Supporting Information

Iridium/Nickel Dual Catalyzed Hydroacylation of Hetero-bicyclic Alkenes under Visible-light Irradiation

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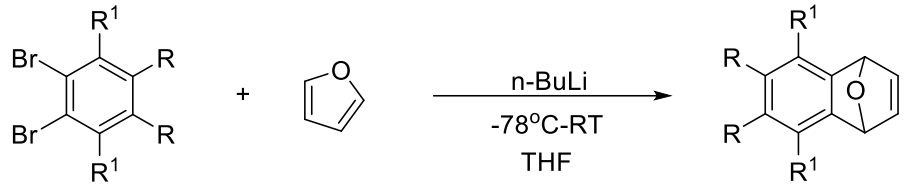
1、General information:

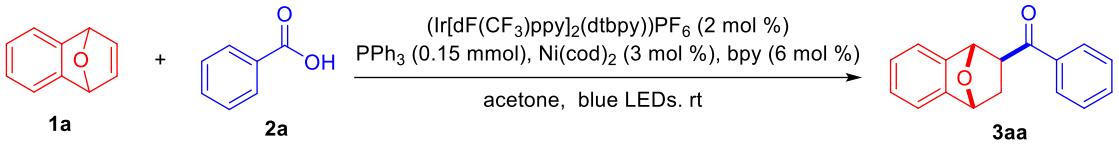
Oxabenzonorbornadienes were synthesized according to the steps in the literature. A series of oxabenzonorbornadienes containing different substituents were synthesized according to literature method. Other commercially available reagents and solvents were purchased and used without further purification. All catalytic experiments were performed under an atmosphere of argon by using Glove Box. ^{1}H NMR (400 MHz) and ^{13}C NMR (101 MHz) spectra were recorded on a Bruker NMR spectrometer in $CDCl_3$ using TMS as an internal reference with chemical shift values reported in ppm. Abbreviations used in the NMR follow-up experiments: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. High-resolution mass spectra (HRMS) were obtained by fast atom bombardment (FAB) using a double focusing magnetic sector mass spectrometer and electrospray ionization electron impact (ESI) ionization technique.

2、General procedure for synthesis of substrates:[1-2]



Preparation of oxabenzonorbornadienes: In a 250 mL three-way flask, 1, 2-dibromobenzene (3 mL, 25 mmol), furan (20 mL, 0.275 mol) and anhydrous tetrahydrofuran (50 mL) were added, and cooled to -78 °C in a low-temperature reactor. *N*-butyl lithium was dropped into the flask (1.6 M in hexane, 16.5 mL, 26.3 mmol), and stirred at -78 °C for 1 hour. Then 50 mL water and 50 mL ethyl acetate were added. The aqueous phase was extracted with ethyl acetate (2×50 mL), the resulting organic phase was combined, washed with saturated salt water. Anhydrous sodium sulfate was added for drying, the solvent was removed by filtration, and the residue was purified by silica gel column chromatography with petroleum ether/ethyl acetate (40:1) to give the product (2.4 g, 69%) as white crystal.

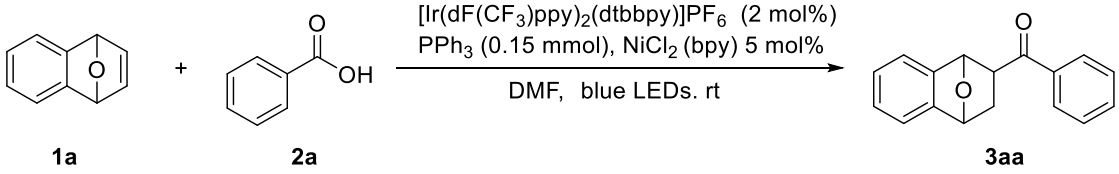
3、General experimental methods:



Under Ar or N_2 atmosphere, a magnetic stirring rod was placed in the oven-dried Schlenk tube. $Ni(cod)_2$ (0.003 mmol, 0.03 equiv) and bpy (0.006 mmol, 0.06 equiv) were added into the tube. After stirred for 30 min, iridium photosensitizer (0.002 mmol, 0.02 equiv), PPh_3(0.15 mmol, 1.5 equiv), benzoic acid (0.1 mmol, 1.0 equiv), oxabenzonorbornadiene (0.2 mmol, 2 equiv) and acetone (1 mL) were added. The mixture was irradiated with a 20 W blue LEDs (450 nm-470 nm) until the starting material disappears monitored the TLC. The reaction mixture was concentrated by vacuum, purified by silica gel chromatography, and eluted by ethyl acetate/petroleum ether to give the products.

4、Investigation of the Key Reaction Parameters

4-1、controlled experiment:

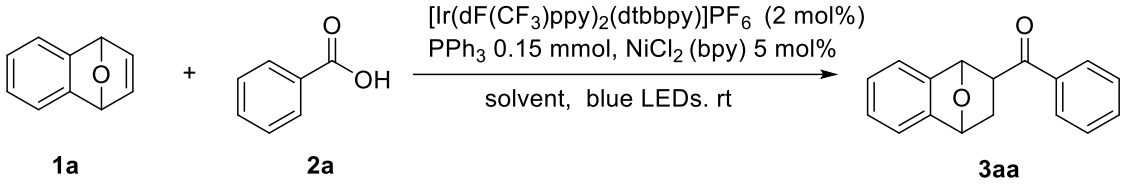


Entry	light source	photocatalyst	additive	Metal	Solvent	Time (h)	Yield(%)[b]
1	Blue LEDs	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	PPh_3	$NiCl_2(bpy)$	DMF	1	30
2	Blue LEDs	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	——	$NiCl_2(bpy)$	DMF	0.5	NR [c]
3	Blue LEDs	——	PPh_3	$NiCl_2(bpy)$	DMF	0.5	NR
4	Blue LEDs	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	PPh_3	——	DMF	1	18
5	Blue LEDs	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	PPh_3	$NiCl_2(bpy)$	DMF	1	28
6	Blue LEDs	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	PPh_3	——	DMF	1.5	9

7	——	[Ir(dF(CF$_3$)ppy)$_2$(dtbbpy)]PF$_6$	PPh_3	$NiCl_2$(bpy)	DMF	20	NR

[a]General conditions: 1a (0.2 mmol), 2a (0.1 mmol,), PPh_3 (0.15 mmol), photocatalyst (2 mol %) and $NiCl_2$ (bpy)(5 mol %) in DMF (1 mL) was irradiated with 20 W blue LEDs at room temperature under argon. [b]Isolated yields are provided. [c]N.R.= no reaction.

4-2、Solvent screening: [a]

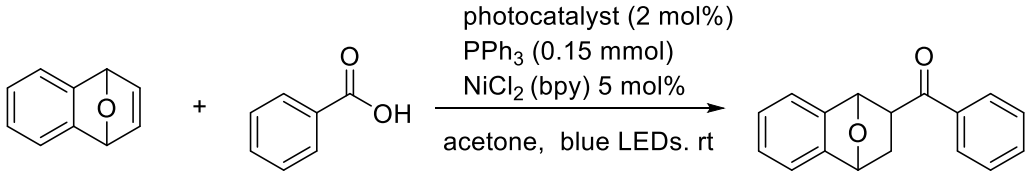


1a **2a** **3aa**

Entry	photocatalyst	additive	Metal	solvent	Time	Yield(%)[b]
1	[Ir(dF(CF$_3$)ppy)$_2$(dtbbpy)]PF$_6$	PPh_3	$NiCl_2$(bpy)	DMF	1	30
2	[Ir(dF(CF$_3$)ppy)$_2$(dtbbpy)]PF$_6$	PPh_3	$NiCl_2$(bpy)	DMAc	4	18
3	[Ir(dF(CF$_3$)ppy)$_2$(dtbbpy)]PF$_6$	PPh_3	$NiCl_2$(bpy)	THF	4	8
4	[Ir(dF(CF$_3$)ppy)$_2$(dtbbpy)]PF$_6$	PPh_3	$NiCl_2$(bpy)	DCE	4	5
5	[Ir(dF(CF$_3$)ppy)$_2$(dtbbpy)]PF$_6$	PPh_3	$NiCl_2$(bpy)	DCM	2	8
6	[Ir(dF(CF$_3$)ppy)$_2$(dtbbpy)]PF$_6$	PPh_3	$NiCl_2$(bpy)	TOl.	12	15
7	[Ir(dF(CF$_3$)ppy)$_2$(dtbbpy)]PF$_6$	PPh_3	$NiCl_2$(bpy)	MeCN	1	33
8	[Ir(dF(CF$_3$)ppy)$_2$(dtbbpy)]PF$_6$	PPh_3	$NiCl_2$(bpy)	$PhCF_3$	15	22
9	[Ir(dF(CF$_3$)ppy)$_2$(dtbbpy)]PF$_6$	PPh_3	$NiCl_2$(bpy)	acetone	18	53
10	[Ir(dF(CF$_3$)ppy)$_2$(dtbbpy)]PF$_6$	PPh_3	$NiCl_2$(bpy)	1,4-dioxane	36	Trace
11	[Ir(dF(CF$_3$)ppy)$_2$(dtbbpy)]PF$_6$	PPh_3	$NiCl_2$(bpy)	EtOAc	10	33

[a]General conditions: 1a (0.2 mmol), 2a (0.1 mmol), PPh_3 (0.15 mmol), photocatalyst (2 mol %) and $NiCl_2$ (bpy)(5 mol %) in solvent (1 mL) was irradiated with 20 W blue LEDs at room temperature under argon. [b]Isolated yields are provided.

4-3、Screening for photosensitizers and sizes: [a]



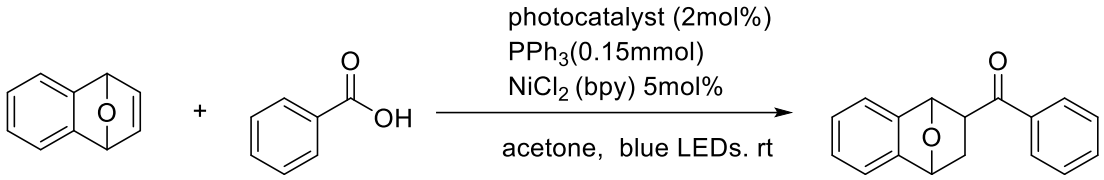
1a **2a** **3aa**

Entry	photocatalyst	PC(%)	additive	Metal	solvent	Time	Yield(%)[b]
1	[Ir(dF(CF$_3$)ppy)$_2$(dtbbpy)]PF$_6$	1	PPh_3	$NiCl_2$(bpy)	acetone	16	24

2	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	2	PPh_3	$NiCl_2(bpy)$	acetone	18	53
3	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	3	PPh_3	$NiCl_2(bpy)$	acetone	16	51
4	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	4	PPh_3	$NiCl_2(bpy)$	acetone	16	48
5	4CZIPN	2	PPh_3	$NiCl_2(bpy)$	acetone	15	trace [d]
6	EosinY	2	PPh_3	$NiCl_2(bpy)$	acetone	38	NR [c]
7	$Ru(bpy)_3{\cdot}6H_2O$	2	PPh_3	$NiCl_2(bpy)$	acetone	38	NR
8	Fac-Ir^{3+}	2	PPh_3	$NiCl_2(bpy)$	acetone	38	NR

[a]General conditions: 1a (0.2 mmol), 2a (0.1 mmol), PPh_3 (0.15 mmol), photocatalyst (2 mol %) and $NiCl_2$ (bpy)(5 mol %) in acetone (1 mL) was irradiated with 20 W blue LEDs at room temperature under argon. [b]Isolated yields are provided. [c] N.R.= no reaction. [d] trace=Raw material is declining but no product is being monitored.

4-4、Screening of the amount of substrate: [a]

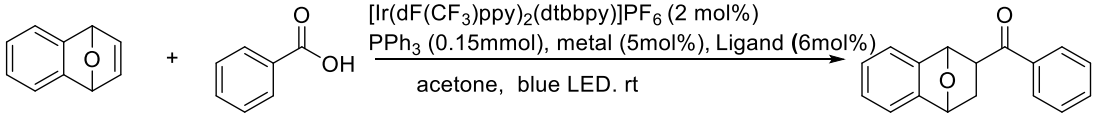


1a **2a** **3aa**

Entry	1a (mmol)	2a (mmol)	photocatalyst	additive	Metal	Solvent	Time	Yield(%)[b]
1	0.1	0.2	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	PPh_3	$NiCl_2(bpy)$	acetone	18	53
2	0.12	0.1	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	PPh_3	$NiCl_2(bpy)$	acetone	18	51
3	0.15	0.1	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	PPh_3	$NiCl_2(bpy)$	acetone	18	60
4	0.2	0.1	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	PPh_3	$NiCl_2(bpy)$	acetone	18	57
5	0.3	0.1	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	PPh_3	$NiCl_2(bpy)$	acetone	18	50

[a]General conditions: 1a (0.1-0.3 mmol), 2a (0.1-0.2 mmol), PPh_3 (0.15 mmol), photocatalyst (2 mol %) and $NiCl_2$ (bpy)(5 mol %) in acetone (1 mL) was irradiated with 20 W blue LEDs at room temperature under argon. [b]Isolated yields are provided.

4-5、Screening of metals and ligands: [a]

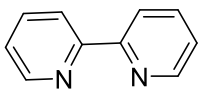


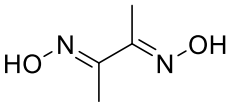
1a **2a** **3aa**

Entry	photocatalyst	additive	Metal	Ligand	solvent	Time	Yield(%)[b]
1	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	PPh_3	$NiCl_2$	**L1**	acetone	18	57

2	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	PPh_3	$Ni(cod)_2$	**L1**	acetone	8	67
3	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	PPh_3	$CoCl_2$	**L2**	acetone	27	37
4	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	PPh_3	$Ni(cod)_2$	**L3**	acetone	6	54
5	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	PPh_3	$Ni(cod)_2$	**L4**	acetone	6	55
6	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	PPh_3	$Ni(cod)_2$	**L5**	acetone	22	25
7	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	PPh_3	$Ni(cod)_2$	**L6**	acetone	22	30
8	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	PPh_3	$Ni(cod)_2$	**L7**	acetone	22	trace[d]
9	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	PPh_3	$Ni(cod)_2$	**L8**	acetone	15	trace

[a]General conditions: 1a (0.15 mmol), 2a (0.1 mmol), PPh_3 (0.15 mmol), photocatalyst (2 mol %) and metal (5 mol %) , ligand(6 mol %) in solvent (1 mL) was irradiated with 20 W blue LEDs at room temperature under argon. [b]Isolated yields are provided. [d] trace=Raw material is declining but no product is being monitored.


L1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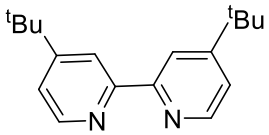
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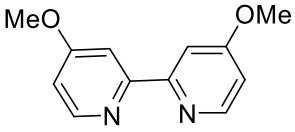
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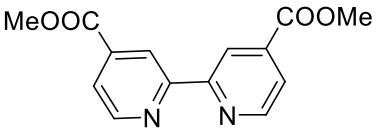
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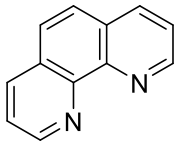
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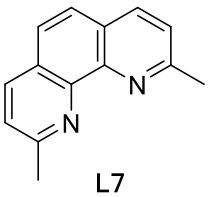
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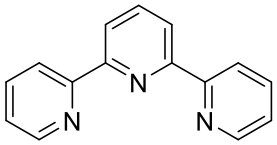
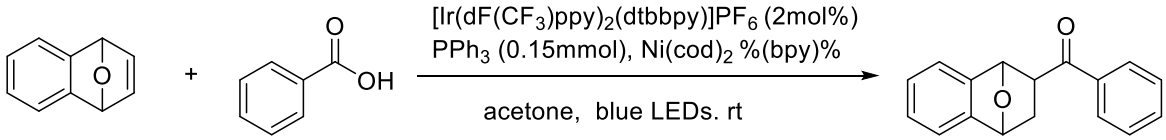

L8

4-6、Screening of metal and composition: [a]



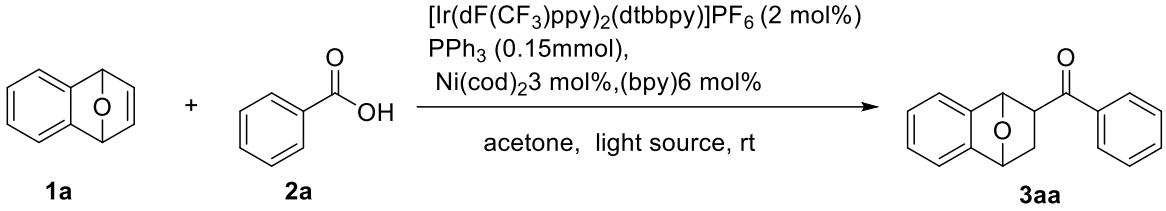
1a **2a** **3aa**

Entry	photocatalyst	additive	Metal (%)	L1 (%)	solvent	Time(h)	Yield(%)[b]
1	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	PPh_3	5	7	acetone	18	68
2	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	PPh_3	5	6	acetone	18	67
3	$[Ir(dF(CF_3)ppy)_2(dtbbpy)]PF_6$	PPh_3	3	4	acetone	18	73
4	$[Ir(dF(CF_3)ppy)_2(d$	PPh_3	3	5	acetone	26	76

	tbbpy)]PF_6						
5	[Ir(dF(CF_3)ppy)$_2$(d tbbpy)]PF_6	PPh_3	3	6	acetone	18	82
6	[Ir(dF(CF_3)ppy)$_2$(d tbbpy)]PF_6	PPh_3	3	7	acetone	26	74
7	[Ir(dF(CF_3)ppy)$_2$(d tbbpy)]PF_6	PPh_3	3	8	acetone	26	69
8	[Ir(dF(CF_3)ppy)$_2$(d tbbpy)]PF_6	PPh_3	3	10	acetone	26	66

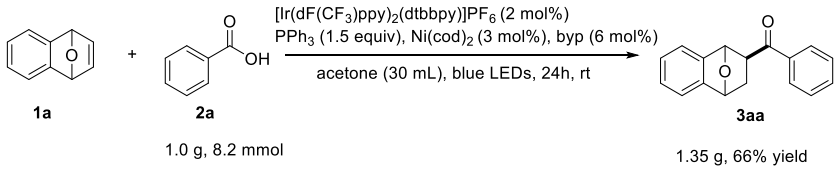
[a]General conditions: 1a (0.15 mmol), 2a (0.1 mmol), PPh_3 (0.15 mmol), photocatalyst (2 mol %) and metal (3 - 5 mol %) , ligand(4 - 10 mol %) in Acetone (1 mL) was irradiated with 20 W blue LEDs at room temperature under argon. [b]Isolated yields are provided.

4-7、Light source screening:



Entry	light source	photocatalyst	additive	Metal (%)	L1 (%)	solvent	Time (h)	Yield(%)[b]
1	Blue LEDs (450nm)	[Ir(dF(CF_3)ppy)$_2$ (dtbbpy)]PF_6	PPh_3	3	6	acetone	18	82
2	purple LEDs (365nm)	[Ir(dF(CF_3)ppy)$_2$ (dtbbpy)]PF_6	PPh_3	3	6	acetone	18	60
3	purple LEDs (390nm)	[Ir(dF(CF_3)ppy)$_2$ (dtbbpy)]PF_6	PPh_3	3	6	acetone	18	61
4	green LEDs (530nm)	[Ir(dF(CF_3)ppy)$_2$ (dtbbpy)]PF_6	PPh_3	3	6	acetone	26	ND

5、Gram-scale reaction

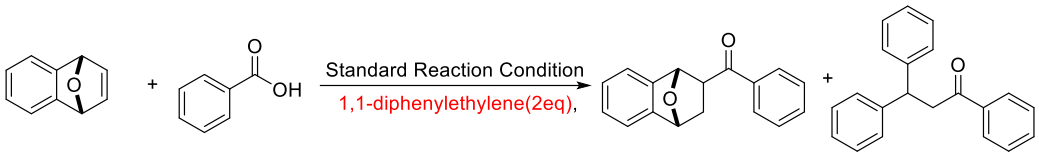


Oxabenzonorbornadiene **1a** (1.78 g, 12.3 mmol, 1.5 equiv), benzoic acid **2a** (1.0 g, 8.2 mmol, 1.5 equiv), (Ir[dF(CF_3)ppy]$_2$(dtbpy))PF_6 (0.18 g, 2 mol%), PPh_3 (3.2 g, 12.3 mmol, 1.5 equiv), Ni(cod)$_2$ (0.074 g, 3 mol%,), bpy (0.082 g, 6 mol%), acetone (30 mL)

were added to a 100 mL round-bottomed flask. The reaction bottle was stirred under 20 W blue light and cooled by a fan. TLC was used to monitor the reaction process, and the reaction liquid was concentrated by vacuum and the reaction products were purified by silica gel column. The phenyl (1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl) methanone was obtained in 66% yield.

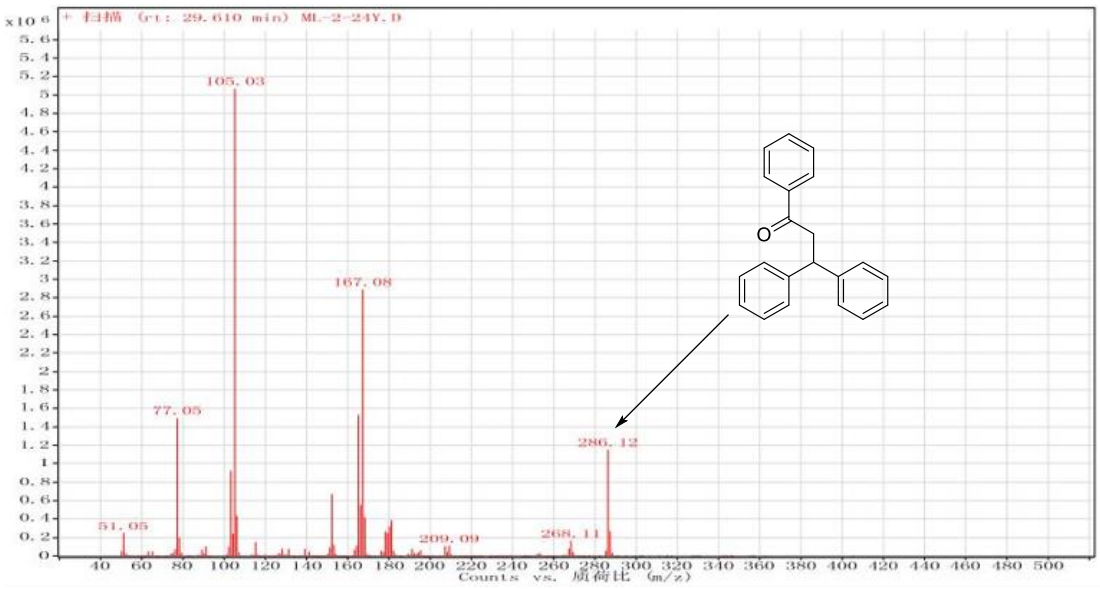
6、Mechanistic studies.

6-1、Free radical trapping experiment：



1a **2a** **3aa(Not Detected)** **6a**,*detected by GC*

Under Ar or N_2 atmosphere, a magnetic stirring rod was placed in the oven-dried Schlenk tube. $Ni(cod)_2$ (0.003 mmol, 0.02 equiv) and bpy (0.006 mmol, 0.06 equiv) were added into the tube and stirred for 30 min. Iridium photosensitizer (0.002 mmol, 0.02 equiv), PPh_3(0.15 mmol,1.5 equiv), benzoic acid (0.1 mmol, 1.0 equiv), oxabenzonorbornadiene (0.15 mmol, 1.5 equiv)、1,1-diphenylethylene (0.2 mmol, 2 equiv) and acetone (1 mL) were added. The mixture was irradiated at room temperature with a 20W blue LED (460 nm-470 nm) until the starting material disappears from the TLC. The analysis of the resulting mixture by GC-MS has detected the molecular weight of **6a**.



6-2、 Measurement of Quantum Yields：

The photon flux of blue LED was determined by standard ferrioxalate actinometry.

0.15 mol/L solution of ferrioxalate was prepared by dissolving potassium ferrioxalate hydrate (328 mg, 0.750 mmol) in 5.0 mL of 0.20 mol/L aqueous sulfuric acid.

0.15 mol/L buffered solution of 1,10-phenanthroline was prepared by dissolving 1,10-phenanthroline (54.1 mg, 0.3 mmol) and sodium acetate (1.23 g, 15.0 mmol) in 20 mL of 0.20 mol/L aqueous sulfuric acid. The actinometry measurements were done as follows: To a reaction tube equipped with a stir bar was added 0.50 mL of the ferrioxalate solution. The reaction tube was sealed and placed 2 cm away from a 20 W blue LEDs. After irradiation for 5 seconds, 1.5 mL of the aqueous sulfuric acid and 2.0 mL of the buffered solution was added to the reaction tube. The solution was then allowed to rest for 1 hour to allow the resultant ferrous ions to react completely with 1,10-phenanthroline. 50 μL of the resulting solution was taken as an aliquot and diluted with 3.0 mL of 0.20 mol/L aqueous sulfuric acid. The absorbance of the resulting solution in a cuvette (l = 1.0 cm) at 510 nm was measured by UV-V is spectrometer. A non-irradiated sample was also prepared and the absorbance at 510 nm was measured.

The amount of ferrous ion formed was calculated as follows:

$$\text{mol}\,Fe^{2+} = \frac{V \times \Delta A}{l \times \varepsilon}$$

where V is the total volume (0.024 L) of the solution that was analyzed, ΔA is the difference in absorbance at 510 nm between the irradiated and non-irradiated samples, l is the path length (1.00 cm), and ε is the molar absorptivity at 510 nm (11,100 L/(mol•cm)).

The photon flux was calculated as follows:

$$\text{photo flux} = \frac{\text{mol } Fe^{2+}}{\Phi \times t \times f}$$

where Φ is the quantum yield for the ferrioxalate actinometer (approximated as 0.845, which was reported for a 0.15 mol/L solution at λ = 457.9 nm) , t is the irradiation time, and f is the fraction of light absorbed at 450 nm (0.4355).

The fraction of light absorbed was determined by the following equation:

$$f = 1.0000\text{-}10^{-A}$$

where A is the measured absorbance (0.2483) of the 0.15 mol/L solution of potassium ferrioxalate at 450 nm.

The photo flux is 1.49 × 10^{-7} Einstein/s.

Determination of quantum yield:

In an oven-dried reaction tube containing a magnetic stirring bar was charged with a sample of oxabenzonorbornadiene **1a** (0.15 mmol), benzoic acid **2a** (0.1 mmol) , (Ir[dF(CF_3)ppy]$_2$(dtbpy))PF_6 (2 mol%), PPh_3 (0.15 mmol), Ni(cod)$_2$ (3 mol%,), bpy (6% mol,), Acetone (1mL). The reaction mixture was stirred under the irradiation of 20 W LED at room temperature and placed 2 cm away from 20 W blue LEDs. After irradiation for 3 hours, the moles of product **3aa** formed for the model reaction were determined by GC measurement using tridecane as internal standard, and revealed 33% yield of **3aa** (3.3×10^{-5} mol).

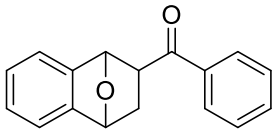
The quantum yield was calculated as follows:

$$\Phi = \frac{\text{mol product}}{flux \times t \times f}$$

where flux is the photon flux determined by ferrioxalate actinometry (**1.49 × 10^{-7}** Einstein/s), t is the time, and f is the fraction of light absorbed by the irradiated reaction system at 450 nm, and the absorbance of the irradiated reaction system at 450 nm was 0.041. The fraction of light absorbed at 450 nm was calculated: f = 1.0000 – 10^{-A} = 1.0000 – $10^{-0.041}$ = 0.090.

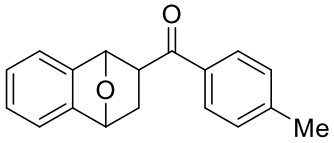
The quantum yield was calculated: Φ = 0.23

7、Characterization data of products.



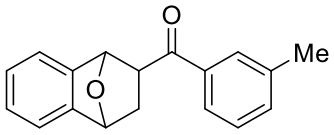
phenyl(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)methanone (3aa)

20.5 mg, 82% yield, yellow oil, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.00 – 7.92 (m, 2H), 7.63 – 7.54 (m, 1H), 7.53 – 7.44 (m,2H), 7.39 – 7.32 (m,1H), 7.31 (dd, *J* = 5.2, 3.2 Hz, 1H), 7.22 (dd, *J* = 5.3, 3.0 Hz, 2H), 5.70 (s, 1H), 5.50 (d, *J* = 4.9 Hz, 1H), 3.44 (dd, *J* = 8.9, 4.7 Hz, 1H), 2.47 (dt, *J* = 11.7, 4.9 Hz, 1H), 1.88 (dd, *J* = 11.7, 8.9 Hz, 1H).
^{13}C NMR (101 MHz, Chloroform-d) δ 198.8, 146.4, 144.9, 136.4, 133.4, 128.9, 128.7, 127.2, 127.0, 119.5, 119.1, 80.8, 79.3, 48.4, 32.5.
HRMS-ESI$^+$ (m/z) calcd for $C_{17}H_{14}O_2Na^+$ $[M+Na]^+$ 273.0886 , found 273.0891.



(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)(*p*-tolyl)methanone (3ab)

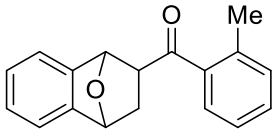
20.6 mg, 78% yield，yellow oil , ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.79 – 7.75 (m, 2H), 7.28 – 7.25 (m, 1H), 7.24 – 7.20 (m, 2H), 7.18 (d, *J* = 2.4 Hz, 1H), 7.13 (dd, *J* = 5.3, 3.1 Hz, 2H), 5.60 (s, 1H), 5.41 (d, *J* = 4.9 Hz, 1H), 3.33 (dd, *J* = 8.9, 4.7 Hz, 1H), 2.41 – 2.36 (m,1H), 2.34 (s, 3H), 1.79 (dd, *J* = 11.6, 8.9 Hz, 1H).
^{13}C NMR (101 MHz, Chloroform-d) δ 198.5, 146.4, 145.0, 144.2, 133.9, 129.6, 128.8, 127.2, 127.0, 119.5, 119.1, 80.8, 79.3, 48.2, 32.5, 21.8.
HRMS-ESI$^+$ (m/z) calcd for $C_{18}H_{17}O_2^+$ $[M+H]^+$ 265.1223, found 265.1225.



(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)(*m*-tolyl)methanone (3ac)

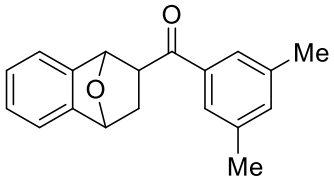
20.4 mg, 77% yield, yellow oil, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.58 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.42 (td, *J* = 7.5, 1.4 Hz, 1H), 7.34 – 7.32 (m, 2H), 7.31 (d, *J* = 4.7 Hz, 2H), 7.28 (d, *J* = 1.7 Hz, 1H), 7.23 – 7.20 (m, 2H), 5.65 (s, 1H), 5.51 (d, *J* = 5.0 Hz, 1H), 3.39 (dd, *J* = 8.8, 4.6 Hz, 1H), 2.57 (s, 3H), 2.47 (dt, *J* = 11.7, 4.9 Hz, 1H), 1.81 (dd, *J* = 11.7, 8.9 Hz, 1H).

^{13}C NMR (101 MHz, Chloroform-d) δ 202.7, 146.4, 144.9, 139.1, 137.5, 132.4, 131.6, 128.5, 127.2, 127.0, 125.9, 119.4, 119.1, 80.8, 79.4, 50.8, 32.1, 21.6.
HRMS-ESI$^+$ (m/z) calcd for $C_{18}H_{16}O_2Na^+$ [M+Na]$^+$ 287.1043, found 287.1044.



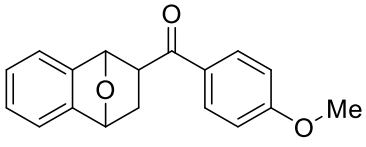
(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)(*o*-tolyl)methanone (3ad)

20.4 mg,72% yield, yellow oil, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.72 – 7.62 (m, 2H), 7.31 (d, *J* = 3.4 Hz, 1H), 7.31 – 7.24 (m, 2H), 7.23 (dd, *J* = 5.2, 3.2 Hz, 1H), 7.19 – 7.11 (m, 2H), 5.61 (s, 1H), 5.42 (d, *J* = 4.9 Hz, 1H), 3.35 (dd, *J* = 8.9, 4.7 Hz, 1H), 2.38 (dt, *J* = 11.8, 4.9 Hz, 1H), 2.34 (s, 3H), 1.80 (dd, *J* = 11.6, 9.0 Hz, 1H).
^{13}C NMR (101 MHz, Chloroform-d) δ 199.1, 146.4, 145.0, 138.8, 136.5, 134.2, 129.2, 128.8, 127.2, 127.1, 125.9, 119.5, 119.1, 80.8, 79.3, 48.4, 32.5, 21.6.
HRMS-ESI$^+$ (m/z) calcd for $C_{18}H_{16}O_2Na^+$ [M+Na]$^+$ 287.1043, found 287.1044.



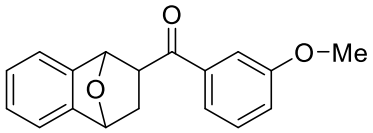
(3,5-dimethylphenyl)(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)methanone (3ae)

17.8 mg, 64% yield, light yellow solid, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55 (d, *J* = 1.7 Hz, 2H), 7.38 – 7.32 (m, 1H), 7.31 (dd, *J* = 5.5, 3.0 Hz, 1H), 7.22 (dd, *J* = 5.3, 3.0 Hz, 3H), 5.69 (s, 1H), 5.50 (d, *J* = 5.0 Hz,1H), 3.42 (dd, *J* = 9.0, 4.8 Hz, 1H), 2.43 (dt, *J* = 11.6, 4.9 Hz, 1H), 2.37 (s, 6H), 1.88 (dd, *J* = 11.7, 9.0 Hz, 1H).
^{13}C NMR (101 MHz, Chloroform-d) δ 199.3, 146.5, 145.2, 138.5, 136.7, 135.0, 127.2, 127.0, 126.5, 119.5, 119.0, 80.9, 79.3, 48.5, 32.6, 21.5.
MP: 139 ^{o}C
HRMS-ESI$^+$ (m/z) calcd for $C_{19}H_{18}O_2Na^+$ [M+Na]$^+$301.1199, found 301.1203.



(4-methoxyphenyl)(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)methanone (3af)

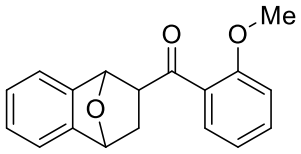
20.5 mg, 73% yield; white solid, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.95 – 7.91 (m, 2H), 7.36 – 7.33 (m, 1H), 7.30 (dd, *J* = 5.2, 3.2 Hz,1H), 7.21 (dd, *J* = 5.3, 3.1 Hz, 2H), 6.97 – 6.93 (m, 2H), 5.68 (s, 1H), 5.49 (d, *J* = 5.0 Hz,1H), 3.87 (s, 3H), 3.38 (dd, *J* = 8.9, 4.7 Hz, 1H), 2.45 (dt, *J* = 11.6, 4.9 Hz, 1H), 1.86 (dd, *J* = 11.6, 8.9 Hz, 1H).
^{13}C NMR (101 MHz, Chloroform-d) δ 197.5, 163.7, 146.4, 145.1, 130.9, 129.4, 127.2, 127.0, 119.5, 119.1, 114.1, 80.9, 79.2, 55.7, 48.0, 32.6.
MP: 102 °C
HRMS-ESI$^+$ (m/z) calcd for $C_{18}H_{17}O_3^+$ [M+H]$^+$ 281.1172, found 281.1177.



(3-methoxyphenyl)(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)methanone

(3ag)

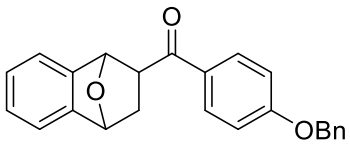
21.9 mg, 78% yield, yellow oil, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.46 – 7.37 (m, 2H), 7.34 – 7.24 (m, 2H), 7.23 (dd, *J* = 5.3, 3.1 Hz, 1H), 7.13 (dd, *J* = 5.3, 3.0 Hz, 2H), 7.04 (ddd, *J* = 8.2, 2.7, 1.0 Hz,1H), 5.60 (s, 1H), 5.42 (d, *J* = 4.9 Hz, 1H), 3.77 (s, 3H), 3.33 (dd, *J* = 8.9, 4.7 Hz,1H), 2.42 – 2.36 (m, 1H), 1.78 (dd, *J* = 11.7, 8.9 Hz, 1H).
^{13}C NMR (101 MHz, Chloroform-d) δ 198.7, 160.1, 146.3, 144.9, 137.8, 129.8, 127.2, 127.0, 121.1, 119.8, 119.5, 119.1, 113.0, 80.8, 79.3, 55.6, 48.5, 32.4.
HRMS-ESI$^+$ (m/z) calcd for $C_{18}H_{16}O_3Na^+$ [M+Na]$^+$ 281.1172, found 281.1177.



(2-methoxyphenyl)(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)methanone

(3ah)

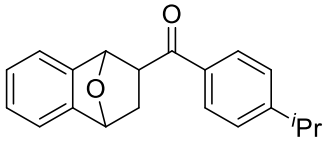
20.8 mg, 74% yield, yellow oil, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.82 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.51 (ddd, *J* = 8.4, 7.3, 1.9 Hz, 1H), 7.30 (ddd, *J* = 8.3, 6.2, 3.3 Hz, 2H), 7.23 – 7.16 (m, 2H), 7.10 – 6.98 (m, 2H), 5.57 (s, 1H), 5.47 (d, *J* = 5.1 Hz, 1H), 3.93 (s, 3H), 3.56 (dd, *J* = 8.5, 4.4 Hz, 1H), 2.67 (d, *J* = 11.7 Hz, 1H), 1.67 (dd, *J* = 11.7, 8.6 Hz, 1H).
^{13}C NMR (101 MHz, Chloroform-d) δ 200.6, 158.5, 146.8, 145.5, 134.0, 131.5, 127.5, 126.9, 126.7, 121.2, 119.3, 119.0, 111.7, 81.8, 79.5, 55.7, 53.2, 31.0.
HRMS-ESI$^+$ (m/z) calcd for $C_{18}H_{17}O_3^+$ [M+H]$^+$ 281.1172, found 281.1173.



(4-(benzyloxy)phenyl)(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)methanone

(3ai)

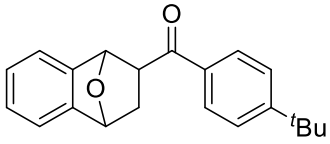
29.6 mg, 83% yield, yellow oil, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.97 – 7.89 (m, 2H), 7.45 (d, *J* = 2.0 Hz, 1H), 7.45 – 7.39 (m, 2H), 7.43 – 7.36 (m, 1H), 7.39 – 7.31 (m, 2H), 7.30 (dd, *J* = 5.2, 3.1 Hz, 1H), 7.21 (dd, *J* = 5.3, 3.0 Hz, 2H), 7.06 – 6.98 (m, 2H), 5.68 (s, 1H), 5.49 (d, *J* = 4.9 Hz, 1H), 5.13 (s, 2H), 3.38 (dd, *J* = 8.9, 4.7 Hz, 1H), 2.45 (dt, *J* = 11.6, 4.9 Hz, 1H), 1.86 (dd, *J* = 11.6, 8.9 Hz, 1H).
^{13}C NMR (101 MHz, Chloroform-d) δ 197.5, 162.9, 146.4, 145.1, 136.3, 130.9, 129.6, 128.9, 128.5, 127.7, 127.2, 127.0, 119.5, 119.1, 114.9, 80.9, 79.2, 70.4, 48.0, 32.6.
HRMS-ESI$^+$ (m/z) calcd for $C_{24}H_{21}O_3^+$ [M+H]$^+$ 357.1485, found 357.1489.



(4-isopropylphenyl)(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)methanone

(3aj)

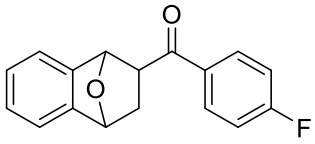
23.4 mg, 80% yield,yellow oil, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.91 – 7.87 (m, 2H), 7.37 – 7.33 (m, 2H), 7.33 – 7.30 (m, 2H), 7.22 (dd, *J* = 5.3, 3.0 Hz, 2H), 5.69 (s, 1H), 5.50 (d, J = 4.9 Hz, 1H), 3.42 (dd, *J* = 8.9, 4.7 Hz, 1H), 2.98 (p, *J* = 6.9 Hz, 1H), 2.48 (dt, *J* = 11.6, 4.9 Hz, 1H), 1.87 (dd, *J* = 11.7, 8.9 Hz, 1H), 1.28 (d, *J* = 6.9 Hz, 6H).
^{13}C NMR (101 MHz, Chloroform-d) δ 198.4, 154.9, 146.5, 145.2, 134.4, 128.9, 127.18, 127.0, 127.0, 119.5, 119.1, 81.0, 79.3, 48.3, 34.5, 32.4, 23.8.
HRMS-ESI$^+$ (m/z) calcd for $C_{20}H_{21}O_2^+$ [M+H]$^+$ 293.1536, found 293.1539.



(4-(tert-butyl)phenyl)(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)methanone

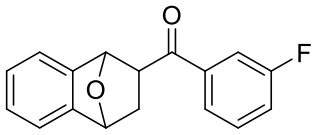
(3ak)

25.1 mg, 82% yield,yellow oil, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.93 – 7.86 (m, 2H), 7.53 – 7.46 (m, 2H), 7.39 – 7.33 (m, 1H), 7.31 (dd, *J* = 5.3, 3.1 Hz, 1H), 7.26 – 7.18 (m, 2H), 5.69 (s, 1H), 5.50 (d, *J* = 4.9 Hz, 1H), 3.43 (dd, *J* = 8.9, 4.7 Hz, 1H), 2.49 (dt, *J* = 11.6, 4.9 Hz, 1H), 1.86 (dd, *J* = 11.6, 8.9 Hz, 1H), 1.35 (s, 9H).
^{13}C NMR (101 MHz, Chloroform-d) δ 198.4, 157.2, 146.5, 145.1, 133.9, 128.7, 127.2, 127.0, 125.9, 119.5, 119.1, 81.0, 79.3, 48.3, 35.3, 32.4, 31.3.
HRMS-ESI$^+$ (m/z) calcd for $C_{21}H_{23}O_2^+$ [M+H]$^+$ 307.1693, found307.1695.



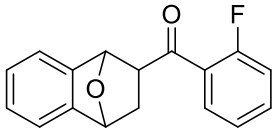
(4-fluorophenyl)(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)methanone (3al)

20.7 mg, 77% yield, yellow oil , 1H NMR (400 MHz, Chloroform-*d*) δ 8.00 – 7.95 (m, 2H), 7.34 (dd, *J* = 5.3, 3.1 Hz, 1H), 7.31 (dd, *J* = 5.3, 3.3 Hz, 1H), 7.22 (dd, *J* = 5.3, 3.0 Hz, 2H), 7.16 – 7.12 (m, 2H), 5.68 (s, 1H), 5.50 (d, *J* = 4.9 Hz, 1H), 3.39 (dd, *J* = 8.9, 4.7 Hz, 1H), 2.43 (dt, *J* = 11.6, 4.9 Hz, 1H), 1.89 (dd, *J* = 11.6, 9.0 Hz, 1H).
^{13}C NMR (101 MHz, Chloroform-d) δ 197.4, 166.0 (d, *J* = 255.1 Hz), 146.3, 144.8, 132.8 (d, *J* = 3.1 Hz), 131.3, 131.2, 127.3, 127.1, 119.6, 119.1, 116.2, 116.0, 80.7, 79.3, 48.3, 32.6.
^{19}F NMR (376 MHz, Chloroform-d) δ -104.84.
HRMS-ESI+ (m/z) calcd for $C_{17}H_{13}FO_2Na^+$ [M+Na]$^+$ 291.0792, found 291.0794.



(3-fluorophenyl)(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)methanone (3am)

17.2 mg, 64% yield, yellow oil, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.72 (dt, *J* = 7.9, 1.3 Hz, 1H), 7.64 (ddd, *J* = 9.4, 2.6, 1.6 Hz, 1H), 7.46 (td, *J* = 8.0, 5.5 Hz, 1H), 7.36 (dd, *J* = 5.2, 3.1 Hz, 1H), 7.31 (dd, *J* = 5.2, 3.2 Hz, 1H), 7.30 – 7.27 (m, 1H), 7.22 (dd, *J* = 5.3, 3.0 Hz, 2H), 5.69 (s, 1H), 5.51 (d, *J* = 4.9 Hz, 1H), 3.39 (dd, *J* = 8.9, 4.7 Hz, 1H), 2.44 (dt, *J* = 11.7, 4.9 Hz, 1H), 1.89 (dd, *J* = 11.6, 9.0 Hz, 1H).
^{13}C NMR (101 MHz, Chloroform-d) δ 196.5 (d, J = 2.1 Hz), 161.9 (d, *J* = 248.3 Hz), 145.0, 143.5, 137.3 (d, *J* = 6.1 Hz), 129.4 (d, *J* = 7.7 Hz), 126.1, 125.9, 123.1 (d, *J* = 3.1 Hz), 119.2 (d, *J* = 21.4 Hz), 118.3, 117.9, 114.3 (d, *J* = 22.4 Hz), 79.4, 78.1, 47.4, 31.3.
^{19}F NMR (376 MHz, Chloroform-d) δ -111.38.
HRMS-ESI$^+$ (m/z) calcd for $C_{17}H_{13}FO_2Na^+$ [M+Na]$^+$ 291.0792, found 291.0793.



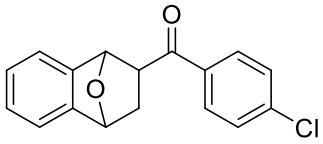
(2-fluorophenyl)(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)methanone (3an)

20.4 mg, 76% yield, yellow oil, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.99 (td, *J* = 7.7, 1.9 Hz, 1H), 7.57 (dddd, *J* = 8.9, 7.2, 5.0, 1.9 Hz, 1H), 7.40 – 7.34 (m, 1H), 7.28 (td, *J* = 7.0, 6.2, 1.3 Hz, 2H), 7.25 – 7.17 (m, 2H), 7.21 – 7.13 (m, 1H), 5.65 (d, *J* = 3.2 Hz, 1H), 5.51 (d, *J* = 5.0 Hz, 1H), 3.42 (dt, *J* = 8.7, 4.4 Hz, 1H), 2.68 (dtd, *J* = 11.4, 4.8, 1.4 Hz, 1H), 1.74 (ddd, *J* = 11.8, 8.6, 0.9 Hz, 1H).

^{13}C NMR (101 MHz, Chloroform-d) δ 195.3 (d, *J* = 4.4 Hz), 160.8 (d, *J* = 253.4 Hz), 145.2, 143.8, 133.9 (d, *J* = 9.2 Hz), 130.5 (d, *J* = 2.6 Hz), 125.9, 125.8, 123.8 (d, *J* = 3.3 Hz), 118.1, 118.1, 115.8, 115.6, 80.1 (d, *J* = 4.1 Hz), 78.3, 52.2 (d, *J* = 6.1 Hz), 29.5 (d, *J* = 1.6 Hz).

^{19}F NMR (376 MHz, Chloroform-d) δ -109.01.

HRMS-ESI$^+$ (m/z) calcd for $C_{17}H_{13}FO_2Na^+$ [M+Na]$^+$ 291.0792, found 291.0796

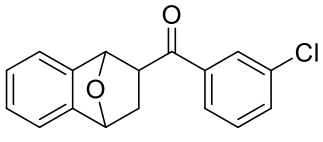


(4-chlorophenyl)(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)methanone (3ao)

14.8 mg, 52% yield, yellow oil, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.93 – 7.85 (m, 2H), 7.49 – 7.42 (m, 2H), 7.34 (dd, *J* = 5.2, 3.1 Hz, 1H), 7.34 – 7.28 (m, 1H), 7.22 (dd, *J* = 5.3, 3.0 Hz, 2H), 5.68 (s, 1H), 5.50 (d, *J* = 4.9 Hz, 1H), 3.38 (dd, *J* = 8.9, 4.7 Hz, 1H), 2.42 (dt, *J* = 11.7, 4.9 Hz, 1H), 1.89 (dd, *J* = 11.6, 9.0 Hz, 1H).

^{13}C NMR (101 MHz, Chloroform-d) δ 197.8, 146.3, 144.8, 139.9, 134.7, 130.1, 129.3, 127.3, 127.2, 119.6, 119.1, 80.6, 79.3, 48.4, 32.6.

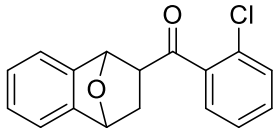
HRMS-ESI$^+$ (m/z) calcd for $C_{17}H_{14}ClO_2^+$ [M+H]$^+$ 285.0677, found 285.0679.



(3-chlorophenyl)(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)methanone (3ap)

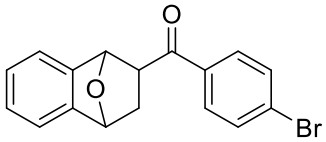
15.7 mg, 55% yield, yellow oil, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.91 (t, *J* = 1.9 Hz, 1H), 7.82 (dt, *J* = 7.8, 1.4 Hz, 1H), 7.56 (ddd, *J* = 7.9, 2.1, 1.1 Hz, 1H), 7.43 (t, *J* = 7.9 Hz, 1H), 7.35 (dd, *J* = 5.1, 3.2 Hz, 1H), 7.32 (dd, *J* = 5.2, 3.2 Hz, 1H), 7.23 (dd, *J* = 5.3, 3.0

Hz, 2H), 5.69 (s, 1H), 5.51 (d, *J* = 4.9 Hz, 1H), 3.38 (dd, *J* = 9.0, 4.7 Hz, 1H), 2.42 (dt, *J* = 11.7, 4.9 Hz, 1H), 1.90 (dd, *J* = 11.7, 9.0 Hz, 1H).
^{13}C NMR (101 MHz, Chloroform-d) δ 197.7, 146.3, 144.7, 138.0, 135.3, 133.4, 130.3, 128.8, 127.4, 127.2, 126.7, 119.6, 119.2, 80.6, 79.3, 48.5, 32.6.
HRMS-ESI$^+$ (m/z) calcd for $C_{17}H_{14}ClO_2^+$ [M+H]$^+$ 285.0677, found 285.0681.



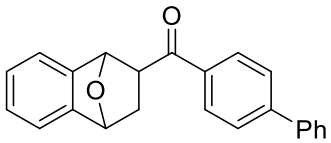
(2-chlorophenyl)(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)methanone (3aq)

16.8 mg, 59% yield, yellow oil, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55 (dd, *J* = 7.5, 1.6 Hz, 1H), 7.51 – 7.41 (m, 2H), 7.39 (td, *J* = 7.0, 2.1 Hz, 1H), 7.35 – 7.26 (m, 2H), 7.26 – 7.16 (m, 2H), 5.65 (s, 1H), 5.53 (d, *J* = 5.0 Hz, 1H), 3.51 (dd, *J* = 8.7, 4.5 Hz, 1H), 2.56 (dt, *J* = 11.9, 4.8 Hz, 1H), 1.80 (dd, *J* = 11.8, 8.7 Hz, 1H).
^{13}C NMR (101 MHz, Chloroform-d) δ 202.5, 146.3, 144.7, 139.2, 132.1 (d, *J* = 2.0 Hz), 131.1, 130.8, 129.9, 127.4, 127.3, 127.0, 119.4, 119.3, 81.2, 79.5, 52.6, 31.9.
HRMS-ESI$^+$ (m/z) calcd for $C_{17}H_{14}ClO_2^+$ [M+H]$^+$ 285.0677, found 285.0678.



(4-bromophenyl)(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)methanone (3ar)

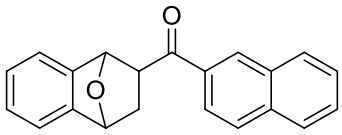
12.5 mg, 38% yield, yellow oil, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.83 – 7.78 (m, 2H), 7.65 – 7.61 (m, 2H), 7.34 (dd, *J* = 5.2, 3.1 Hz, 1H), 7.32 – 7.29 (m, 1H), 7.22 (dd, *J* = 5.3, 3.0 Hz, 2H), 5.68 (s, 1H), 5.50 (d, *J* = 4.9 Hz, 1H), 3.37 (dd, J = 8.9, 4.7 Hz, 1H), 2.42 (dt, *J* = 11.6, 4.9 Hz, 1H), 1.88 (dd, *J* = 11.7, 9.0 Hz, 1H).
^{13}C NMR (101 MHz, Chloroform-d) δ 198.0, 146.3, 144.8, 135.1, 132.3, 130.2, 128.6, 127.4, 127.2, 119.6, 119.1, 80.6, 79.3, 48.4, 32.6.
HRMS-ESI$^+$ (m/z) calcd for $C_{17}H_{14}BrO_2^+$ [M+H]$^+$ 329.0172, found 329.0175.



[1,1'-biphenyl]-4-yl(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)methanone

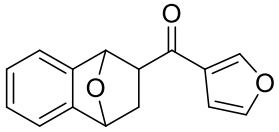
(3as)

21.2 mg, 65% yield, white solid, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.06 – 7.99 (m, 2H), 7.74 – 7.66 (m, 2H), 7.67 – 7.59 (m, 2H), 7.52 – 7.43 (m, 2H), 7.45 – 7.38 (m, 1H), 7.41 – 7.34 (m, 1H), 7.32 (dd, *J* = 5.4, 3.0 Hz, 1H), 7.24 – 7.21 (m, 2H), 5.73 (s, 1H), 5.52 (d, *J* = 4.9 Hz, 1H), 3.47 (dd, *J* = 8.9, 4.7 Hz, 1H), 2.50 (dt, *J* = 11.6, 4.8 Hz, 1H), 1.91 (dd, *J* = 11.6, 9.0 Hz,1H).
^{13}C NMR (101 MHz, Chloroform-d) δ 198.5, 146.4, 146.1, 145.0, 140.0, 135.1, 129.28, 129.2, 128.5, 127.6, 127.5, 127.3, 127.1, 119.6, 119.1, 80.9, 79.3, 48.5, 32.6.
MP:102 °C
HRMS-ESI$^+$ (m/z) calcd for $C_{23}H_{19}O_2^+$ [M+H]$^+$ 327.1380, found 327.1383.



naphthalen-2-yl(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)methanone (3at)

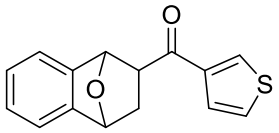
16.5 mg, 55% yield, light yellow soild, 1H NMR (400 MHz, Chloroform-*d*) δ 8.42 (d, *J* = 1.7 Hz, 1H), 8.05 (dd, *J* = 8.7, 1.8 Hz, 1H), 7.96 – 7.92 (m, 2H), 7.92 – 7.87 (m, 2H), 7.62 (ddd, *J* = 8.2, 6.9, 1.4 Hz, 1H), 7.56 (ddd, *J* = 8.1, 6.9, 1.4 Hz, 1H), 7.39 (dd, *J* = 5.6, 2.7 Hz, 1H), 7.34 (dd, *J* = 5.5, 2.8 Hz, 1H), 7.26 – 7.23 (m, 1H), 5.77 (s, 1H), 5.53 (d, *J* = 4.9 Hz, 1H), 3.60 (dd, *J* = 9.0, 4.8 Hz, 1H), 2.51 (dt, *J* = 11.7, 4.9 Hz, 1H), 1.98 (dd, *J* = 11.6, 9.0 Hz, 1H).
^{13}C NMR (101 MHz, Chloroform-d) δ 198.9, 146.5, 145.1, 135.9, 133.9, 132.8, 130.3, 129.8, 128.9, 128.8, 128.0, 127.3, 127.1, 127.1, 124.5, 119.6, 119.2, 80.9, 79.4, 48.5, 32.8.
MP: 164 °C
HRMS-ESI$^+$ (m/z) calcd for $C_{21}H_{16}O_2Na^+$ [M+Na]$^+$ 323.1043, found 323.1045.



furan-3-yl(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)methanone (3au)

14.4 mg, 60% yield, white solid, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.00 (t, *J* = 1.1 Hz, 1H), 7.47 (t, *J* = 1.7 Hz, 1H), 7.31 (ddd, *J* = 8.4, 5.2, 3.5 Hz, 2H), 7.21 (dd, *J* = 5.3, 3.0 Hz, 2H), 6.81 (dd, *J* = 1.9, 0.8 Hz, 1H), 5.64 (s, 1H), 5.51 (d, *J* = 4.9 Hz, 1H), 3.08 (dd, *J* = 8.8, 4.6 Hz, 1H), 2.50 (dt, *J* = 11.6, 4.8 Hz, 1H), 1.79 (dd, *J* = 11.6, 8.8 Hz, 1H).
^{13}C NMR (101 MHz, Chloroform-d) δ 193.6, 147.1, 146.0, 144.7, 144.3, 127.1, 126.9, 119.4, 118.8, 109.0, 80.8, 79.1, 50.3, 31.8.
MP: 110 °C

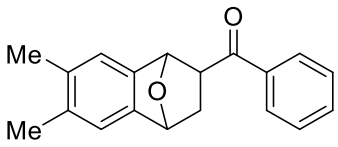
HRMS-ESI$^+$ (m/z) calcd for $C_{15}H_{12}O_3Na^+$ [M+Na]$^+$263.0679, found 263.0681.



(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)(thiophen-3-yl)methanone (3av)

14.1 mg, 55% yield, yellow oil, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.02 (dd, *J* = 2.9, 1.3 Hz, 1H), 7.57 (dd, *J* = 5.1, 1.3 Hz, 1H), 7.39 – 7.33 (m, 1H), 7.37 – 7.31 (m, 1H), 7.30 (dd, *J* = 5.2, 3.2 Hz, 1H), 7.21 (dd, *J* = 5.3, 3.1 Hz, 2H), 5.66 (s, 1H), 5.51 (d, *J* = 5.0 Hz, 1H), 3.29 (dd, *J* = 8.9, 4.7 Hz, 1H), 2.51 (dt, *J* = 11.7, 4.9 Hz, 1H), 1.83 (dd, *J* = 11.6, 8.9 Hz, 1H).
^{13}C NMR (101 MHz, Chloroform-d) δ 193.2, 146.4, 145.0, 141.8, 132.2, 127.4, 127.3, 127.1, 126.8, 119.5, 119.0, 81.1, 79.3, 49.9, 32.2.

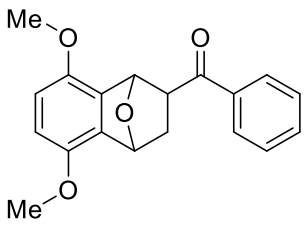
HRMS-ESI$^+$ (m/z) calcd for $C_{15}H_{13}SO_2^+$ [M+H]$^+$ 257.0631, found 257.0635.



(6,7-dimethyl-1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)(phenyl)methanone

(3ba)

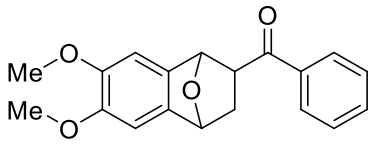
19.8 mg, 71% yield, yellow oil, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.99 – 7.92 (m, 2H), 7.63 – 7.54 (m, 1H), 7.48 (dd, *J* = 8.3, 7.0 Hz, 2H), 7.14 (s, 1H), 7.10 (s, 1H), 5.62 (s, 1H), 5.45 (d, *J* = 4.9 Hz, 1H), 3.42 (dd, *J* = 8.9, 4.7 Hz, 1H), 2.46 (dt, *J* = 11.6, 4.8 Hz, 1H), 2.28 (d, *J* = 3.5 Hz, 6H), 1.84 (dd, *J* = 11.5, 8.9 Hz, 1H).
^{13}C NMR (101 MHz, Chloroform-d) δ 197.9, 147.4, 147.2, 137.7, 136.1, 135.4, 132.1, 127.7, 127.4, 103.3, 102.9, 79.8, 78.3, 55.4, 55.4, 47.6, 31.9.
HRMS-ESI$^+$ (m/z) calcd for $C_{19}H_{19}O_2^+$ [M+H]$^+$ 279.1380, found 279.1382.



(5,8-dimethoxy-1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)(phenyl)methanone

(3ca)

22.4 mg, 72% yield, light yellow soild, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.06 – 7.99 (m, 2H), 7.62 – 7.53 (m, 1H), 7.53 – 7.43 (m, 2H), 6.70 (s, 2H), 5.83 (d, *J* = 0.9 Hz, 1H), 5.70 – 5.64 (m, 1H), 3.85 (s, 3H), 3.82 (s, 3H), 3.46 (dd, *J* = 8.7, 4.6 Hz, 1H), 2.53 (dt, *J* = 11.6, 4.8 Hz, 1H), 1.83 (dd, *J* = 11.7, 8.8 Hz, 1H).
^{13}C NMR (101 MHz, Chloroform-d) δ 198.4, 147.1, 146.7, 136.5, 135.6, 134.3, 133.3, 128.8, 128.8, 111.9, 111.5, 79.4, 77.5, 56.4 (d, *J* = 4.0 Hz) , 48.2, 31.4.
MP: 126 ^{o}C
HRMS-ESI$^+$ (m/z) calcd for $C_{19}H_{18}O_4Na^+$ [M+Na]$^+$ 333.1097, found 333.1101.

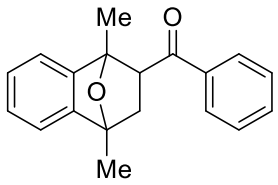


(6,7-dimethoxy-1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)(phenyl)methanone

(3da)

10.9 mg, 35% yield, yellow oil , ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.98 – 7.91 (m,2H), 7.63 – 7.54 (m, 1H), 7.48 (dd, *J* = 8.3, 6.9 Hz, 2H), 6.94 (d, *J* = 10.7 Hz, 2H), 5.66 (s, 1H), 5.45 (d, *J* = 4.7 Hz, 1H), 3.90 (d, *J* = 5.0 Hz, 6H), 3.39 (dd, *J* = 9.0, 4.6 Hz, 1H), 2.40 (dt, *J* = 11.5, 4.7 Hz, 1H), 1.85 (dd, *J* = 11.5, 8.9 Hz, 1H).
^{13}C NMR (101 MHz, Chloroform-d) δ 199.1, 148.6, 148.4, 138.9, 137.3, 136.6, 133.3, 128.9, 128.7, 104.5, 104.1, 81.1, 79.5, 56.6 (d, *J* = 10.0 Hz), 48.9, 33.1.

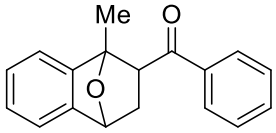
HRMS-ESI$^+$ (m/z) calcd for $C_{19}H_{18}O_4Na^+$ [M+Na]$^+$ 333.1097, found 333.1099.



(1,4-dimethyl-1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)(phenyl)methanone

(3ea)

12.5 mg, 45% yield， yellow oil, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.00 – 7.92 (m, 2H), 7.63 – 7.54 (m, 1H), 7.49 (dd, *J* = 8.4, 6.9 Hz, 2H), 7.24 (s, 2H), 7.25 – 7.18 (m,2H), 3.73 (dd, *J* = 8.1, 4.7 Hz, 1H), 2.52 (dd, *J* = 11.5, 4.7 Hz, 1H), 1.94 (s, 3H), 1.83 (dd, *J* = 11.5, 8.1 Hz, 1H), 1.62 (s, 3H).
^{13}C NMR (101 MHz, Chloroform-d) δ 200.2, 149.1, 149.0, 138.5, 133.3, 129.0, 128.5, 127.2, 126.9, 118.1, 117.4, 87.6, 85.1, 51.5, 40.8, 17.5, 16.0.
HRMS-ESI$^+$ (m/z) calcd for $C_{19}H_{18}O_2Na^+$ [M+Na]$^+$ 301.1199, found 301.1200.

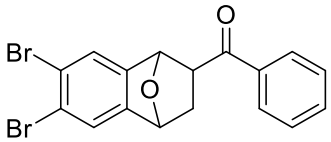


(1-methyl-1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)(phenyl)methanone (3fa)

8.7 mg, 33% yield，yellow oil, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.01 – 7.94 (m, 2H), 7.64 – 7.55 (m, 1H), 7.50 (dd, *J* = 8.4, 7.0 Hz,2H), 7.33 – 7.26 (m, 1H), 7.27 – 7.20 (m, 3H), 5.54 (d, *J* = 5.0 Hz, 1H), 3.65 (dd, *J* = 8.2, 4.6 Hz, 1H), 2.77 (dt, *J* = 11.6, 4.9 Hz, 1H), 1.79 (dd, *J* = 11.6, 8.2 Hz, 1H), 1.64 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-d) δ 200.3, 148.1, 146.7, 138.3, 133.2, 128.8, 128.3, 127.0, 126.8, 119.2, 117.4, 88.4, 78.3, 48.4, 35.1, 15.6.

HRMS-ESI$^+$ (m/z) calcd for $C_{18}H_{16}O_2Na^+$ [M+Na]$^+$287.1043, found 287.1047.



(6,7-dibromo-1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)(phenyl)methanone

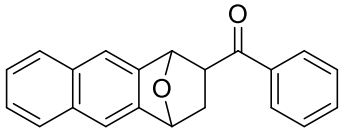
(3ga)

27.4 mg, 67% yield, white solid, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.92 (d, *J* = 7.7 Hz, 2H), 7.60 (d, *J* = 4.2 Hz, 2H), 7.57 (d, *J* = 6.4 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 2H), 5.66 (s, 1H), 5.44 (d, *J* = 5.0 Hz, 1H), 3.43 (dd, *J* = 9.0, 4.8 Hz, 1H), 2.49 – 2.42 (m, 1H), 1.89 (dd, *J* = 11.9, 9.0 Hz, 1H).

^{13}C NMR (101 MHz, Chloroform-d) δ 197.9, 147.4, 146.1, 136.1, 133.6, 129.0, 128.7, 125.0, 124.6, 123.0, 123.0, 80.3, 78.8, 48.0, 32.2.

MP: 155 °C

HRMS-ESI$^+$ (m/z) calcd for $C_{17}H_{13}Br_2O_2^+$ [M+H]$^+$ 408.9256, found 408.9258.

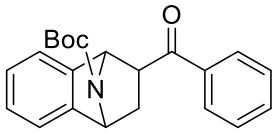


phenyl(1,2,3,4-tetrahydro-1,4-epoxyanthracen-2-yl)methanone (3ha)

18.0 mg, 68% yield, white solid, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.01 – 7.94 (m, 2H), 7.89 – 7.81 (m, 2H), 7.74 (s, 1H), 7.69 (s, 1H), 7.64 – 7.55 (m, 1H), 7.52 (d, *J* = 1.5 Hz, 1H), 7.50 (d, *J* = 3.0 Hz, 2H), 7.49 (d, *J* = 3.4 Hz, 1H), 5.81 (s, 1H), 5.62 (d, *J* = 5.0 Hz, 1H), 3.56 (dd, *J* = 8.9, 4.9 Hz, 1H), 2.59 (dt, *J* = 11.8, 5.0 Hz, 1H), 1.97 (dd, *J* = 11.8, 9.0 Hz, 1H).

^{13}C NMR (101 MHz, Chloroform-d) δ 198.5, 144.0, 142.9, 136.3, 133.5, 133.1, 133.0, 129.0, 128.7, 128.5, 128.4, 126.3, 126.2, 117.9, 117.4, 80.8, 79.3, 49.1, 33.1.
MP: 174 °C

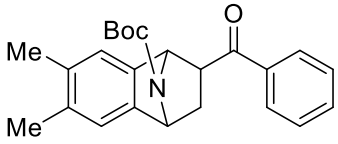
HRMS-ESI$^+$ (m/z) calcd for $C_{21}H_{16}O_2Na^+$ [M+Na]$^+$323.1043, found 323.1045.



tert-butyl-2-benzoyl-1,2,3,4-tetrahydro-1,4-epiminonaphthalene-9-carboxylate (3ia)

27.2 mg, 78% yield, light yellow soild, ^{1}H NMR (400 MHz,Chloroform-*d*) δ 7.98 (d, *J* = 7.6 Hz, 2H), 7.59 (d, *J* = 7.4 Hz, 1H), 7.51 (t, *J* = 7.6 Hz, 2H), 7.41 – 7.35 (m, 1H), 7.30 (q, *J* = 5.4, 5.0 Hz, 1H), 7.25 – 7.15 (m,2H), 5.37 (s, 1H), 5.34 – 5.14 (m, 1H), 3.40 (dd, *J* = 8.6, 4.7 Hz, 1H), 2.81 (dt, *J* = 11.9, 4.7 Hz, 1H), 1.64 (dd, *J* = 11.9, 8.5 Hz, 1H), 1.33 (s, 9H).
^{13}C NMR (101 MHz, Chloroform-d) δ 197.6, 145.8, 144.6, 136.2, 133.2, 128.8, 128.5, 127.0, 126.6, 120.1, 119.1, 80.4, 63.9, 30.5, 28.1.
MP: 136 °C

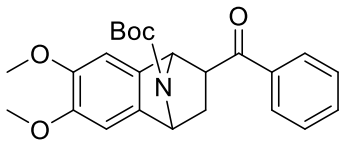
HRMS-ESI$^+$ (m/z) calcd for $C_{22}H_{23}O_3NNa^+$ [M+Na]$^+$ 372.1570, found 372.1570.



tert-butyl-2-benzoyl-6,7-dimethyl-1,2,3,4-tetrahydro-1,4-epiminonaphthalene-9-carboxylate (3ja)

30.6 mg, 81% yield, yellow oil, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.97 (d, *J* = 7.6 Hz, 2H), 7.59 (t, *J* = 7.3 Hz, 1H), 7.51 (t, *J* = 7.6 Hz, 2H), 7.15 (s, 1H), 7.09 (s, 1H), 5.29 (s, 1H), 5.24 (s, 1H), 3.37 (dd, *J* = 8.7, 4.7 Hz, 1H), 2.78 (dt, *J* = 10.2, 4.6 Hz, 1H), 2.27 (d, *J* = 7.3 Hz, 6H), 1.60 (dd, *J* = 11.9, 8.4 Hz, 1H), 1.33 (s, 9H).
^{13}C NMR (101 MHz, Chloroform-d) δ 197.9, 143.7, 142.5, 136.4, 135.1, 134.8, 133.2, 128.9, 128.6, 121.6, 120.6, 80.3, 63.9, 31.0, 28.3, 20.2.

HRMS-ESI$^+$ (m/z) calcd for $C_{24}H_{28}NO_3^+$ [M+H]$^+$378.2064, found 378.2065.



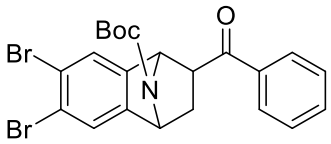
tert-butyl-2-benzoyl-6,7-dimethoxy-1,2,3,4-tetrahydro-1,4-epiminonaphthalene-9-carboxylate (3ka)

25.0 mg, 61% yield, white solid . ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.96 (d, *J* = 7.6 Hz, 2H), 7.60 (t, *J* = 7.4 Hz, 1H), 7.51 (t, *J* = 7.5 Hz, 2H), 6.96 (d, *J* = 17.8 Hz, 2H), 5.33 (s, 1H), 5.19 (d, *J* = 37.3 Hz, 1H), 3.91 (d, *J* = 10.3 Hz, 6H), 3.35 (d, *J* = 8.5 Hz, 1H), 2.72 (d, *J* = 11.8 Hz, 1H), 1.67 – 1.58 (m, 1H), 1.34 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-d) δ 198.0, 148.3, 148. 0, 138.6, 136.5, 133.2, 128.9, 128.6, 105.1, 80.5, 64.1, 56.5 (d, *J* = 13.7 Hz), 31.8, 28.3, 22.8, 14.0.

MP: 125 °C

HRMS-ESI^{+} (m/z) calcd for $C_{24}H_{28}O_5N^{+}$ $[M+H]^{+}$410.1962, found 410.1963.



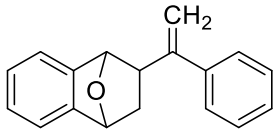
tert-butyl-2-benzoyl-6,7-dibromo-1,2,3,4-tetrahydro-1,4-epiminonaphthalene-9-carboxylate (3la)

35.5 mg, 70% yield, white solid, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.96 – 7.90 (m, 2H), 7.64 (s, 1H), 7.59 (d, *J* = 13.1 Hz, 2H), 7.51 (t, *J* = 7.6 Hz,2H), 5.34 (s, 1H), 5.21 (s, 1H), 3.36 (dd, *J* = 8.6, 4.7 Hz, 1H), 2.81 – 2.68 (m, 1H), 1.71 – 1.59 (m, 1H), 1.34 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-d) δ 197.1, 168.0, 147.0, 145.7, 136.0, 133.6, 131.1, 129.7, 129.1, 128.6, 123.1, 122.7, 81.2, 63.3, 30.8, 28.2.

MP: 168 °C

HRMS-ESI^{+} (m/z) calcd for $C_{22}H_{21}BrNO_3Na^{+}$ $[M+Na]^{+}$529.9760, found 529.9763.

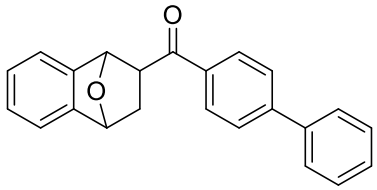


2-(1-phenylvinyl)-1,2,3,4-tetrahydro-1,4-epoxynaphthalene (4aa)

20.1 mg, 81% yield, light yellow oil, ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.38 (d, J = 7.4 Hz, 2H), 7.32 (d, J = 6.5 Hz, 2H), 7.28 (s, 1H), 7.29 – 7.23 (m, 2H), 7.17 (q, *J* = 4.8, 4.0 Hz, 2H), 5.47 (d, *J* = 5.0 Hz, 1H), 5.42 (d, *J* = 10.0 Hz, 2H), 5.37 (s, 1H), 2.82 (dd, *J* = 8.6, 4.5 Hz, 1H), 2.02 (dt, *J* = 11.7, 4.8 Hz, 1H), 1.89 (dd, *J* = 11.7, 8.5 Hz, 1H).

^{13}C NMR (101 MHz, Chloroform-d) δ 151.0, 146.4, 146.1, 142.4, 128.6, 127.7, 126.9, 126.8, 126.4, 119.3, 119.1, 112.0, 83.4, 79.5, 44.2, 36.6.

TOFMS-ESI$^+$ (m/z) calcd for $C_{18}H_{17}O$ [M+H]$^+$249.1274， found 249.1276.



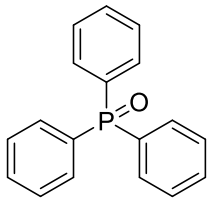
[1,1'-biphenyl]-4-yl(1,2,3,4-tetrahydro-1,4-epoxynaphthalen-2-yl)methanone (5ar)

29.7 mg, 91% yield, white solid , ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.05 – 7.99 (m, 2H), 7.73 – 7.68 (m, 2H), 7.66 – 7.61 (m, 2H), 7.50 – 7.45 (m, 2H), 7.43 – 7.35 (m, 2H), 7.32 (dd, *J* = 5.4, 3.0 Hz, 1H), 7.23 (dd, *J* = 5.3, 3.0 Hz, 2H), 5.73 (s, 1H), 5.52 (d, *J* = 5.0 Hz, 1H), 3.47 (dd, *J* = 8.9, 4.7 Hz, 1H), 2.50 (dt, *J* = 11.6, 4.9 Hz, 1H), 1.91 (dd, *J* = 11.6, 8.9 Hz, 1H).

^{13}C NMR (101 MHz, Chloroform-d) δ 198.7, 146.6, 146.3, 145.2, 140.2, 135.3, 129.5, 129.4, 128.7, 127.8, 127.7, 127.5, 127.3, 119.8, 119.4, 81.1, 79.5, 48.7, 32.8.

MP: 165 ^{o}C

HRMS-ESI$^+$ (m/z) calcd for $C_{23}H_{18}NaO_2^+$ [M+Na]$^+$349.1199, found 349.1204.



triphenylphosphine oxide （6）

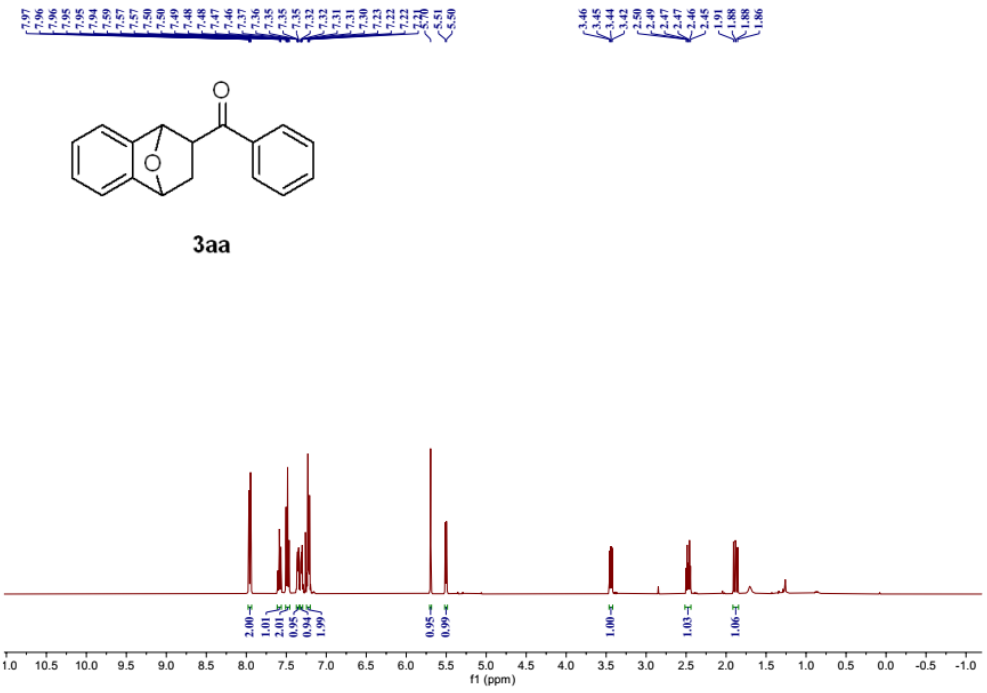
23.6 mg, 85% yield, white solid, ^{1}H NMR (400 MHz, Chloroform-d) δ 7.72 – 7.62 (m, 6H), 7.52 (tt, J = 7.4, 2.1 Hz, 3H), 7.44 (td, J = 7.6, 3.9 Hz, 6H).
^{13}C NMR (101 MHz, Chloroform-d) δ 133.0, 132.2 – 131.9 (m), 128.5 (d, J = 12.1 Hz).
^{31}P NMR (162 MHz, Chloroform-d) δ 29.07.

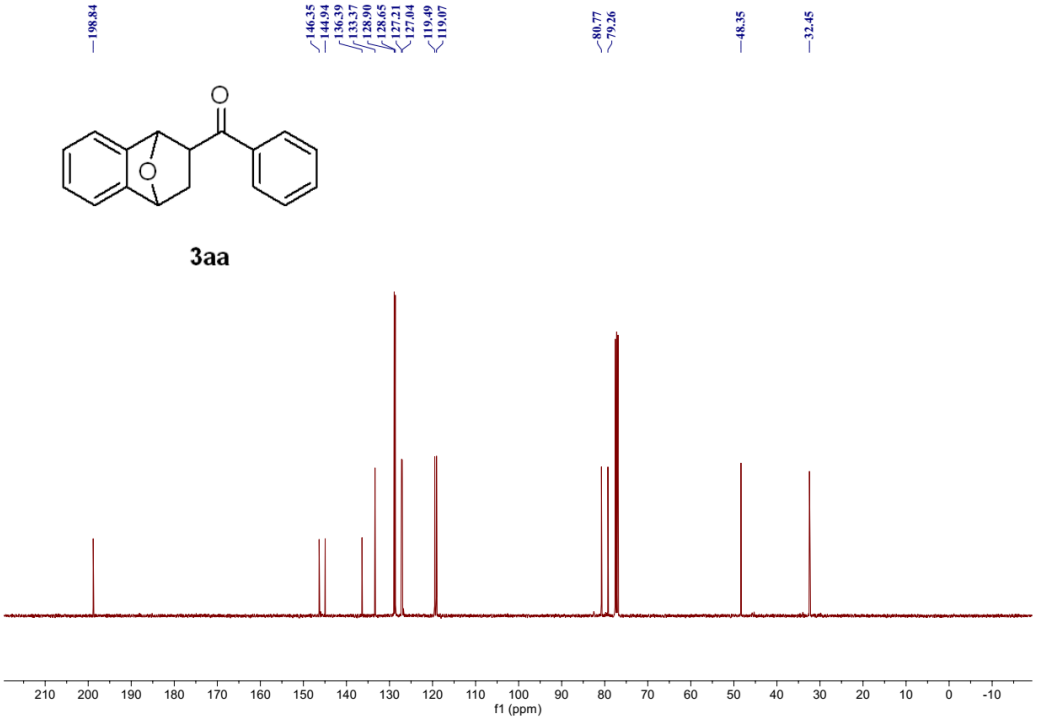
8、Reference

[1] Gandeepan P , Rajamalli P , Cheng C H .Diastereoselective [3+2] Annulation of Aromatic/Vinylic Amides with Bicyclic Alkenes through Cobalt‐Catalyzed CH A ctivation and Intramolecular Nucleophilic Addition[J].*Angewandte Chemie*, **2016**, *55*(13):4368-4368.DOI:10.1002/anie.201601760.

[2] Luo R , Liao J , Xie L , et al. Asymmetric ring-opening of oxabenzonorbornadiene with amines promoted by a chiral iridium-monophosphine catalyst[J]. *Chemical Communications*, **2013**, *49*(85):9959-9961.

9、NMR Spectral of Products:

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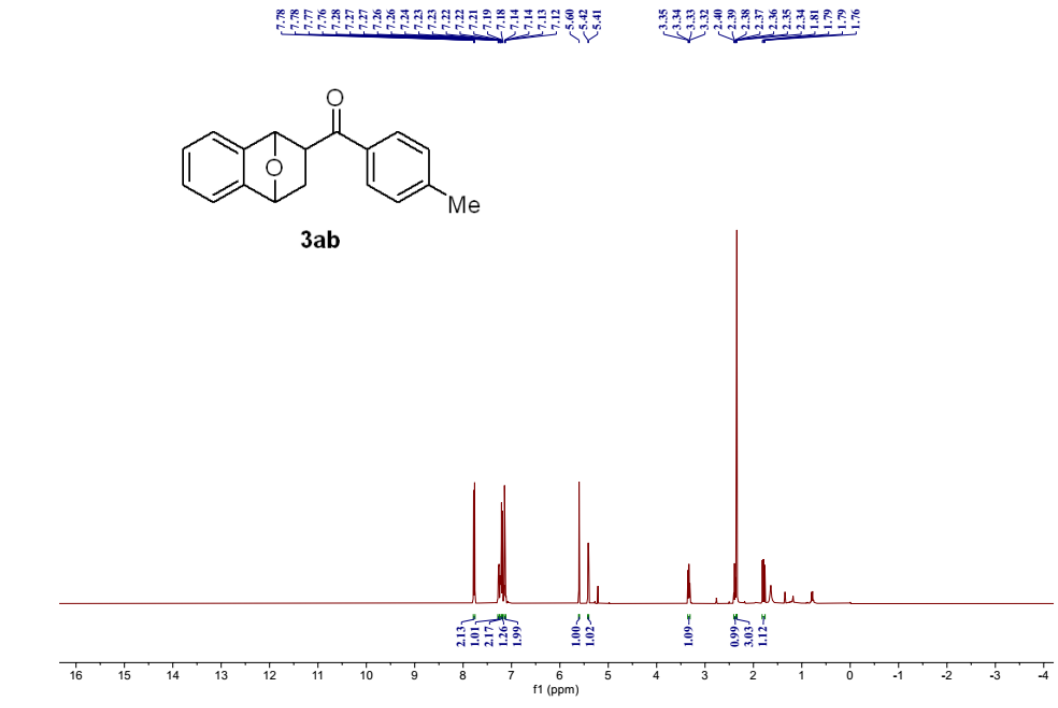
O
3aa
f1 (ppm)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1.76
O
O
Me
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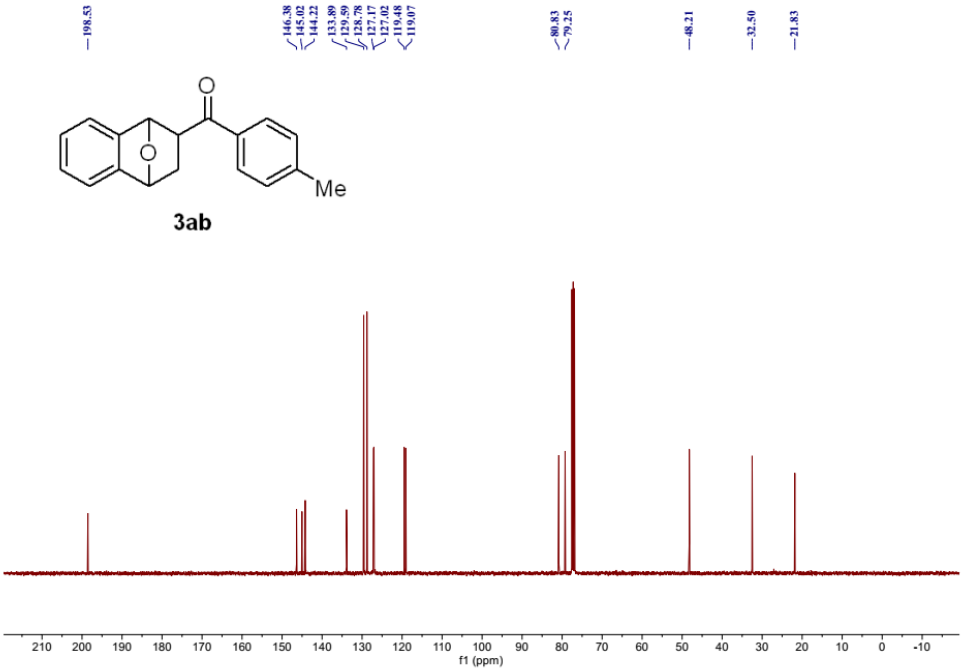
f1 (ppm)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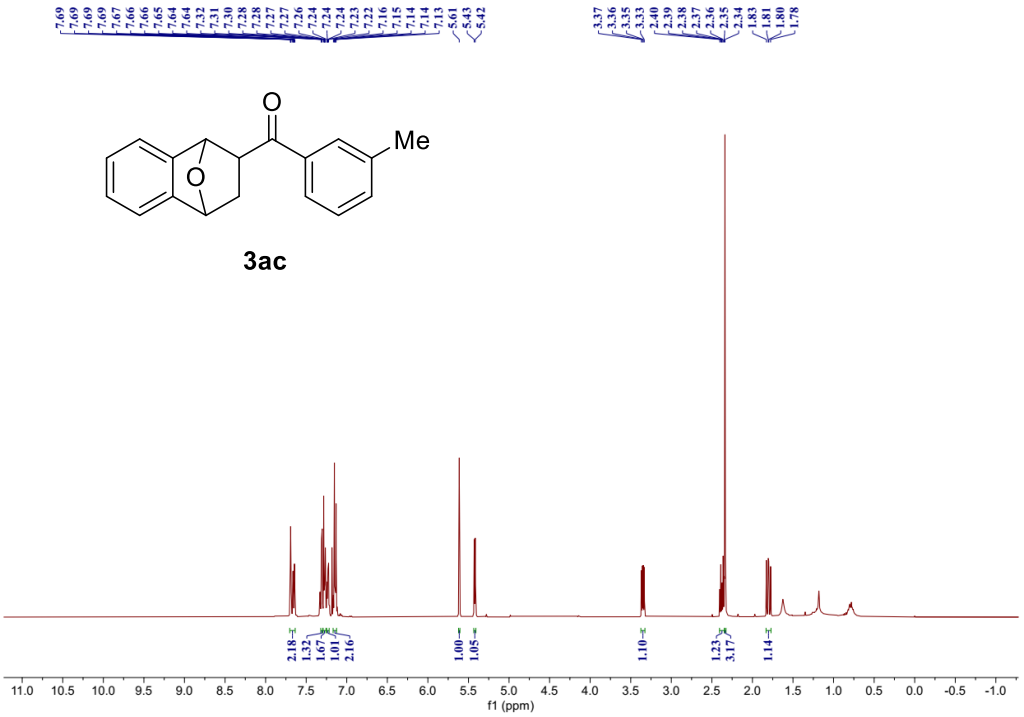
O
Me
f1 (ppm)

3ab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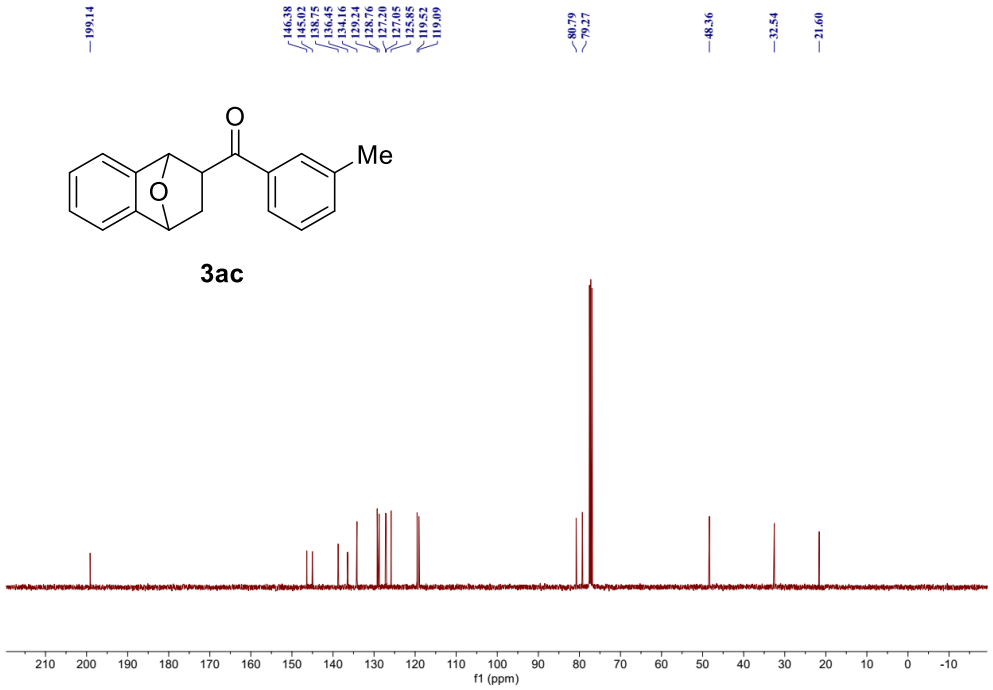
3ac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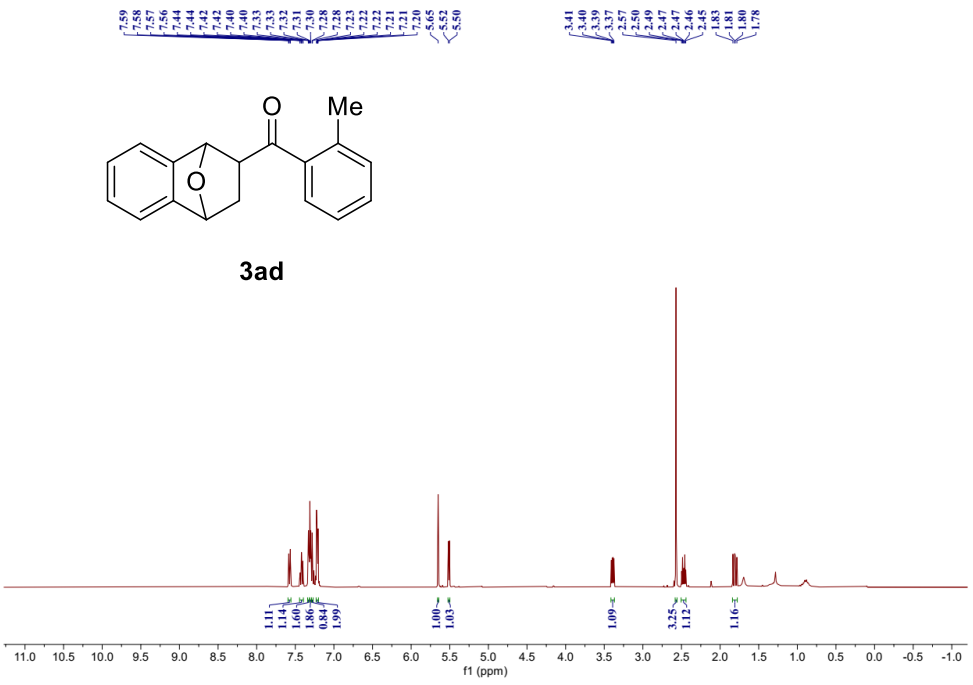
3ac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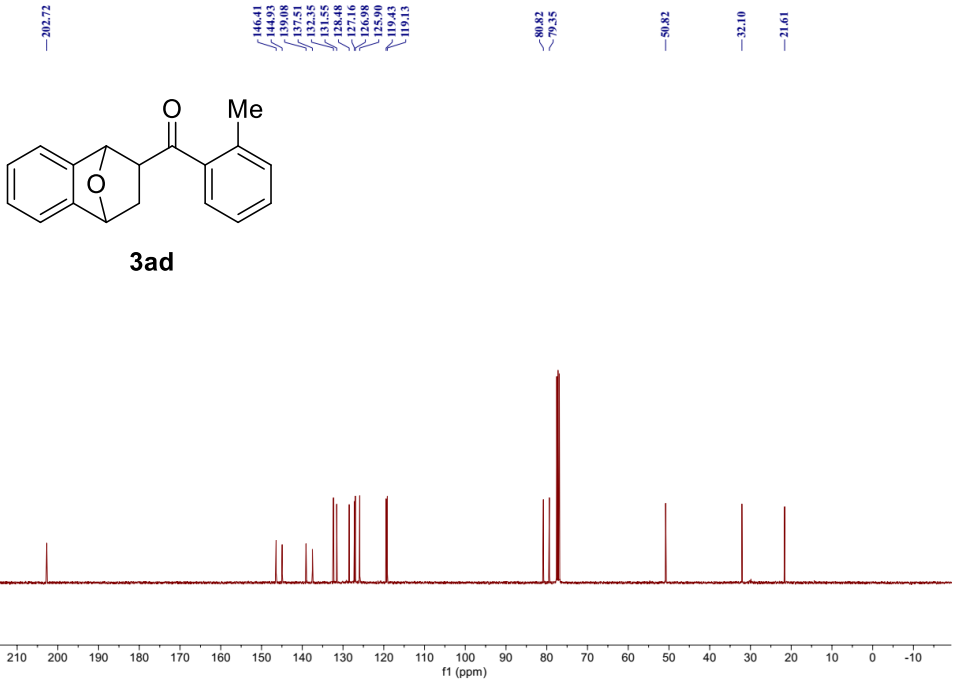
f1 (ppm)

3ad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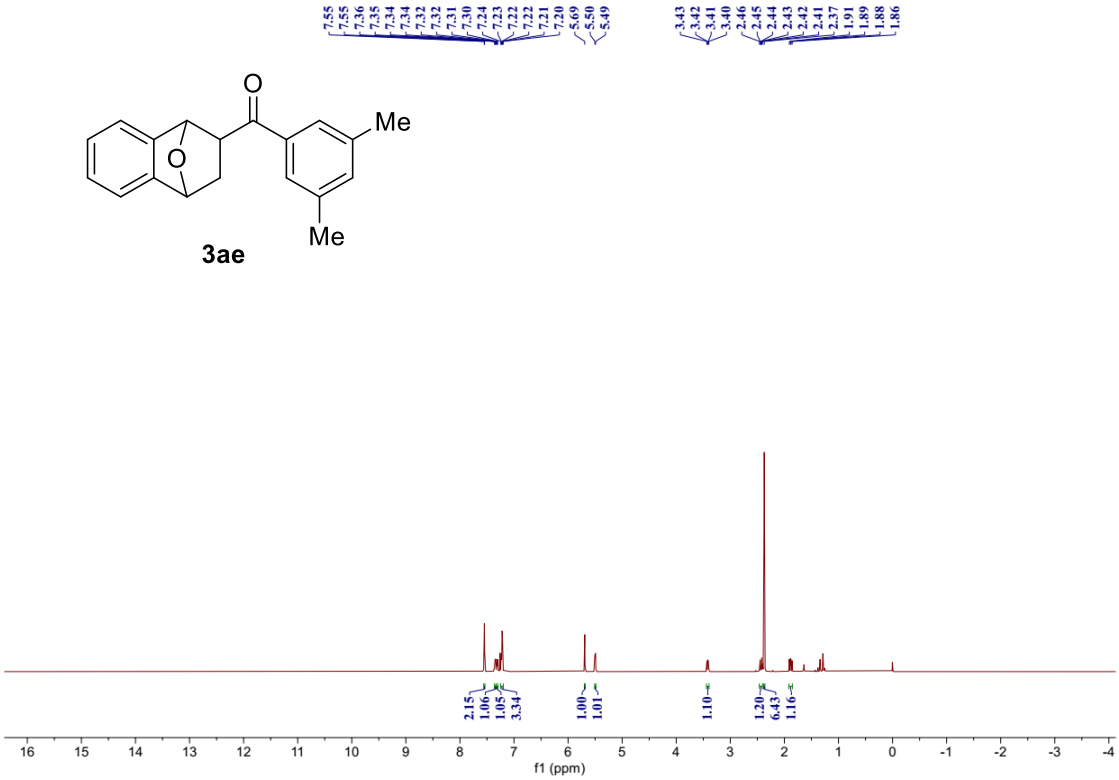
f1 (ppm)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5.49
3.43
3.42
3.41
3.40
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2.45
2.44
2.43
2.42
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1.88
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Me
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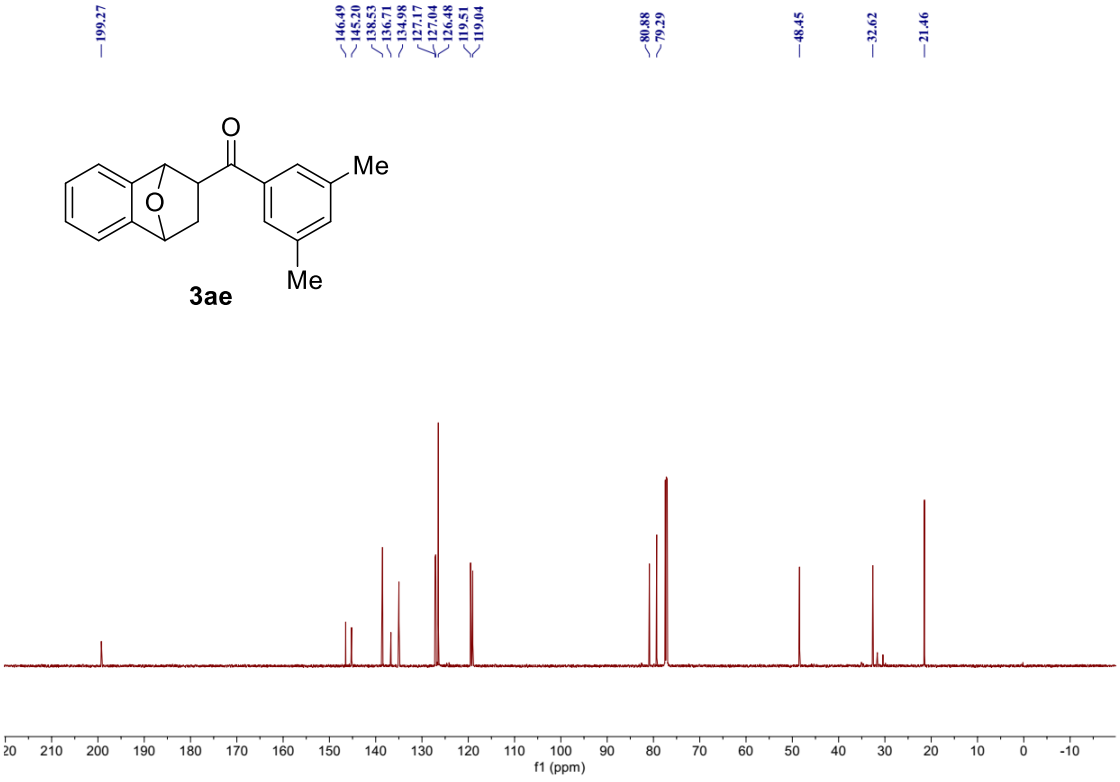
f1 (ppm)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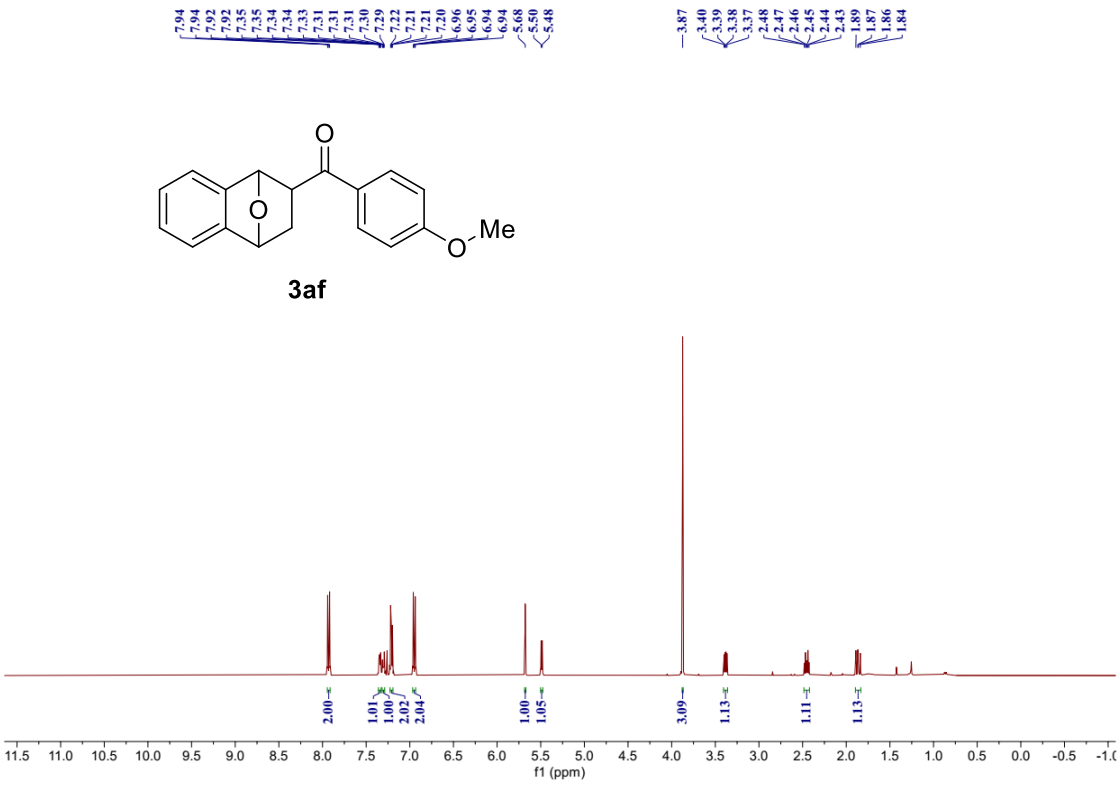
f1 (ppm)

3ae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1.86
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3.09
1.13
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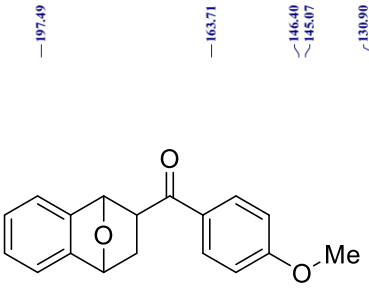
1.13
f1 (ppm)

3af


O
O
O
Me

3af

197.49
163.71
146.40
145.07
130.90
129.40
127.15
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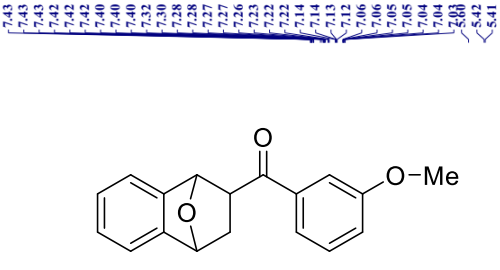
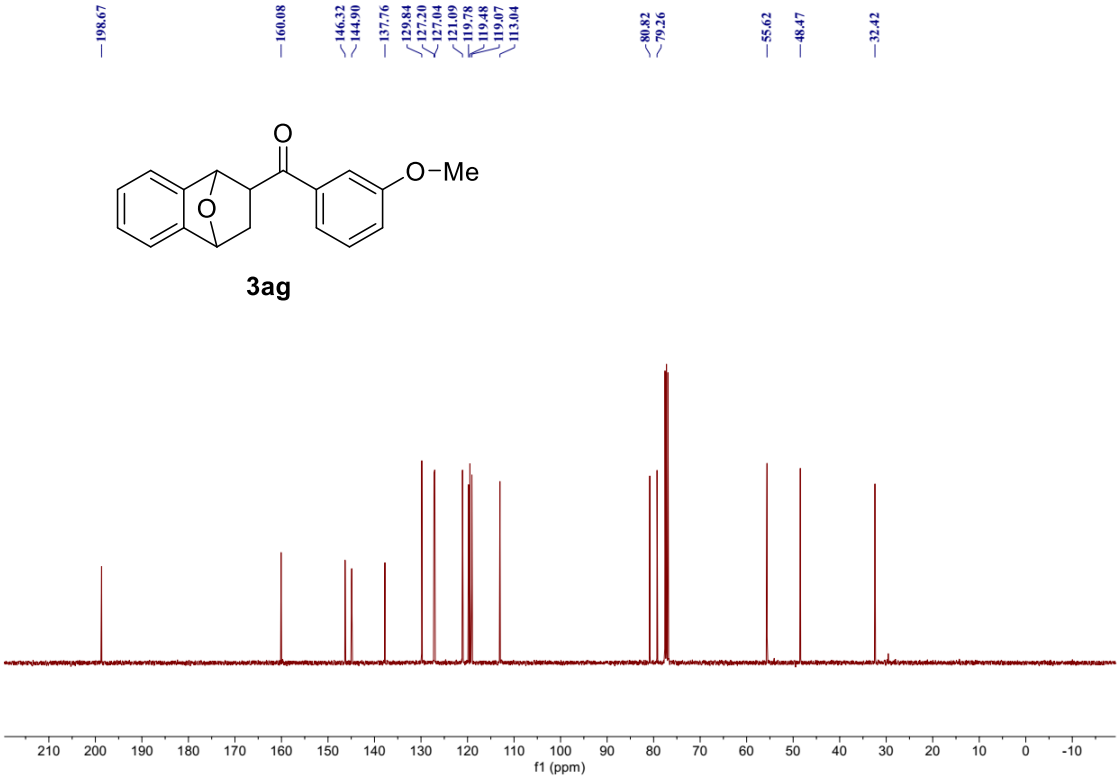
119.48
119.05
114.05
80.90
79.22
55.68
47.97
32.60
f1 (ppm)

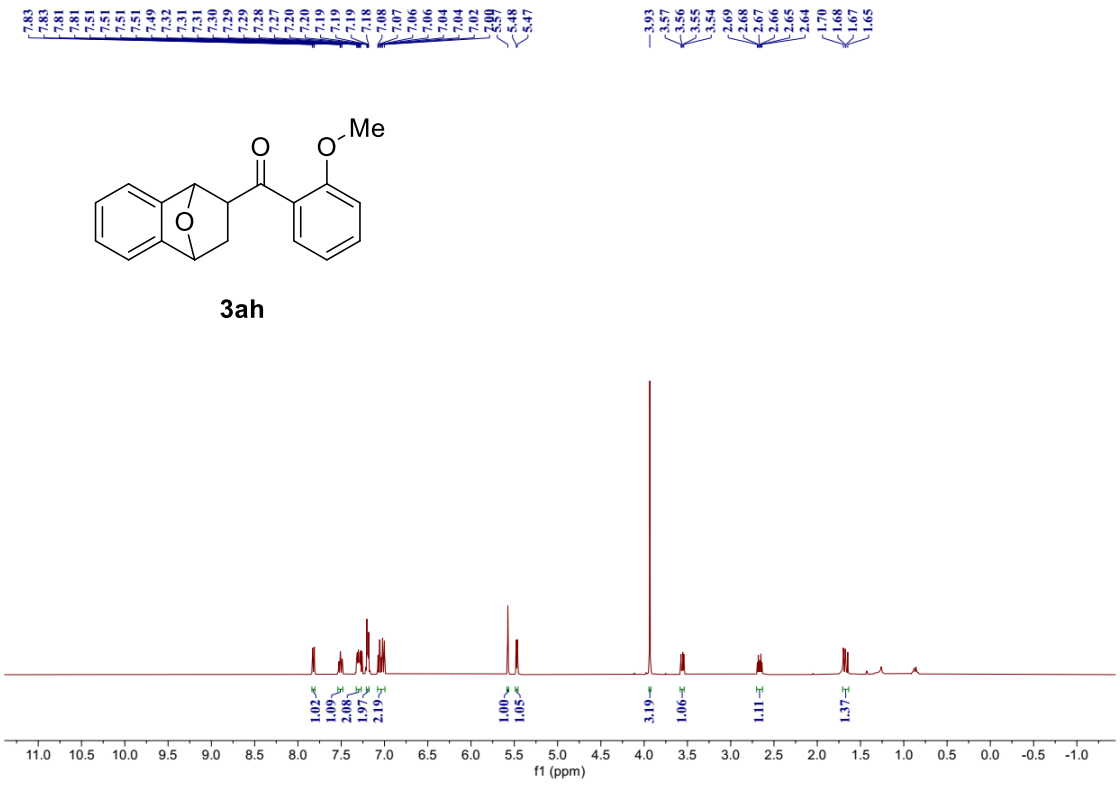

O
O
O−Me

3ag

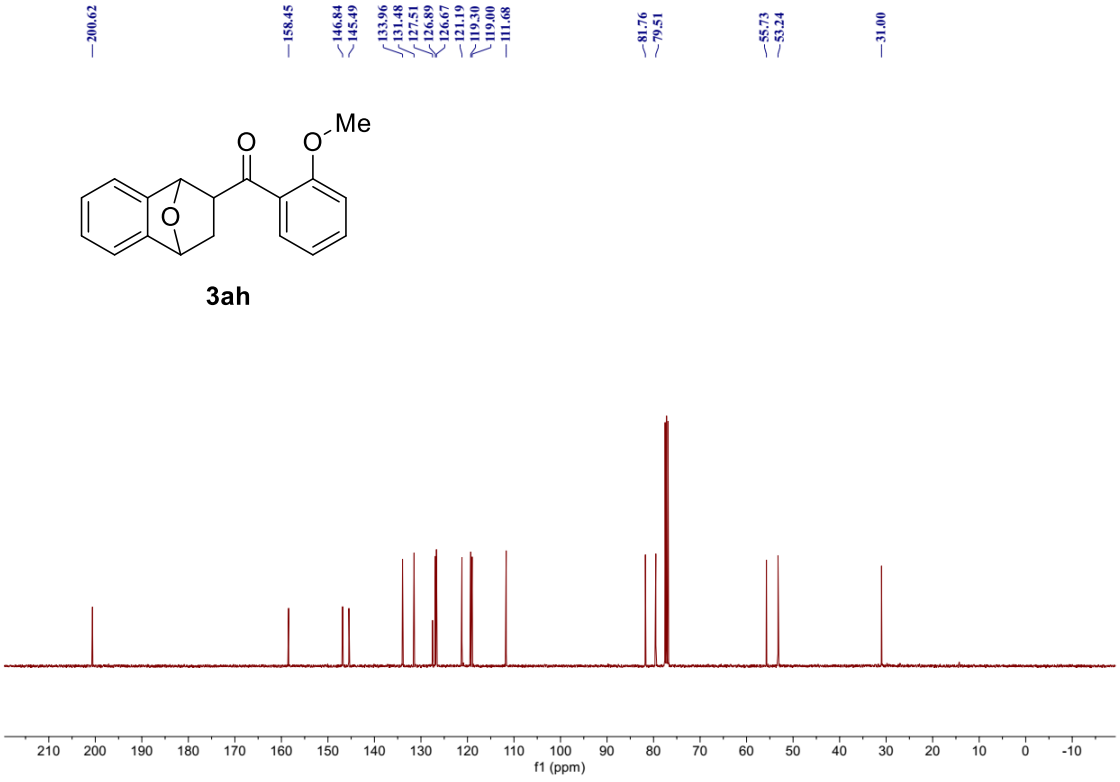
7.43
7.43
7.42
7.42
7.42
7.40
7.40
7.40
7.32
7.30
7.28
7.28
7.27
7.27
7.26
7.23
7.22
7.22
7.14
7.14
7.13
7.12
7.06
7.06
7.05
7.05
7.04
7.04
5.42
5.41
3.77
3.35
3.34
3.33
3.32
2.42
2.40
2.39
2.39
2.38
2.36
1.81
1.79
1.78
1.76
2.21
2.18
1.02
1.98
1.07
1.00
1.03
3.21
1.10
1.11
1.17
f1 (ppm)


198.67
160.08
146.32
144.90
137.76
129.84
127.20
127.04
121.09
119.78
119.48
119.07
113.04
80.82
79.26
55.62
48.47
32.42
O
O
O−Me
f1 (ppm)

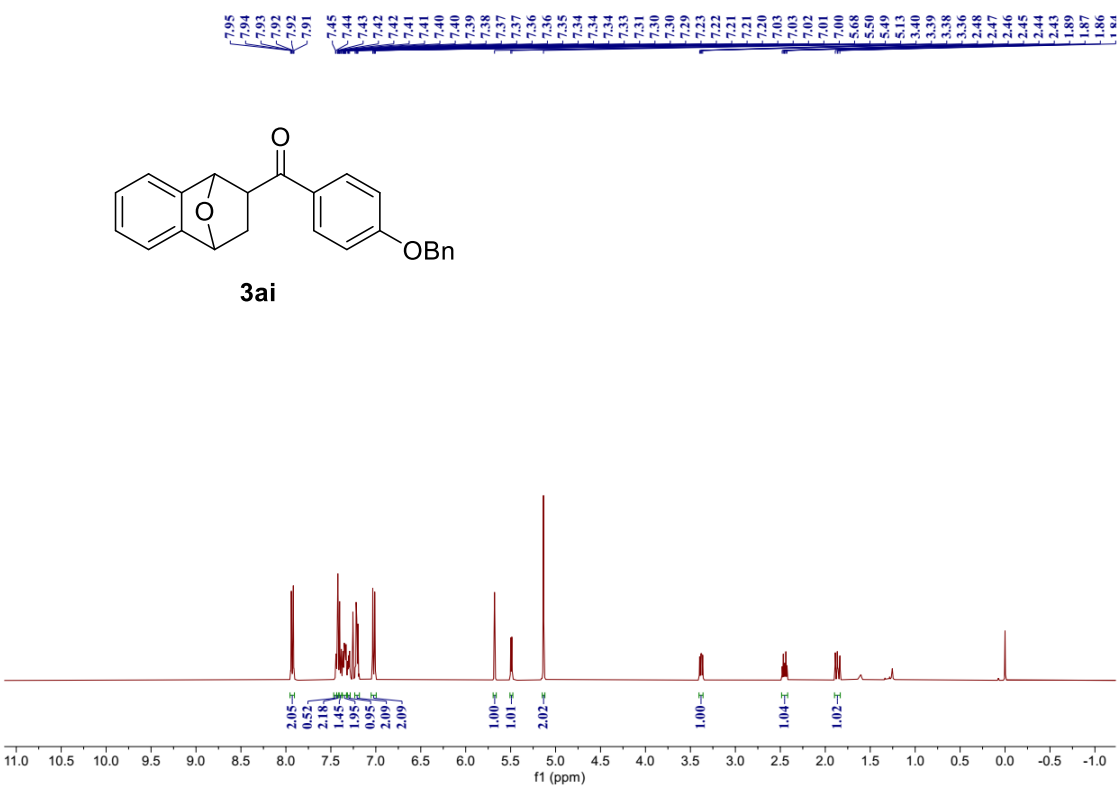
3ag


7.83
7.83
7.81
7.81
7.51
7.51
7.51
7.51
7.49
7.32
7.31
7.31
7.30
7.29
7.29
7.28
7.27
7.20
7.20
7.19
7.19
7.19
7.18
7.08
7.07
7.06
7.06
7.04
7.04
7.02
5.48
5.47
3.93
3.57
3.56
3.55
3.54
2.69
2.68
2.67
2.66
2.65
2.64
1.70
1.68
1.67
1.65
O
O
Me
1.02
1.09
2.08
1.97
2.19
1.00
1.05
3.19
1.06
1.11
1.37
f1 (ppm)

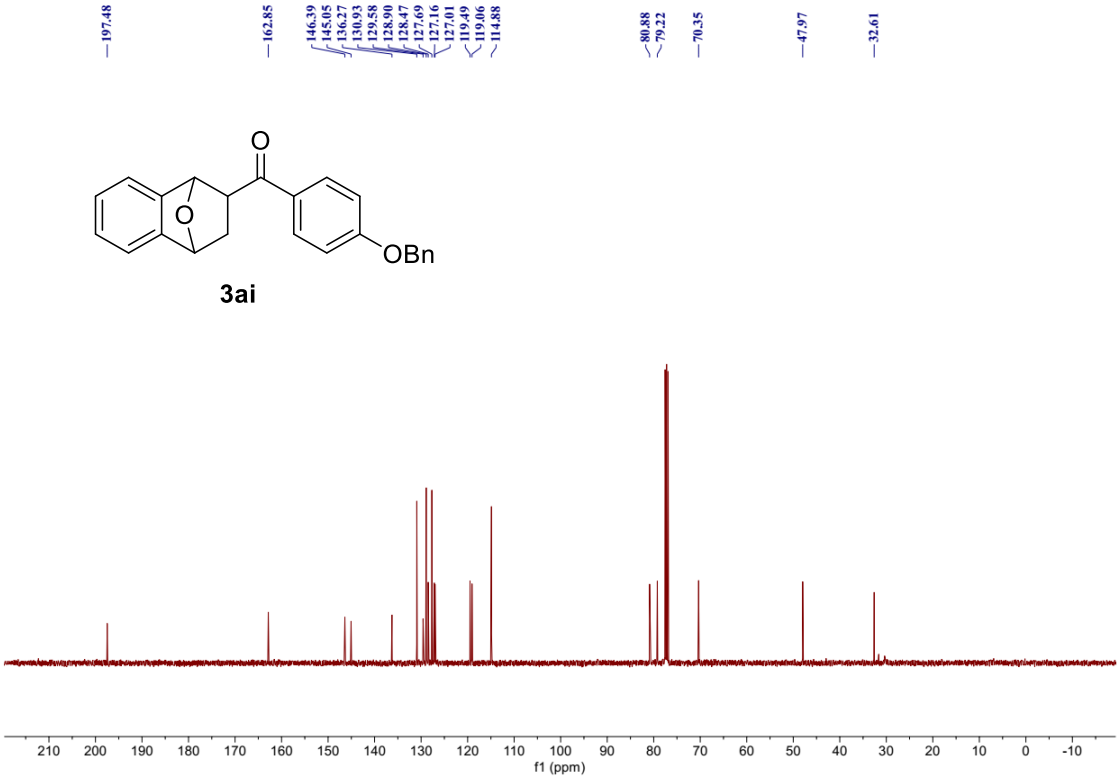
3ah


200.62
158.45
146.84
145.49
133.96
131.48
127.51
126.89
126.67
121.19
119.30
119.00
111.68
81.76
79.51
55.73
53.24
31.00
O
O
Me
O
f1 (ppm)

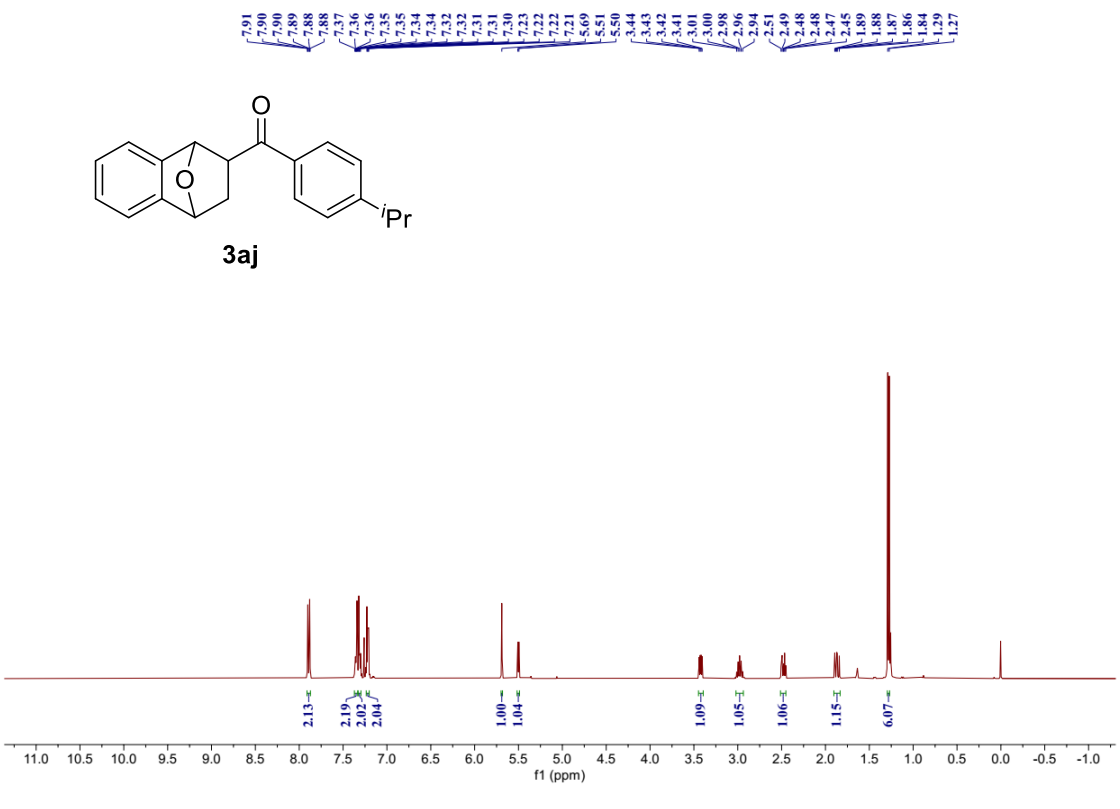
3ah


7.95
7.94
7.93
7.92
7.92
7.91
7.45
7.44
7.43
7.42
7.42
7.41
7.41
7.40
7.40
7.39
7.38
7.37
7.37
7.36
7.36
7.35
7.34
7.34
7.34
7.33
7.31
7.30
7.30
7.29
7.23
7.22
7.21
7.21
7.20
7.03
7.03
7.02
7.01
7.00
5.68
5.50
5.49
5.13
3.40
3.39
3.38
3.36
2.48
2.47
2.46
2.45
2.44
2.43
1.89
1.87
1.86
O
O
OBn
2.05
0.52
2.18
1.45
1.95
0.95
2.09
2.09
1.00
1.01
2.02
1.00
1.04
1.02
f1 (ppm)

3ai


197.48
162.85
146.39
145.05
136.27
130.93
129.58
128.90
128.47
127.69
127.16
127.01
119.49
119.06
114.88
80.88
79.22
70.35
47.97
32.61
O
O
OBn
f1 (ppm)

3ai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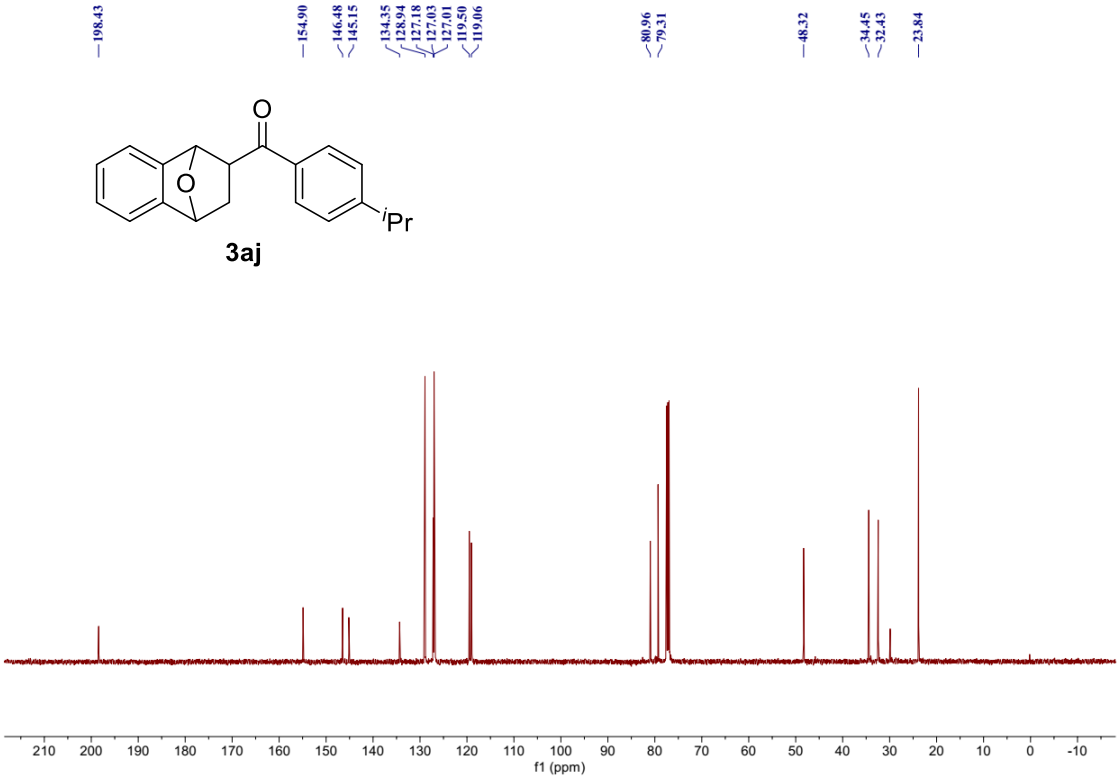
7.90
7.90
7.89
7.88
7.88
7.37
7.36
7.36
7.35
7.35
7.34
7.34
7.32
7.32
7.31
7.31
7.30
7.23
7.22
7.22
7.21
5.69
5.51
5.50
3.44
3.43
3.42
3.41
3.01
3.00
2.98
2.96
2.94
2.51
2.49
2.48
2.48
2.47
2.45
1.89
1.88
1.87
1.86
1.84
1.29
1.27
O
O
iPr
2.13
2.19
2.02
2.04
1.00
1.04
1.09
1.05
1.06
1.15
6.07
f1 (ppm)

3aj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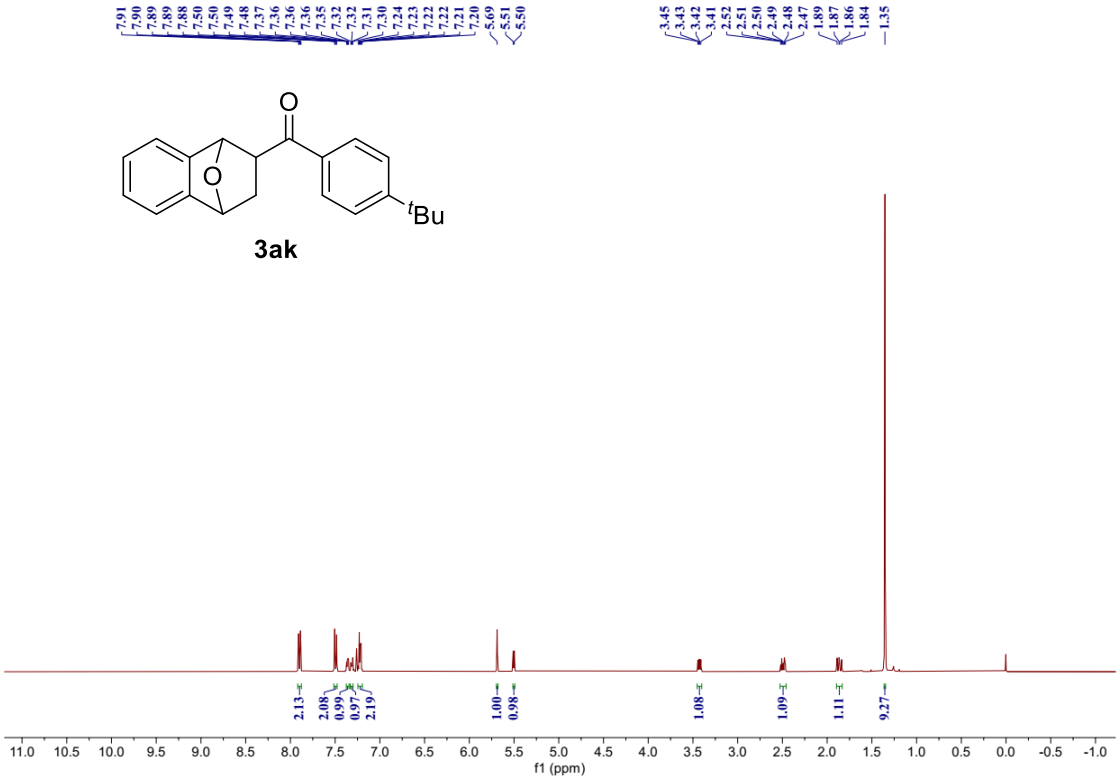
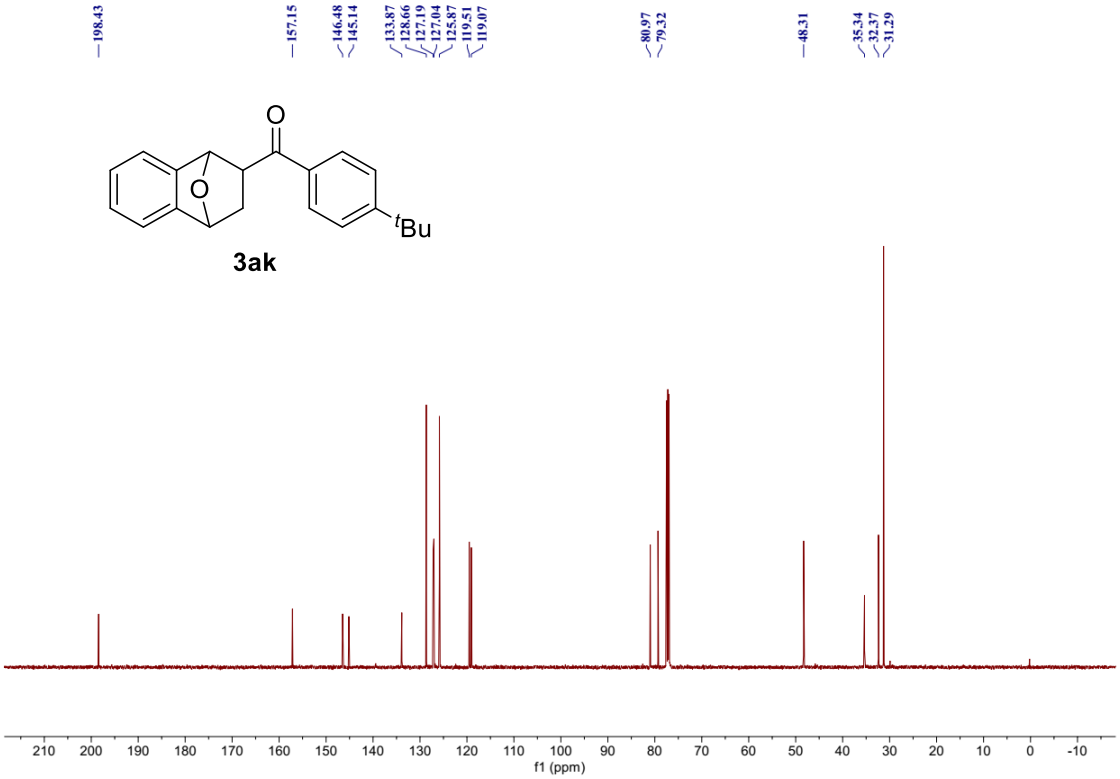
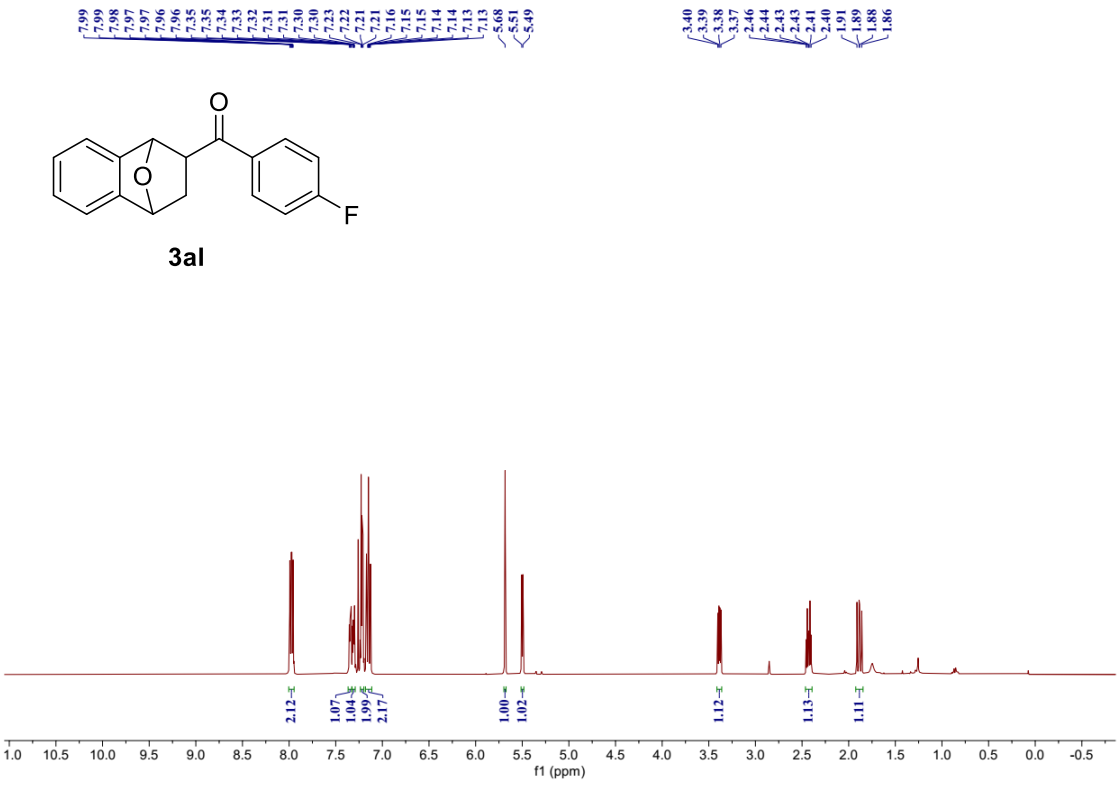
154.90
146.48
145.15
134.35
128.94
127.18
127.03
127.01
119.50
119.06
80.96
79.31
48.32
34.45
32.43
23.84
O
O
iPr
3aj
f1 (ppm)


7.91
7.90
7.89
7.89
7.88
7.50
7.50
7.49
7.48
7.37
7.36
7.36
7.36
7.35
7.32
7.32
7.31
7.30
7.24
7.23
7.22
7.22
7.21
7.20
5.69
5.51
5.50
3.45
3.43
3.42
3.41
2.52
2.51
2.50
2.49
2.48
2.47
1.89
1.87
1.86
1.84
1.35
O
O
tBu
3ak
2.13
2.08
0.99
0.97
2.19
1.00
0.98
1.08
1.09
1.11
9.27
f1 (ppm)


198.43
157.15
146.48
145.14
133.87
128.66
127.19
127.04
125.87
119.51
119.07
80.97
79.32
48.31
35.34
32.37
31.29
O
O
tBu
3ak
f1 (ppm)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7.99
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7.97
7.96
7.96
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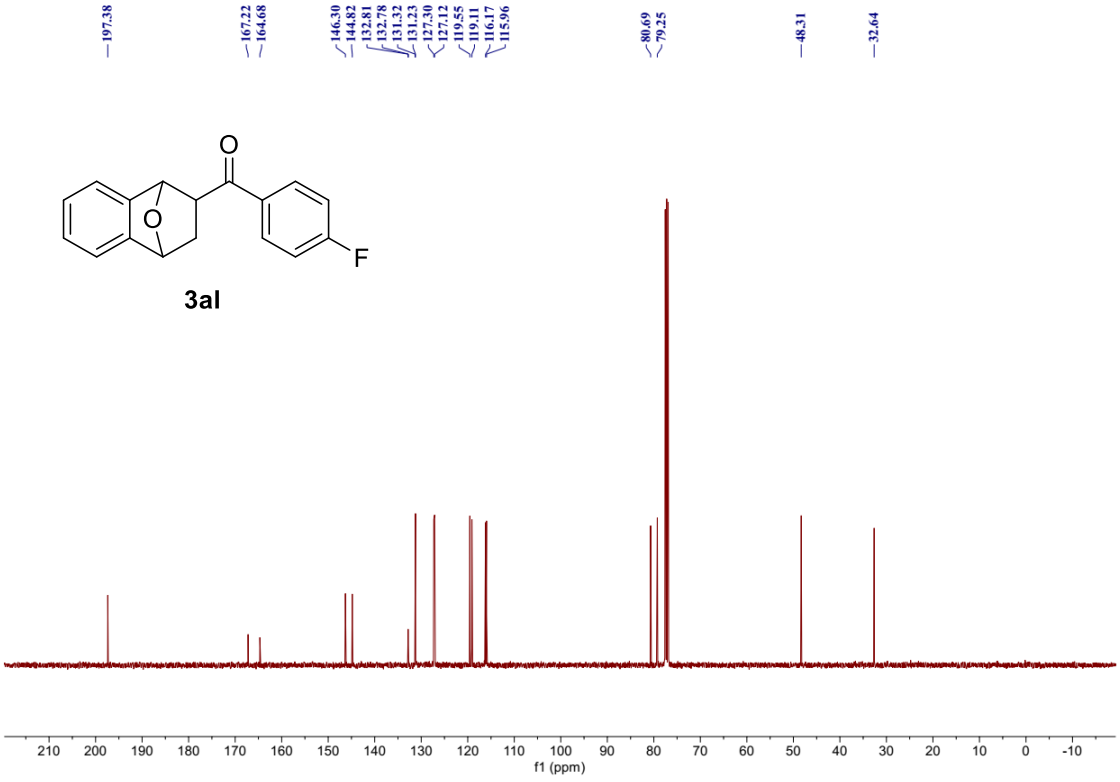
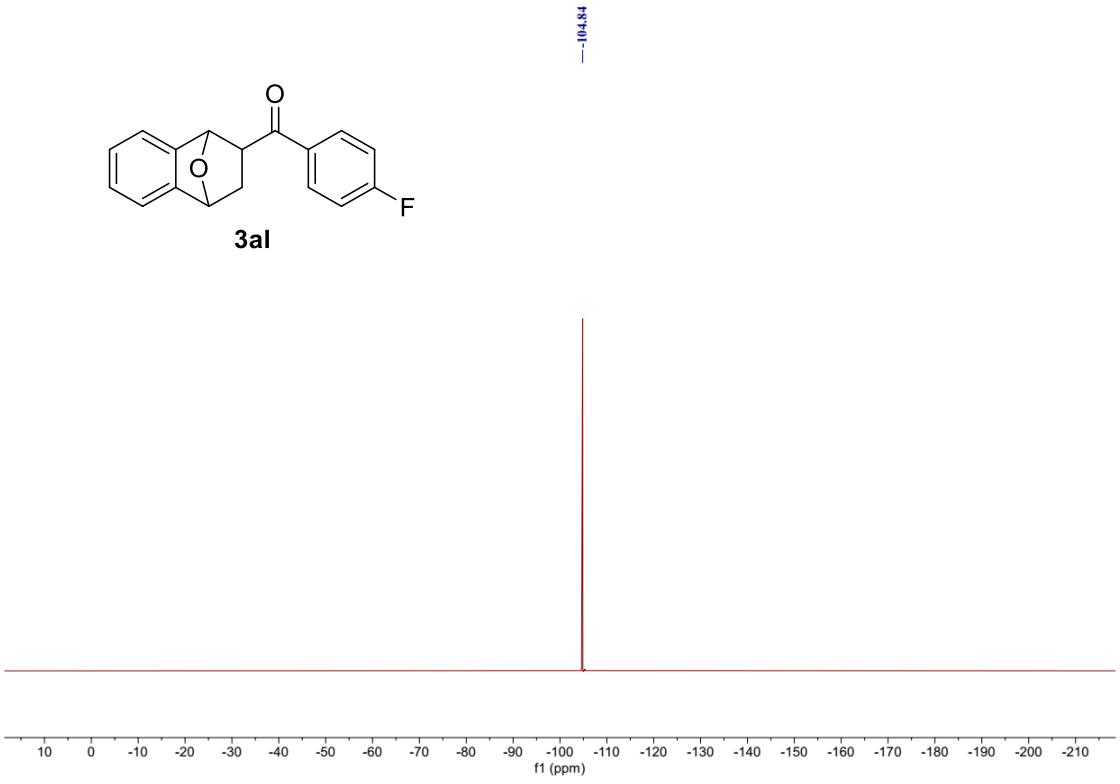
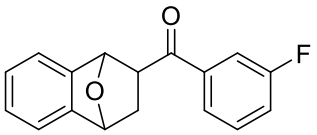
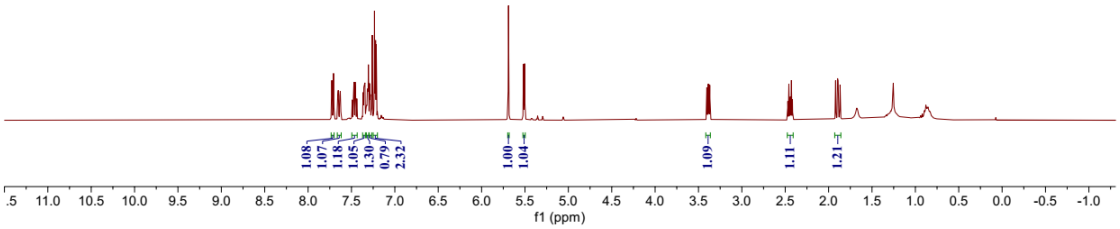
7.35
7.34
7.33
7.32
7.31
7.31
7.30
7.30
7.23
7.22
7.21
7.21
7.16
7.15
7.15
7.14
7.14
7.13
7.13
5.68
5.51
5.49
3.40
3.39
3.38
3.37
2.46
2.44
2.43
2.43
2.41
2.40
1.91
1.89
1.88
1.86
O
O
F
3al
2.12
1.07
1.04
1.99
2.17
1.00
1.02
1.12
1.13
1.11
f1 (ppm)

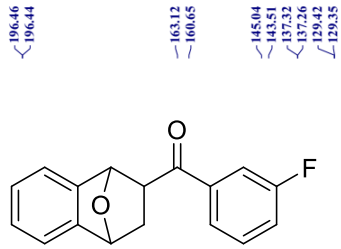

197.38
167.22
164.68
146.30
144.82
132.81
132.78
131.32
131.23
127.30
127.12
119.55
119.11
116.17
115.96
80.69
79.25
48.31
32.64
O
O
F
3al
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


-104.84
O
O
F
3al
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

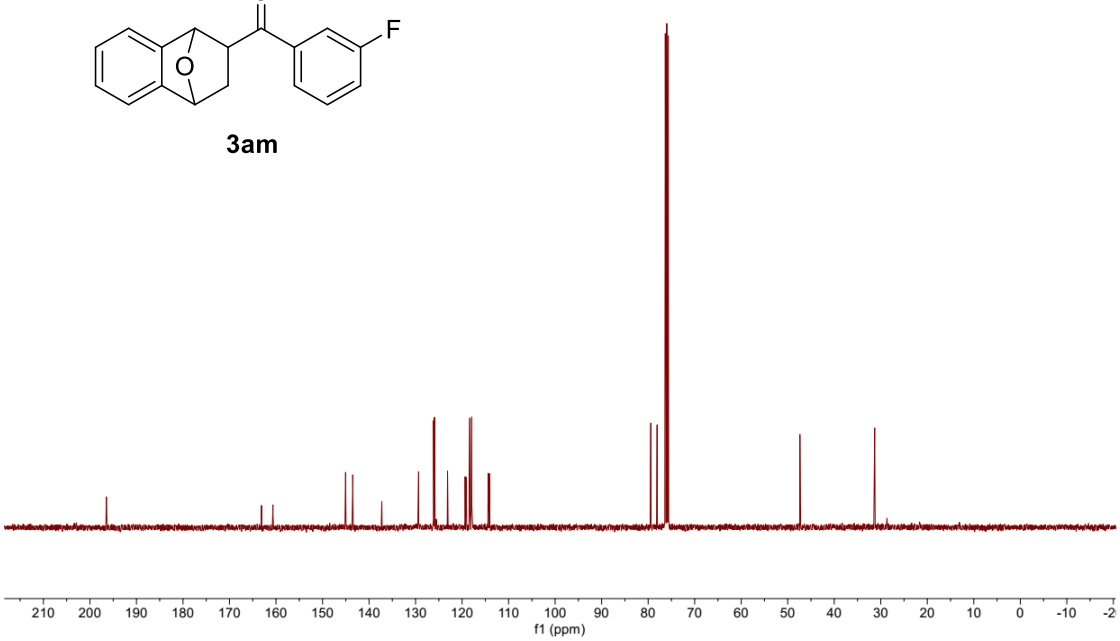


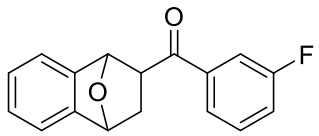
3am





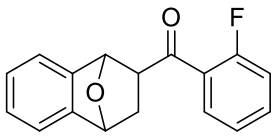
3am



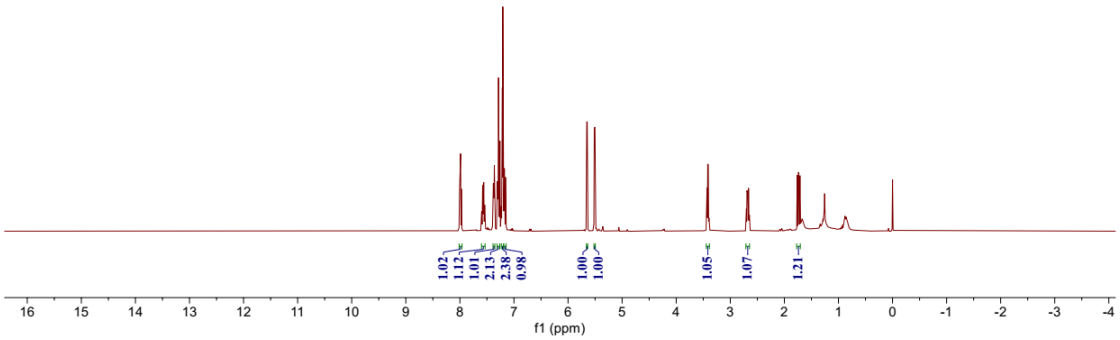


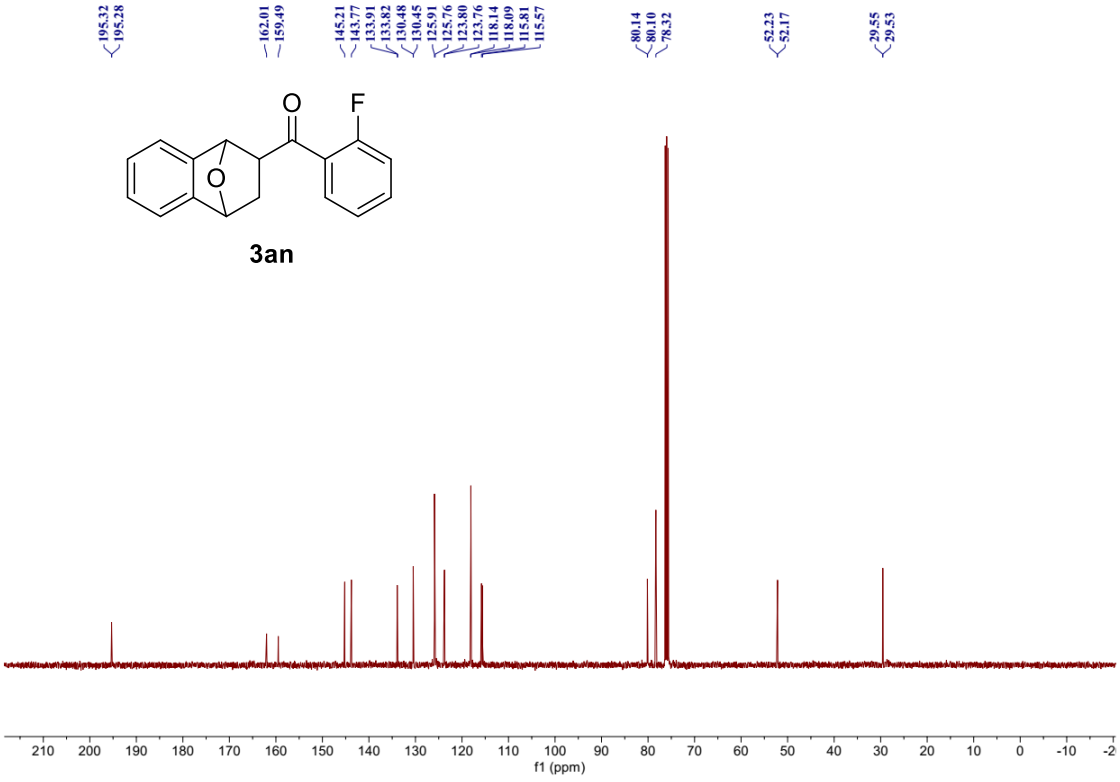
3am

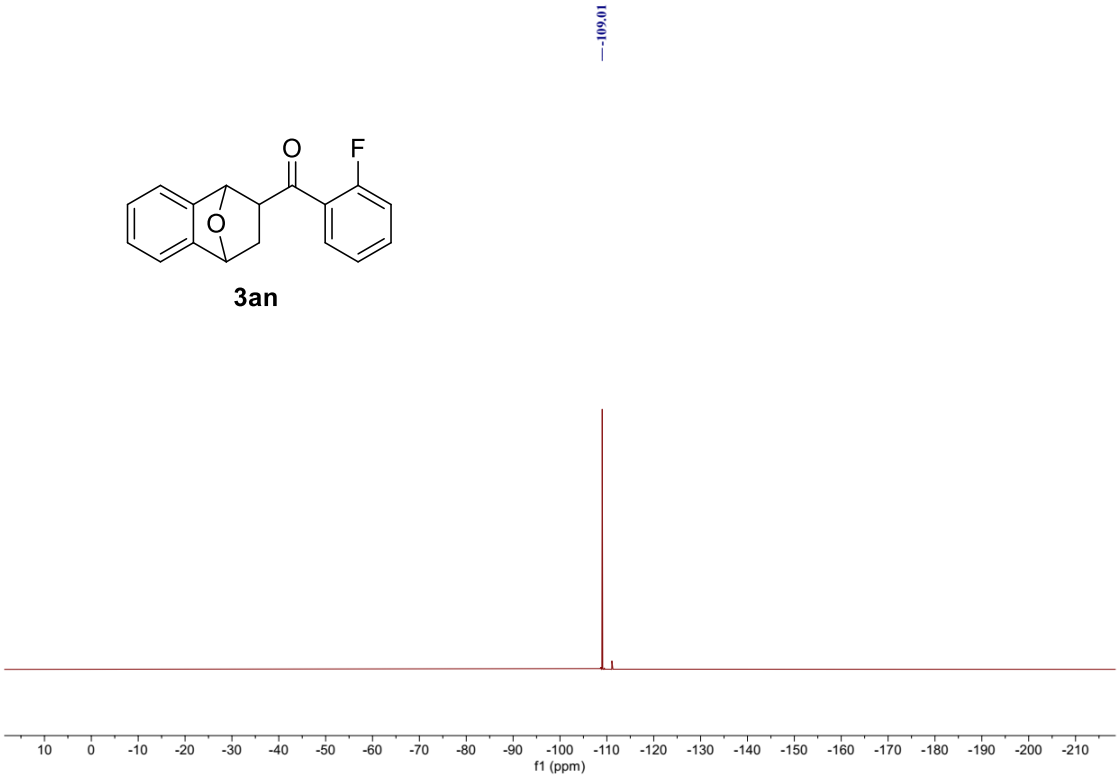
-111.38
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)



3an


1.02 1.12 1.01 2.13 2.38 0.98 1.00 1.00 1.05 1.07 1.21
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4
f1 (ppm)


195.32
195.28
162.01
159.49
145.21
143.77
133.91
133.82
130.48
130.45
125.91
125.76
123.80
123.76
118.14
118.09
115.81
115.57
80.14
80.10
78.32
52.23
52.17
29.55
29.53
O
F
O
3an
f1 (ppm)


-109.01
O
F
O
3an
f1 (ppm)


7.90
7.90
7.89
7.88
7.88
7.87
7.47
7.47
7.46
7.46
7.45
7.44
7.44
7.35
7.35
7.34
7.33
7.32
7.31
7.31
7.31
7.30
7.23
7.22
7.22
7.21
5.68
5.51
5.50
3.40
3.39
3.38
3.36
2.45
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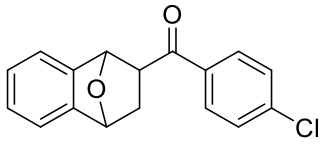
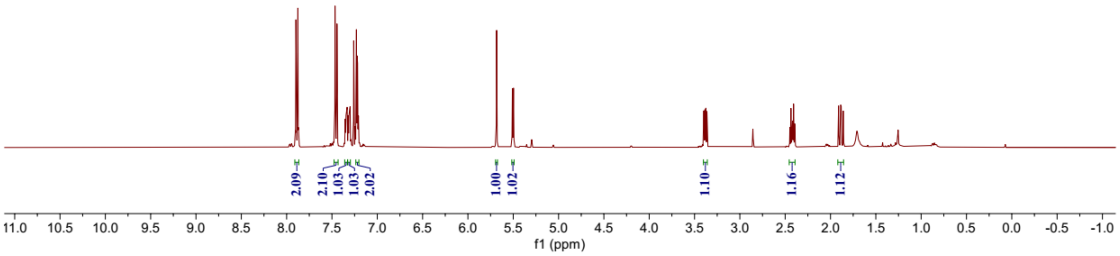
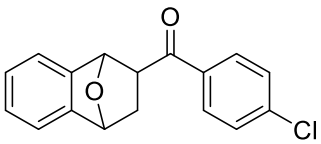
2.41
2.40
1.91
1.89
1.88
1.86


O
O
Cl

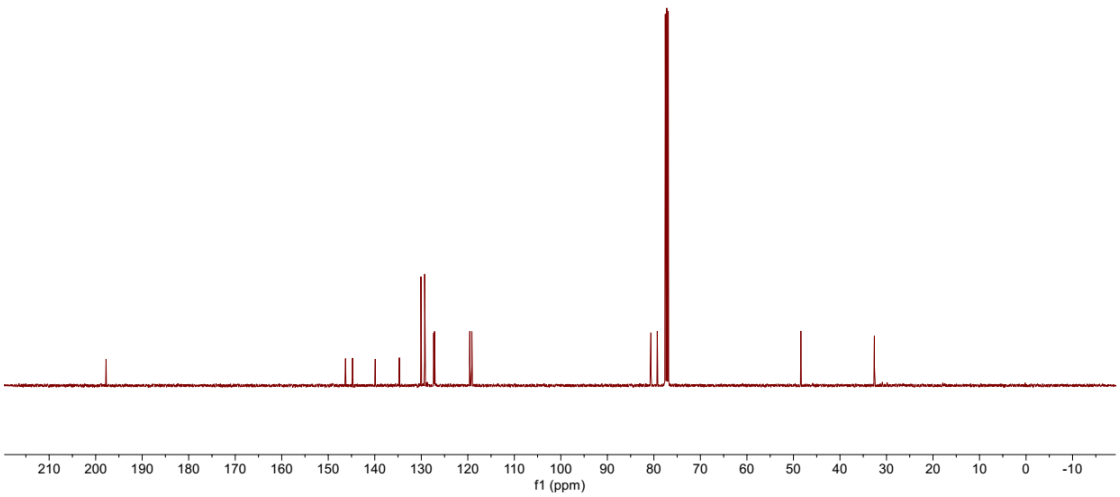
3ao

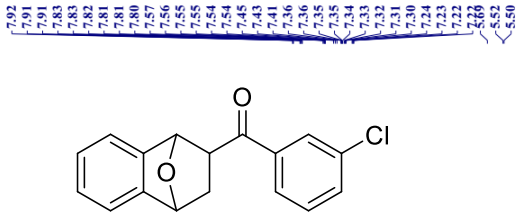

2.09
2.10
1.03
1.03
2.02
1.00
1.02
1.10
1.16
1.12
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


197.76
146.29
144.78
139.90
134.71
130.07
129.28
127.34
127.15
119.57
119.13
80.64
79.28
48.40
32.61

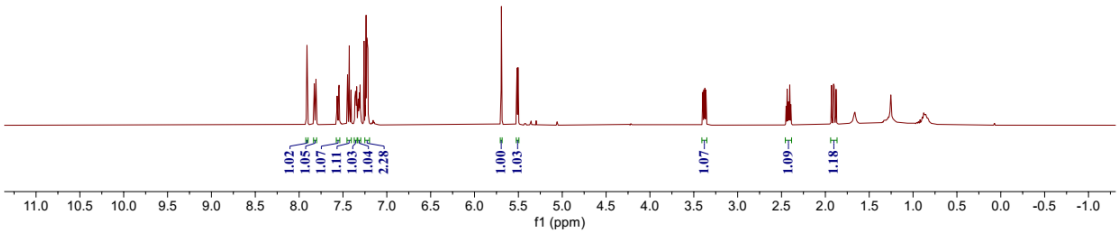

O
O
Cl

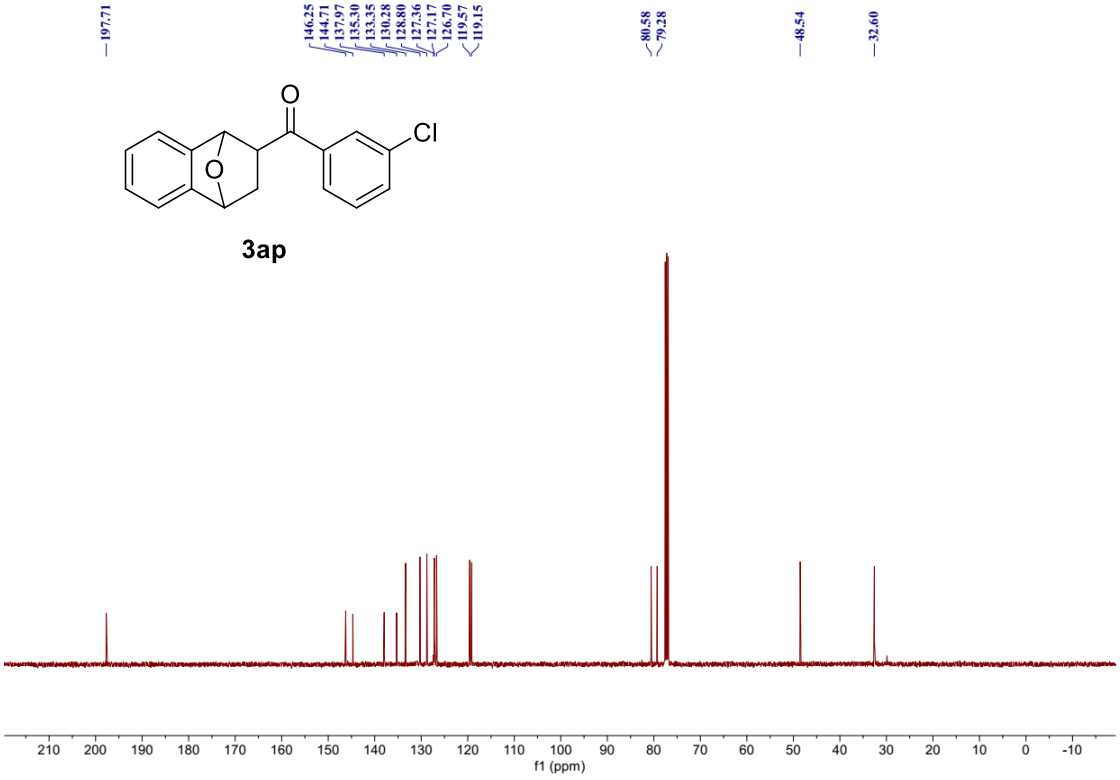
3ao


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)

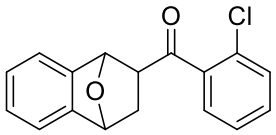

O
Cl
O

3ap

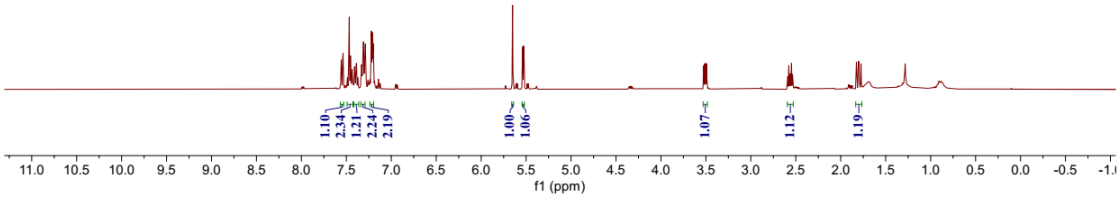

1.02
1.05
1.07
1.11
1.03
1.04
2.28
1.00
1.03
1.07
1.09
1.18
f1 (ppm)

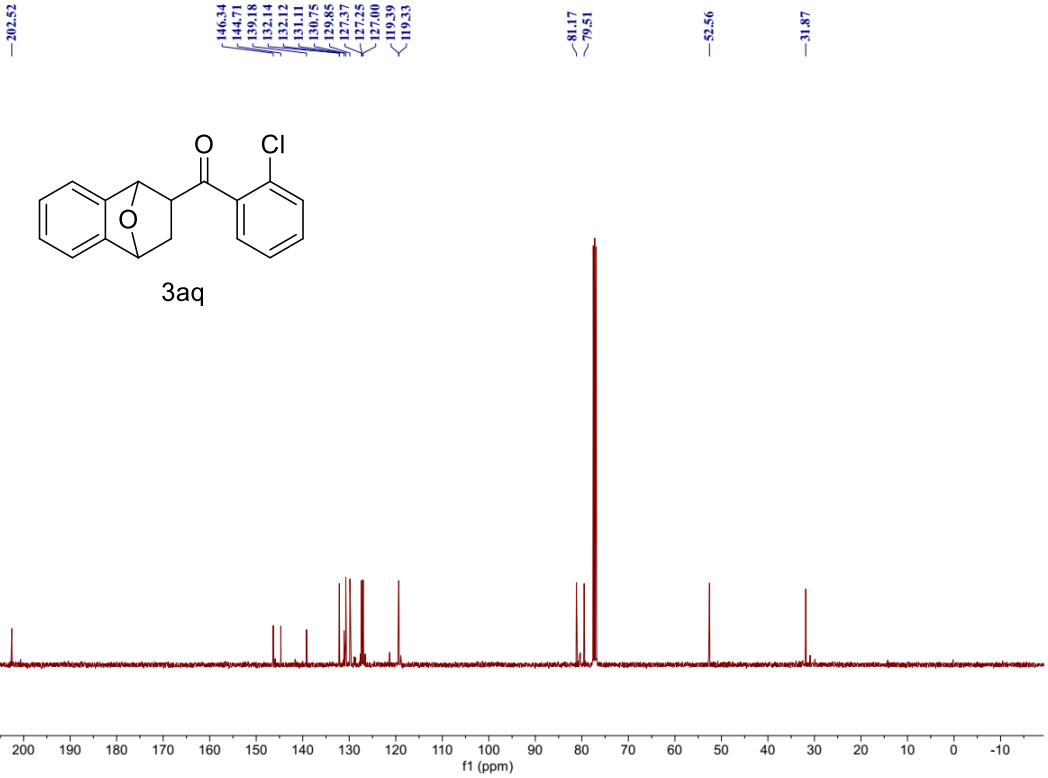

197.71
146.25
144.71
137.97
135.30
133.35
130.28
128.80
127.36
127.17
126.70
119.57
119.15
80.58
79.28
48.54
32.60
O
Cl
O
f1 (ppm)

3ap

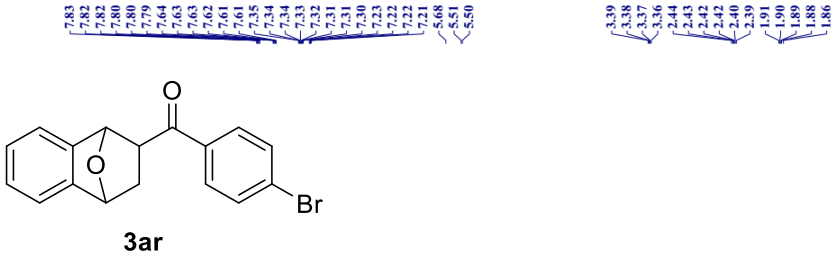


3aq

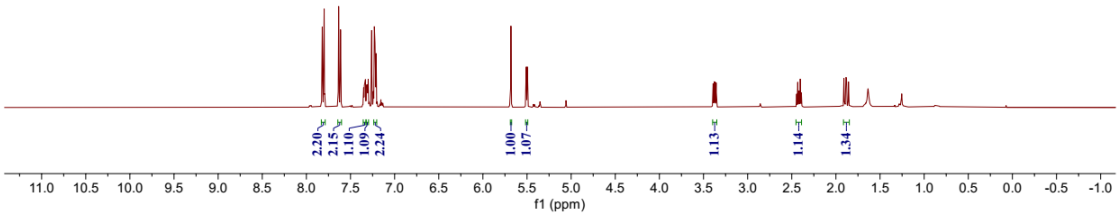


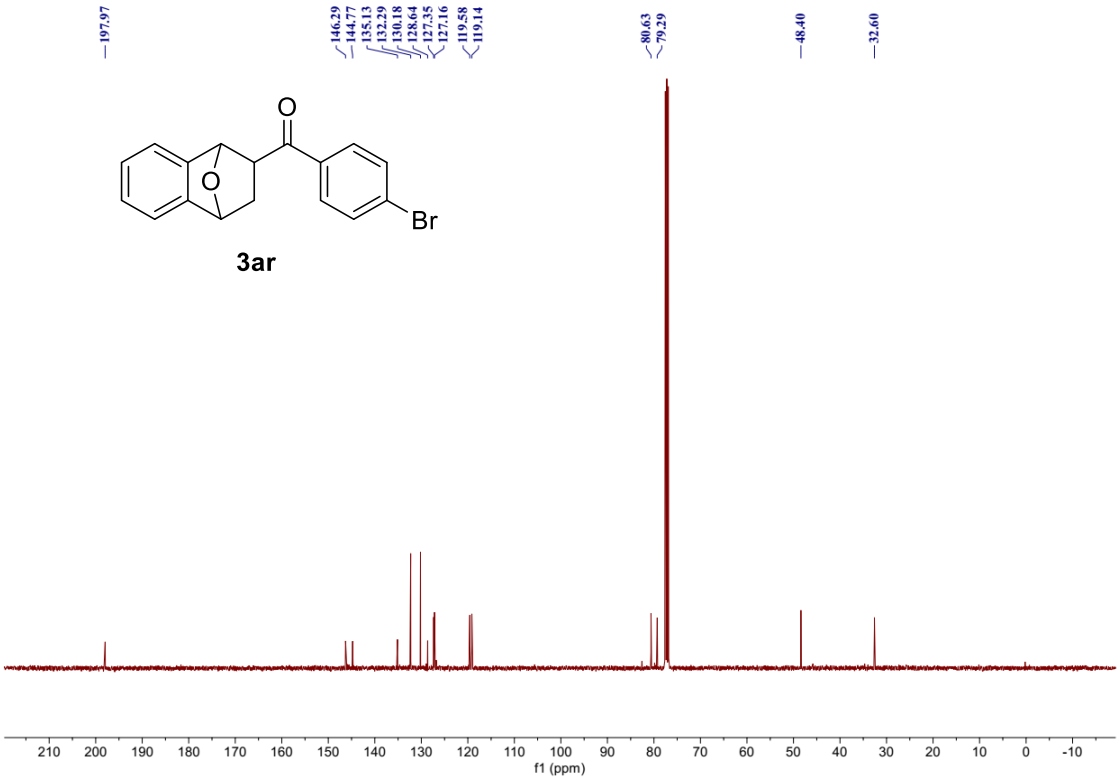


3aq

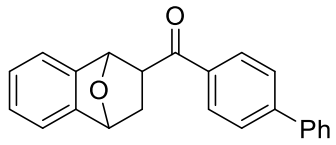

7.83
7.82
7.82
7.80
7.80
7.79
7.64
7.63
7.63
7.62
7.61
7.61
7.35
7.34
7.34
7.33
7.32
7.31
7.31
7.30
7.23
7.22
7.22
7.21
5.68
5.51
5.50
3.39
3.38
3.37
3.36
2.44
2.43
2.42
2.42
2.40
2.39
1.91
1.90
1.89
1.88
1.86
O
O
Br

3ar

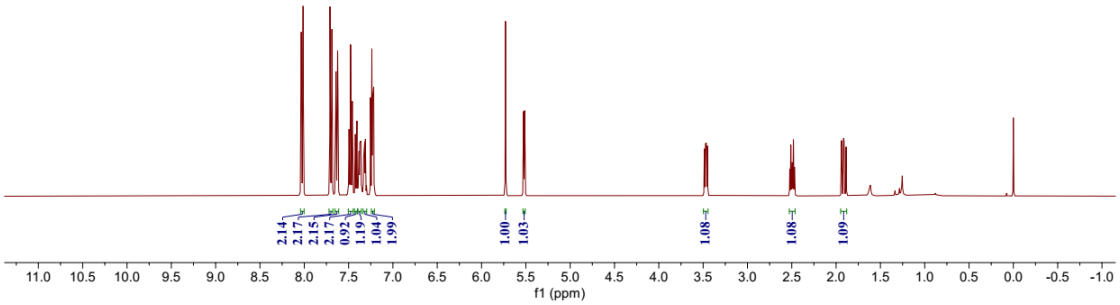

2.20
2.15
1.10
1.09
2.24
1.00
1.07
1.13
1.14
1.34
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

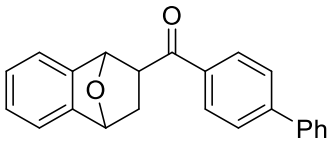

197.97
146.29
144.77
135.13
132.29
130.18
128.64
127.35
127.16
119.58
119.14
80.63
79.29
48.40
32.60
O
O
Br
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

3ar

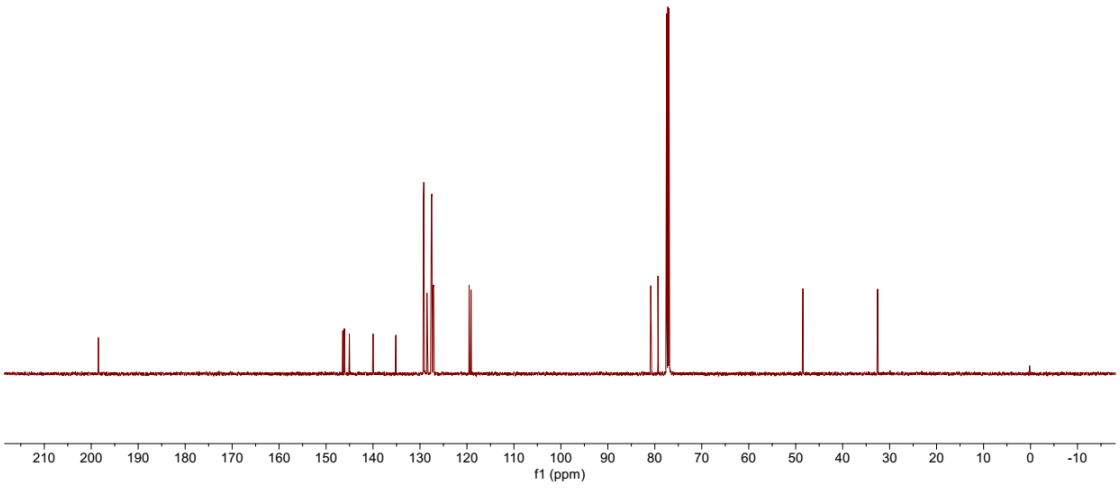

O
O
Ph

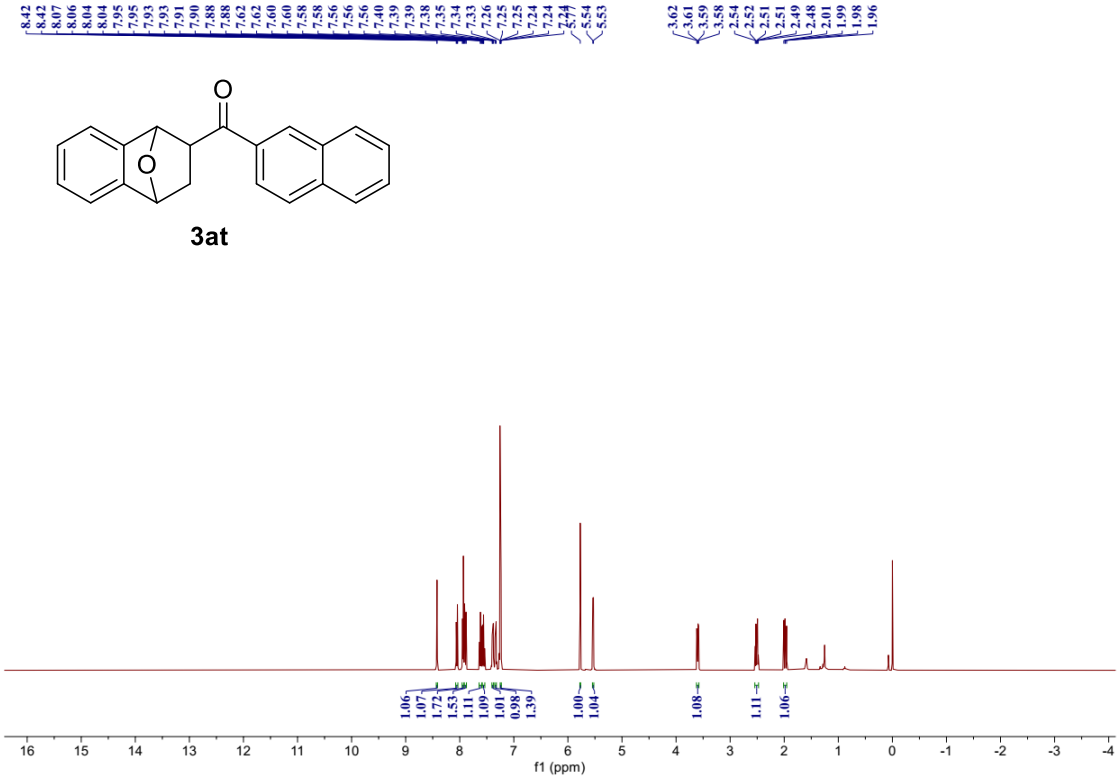
3as

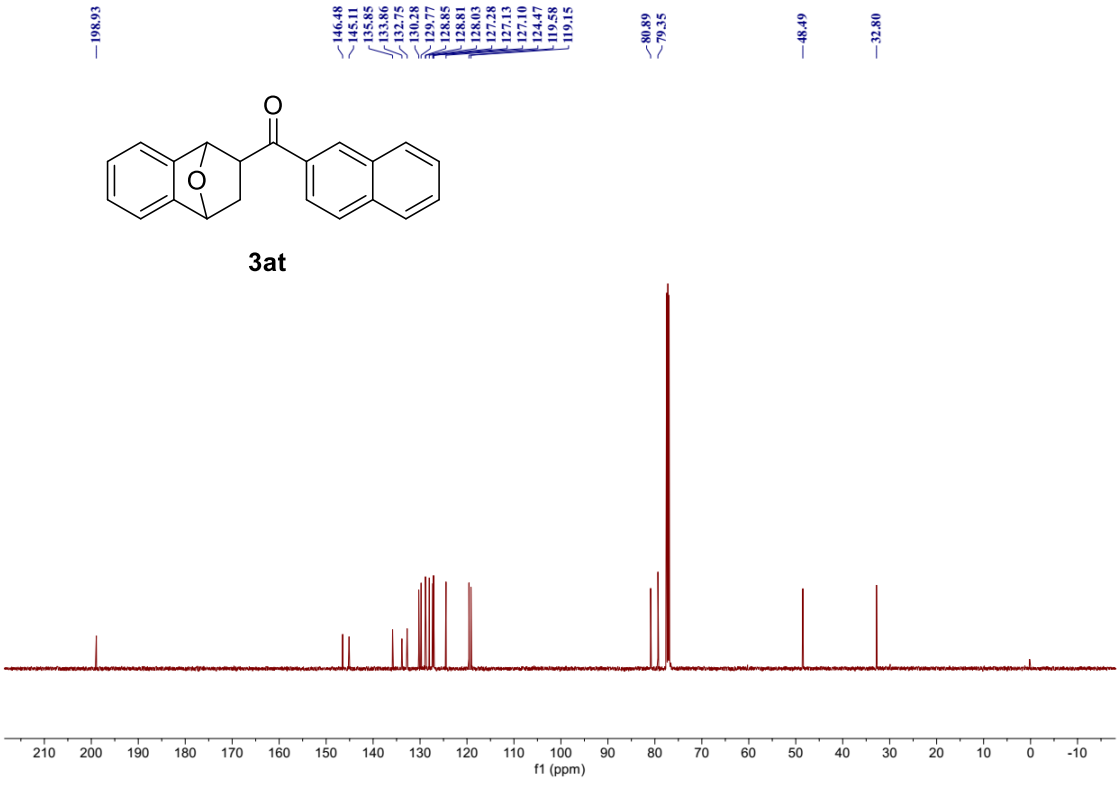

2.14
2.17
2.15
2.17
0.92
1.19
1.04
1.99
1.00
1.03
1.08
1.08
1.09
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

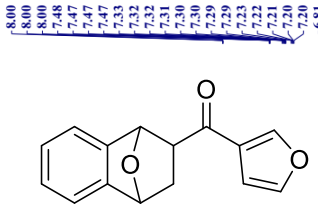

O
O
Ph

3as

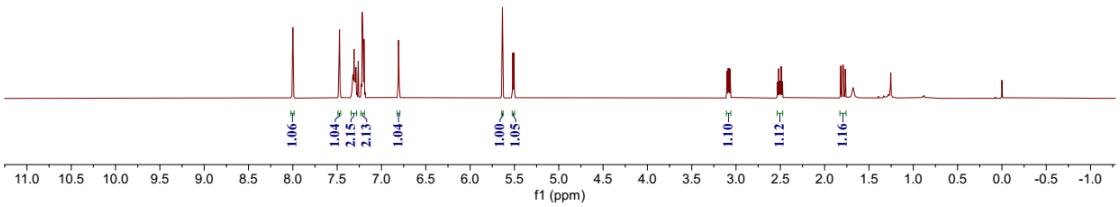

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

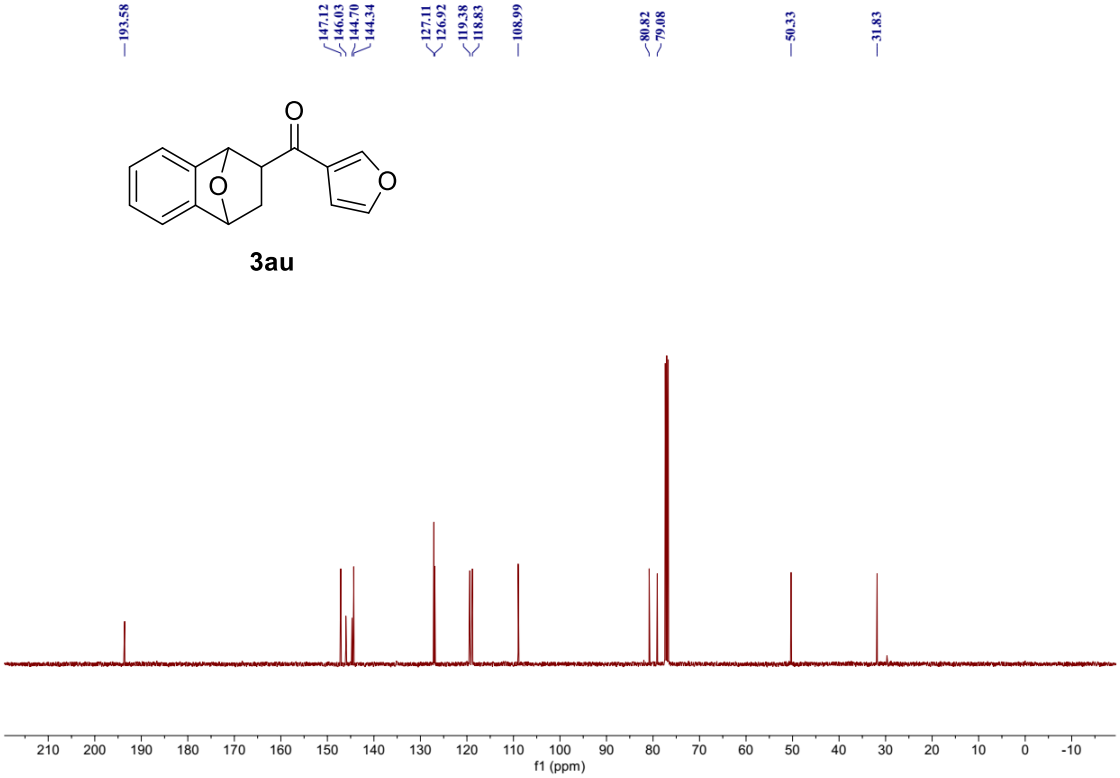

8.42
8.42
8.07
8.06
8.04
8.04
7.95
7.95
7.93
7.93
7.91
7.90
7.88
7.88
7.62
7.62
7.60
7.60
7.58
7.58
7.56
7.56
7.56
7.40
7.39
7.39
7.38
7.35
7.34
7.33
7.26
7.25
7.25
7.24
7.24
7.24
5.77
5.54
5.53
3.62
3.61
3.59
3.58
2.54
2.52
2.51
2.51
2.49
2.48
2.01
1.99
1.98
1.96
O
O
3at
1.06
1.07
1.72
1.53
1.11
1.09
1.01
0.98
1.39
1.00
1.04
1.08
1.11
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)


198.93
146.48
145.11
135.85
133.86
132.75
130.28
129.77
128.85
128.81
128.03
127.28
127.13
127.10
124.47
119.58
119.15
80.89
79.35
48.49
32.80
O
O
3at
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)



3au





3au


8.03
8.02
8.02
8.01
7.57
7.57
7.56
7.56
7.36
7.36
7.35
7.34
7.34
7.34
7.33
7.32
7.31
7.30
7.30
7.29
7.23
7.22
7.21
7.21
7.20
7.19
5.66
5.51
5.50
3.30
3.29
3.28
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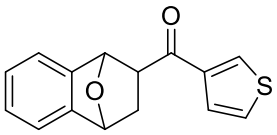
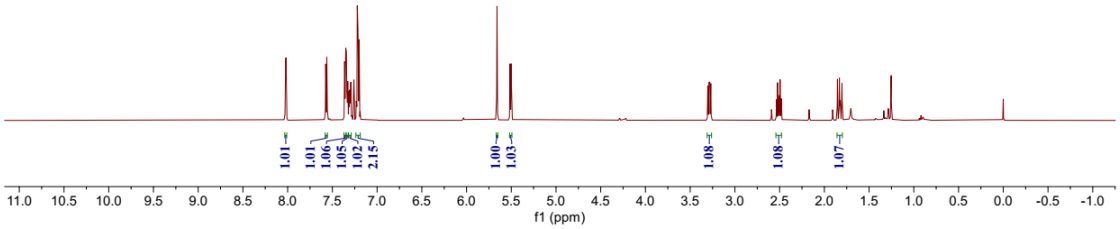
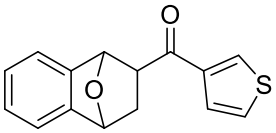
2.49
2.48
1.85
1.83
1.83
1.80


O
O
S

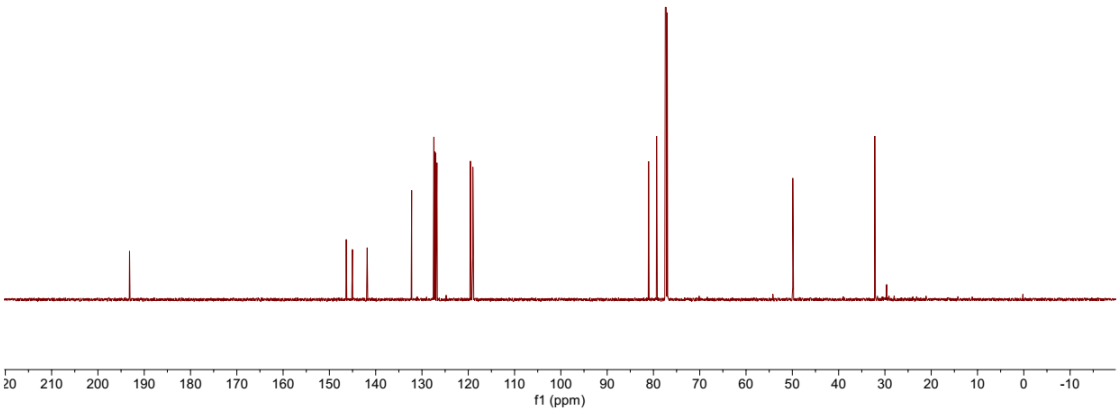
3av

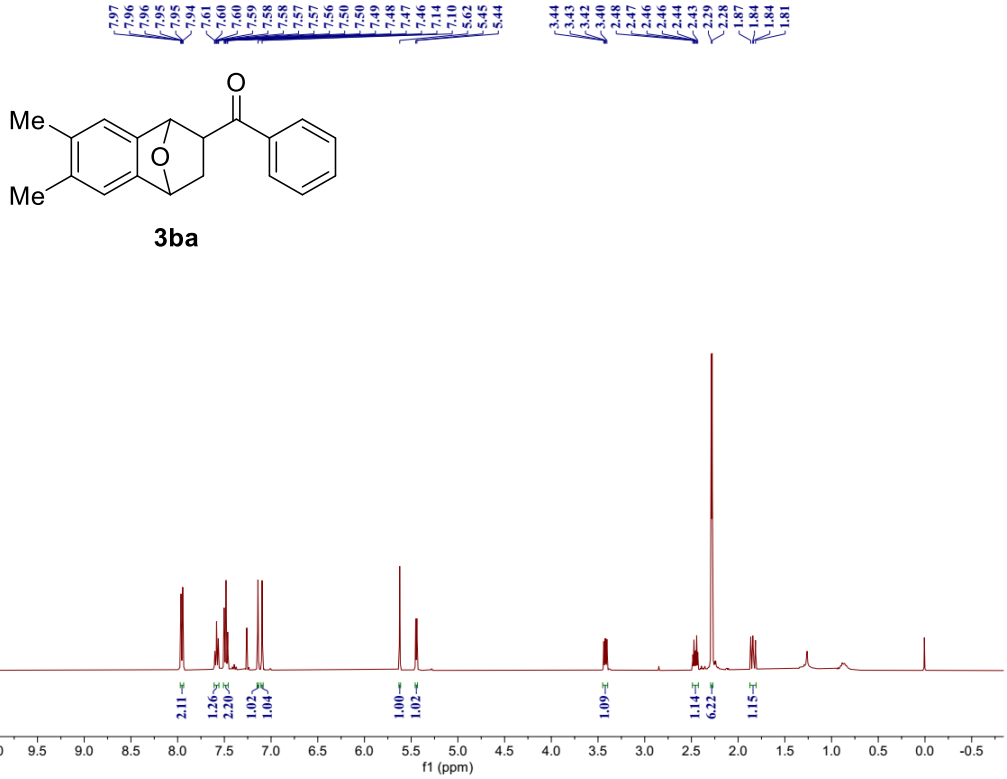

1.01
1.01
1.06
1.05
1.02
2.15
1.00
1.03
1.08
1.08
1.07
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

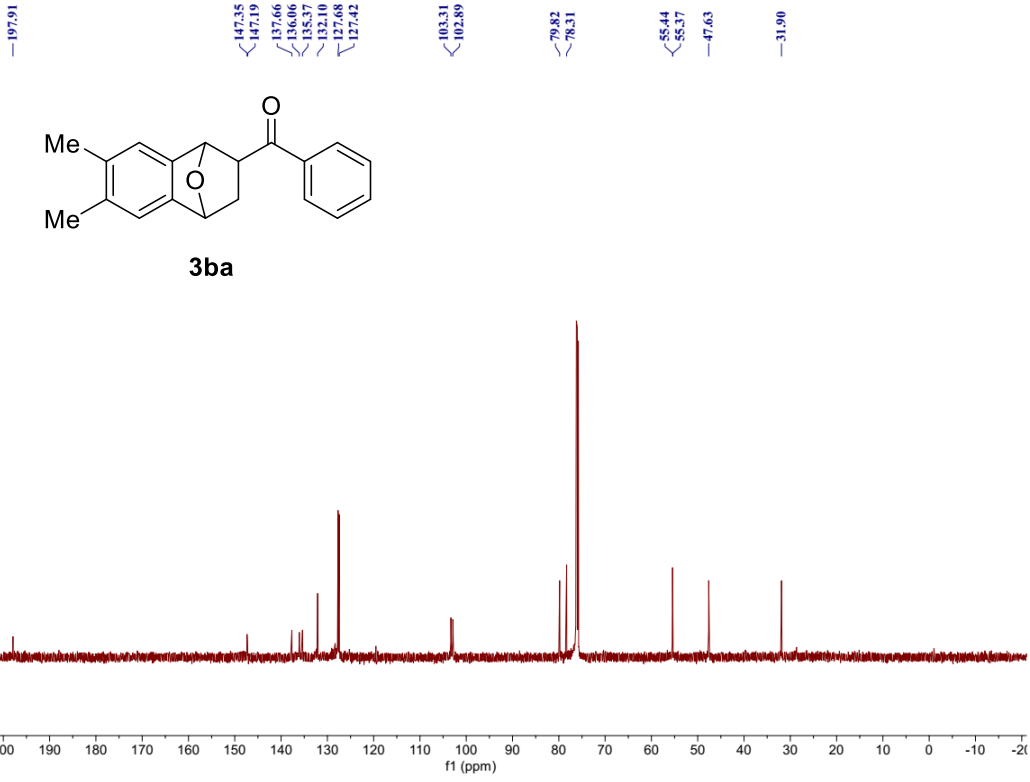

193.17
146.36
145.02
141.84
132.23
127.43
127.26
127.07
126.79
126.77
119.54
119.01
81.05
79.31
49.90
32.19

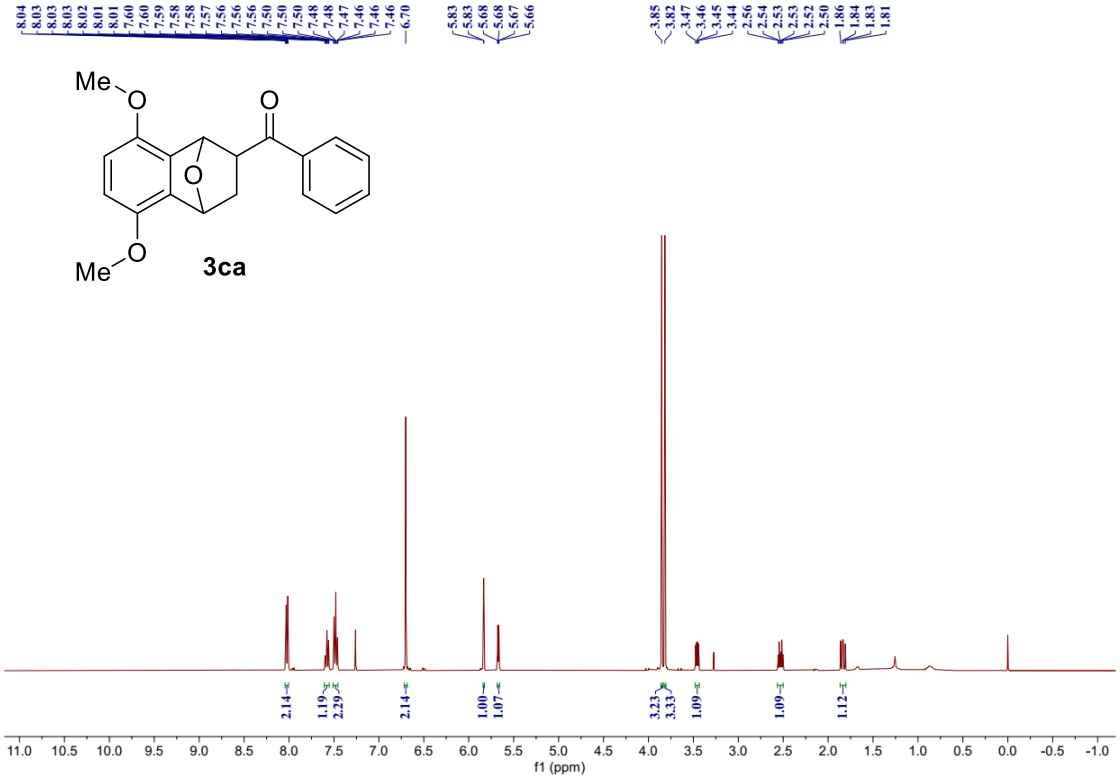

O
O
S

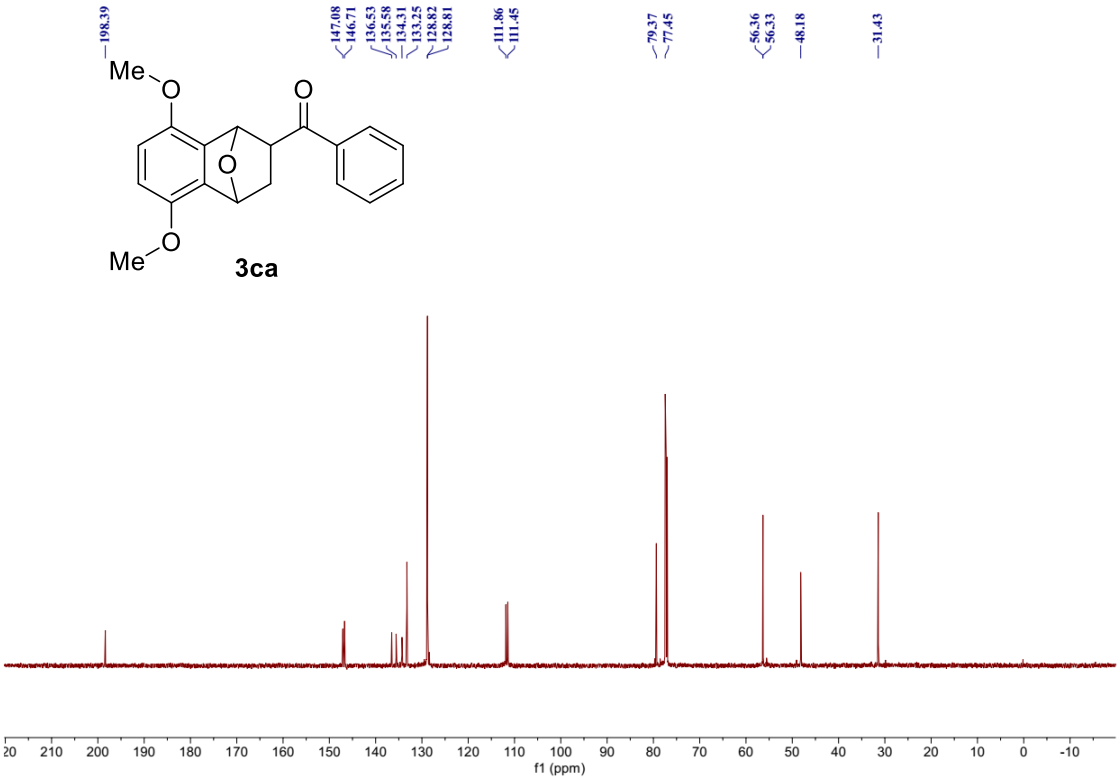
3av

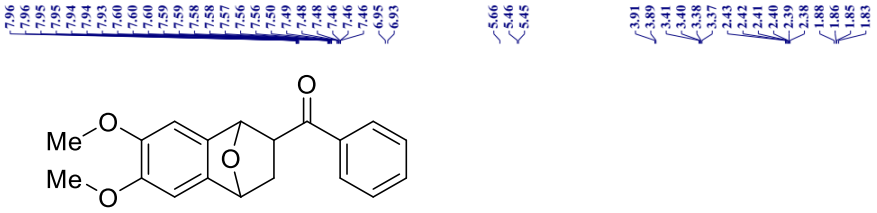

220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.97
7.96
7.96
7.95
7.95
7.94
7.61
7.60
7.60
7.59
7.58
7.58
7.57
7.57
7.56
7.50
7.50
7.49
7.48
7.47
7.46
7.14
7.10
5.62
5.45
5.44
3.44
3.43
3.42
3.40
2.48
2.47
2.46
2.46
2.44
2.43
2.29
2.28
1.87
1.84
1.84
1.81
O
Me
O
Me
3ba
2.11
1.26
2.20
1.02
1.04
1.00
1.02
1.09
1.14
6.22
1.15
1.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


197.91
147.35
147.19
137.66
136.06
135.37
132.10
127.68
127.42
103.31
102.89
79.82
78.31
55.44
55.37
47.63
31.90
O
Me
O
Me
3ba
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

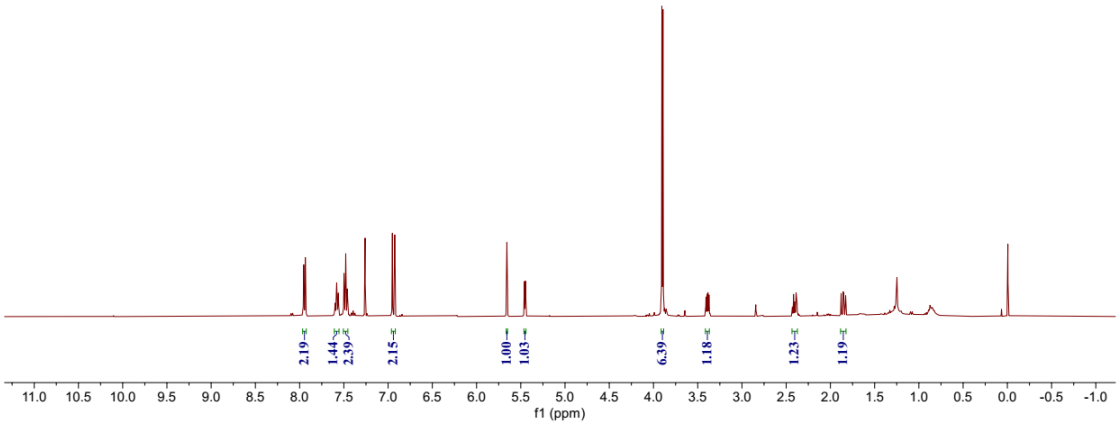

8.04 8.03 8.03 8.03 8.02 8.01 8.01 7.60 7.60 7.59 7.58 7.58 7.57 7.56 7.56 7.56 7.50 7.50 7.50 7.48 7.47 7.46 7.46 7.46 6.70
5.83 5.83 5.68 5.68 5.67 5.66
3.85 3.82 3.47 3.46 3.45 3.44 2.56 2.54 2.53 2.53 2.52 2.50 1.86 1.84 1.83 1.81
Me
O
O
O
Me
O
3ca
2.14 1.19 2.29 2.14 1.00 1.07 3.23 3.33 1.09 1.09 1.12
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


198.39
147.08 146.71 136.53 135.58 134.31 133.25 128.82 128.81 111.86 111.45 79.37 77.45 56.36 56.33 48.18 31.43
Me
O
O
O
Me
O
3ca
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)

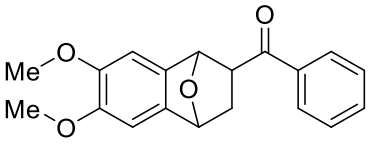

7.96
7.96
7.95
7.95
7.94
7.94
7.93
7.60
7.60
7.60
7.59
7.59
7.58
7.58
7.57
7.56
7.56
7.50
7.49
7.48
7.48
7.46
7.46
7.46
6.95
6.93
5.66
5.46
5.45
3.91
3.89
3.41
3.40
3.38
3.37
2.43
2.42
2.42
2.41
2.40
2.39
2.38
1.88
1.86
1.85
1.83

O
Me
O
Me
O
O

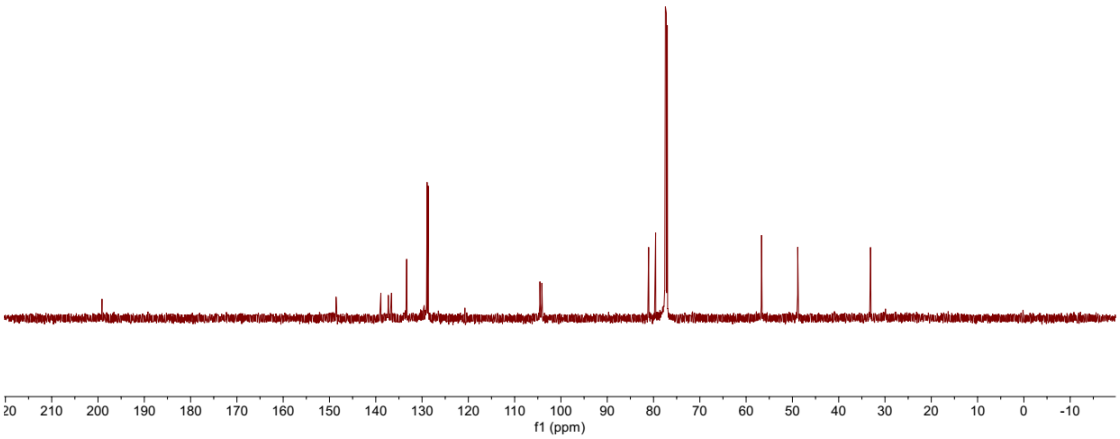
3da


2.19
1.44
2.39
2.15
1.00
1.03
6.39
1.18
1.23
1.19
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

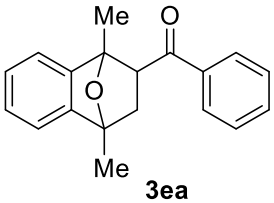

199.14
148.58
148.42
138.89
137.29
136.60
133.33
128.91
128.65
104.54
104.12
81.05
79.54
56.67
56.60
48.86
33.13


O
Me
O
Me
O
O

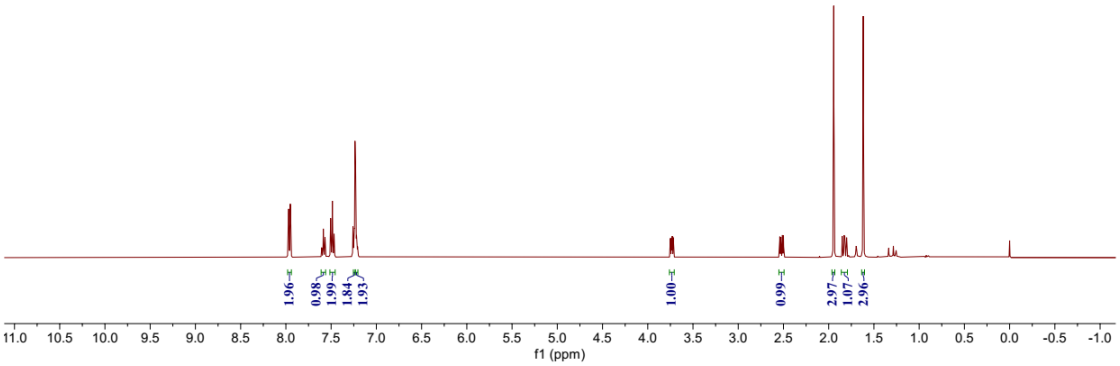
3da

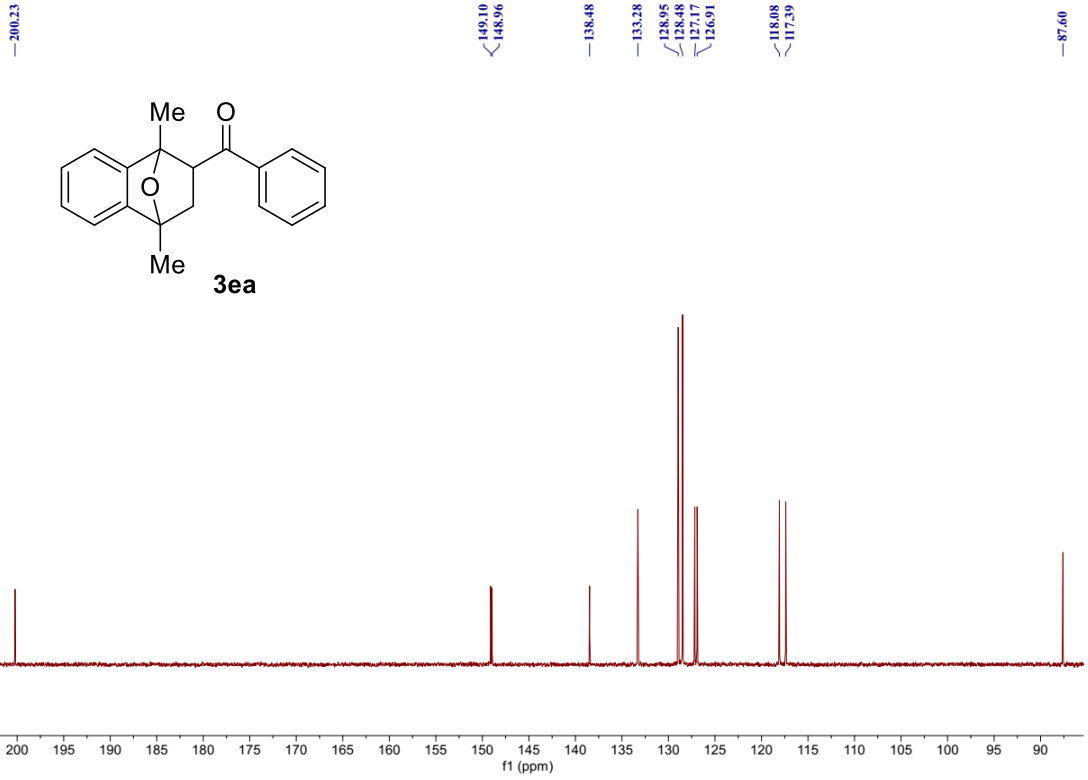

220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.98
7.97
7.97
7.97
7.96
7.95
7.95
7.61
7.60
7.60
7.59
7.59
7.58
7.57
7.57
7.56
7.52
7.51
7.50
7.49
7.49
7.47
7.47
7.25
7.25
7.24
7.24
7.23
7.22
7.21
7.21
3.75
3.74
3.73
3.72
2.54
2.53
2.51
2.50
1.94
1.85
1.84
1.83
1.82
1.80
1.62


Me
O
O
Me

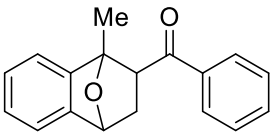
3ea


1.96
0.98
1.99
1.84
1.93
1.00
0.99
2.97
1.07
2.96
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

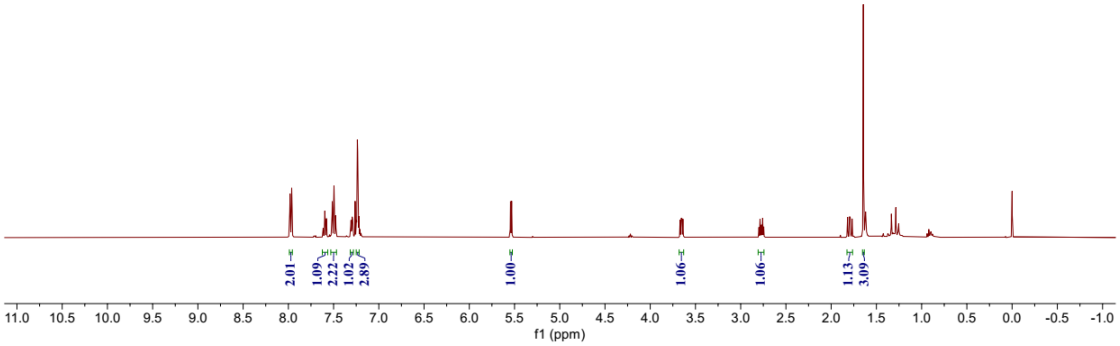

200.23
149.10
148.96
138.48
133.28
128.95
128.48
127.17
126.91
118.08
117.39
87.60
Me
O
O
Me
200 195 190 185 180 175 170 165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90
f1 (ppm)

3ea


7.99
7.98
7.98
7.98
7.97
7.96
7.96
7.62
7.61
7.60
7.60
7.59
7.58
7.58
7.58
7.52
7.51
7.50
7.50
7.48
7.48
7.31
7.31
7.30
7.30
7.29
7.29
7.28
7.25
7.24
7.24
7.23
7.23
7.22
5.54
5.53
3.67
3.66
3.65
3.64
2.80
2.79
2.77
2.77
2.76
2.75
1.82
1.80
1.79
1.78
1.77
1.64


Me
O
O

3fa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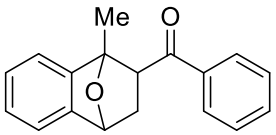
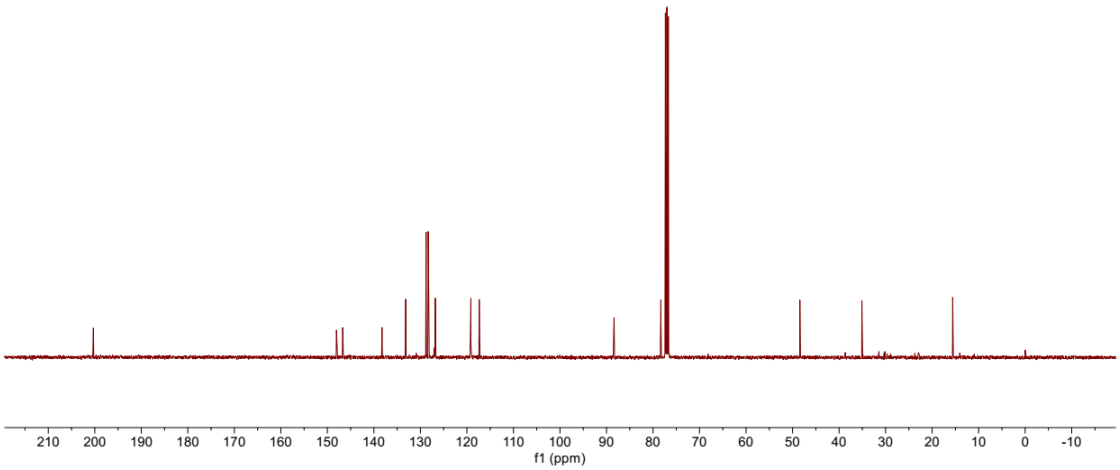
1.09
2.22
1.02
2.89
1.00
1.06
1.06
1.13
3.09
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


200.30
148.05
146.70
138.26
133.16
128.78
128.32
126.95
126.81
119.15
117.35
88.38
78.30
48.43
35.10
15.58


Me
O
O

3fa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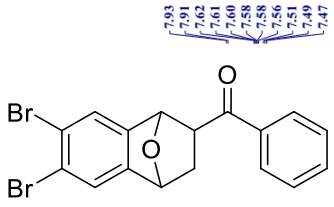
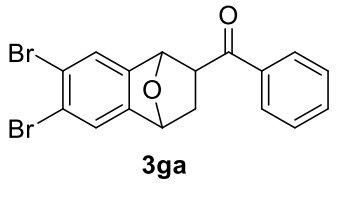
f1 (ppm)


Br
Br
O
O

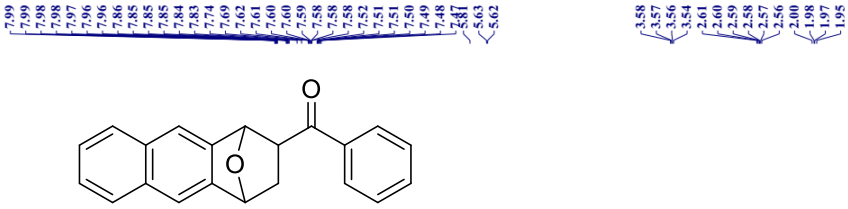
3ga

7.93
7.91
7.62
7.61
7.60
7.58
7.58
7.56
7.51
7.49
7.47
5.66
5.45
5.44
3.44
3.43
3.42
3.41
2.49
2.48
2.48
2.47
2.47
2.46
2.45
2.45
2.44
2.44
2.43
1.91
1.89
1.88
1.86
2.19
1.82
1.44
2.26
1.00
1.05
1.09
1.12
1.17
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


Br
Br
O
O

3ga

197.91
147.44
146.06
136.14
133.60
129.03
128.65
124.97
124.55
123.22
122.98
80.26
78.79
47.98
32.20
20 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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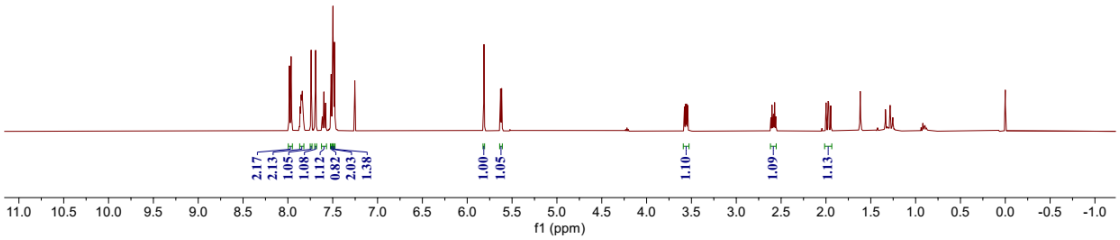
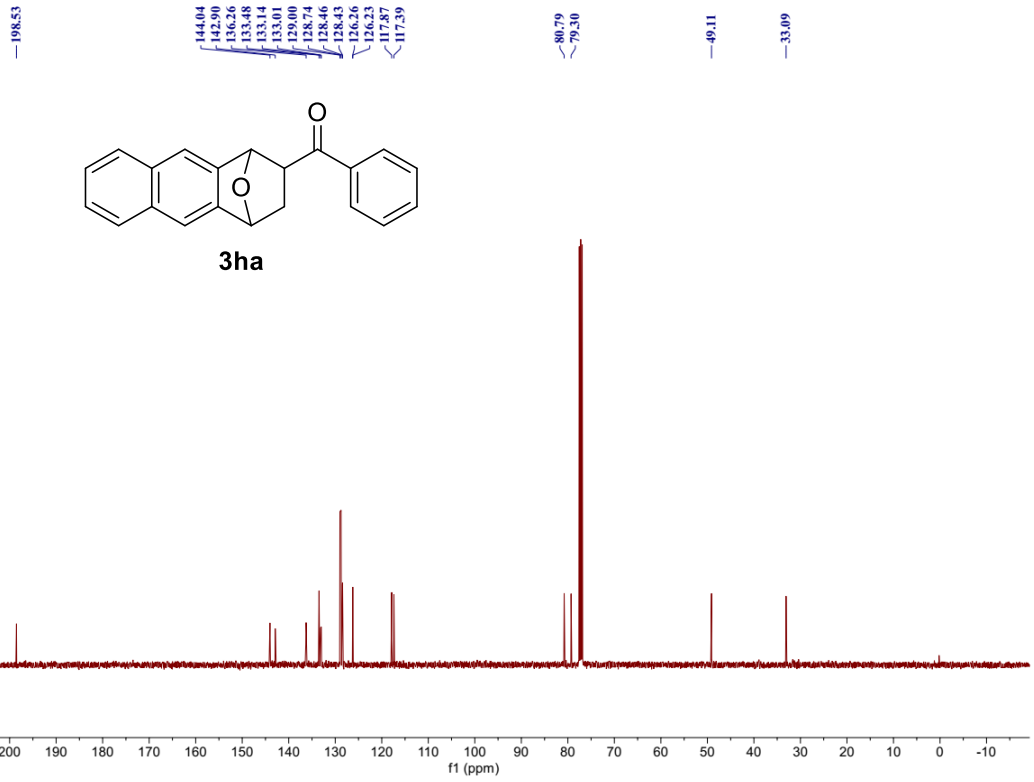
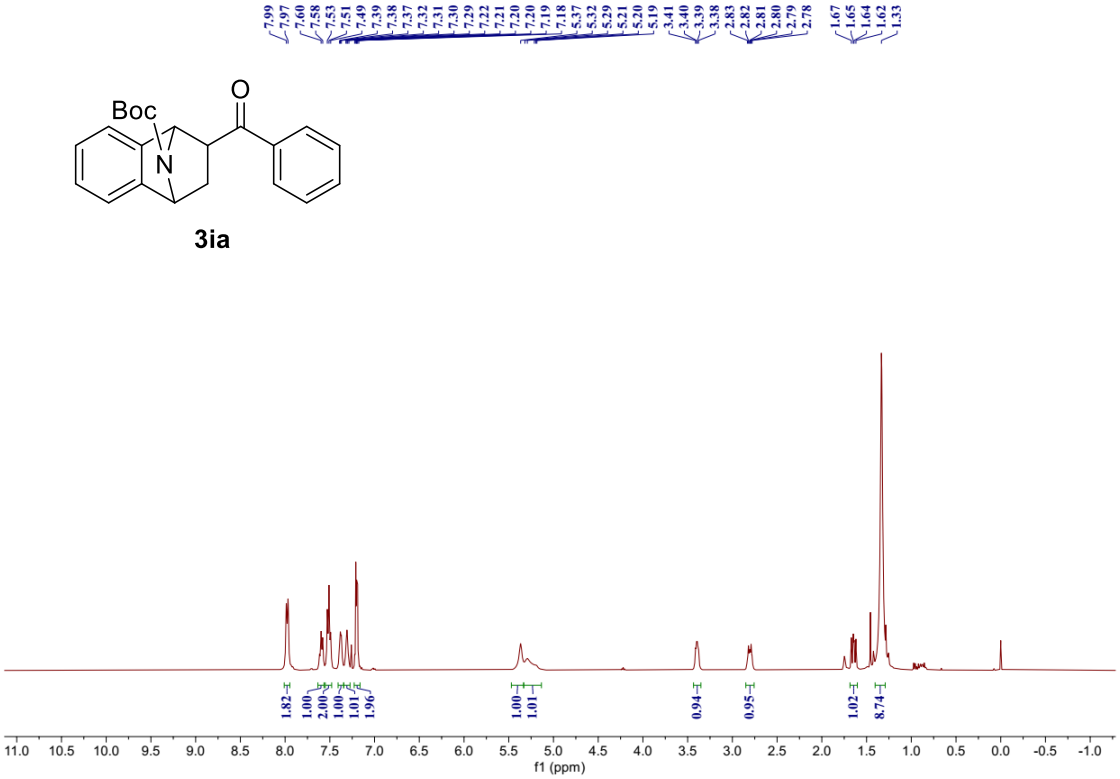
7.99
7.98
7.98
7.98
7.97
7.96
7.96
7.86
7.85
7.85
7.85
7.84
7.83
7.74
7.69
7.62
7.61
7.60
7.60
7.60
7.59
7.58
7.58
7.58
7.52
7.51
7.51
7.50
7.49
7.48
7.47
5.81
5.63
5.62
3.58
3.57
3.56
3.54
2.61
2.60
2.59
2.58
2.57
2.56
2.00
1.98
1.97
1.95
O
O

3ha


2.17
2.13
1.05
1.08
1.12
0.82
2.03
1.38
1.00
1.05
1.10
1.09
1.13
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


198.53
144.04
142.90
136.26
133.48
133.14
133.01
129.00
128.74
128.46
128.43
126.26
126.23
117.87
117.39
80.79
79.30
49.11
33.09
O
O
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)

3ha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7.97
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7.58
7.53
7.51
7.49
7.39
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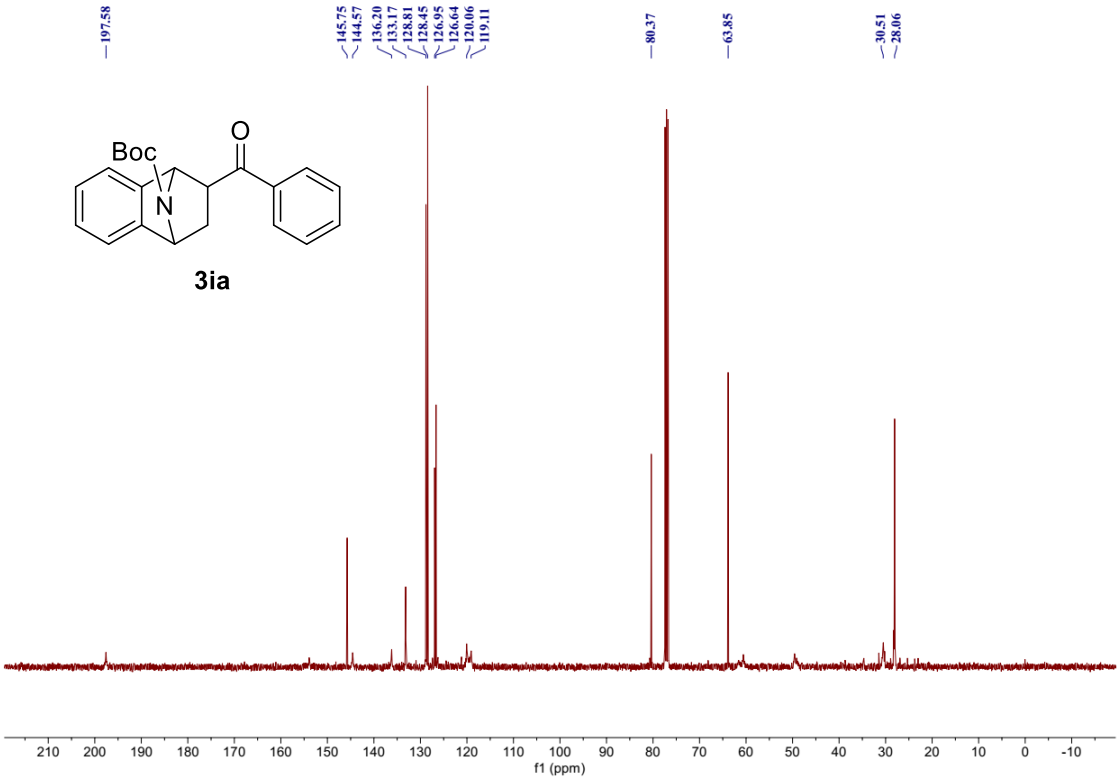
7.37
7.32
7.31
7.30
7.29
7.22
7.21
7.20
7.20
7.19
7.18
5.37
5.32
5.29
5.21
5.20
5.19
3.41
3.40
3.39
3.38
2.83
2.82
2.81
2.80
2.79
2.78
1.67
1.65
1.64
1.62
1.33
Boc
O
N
3ia
1.82
1.00
2.00
1.00
1.01
1.96
1.00
1.01
0.94
0.95
1.02
8.74
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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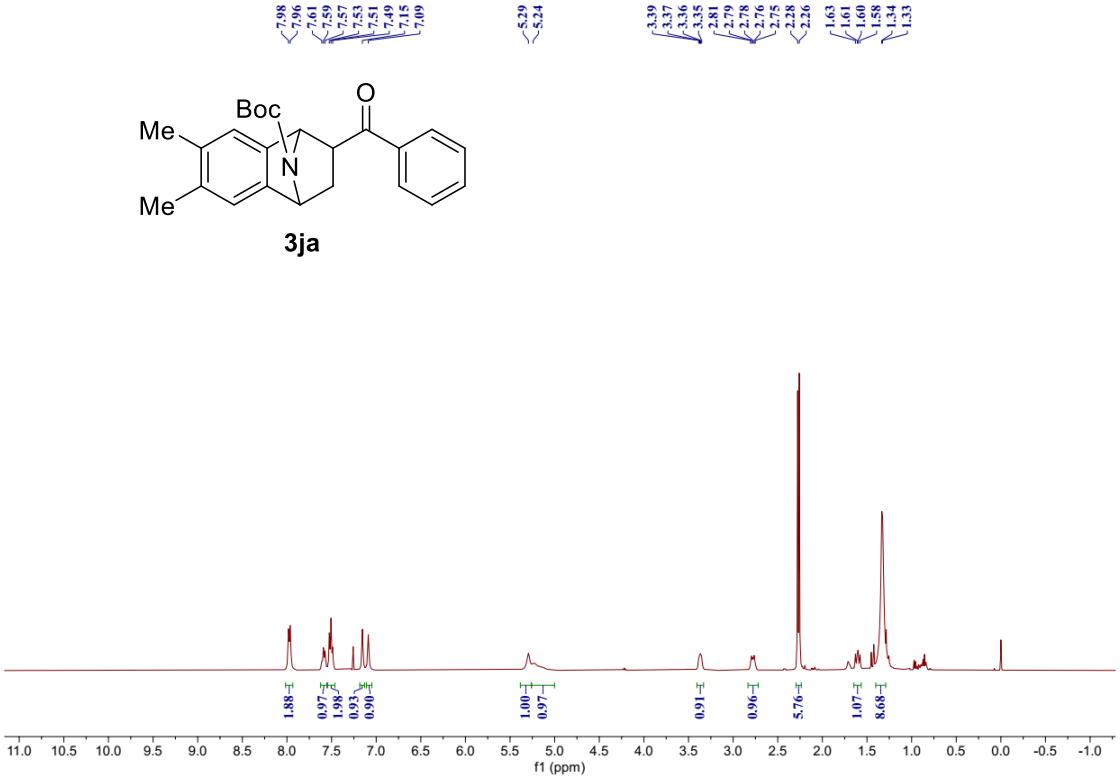
145.75
144.57
136.20
133.17
128.81
128.45
126.95
126.64
120.06
119.11
80.37
63.85
30.51
28.06
Boc
O
N
3ia
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


7.98
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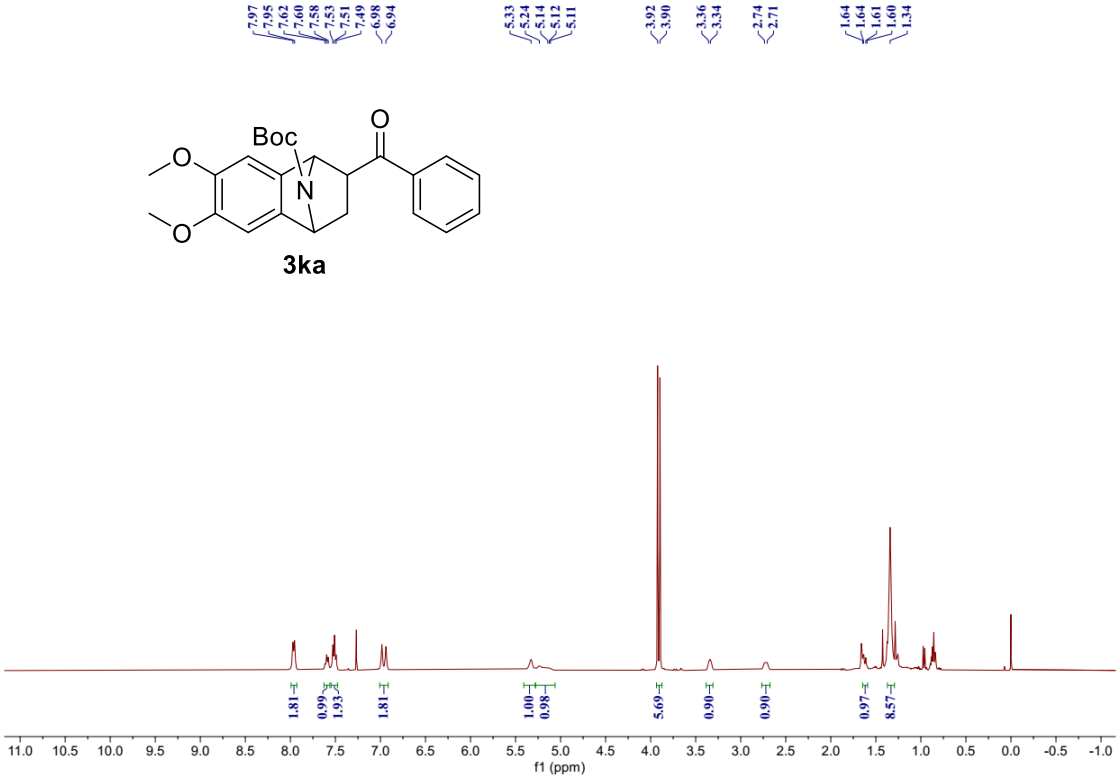
7.61
7.59
7.57
7.53
7.51
7.49
7.15
7.09
5.29
5.24
3.39
3.37
3.36
3.35
2.81
2.79
2.78
2.76
2.75
2.28
2.26
1.63
1.61
1.60
1.58
1.34
1.33
Boc
O
Me
N
Me
3ja
1.88
0.97
1.98
0.93
0.90
1.00
0.97
0.91
0.96
5.76
1.07
8.68
f1 (ppm)
197.89
143.68
142.48
136.43
135.10
134.76
133.24
128.93
128.60
121.56
120.61
80.34
63.88
31.00
28.25
20.16
Boc
O
Me
N
Me
3ja
f1 (ppm)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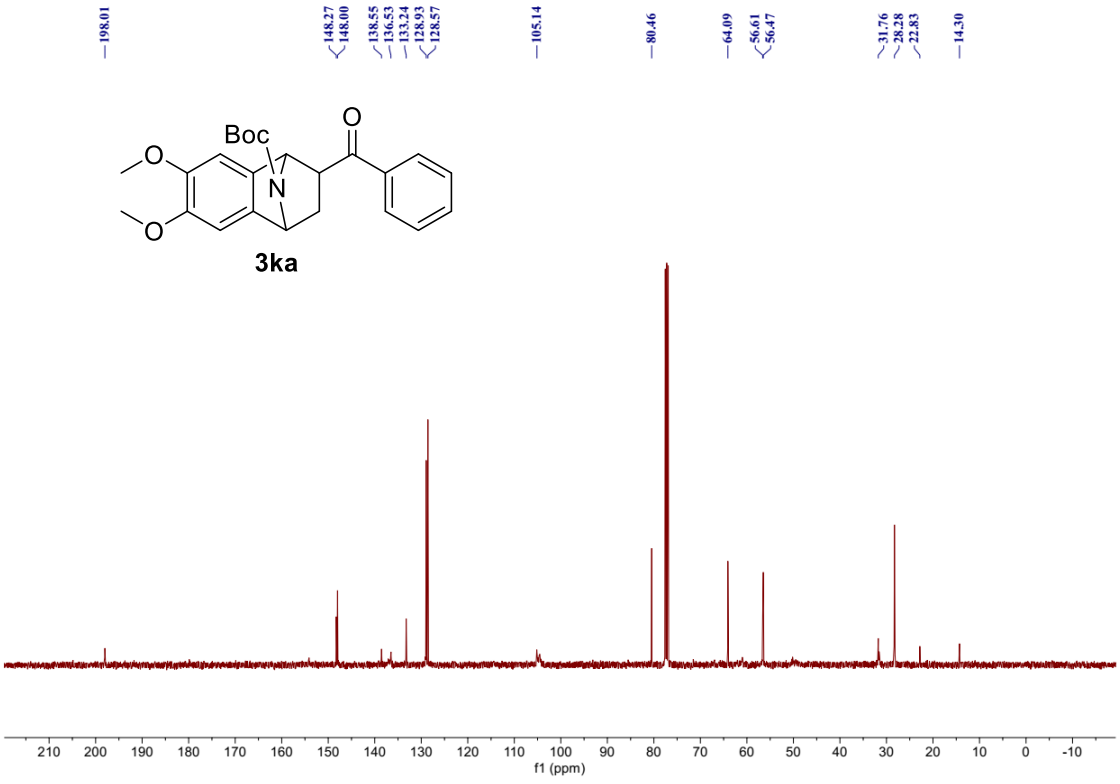
7.62
7.60
7.58
7.53
7.51
7.49
6.98
6.94
5.33
5.24
5.14
5.12
5.11
3.92
3.90
3.36
3.34
2.74
2.71
1.64
1.64
1.61
1.60
1.34
Boc
O
O
O
N
3ka
1.81
0.99
1.93
1.81
1.00
0.98
5.69
0.90
0.90
0.97
8.57
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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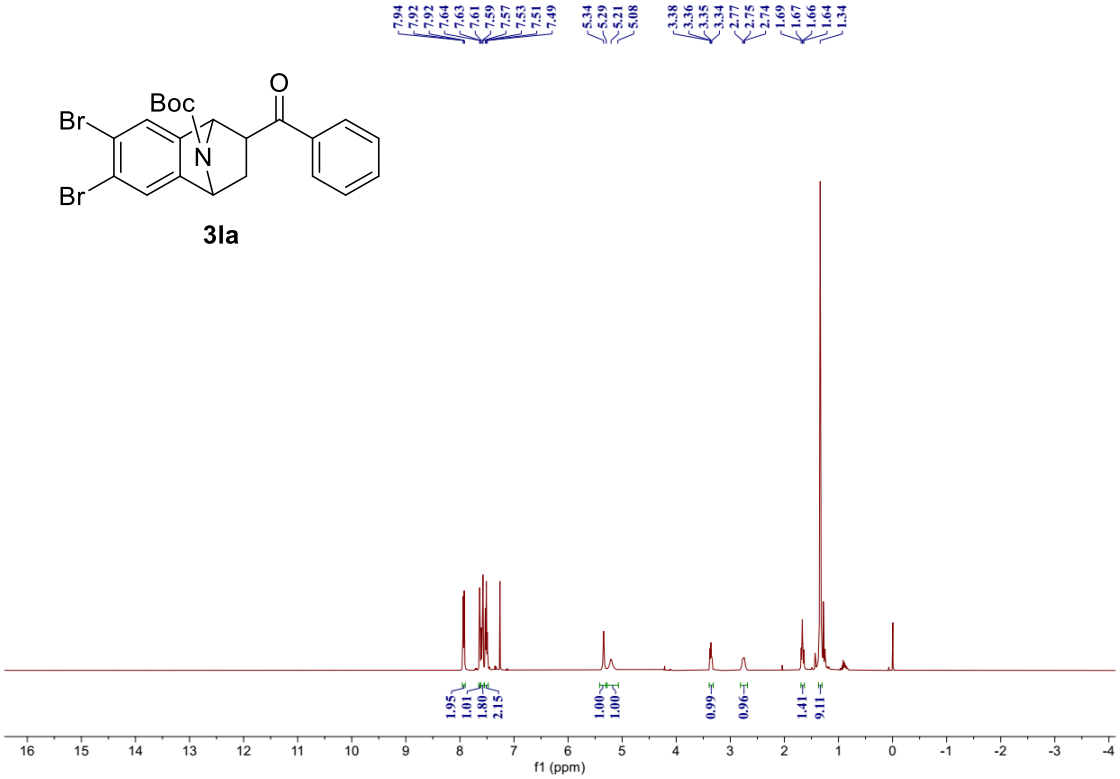
148.27
148.00
138.55
136.53
133.24
128.93
128.57
105.14
80.46
64.09
56.61
56.47
31.76
28.28
22.83
14.30
Boc
O
O
O
N
3ka
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.92
7.92
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7.61
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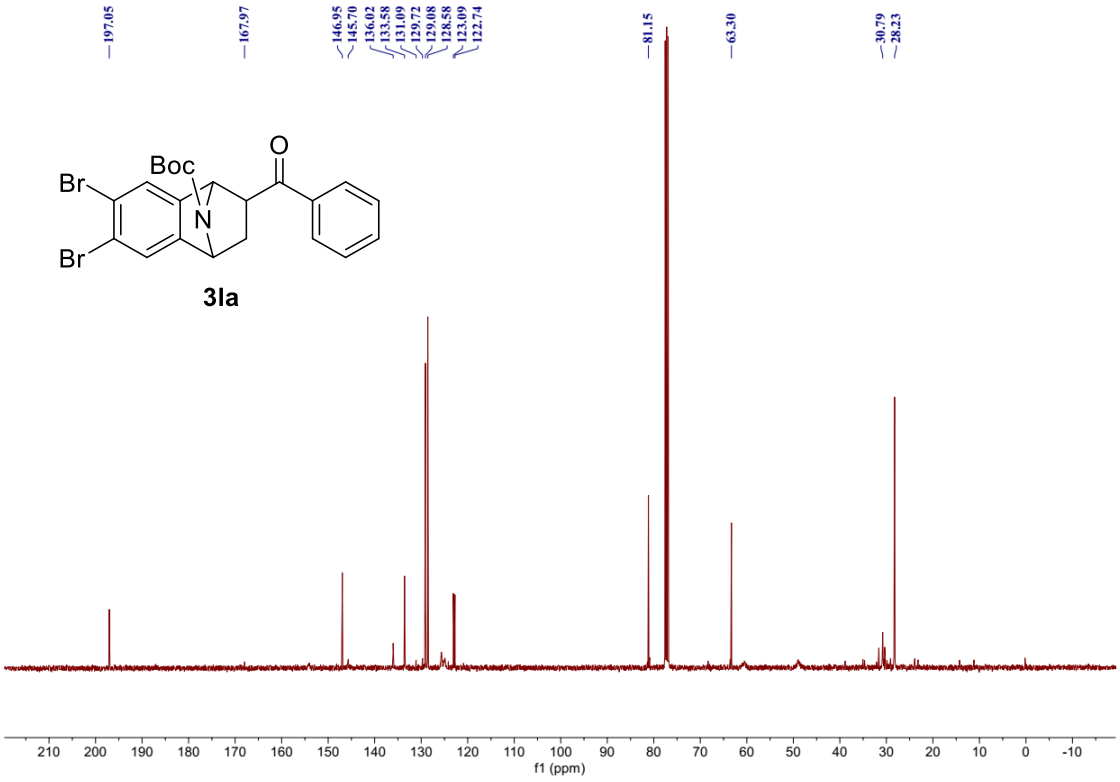
7.53
7.51
7.49
5.34
5.29
5.21
5.08
3.38
3.36
3.35
3.34
2.77
2.75
2.74
1.69
1.67
1.66
1.64
1.34
Boc
O
Br
N
Br
3la
1.95
1.01
1.80
2.15
1.00
1.00
0.99
0.96
1.41
9.11
f1 (ppm)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167.97
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145.70
136.02
133.58
131.09
129.72
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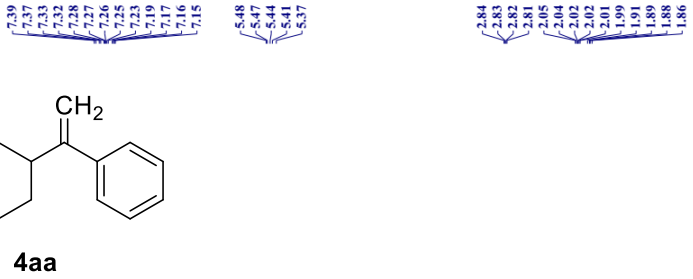
81.15
63.30
30.79
28.23
Boc
O
Br
N
Br
3la
f1 (ppm)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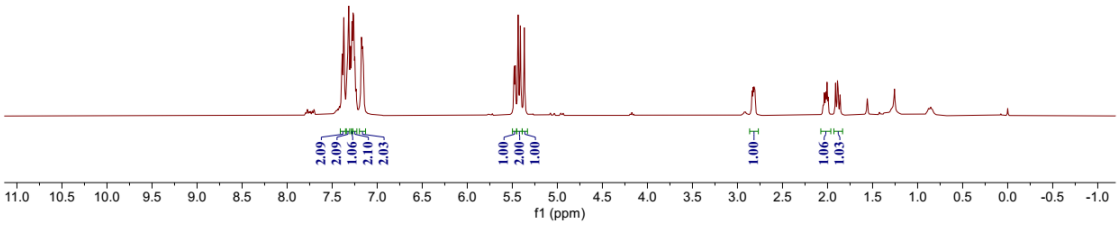
7.23
7.19
7.17
7.16
7.15
5.48
5.47
5.44
5.41
5.37
2.84
2.83
2.82
2.81
2.05
2.04
2.02
2.02
2.01
1.99
1.91
1.89
1.88
1.86
CH2
O

4aa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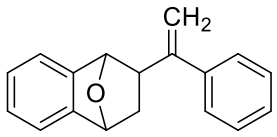
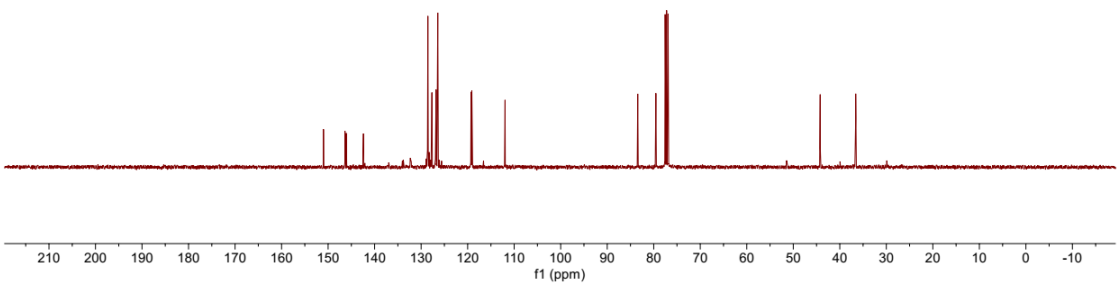
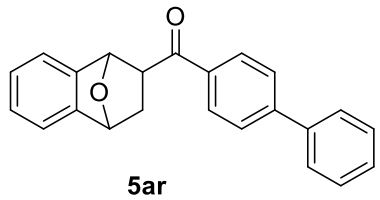
2.09
1.06
2.10
2.03
1.00
2.00
1.00
1.00
1.06
1.03
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


150.99
146.37
146.10
142.40
128.55
127.68
126.90
126.79
126.42
119.27
119.10
111.96
83.43
79.54
44.22
36.58

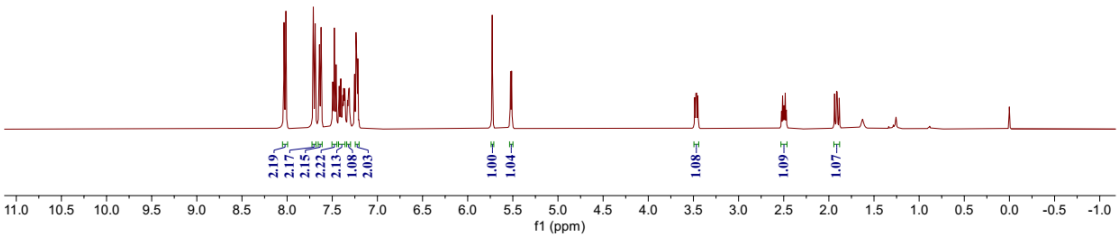

CH2
O

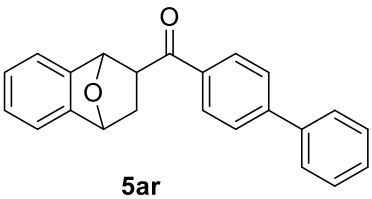
4aa


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)



5ar





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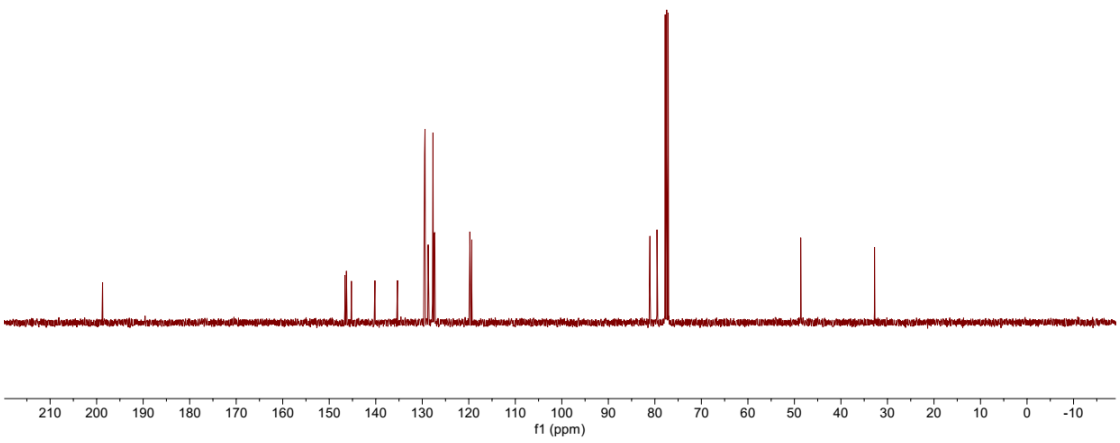

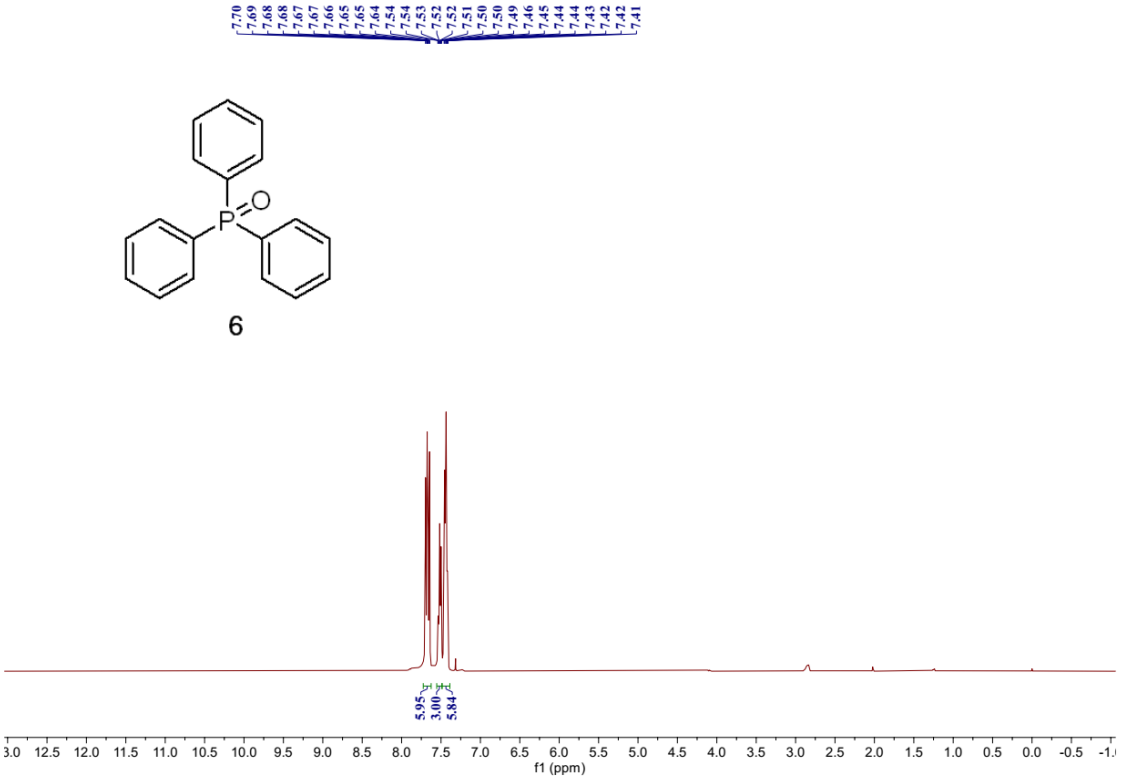
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7.53
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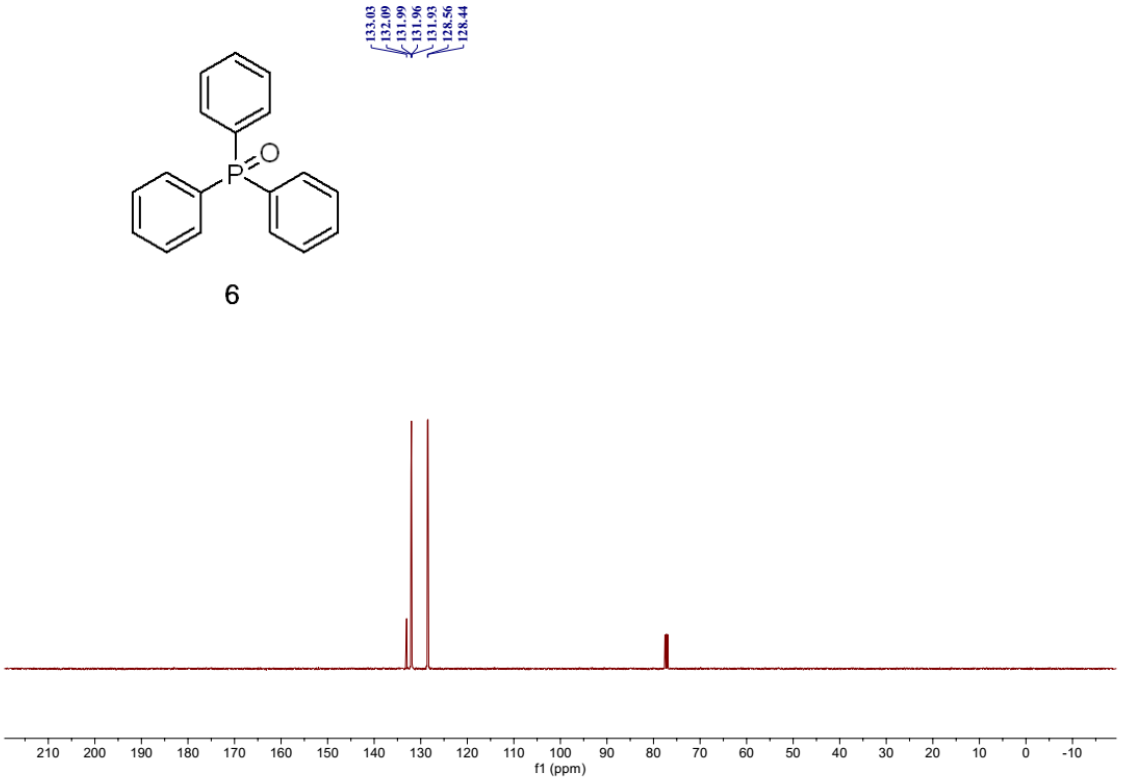
7.51
7.50
7.50
7.49
7.46
7.45
7.44
7.44
7.43
7.42
7.42
7.41
O
P
6
5.95
3.00
5.84
f1 (ppm)


133.03
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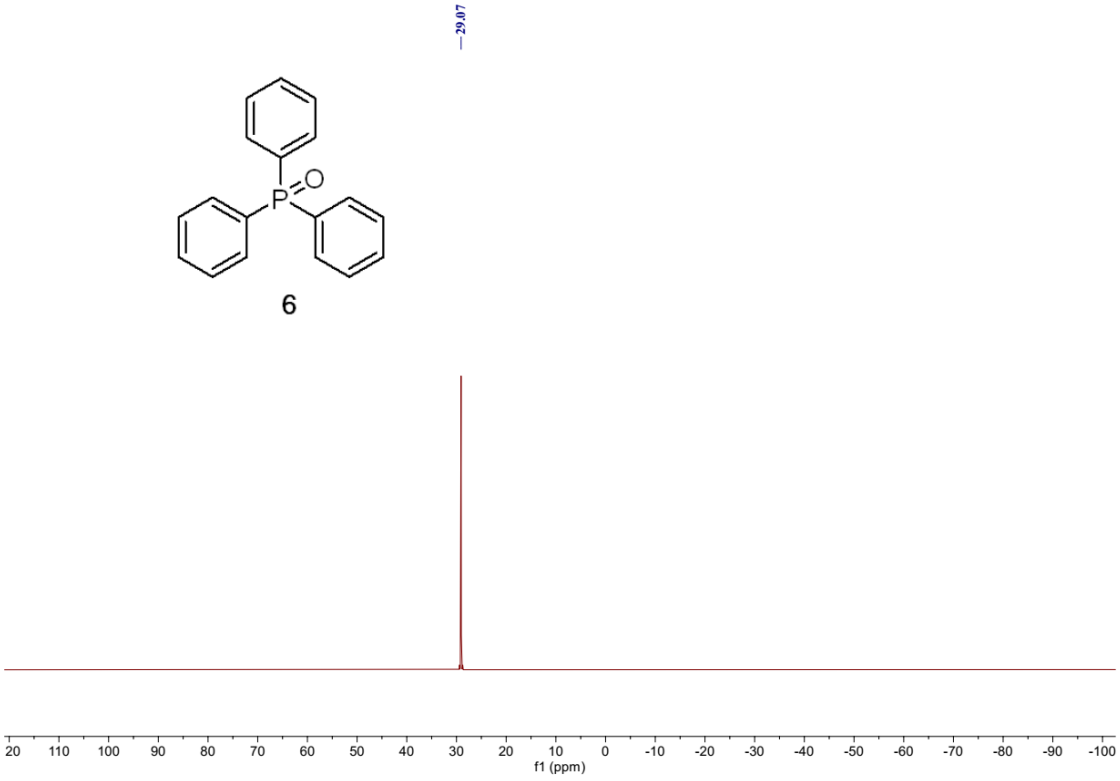
131.99
131.96
131.93
128.56
128.44
O
P
6
f1 (ppm)


29.07
P
O
6
20
110
100
90
80
70
60
50
40
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
f1 (ppm)