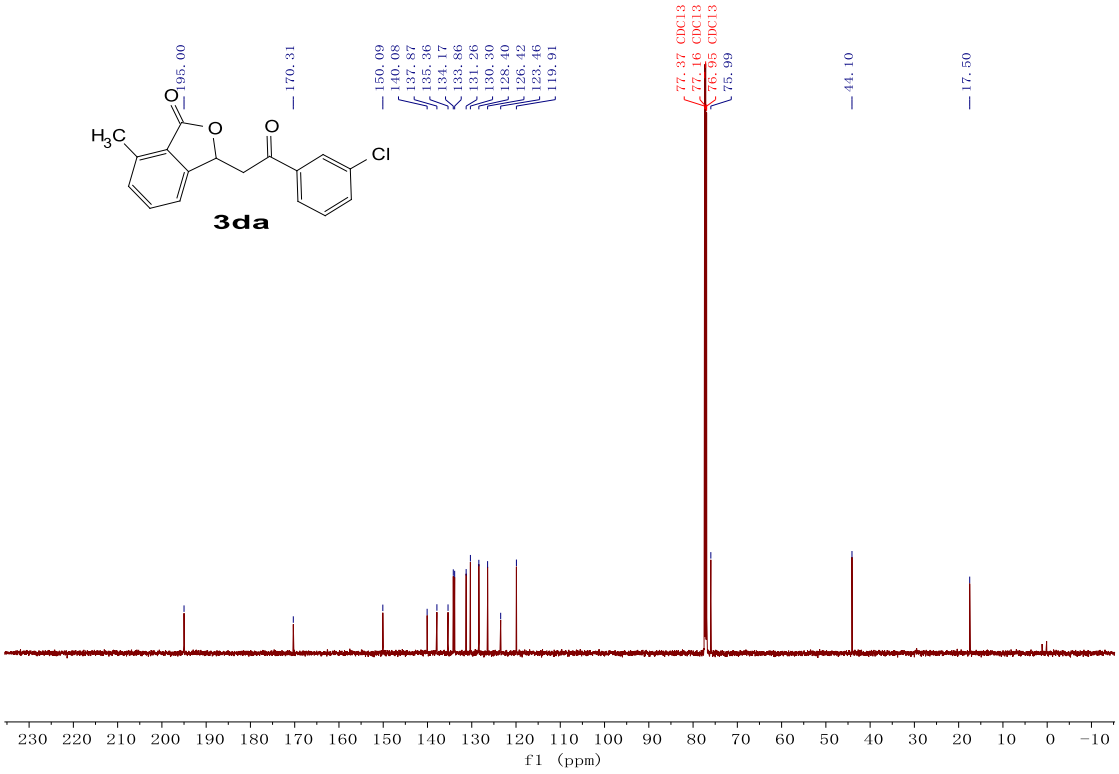
7.59
7.59
7.58
7.58
7.57
7.57
7.54
7.53
7.52
7.45
7.44
7.43
7.34
7.33
7.31
7.30
7.28 CDCl3
6.11
6.10
6.09
3.70
3.69
3.67
3.66
3.40
3.39
3.37
3.36
2.71
O
O
O
H3C
Cl
3da
0.91
0.95
0.94
0.97
1.01
1.97
0.94
1.00
0.99
2.91
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
f1 (ppm)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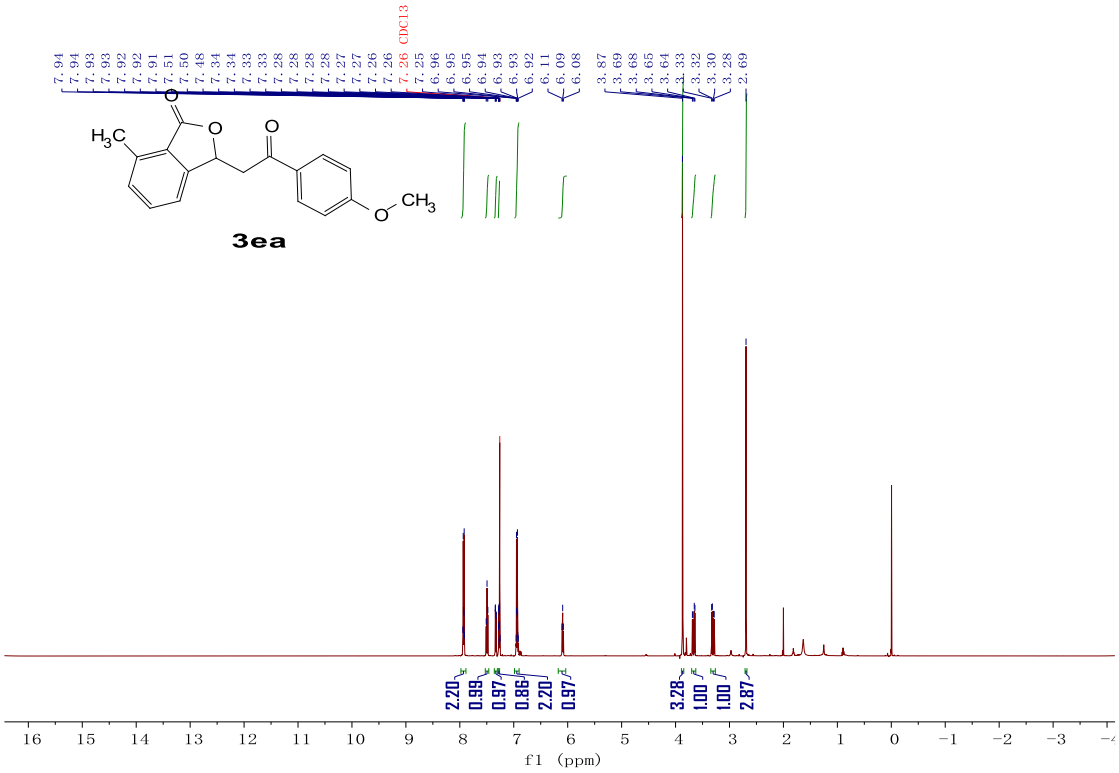
140.08
137.87
135.36
134.17
133.86
131.26
130.30
128.40
126.42
123.46
119.91
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
75.99
44.10
17.50
O
O
O
H3C
Cl
3da
f1 (ppm)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7.48
7.34
7.34
7.33
7.33
7.28
7.28
7.28
7.28
7.27
7.27
7.26
7.26 CDCl3
7.25
6.96
6.95
6.95
6.94
6.93
6.93
6.92
6.11
6.09
6.08
3.87
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3.64
3.33
3.32
3.30
3.28
2.69
O
O
O
H3C
O
CH3
3ea
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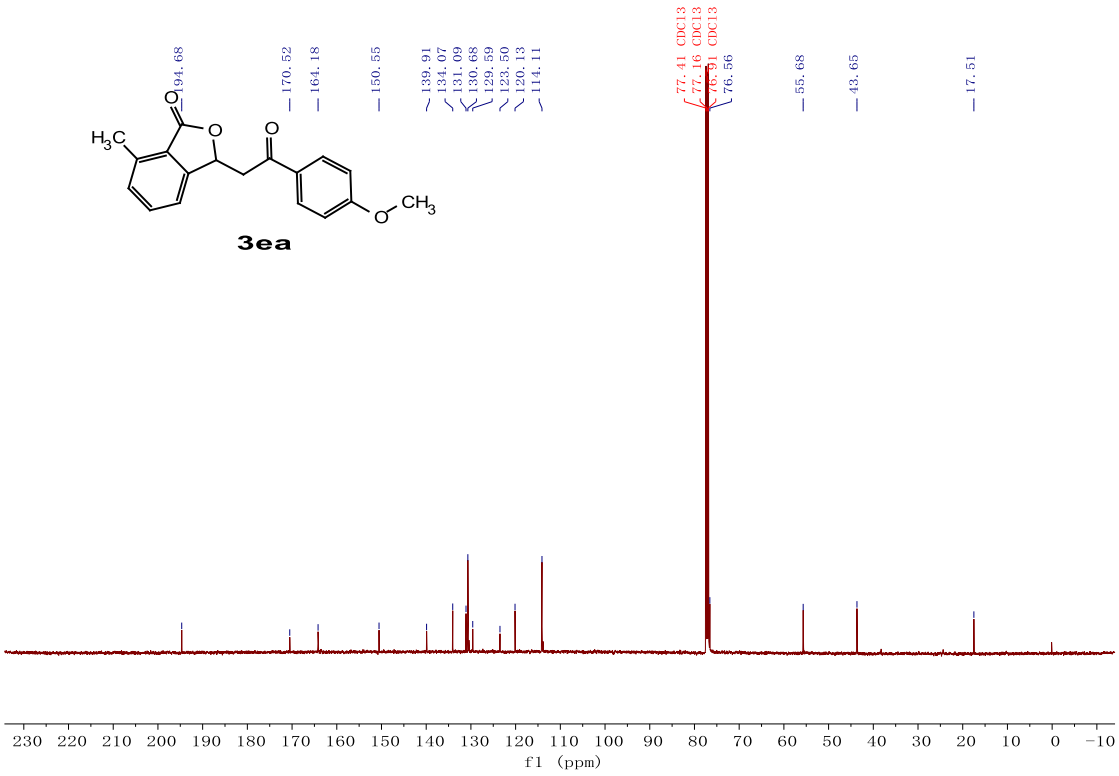
0.97
0.86
2.20
0.97
3.28
1.00
1.00
2.87
f1 (ppm)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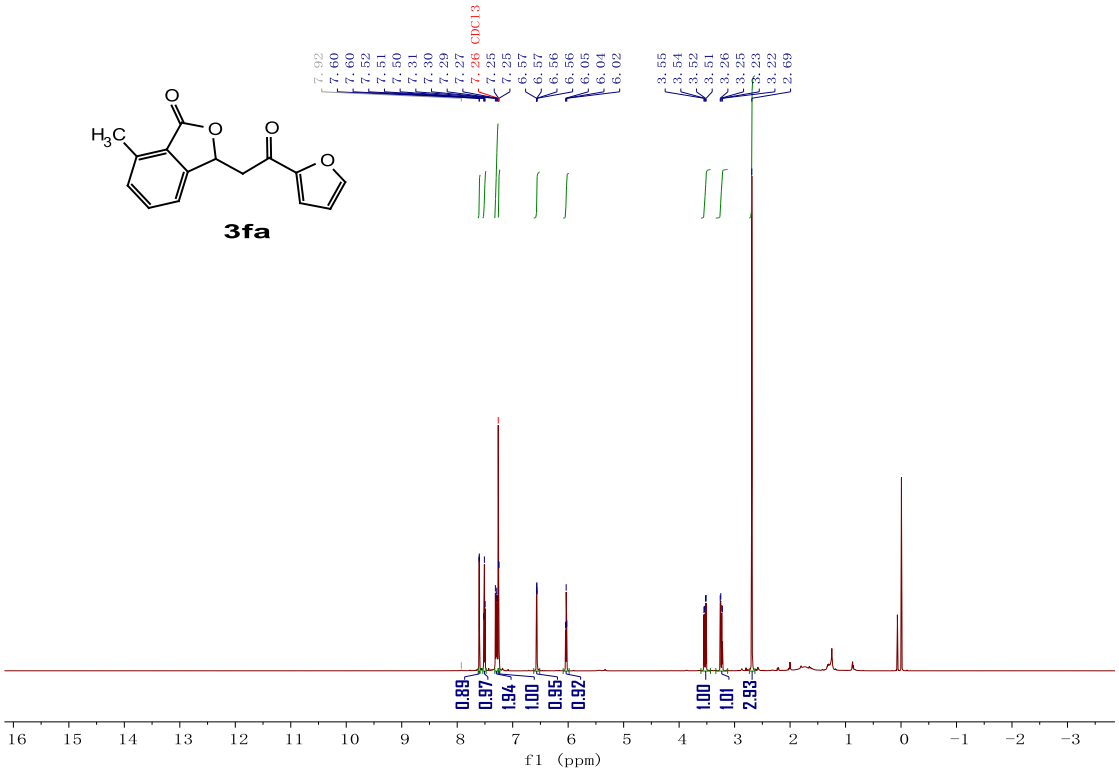
164.18
150.55
139.91
134.07
131.09
130.68
129.59
123.50
120.13
114.11
77.41 CDCl3
77.16 CDCl3
76.91 CDCl3
76.56
55.68
43.65
17.51
H3C
O
O
O
O
CH3
3ea
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)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7.52
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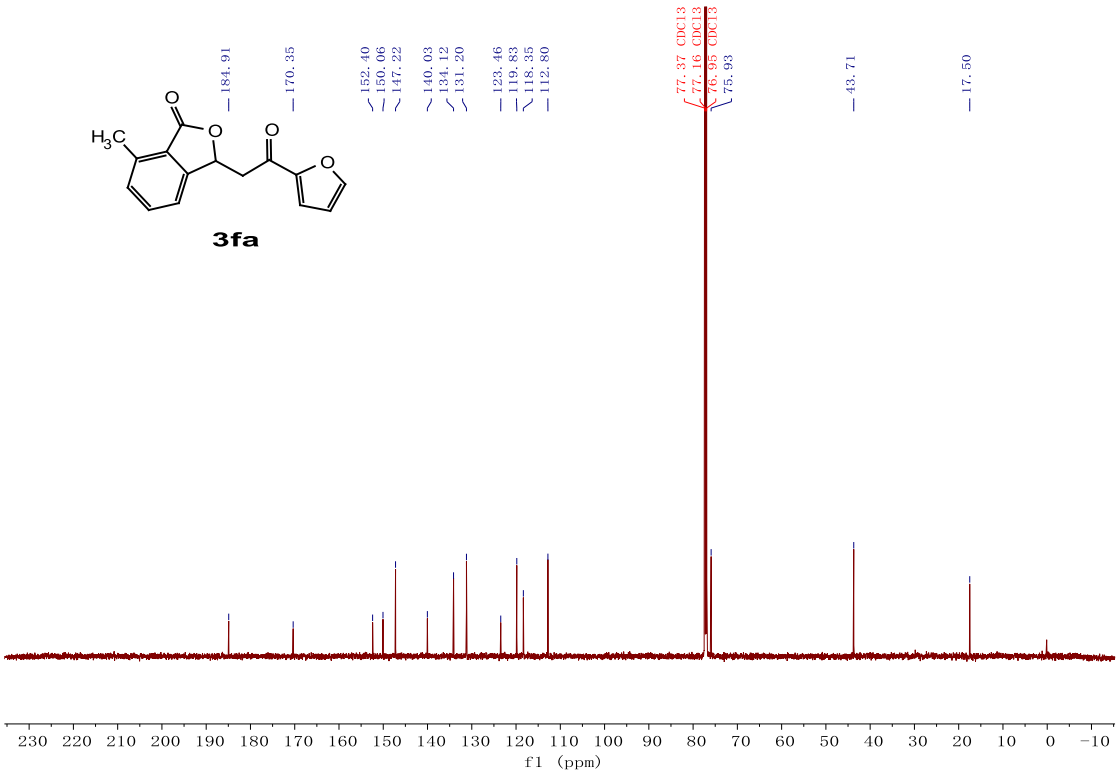
7.50
7.31
7.30
7.29
7.27
7.26 CDCl3
7.25
7.25
6.57
6.57
6.56
6.56
6.05
6.04
6.02
3.55
3.54
3.52
3.51
3.26
3.25
3.23
3.22
2.69
H3C
O
O
O
O
3fa
0.89
0.97
1.94
1.00
0.95
0.92
1.00
1.01
2.93
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3
f1 (ppm)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170.35
152.40
150.06
147.22
140.03
134.12
131.20
123.46
119.83
118.35
112.80
77.37 CDCl3
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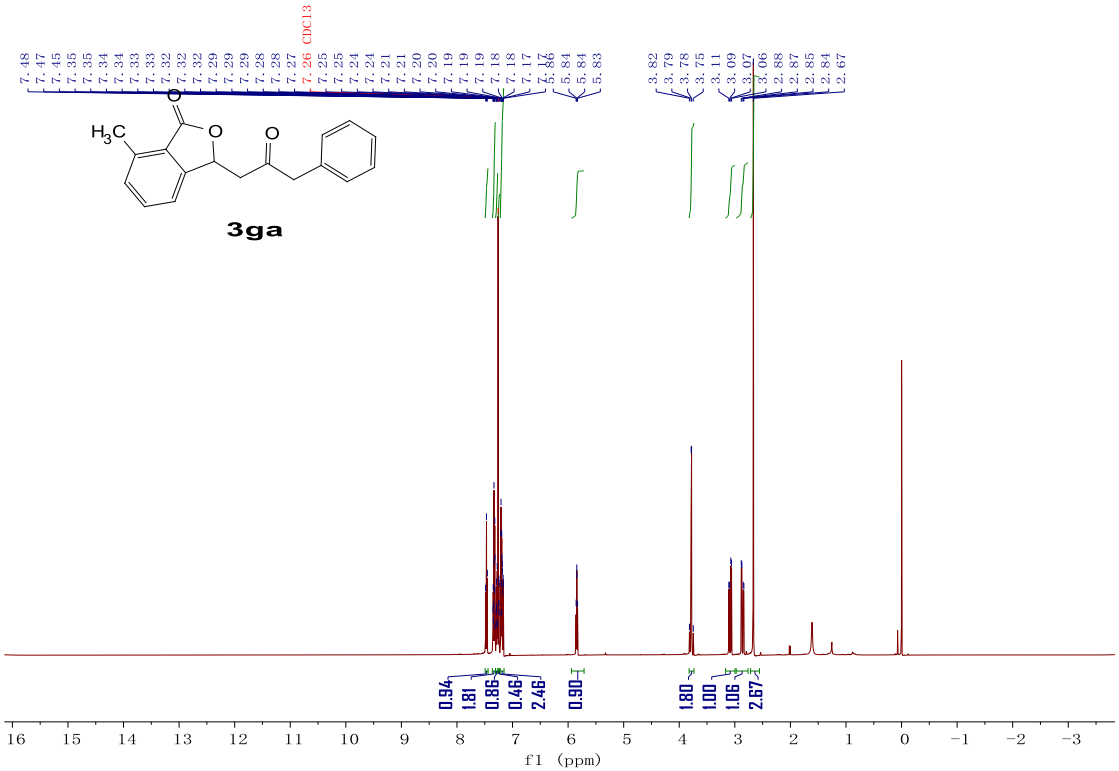
H3C
O
O
O
O
3fa
f1 (ppm)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7.32
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7.28
7.28
7.27
7.26 CDCl3
7.25
7.25
7.24
7.24
7.21
7.21
7.20
7.20
7.19
7.19
7.19
7.18
7.18
7.17
5.86
5.84
5.84
5.83
3.82
3.79
3.78
3.75
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2.85
2.84
2.67
H3C
O
O
O
3ga
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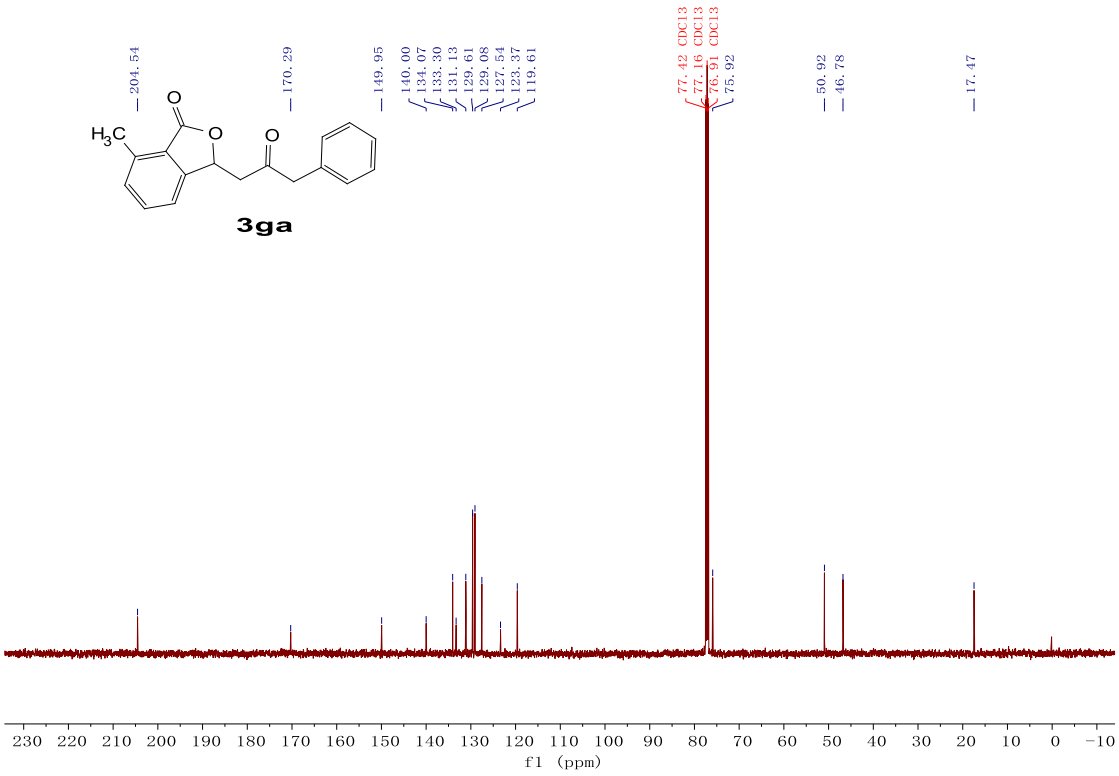
0.86
0.46
2.46
0.90
1.80
1.00
1.06
2.67
f1 (ppm)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140.00
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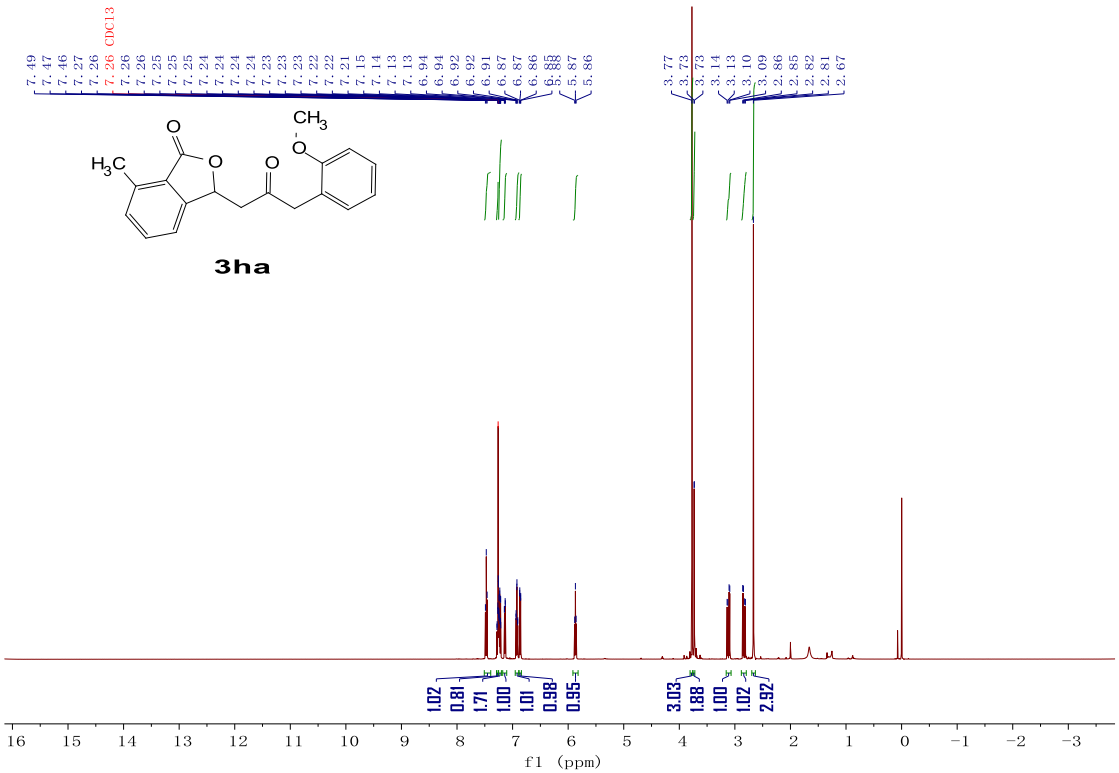
133.30
131.13
129.61
129.08
127.54
123.37
119.61
77.42 CDCl3
77.16 CDCl3
76.91 CDCl3
75.92
50.92
46.78
17.47
O
O
O
H3C
3ga
f1 (ppm)


7.49
7.47
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7.27
7.26 CDCl3
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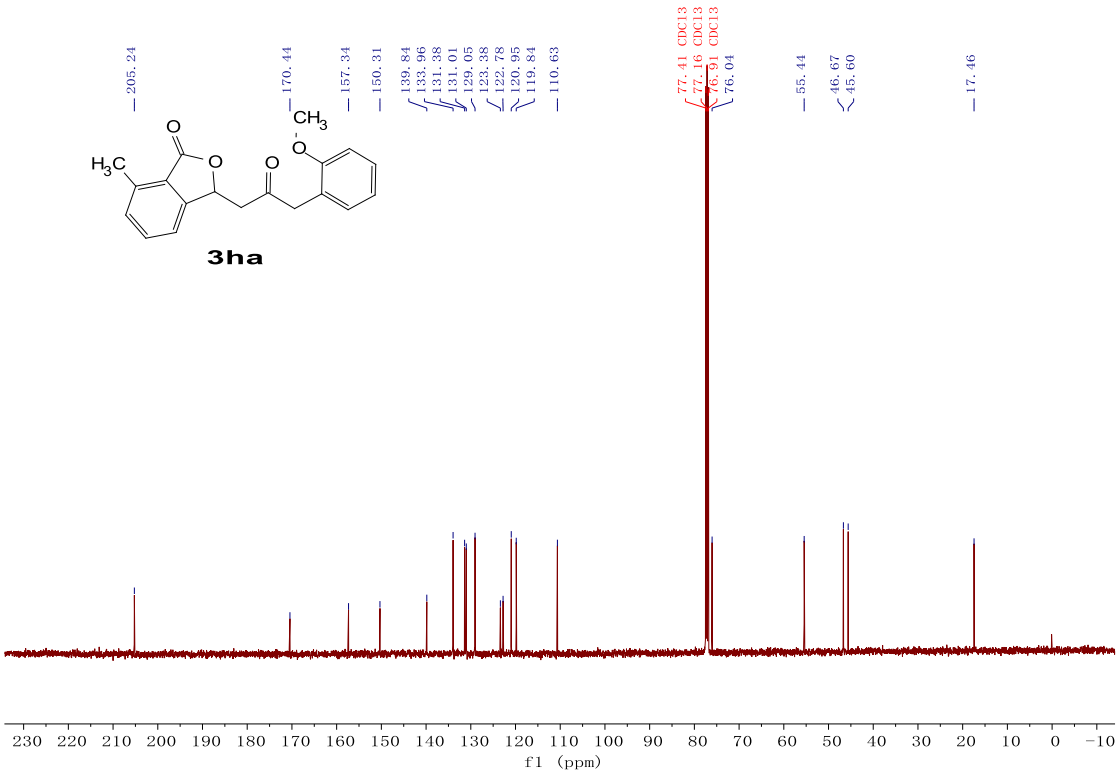
7.14
7.13
7.13
6.94
6.94
6.92
6.92
6.91
6.87
6.87
6.86
6.85
6.85
5.87
5.86
3.77
3.73
3.73
3.14
3.13
3.10
3.09
2.86
2.85
2.82
2.81
2.67
CH3
O
O
O
O
H3C
3ha
1.02
0.81
1.71
1.00
1.01
0.98
0.95
3.03
1.88
1.00
1.02
2.92
f1 (ppm)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170.44
157.34
150.31
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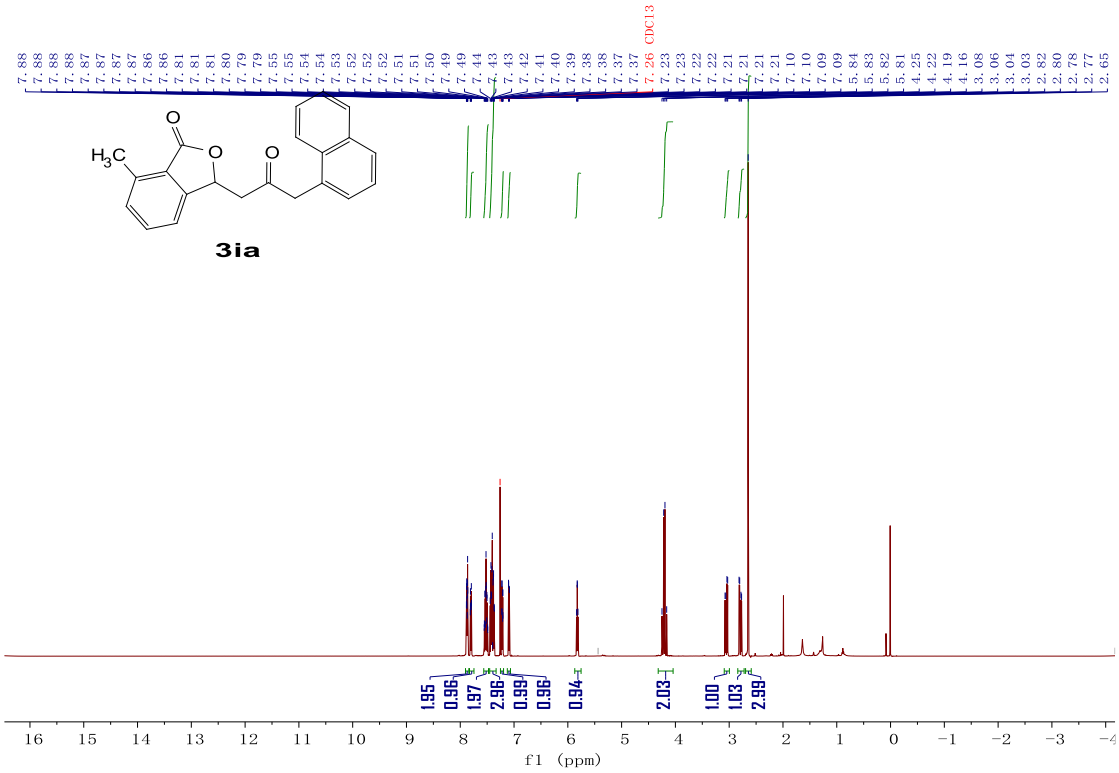
133.96
131.38
131.01
129.05
123.38
122.78
120.95
119.84
110.63
77.41 CDCl3
77.16 CDCl3
76.91 CDCl3
76.04
55.44
46.67
45.60
17.46
3ha
f1 (ppm)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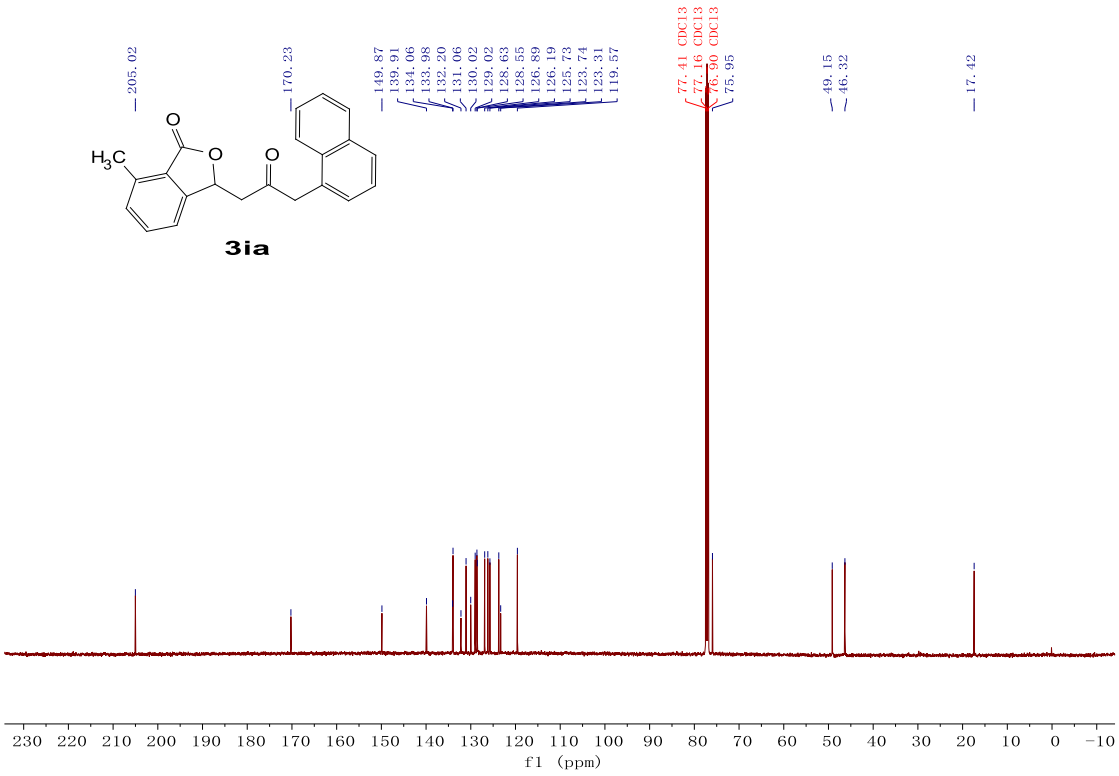
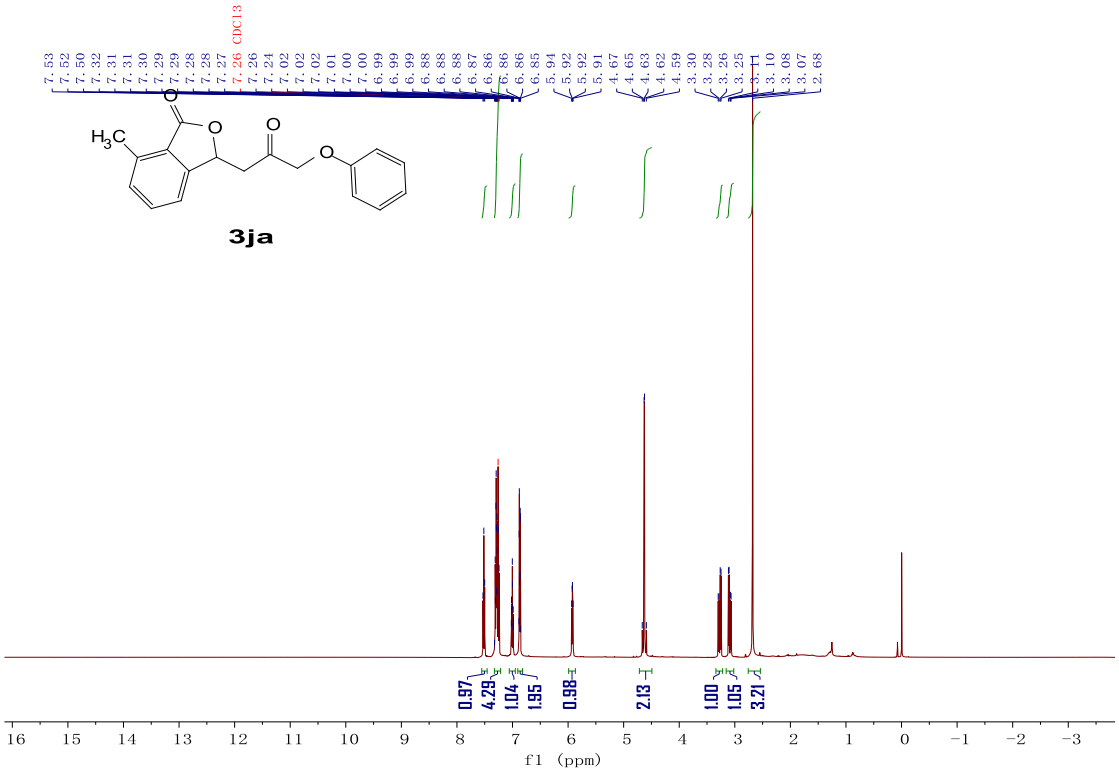
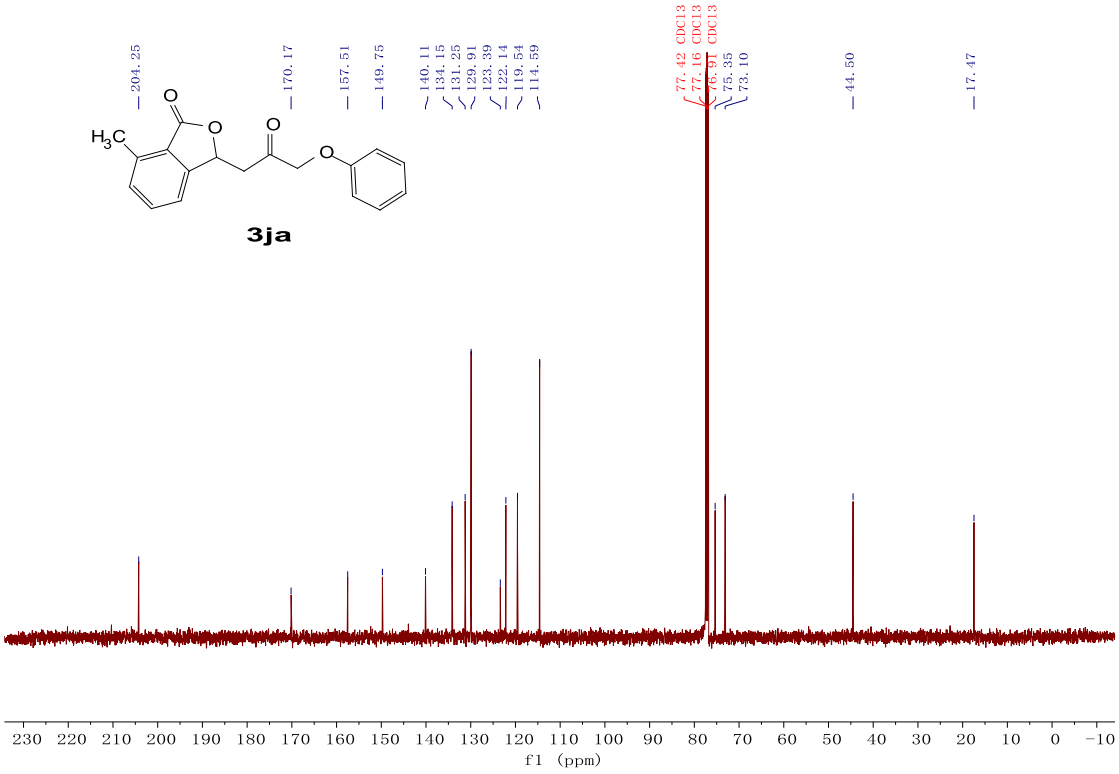
3ia
1.95
0.96
1.97
2.96
0.99
0.96
0.94
2.03
1.00
1.03
2.99
f1 (ppm)


205.02
170.23
149.87
139.91
134.06
133.98
132.20
131.06
130.02
129.02
128.63
128.55
126.89
126.19
125.73
123.74
123.31
119.57
77.41 CDCl3
77.16 CDCl3
76.90 CDCl3
75.95
49.15
46.32
17.42
O
O
O
H3C
3ia
f1 (ppm)


7.53
7.52
7.50
7.32
7.31
7.31
7.30
7.29
7.29
7.28
7.28
7.27
7.26 CDCl3
7.26
7.24
7.02
7.02
7.02
7.01
7.00
7.00
6.99
6.99
6.99
6.88
6.88
6.87
6.86
6.86
6.86
6.85
5.94
5.92
5.92
5.91
4.67
4.65
4.63
4.62
4.59
3.30
3.28
3.26
3.25
3.11
3.10
3.08
3.07
2.68
O
O
O
O
H3C
3ja
0.97
4.29
1.04
1.95
0.98
2.13
1.00
1.05
3.21
f1 (ppm)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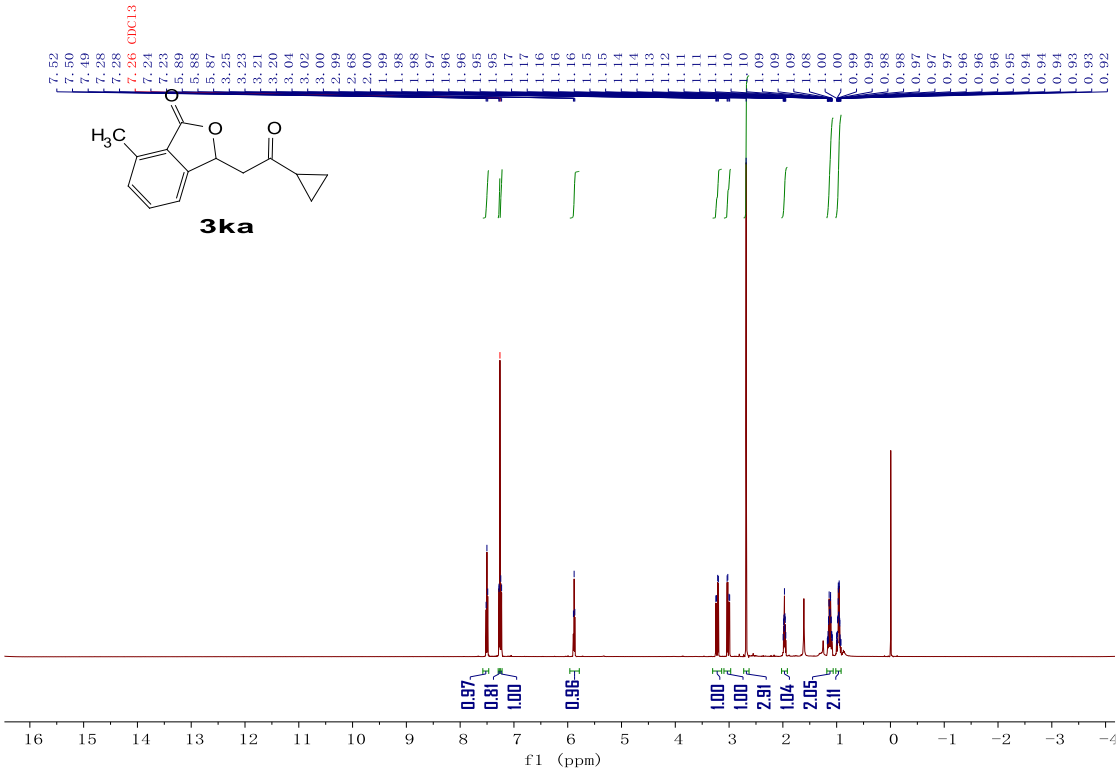
170.17
157.51
149.75
140.11
134.15
131.25
129.91
123.39
122.14
119.54
114.59
77.42 CDCl3
77.16 CDCl3
76.91 CDCl3
75.35
73.10
44.50
17.47
3ja
f1 (ppm)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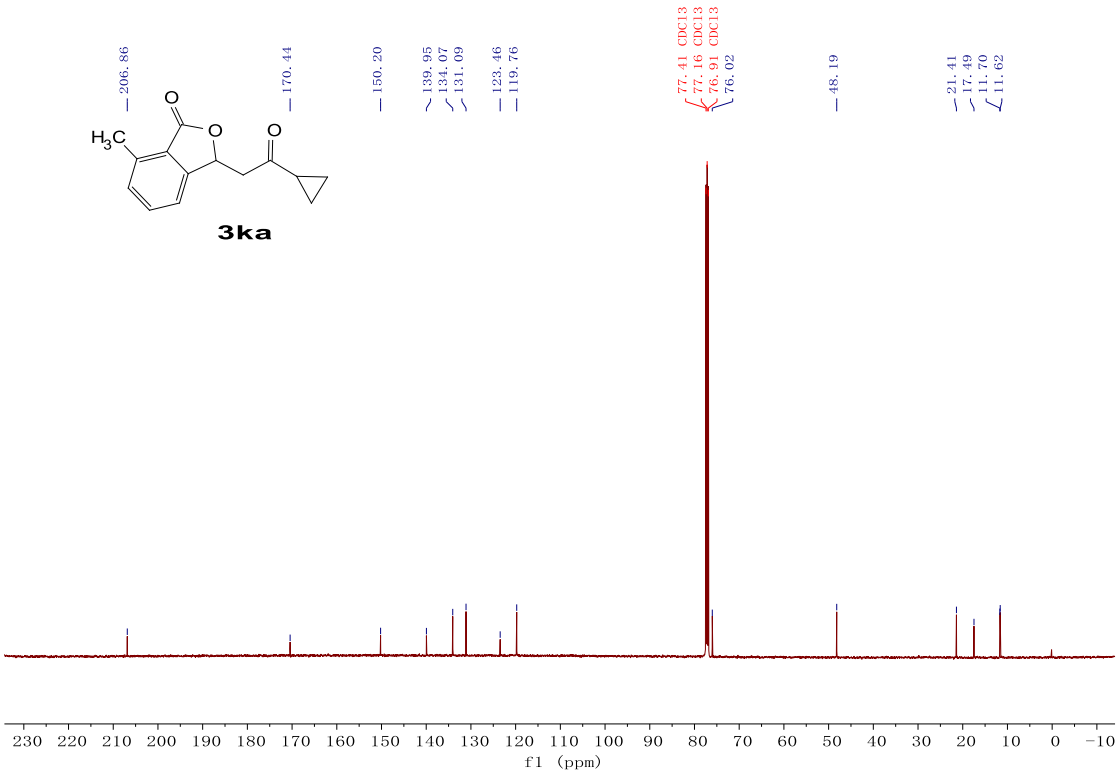
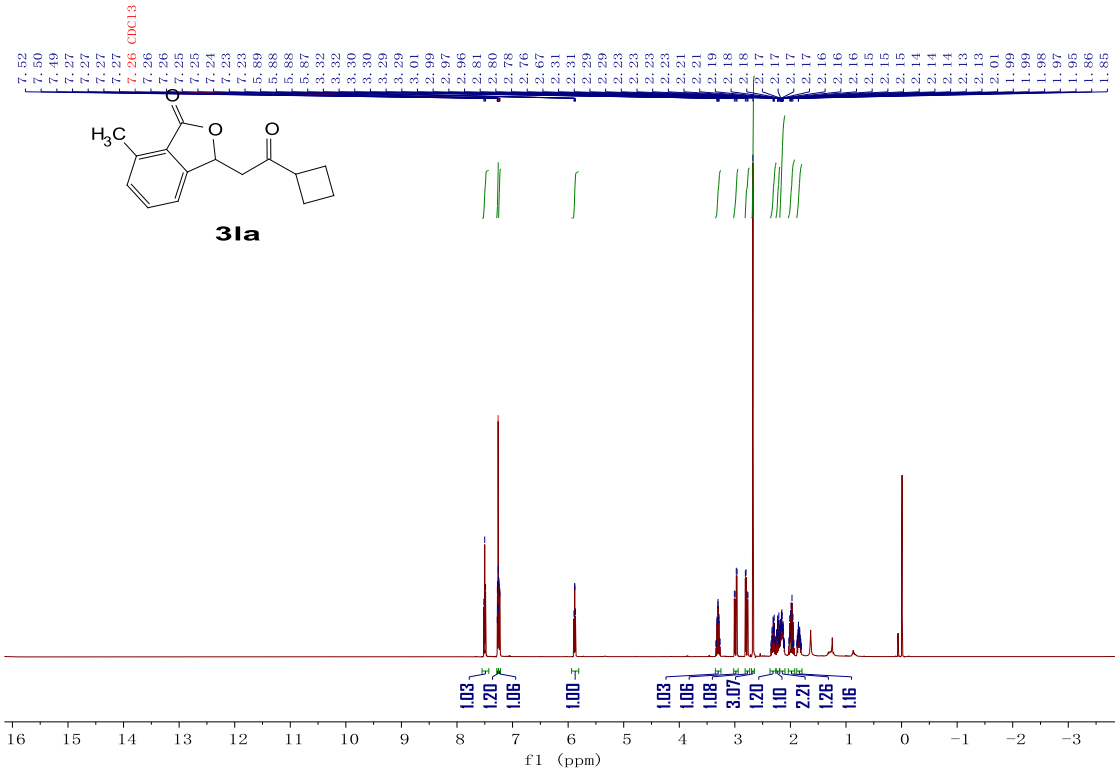
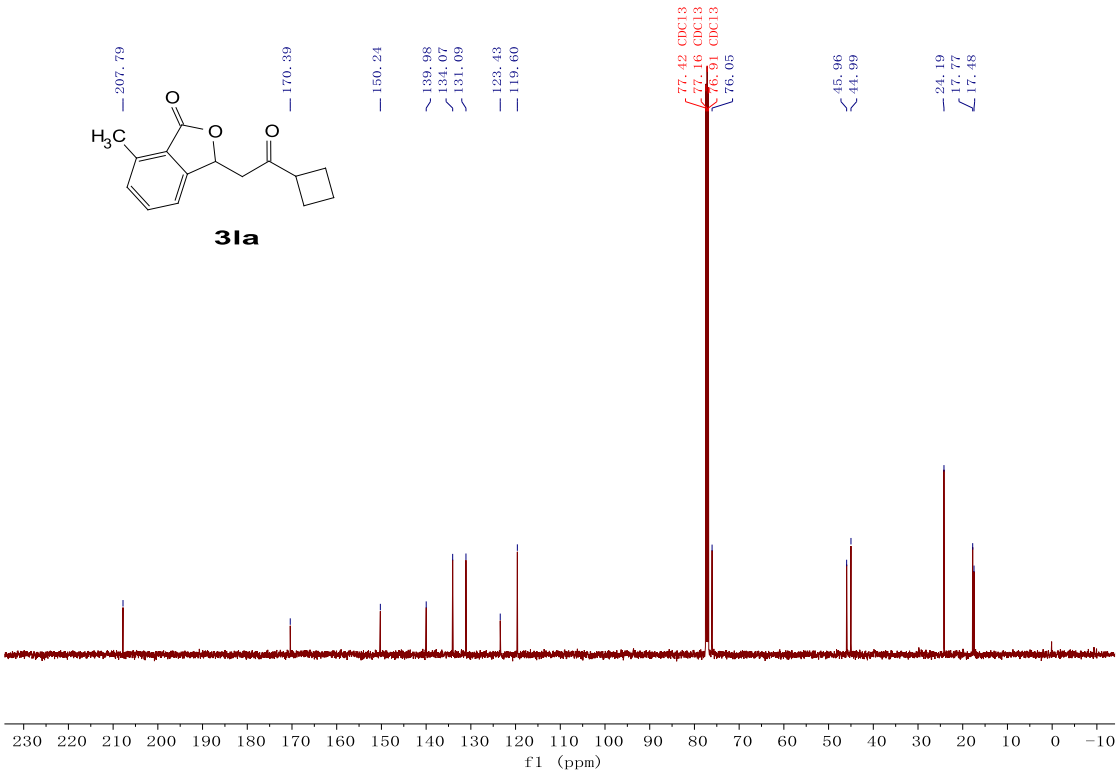
3ka
0.97
0.81
1.00
0.96
1.00
1.00
2.91
1.04
2.05
2.11
f1 (ppm)


206.86
170.44
150.20
139.95
134.07
131.09
123.46
119.76
77.41 CDCl3
77.16 CDCl3
76.91 CDCl3
76.02
48.19
21.41
17.49
11.70
11.62
3ka
f1 (ppm)


7.26 CDCl3
3la
1.03
1.20
1.06
1.00
1.03
1.06
1.08
3.07
1.20
1.10
2.21
1.26
1.16
f1 (ppm)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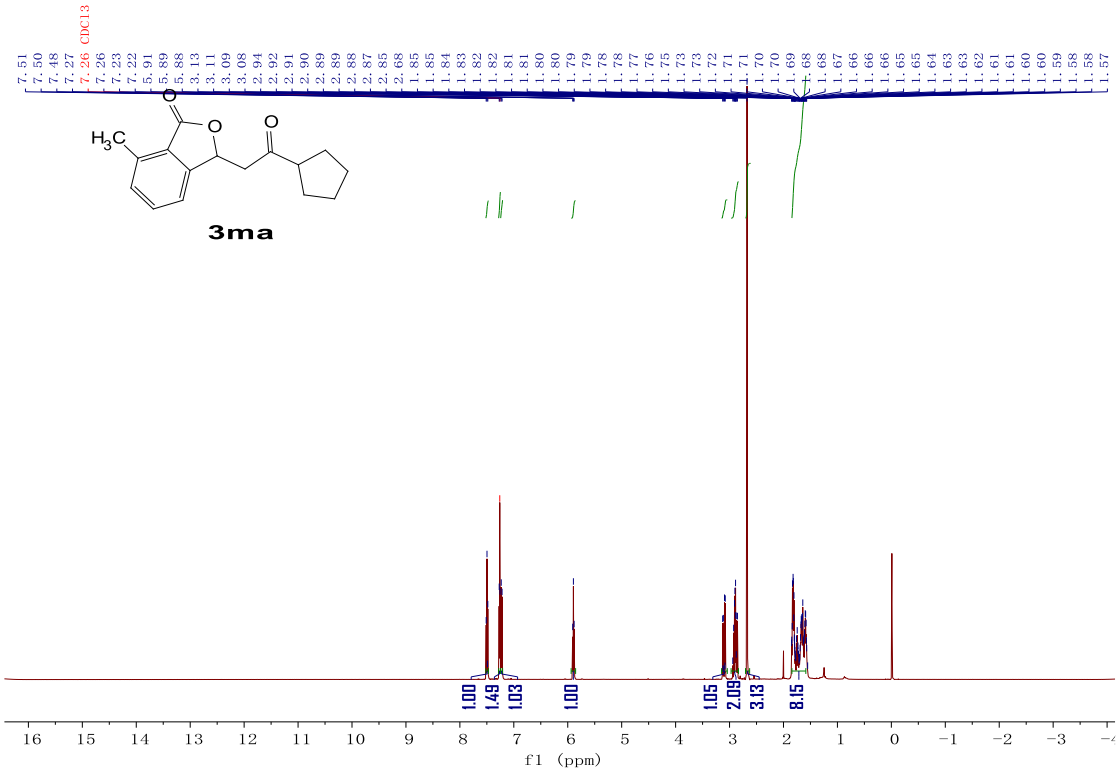
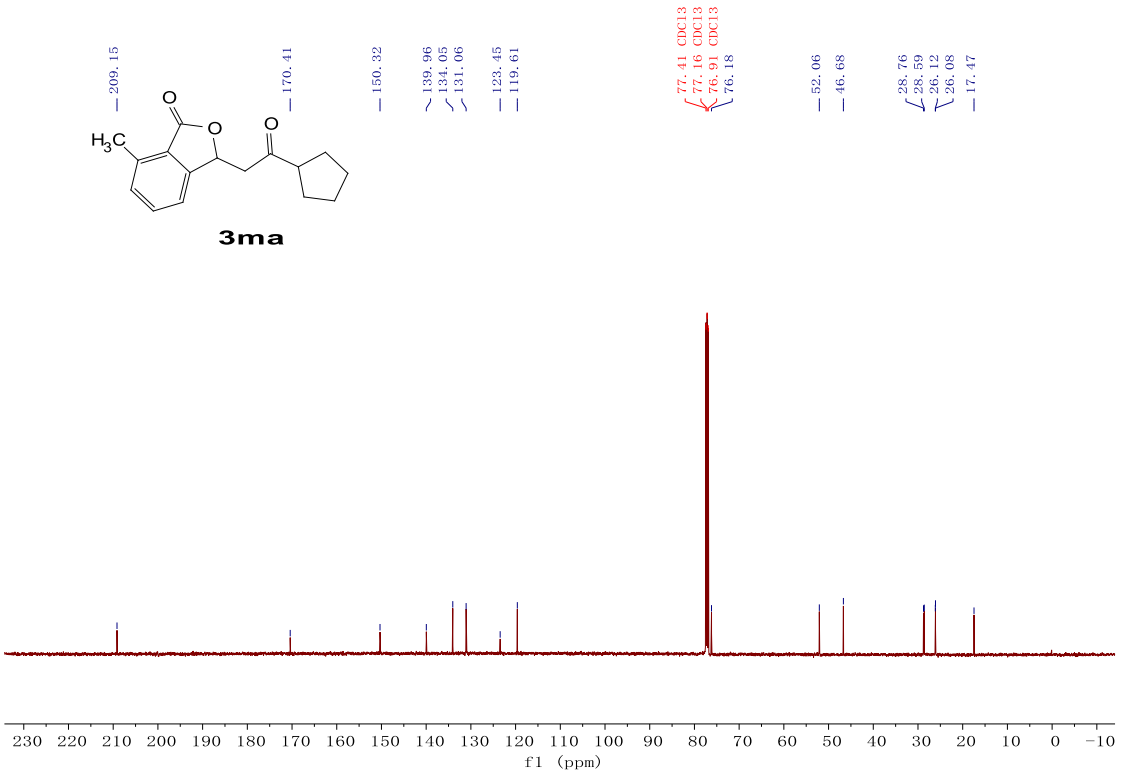
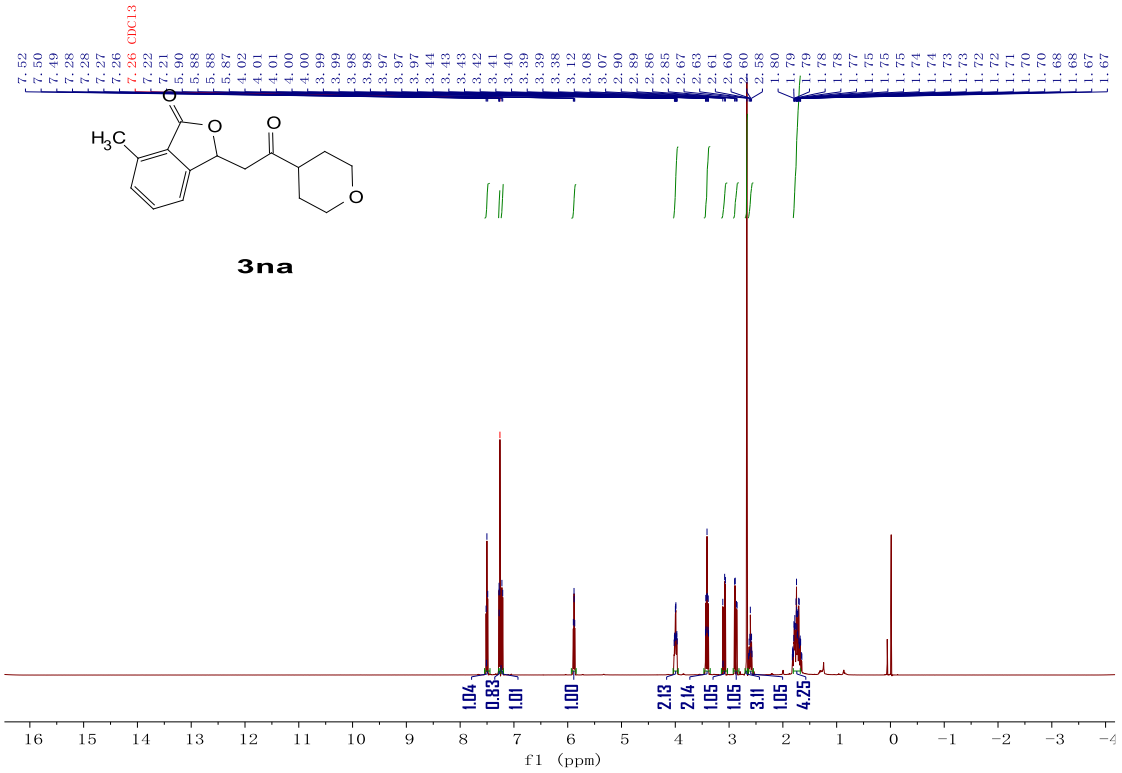
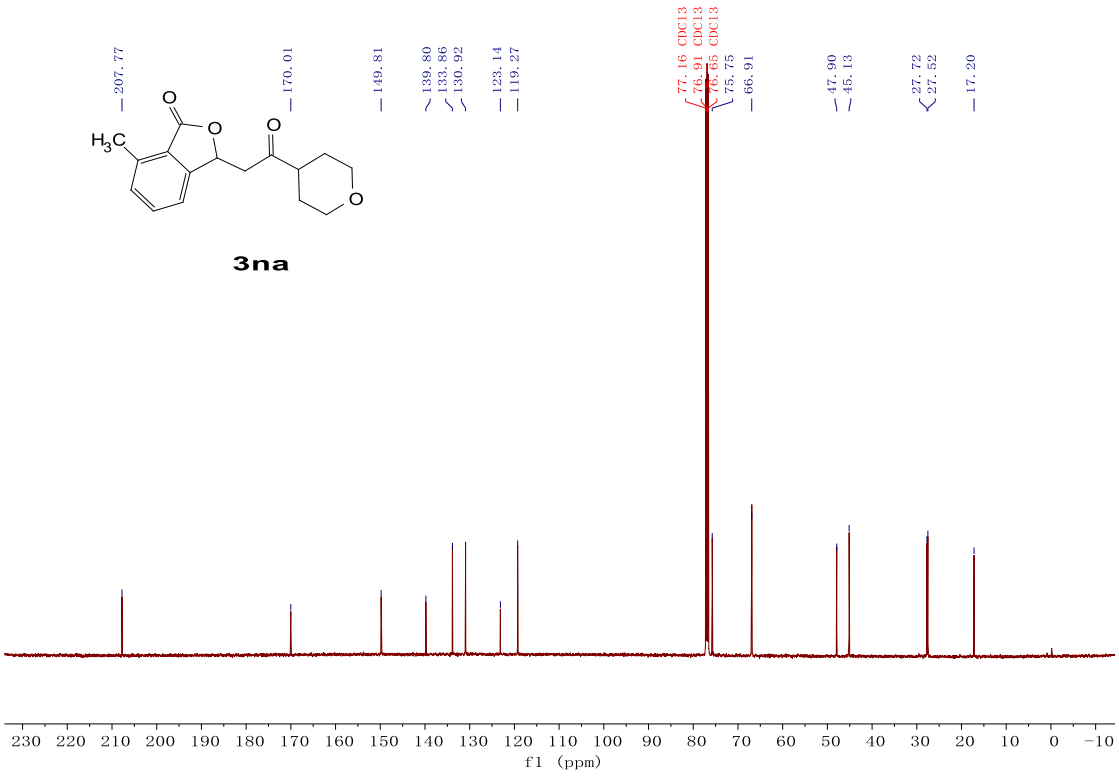
170.39
150.24
139.98
134.07
131.09
123.43
119.60
77.42 CDCl3
77.16 CDCl3
76.91 CDCl3
76.05
45.96
44.99
24.19
17.77
17.48
O
O
O
H3C
3la
f1 (ppm)


7.26 CDCl3
O
O
O
H3C
3ma
1.00
1.49
1.03
1.00
1.05
2.09
3.13
8.15
f1 (ppm)


209.15
170.41
150.32
139.96
134.05
131.06
123.45
119.61
77.41 CDCl3
77.16 CDCl3
76.91 CDCl3
76.18
52.06
46.68
28.76
28.59
26.12
26.08
17.47
O
O
O
H3C
3ma
f1 (ppm)


7.52
7.50
7.49
7.28
7.27
7.26 CDCl3
7.22
7.21
5.90
5.88
5.88
5.87
4.02
4.01
4.01
4.00
4.00
3.99
3.99
3.98
3.98
3.97
3.97
3.97
3.44
3.43
3.43
3.42
3.41
3.40
3.39
3.39
3.38
3.12
3.08
3.07
2.90
2.89
2.86
2.85
2.67
2.63
2.61
2.60
2.60
2.58
1.80
1.79
1.79
1.78
1.78
1.77
1.75
1.75
1.75
1.74
1.74
1.73
1.73
1.72
1.72
1.71
1.70
1.70
1.68
1.68
1.67
1.67
O
O
O
O
H3C
3na
1.04
0.83
1.01
1.00
2.13
2.14
1.05
1.05
3.11
1.05
4.25
f1 (ppm)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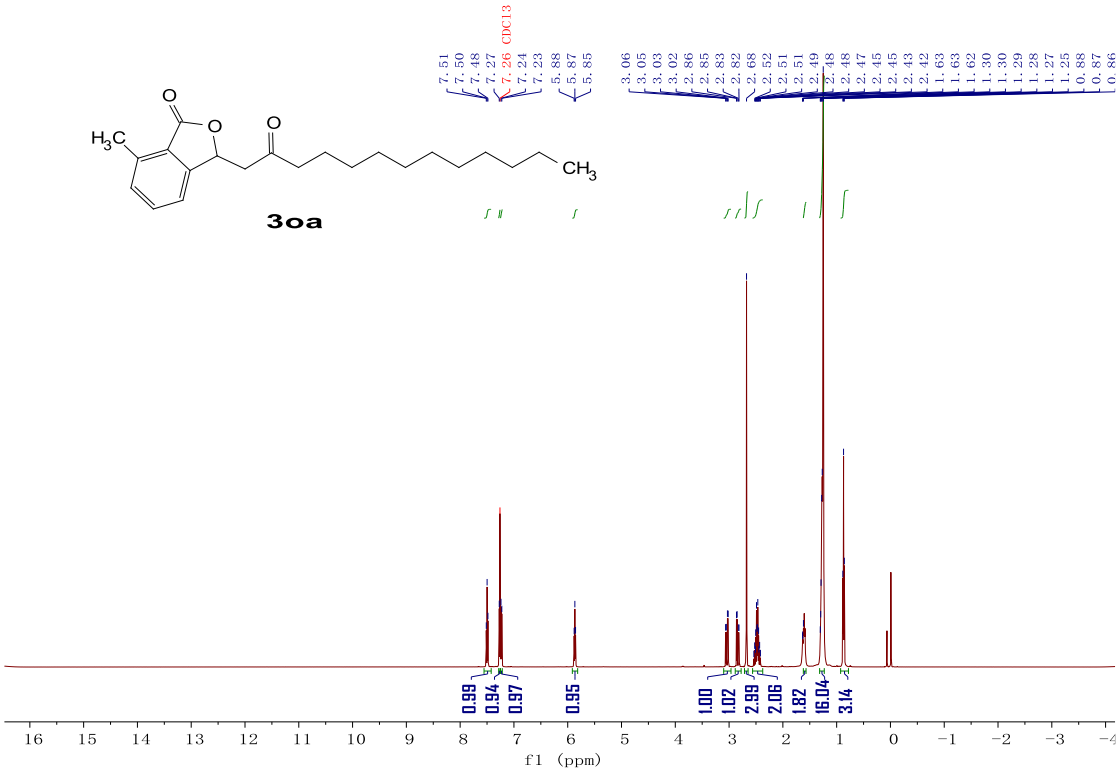
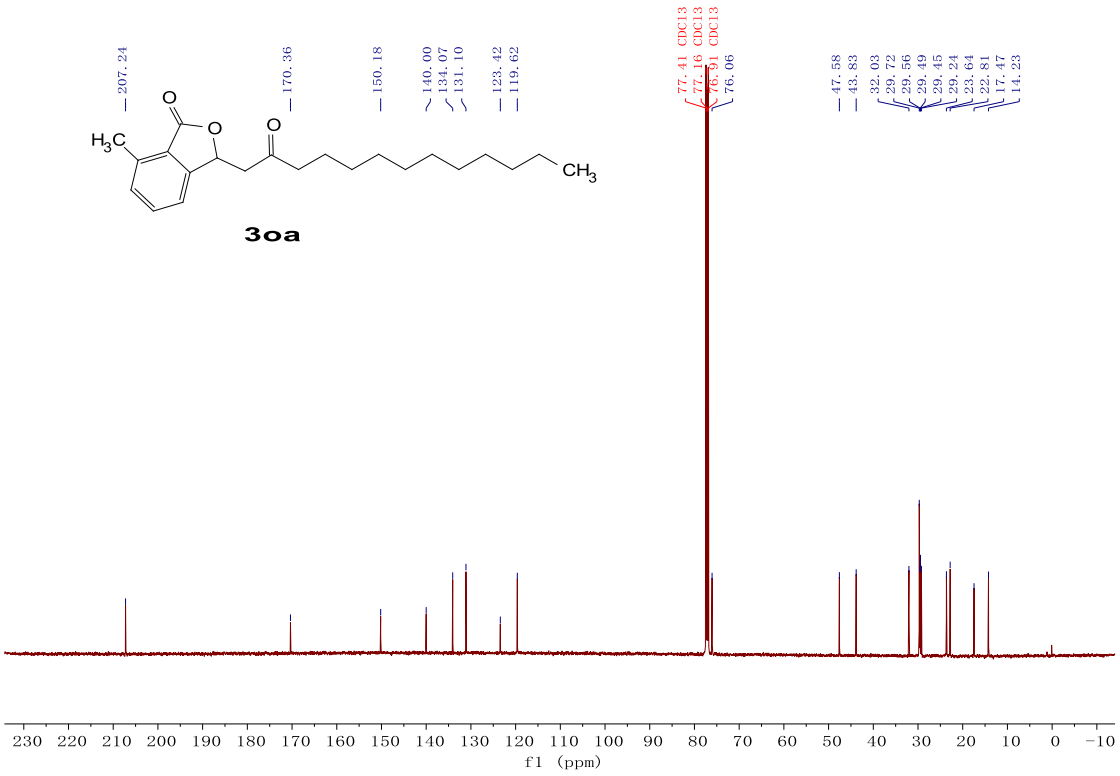
170.01
149.81
139.80
133.86
130.92
123.14
119.27
77.16 CDCl3
76.91 CDCl3
76.65 CDCl3
75.75
66.91
47.90
45.13
27.72
27.52
17.20
3na
f1 (ppm)


7.51
7.50
7.48
7.27
7.26 CDCl3
7.24
7.23
5.88
5.87
5.85
3.06
3.05
3.03
3.02
2.86
2.85
2.83
2.82
2.68
2.52
2.51
2.49
2.48
2.48
2.47
2.45
2.45
2.43
2.42
1.63
1.63
1.62
1.30
1.30
1.29
1.28
1.27
1.25
0.88
0.87
0.86
CH3
3oa
0.99
0.94
0.97
0.95
1.00
1.02
2.99
2.06
1.82
16.04
3.14
f1 (ppm)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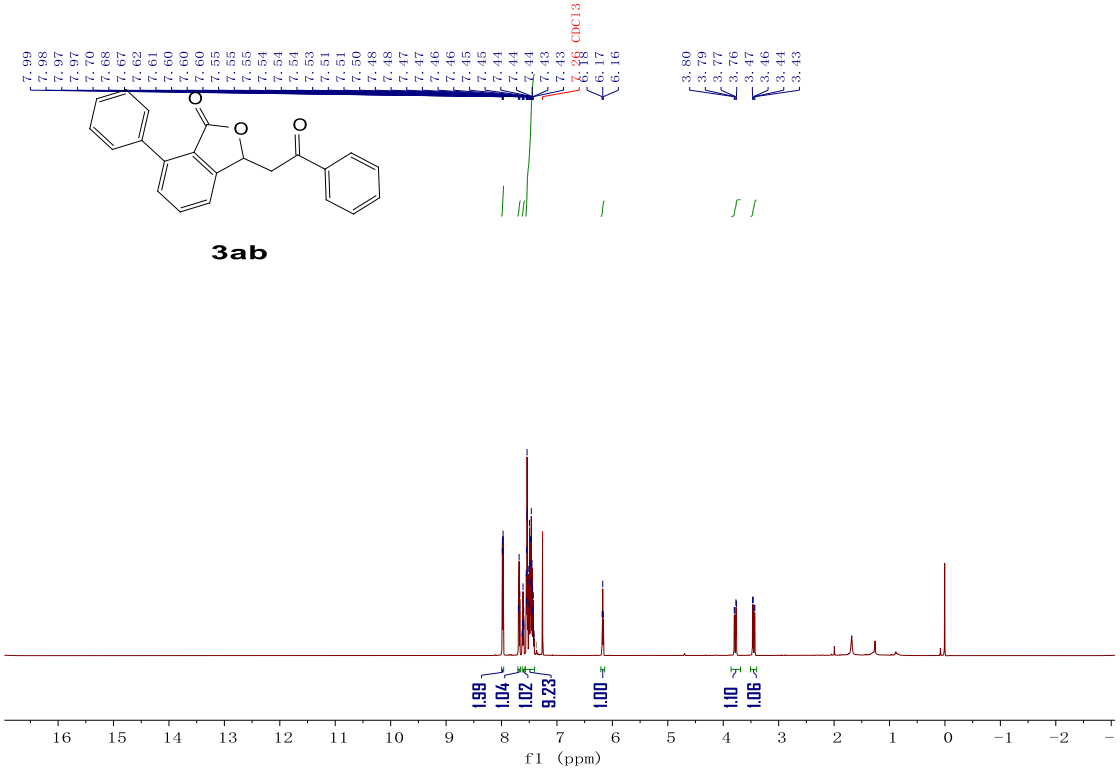
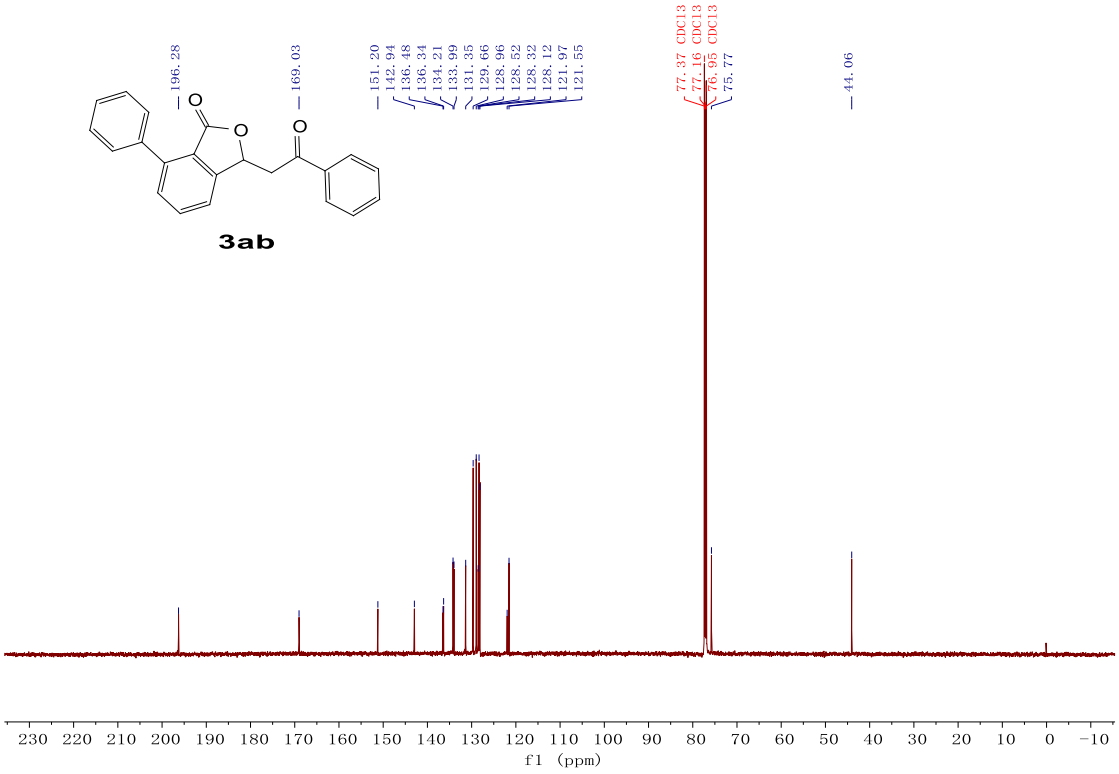
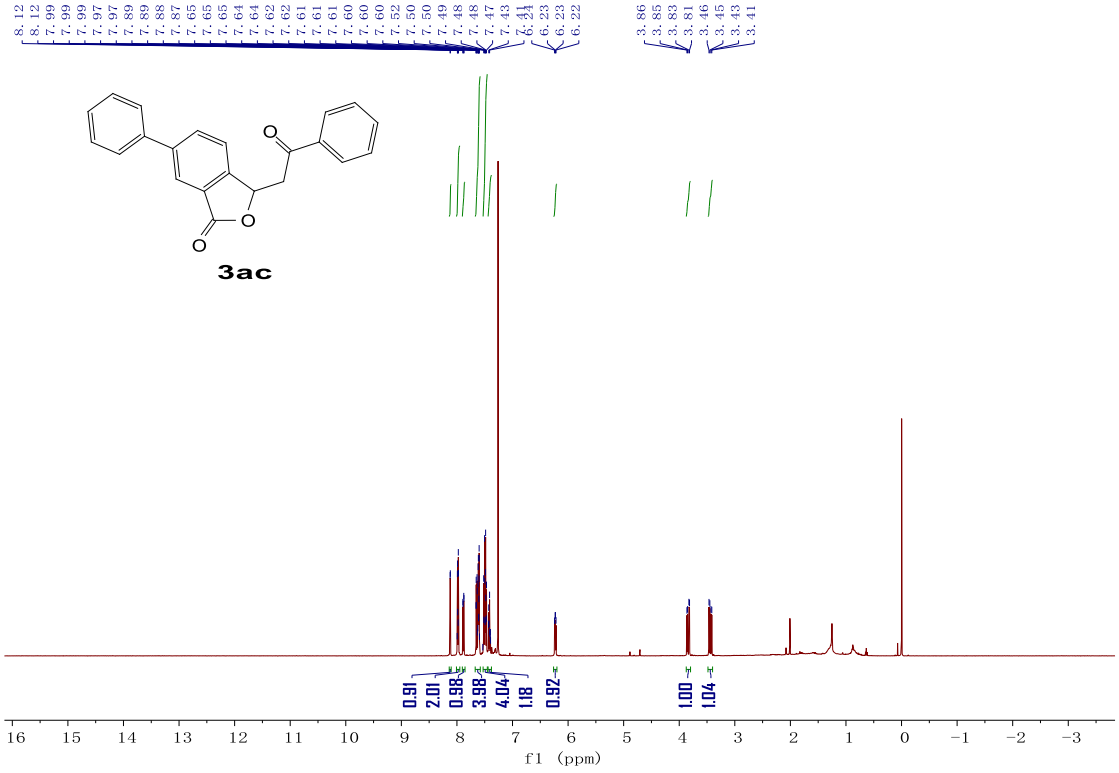
170.36
150.18
140.00
134.07
131.10
123.42
119.62
77.41 CDCl3
77.16 CDCl3
76.91 CDCl3
76.06
47.58
43.83
32.03
29.72
29.56
29.49
29.45
29.24
23.64
22.81
17.47
14.23
O
O
O
H3C
CH3
f1 (ppm)

3oa


7.99
7.98
7.97
7.97
7.70
7.68
7.67
7.62
7.61
7.60
7.60
7.60
7.55
7.55
7.54
7.54
7.54
7.53
7.51
7.51
7.50
7.48
7.48
7.47
7.47
7.46
7.46
7.45
7.45
7.44
7.44
7.44
7.43
7.43
7.26 CDCl3
6.18
6.17
6.16
3.80
3.79
3.77
3.76
3.47
3.46
3.44
3.43
1.99
1.04
1.02
9.23
1.00
1.10
1.06
f1 (ppm)

3ab


196.28
169.03
151.20
142.94
136.48
136.34
134.21
133.99
131.35
129.66
128.96
128.52
128.32
128.12
121.97
121.55
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
75.77
44.06
3ab
f1 (ppm)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8.12
7.99
7.99
7.99
7.97
7.97
7.89
7.89
7.88
7.87
7.65
7.65
7.65
7.64
7.64
7.62
7.62
7.61
7.61
7.61
7.60
7.60
7.60
7.52
7.50
7.50
7.49
7.48
7.48
7.47
7.43
6.24
6.23
6.23
6.22
3.86
3.85
3.83
3.81
3.46
3.45
3.43
3.41
3ac
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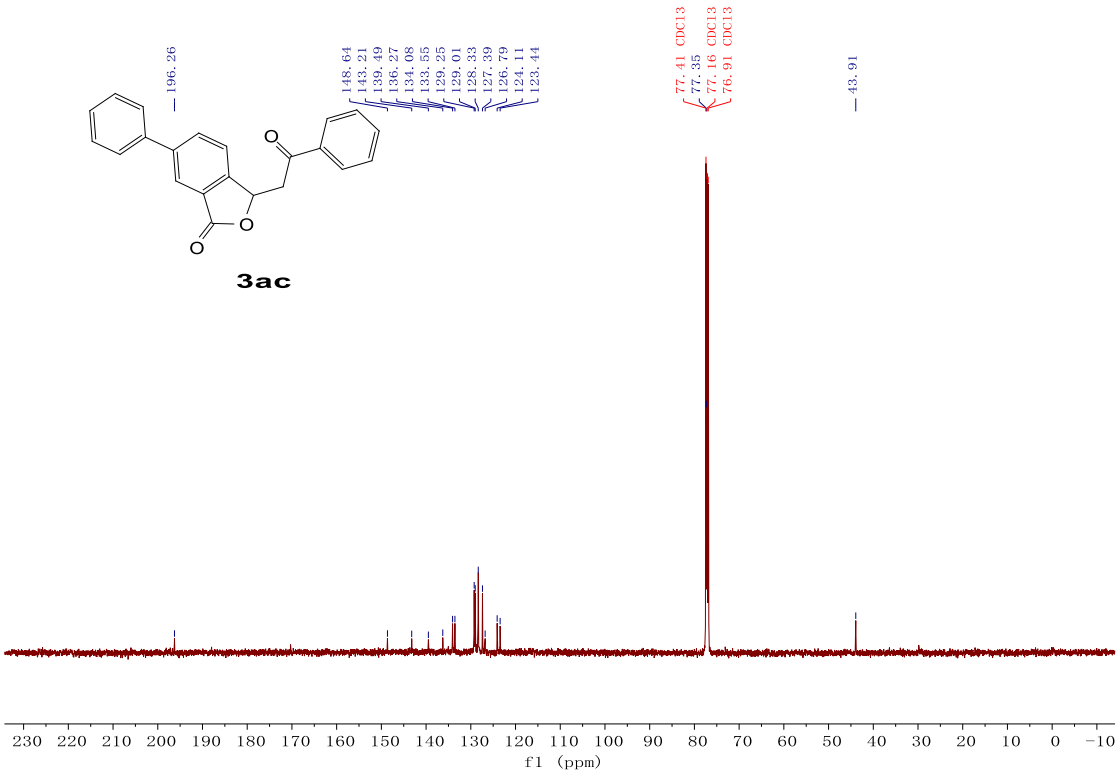
2.01
0.98
3.98
4.04
1.18
0.92
1.00
1.04
f1 (ppm)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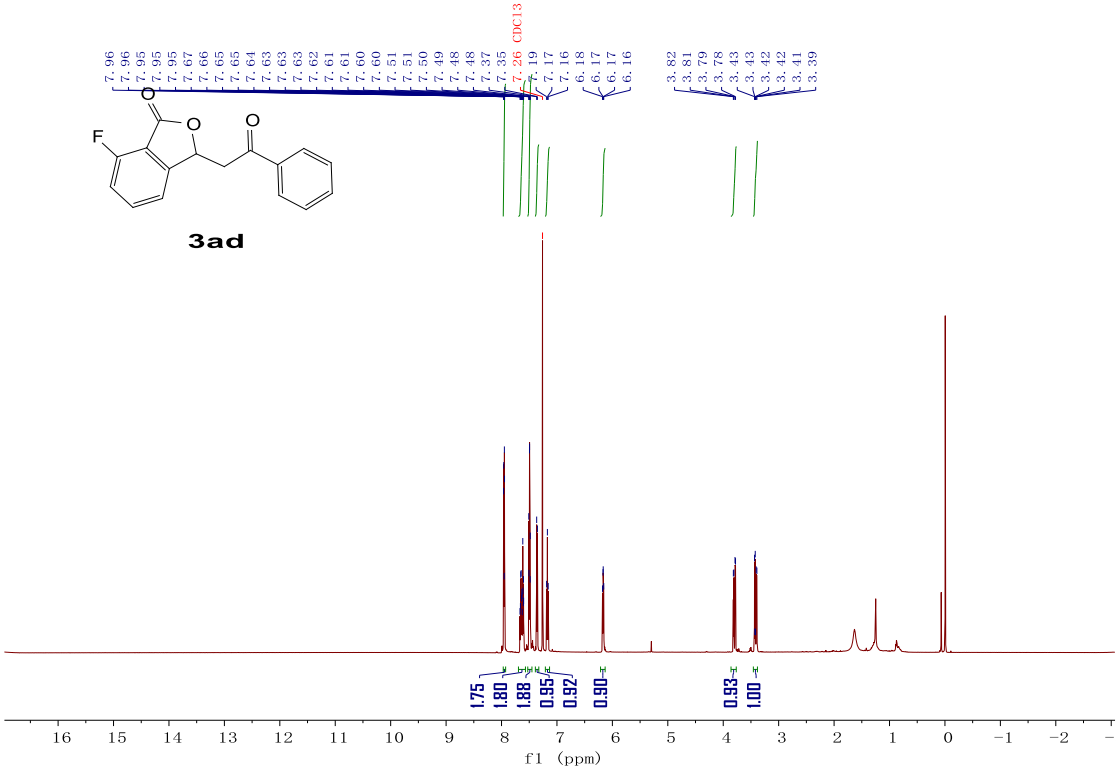
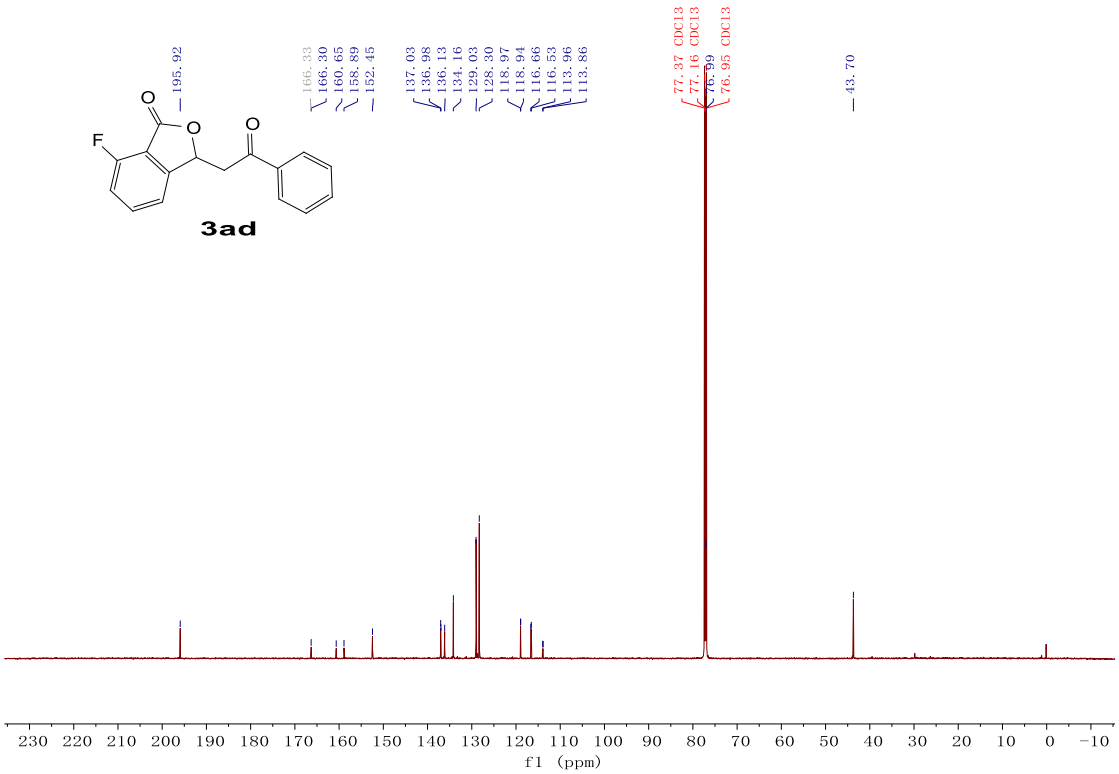
148.64
143.21
139.49
136.27
134.08
133.55
129.25
129.01
128.33
127.39
126.79
124.11
123.44
77.41 CDCl3
77.35
77.16 CDCl3
76.91 CDCl3
43.91
O
O
O
3ac
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)


7.96
7.96
7.95
7.95
7.95
7.67
7.66
7.65
7.65
7.64
7.63
7.63
7.63
7.62
7.61
7.61
7.60
7.60
7.51
7.51
7.50
7.49
7.48
7.48
7.37
7.35
7.26 CDCl3
7.19
7.17
7.16
6.18
6.17
6.17
6.16
3.82
3.81
3.79
3.78
3.43
3.43
3.42
3.42
3.41
3.39
O
F
O
O
3ad
1.75
1.80
1.88
0.95
0.92
0.90
0.93
1.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −
f1 (ppm)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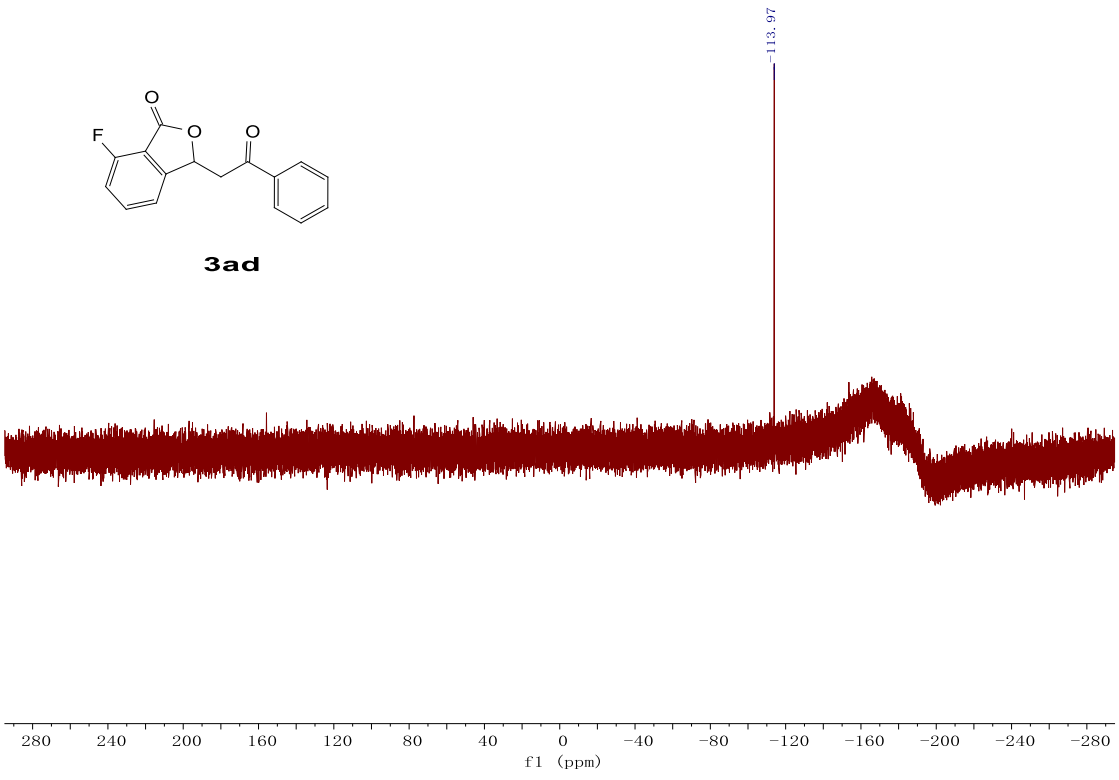
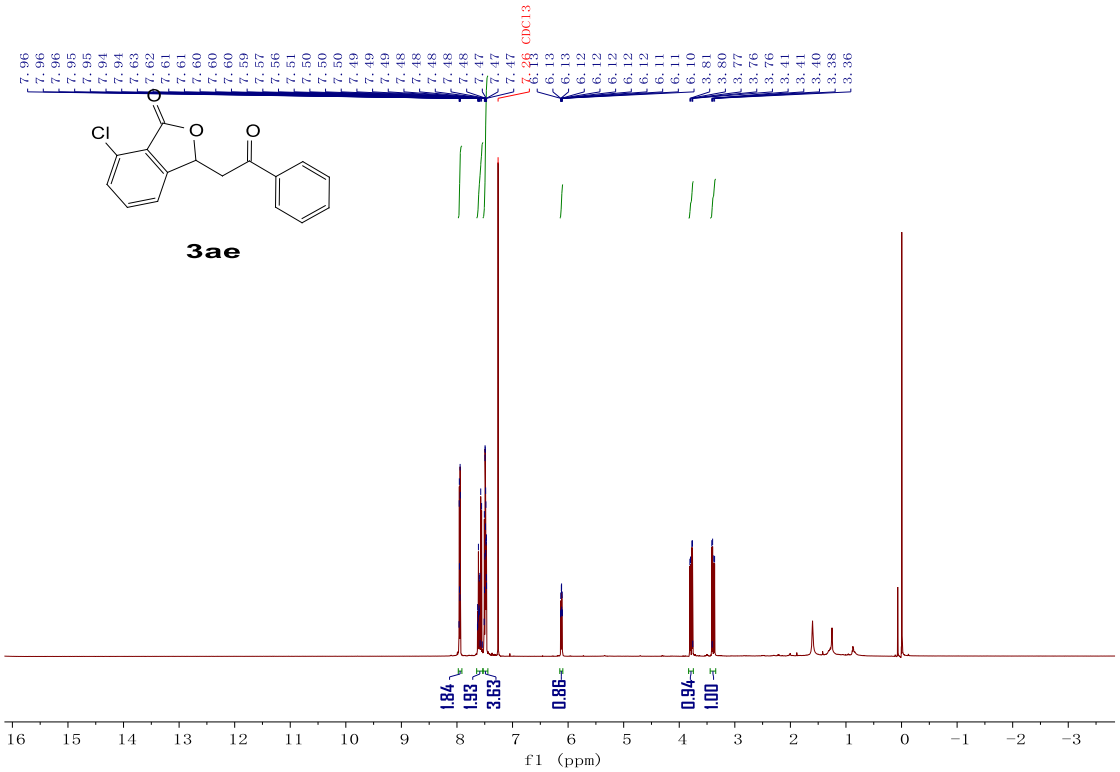
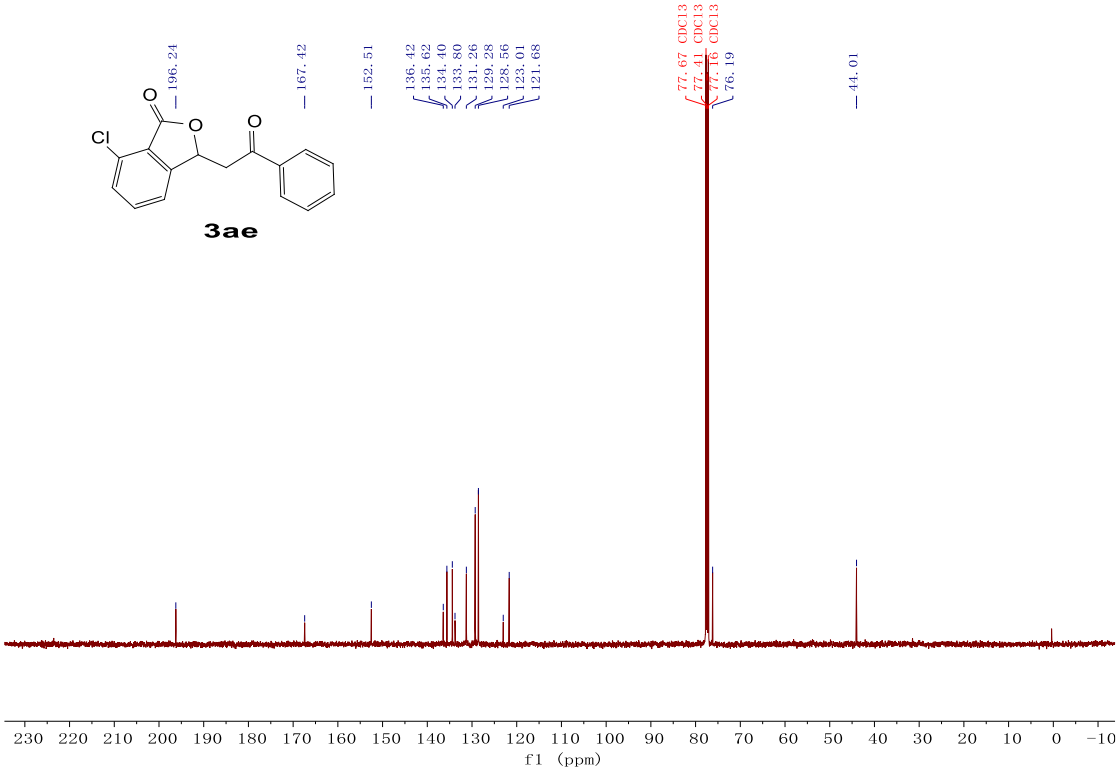
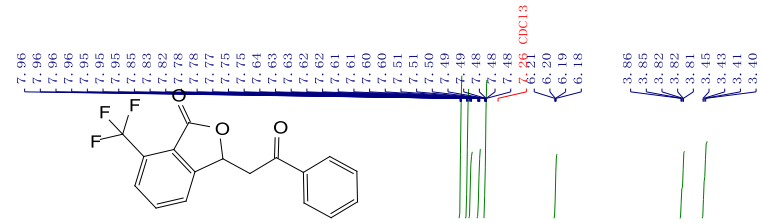
166.33
166.30
160.65
158.89
152.45
137.03
136.98
136.13
134.16
129.03
128.30
118.97
118.94
116.66
116.53
113.96
113.86
77.37 CDCl3
77.16 CDCl3
76.99
76.95 CDCl3
43.70
F
O
O
O
3ad
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)


−113.97
F
O
O
O
3ad
280 240 200 160 120 80 40 0 −40 −80 −120 −160 −200 −240 −280
f1 (ppm)


7.96 7.96 7.96 7.95 7.95 7.94 7.94 7.63 7.62 7.61 7.61 7.60 7.60 7.60 7.59 7.57 7.56 7.51 7.50 7.50 7.50 7.49 7.49 7.49 7.48 7.48 7.48 7.48 7.48 7.47 7.47 7.47 7.26 CDCl3 6.13 6.13 6.12 6.12 6.12 6.12 6.12 6.12 6.11 6.11 6.10 3.81 3.80 3.77 3.76 3.76 3.41 3.40 3.38 3.36
Cl
O
O
O
3ae
1.84
1.93
3.63
0.86
0.94
1.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3
f1 (ppm)


196.24
167.42
152.51
136.42
135.62
134.40
133.80
131.26
129.28
128.56
123.01
121.68
77.67 CDCl3
77.41 CDCl3
77.16 CDCl3
76.19
44.01
Cl
O
O
O
3ae
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)


7.96
7.96
7.96
7.96
7.95
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7.95
7.85
7.83
7.82
7.78
7.78
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7.75
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7.63
7.63
7.62
7.62
7.61
7.61
7.60
7.60
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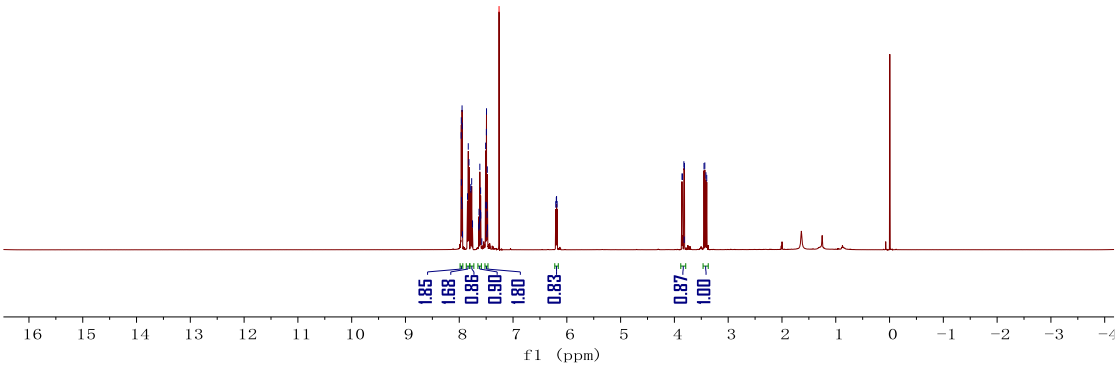
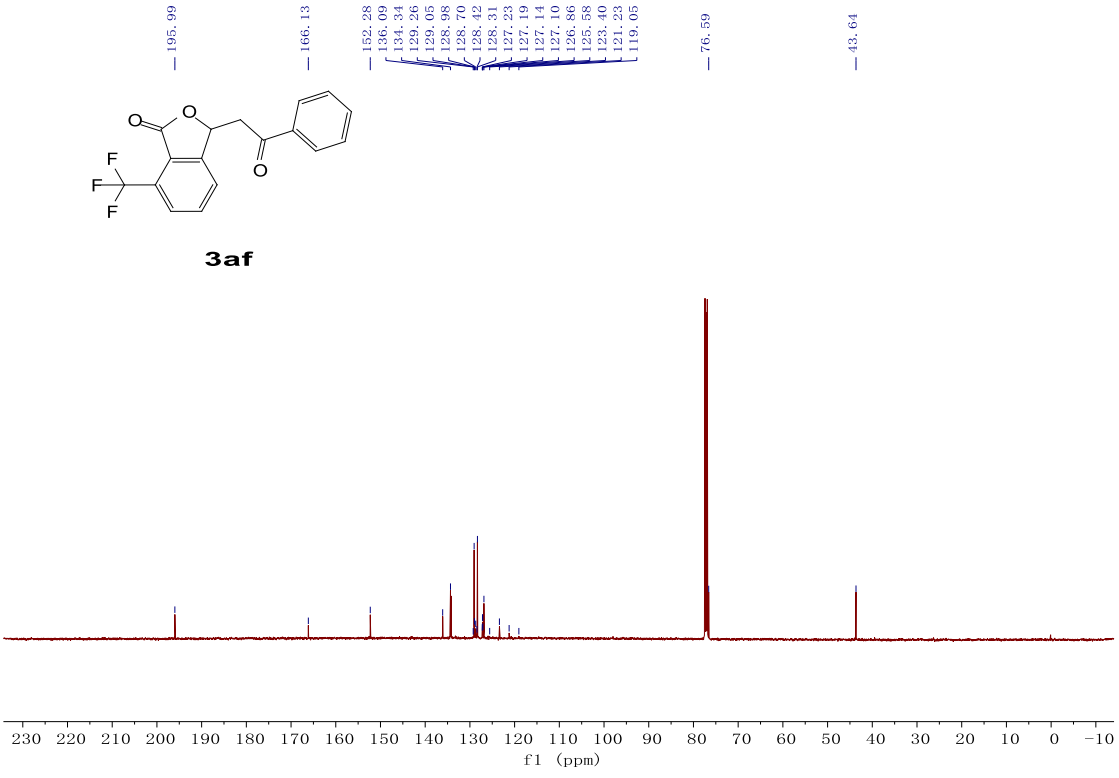
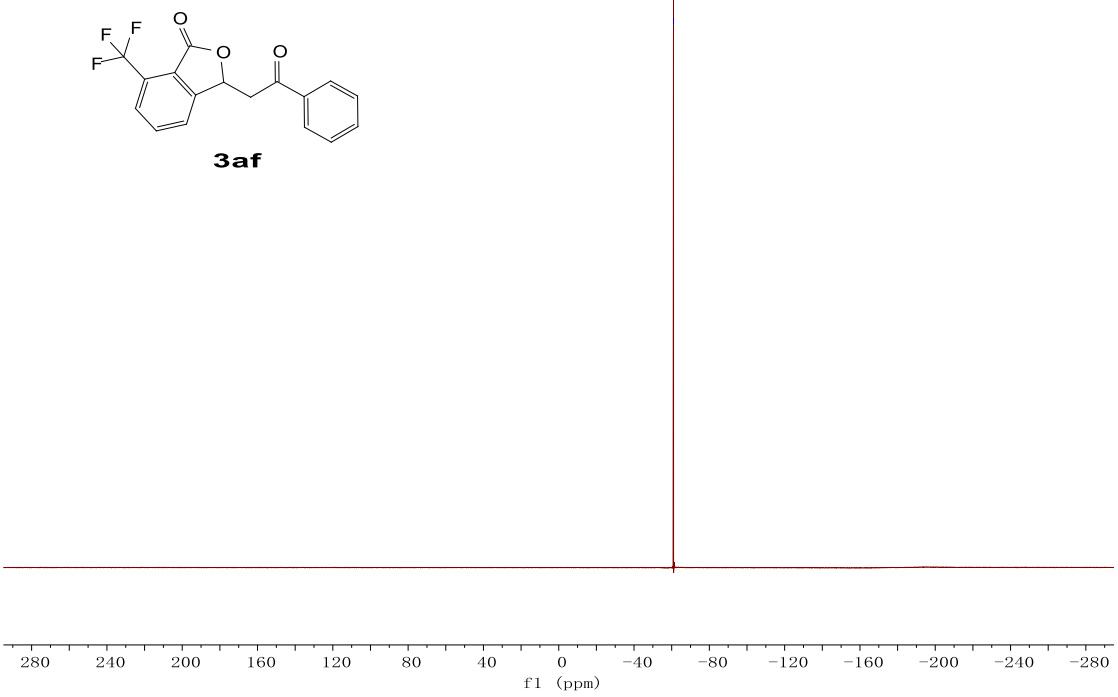
7.49
7.48
7.48
7.48
7.26
CDCl3
6.21
6.20
6.19
6.18
3.86
3.85
3.82
3.82
3.81
3.45
3.43
3.41
3.40
F
F
F
O
O
O

3af

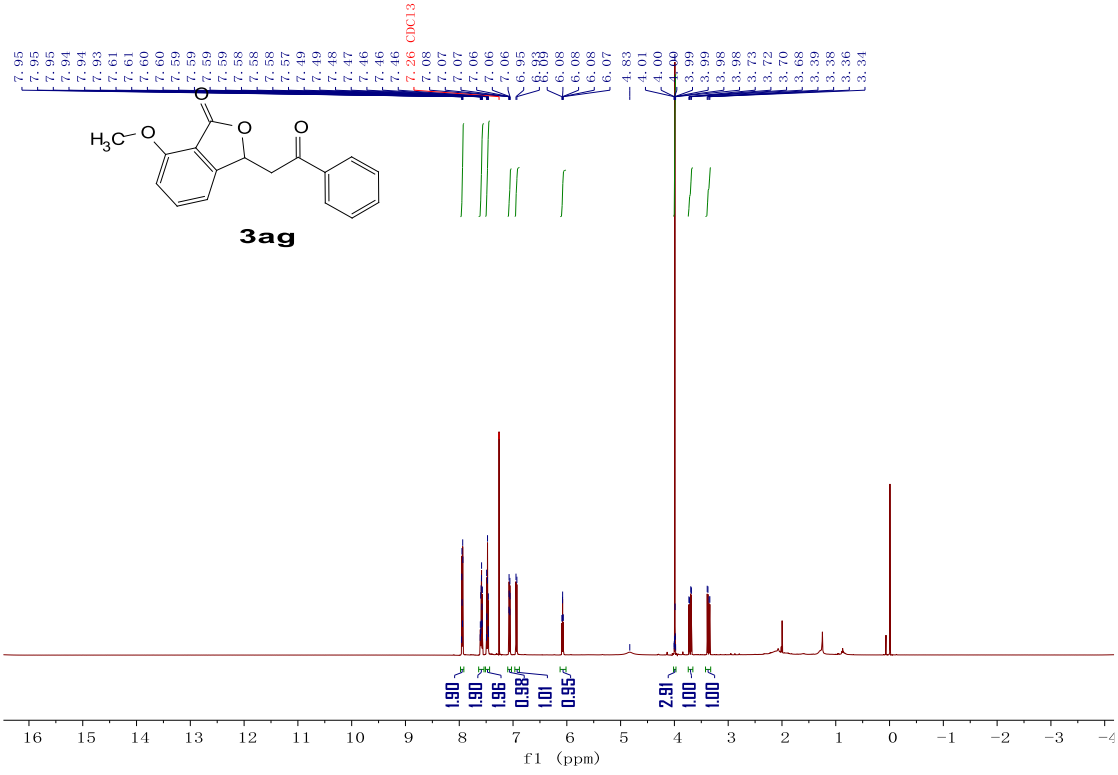

1.85
1.68
0.86
0.90
1.80
0.83
0.87
1.00
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
−2
−3
f1 (ppm)


195.99
166.13
152.28
136.09
134.34
129.26
129.05
128.98
128.70
128.42
128.31
127.23
127.19
127.14
127.10
126.86
125.58
123.40
121.23
119.05
76.59
43.64
O
O
O
F
F
F
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

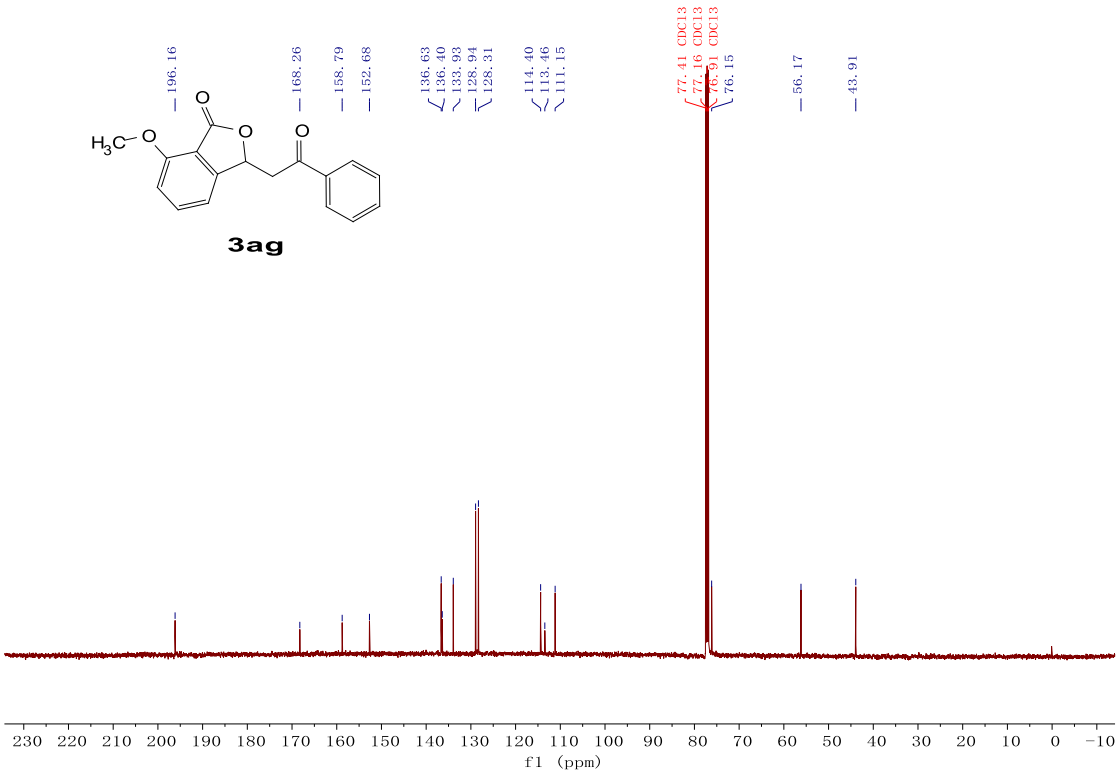
3af

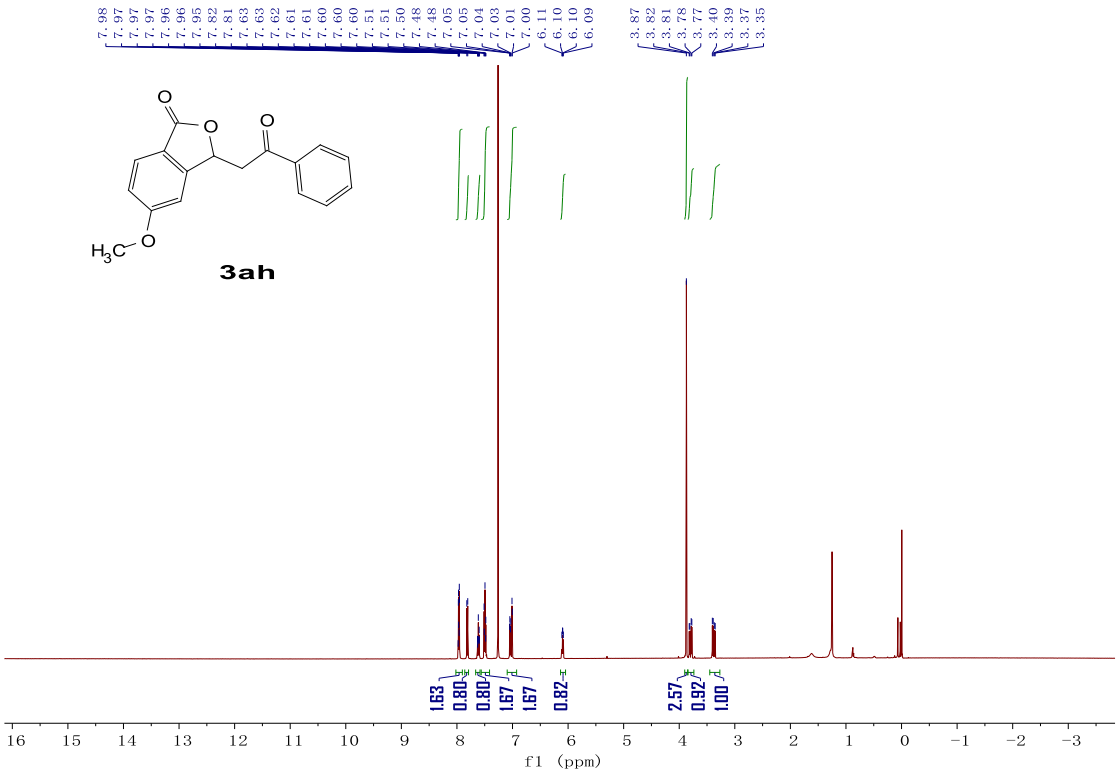

-60.97
280 240 200 160 120 80 40 0 -40 -80 -120 -160 -200 -240 -280
f1 (ppm)

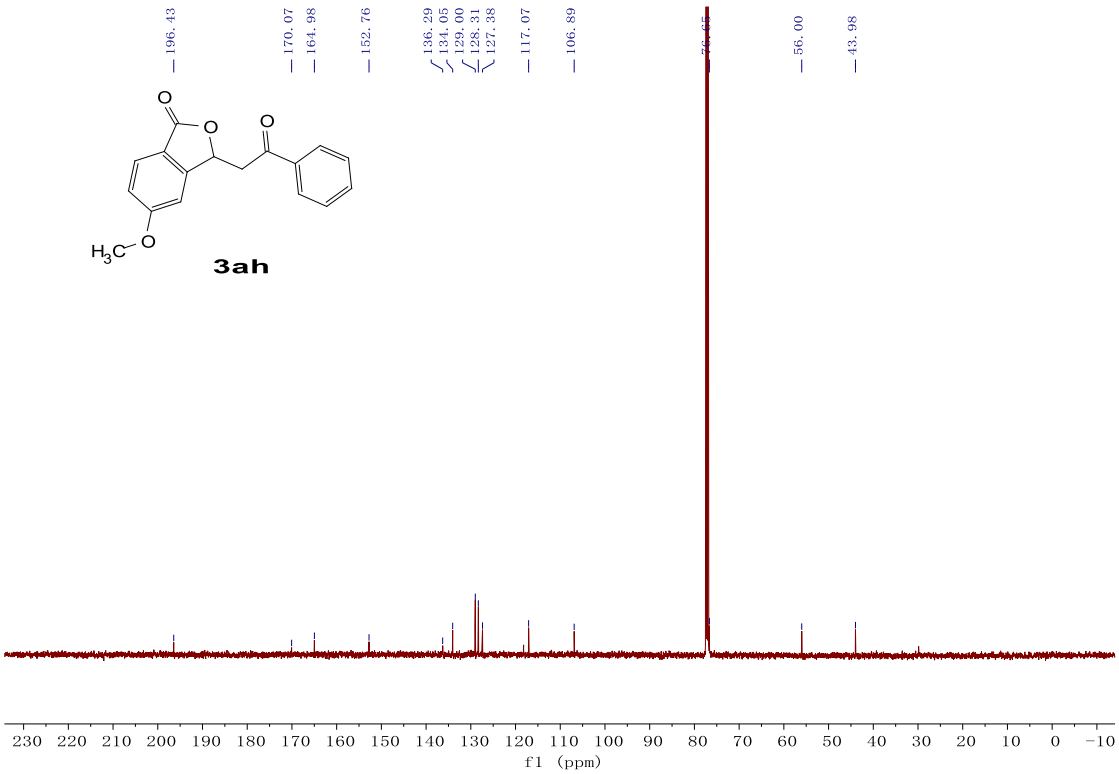
3af

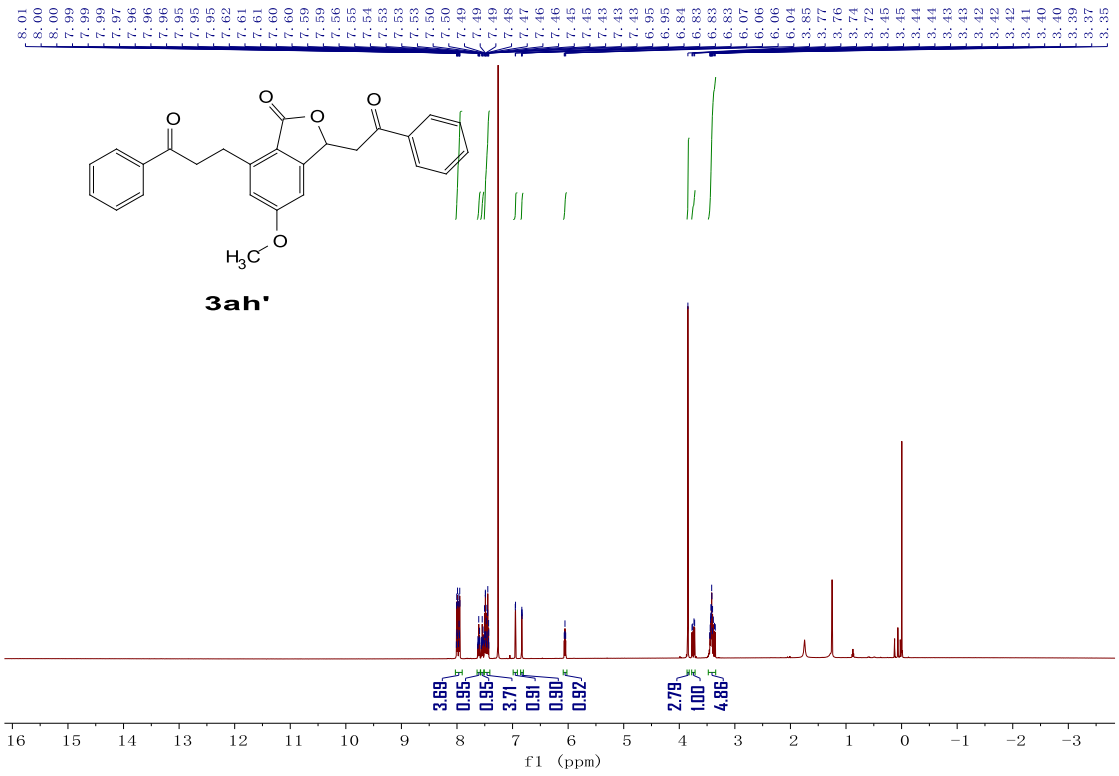

7.95 7.95 7.95 7.94 7.94 7.93 7.61 7.61 7.60 7.60 7.59 7.59 7.59 7.59 7.58 7.58 7.58 7.57 7.49 7.49 7.48 7.47 7.46 7.46 7.46 7.26 CDCl3 7.08 7.07 7.07 7.06 7.06 7.06 6.95 6.93 6.08 6.08 6.08 6.07 4.83 4.01 4.00 4.00 3.99 3.99 3.98 3.98 3.73 3.72 3.70 3.68 3.39 3.38 3.36 3.34
1.90 1.90 1.96 0.98 1.01 0.95 2.91 1.00 1.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
f1 (ppm)

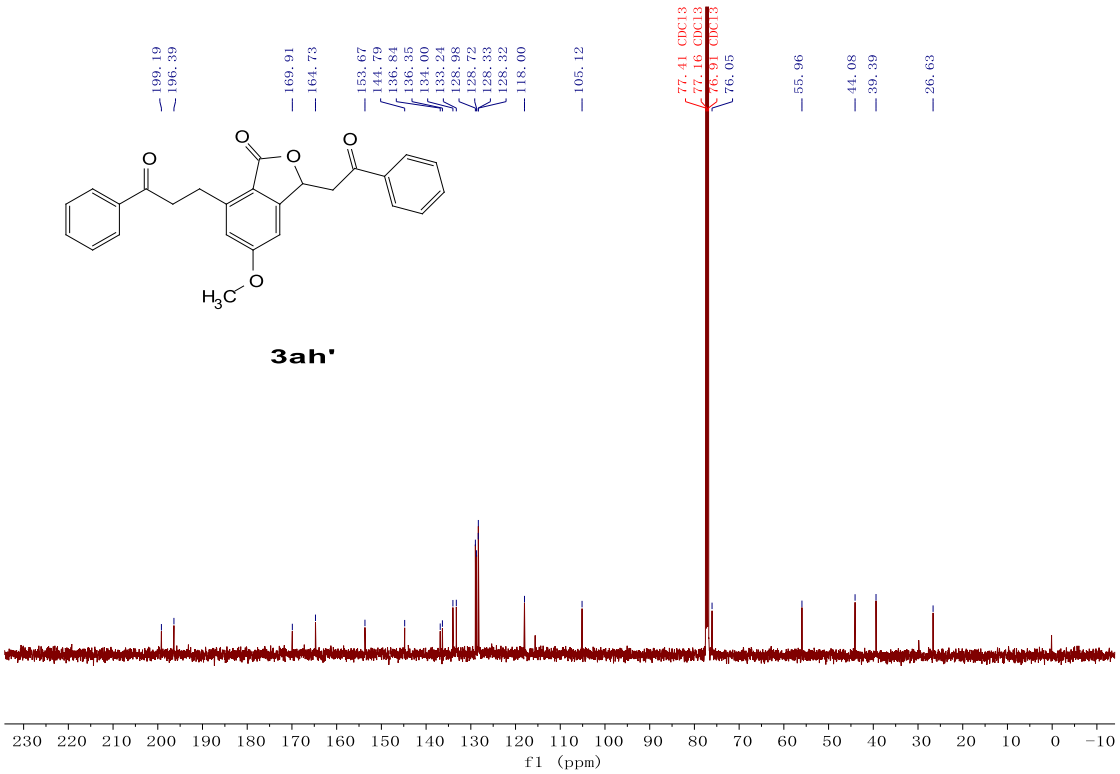
3ag

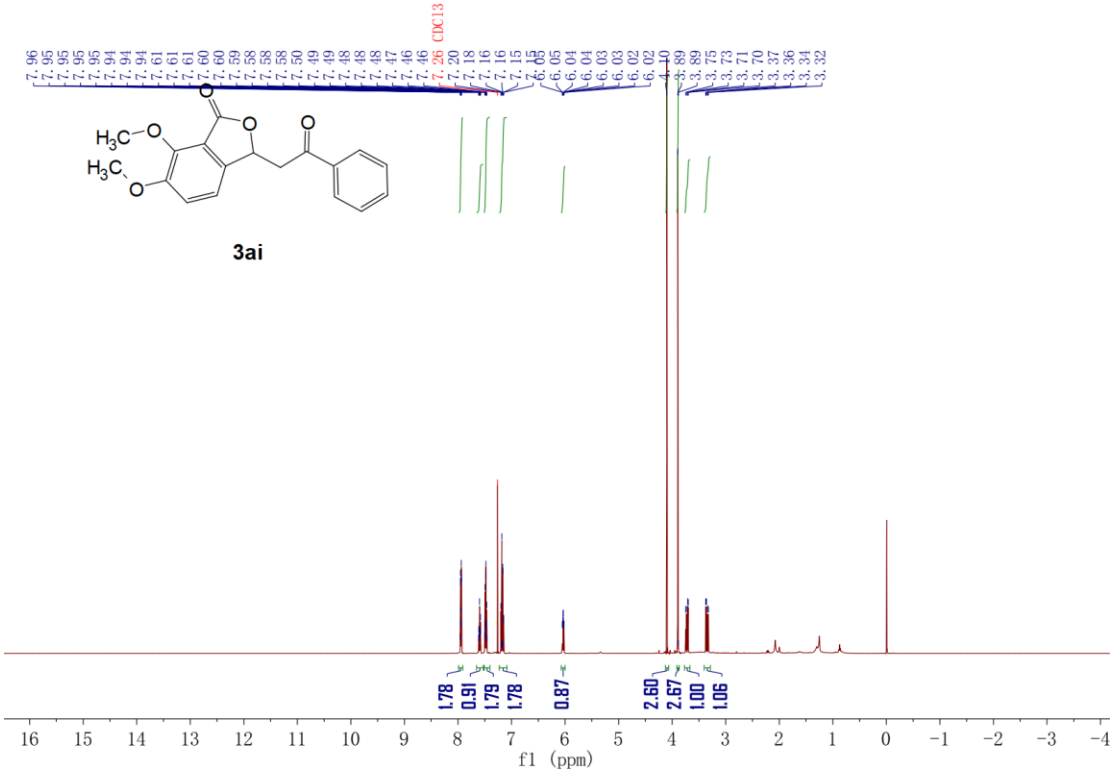

196.16
168.26
158.79
152.68
136.63
136.40
133.93
128.94
128.31
114.40
113.46
111.15
77.41 CDCl3
77.16 CDCl3
76.91 CDCl3
76.15
56.17
43.91
3ag
f1 (ppm)

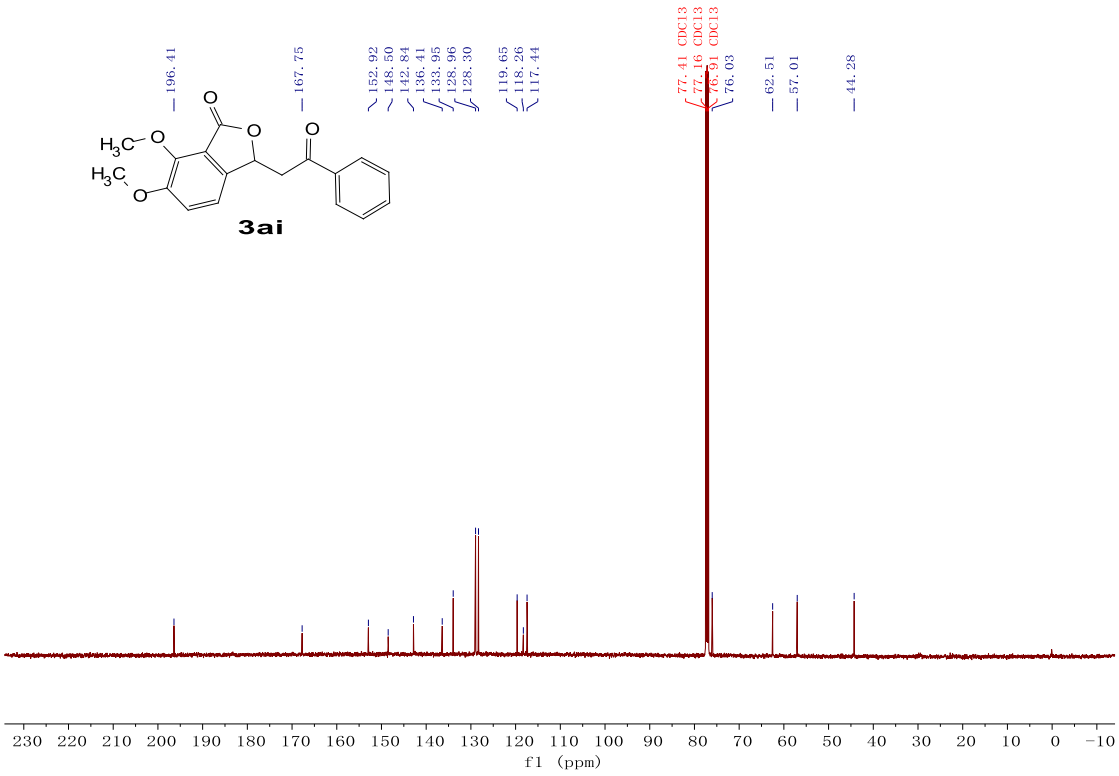

7.98
7.97
7.97
7.97
7.96
7.96
7.95
7.82
7.81
7.63
7.63
7.62
7.61
7.61
7.60
7.60
7.60
7.51
7.51
7.50
7.48
7.48
7.05
7.05
7.04
7.03
7.01
7.00
6.11
6.10
6.10
6.09
3.87
3.82
3.81
3.78
3.77
3.40
3.39
3.37
3.35
1.63
0.80
0.80
1.67
1.67
0.82
2.57
0.92
1.00
3ah
f1 (ppm)

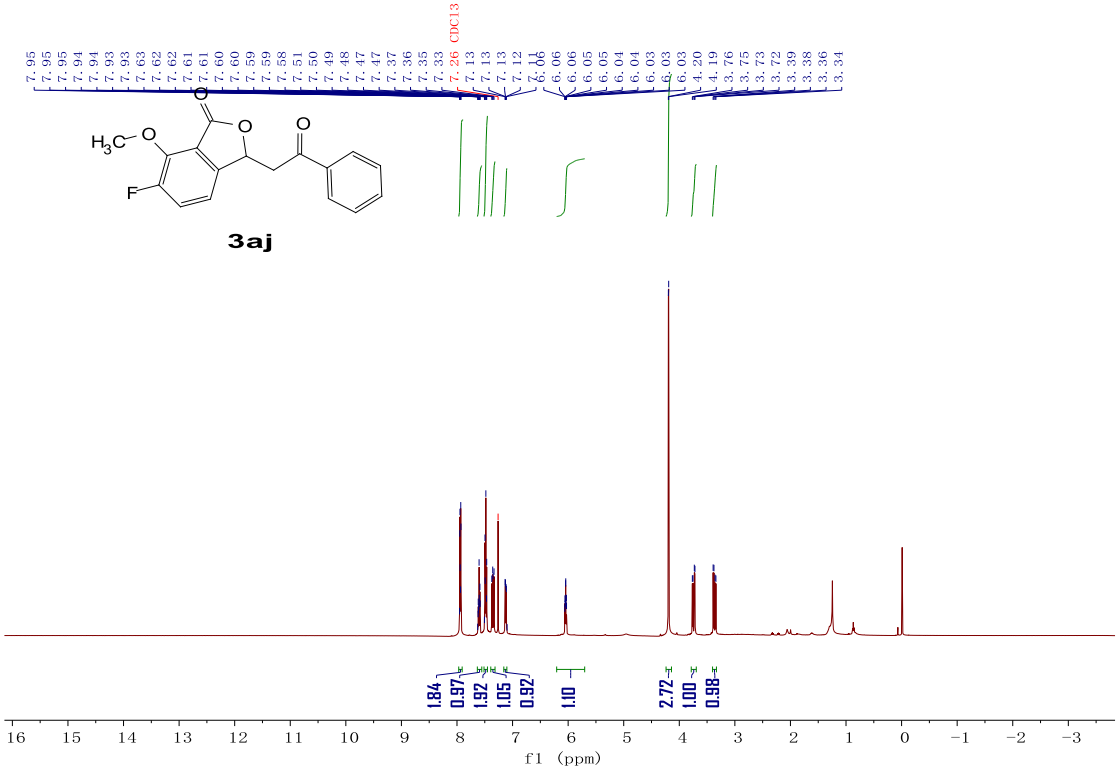

196.43
170.07
164.98
152.76
136.29
134.05
129.00
128.31
127.38
117.07
106.89
77.45
56.00
43.98
3ah
f1 (ppm)

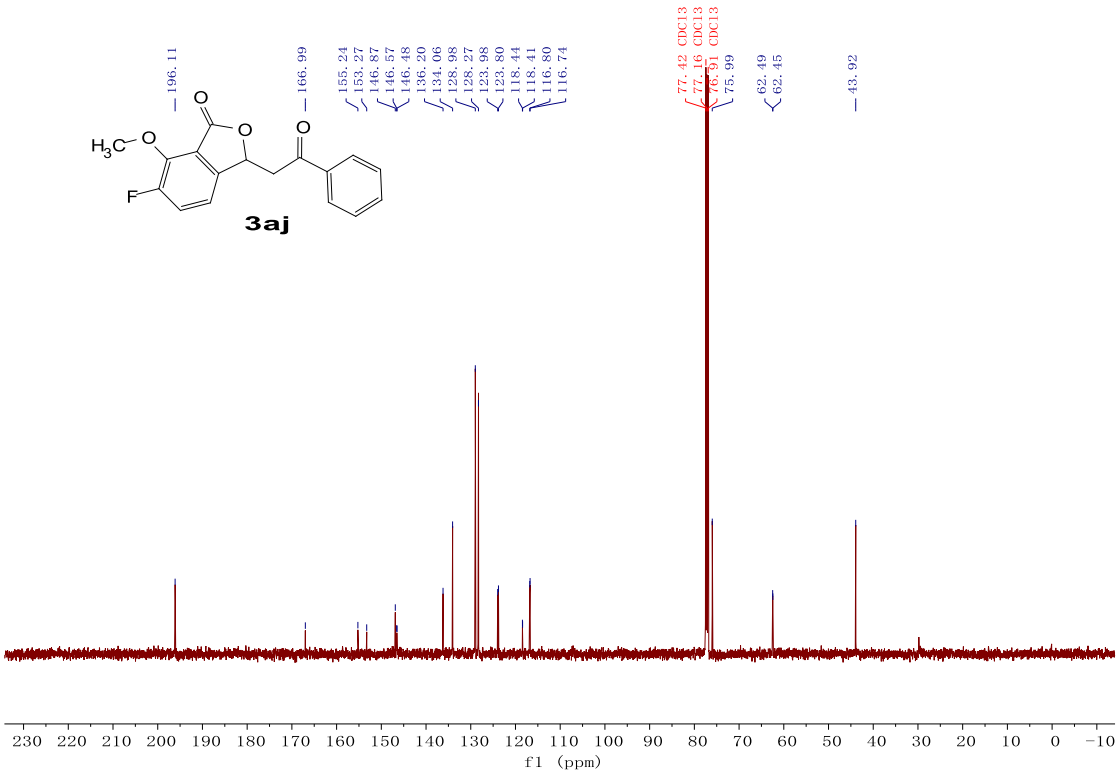

3ah'
3.69
0.95
0.95
3.71
0.91
0.90
0.92
2.79
1.00
4.86
f1 (ppm)

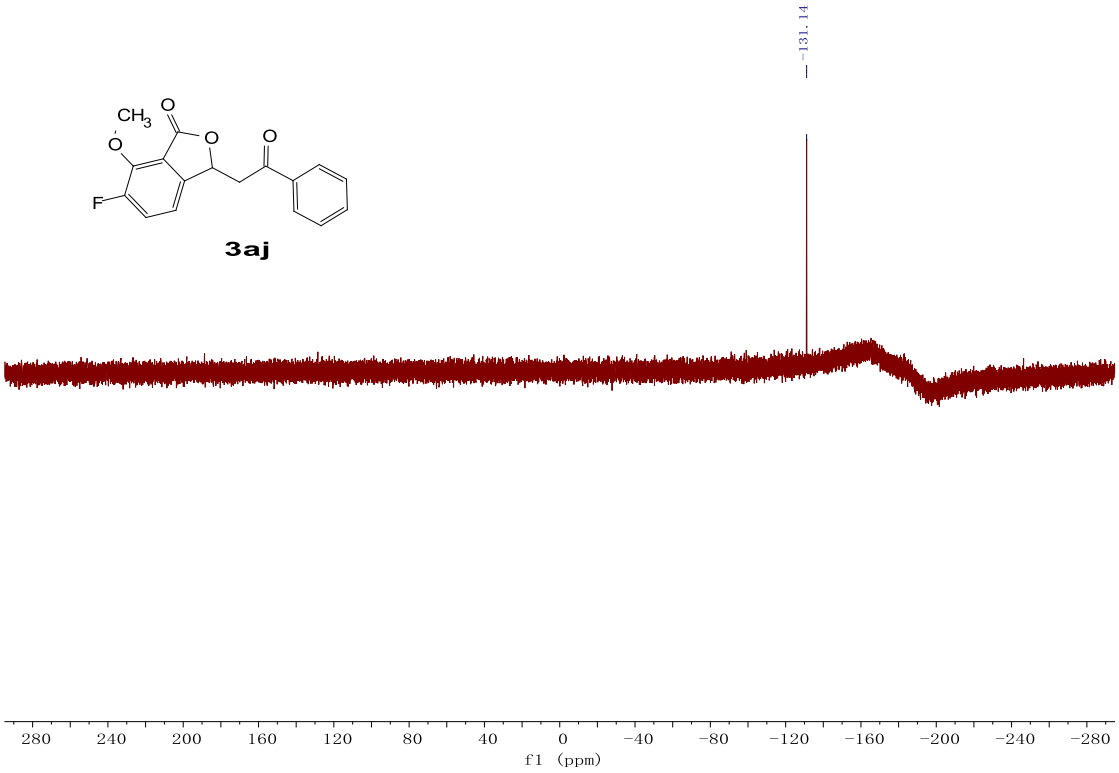

199.19
196.39
169.91
164.73
153.67
144.79
136.84
136.35
134.00
133.24
128.98
128.72
128.33
128.32
118.00
105.12
77.41 CDCl3
77.16 CDCl3
76.91 CDCl3
76.05
55.96
44.08
39.39
26.63
O
O
O
O
O
H3C
3ah'
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

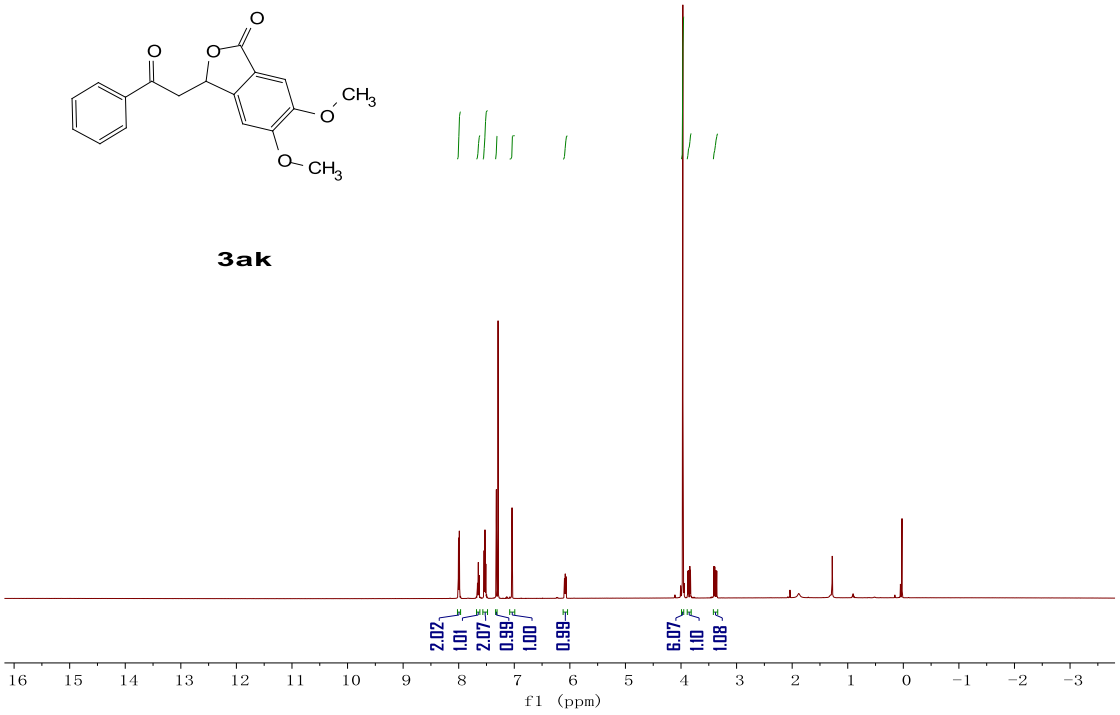

7.96
7.95
7.95
7.95
7.95
7.94
7.94
7.94
7.61
7.61
7.61
7.60
7.60
7.59
7.58
7.58
7.58
7.50
7.49
7.49
7.48
7.48
7.48
7.47
7.46
7.46
7.26 CDCl3
7.20
7.18
7.16
7.16
7.15
7.15
6.05
6.04
6.04
6.03
6.03
6.02
6.02
4.10
3.89
3.89
3.75
3.73
3.71
3.70
3.37
3.36
3.34
3.32
O
O
O
O
H3C
H3C
3ai
1.78
0.91
1.79
1.78
0.87
2.60
2.67
1.00
1.06
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3 −4
f1 (ppm)

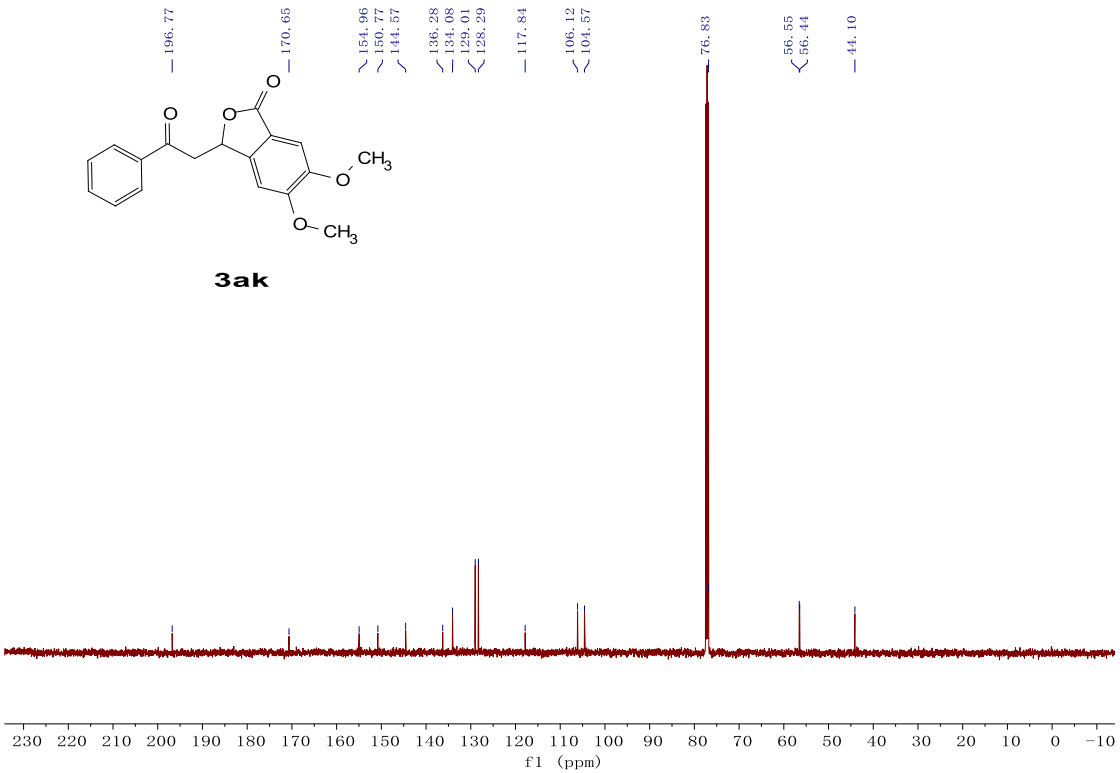

196.41
167.75
152.92
148.50
142.84
136.41
133.95
128.96
128.30
119.65
118.26
117.44
77.41 CDCl3
77.16 CDCl3
76.91 CDCl3
76.03
62.51
57.01
44.28
3ai
f1 (ppm)

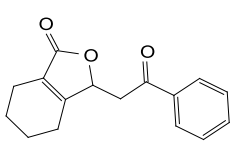

7.26 CDCl3
1.84
0.97
1.92
1.05
0.92
1.10
2.72
1.00
0.98
3aj
f1 (ppm)


196.11
166.99
155.24
153.27
146.87
146.57
146.48
136.20
134.06
128.98
128.27
123.98
123.80
118.44
118.41
116.80
116.74
77.42 CDCl3
77.16 CDCl3
76.91 CDCl3
75.99
62.49
62.45
43.92
3aj
f1 (ppm)

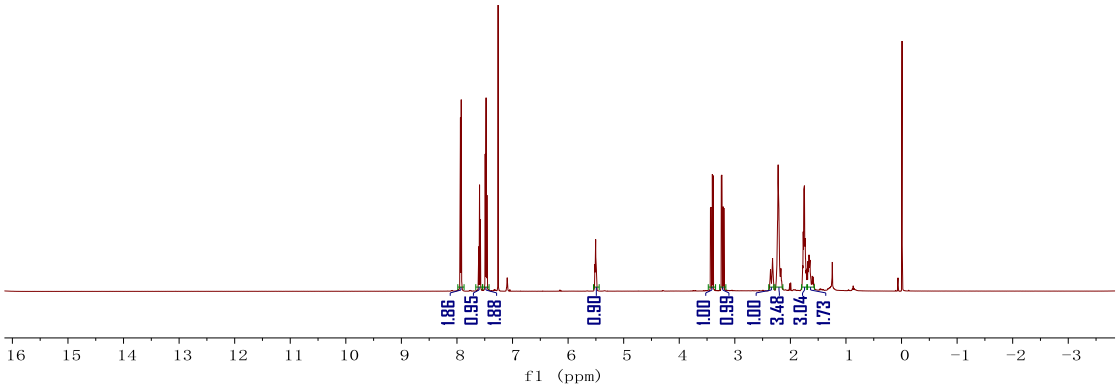

-131.14
3aj
f1 (ppm)

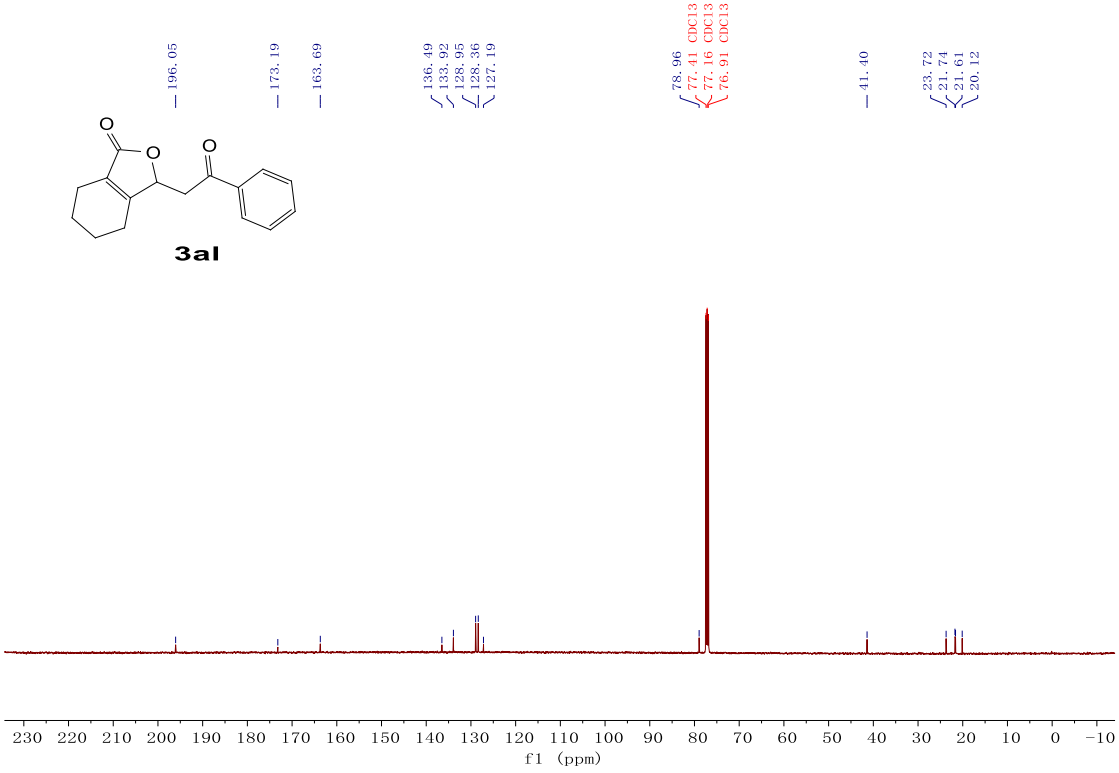

O
O
O
CH3
O
O
CH3
3ak
2.02
1.01
2.07
0.99
1.00
0.99
6.07
1.10
1.08
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 −1 −2 −3
f1 (ppm)


196.77
170.65
154.96
150.77
144.57
136.28
134.08
129.01
128.29
117.84
106.12
104.57
76.83
56.55
56.44
44.10
O
O
O
CH3
O
O
CH3
3ak
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

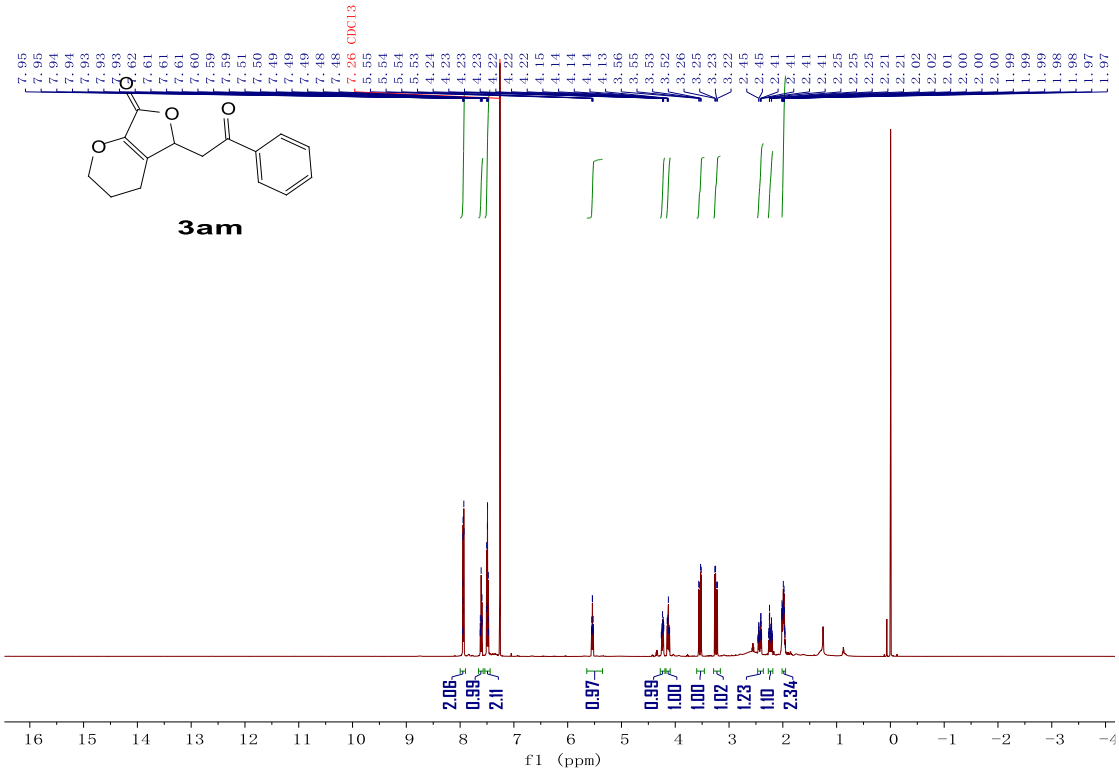


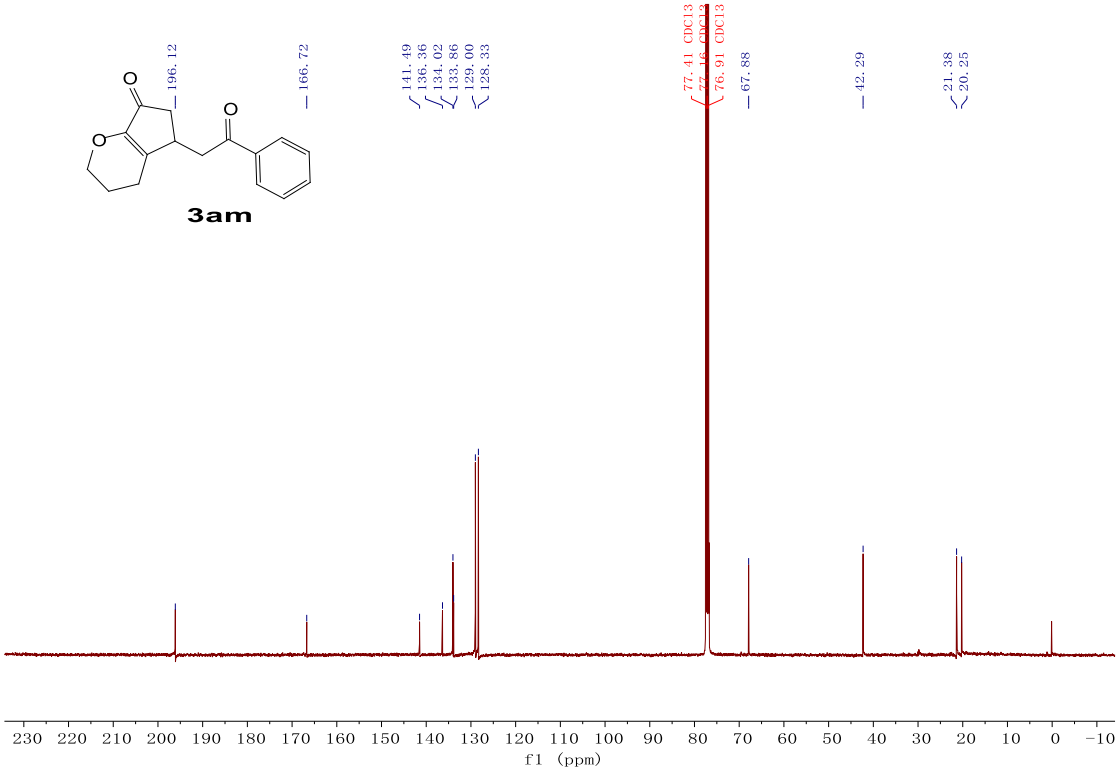
3al

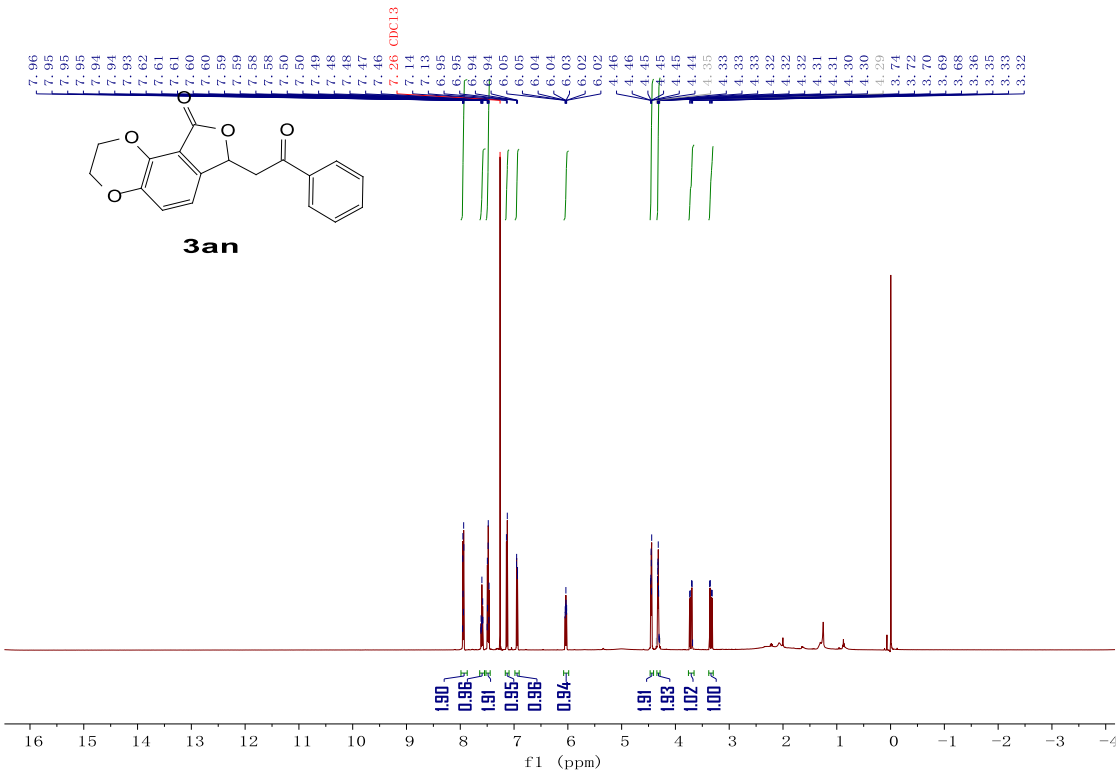

1.86
0.95
1.88
0.90
1.00
0.99
1.00
3.48
3.04
1.73
f1 (ppm)

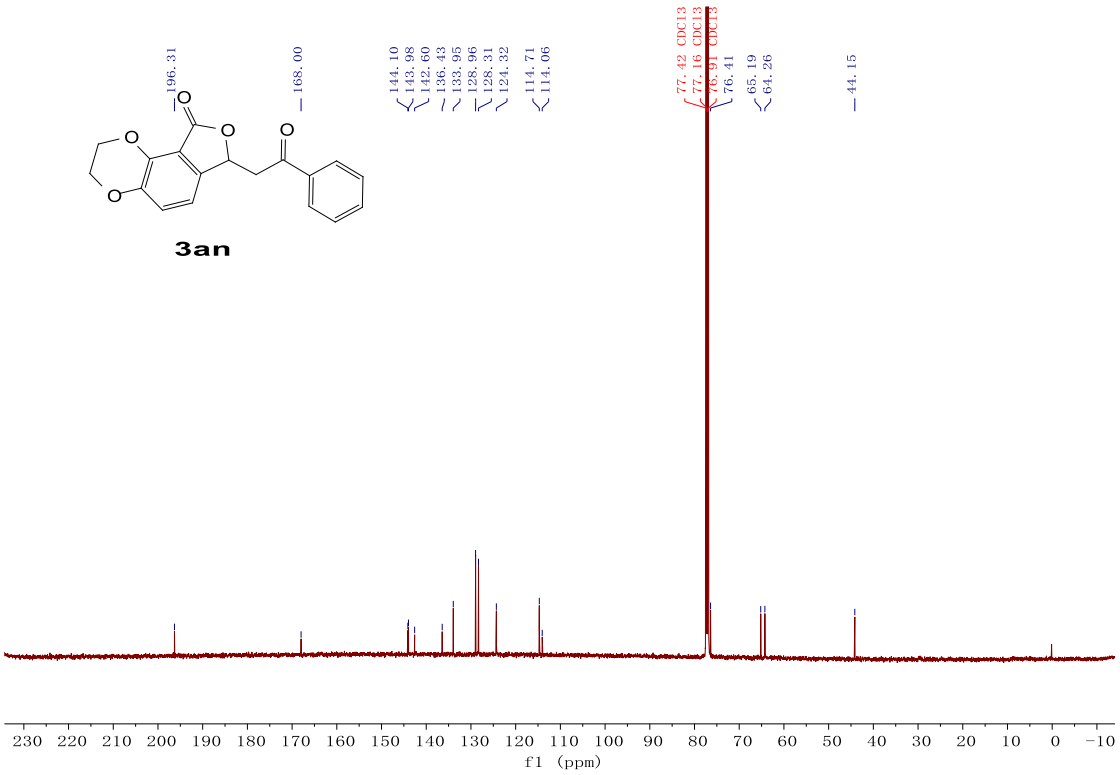

196.05
173.19
163.69
136.49
133.92
128.95
128.36
127.19
78.96
77.41 CDCl3
77.16 CDCl3
76.91 CDCl3
41.40
23.72
21.74
21.61
20.12
f1 (ppm)

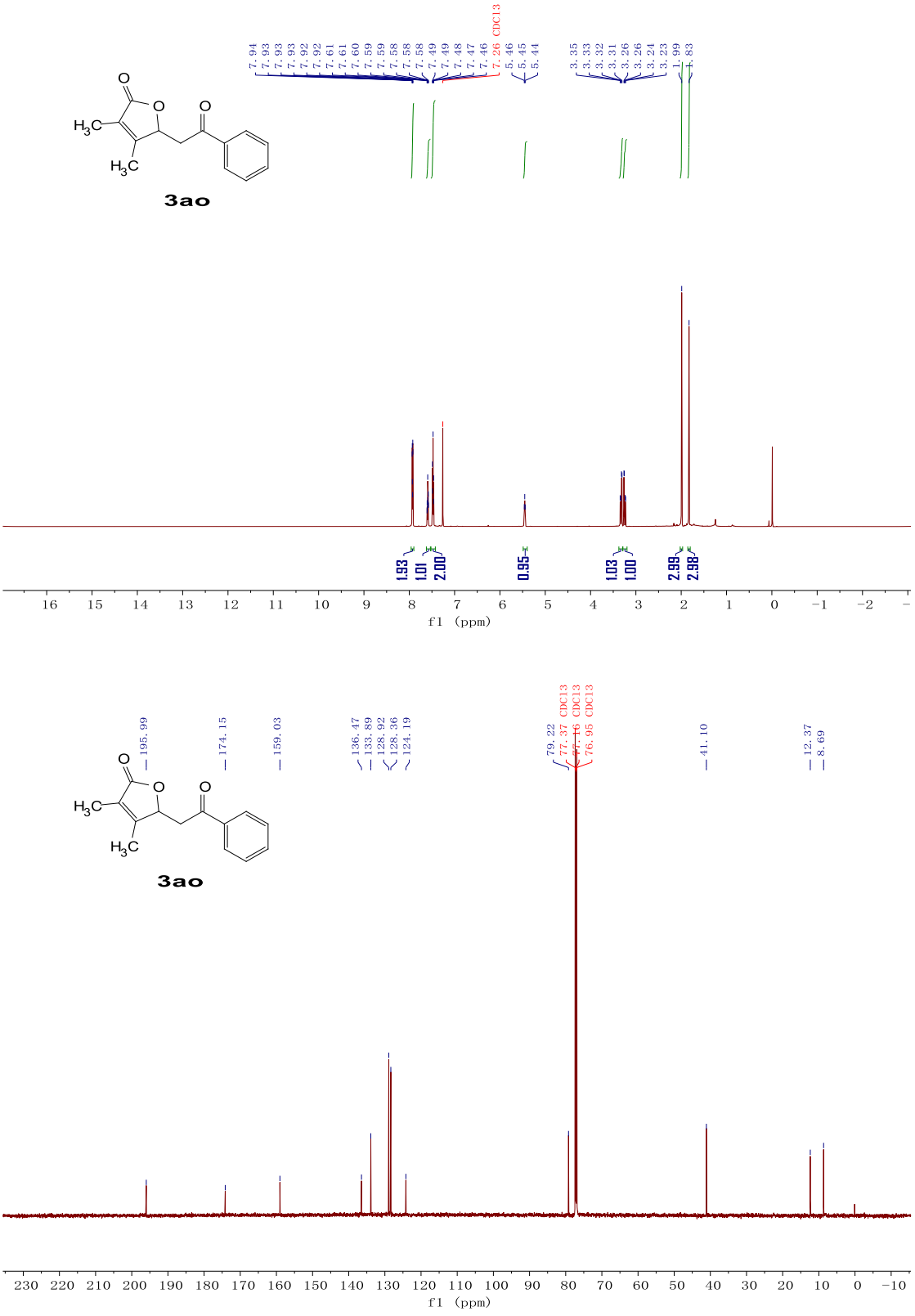
3al

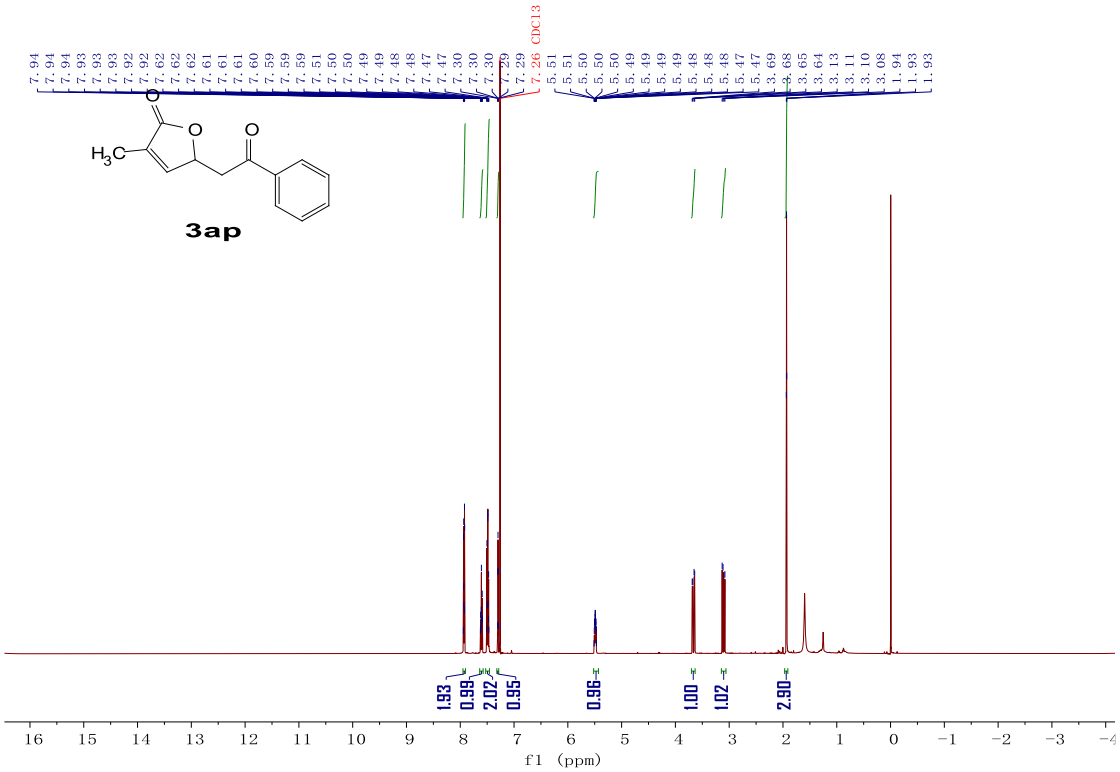

3am
f1 (ppm)

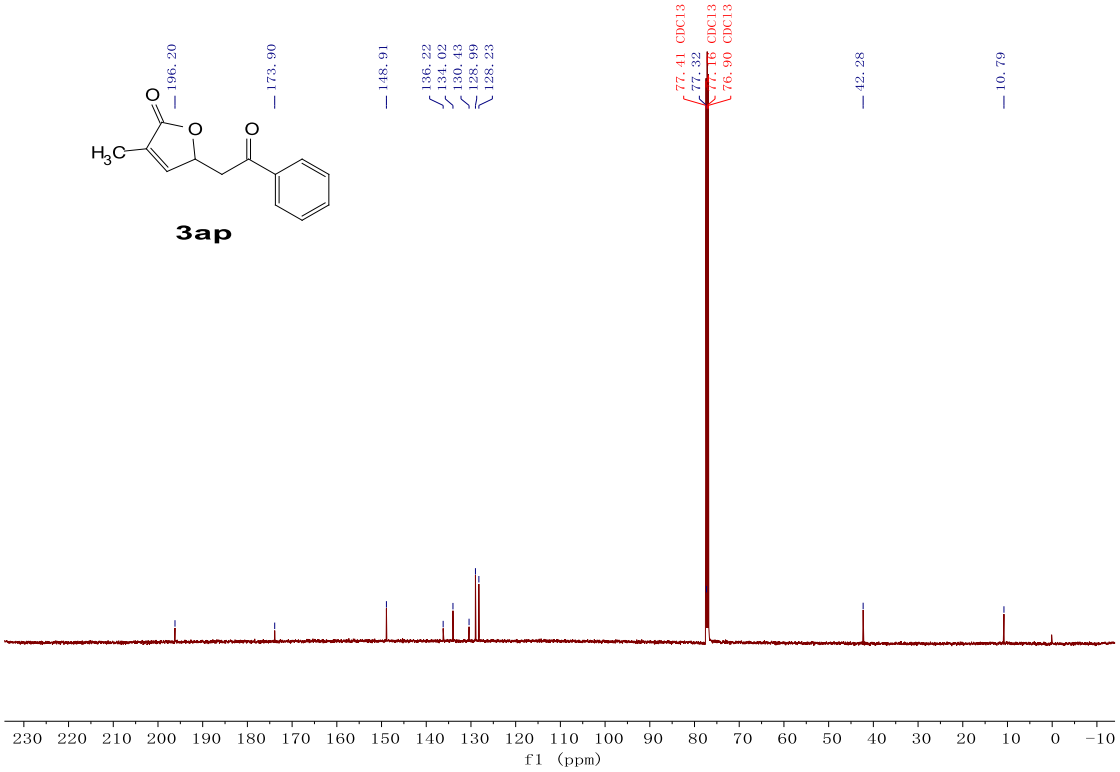

3am
f1 (ppm)

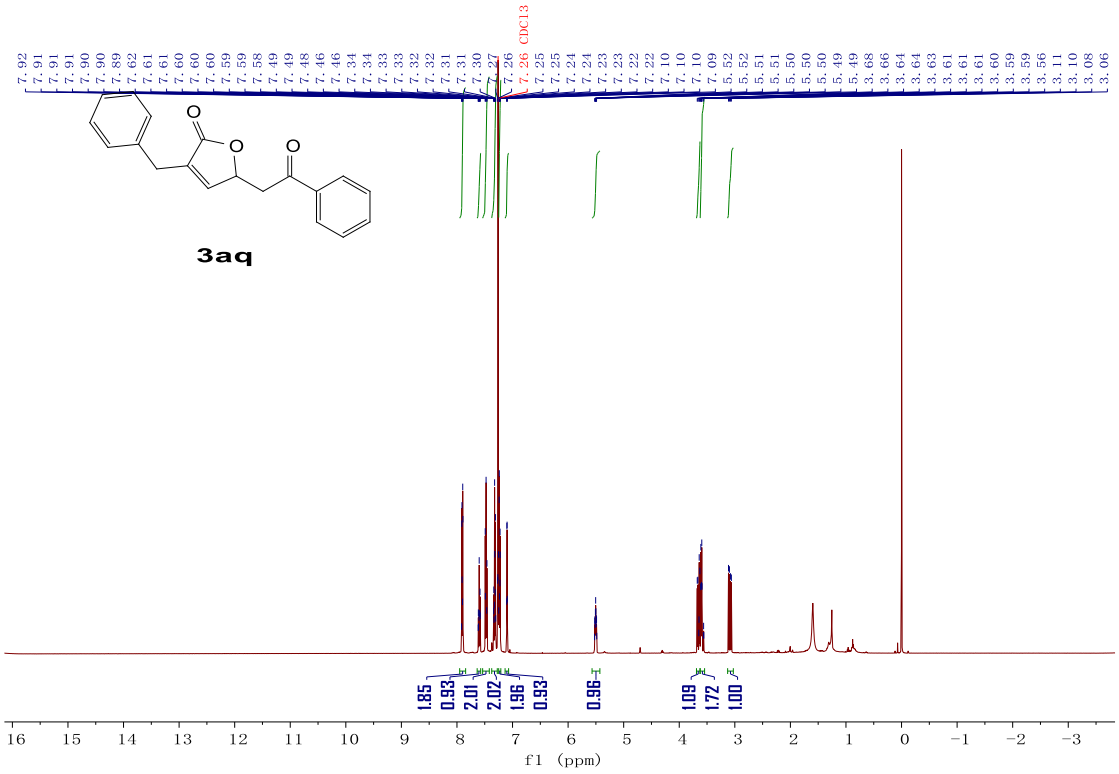

7.96 7.95 7.95 7.95 7.94 7.94 7.93 7.62 7.61 7.61 7.60 7.60 7.59 7.59 7.58 7.58 7.50 7.50 7.49 7.48 7.48 7.47 7.46 7.26 CDCl3 7.14 7.13 6.95 6.95 6.94 6.94 6.05 6.05 6.04 6.04 6.03 6.02 6.02 4.46 4.46 4.45 4.45 4.45 4.44 4.35 4.33 4.33 4.33 4.32 4.32 4.31 4.31 4.30 4.30 4.29 3.74 3.72 3.70 3.69 3.68 3.36 3.35 3.33 3.32
3an
1.90 0.96 1.91 0.95 0.96 0.94 1.91 1.93 1.02 1.00
f1 (ppm)

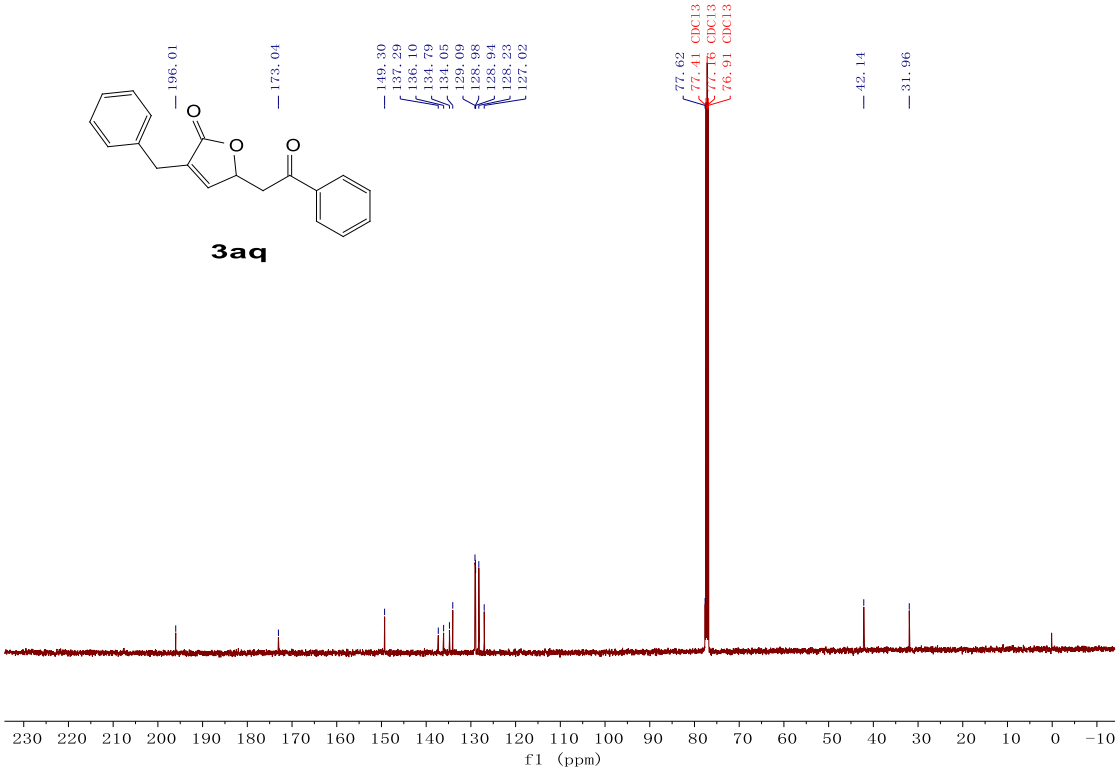

196.31 168.00 144.10 143.98 142.60 136.43 133.95 128.96 128.31 124.32 114.71 114.06 77.42 CDCl3 77.16 CDCl3 76.91 CDCl3 76.41 65.19 64.26 44.15
3an
f1 (ppm)

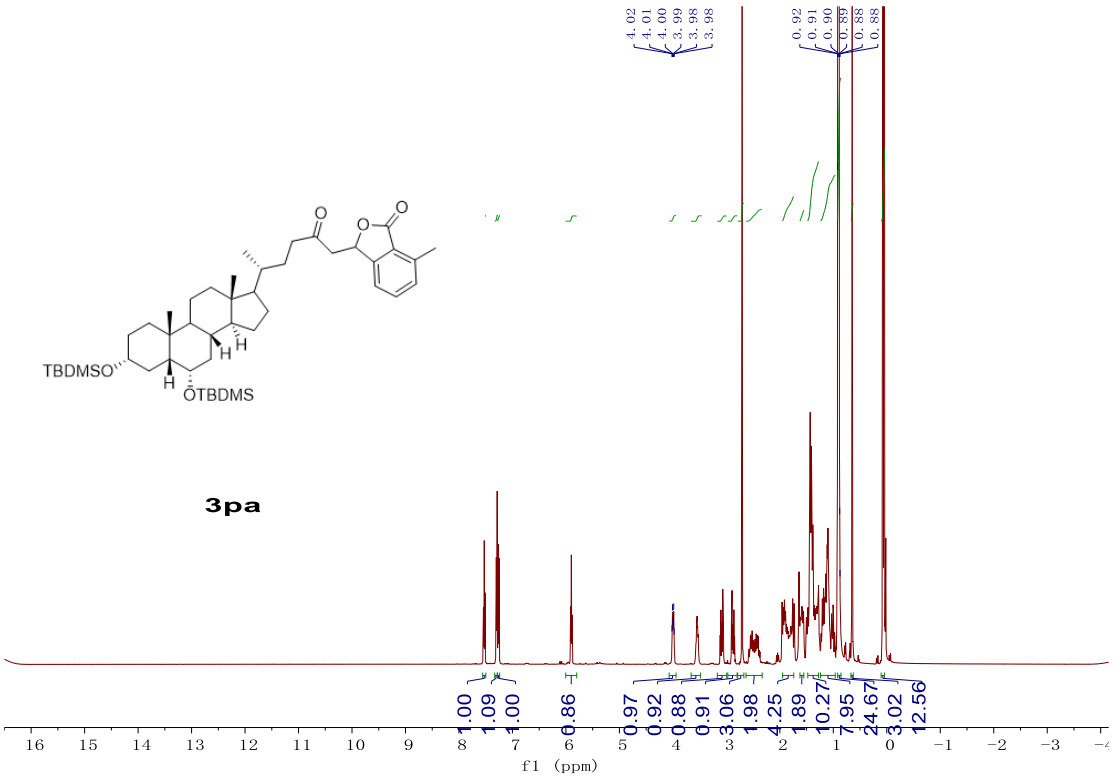

7.94
7.93
7.93
7.93
7.92
7.92
7.61
7.61
7.60
7.59
7.59
7.58
7.58
7.58
7.49
7.49
7.48
7.47
7.46
7.26 CDCl3
5.46
5.45
5.44
3.35
3.33
3.32
3.31
3.26
3.26
3.24
3.23
1.99
1.83
O
O
O
H3C
H3C
3ao
1.93
1.01
2.00
0.95
1.03
1.00
2.99
2.98
f1 (ppm)
195.99
174.15
159.03
136.47
133.89
128.92
128.36
124.19
79.22
77.37 CDCl3
77.16 CDCl3
76.95 CDCl3
41.10
12.37
8.69
3ao
f1 (ppm)

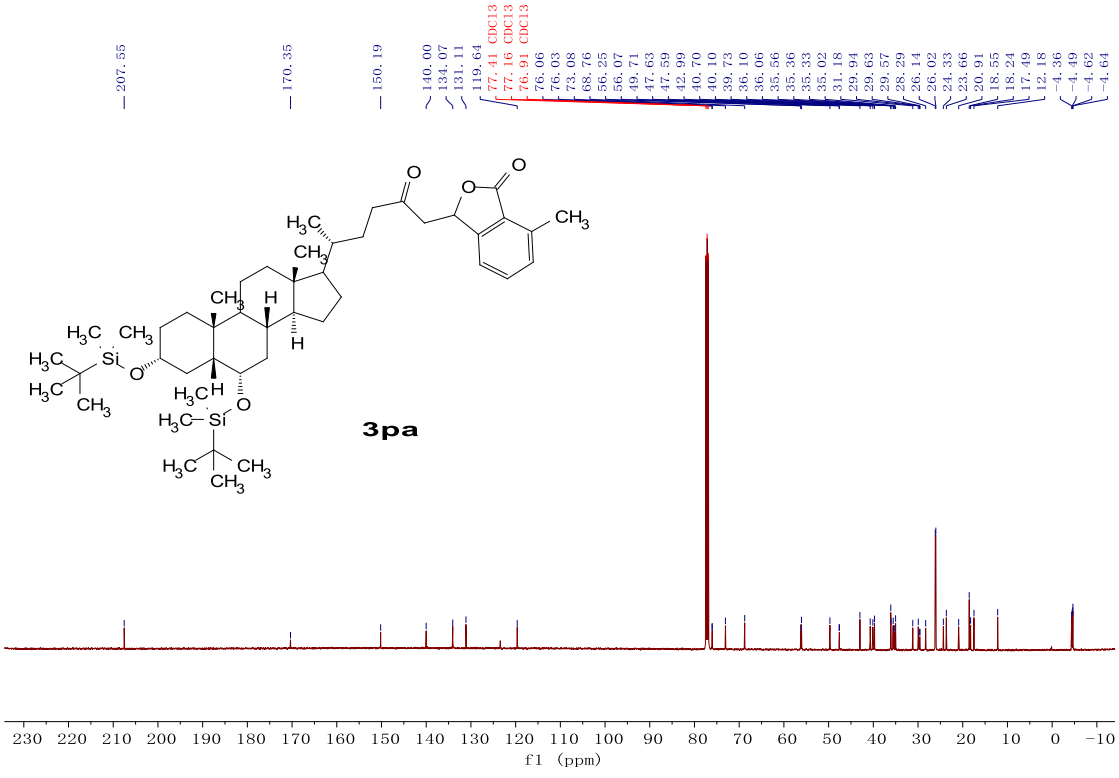

3ap
1.93
0.99
2.02
0.95
0.96
1.00
1.02
2.90
f1 (ppm)

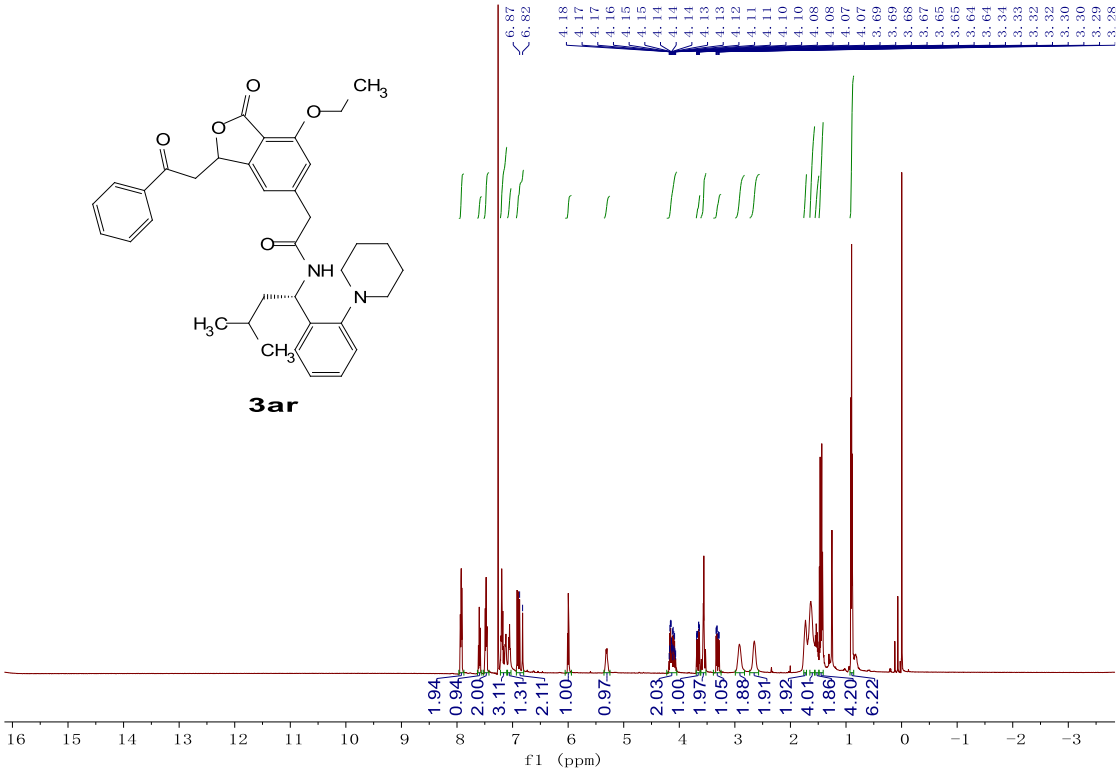

3ap
196.20
173.90
148.91
136.22
134.02
130.43
128.99
128.23
77.41 CDCl3
77.32
77.16 CDCl3
76.90 CDCl3
42.28
10.79
f1 (ppm)

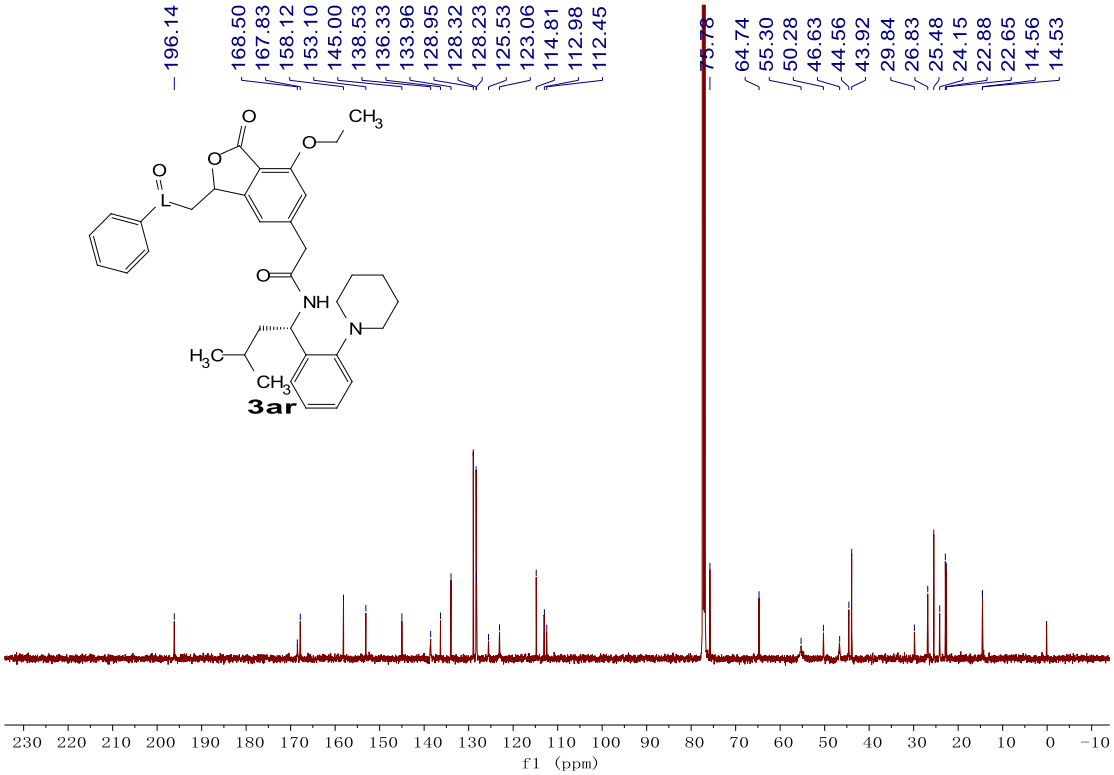

3aq
f1 (ppm)

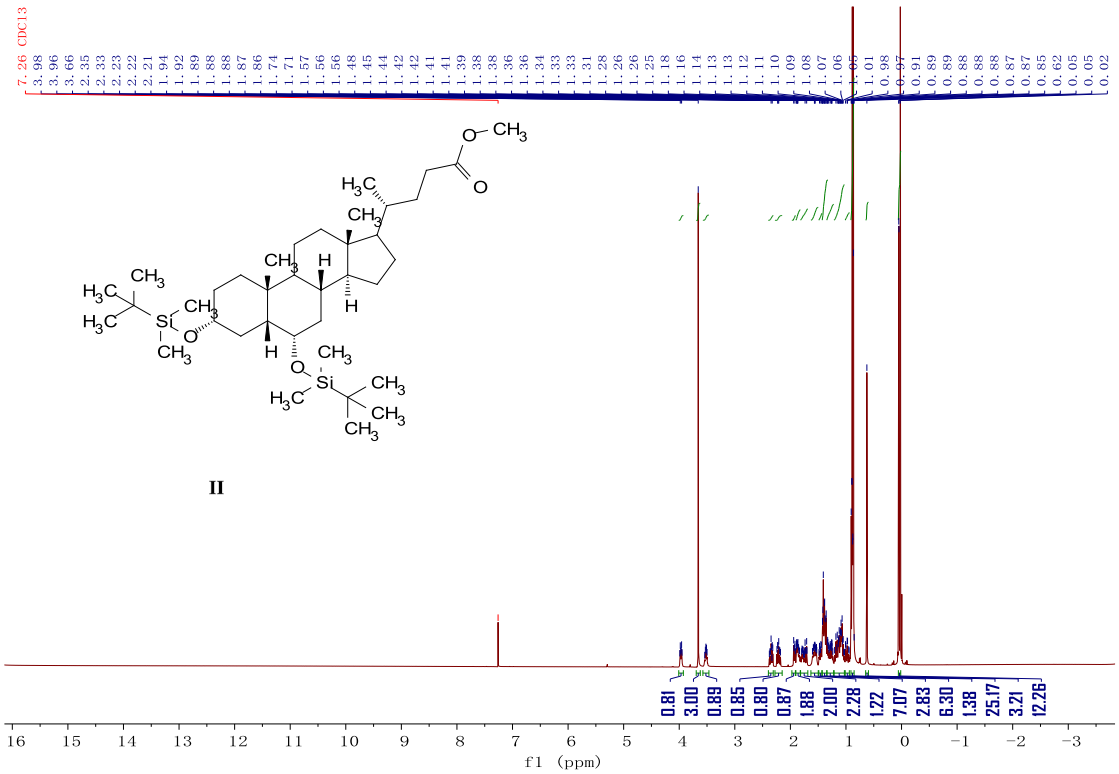

3aq
f1 (ppm)


TBDMSO
OTBDMS
3pa
f1 (ppm)

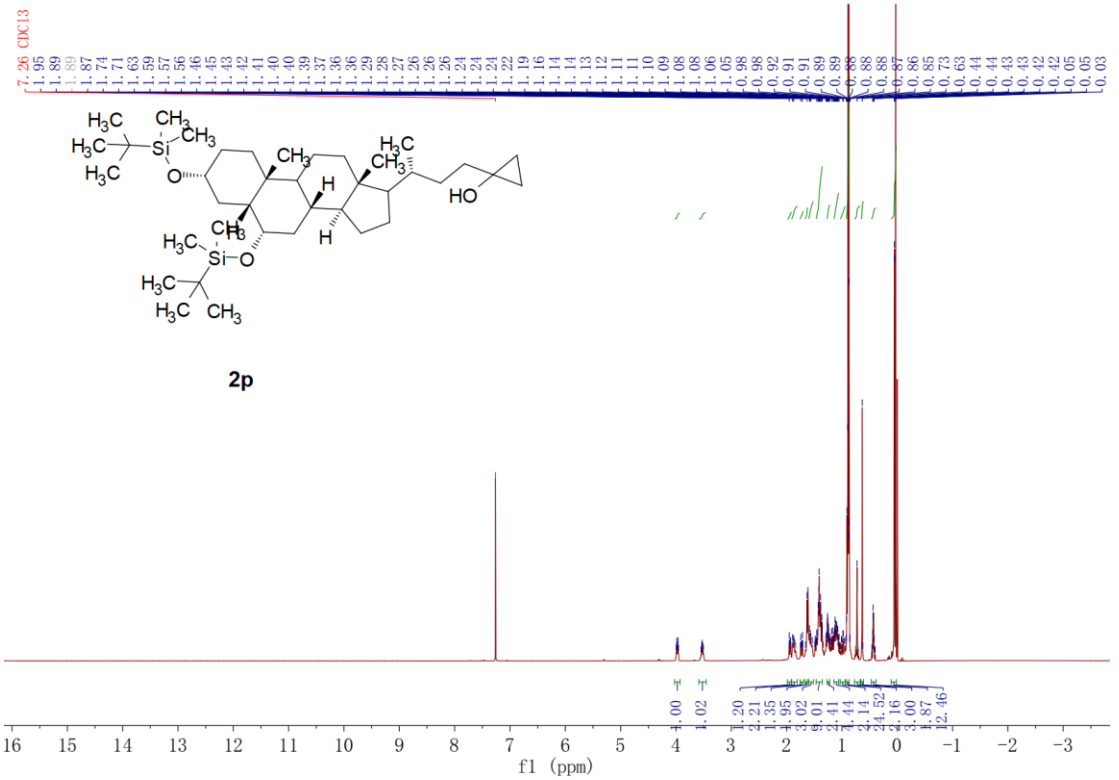

3pa
f1 (ppm)


3ar
f1 (ppm)

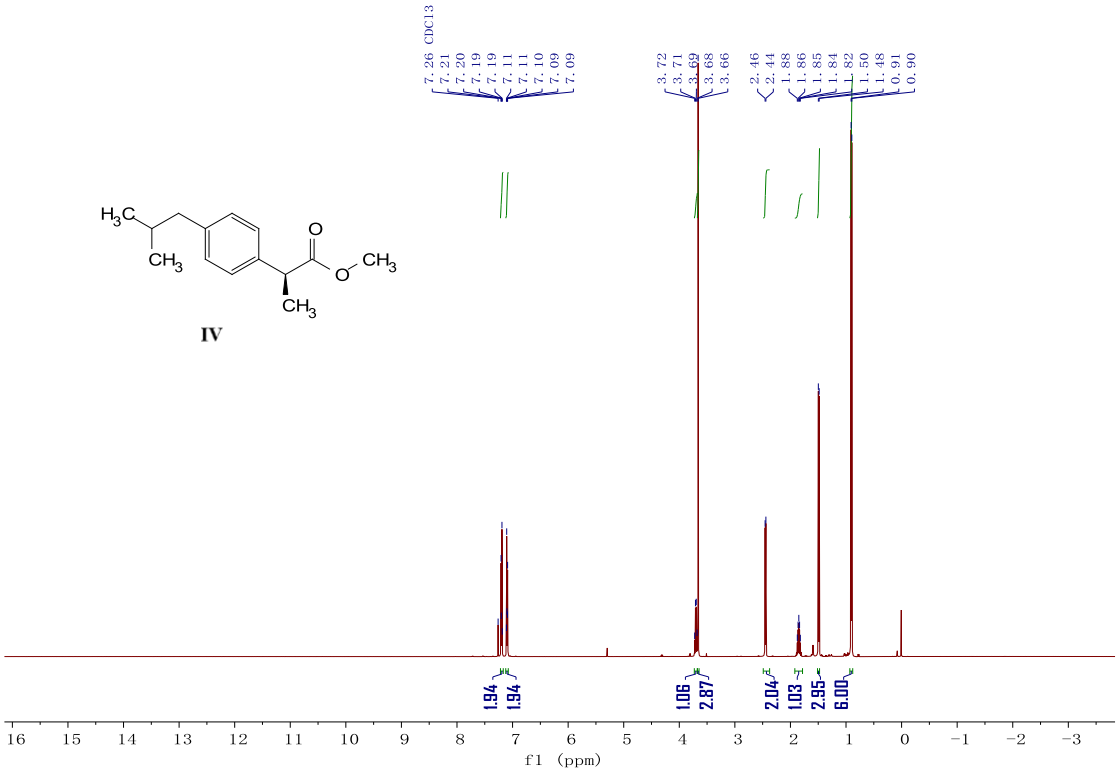

3ar
f1 (ppm)



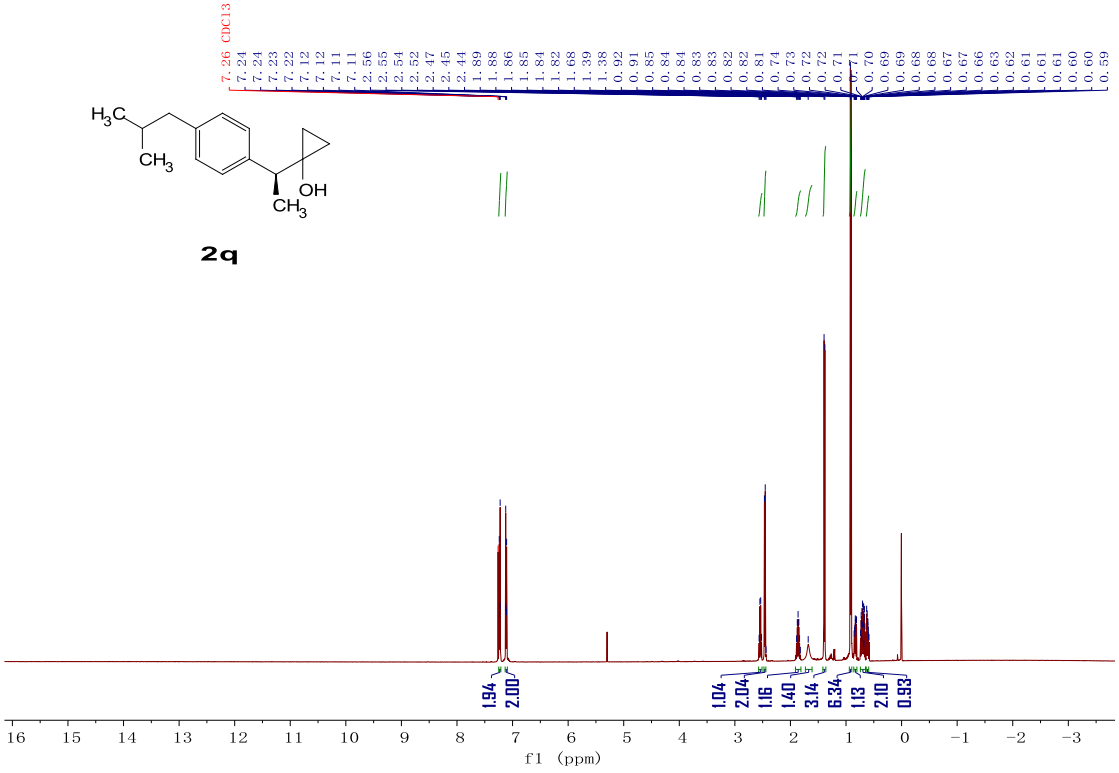
II



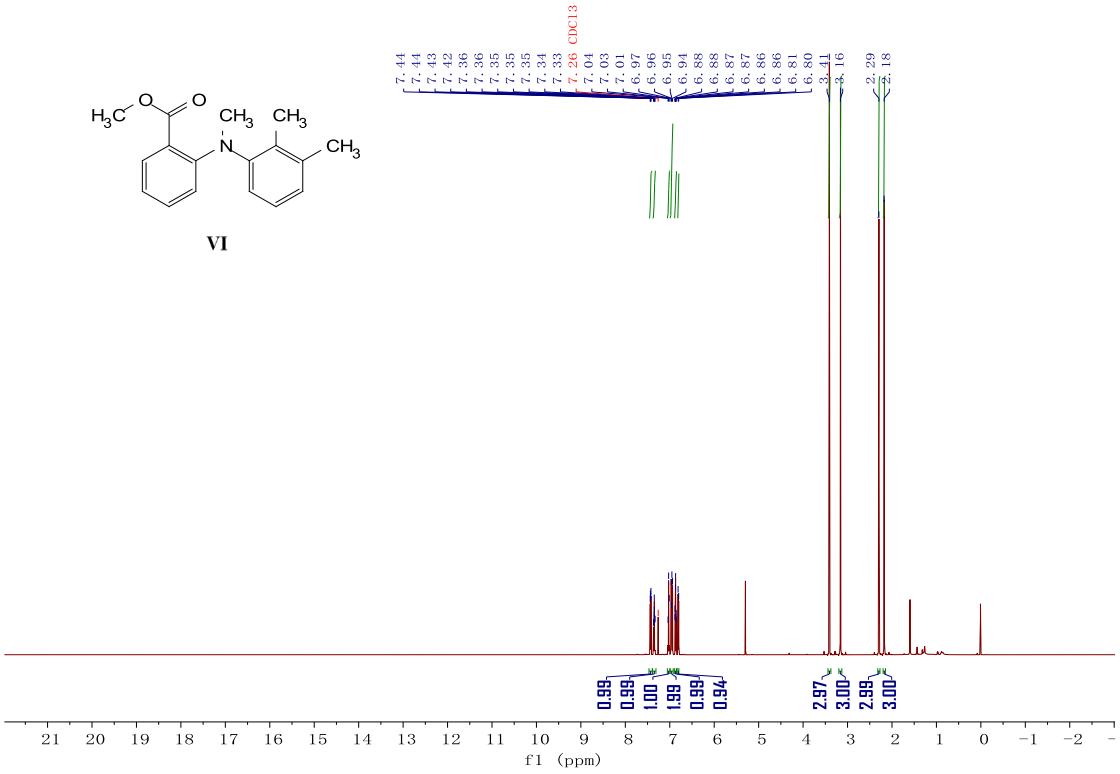
2p


7.26 CDCl3
7.21
7.20
7.19
7.11
7.10
7.09
3.72
3.71
3.69
3.68
3.66
2.46
2.44
1.88
1.86
1.85
1.84
1.82
1.50
1.48
0.91
0.90
1.94
1.94
1.06
2.87
2.04
1.03
2.95
6.00
f1 (ppm)

IV


7.26 CDCl3
7.24
7.23
7.22
7.12
7.11
2.56
2.55
2.54
2.52
2.47
2.45
2.44
1.89
1.88
1.86
1.85
1.84
1.82
1.68
1.39
1.38
0.92
0.91
0.85
0.84
0.84
0.83
0.83
0.82
0.82
0.81
0.74
0.73
0.72
0.72
0.71
0.71
0.70
0.69
0.69
0.68
0.68
0.67
0.67
0.66
0.63
0.62
0.61
0.61
0.60
0.60
0.59
1.94
2.00
1.04
2.04
1.16
1.40
3.14
6.34
1.13
2.10
0.93
f1 (ppm)

2q


VI
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7.44
7.43
7.42
7.36
7.36
7.35
7.35
7.35
7.34
7.33
7.26 CDCl3
7.04
7.03
7.01
6.97
6.96
6.95
6.94
6.88
6.88
6.87
6.87
6.86
6.86
6.81
6.80
3.41
3.16
2.29
2.18
0.99
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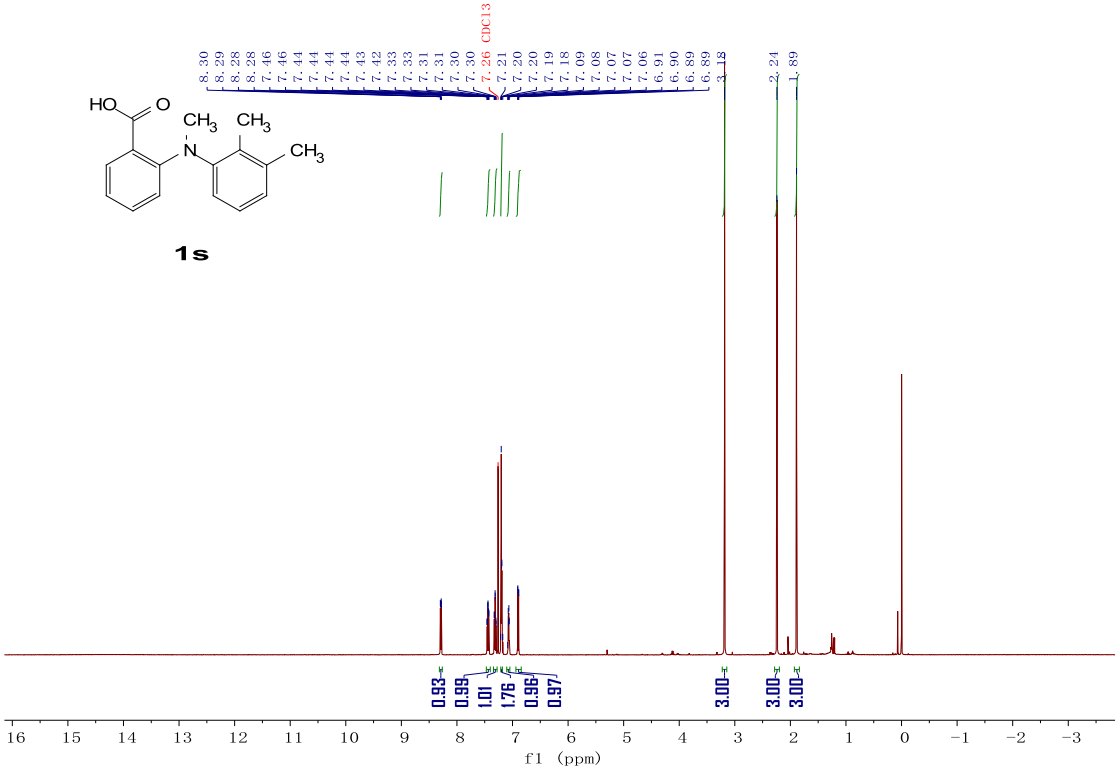
1.00
1.99
0.99
0.94
2.97
3.00
2.95
3.00
f1 (ppm)


1s
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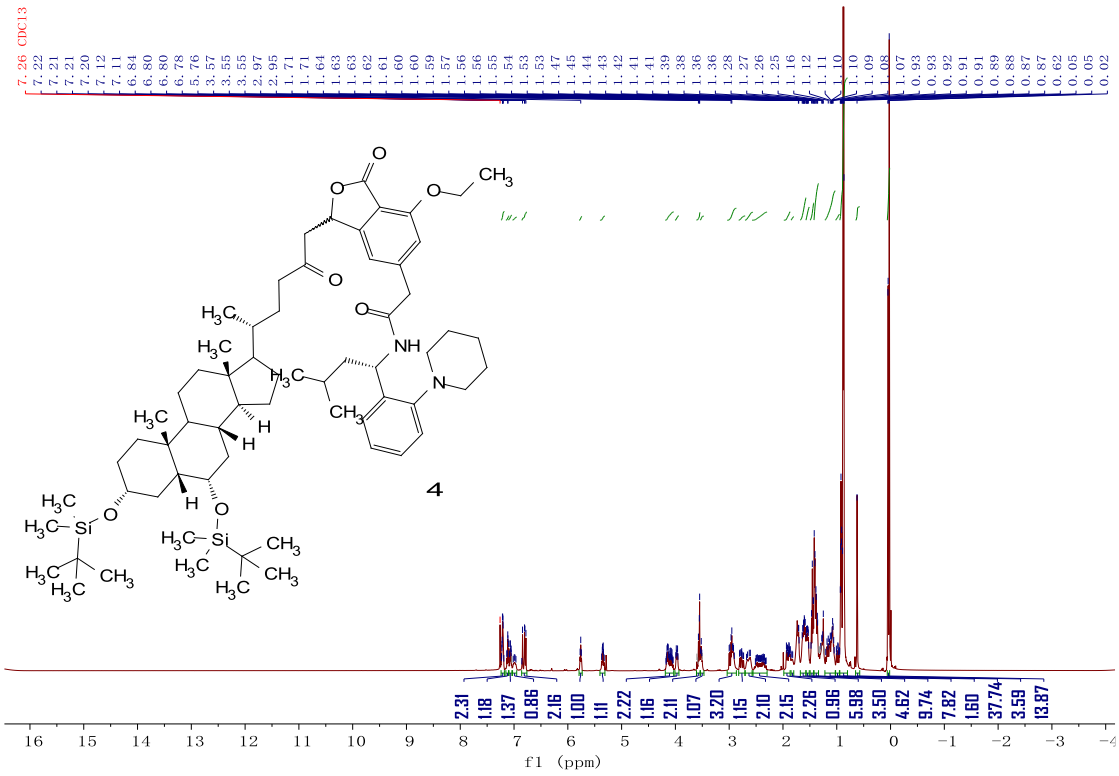
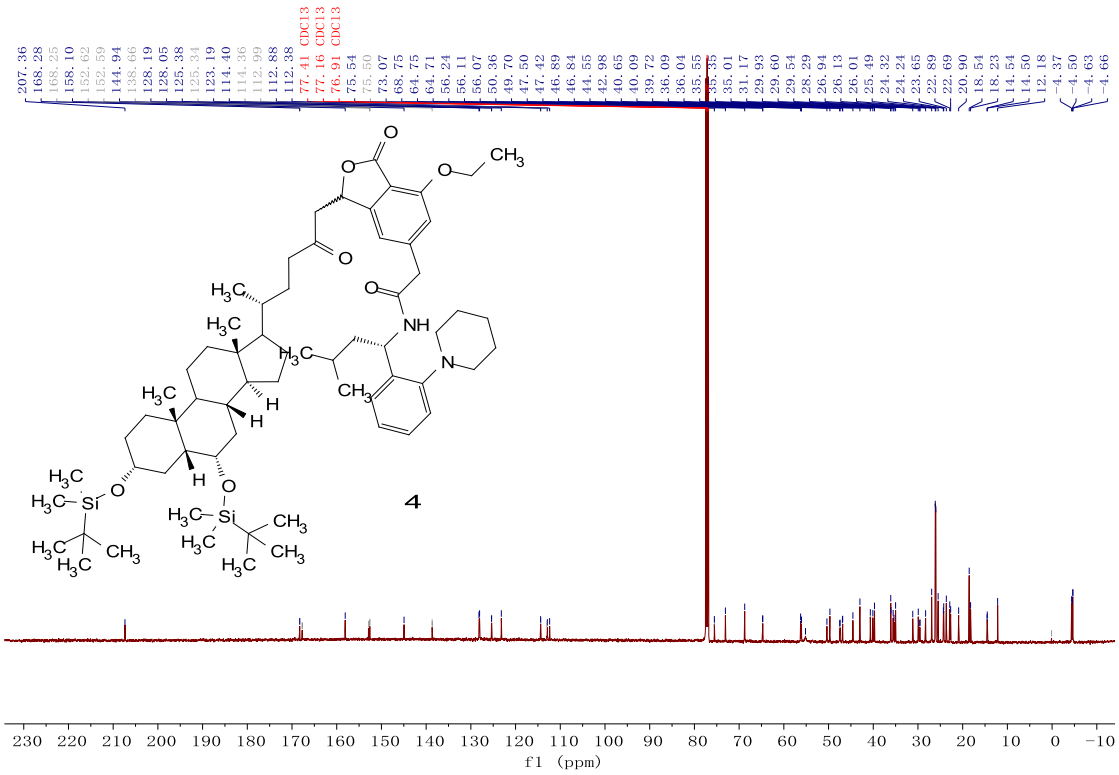
8.29
8.28
8.28
7.46
7.46
7.44
7.44
7.44
7.43
7.42
7.33
7.33
7.31
7.31
7.30
7.26 CDCl3
7.21
7.20
7.20
7.19
7.18
7.09
7.08
7.07
7.07
7.06
6.91
6.90
6.89
6.89
3.18
2.24
1.89
0.93
0.99
1.01
1.76
0.96
0.97
3.00
3.00
3.00
f1 (ppm)


4
f1 (ppm)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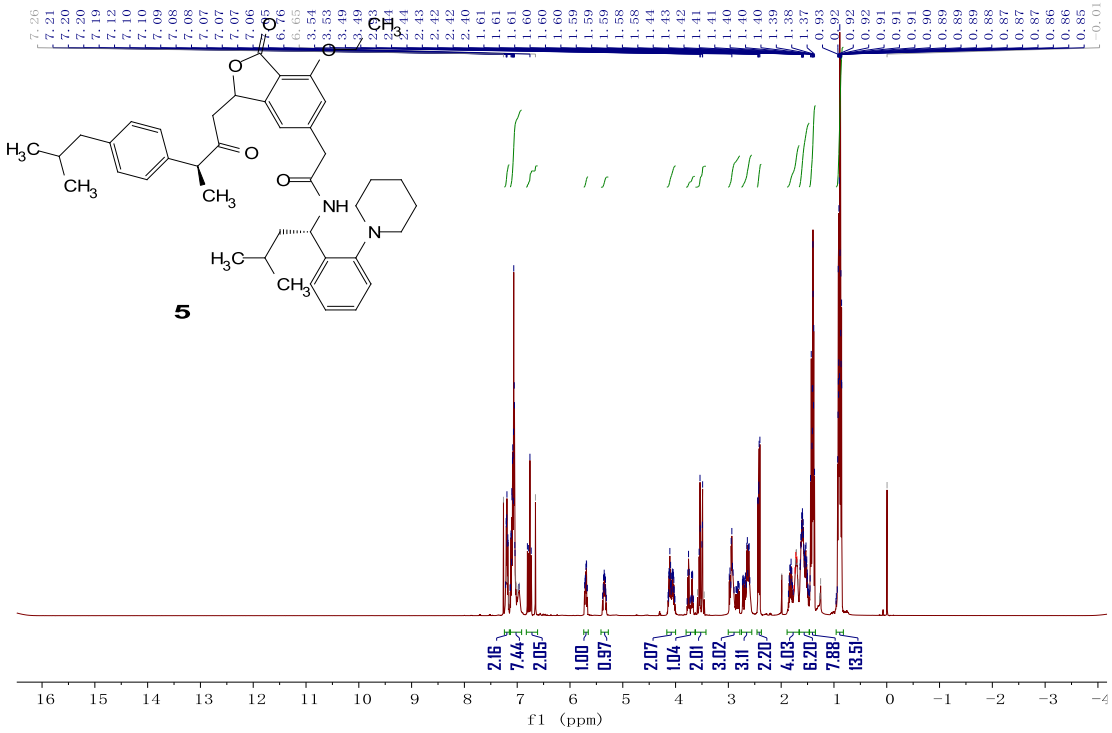
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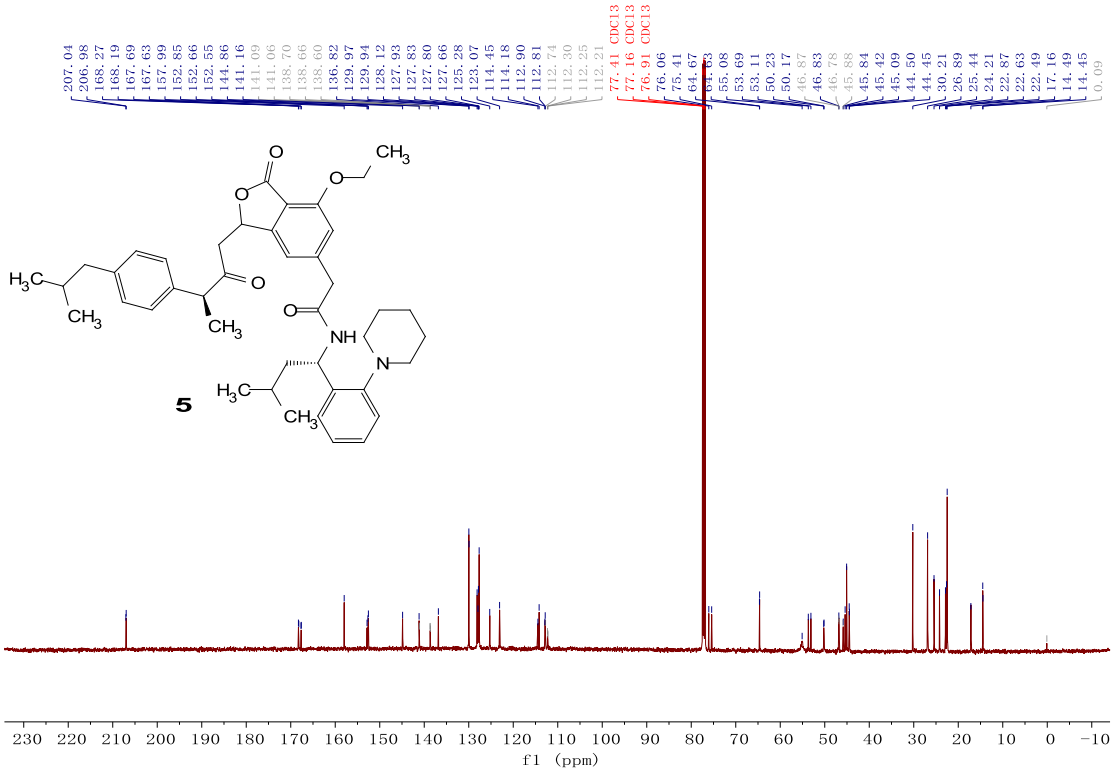
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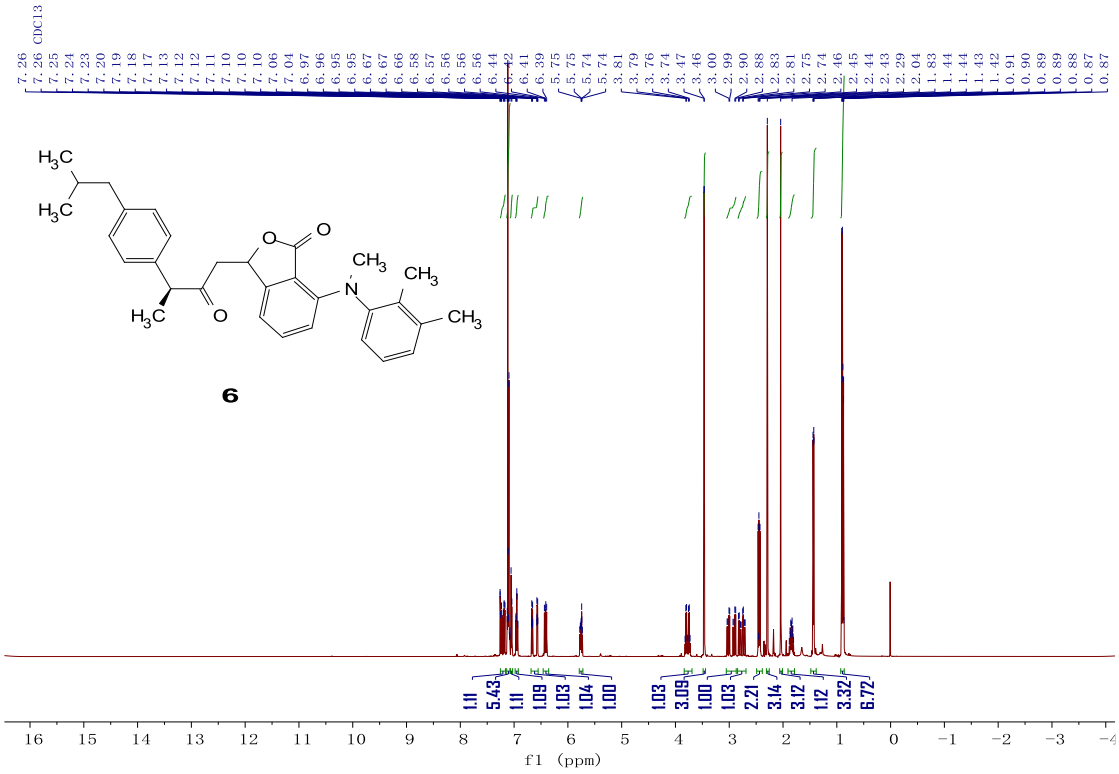
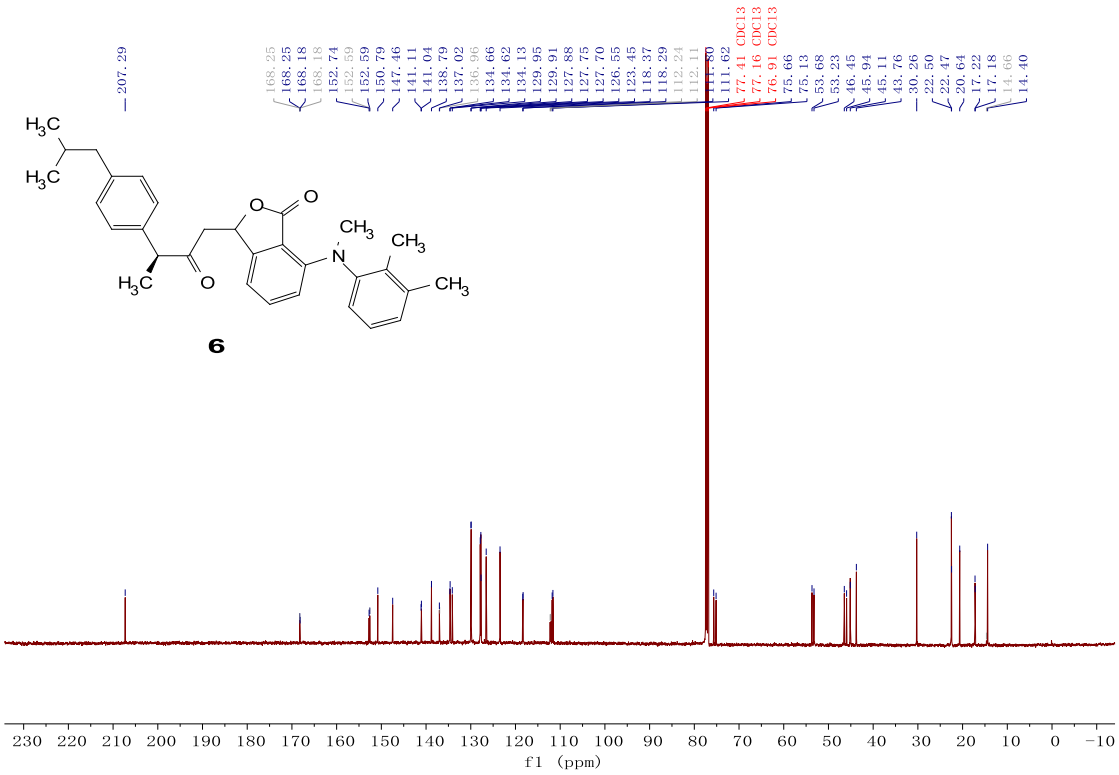
f1 (ppm)



6



6

Reference

1. Zhou, X.; Yu, S.; Kong, L.;Li, X., Rhodium(III)-Catalyzed Coupling of Arenes with Cyclopropanols via C–H Activation and Ring Opening. *ACS Catal.* **2016,** *6*, 647-651.