Supporting Information

Synthesis of oxa-bridged carbocycles via rhodium/Lewis acid catalyzed [3+3] and [4+3] cycloadditions of carbonyl ylides with donor-acceptor strained carbocycles

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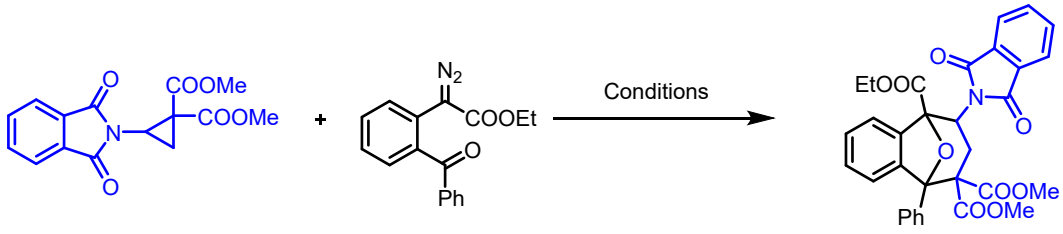
Table of contents

1. General information

Unless otherwise noted, all reactions were carried out under air. All reagents were from commercial sources and used as received without further purification. All solvents were dried by standard techniques and distilled prior to use. Column chromatography was performed on silica gel (200-300 meshes) using petroleum ether (bp. 60~90 °C) and ethyl acetate as eluent. Chemical shifts (δ, ppm) in the ^{1}H NMR spectra were recorded using TMS as internal standard or internally referenced to $CDCl_3$ (δ = 7.26 ppm), while the ^{13}C NMR spectra were internally $CDCl_3$ (δ = 77.16 ppm). All coupling constants (*J*) are reported in Hz. The following abbreviations were used to describe peak splitting patterns when appropriate: s = singlet, d = doublet, dd = double doublet, ddd = double doublet of doublets, t = triplet, dt = double triplet, q = quatriplet, m = multiplet, br = broad. HRMS all data were obtained using ESI-TOF (Electrospray ionization-time of flight). The X-ray diffraction patterns was recorded on a Bruker D8 Venture (Ga) Single Crystal XRD system. All melting points were measured with the samples after column chromatography and uncorrected.

2. Optimization of the reaction conditions

Table S1. Optimization of direct [3+3] cycloaddition conditions[a]

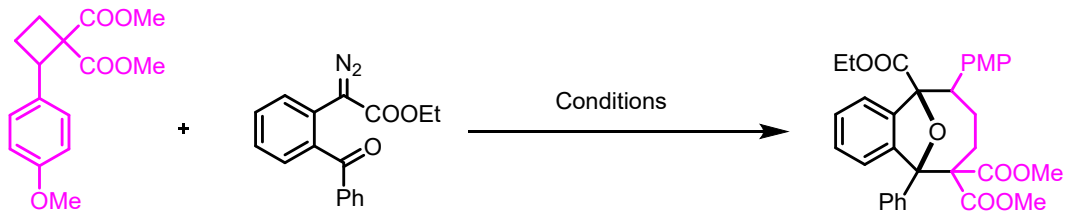


Entry	Catalyst	Lewis acid	Solvent	Yield (%)	d.r.
1[b]	$Rh_2(OAc)_4$	$Sc(OTf)_3$	Toluene	Trace	-
2	$Rh_2(OAc)_4$	$Sc(OTf)_3$	Toluene	66	>20:1
3	$Rh_2(OAc)_4$	$Yb(OTf)_3$	Toluene	6	8:1
4	$Rh_2(OAc)_4$	$Mg(OTf)_2$	Toluene	-	-
5	$Rh_2(OAc)_4$	$Sc(OTf)_3$, $Yb(OTf)_3$	Toluene	78	>20:1
6	$Rh_2(OAc)_4$	$Sc(OTf)_3$, $In(OTf)_3$	Toluene	84	11:1
7	$Rh_2(OAc)_4$	$Sc(OTf)_3$, $Mg(OTf)_2$	Toluene	62	10:1
8	$Rh_2(Oct)_4$	$Sc(OTf)_3$, $Yb(OTf)_3$	Toluene	62	>20:1
9	$Rh(esp)_2$	$Sc(OTf)_3$, $Yb(OTf)_3$	Toluene	72	>20:1
10	$Rh_2(OPiv)_4$	$Sc(OTf)_3$, $Yb(OTf)_3$	Toluene	65	>20:1
11	$Rh_2(OAc)_4$	$Sc(OTf)_3$, $Yb(OTf)_3$	$CHCl_3$	55	>20:1
12	$Rh_2(OAc)_4$	$Sc(OTf)_3$, $Yb(OTf)_3$	DCM	-	-
13	$Rh_2(OAc)_4$	$Sc(OTf)_3$, $Yb(OTf)_3$	THF	-	-
14	$Rh_2(OAc)_4$	$Sc(OTf)_3$, $Yb(OTf)_3$	EtOH	-	-
15	$Rh_2(OAc)_4$	$Sc(OTf)_3$, $Yb(OTf)_3$	Et_2O	-	-
16	$Rh_2(OAc)_4$	$Sc(OTf)_3$, $Yb(OTf)_3$	PhCl	88	>20:1
17	$Rh_2(OAc)_4$	$Sc(OTf)_3$, $Yb(OTf)_3$	*o*-xylene	74	>20:1
18[c]	$Rh_2(OAc)_4$	$Sc(OTf)_3$, $Yb(OTf)_3$	PhCl	Trace	-
19[d]	$Rh_2(OAc)_4$	$Sc(OTf)_3$, $Yb(OTf)_3$	PhCl	72	>20:1
20[e]	$Rh_2(OAc)_4$	$Sc(OTf)_3$, $Yb(OTf)_3$	PhCl	-	-

21[f]	$Rh_2(OAc)_4$	$Sc(OTf)_3$, $Yb(OTf)_3$	PhCl	33	>20:1

[a]Reaction conditions: **1a** (0.05 mmol), **2a** (0.075 mmol), $Sc(OTf)_3$ (10 mol%), $Yb(OTf)_3$ (2 mol%), 4 Å MS (60 mg) and solvent (1 mL) at room temperature for 4 h. The yields and dr values were determined by ^1H NMR analysis using 1,3,5-triisopropylbenzene as an internal standard. [b]No 4Å MS was added. [c]**1a** (0.05 mmol), **2a** (0.10 mmol). [d]**1a** (0.075 mmol), **2a** (0.05 mmol). [e]At 0 °C. [f]At 40 °C.

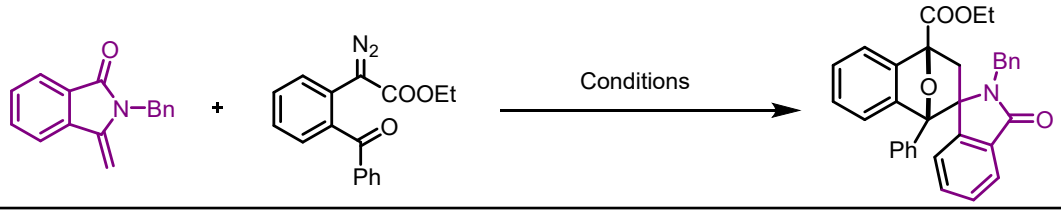
Table S2. Optimization of direct [4+3] cycloaddition conditions[a]



Entry	Catalyst	Solvent	Lewis acid	Yield (%)	d.r.
1	$Rh_2(OAc)_4$	Toluene	$Sc(OTf)_3$,$Yb(OTf)_3$	47	7:1
2	$Rh_2(Oct)_4$	Toluene	$Sc(OTf)_3$,$Yb(OTf)_3$	42	>20:1
3	$Rh(esp)_2$	Toluene	$Sc(OTf)_3$,$Yb(OTf)_3$	20	10:1
4	$Rh_2(OPiv)_4$	Toluene	$Sc(OTf)_3$,$Yb(OTf)_3$	53	8:1
5	$Rh_2(Oct)_4$	Toluene	$Sc(OTf)_3$	68	>20:1
6	$Rh_2(Oct)_4$	Toluene	$Yb(OTf)_2$	Trace	-
7	$Rh_2(Oct)_4$	Toluene	$Lu(OTf)_3$	20	3:1
8	$Rh_2(Oct)_4$	Toluene	$Cu(OTf)_2$	33	4:1
9	$Rh_2(Oct)_4$	Toluene	$Ga(OTf)_3$	Trace	-
10	$Rh_2(Oct)_4$	$CHCl_3$	$Sc(OTf)_3$	43	>20:1
11	$Rh_2(Oct)_4$	PhCl	$Sc(OTf)_3$	27	9:1
12	$Rh_2(Oct)_4$	$PhCF_3$	$Sc(OTf)_3$	24	5:1
13[b]	$Rh_2(Oct)_4$	Toluene	$Sc(OTf)_3$	66	>20:1
14[c]	$Rh_2(Oct)_4$	Toluene	$Sc(OTf)_3$	21	>20:1
15[d]	$Rh_2(Oct)_4$	Toluene	$Sc(OTf)_3$	72	>20:1
16[e]	$Rh_2(Oct)_4$	Toluene	$Sc(OTf)_3$	53	16:1

[a]Reaction conditions: **1a** (0.05 mmol), **2a** (0.075 mmol), $Sc(OTf)_3$ (10 mol%), $Yb(OTf)_3$ (2 mol%), 4 Å MS (60 mg) and solvent (1.0 mL) at room temperature for 12 h. The yields and dr values were determined by ^1H NMR analysis using 1,3,5-triisopropylbenzene as an internal standard. [b]**1a** (0.05 mmol), **2a** (0.10 mmol). [c]**1a** (0.075 mmol), **2a** (0.05 mmol). [d]At 30 °C. [e]At 40 °C.

Table S3. Optimization of direct [2+3] cycloaddition conditions[a]



Entry	Catalyst	Solvent	Lewis acid	Yield (%)	d.r.
1	$Rh_2(OAc)_4$	Toluene	$Sc(OTf)_3$	43	>20:1
2	$Rh_2(OAc)_4$	Toluene	$Yb(OTf)_3$	19	>20:1
3	$Rh_2(OAc)_4$	Toluene	$Cu(OTf)_2$	Trace	-
4	$Rh_2(OAc)_4$	Toluene	$Zn(OTf)_2$	-	-
5	$Rh_2(OAc)_4$	Toluene	$(PhO)_2POOH$	Trace	-
6	$Rh_2(oct)_4$	Toluene	$Sc(OTf)_3$	30	>20:1
7	$Rh_2(esp)_2$	Toluene	$Sc(OTf)_3$	21	>20:1
8	$Rh_2(opiv)_4$	Toluene	$Sc(OTf)_3$	33	>20:1
9	$Rh_2(OAc)_4$	PhCl	$Sc(OTf)_3$	31	>20:1
10	$Rh_2(OAc)_4$	DCM	$Sc(OTf)_3$	18	>20:1
11	$Rh_2(OAc)_4$	CF_3Ph	$Sc(OTf)_3$	27	>20:1
12[b]	$Rh_2(OAc)_4$	Toluene	$Sc(OTf)_3$	47	>20:1
13[c]	$Rh_2(OAc)_4$	Toluene	$Sc(OTf)_3$	54	>20:1
14[d]	$Rh_2(OAc)_4$	Toluene	$Sc(OTf)_3$	59	12:1
15[c,e]	$Rh_2(OAc)_4$	Toluene	$Sc(OTf)_3$	68	>20:1
16[c,f]	$Rh_2(OAc)_4$	Toluene	$Sc(OTf)_3$	59	>20:1
17[c,e,g]	$Rh_2(OAc)_4$	Toluene	$Sc(OTf)_3$	74	>20:1

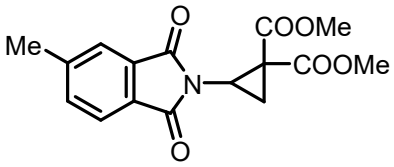
[a]Reaction conditions: **1a** (0.05 mmol), **2a** (0.075 mmol), $Sc(OTf)_3$ (10 mol%), 4 Å MS (60 mg) and solvent (1 mL) at room temperature for 12 h. The yields and dr values were determined by ^1H NMR analysis using 1,3,5-triisopropylbenzene as an internal standard. [b]With $Sc(OTf)_3$ (20 mol%). [c]With $Sc(OTf)_3$ (30 mol%). [d]With $Sc(OTf)_3$ (100 mol%). [e]**1a** (0.05 mmol), **2a** (0.10 mmol). [f]**1a** (0.05 mmol), **2a** (0.15 mmol). [g]24 h.

3. Synthesis of Substrates

The D–A cyclopropanes **1a-1l**,[1] **1m**,[2] α-diazoester **2a-o**,[3] D–A cyclobutanes **4a-e**,[4] were synthesized according to the published procedures.

S1 **S2** **1**

General Procedure A: Following a modified procedure,[1] a solution of **S2** (1.1 equiv.) in CH_2Cl_2 (4M) was added over 5 minutes at 0 °C to a solution of $Rh_2(esp)_2$ (0.2 mol%) and **S1** (1 equiv.) in CH_2Cl_2. The reaction mixture was stirred for 16 hours while warming to room temperature. Thereafter the solvent was evaporated and the residue was purified by column chromatography (silica, pentane: EtOAc 10:1 to pentane: EtOAc 3:1) affording pure product **1**.



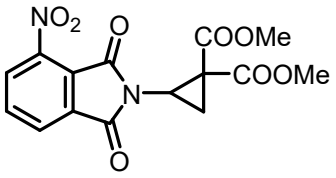
1b

Dimethyl 2-(5-methyl-1,3-dioxoisoindolin-2-yl)cyclopropane-1,1-dicarboxylate (1b) was prepared as a white foam from 5-methyl-2-vinylisoindoline-1,3-dione and dimethyl 2-diazomalonate according to the General Procedure A (eluent: PE/EA = 5:1) in 95% yield (602 mg).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.71 (d, *J* = 7.6 Hz, 1H), 7.63 (s, 1H), 7.50 (d, *J* = 7.6 Hz, 1H), 3.82 (s, 3H), 3.68 (dd, *J* = 8.4, 6.7 Hz, 1H), 3.60 (s, 3H), 2.70 (t, *J* = 76.5 Hz, 1H), 2.50 (s, 3H), 2.02 (dd, *J* = 8.5, 6.4 Hz, 1H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 168.7, 168.1, 168.0, 167.0, 145.8, 135.0, 131.9, 128.9, 124.1, 123.5, 53.1, 53.0, 35.0, 33.2, 22.1, 19.7.

HRMS (ESI+) Calcd for $C_{16}H_{16}NO_6$ $(M+H)^+$ requires m/z 318.0972, found m/z 318.0972.



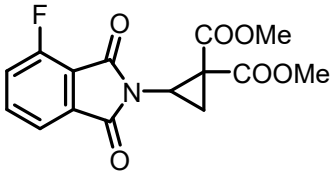
1f

Dimethyl 2-(4-nitro-1,3-dioxoisoindolin-2-yl)cyclopropane-1,1-dicarboxylate (1f) was prepared as a colorless oil from 4-nitro-2-vinylisoindoline-1,3-dione and dimethyl 2-diazomalonate according to the General Procedure A (eluent: PE/EA = 3:1) in 94% yield (326 mg).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.11 (dd, *J* = 7.6, 5.1 Hz, 2H), 7.92 (t, *J* = 7.8 Hz, 1H), 3.83 (s, 3H), 3.69 – 3.66 (m, 1H), 3.66 (s, 3H), 2.62 (t, *J* = 6.5 Hz, 1H), 2.07 (dd, *J* = 8.4, 6.6 Hz, 1H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 168.2, 167.2, 165.4, 162.7, 145.2, 135.8, 133.6, 129.0, 127.4, 123.2, 53.3, 53.2, 34.9, 33.0, 19.8.

HRMS (ESI+) Calcd for $C_{15}H_{13}N_2O_8$ $(M+H)^+$ requires m/z 349.0667, found m/z 349.0667.



1g

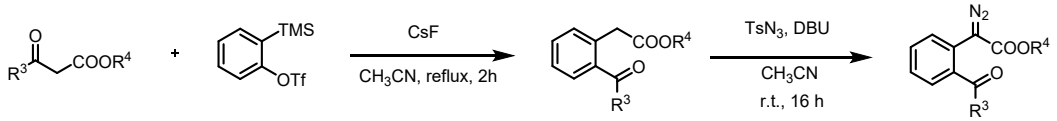
Dimethyl 2-(4-fluoro-1,3-dioxoisoindolin-2-yl)cyclopropane-1,1-dicarboxylate (1g) was prepared as a colorless oil from 4-fluoro-2-vinylisoindoline-1,3-dione and dimethyl 2-diazomalonate according to the General Procedure A (eluent: PE/EA = 3:1) in 94% yield (326 mg).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.72 (td, *J* = 7.8, 4.3 Hz, 1H), 7.65 (d, *J* = 7.3 Hz, 1H), 7.38 (t, *J* = 8.5 Hz, 1H), 3.82 (s, 3H), 3.67 (dd, *J* = 3.8 Hz, 1H), 3.64 (s, 3H), 2.65 (t, *J* = 6.5 Hz, 1H), 2.04 (dd, *J* = 8.3, 6.6 Hz, 1H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 168.3, 166.9, 166.6 (d, J_{C-F} = 2.9 Hz), 164.5, 158.9, 156.3, 137.0 (d, J_{C-F} = 7.7 Hz), 133.6, 122.7 (d, J_{C-F} = 19.6 Hz), 119.7 (d, J_{C-F} = 3.8 Hz), 117.2 (d, J_{C-F} = 12.4 Hz), 53.1, 53.0, 34.7, 32.9, 19.5.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -112.60.

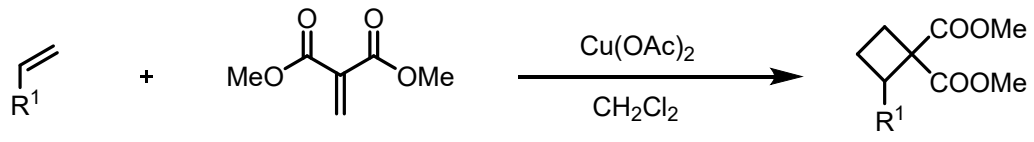
HRMS (ESI+) Calcd for $C_{15}H_{13}FNO_6$ $(M+H)^+$ requires m/z 322.0722, found m/z 322.0720.



S3 **S4** **2**

General Procedure B: Following a modified procedure,[3] to a solution of benzoylacetates **S3** (1 equiv.) and 2-(trimethylsilyl)phenyl triflate (1.3 equiv.) in CH_3CN (4M) under Ar was added CsF (2.5 equiv.). The reaction mixture was refluxed for 2 h. After completion of the reaction (monitored by TLC), the reaction mixture was cooled to RT and quenched by a aqueous solution of saturated NaCl. The aqueous layer was extracted twice with EtOAc. The combined organic layers were dried over Na_2SO_4 and conc. in vacuo to obtain crude product **S4**. The residue was purified over silica gel column chromatography using pentane: EtOAc as eluent.

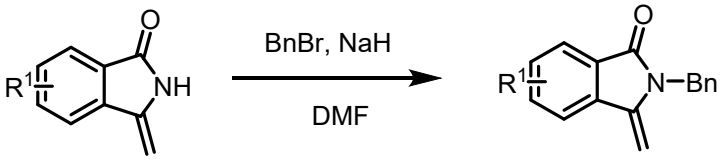
To a solution of acyl-alkyl derivative **S4** (1 equiv.) and TsN_3 (1.2 equiv.) in CH_3CN (3 M) under Ar at 0 °C was slowly added DBU (1.3 equiv.). The reaction mixture was then stirred for 16 h at RT. Upon completion of the reaction (monitored by TLC), the mixture was concentrated under reduced pressure to remove excess of CH_3CN. The residue was then purified by flash chromatography (using pentane: EtOAc as eluent) to afford the desired diazoesters **2** in high yields.



S5 **S6** **4**

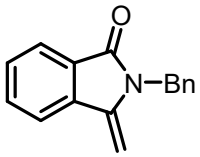
General Procedure C: Following a modified procedure,[4] A flame-dried schlenk

tube (50 mL) was evacuated and recharged with N_2 for 3 times. Under N_2, the tube (50 mL) was charged with 5 mol% $Cu(OAc)_2$, 200 mg of activated 4Å molecular sieves powder (MS), dry CH_2Cl_2 (3 mL) at room temperature. In another flame-dried schlenk tube (25 mL), vinylbenzene **S5** (10 mmol, 1.35 g) were dissolved in dry CH_2Cl_2 (6 mL) and cooled to 0 ºC for 10 min. Then a solution of dimethyl methylidenemalonate **S6** (unpurified product, 20 mmol, 2 equiv, 2.90 g. Mass calculation based on pure dimethyl methylidene malonate molecular mass) in dry CH_2Cl_2 (6 mL) was added to the solution of vinylbenzene **S5** via syringe at 0 ºC. This mixed solution was then added dropwise to the $Cu(OAc)_2$ solution via syringe in three portions (4 mL/portion) every 10 min. After the addition was complete, the reaction mixture was stirred at rt for 4 h until the reaction was complete (monitored by TLC). The reaction mixture was quenched with Et_3N (2-3 mL), and then passed over a short pad of silica gel with 50 mL of CH_2Cl_2. The solvent was removed under reduced pressure and the residue was purified by silica gel column chromatography (silica gel was pretreated with 1-2% Et_3N in pentane), eluting with (pentane: EtOAc = 10:1) to afford **4**.



S7 **6**

General Procedure D: Under the protection of argon gas at 0 °C, NaH (120 mg, 60% mineral oil, 3 mmol) was added to DMF (10 mL) solution **S7** (290 mg, 2 mmol, 1 equiv). After stirring for 30 min, Benzyl bromide (513 mg, 3 mmol, 1.5 equiv) was added dropwise, and the resulting mixture was kept stirring for another 2 h. The mixture was poured into ice water (30 mL) and EtOAc (10 mL) and extracted with EtOAc (2×10 mL). The combined organic layer was dried over anhydrous Na_2SO_4, concentrated and further purified by column chromatography (PE/EA = 10:1) to obtain the product.



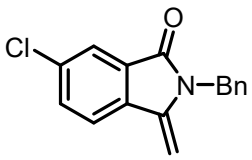
6a

2-Benzyl-3-methyleneisoindolin-1-one (6a) was prepared as a white foam from 3-methyleneisoindolin-1-one and benzyl bromide according to the General Procedure D (eluent: PE/EA = 10:1) in 62% yield (292 mg)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.88 (d, *J* = 7.5 Hz, 1H), 7.65 (d, *J* = 7.6 Hz, 1H), 7.57 (td, *J* = 7.5, 1.1 Hz, 1H), 7.50 (td, *J* = 7.4, 0.8 Hz, 1H), 7.31 – 7.21 (m, 5H), 5.13 (d, *J* = 2.4 Hz, 1H), 4.99 (s, 2H), 4.78 (d, *J* = 2.4 Hz, 1H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 167.4, 141.7, 137.0, 136.5, 132.2, 129.6, 129.3, 128.8, 127.5, 127.2, 123.5, 120.0, 90.1, 43.2.

HRMS (ESI+) Calcd for $C_{16}H_{14}NO$ $(M+H)^+$ requires m/z 231.1070, found m/z 231.1072.



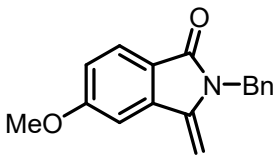
6b

2-Benzyl-6-chloro-3-methyleneisoindolin-1-one (6b) was prepared as a colorless oil from 6-chloro-3-methyleneisoindolin-1-one and benzyl bromide according to the General Procedure D (eluent: PE/EA = 10:1) in 64% yield (344 mg)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.85 (d, *J* = 1.6 Hz, 1H), 7.60 – 7.51 (m, 2H), 7.33 – 7.28 (m, 2H), 7.27 – 7.23 (m, 3H), 5.13 (d, *J* = 2.6 Hz, 1H), 4.99 (s, 2H), 4.83 (d, *J* = 2.6 Hz, 1H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 166.0, 140.9, 136.6, 135.9, 134.7, 132.4, 130.9, 128.8, 127.6, 127.2, 123.6, 121.4, 91.0, 43.4.

HRMS (ESI+) Calcd for $C_{16}H_{13}ClNO$ $(M+H)^+$ requires m/z 270.0680 (Cl, 34.9686), found m/z 270.0674.



6c

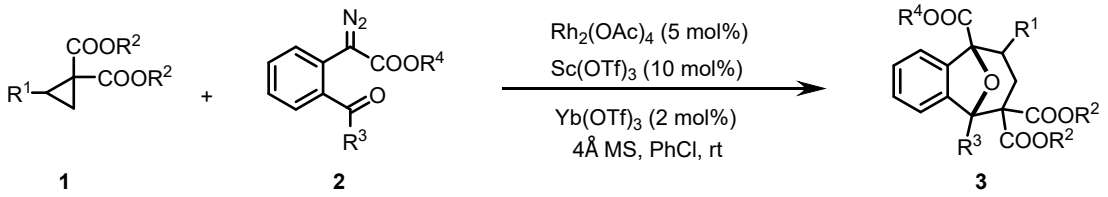
2-Benzyl-5-methoxy-3-methyleneisoindolin-1-one (6c) was prepared as a white foam from 5-methoxy-3-methyleneisoindolin-1-one and benzyl bromide according to the General Procedure D (eluent: PE/EA = 10:1) in 53% yield (282 mg)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.55 (d, *J* = 8.4 Hz, 1H), 7.35 (d, *J* = 2.3 Hz, 1H), 7.32 – 7.22 (m, 5H), 7.12 (dd, *J* = 8.4, 2.4 Hz, 1H), 5.02 (d, *J* = 2.3 Hz, 1H), 4.99 (s, 2H), 4.71 (d, *J* = 2.3 Hz, 1H), 3.88 (s, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 167.3, 161.3, 141.5, 137.1, 131.0, 129.3, 128.8, 127.5, 127.2, 121.3, 120.4, 106.2, 89.0, 55.9, 43.3.

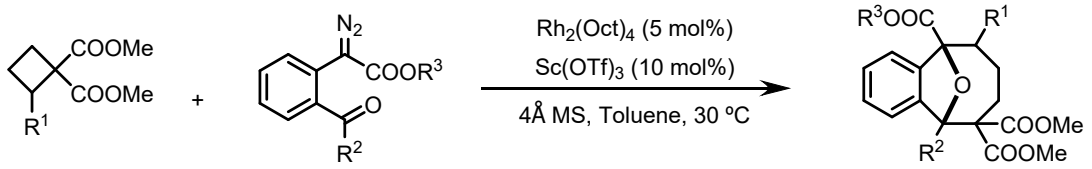
HRMS (ESI+) Calcd for $C_{17}H_{16}NO_2$ $(M+H)^+$ requires m/z 266.1176, found m/z 266.1181.

4. Catalytic Synthesis of compounds 3, 5 and 7



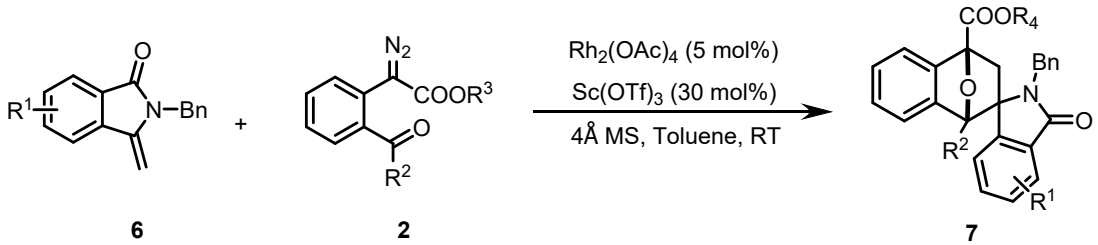
1 2 3

General Procedure E: A solution of **1** (0.1 mmol), $Sc(OTf)_3$ (4.9 mg, 0.01 mmol) , $Yb(OTf)_3$ (1.2 mg, 0.002 mmol), 4Å MS (60 mg) and $Rh_2(OAc)_4$ (2.2 mg, 0.005 mmol) were dissolved in PhCl (1 mL). A solution of diazo compound **2** (0.15 mmol) in PhCl (1 mL) was then added to the reaction mixture for a period of 1 h by using syringe pump and stirred at the indicated temperature in a closed vessel for 3 h (monitored by TLC). The product **3** was purified over silica gel by flash column chromatography using pentane: EtOAc mixtures as eluent.



4 2 5

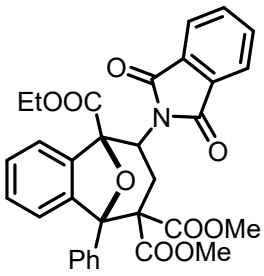
General Procedure F: At 30°C, a solution of **4** (0.1 mmol), $Sc(OTf)_3$ (4.9 mg, 0.01 mmol), 4Å MS (60 mg) and $Rh_2(Oct)_4$ (3.9 mg, 0.005 mmol) were dissolved in toluene (1 mL). A solution of diazo compound **2** (0.15 mmol) in toluene (1 mL) was then added to the reaction mixture for a period of 1 h by using syringe pump and stirred at the indicated temperature in a closed vessel for 12 h (monitored by TLC). The product **5** was purified over silica gel by flash column chromatography using pentane: EtOAc mixtures as eluent.



6 2 7

General Procedure G: A solution of **6** (0.1 mmol), $Sc(OTf)_3$ (14.8 mg, 0.03 mmol) ,

4Å MS (60 mg) and $Rh_2(OAc)_4$ (2.2 mg, 0.005 mmol) were dissolved in toluene (1 mL). A solution of diazo compound **2** (0.2 mmol) in toluene (1 mL) was then added to the reaction mixture for a period of 1 h by using syringe pump and stirred at the indicated temperature in a closed vessel for 24 h (monitored by TLC). The product **7** was purified over silica gel by flash column chromatography using pentane: EtOAc mixtures as eluent.



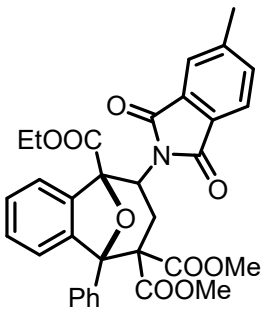
3a

5-Ethyl 8,8-dimethyl 6-(1,3-dioxoisoindolin-2-yl)-9-phenyl-6,7-dihydro-5*H*-5,9-epoxybenzo[7]annulene-5,8,8(9*H*)-tricarboxylate (3a) was prepared as a white foam from dimethyl 2-(1,3-dioxoisoindolin-2-yl)cyclopropane-1,1-dicarboxylate **1a** (30.3 mg, 0.1 mmol) and ethyl 2-(2-benzoylphenyl)-2-diazoacetate **2a** (44.1 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 5:1) in 84% yield (47.9 mg, >20:1 dr).

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.85 – 7.80 (m, 4H), 7.72 – 7.68 (m, 2H), 7.48 – 7.45 (m, 1H), 7.38 (t, *J* = 7.8 Hz, 2H), 7.35 – 7.31 (m, 2H), 7.31 – 7.27 (m, 2H), 4.73 (dd, *J* = 6.1, 2.3 Hz, 1H), 4.29 (q, *J* = 7.1 Hz, 2H), 3.68 (s, 3H), 3.19 (s, 3H), 3.02 (dd, *J* = 15.1, 2.3 Hz, 1H), 2.09 (dd, *J* = 15.1, 6.2 Hz, 1H), 1.27 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (150 MHz, Chloroform-*d*) δ 170.5, 169.1, 168.6, 143.0, 141.9, 141.2, 134.0, 128.9, 128.9, 128.1, 127.3, 125.8, 125.4, 123.2, 119.5, 89.7, 86.1, 62.0, 60.9, 52.9, 52.4, 48.7, 33.3, 14.2.

HRMS (ESI+) Calcd for $C_{33}H_{27}NO_9Na$ $(M+Na)^+$ requires m/z 592.1578, found m/z 592.1586.



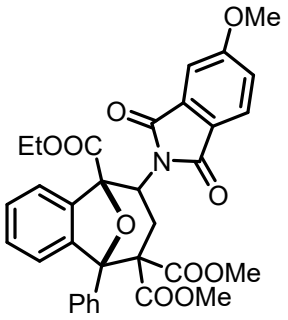
3b

5-Ethyl 8,8-dimethyl 6-(5-methyl-1,3-dioxoisoindolin-2-yl)-9-phenyl- 6,7-dihydro-5*H*-5,9-epoxybenzo[7]annulene-5,8,8(9*H*)-tricarboxylate (3b) was prepared as a white foam from dimethyl 2-(5-methyl-1,3-dioxoisoindolin-2-yl) cyclopropane-1,1-dicarboxylate **1b** (31.7 mg, 0.1 mmol) and ethyl 2-(2-benzoylphenyl)-2-diazoacetate **2a** (44.1 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 5:1) in 67% yield (39.2 mg, >20:1 dr).

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.82 (d, *J* = 7.4 Hz, 2H), 7.70 (d, *J* = 7.6 Hz, 1H), 7.62 (s, 1H), 7.48 (d, *J* = 7.6 Hz, 1H), 7.45 (dd, *J* = 5.8, 2.8 Hz, 1H), 7.38 (t, *J* = 7.8 Hz, 2H), 7.33 – 7.30 (m, 2H), 7.30 – 7.27 (m, 2H), 4.71 (dd, *J* = 6.1, 2.5 Hz, 1H), 4.28 (q, *J* = 7.1 Hz, 2H), 3.68 (s, 3H), 3.19 (s, 3H), 3.03 (dd, *J* = 15.1, 2.5 Hz, 1H), 2.49 (s, 3H), 2.09 (dd, *J* = 15.1, 6.2 Hz, 1H), 1.26 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (150 MHz, Chloroform-*d*) δ 170.5, 169.1, 168.5, 145.2, 143.0, 142.0, 141.2, 134.6, 128.9, 128.8, 128.1, 127.3, 125.7, 125.4, 123.7, 123.1, 119.5, 89.6, 86.1, 61.9, 60.9, 52.8, 52.4, 48.6, 33.3, 22.1, 14.2.

HRMS (ESI+) Calcd for $C_{33}H_{30}NO_9$ $(M+H)^+$ requires m/z 584.1915, found m/z 584.1910.



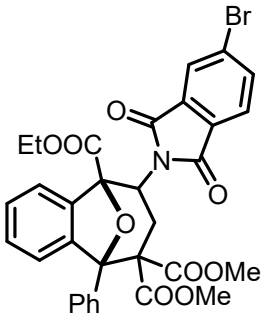
3c

5-Ethyl 8,8-dimethyl 6-(5-methoxy-1,3-dioxoisoindolin-2-yl)-9-phenyl-6,7-dihydro-5*H*-5,9-epoxybenzo[7]annulene-5,8,8(9*H*)-tricarboxylate (3c) was prepared as a white foam from dimethyl 2-(5-methoxy-1,3-dioxoisoindolin-2-yl) cyclopropane-1,1-dicarboxylate **1c** (33.3 mg, 0.1 mmol) and ethyl 2-(2-benzoylphenyl)-2-diazoacetate **2a** (44.1 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 5:1) in 52% yield (31.2 mg, >20:1 dr).

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.82 (d, *J* = 7.7 Hz, 2H), 7.73 (d, *J* = 8.2 Hz, 1H), 7.47 – 7.43 (m, 1H), 7.38 (t, *J* = 7.7 Hz, 2H), 7.34 – 7.30 (m, 3H), 7.30 – 7.27 (m, 2H), 7.15 (dd, *J* = 8.3, 2.1 Hz, 1H), 4.70 (dd, *J* = 6.0, 2.3 Hz, 1H), 4.29 (q, *J* = 7.1 Hz, 2H), 3.91 (s, 3H), 3.68 (s, 3H), 3.20 (s, 3H), 3.03 (dd, *J* = 15.1, 2.3 Hz, 1H), 2.08 (dd, *J* = 15.1, 6.1 Hz, 1H), 1.26 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (150 MHz, Chloroform-*d*) δ 170.5, 169.1, 168.6, 164.8, 143.0, 142.0, 141.2, 128.9, 128.8, 128.1, 127.3, 125.7, 125.4, 124.9, 112.0, 119.6, 107.9, 89.6, 86.1, 61.9, 60.9, 56.2, 52.9, 52.4, 48.6, 33.3, 14.2.

HRMS (ESI+) Calcd for $C_{33}H_{30}NO_{10}$ $(M+H)^+$ requires m/z 600.1864, found m/z 600.1862.



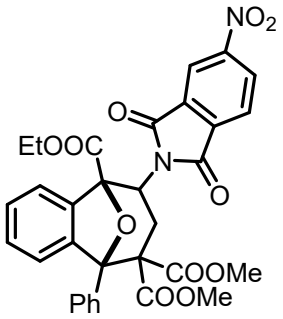
3d

5-Ethyl 8,8-dimethyl 6-(5-bromo-1,3-dioxoisoindolin-2-yl)-9-phenyl-6,7-dihydro-5*H*-5,9-epoxybenzo[7]annulene-5,8,8(9*H*)-tricarboxylate (3d) was prepared as a white foam from dimethyl 2-(5-bromo-1,3-dioxoisoindolin-2-yl)cyclopropane-1,1-dicarboxylatee **1d** (38.0 mg, 0.1 mmol) and ethyl 2-(2-benzoylphenyl)-2-diazoacetate **2a** (44.1 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 5:1) in 87% yield (56.2 mg, 8:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.96 (dd, *J* = 4.8, 1.7 Hz, 1H), 7.85 – 7.79 (m, 3H), 7.69 (d, *J* = 7.9 Hz, 1H), 7.51 – 7.47 (m, 1H), 7.38 (t, *J* = 7.8 Hz, 2H), 7.35 – 7.26 (m, 4H), 4.70 (dd, *J* = 6.2, 2.1 Hz, 1H), 4.29 (q, *J* = 7.1 Hz, 2H), 3.69 (s, 3H), 3.20 (s, 3H), 2.97 (dd, *J* = 15.3, 2.2 Hz, 1H), 2.05 (dd, *J* = 15.2, 6.2 Hz, 1H), 1.27 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 170.4, 169.1, 168.5, 143.0, 141.7, 141.1, 137.0, 129.0, 128.93, 128.88, 128.1, 127.3, 126.5, 125.8, 125.4, 124.6, 119.5, 89.7, 85.9, 62.0, 60.9, 52.9, 52.4, 48.9, 33.2, 14.2.

HRMS (ESI+) Calcd for $C_{32}H_{27}BrNO_9$ $(M+H)^+$ requires m/z 648.0864 (Br, 78.9183), found m/z 648.0874.



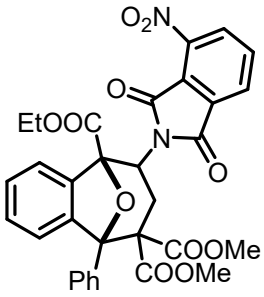
3e

5-Ethyl 8,8-dimethyl 6-(5-nitro-1,3-dioxoisoindolin-2-yl)-9-phenyl-6,7-dihydro -5*H*-5,9-epoxybenzo[7]annulene-5,8,8(9*H*)-tricarboxylate (3e) was prepared as a white foam from dimethyl 2-(5-nitro-1,3-dioxoisoindolin-2-yl)cyclopropane -1,1-dicarboxylate **1e** (34.8 mg, 0.1 mmol) and ethyl 2-(2-benzoylphenyl) -2-diazoacetate **2a** (44.1 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 5:1) in 88% yield (54 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.64 (d, *J* = 1.7 Hz, 1H), 8.58 (dd, *J* = 8.1, 2.0 Hz, 1H), 8.02 (d, *J* = 8.1 Hz, 1H), 7.81 (d, *J* = 7.3 Hz, 2H), 7.56 – 7.51 (m, 1H), 7.42 – 7.34 (m, 4H), 7.33 – 7.26 (m, 2H), 4.73 (dd, *J* = 6.2, 1.8 Hz, 1H), 4.31 (q, *J* = 7.1 Hz, 2H), 3.69 (s, 3H), 3.19 (s, 3H), 2.95 (dd, *J* = 15.4, 1.8 Hz, 1H), 2.04 (dd, *J* = 15.3, 6.2 Hz, 1H), 1.29 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 170.3, 169.3, 168.6, 151.8, 142.8, 141.4, 140.9, 129.3, 129.1, 129.1, 128.1, 127.4, 125.9, 125.4, 124.4, 119.4, 118.6, 89.7, 85.7, 77.4, 62.2, 60.8, 53.0, 52.4, 49.3, 33.1, 14.2.

HRMS (ESI+) Calcd for $C_{32}H_{30}N_3O_{11}$ $(M+NH_4)^+$ requires m/z 632.1875, found m/z 632.1869.



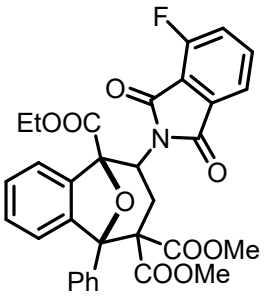
3f

5-Ethyl 8,8-dimethyl 6-(4-nitro-1,3-dioxoisoindolin-2-yl)-9-phenyl-6,7-dihydro -5*H*-5,9-epoxybenzo[7]annulene-5,8,8(9*H*)-tricarboxylate (3f) was prepared as a white foam from dimethyl 2-(4-nitro-1,3-dioxoisoindolin-2-yl)cyclopropane -1,1-dicarboxylate **1f** (34.8 mg, 0.1 mmol) and ethyl 2-(2-benzoylphenyl) -2-diazoacetate **2a** (44.1 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 5:1) in 97% yield (60.0 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.07 (t, *J* = 7.9 Hz, 2H), 7.88 (t, *J* = 7.8 Hz, 1H), 7.80 (d, *J* = 7.9 Hz, 2H), 7.54 – 7.46 (m, 1H), 7.39 (t, *J* = 7.7 Hz, 2H), 7.36 – 7.32 (m, 2H), 7.30 (d, *J* = 7.4 Hz, 1H), 7.26 – 7.24 (m, 1H), 4.72 (dd, 1H), 4.30 (q, *J* = 7.1 Hz, 2H), 3.69 (s, 3H), 3.25 (s, 3H), 2.96 (dd, *J* = 15.3, 1H), 2.05 (dd, *J* = 15.3, 6.2 Hz, 1H), 1.28 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (150 MHz, Chloroform-*d*) δ 170.4, 169.4, 168.6, 145.2, 143.0, 141.6, 141.1, 135.3, 129.1, 128.4, 128.1, 127.4, 126.9, 125.9, 125.4, 119.4, 89.8, 85.9, 62.2, 60.9, 53.0, 52.5, 49.3, 33.2, 14.2.

HRMS (ESI+) Calcd for $C_{32}H_{30}N_3O_{11}$ $(M+NH_4)^+$ requires m/z 632.1875, found m/z 632.1872.



3g

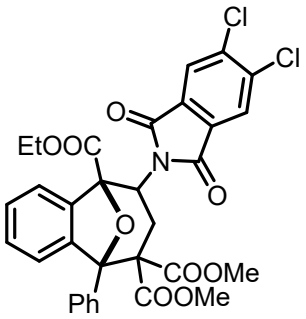
5-Ethyl 8,8-dimethyl 6-(4-fluoro-1,3-dioxoisoindolin-2-yl)-9-phenyl-6,7-dihydro-5*H*-5,9-epoxybenzo[7]annulene-5,8,8(9*H*)-tricarboxylate (3g) was prepared as a white foam from dimethyl 2-(4-fluoro-1,3-dioxoisoindolin-2-yl)cyclopropane-1,1-dicarboxylate **1g** (32.1 mg, 0.1 mmol) and ethyl 2-(2-benzoylphenyl)-2-diazoacetate **2a** (44.1 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 5:1) in 96% yield (56.6 mg, >20:1 dr).

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.82 (d, *J* = 7.6 Hz, 2H), 7.69 (td, *J* = 7.8, 4.1 Hz, 1H), 7.64 (d, *J* = 7.2 Hz, 1H), 7.52 – 7.48 (m, 1H), 7.40 – 7.32 (m, 5H), 7.31 – 7.26 (m, 2H), 4.70 (dd, *J* = 6.1, 1.9 Hz, 1H), 4.30 (q, *J* = 7.1 Hz, 2H), 3.69 (s, 3H), 3.22 (s, 3H), 2.98 (dd, *J* = 15.2, 2.0 Hz, 1H), 2.06 (dd, *J* = 15.2, 6.2 Hz, 1H), 1.28 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (150 MHz, Chloroform-*d*) δ 170.4, 169.2, 168.6, 158.5, 156.8, 143.0, 141.8, 141.1, 136.5 (d, $J_{C\text{-}F}$ = 7.2 Hz), 129.0 (d, $J_{C\text{-}F}$ = 7.3 Hz), 128.1, 127.3, 125.8, 125.4, 122.4, 122.3, 119.5, 119.4 (d, $J_{C\text{-}F}$ = 2.5 Hz), 89.7, 85.9, 62.0, 60.9, 52.9, 52.4, 48.8, 33.2, 14.2.

^{19}F NMR (565 MHz, Chloroform-*d*) δ -113.61.

HRMS (ESI+) Calcd for $C_{32}H_{27}FNO_9$ $(M+H)^+$ requires m/z 588.1664, found m/z 588.1667.



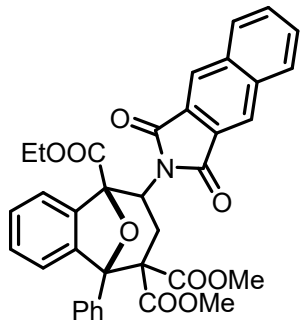
3h

5-Ethyl 8,8-dimethyl 6-(5,6-dichloro-1,3-dioxoisoindolin-2-yl)-9-phenyl-6,7-dihydro-5*H*-5,9-epoxybenzo[7]annulene-5,8,8(9*H*)-tricarboxylate (3h) was prepared as a white foam from dimethyl 2-(5,6-dichloro-1,3-dioxoisoindolin-2-yl) cyclopropane-1,1-dicarboxylate **1h** (37.1 mg, 0.1 mmol) and ethyl 2-(2-benzoylphenyl)-2-diazoacetate **2a** (44.1 mg, 0.15 mmol) according to the General Procedure E(eluent: PE/EA = 3:1) in 72% yield (46 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.90 (s, 2H), 7.81 (d, *J* = 1.5 Hz, 2H), 7.52 – 7.48 (m, 1H), 7.41 – 7.32 (m, 4H), 7.31 – 7.28 (m, 1H), 7.26 – 7.23 (m, 1H), 4.68 (dd, *J* = 6.2, 2.0 Hz, 1H), 4.29 (q, *J* = 7.1 Hz, 2H), 3.69 (s, 3H), 3.21 (s, 3H), 2.94 (dd, *J* = 15.3, 2.0 Hz, 1H), 2.03 (dd, *J* = 15.2, 6.2 Hz, 1H), 1.28 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 170.4, 169.2, 168.5, 142.9, 141.6, 141.0, 138.9, 129.0, 128.1, 127.4, 125.9, 125.8, 125.4, 125.3, 119.5, 89.7, 85.8, 62.1, 60.8, 52.9, 52.5, 49.1, 33.1, 14.2.

HRMS (ESI+) Calcd for $C_{32}H_{29}Cl_2N_2O_9$ $(M+NH_4)^+$ requires m/z 655.1245 (Cl, 34.9686), found m/z 655.1238.



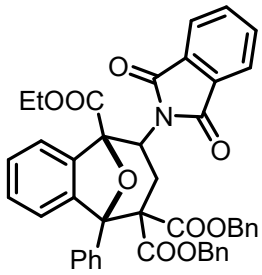
3i

5-Ethyl 8,8-dimethyl6-(1,3-dioxo-1,3-dihydro-2H-benzo[f]isoindol-2-yl)-9-phenyl -6,7-dihydro-5*H*-5,9-epoxybenzo[7]annulene-5,8,8(9*H*)-tricarboxylate (3i) was prepared as a white foam from dimethyl 2-(1,3-dioxo-1,3-dihydro-2H-benzo[f] isoindol-2-yl)cyclopropane-1,1-dicarboxylate **1i** (35.3 mg, 0.1 mmol) and ethyl 2-(2-benzoylphenyl)-2-diazoacetate **2a** (44.1 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 2:1) in 71% yield (44.1 mg, 17:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.32 (s, 2H), 8.05 (dd, *J* = 6.2, 3.3 Hz, 2H), 7.85 (d, *J* = 7.4 Hz, 2H), 7.68 (dd, *J* = 6.2, 3.2 Hz, 2H), 7.47 (dd, *J* = 5.6, 2.9 Hz, 1H), 7.40 (t, *J* = 7.7 Hz, 2H), 7.36 – 7.28 (m, 4H), 4.81 (dd, *J* = 6.2, 2.5 Hz, 1H), 4.31 (q, *J* = 7.1 Hz, 2H), 3.68 (s, 3H), 3.18 (s, 3H), 3.10 (dd, *J* = 15.1, 2.5 Hz, 1H), 2.13 (dd, *J* = 15.1, 6.2 Hz, 1H), 1.27 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 170.5, 169.1, 168.5, 143.0, 142.0, 141.2, 135.7, 130.4, 129.1, 128.9, 128.8, 128.1, 128.0, 127.3, 125.7, 125.4, 124.6, 119.6, 89.7, 86.1, 62.0, 60.9, 52.9, 52.4, 48.9, 33.2, 14.2.

HRMS (ESI+) Calcd for $C_{36}H_{30}NO_9$ $(M+H)^+$ requires m/z 620.1915, found m/z 620.1915.



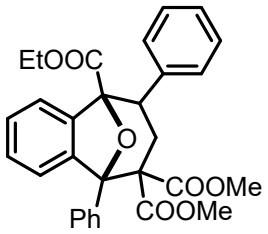
3j

8,8-Dibenzyl 5-ethyl 6-(1,3-dioxoisoindolin-2-yl)-9-phenyl-6,7-dihydro-5*H*-5,9 -epoxybenzo[7]annulene-5,8,8(9*H*)-tricarboxylate (3j) was prepared as a white foam from dibenzyl 2-(1,3-dioxoisoindolin-2-yl)cyclopropane-1,1-dicarboxylate **1j** (45.5 mg, 0.1 mmol) and ethyl 2-(2-benzoylphenyl)-2-diazoacetate **2a** (44.1 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 5:1) in 71% yield (54 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.82 – 7.77 (m, 2H), 7.72 – 7.66 (m, 4H), 7.36 (d, *J* = 7.5 Hz, 1H), 7.34 – 7.27 (m, 4H), 7.25 – 7.21 (m, 2H), 7.19 – 7.11 (m, 6H), 7.07 (t, *J* = 7.4 Hz, 2H), 6.61 (d, *J* = 7.1 Hz, 2H), 5.15 (d, *J* = 12.2 Hz, 1H), 5.00 (d, *J* = 12.1 Hz, 1H), 4.75 – 4.71 (m, 1H), 4.68 (d, *J* = 3.2 Hz, 2H), 4.30 (q, *J* = 7.1 Hz, 2H), 3.04 (dd, *J* = 15.3, 1.8 Hz, 1H), 2.11 (dd, *J* = 15.3, 6.3 Hz, 1H), 1.28 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 170.0, 168.6, 168.4, 143.1, 141.7, 141.1, 134.9, 134.6, 133.9, 128.8, 128.7, 128.6, 128.5, 128.1, 127.8, 127.7, 127.2, 126.0, 125.4, 123.2, 119.4, 89.7, 86.1, 67.7, 67.3, 61.9, 60.8, 48.6, 33.6, 29.8, 14.2.

HRMS (ESI+) Calcd for $C_{44}H_{36}NO_9$ (M+H)+ requires m/z 722.2385, found m/z 722.2394.



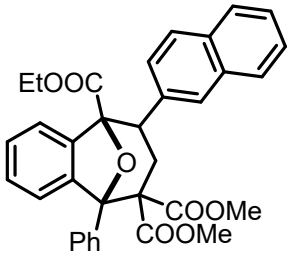
3k

6-Ethyl 8,8-dimethyl 6,9-diphenyl-6,7-dihydro-5*H*-5,9-epoxybenzo[7]annulene -5,8,8(9*H*)-tricarboxylate (3k) was prepared as a white foam from dimethyl 2-phenylcyclopropane-1,1-dicarboxylate **1k** (23.4 mg, 0.1 mmol) and ethyl 2-(2-benzoylphenyl)-2-diazoacetate **2a** (44.1 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 10:1) in 94% yield (47 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.66 (d, *J* = 8.0 Hz, 2H), 7.50 (d, *J* = 7.7 Hz, 2H), 7.44 – 7.37 (m, 3H), 7.34 – 7.28 (m, 4H), 7.24 (s, 2H), 7.15 (t, *J* = 7.4 Hz, 1H), 4.27 – 4.17 (m, 1H), 4.17 – 4.09 (m, 1H), 3.72 (s, 3H), 3.48 (d, *J* = 6.7 Hz, 1H), 2.91 (s, 3H), 2.61 (d, *J* = 14.4 Hz, 1H), 2.37 – 2.31 (m, 1H), 1.11 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (150 MHz, Chloroform-*d*) δ 170.7, 169.6, 168.5, 142.8, 142.2, 141.2, 128.6, 128.5, 128.3, 128.2, 128.0, 127.2, 126.5, 125.10, 125.07, 119.7, 89.5, 88.5, 61.5, 58.5, 52.8, 51.5, 42.9, 36.0, 14.1.

HRMS (ESI+) Calcd for $C_{30}H_{29}O_7$ (M+H)+ requires m/z 501.1908, found m/z 501.1903.



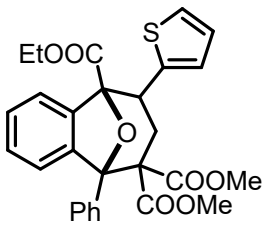
3l

5-Ethyl 8,8-dimethyl 6-(naphthalen-2-yl)-9-phenyl-6,7-dihydro-5*H*-5,9-epoxybenzo [7]annulene-5,8,8(9*H*)-tricarboxylate (3l) was prepared as a white foam from dimethyl 2-(naphthalen-2-yl)cyclopropane-1,1-dicarboxylate **1l** (28.4 mg, 0.1 mmol) and ethyl 2-(2-benzoylphenyl)-2-diazoacetate **2a** (44.1 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 10:1) in 85% yield (46.5 mg, >20:1 dr).

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.08 (s, 1H), 7.86 (d, *J* = 7.6 Hz, 1H), 7.80 – 7.75 (m, 2H), 7.72 (d, *J* = 7.6 Hz, 2H), 7.54 (d, *J* = 7.8 Hz, 1H), 7.48 (d, *J* = 7.1 Hz, 1H), 7.46 – 7.40 (m, 4H), 7.39 – 7.29 (m, 4H), 4.24 (dq, *J* = 10.9, 7.1 Hz, 1H), 4.10 (dq, *J* = 10.8, 7.1 Hz, 1H), 3.74 (s, 3H), 3.66 (d, *J* = 6.7 Hz, 1H), 2.74 – 2.69 (m, 1H), 2.66 (s, 3H), 2.42 (dd, *J* = 14.4, 7.0 Hz, 1H), 1.11 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (150 MHz, Chloroform-*d*) δ 170.8, 169.6, 168.5, 143.0, 142.2, 141.3, 138.8, 133.5, 132.2, 128.7, 128.3, 128.2, 128.0, 127.8, 127.5, 127.3, 127.2, 126.8, 126.0, 125.6, 125.2, 119.7, 89.7, 88.5, 61.7, 58.5, 52.8, 51.4, 43.1, 35.9, 14.2.

HRMS (ESI+) Calcd for $C_{34}H_{31}O_7$ (M+H)+ requires m/z 551.2065, found m/z 551.2057.



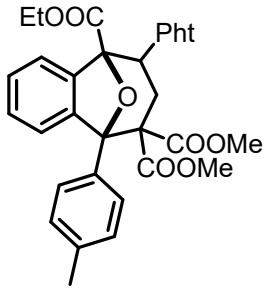
3m

5-Ethyl 8,8-dimethyl 9-phenyl-6-(thiophen-2-yl)-6,7-dihydro-5*H*-5,9-epoxybenzo [7]annulene-5,8,8(9*H*)-tricarboxylate (3m) was prepared as a white foam from dimethyl 2-(thiophen-2-yl)cyclopropane-1,1-dicarboxylate **1m** (24.1 mg, 0.1mmol) and ethyl 2-(2-benzoylphenyl)-2-diazoacetate **2a** (44.1 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 10:1) in 57% yield (28.8 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.65 (d, *J* = 7.8 Hz, 2H), 7.46 – 7.38 (m, 3H), 7.37 – 7.27 (m, 5H), 7.11 (d, *J* = 5.0 Hz, 1H), 6.92 – 6.86 (m, 1H), 4.31 – 4.15 (m, 2H), 3.75 (s, 3H), 3.73 (s, 1H), 3.11 (s, 3H), 2.67 (d, *J* = 14.4 Hz, 1H), 2.31 (dd, *J* = 14.4, 6.4 Hz, 1H), 1.18 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (150 MHz, Chloroform-*d*) δ 170.5, 169.1, 168.5, 143.1, 142.5, 141.4, 141.1, 128.7, 128.4, 128.0, 127.3, 126.8, 126.4, 125.1, 123.7, 119.8, 89.7, 88.8, 61.6, 58.2, 52.9, 51.8, 38.6, 36.0, 14.1.

HRMS (ESI+) Calcd for $C_{28}H_{27}O_7S$ (M+H)+ requires m/z 507.1472, found m/z 507.1477.



3n

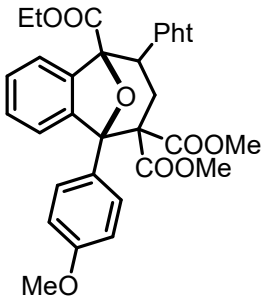
5-Ethyl 8,8-dimethyl 6-(1,3-dioxoisoindolin-2-yl)-9-(p-tolyl)-6,7-dihydro-5H-5,9 -epoxybenzo[7]annulene-5,8,8(9H)-tricarboxylate (3n) was prepared as a white foam from dimethyl 2-(1,3-dioxoisoindolin-2-yl)cyclopropane-1,1-dicarboxylate **1a**

(30.3 mg, 0.1 mmol) and ethyl 2-diazo-2-(2-(4-methylbenzoyl)phenyl)acetate **2b** (46.2 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 5:1) in 48% yield (30.1 mg, >20:1 dr).

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.83 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.73 – 7.67 (m, 4H), 7.49 – 7.44 (m, 1H), 7.35 – 7.29 (m, 2H), 7.27 (q, *J* = 2.9 Hz, 1H), 7.19 (d, *J* = 8.1 Hz, 2H), 4.71 (dd, *J* = 6.2, 2.3 Hz, 1H), 4.33 – 4.24 (m, 2H), 3.68 (s, 3H), 3.19 (s, 3H), 3.00 (dd, *J* = 15.3, 2.4 Hz, 1H), 2.34 (s, 3H), 2.08 (dd, *J* = 15.2, 6.2 Hz, 1H), 1.26 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (150 MHz, Chloroform-*d*) δ 170.5, 169.1, 168.6, 143.2, 141.9, 138.2, 136.8, 134.0, 132.2, 128.8, 128.8, 125.7, 125.3, 123.2, 119.5, 89.7, 86.0, 61.9, 60.7, 52.8, 48.7, 33.2, 21.2, 14.2.

HRMS (ESI+) Calcd for $C_{33}H_{33}N_2O_9$ $(M+NH_4)^+$ requires m/z 601.2180, found m/z 601.2168.



3o

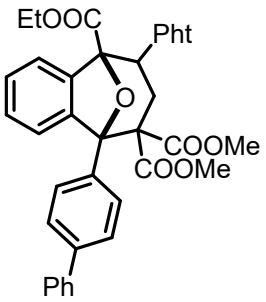
5-Ethyl 8,8-dimethyl 6-(1,3-dioxoisoindolin-2-yl)-9-(4-methoxyphenyl)-6,7-dihydro-5*H*-5,9-epoxybenzo[7]annulene-5,8,8(9*H*)-tricarboxylate (3o) was prepared as a white foam from dimethyl 2-(1,3-dioxoisoindolin-2-yl)cyclopropane-1,1-dicarboxylate **1a** (30.3 mg, 0.1 mmol) and ethyl 2-diazo-2-(2-(4-methoxybenzoyl) phenyl)acetate **2c** (48.6 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 5:1) in 43% yield (25.8 mg, >20:1 dr).

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.85 – 7.80 (m, 2H), 7.75 (d, *J* = 9.0 Hz, 2H), 7.73 – 7.68 (m, 2H), 7.46 (dd, *J* = 6.3, 2.4 Hz, 1H), 7.35 – 7.29 (m, 2H), 7.28 – 7.26 (m, 1H), 6.94 – 6.90 (m, 2H), 4.71 (dd, *J* = 6.2, 2.4 Hz, 1H), 4.28 (q, *J* = 7.1 Hz, 2H),

3.81 (s, 3H), 3.67 (s, 3H), 3.21 (s, 3H), 3.00 (dd, *J* = 15.1, 2.4 Hz, 1H), 2.06 (dd, *J* = 15.2, 6.2 Hz, 1H), 1.26 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (150 MHz, Chloroform-d) δ 170.5, 168.6, 158.8, 143.4, 141.9, 134.0, 133.5, 132.2, 128.9, 126.9, 125.7, 123.2, 119.5, 113.4, 89.6, 86.0, 62.0, 60.8, 55.3, 52.9, 52.5, 48.7, 33.2, 14.2.

HRMS (ESI+) Calcd for $C_{33}H_{33}N_2O_{10}$ $(M+NH_4)^+$ requires m/z 617.2309, found m/z 617.2112.



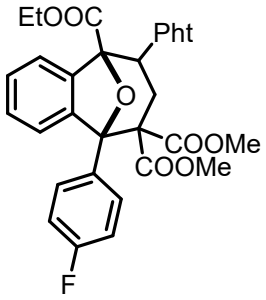
3p

5-Ethyl 8,8-dimethyl 9-([1,1'-biphenyl]-4-yl)-6-(1,3-dioxoisoindolin-2-yl) -6,7-dihydro-5*H*-5,9-epoxybenzo[7]annulene-5,8,8(9*H*)-tricarboxylate (3p) was preparedas a white foam from dimethyl 2-(1,3-dioxoisoindolin-2-yl)cyclopropane-1,1-dicarboxylate **1a** (30.3 mg, 0.1 mmol) and ethyl 2-(2-([1,1'-biphenyl]-4-carbonyl) phenyl)-2-diazoacetate **2d** (55.5 mg, 0.15 mmol) according to the General ProcedureE (eluent: PE/EA = 5:1) in 79% yield (51.1 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.90 (d, *J* = 8.2 Hz, 2H), 7.86 – 7.82 (m, 2H), 7.73 – 7.68 (m, 2H), 7.63 (dd, *J* = 8.3, 3.4 Hz, 4H), 7.55 – 7.50 (m, 1H), 7.44 (t, *J* = 7.5 Hz, 2H), 7.33 (dd, *J* = 13.3, 6.8 Hz, 4H), 4.74 (dd, *J* = 6.3, 2.3 Hz, 1H), 4.31 (q, *J* = 7.1 Hz, 2H), 3.77 (s, 1H), 3.71 (s, 3H), 3.22 (s, 3H), 3.04 (dd, *J* = 15.3, 2.3 Hz, 1H), 2.11 (dd, *J* = 15.2, 6.2 Hz, 1H), 1.28 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 170.5, 169.1, 168.6, 143.0, 141.9, 141.0, 140.3, 139.9, 134.1, 132.2, 129.0, 128.9, 128.9, 127.4, 127.2, 126.8, 125.9, 125.8, 123.2, 119.6, 89.7, 86.1, 62.0, 60.8, 52.9, 52.5, 48.7, 33.3, 14.2.

HRMS (ESI+) Calcd for $C_{38}H_{32}NO_9$ $(M+H)^+$ requires m/z 646.2072, found m/z 646.2063.



3q

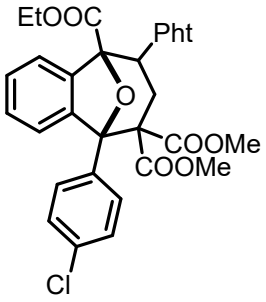
5-Ethyl 8,8-dimethyl 6-(1,3-dioxoisoindolin-2-yl)-9-(4-fluorophenyl)-6,7-dihydro-5*H*-5,9-epoxybenzo[7]annulene-5,8,8(9*H*)-tricarboxylate (3q) was prepared as a white foam from dimethyl 2-(1,3-dioxoisoindolin-2-yl)cyclopropane-1,1-dicarboxylate **1a** (30.3 mg, 0.1 mmol) and ethyl 2-diazo-2-(2-(4-fluorobenzoyl) phenyl)acetate **2e** (46.8 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 5:1) in 76% yield (44.8 mg, 9:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.85 – 7.81 (m, 4H), 7.72 – 7.68 (m,2H), 7.48 – 7.44 (m, 1H), 7.37 – 7.31 (m, 2H), 7.30 – 7.27 (m, 1H), 7.07 (t, *J* = 8.8 Hz, 2H), 4.72 (dd, *J* = 6.2, 2.2 Hz, 1H), 4.29 (q, *J* = 7.1 Hz, 2H), 3.67 (s, 3H), 3.20 (s, 3H), 3.00 (dd, *J* = 15.2, 2.3 Hz, 1H), 2.05 (dd, *J* = 15.2, 6.2 Hz, 1H), 1.26 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (150 MHz, Chloroform-*d*) δ 170.4, 168.9, 168.4, 162.9, 161.3,142.8, 141.9, 134.1, δ 128.9 (d, $J_{C\text{-}F}$ = 18.3 Hz), 127.4 (d, $J_{C\text{-}F}$ = 8.1 Hz), 125.7, 123.2, 119.6, 114.9, 114.8, 89.3, 86.0, 62.0, 60.8, 52.9, 52.4, 48.6, 33.2, 14.2.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -115.63.

HRMS (ESI+) Calcd for $C_{32}H_{27}FNO_9$ $(M+H)^+$ requires m/z 588.1665, found m/z 588.1658.



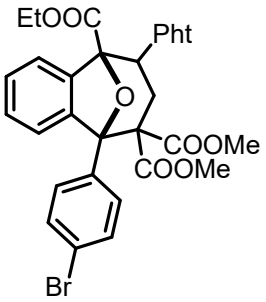
3r

5-Ethyl 8,8-dimethyl 9-(4-chlorophenyl)-6-(1,3-dioxoisoindolin-2-yl)-6,7-dihydro-5*H*-5,9-epoxybenzo[7]annulene-5,8,8(9*H*)-tricarboxylate (3r) was prepared as a white foam from dimethyl 2-(1,3-dioxoisoindolin-2-yl)cyclopropane-1,1-dicarboxylate **1a** (30.3 mg, 0.1 mmol) and ethyl 2-(2-(4-chlorobenzoyl)phenyl)-2-diazoacetate **2f** (49.2 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 5:1) in 89% yield (53.1 mg, 12:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.83 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.78 (d, *J* = 8.8 Hz, 2H), 7.70 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.46 – 7.42 (m, 1H), 7.37 – 7.33 (m, 4H), 7.30 – 7.27 (m, 1H), 4.71 (dd, *J* = 6.2, 2.2 Hz, 1H), 4.29 (q, *J* = 7.1 Hz, 2H), 3.67 (s, 3H), 3.21 (s, 3H), 2.99 (dd, *J* = 15.2, 2.2 Hz, 1H), 2.05 (dd, *J* = 15.2, 6.2 Hz, 1H), 1.25 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 170.34, 168.82, 168.34, 142.52, 141.82, 139.71, 134.08, 133.28, 129.12, 128.96, 128.22, 127.09, 125.60, 123.23, 119.63, 89.27, 86.05, 62.04, 60.75, 52.95, 52.50, 48.57, 33.18, 14.16.

HRMS (ESI+) Calcd for $C_{32}H_{27}ClNO_9$ $(M+H)^+$ requires m/z 604.1369 (Cl, 34.9686), found m/z 604.1367.



3s

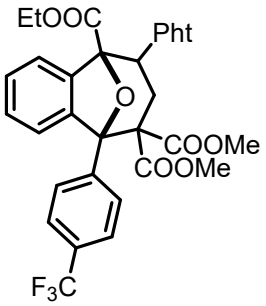
5-Ethyl 8,8-dimethyl 9-(4-bromophenyl)-6-(1,3-dioxoisoindolin-2-yl)-6,7-dihydro

-5*H*-5,9-epoxybenzo[7]annulene-5,8,8(9*H*)-tricarboxylate (3s) was prepared as a white foam from dimethyl 2-(1,3-dioxoisoindolin-2-yl)cyclopropane-1,1-dicarboxylate **1a** (30.3 mg, 0.1 mmol) and ethyl 2-(2-(4-bromobenzoyl) phenyl)-2-diazoacetate **2g** (45.0 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 5:1) in 94% yield (61 mg, 11:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.83 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.74 – 7.69 (m, 4H), 7.51 (d, *J* = 8.8 Hz, 2H), 7.46 – 7.42 (m, 1H), 7.34 (dd, *J* = 5.9, 2.9 Hz, 2H), 7.30 – 7.26 (m, 1H), 4.71 (dd, *J* = 6.2, 2.2 Hz, 1H), 4.28 (q, *J* = 7.1 Hz, 2H), 3.67 (s, 3H), 3.21 (s, 3H), 2.99 (dd, *J* = 15.2, 2.2 Hz, 1H), 2.05 (dd, *J* = 15.2, 6.2 Hz, 1H), 1.25 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 170.3, 168.8, 168.3, 142.4, 141.8, 140.2, 134.1, 131.2, 129.1, 129.0, 127.4, 125.6, 123.2, 121.5, 119.6, 89.3, 86.1, 62.0, 60.7, 52.9, 52.5, 48.6, 33.2, 14.2.

HRMS (ESI+) Calcd for $C_{32}H_{27}BrNO_9$ $(M+H)^+$ requires m/z 648.0864 (Br, 78.9183), found m/z 648.0869.



3t

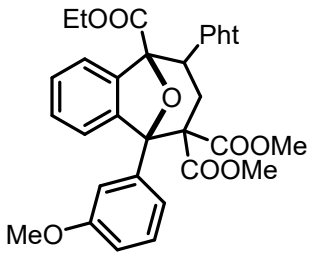
5-Ethyl 8,8-dimethyl 6-(1,3-dioxoisoindolin-2-yl)-9-(4-(trifluoromethyl)phenyl)-6,7-dihydro-5*H*-5,9-epoxybenzo[7]annulene-5,8,8(9*H*)-tricarboxylate (3t) was preparedas a white foam from dimethyl 2-(1,3-dioxoisoindolin-2-yl)cyclopropane-1,1-dicarboxylate **1a** (30.3 mg, 0.1 mmol) and ethyl 2-diazo-2-(2-(4-(trifluoromethyl) benzoyl)phenyl)acetate **2h** (54.3 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 5:1) in 99% yield (63.3 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.97 (d, *J* = 8.3 Hz, 2H), 7.83 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.71 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.65 (d, *J* = 8.4 Hz, 2H), 7.48 – 7.42 (m, 1H), 7.35 (dd, *J* = 5.5, 3.3 Hz, 2H), 7.33 – 7.27 (m, 1H), 4.73 (dd, *J* = 6.2, 2.0 Hz, 1H), 4.30 (q, *J* = 7.1 Hz, 2H), 3.69 (s, 3H), 3.19 (s, 3H), 3.01 (dd, *J* = 15.3, 2.0 Hz, 1H), 2.08 (dd, *J* = 15.3, 6.2 Hz, 1H), 1.28 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 170.3, 168.7, 168.3, 145.1, 142.1, 141.8, 134.1, 132.1, 129.3, 129.0, 126.0, 126.0, 125.0 (q, $J_{C\text{-}F}$ = 3.7 Hz) ,123.2, 119.7, 89.3, 86.2, 62.1, 60.8, 53.0, 52.5, 48.5, 33.2, 14.2.

^{19}F NMR (376 MHz, Chloroform-*d*) δ -62.46.

HRMS (ESI+) Calcd for $C_{33}H_{27}F_3NO_9$ $(M+H)^+$ requires m/z 638.1633, found m/z 638.1627.



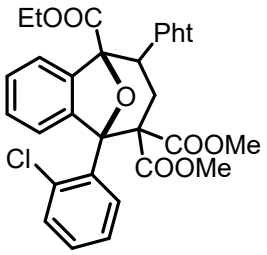
3u

5-Ethyl 8,8-dimethyl 6-(1,3-dioxoisoindolin-2-yl)-9-(3-methoxyphenyl)-6,7-dihydro-5*H*-5,9-epoxybenzo[7]annulene-5,8,8(9*H*)-tricarboxylate (3u) was preparedas a white foam from dimethyl 2-(1,3-dioxoisoindolin-2-yl)cyclopropane-1,1-dicarboxylate **1a** (30.3 mg, 0.1 mmol) and ethyl 2-diazo-2-(2-(3-methoxybenzoyl)phenyl)acetate **2i** (48.6 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 3:1) in 80% yield (48 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.83 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.70 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.48 – 7.39 (m, 2H), 7.39 – 7.26 (m, 5H), 6.83 (dd, *J* = 8.0, 1.9 Hz, 1H), 4.72 (dd, *J* = 6.1, 2.5 Hz, 1H), 4.27 (qd, *J* = 7.1, 1.4 Hz, 2H), 3.83 (s, 3H), 3.69 (s, 3H), 3.22 (s, 3H), 3.02 (dd, *J* = 15.1, 2.5 Hz, 1H), 2.09 (dd, *J* = 15.1, 6.2 Hz, 1H), 1.27 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 170.5, 169.1, 168.6, 159.5, 143.0, 142.8, 142.0, 134.0, 132.2, 129.1, 129.0, 128.9, 125.7, 123.2, 119.5, 117.7, 112.8, 111.4, 89.6, 86.1, 61.9, 60.9, 55.3, 52.9, 52.5, 48.7, 33.4, 14.2.

HRMS (ESI+) Calcd for $C_{33}H_{30}NO_{10}$ $(M+H)^+$ requires m/z 600.1864, found m/z 600.1856.



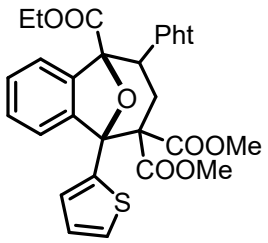
3v

5-Ethyl 8,8-dimethyl 9-(2-chlorophenyl)-6-(1,3-dioxoisoindolin-2-yl)-6,7-dihydro-5*H*-5,9-epoxybenzo[7]annulene-5,8,8(9*H*)-tricarboxylate (3v) was preparedas a white foam from dimethyl 2-(1,3-dioxoisoindolin-2-yl)cyclopropane-1,1-dicarboxylate **1a** (30.3 mg, 0.1 mmol) and ethyl 2-(2-(2-chlorobenzoyl)phenyl)-2-diazoacetate **2j** (49.2 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 5:1) in 41% yield (24.6 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.85 – 7.77 (m, 3H), 7.73 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.50 (dd, *J* = 5.7, 2.9 Hz, 1H), 7.38 – 7.30 (m, 2H), 7.30 – 7.26 (m, 1H), 7.25 – 7.19 (m, 2H), 7.09 (dd, *J* = 5.6, 2.9 Hz, 1H), 5.26 (dd, *J* = 12.1, 4.8 Hz, 1H), 4.25 (q, *J* = 7.1 Hz, 2H), 3.82 (s, 3H), 3.77 (s, 3H), 3.31 – 3.20 (m, 1H), 2.57 (dd, *J* = 13.7, 4.8 Hz, 1H), 1.19 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 167.0, 169.4, 168.1, 167.7, 143.4, 138.4, 135.8, 135.1, 134.3, 132.6, 131.7, 130.9, 129.4, 128.8, 128.1, 125.0, 124.8, 123.6, 123.3, 90.8, 87.0, 62.2, 61.2, 53.4, 53.0, 49.4, 30.5, 14.1.

HRMS (ESI+) Calcd for $C_{32}H_{27}ClNO_9$ $(M+H)^+$ requires m/z 604.1369 (Cl, 34.9686), found m/z 604.1371.



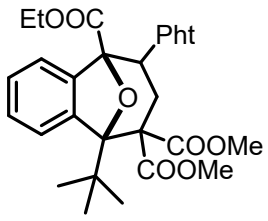
3w

5-Ethyl 8,8-dimethyl 6-(1,3-dioxoisoindolin-2-yl)-9-(thiophen-2-yl)-6,7- dihydro-5*H*-5,9-epoxybenzo[7]annulene-5,8,8(9*H*)-tricarboxylate (3w) was prepared as a white foam from dimethyl 2-(1,3-dioxoisoindolin-2-yl)cyclopropane- 1,1-dicarboxylate **1a** (30.3 mg, 0.1 mmol) and ethyl 2-diazo-2-(2-(thiophene-2-carbonyl)phenyl)acetate **2k** (45.0 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 5:1) in 65% yield (37.5 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.84 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.74 – 7.66 (m, 3H), 7.42 (d, *J* = 3.5 Hz, 1H), 7.35 (dt, *J* = 15.0, 7.2 Hz, 2H), 7.27 (d, *J* = 3.6 Hz, 1H), 7.24 (s, 1H), 7.04 – 6.99 (m, 1H), 4.67 (d, *J* = 5.5 Hz, 1H), 4.28 (q, *J* = 7.0 Hz, 2H), 3.67 (s, 3H), 3.26 (s, 3H), 2.93 (d, *J* = 15.6 Hz, 1H), 1.93 (dd, *J* = 15.5, 6.4 Hz, 1H), 1.27 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 169.7, 168.5, 168.4, 143.6, 142.5, 141.4, 134.0, 132.2, 129.1, 129.1, 126.6, 126.0, 125.0, 124.3, 123.2, 119.5, 89.1, 86.1, 62.01, 60.8, 52.8, 52.7, 48.6, 32.5, 14.2.

HRMS (ESI+) Calcd for $C_{30}H_{29}N_2O_9S$ $(M+NH_4)^+$ requires m/z 593.1588, found m/z 593.1581.



3x

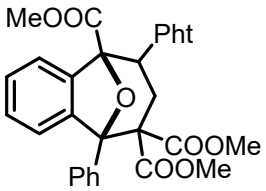
6-Ethyl 8,8-dimethyl 9-(tert-butyl)-6-(1,3-dioxoisoindolin-2-yl)-6,7-dihydro-5*H* -5,9-epoxybenzo[7]annulene-5,8,8(9*H*)-tricarboxylate (3x) was preparedas a white

foam from dimethyl 2-(1,3-dioxoisoindolin-2-yl)cyclopropane-1,1-dicarboxylate **1a** (30.3 mg,0.1 mmol) and methyl ethyl 2-diazo-2-(2-pivaloylphenyl)acetate **2l** (41.1 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 5:1) in 37% yield (20.5 mg, 11:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.82 – 7.74 (m, 1H), 7.72 – 7.63 (m, 3H), 7.53 – 7.46 (m, 1H), 7.38 – 7.34 (m, 1H), 7.24 – 7.14 (m, 2H), 4.94 (dd, *J* = 11.1, 3.7 Hz, 1H), 4.40 – 4.22 (m, 2H), 3.67 (s, 3H), 3.62 (s, 3H), 3.25 – 3.21 (m, 1H), 2.73 (dtt, *J* = 15.6, 8.0, 3.6 Hz, 1H), 1.36 (t, *J* = 7.1 Hz, 3H), 1.02 (s, 9H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 170.20, 169.01, 168.82, 141.39, 137.33, 134.52, 134.41, 129.18, 128.57, 124.31, 123.53, 114.13, 89.78, 62.22, 57.08, 52.83, 52.80, 52.78, 49.19, 40.10, 25.76, 25.16, 14.10.

HRMS (ESI+) Calcd for $C_{30}H_{32}NO_9$ $(M+H)^+$ requires m/z 550.2072, found m/z 550.2062.



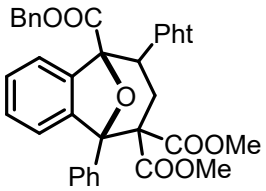
3y

Trimethyl 6-(1,3-dioxoisoindolin-2-yl)-9-phenyl-6,7-dihydro-5*H*-5,9-epoxybenzo [7]annulene-5,8,8(9*H*)-tricarboxylate (3y) was preparedas a white foam from dimethyl 2-(1,3-dioxoisoindolin-2-yl)cyclopropane-1,1-dicarboxylate **1a** (30.3 mg,0.1 mmol) and methyl 2-(2-benzoylphenyl)-2-diazoacetate **2m** (42.0 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 5:1) in 83% yield (46.1 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.86 – 7.77 (m, 4H), 7.70 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.50 – 7.46 (m, 1H), 7.43 – 7.27 (m, 6H), 4.72 (dd, *J* = 6.1, 2.1 Hz, 1H), 3.84 (s, 3H), 3.69 (s, 3H), 3.18 (s, 3H), 3.02 (dd, *J* = 15.2, 2.2 Hz, 1H), 2.07 (dd, *J* = 15.2, 6.1 Hz, 1H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 169.3, 168.1, 167.9, 141.8, 140.4, 139.9, 132.9, 131.0, 127.8, 127.0, 126.2, 124.7, 124.2, 122.1, 118.4, 88.6, 84.9, 59.6, 51.9, 51.8, 51.3, 47.6, 32.0.

HRMS (ESI+) Calcd for $C_{31}H_{26}NO_9$ $(M+H)^+$ requires m/z 556.1602, found m/z 556.1606.



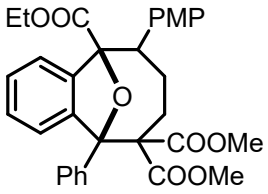
3z

5-Benzyl 8,8-dimethyl 6-(1,3-dioxoisoindolin-2-yl)-9-phenyl-6,7-dihydro-5*H* -5,9-epoxybenzo[7]annulene-5,8,8(9*H*)-tricarboxylate (3z) was preparedas a white foam from dimethyl 2-(1,3-dioxoisoindolin-2-yl)cyclopropane-1,1-dicarboxylate **1a** (30.3 mg, 0.1 mmol) and benzyl 2-(2-benzoylphenyl)-2-diazoacetate **2n** (53.4 mg, 0.15 mmol) according to the General Procedure E (eluent: PE/EA = 5:1) in 64% yield (40.5 mg, >20:1 dr).

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.82 (d, *J* = 7.5 Hz, 2H), 7.80 – 7.76 (m, 2H), 7.70 – 7.68 (m, 2H), 7.46 (d, *J* = 7.6 Hz, 1H), 7.37 (t, *J* = 7.8 Hz, 2H), 7.33 – 7.27 (m, 5H), 7.21 – 7.17 (m, 4H), 5.38 (d, *J* = 12.4 Hz, 1H), 5.18 (d, *J* = 12.4 Hz, 1H), 4.72 (dd, *J* = 6.2, 2.1 Hz, 1H), 3.68 (s, 3H), 3.19 (s, 3H), 2.99 (dd, *J* = 15.2, 2.1 Hz, 1H), 2.09 (dd, *J* = 15.2, 6.2 Hz, 1H).

^{13}C NMR (150 MHz, Chloroform-*d*) δ 170.5, 169.0, 168.2, 143.0, 141.7, 141.2, 135.5, 133.9, 128.9, 128.9, 128.4, 128.3, 128.2, 128.1, 127.3, 125.8, 125.4, 123.2, 119.6, 89.7, 86.1, 67.3, 60.8, 52.9, 52.4, 48.7, 33.3.

HRMS (ESI+) Calcd for $C_{37}H_{30}NO_9$ $(M+H)^+$ requires m/z 632.1915, found m/z 632.1912.



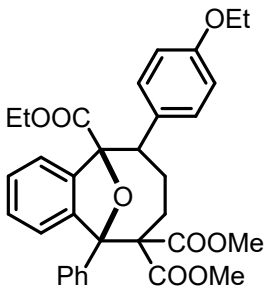
5a

5-Ethyl 9,9-dimethyl 6-(4-methoxyphenyl)-10-phenyl-7,8-dihydro-5,10-epoxybenzo[8]annulene-5,9,9(6*H*,10*H*)-tricarboxylate (5a) was prepared as a white foam from dimethyl 2-(4-methoxyphenyl)cyclobutane-1,1-dicarboxylate **4a** (55.6 mg, 0.2 mmol) and ethyl 2-(2-benzoylphenyl)-2-diazoacetate **2a** (88.2 mg, 0.3 mmol) according to the General Procedure F (eluent: PE/EA = 10:1) in 70% yield (76.2 mg, >20:1 dr).

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.30 (d, *J* = 7.8 Hz, 2H), 7.58 (d, *J* = 8.5 Hz, 2H), 7.52 (d, *J* = 7.5 Hz, 1H), 7.37 (t, *J* = 7.8 Hz, 2H), 7.30 (q, *J* = 7.6 Hz, 2H), 7.26 – 7.21 (m, 2H), 6.90 (d, *J* = 8.6 Hz, 2H), 4.00 (qd, *J* = 7.0, 1.7 Hz, 2H), 3.81 (s, 3H), 3.74 (s, 3H), 3.72 (t, *J* = 4.8 Hz, 1H), 3.40 (s, 3H), 2.56 (dd, *J* = 14.4, 10.4 Hz, 1H), 2.01 – 1.92 (m, 2H), 1.81 – 1.72 (m, 1H), 1.05 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (150 MHz, Chloroform-*d*) δ 171.6, 170.9, 170.2, 158.4, 144.8, 142.7, 140.8, 134.9, 129.9, 128.9, 128.6, 128.0, 126.9, 125.9, 125.6, 121.4, 113.8, 94.1, 93.9, 71.7, 61.4, 55.3, 53.8, 52.4, 52.3, 31.1, 30.2, 13.9.

HRMS (ESI+) Calcd for $C_{32}H_{33}O_8$ $(M+H)^+$ requires m/z 559.2327, found m/z 559.2324.



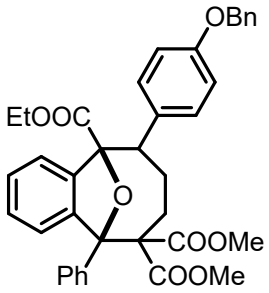
5b

5-Ethyl 9,9-dimethyl 6-(4-ethoxyphenyl)-10-phenyl-7,8-dihydro-5,10-epoxybenzo[8]annulene-5,9,9(6*H*,10*H*)-tricarboxylate (5b) was prepared as a white foam from dimethyl 2-(4-ethoxyphenyl)cyclobutane-1,1-dicarboxylate **4b** (58.2 mg, 0.2 mmol) and ethyl 2-(2-benzoylphenyl)-2-diazoacetate **2a** (88.2 mg, 0.30 mmol) according to the General Procedure F (eluent: PE/EA = 10:1) in 82% yield (91.6 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.28 (d, *J* = 7.3 Hz, 2H), 7.53 (dd, *J* = 12.2, 8.5 Hz, 3H), 7.39 – 7.34 (m, 2H), 7.33 – 7.27 (m, 2H), 7.26 – 7.20 (m, 2H), 6.87 (d, *J* = 8.8 Hz, 2H), 4.06 – 4.01 (m, 2H), 4.01 – 3.95 (m, 2H), 3.73 (s, 3H), 3.70 (t, *J* = 5.0 Hz, 1H), 3.39 (s, 3H), 2.55 (dd, *J* = 14.8, 10.5 Hz, 1H), 1.99 – 1.91 (m, 2H), 1.80 – 1.70 (m, 1H), 1.41 (t, *J* = 7.0 Hz, 3H), 1.04 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 171.6, 170.9, 170.3, 157.8, 144.8, 142.7, 140.9, 134.8, 129.9, 128.9, 128.6, 128.1, 127.0, 126.0, 125.6, 121.4, 114.4, 94.1, 94.0, 71.7, 63.5, 61.4, 53.9, 52.4, 52.3, 31.2, 30.3, 15.0, 14.0.

HRMS (ESI+) Calcd for $C_{33}H_{35}O_8$ $(M+H)^+$ requires m/z 559.2327, found m/z 559.2324.



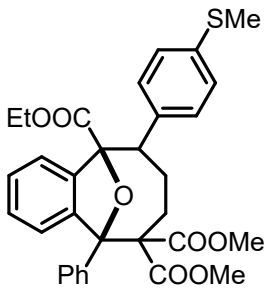
5c

5-Ethyl 9,9-dimethyl 6-(4-(benzyloxy)phenyl)-10-phenyl-7,8-dihydro-5,10-epoxybenzo[8]annulene-5,9,9(6*H*,10*H*)-tricarboxylate (5c) was prepared as a white foam from dimethyl 2-(4-(benzyloxy)phenyl)cyclobutane-1,1-dicarboxylate **4c** (70.8 mg, 0.2 mmol) and ethyl 2-(2-benzoylphenyl)-2-diazoacetate **2a** (88.2 mg, 0.30 mmol) according to the General Procedure F (eluent: PE/EA = 10:1) in 61% yield (75.8 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.29 (d, *J* = 7.3 Hz, 2H), 7.57 (d, *J* = 8.7 Hz, 2H), 7.52 (d, *J* = 8.7 Hz, 1H), 7.45 (d, *J* = 6.8 Hz, 2H), 7.42 – 7.35 (m, 4H), 7.35 – 7.28 (m, 3H), 7.26 – 7.21 (m, 2H), 6.97 (d, *J* = 8.7 Hz, 2H), 5.07 (s, 2H), 3.98 (qd, *J* = 7.1, 1.8 Hz, 2H), 3.74 (s, 3H), 3.71 (t, *J* = 5.0 Hz, 1H), 3.40 (s, 3H), 2.56 (dd, *J* = 14.6, 10.2 Hz, 1H), 2.02 – 1.91 (m, 2H), 1.82 – 1.71 (m, 1H), 1.02 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 171.6, 170.9, 170.3, 157.7, 144.8, 142.7, 140.8, 137.3, 135.2, 129.9, 128.9, 128.7, 128.6, 128.1, 127.7, 127.0, 126.0, 125.6, 121.4, 114.8, 94.1, 94.0, 71.7, 70.1, 61.4, 53.9, 52.4, 52.3, 31.2, 30.23, 14.0.

HRMS (ESI+) Calcd for $C_{38}H_{37}O_8$ $(M+H)^+$ requires m/z 621.248, found m/z 621.2477.



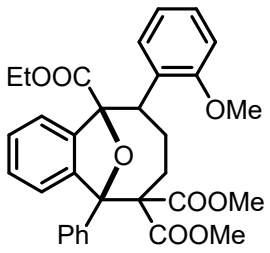
5d

5-Ethyl 9,9-dimethyl 6-(4-(methylthio)phenyl)-10-phenyl-7,8-dihydro-5,10-epoxybenzo[8]annulene-5,9,9(6*H*,10*H*)-tricarboxylate (5d) was prepared as a white foam from dimethyl 2-(4-(methylthio)phenyl)cyclobutane-1,1-dicarboxylate **4d** (58.8 mg, 0.2 mmol) and ethyl 2-(2-benzoylphenyl)-2-diazoacetate **2a** (88.2 mg, 0.30 mmol) according to the General Procedure F (eluent: PE/EA = 10:1) in 73% yield (81.8 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.29 (d, *J* = 7.3 Hz, 2H), 7.57 (d, *J* = 8.4 Hz, 2H), 7.50 (d, *J* = 7.6 Hz, 1H), 7.40 – 7.34 (m, 2H), 7.34 – 7.27 (m, 2H), 7.26 – 7.20 (m, 4H), 4.00 (q, *J* = 7.1 Hz, 2H), 3.74 (s, 3H), 3.71 (t, *J* = 5.0 Hz, 1H), 3.39 (s, 3H), 2.53 (dd, *J* = 14.7, 10.3 Hz, 1H), 2.48 (s, 3H), 2.01 – 1.92 (m, 2H), 1.80 – 1.69 (m, 1H), 1.05 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 171.5, 170.8, 170.2, 144.7, 142.7, 140.7, 139.7, 136.5, 129.3, 128.9, 128.7, 128.1, 127.0, 126.8, 126.0, 125.7, 121.3, 94.0, 93.9, 71.6, 61.5, 54.1, 52.5, 52.3, 31.1, 30.1, 16.1, 14.0.

HRMS (ESI+) Calcd for $C_{32}H_{36}N_1O_7S$ $(M+NH_4)^+$ requires m/z 578.2207, found m/z 578.2191.



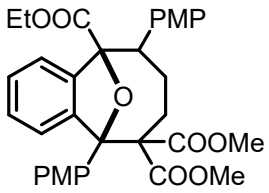
5e

5-Ethyl 9,9-dimethyl 6-(2-methoxyphenyl)-10-phenyl-7,8-dihydro-5,10-epoxybenzo[8]annulene-5,9,9(6*H*,10*H*)-tricarboxylate (5e) was prepared as a white foam from dimethyl 2-(2-methoxyphenyl)cyclobutane-1,1-dicarboxylate **4e** (55.8 mg, 0.2 mmol) and ethyl 2-(2-benzoylphenyl)-2-diazoacetate **2a** (88.2 mg, 0.3 mmol) according to the General Procedure F (eluent: PE/EA = 10:1) in 33% yield (36.1 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.34 (d, *J* = 8.0 Hz, 2H), 8.08 (d, *J* = 7.6 Hz, 1H), 7.52 (d, *J* = 7.4 Hz, 1H), 7.37 (t, *J* = 7.7 Hz, 2H), 7.30 (t, *J* = 6.6 Hz, 2H), 7.25 – 7.20 (m, 3H), 6.99 (t, *J* = 7.7 Hz, 1H), 6.88 (d, *J* = 8.6 Hz, 1H), 4.25 (t, *J* = 4.6 Hz, 1H), 4.08 – 3.96 (m, 2H), 3.85 (s, 3H), 3.72 (s, 3H), 3.35 (s, 3H), 2.53 (dd, *J* = 14.1, 10.0 Hz, 1H), 1.97 (dd, *J* = 14.6, 8.5 Hz, 1H), 1.89 – 1.76 (m, 2H), 1.01 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 171.9, 171.1, 170.3, 156.8, 145.1, 143.0, 140.8, 130.8, 129.2, 128.7, 128.5, 128.0, 127.7, 126.9, 126.0, 125.6, 121.4, 120.6, 110.2, 94.3, 93.9, 72.0, 61.3, 55.6, 52.4, 52.2, 46.1, 31.5, 27.1, 13.9.

HRMS (ESI+) Calcd for $C_{32}H_{33}O_8$ $(M+H)^+$ requires m/z 545.217, found m/z 545.2173.



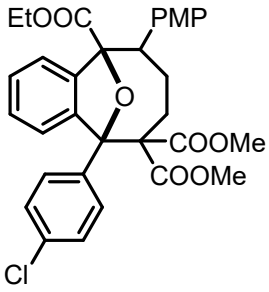
5f

5-Ethyl 9,9-dimethyl 6,10-bis(4-methoxyphenyl)-7,8-dihydro-5,10-epoxybenzo [8]annulene-5,9,9(6*H*,10*H*)-tricarboxylate (5f) was prepared as a white foam from dimethyl 2-(4-methoxyphenyl)cyclobutane-1,1-dicarboxylate **4a** (55.6 mg, 0.2 mmol) and ethyl 2-diazo-2-(2-(4-methoxybenzoyl)phenyl)acetate **2c** (97.2 mg, 0.3 mmol) according to the General Procedure F (eluent: PE/EA = 10:1) in 47% yield (54.0 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.20 (d, *J* = 9.0 Hz, 2H), 7.56 (d, *J* = 8.7 Hz, 2H), 7.50 (d, *J* = 7.0 Hz, 1H), 7.31 – 7.27 (m, 2H), 7.26 – 7.21 (m, 1H), 6.89 (dd, *J* = 8.8, 3.9 Hz, 4H), 3.99 (q, *J* = 7.1 Hz, 2H), 3.80 (s, 3H), 3.79 (s, 3H), 3.73 (s, 3H), 3.69 (t, *J* = 4.8 Hz, 1H), 3.42 (s, 3H), 2.55 (dd, *J* = 14.6, 10.3 Hz, 1H), 1.99 – 1.88 (m, 2H), 1.77 – 1.68 (m, 1H), 1.03 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (150 MHz, Chloroform-*d*) δ 171.7, 171.0, 170.3, 158.4, 143.1, 140.7, 137.2, 134.9, 129.9, 128.8, 128.6, 127.4, 125.5, 121.4, 113.8, 113.3, 94.0, 93.8, 71.6, 61.4, 55.33, 55.29, 53.8, 52.41, 52.40, 31.0, 30.1, 14.0.

HRMS (ESI+) Calcd for $C_{32}H_{33}O_8$ $(M+H)^+$ requires m/z 545.217, found m/z 545.2173.



5g

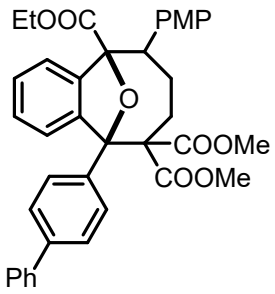
6-Ethyl 9,9-dimethyl 10-(4-chlorophenyl)-6-(4-methoxyphenyl)-7,8-dihydro-5,10

-epoxybenzo[8]annulene-5,9,9(6*H*,10*H*)-tricarboxylate (5g) was prepared as a white foam from dimethyl 2-(4-methoxyphenyl)cyclobutane-1,1-dicarboxylate **4a** (55.6mg, 0.2 mmol) and ethyl 2-(2-(4-chlorobenzoyl)phenyl)-2-diazoacetate **2f** (98.4 mg, 0.3 mmol) according to the General Procedure F (eluent: PE/EA = 10:1) in 64% yield (73.8 mg, >20:1 dr).

^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.27 (d, *J* = 8.7 Hz, 2H), 7.55 (d, *J* = 8.3 Hz, 2H), 7.51 (d, *J* = 7.6 Hz, 1H), 7.35 – 7.28 (m, 3H), 7.26 (s, 2H), 6.89 (d, *J* = 8.3 Hz, 2H), 4.04 – 3.95 (m, 2H), 3.80 (s, 3H), 3.72 (s, 3H), 3.70 (t, *J* = 4.9 Hz, 1H), 3.43 (s, 3H), 2.54 (dd, *J* = 14.6, 10.4 Hz, 1H), 2.00 – 1.89 (m, 2H), 1.79 – 1.71 (m, 1H), 1.03 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (150 MHz, Chloroform-*d*) δ 171.4, 170.7, 170.2, 158.5, 143.5, 142.3, 140.7, 134.6, 132.9, 129.8, 129.1, 128.7, 128.2, 127.7, 125.4, 121.4, 113.9, 94.2, 93.5, 71.5, 61.5, 55.3, 53.7, 52.5, 52.4, 31.1, 30.0, 14.0.

HRMS (ESI+) Calcd for $C_{32}H_{31}ClO_8$ $(M+H)^+$ requires m/z 579.178 (Cl, 34.9686), found m/z 579.1781.



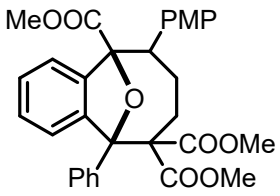
5h

7-Ethyl 9,9-dimethyl 10-([1,1'-biphenyl]-4-yl)-6-(4-methoxyphenyl)-7,8-dihydro -5,10-epoxybenzo[8] annulene-5,9,9(6*H*,10*H*)-tricarboxylate (5h) was preparedas a white foam from dimethyl 2-(4-methoxyphenyl)cyclobutane-1,1-dicarboxylate **4a** (55.6 mg, 0.2 mmol) and ethyl 2-(2-([1,1'-biphenyl]-4-carbonyl)phenyl)-2-diazoacetate **2d** (111 mg, 0.30 mmol) according to the General Procedure F (eluent: PE/EA = 10:1) in 64% yield (73.8 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.35 (d, *J* = 8.4 Hz, 2H), 7.64 – 7.56 (m, 6H), 7.53 (d, *J* = 8.1 Hz, 1H), 7.43 (t, *J* = 7.5 Hz, 2H), 7.37 – 7.27 (m, 4H), 6.90 (d, *J* = 8.7 Hz, 2H), 4.00 (q, *J* = 7.1 Hz, 2H), 3.81 (s, 3H), 3.76 (s, 3H), 3.72 (t, *J* = 4.9 Hz, 1H), 3.43 (s, 3H), 2.64 – 2.51 (m, 1H), 2.01 – 1.93 (m, 2H), 1.81 – 1.72 (m, 1H), 1.06 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 171.6, 170.9, 170.3, 158.4, 143.9, 142.6, 141.1, 140.9, 139.5, 134.9, 129.9, 129.0, 128.8, 128.7, 127.24, 127.16, 126.7, 126.47, 125.6, 121.5, 113.9, 94.2, 93.9, 71.6, 61.4, 55.3, 53.9, 52.5, 52.4, 31.2, 30.3, 14.0.

HRMS (ESI+) Calcd for $C_{38}H_{37}O_8$ $(M+H)^+$ requires m/z 621.2483, found m/z 621.2483.



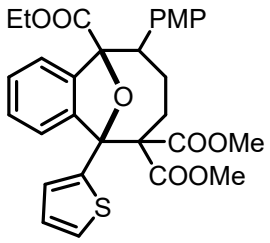
5i

7-Ethyl 9,9-dimethyl 10-([1,1'-biphenyl]-4-yl)-6-(4-methoxyphenyl)-7,8-dihydro -5,10-epoxybenzo[8] annulene-5,9,9(6*H*,10*H*)-tricarboxylate (5i) was preparedas a white foam from dimethyl 2-(4-methoxyphenyl)cyclobutane-1,1-dicarboxylate **3a** (55.6 mg, 0.2 mmol) and methyl 2-(2-benzoylphenyl)-2-diazoacetate **2y** (84.0 mg, 0.3 mmol) according to the General Procedure F (eluent: PE/EA = 10:1) in 48% yield (50.7 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.25 (d, *J* = 7.4 Hz, 2H), 7.54 (dd, *J* = 13.9, 7.9 Hz, 3H), 7.40 – 7.34 (m, 2H), 7.33 – 7.28 (m, 2H), 7.26 – 7.21 (m, 2H), 6.92 – 6.87 (m, 2H), 3.81 (s, 3H), 3.73 (s, 3H), 3.71 (t, *J* = 5.0 Hz, 1H), 3.54 (s, 3H), 3.39 (s, 3H), 2.54 (dd, *J* = 14.6, 10.3 Hz, 1H), 2.00 – 1.91 (m, 2H), 1.81 – 1.71 (m, 1H).

^{13}C NMR (150 MHz, Chloroform-*d*) δ 171.6, 171.5, 170.2, 158.4, 144.7, 142.7, 140.7, 134.8, 129.8, 128.9, 128.6, 128.1, 127.0, 125.9, 125.6, 121.4, 113.9, 94.3, 94.0, 71.6, 55.3, 54.0, 52.4, 52.4, 52.3, 31.1, 30.2.

HRMS (ESI+) Calcd for $C_{31}H_{31}O_8$ $[M+Na]^+$ requires m/z 531.2014, found m/z 531.2010.



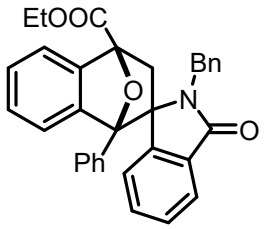
5j

7-Ethyl 9,9-dimethyl 6-(4-methoxyphenyl)-10-(thiophen-2-yl)-7,8-dihydro-5,10-epoxybenzo[8]annulene-5,9,9(6*H*,10*H*)-tricarboxylate (5j) was preparedas a white foam from dimethyl 2-(4-methoxyphenyl)cyclobutane-1,1-dicarboxylate **3a** (55.6 mg, 0.2 mmol) and ethyl 2-diazo-2-(2-(thiophene-2-carbonyl)phenyl)acetate **2k** (90.0 mg, 0.3 mmol) according to the General Procedure F (eluent: PE/EA = 10:1) in 62% yield (68.5 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.70 (d, *J* = 3.6 Hz, 1H), 7.58 (d, *J* = 8.6 Hz, 2H), 7.50 (d, *J* = 7.2 Hz, 1H), 7.36 – 7.27 (m, 3H), 7.19 (d, *J* = 5.1 Hz, 1H), 6.97 (t, 1H), 6.89 (d, *J* = 8.7 Hz, 2H), 4.02 (q, *J* = 7.5, 6.7 Hz, 2H), 3.81 (s, 3H), 3.78 (s, 3H), 3.69 (t, *J* = 4.5 Hz, 1H), 3.47 (s, 3H), 2.55 (dd, *J* = 14.6, 10.7 Hz, 1H), 2.04 – 1.94 (m, 1H), 1.92 – 1.80 (m, 1H), 1.76 – 1.65 (m, 1H), 1.05 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 171.4, 170.4, 169.7, 158.5, 149.7, 142.4, 140.5, 134.2, 130.0, 129.2, 128.8, 126.8, 125.2, 124.8, 123.0, 121.5, 113.7, 94.4, 92.5, 71.6, 61.5, 55.3, 53.7, 52.5, 52.3, 30.4, 29.2, 14.0.

HRMS (ESI+) Calcd for $C_{30}H_{31}O_8S$ $(M+H)^+$ requires m/z 551.1734, found m/z 551.1736.



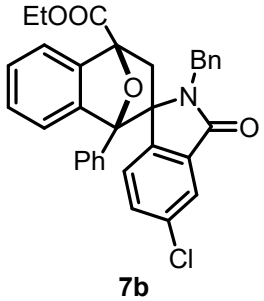
7a

Ethyl 2-benzyl-3-oxo-1'-phenyl-1'*H*-spiro[isoindoline-1,2'-[1,4] epoxynaphthalene]-4'(3'*H*)-carboxylate (7a) was preparedas a white foam from 2-benzyl-3- methyleneisoindolin-1-one **6a** (47.1 mg, 0.2 mmol) and ethyl 2-(2-benzoylphenyl) -2-diazoacetate **2a** (88.2 mg, 0.3 mmol) according to the General Procedure G (eluent: PE/EA = 10:1) in 70% yield (70.5 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.94 (d, *J* = 7.5 Hz, 1H), 7.70 (d, *J* = 7.4 Hz, 1H), 7.53 – 7.44 (m, 4H), 7.41 – 7.35 (m, 3H), 7.34 – 7.26 (m, 5H), 7.09 (s, 2H), 6.96 (d, *J* = 7.4 Hz, 1H), 5.37 (d, *J* = 7.9 Hz, 1H), 4.92 (d, *J* = 15.5 Hz, 1H), 4.68 (d, *J* = 15.5 Hz, 1H), 4.59 – 4.44 (m, 2H), 2.88 (d, *J* = 13.0 Hz, 1H), 2.29 (d, *J* = 13.0 Hz, 1H), 1.50 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (150 MHz, Chloroform-*d*) δ 168.7, 168.0, 145.7, 145.4, 144.1, 138.9, 133.8, 132.8, 131.3, 128.9, 128.8, 128.6, 128.5, 128.4, 127.4, 127.3, 124.8, 123.5, 122.8, 122.6, 119.6, 85.5, 75.1, 62.1, 44.5, 44.3, 14.5.

HRMS (ESI+) Calcd for $C_{33}H_{28}NO_4$ $(M+H)^+$ requires m/z 502.2013, found m/z 502.2013.



7b

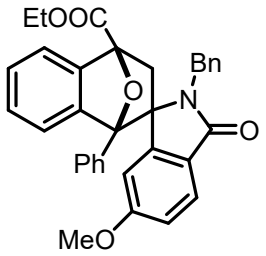
Ethyl 2-benzyl-5-chloro-3-oxo-1'-phenyl-1'*H*-spiro[isoindoline-1,2'-[1,4] epoxynaphthalene]-4'(3'*H*)-carboxylate (7b) was preparedas a white foam from 2-benzyl-6-chloro-3-methyleneisoindolin-1-one **6b** (53.8 mg, 0.2 mmol) and ethyl 2-

(2-benzoylphenyl)-2-diazoacetate **2a** (88.2 mg, 0.30 mmol) according to the General Procedure G (eluent: PE/EA = 10:1) in 63% yield (67.1 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.83 (d, *J* = 2.0 Hz, 1H), 7.64 (d, *J* = 6.5 Hz, 1H), 7.43 – 7.32 (m, 6H), 7.26 – 7.20 (m, 4H), 7.17 (dd, *J* = 8.3, 2.0 Hz, 1H), 7.04 (s, 2H), 6.91 (d, *J* = 7.5 Hz, 1H), 5.19 (d, *J* = 8.2 Hz, 1H), 4.82 (d, *J* = 15.5 Hz, 1H), 4.59 (d, *J* = 15.5 Hz, 1H), 4.52 – 4.39 (m, 2H), 2.80 (d, *J* = 13.1 Hz, 1H), 2.18 (d, *J* = 13.0 Hz, 1H), 1.43 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (150 MHz, Chloroform-*d*) δ 167.8, 167.4, 145.6, 144.0, 143.7, 138.5, 135.3, 134.5, 133.5, 131.5, 128.9, 128.7, 128.62, 128.59, 128.5, 127.5, 127.4, 124.7, 123.9, 123.6, 122.7, 119.7, 95.2, 85.5, 75.0, 62.1, 44.3, 44.2, 14.5.

HRMS (ESI+) Calcd for $C_{33}H_{27}ClNO_4$ $(M+H)^+$ requires m/z 536.1623 (Cl, 34.9686), found m/z 536.1621.



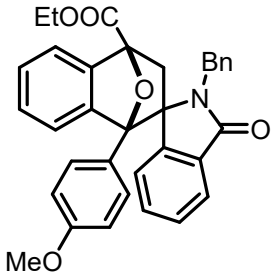
7c

Ethyl 2-benzyl-6-methoxy-3-oxo-1'-phenyl-1'*H*-spiro[isoindoline-1,2'-[1,4] epoxynaphthalene]-4'(3'*H*)-carboxylate (7c) was preparedas a white foam from 2-benzyl-5-methoxy-3-methyleneisoindolin-1-one **6c** (53 mg, 0.2 mmol) and ethyl 2-(2-benzoylphenyl)-2-diazoacetate **2a** (88.2 mg, 0.3 mmol) according to the General Procedure G (eluent: PE/EA = 10:1) in 64% yield (68 mg, 11:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.62 (d, *J* = 7.5 Hz, 1H), 7.41 – 7.35 (m, 4H), 7.33 – 7.29 (m, 3H), 7.26 – 7.17 (m, 4H), 7.07 (s, 2H), 6.91 (d, *J* = 7.6 Hz, 1H), 6.76 (dd, *J* = 8.5, 2.5 Hz, 1H), 5.19 (d, *J* = 8.5 Hz, 1H), 4.83 (d, *J* = 15.5 Hz, 1H), 4.62 (s, 1H), 4.50 – 4.40 (m, 2H), 3.87 (s, 3H), 2.79 (d, *J* = 13.0 Hz, 1H), 2.19 (d, *J* = 13.0 Hz, 1H), 1.43 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 168.7, 168.1, 160.6, 145.7, 144.3, 138.9, 137.5, 134.2, 133.9, 128.7, 128.54, 128.50, 128.4, 127.4, 127.2, 124.8, 123.6, 122.7, 119.6, 119.4, 106.2, 95.1, 85.4, 74.7, 62.0, 55.8, 44.3, 44.2, 14.5.

HRMS (ESI+) Calcd for $C_{34}H_{30}NO_5$ $(M+H)^+$ requires m/z 532.2119, found m/z 532.2128.



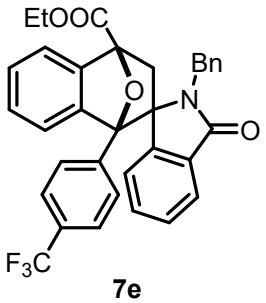
7d

Ethyl 2-benzyl-1'-(4-methoxyphenyl)-3-oxo-1'*H*-spiro[isoindoline-1,2'-[1,4] epoxynaphthalene]-4'(3'*H*)-carboxylate (7d) was preparedas a white foam from 2-benzyl-3-methyleneisoindolin-1-one **6a** (47 mg, 0.2 mmol) and ethyl 2-diazo -2-(2-(4-methoxybenzoyl)phenyl)acetate **2c** (97.2 mg, 0.3 mmol) according to the General Procedure G (eluent: PE/EA = 10:1) in 34% yield (36 mg, >20:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.93 (d, *J* = 7.5 Hz, 1H), 7.68 (d, *J* = 7.4 Hz, 1H), 7.53 – 7.42 (m, 4H), 7.35 – 7.22 (m, 6H), 7.00 (d, *J* = 8.5 Hz, 2H), 6.93 (d, *J* = 7.4 Hz, 1H), 6.89 (d, *J* = 9.0 Hz, 2H), 5.36 (d, *J* = 7.8 Hz, 1H), 4.94 (d, *J* = 15.5 Hz, 1H), 4.67 (d, *J* = 15.5 Hz, 1H), 4.57 – 4.46 (m, 2H), 3.84 (s, 3H), 2.86 (d, *J* = 13.0 Hz, 1H), 2.27 (d, *J* = 13.0 Hz, 1H), 1.49 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (150 MHz, Chloroform-*d*) δ 168.8, 168.1, 159.5, 145.7, 145.5, 144.5, 138.9, 132.7, 131.3, 128.9, 128.54, 128.47, 127.4, 127.2, 126.0, 125.9, 123.5, 122.8, 122.7, 119.5, 114.2, 95.2, 85.5, 75.1, 62.0, 55.3, 44.5, 44.1, 14.5.

HRMS (ESI+) Calcd for $C_{34}H_{30}NO_5$ $(M+H)^+$ requires m/z 532.2119, found m/z 532.2126.



7e

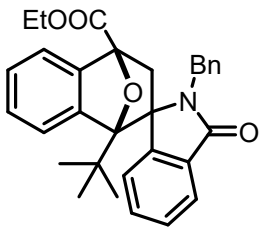
Ethyl 2-benzyl-3-oxo-1'-(4-(trifluoromethyl)phenyl)-1'*H*-spiro[isoindoline-1,2'-[1,4]epoxynaphthalene]-4'(3'*H*)-carboxylate (7e) was preparedas a white foam from 2-benzyl-3-methyleneisoindolin-1-one **6a** (47 mg, 0.2 mmol) and ethyl 2-diazo-2-(2-(4-(trifluoromethyl)benzoyl)phenyl)acetate **2h** (108.6 mg, 0.3 mmol) according to the General Procedure G (eluent: PE/EA = 10:1) in 53% yield (60.1 mg, >20:1 dr).

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.89 (d, *J* = 7.5 Hz, 1H), 7.66 (d, *J* = 7.5 Hz, 1H), 7.54 (d, *J* = 8.4 Hz, 2H), 7.47 (t, *J* = 7.4 Hz, 1H), 7.43 (t, *J* = 7.4 Hz, 1H), 7.35 (d, *J* = 6.9 Hz, 2H), 7.26 – 7.18 (m, 5H), 7.10 (s, 2H), 6.82 (d, *J* = 7.5 Hz, 1H), 5.33 (d, *J* = 7.8 Hz, 1H), 4.84 (d, *J* = 15.4 Hz, 1H), 4.59 (d, *J* = 15.4 Hz, 1H), 4.54 – 4.41 (m, 2H), 2.85 (d, *J* = 13.1 Hz, 1H), 2.25 (d, *J* = 13.1 Hz, 1H), 1.45 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (150 MHz, Chloroform-*d*) δ 168.7, 167.8, 145.5, 145.1, 143.6, 138.5, 137.9, 132.6, 131.6, 129.2, 128.9, 128.6, 128.5, 127.5, 127.4, δ 125.8 (q, *J* = 4.0 Hz), 125.3, 123.7, 122.7, 122.6, 119.8, 94.8, 85.6, 75.0, 62.2, 44.6, 44.2, 29.8, 14.5.

^{19}F NMR (565 MHz, Chloroform-*d*) δ -62.65.

HRMS (ESI+) Calcd for $C_{34}H_{27}F_3NO_4$ $(M+H)^+$ requires m/z 570.1887, found m/z 570.1889.



7f

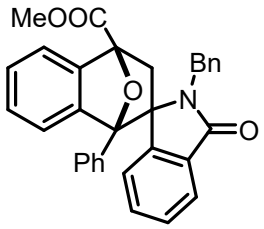
Ethyl 2-benzyl-1'-(tert-butyl)-3-oxo-1'*H*-spiro[isoindoline-1,2'-[1,4] epoxynaphthalene]-4'(3'*H*)-carboxylate (7f) was preparedas a white foam from 2-

benzyl-3-methyleneisoindolin-1-one **6a** (47 mg, 0.2 mmol) and ethyl 2- diazo-2-(2-pivaloylphenyl)acetate **2l** (82.2 mg, 0.3 mmol) according to the General Procedure G (eluent: PE/EA = 10:1) in 58% yield (55.6 mg, 14:1 dr).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.83 (t, *J* = 6.9 Hz, 2H), 7.61 – 7.54 (m, 2H), 7.52 – 7.40 (m, 3H), 7.29 (d, *J* = 6.5 Hz, 1H), 7.15 – 7.09 (m, 3H), 6.77 – 6.70 (m, 2H), 4.47 (d, *J* = 16.4 Hz, 1H), 3.97 (dq, *J* = 10.6, 7.1 Hz, 1H), 3.85 (dq, *J* = 10.7, 7.1 Hz, 1H), 2.66 (d, *J* = 13.0 Hz, 1H), 2.51 (d, *J* = 16.5 Hz, 1H), 1.87 (d, *J* = 13.0 Hz, 1H), 1.31 (s, 9H), 0.93 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (150 MHz, Chloroform-*d*) δ 169.8, 165.8, 148.3, 147.1, 141.7, 138.3, 132.3, 131.4, 128.9, 128.7, 128.4, 127.2, 126.9, 126.7, 123.7, 123.2, 122.5, 121.3, 94.1, 89.9, 74.1, 61.5, 43.8, 37.7, 34.0, 26.2, 14.0.

HRMS (ESI+) Calcd for $C_{31}H_{32}NO_4$ $(M+H)^+$ requires m/z 482.2326, found m/z 482.2321.



7g

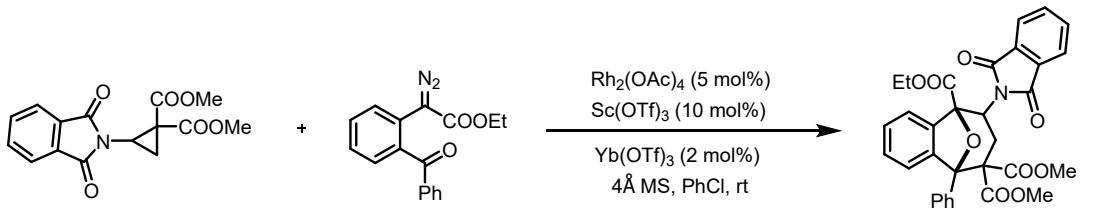
Methyl 2-benzyl-3-oxo-1'-phenyl-1'*H*-spiro[isoindoline-1,2'-[1,4]epoxynaphthalene]-4'(3'*H*)-carboxylate (7g) was preparedas a white foam from 2-benzyl-3-methyleneisoindolin-1-one **6a** (47 mg, 0.2 mmol) and methyl 2-(2-benzoylphenyl)-2-diazoacetate **2y** (84.1 mg, 0.3 mmol) according to the General Procedure G (eluent: PE/EA = 10:1) in 56% yield (54.6 mg, >20:1 dr).

^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.87 (d, *J* = 7.5 Hz, 1H), 7.63 (d, *J* = 7.5 Hz, 1H), 7.45 (t, *J* = 7.5 Hz, 1H), 7.42 – 7.37 (m, 3H), 7.33 – 7.29 (m, 3H), 7.26 – 7.19 (m, 5H), 7.02 (s, 2H), 6.89 (d, *J* = 7.5 Hz, 1H), 5.30 (d, *J* = 7.8 Hz, 1H), 4.86 (d, *J* = 15.5 Hz, 1H), 4.59 (d, *J* = 15.5 Hz, 1H), 3.99 (s, 3H), 2.80 (d, *J* = 13.0 Hz, 1H), 2.22 (d, *J* = 13.0 Hz, 1H).

^{13}C NMR (150 MHz, Chloroform-*d*) δ 168.7, 168.5, 145.6, 145.4, 144.1, 138.8, 133.7, 132.8, 131.4, 128.9, 128.8, 128.6, 128.52, 128.46, 127.4, 127.3, 124.7, 123.5, 122.9, 122.6, 119.6, 95.4, 85.5, 75.0, 52.8, 44.5, 44.0.

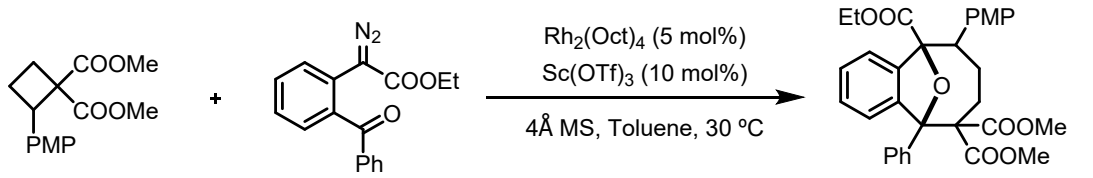
HRMS (ESI+) Calcd for $C_{32}H_{26}NO_4$ $(M+H)^+$ requires m/z 488.1857, found m/z 488.1853.

5. Scale-up reactions and synthetic applications



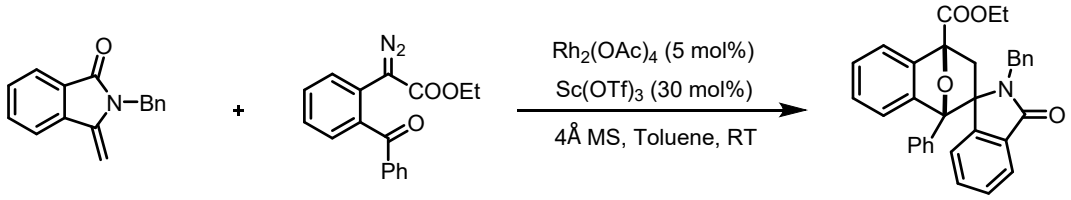
1a **2a** **3a**, 1.01 g, 88%, >20:1 dr

A solution of **1a** (2 mmol), $Sc(OTf)_3$ (4.9 mg, 0.2 mmol) , $Yb(OTf)_3$ (1.2 mg, 0.04 mmol), 4 Å MS (200 mg) and $Rh_2(OAc)_4$ (2.2 mg, 0.1 mmol) were dissolved in PhCl (10 mL). A solution of diazo compound **2a** (3 mmol) in PhCl (10 mL) was then added to the reaction mixture for a period of 1 h by using syringe pump and stirred at the indicated temperature in a closed vessel for 3 h (monitored by TLC). After reaction finished, the reaction solution was evaporated under reduced pressure and purified by column chromatography (PE/EA = 3:1) to afford the pure product **3a** (1.01 g, 88%) as a white foam.



4a **2a** **5a**, 391 mg, 72%, >20:1 dr

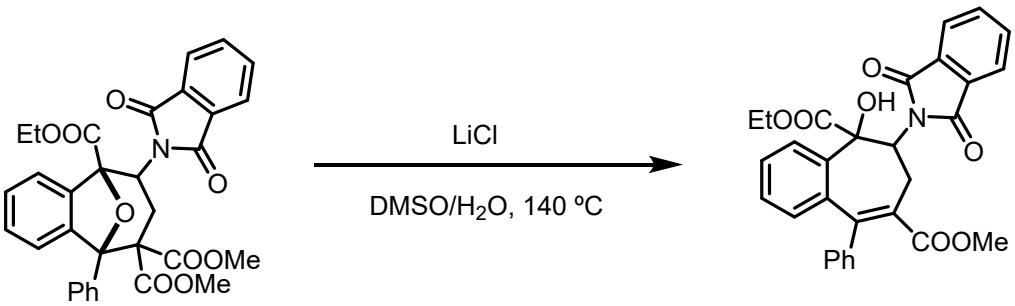
At 30°C, a solution of **4a** (1 mmol), $Sc(OTf)_3$ (4.9 mg, 0.1 mmol), 4 Å MS (100 mg) and $Rh_2(Oct)_4$ (3.9 mg, 0.05 mmol) were dissolved in toluene (5 mL). A solution of diazo compound **2a** (1.5 mmol) in toluene (5 mL) was then added to the reaction mixture for a period of 1 h by using syringe pump and stirred at the indicated temperature in a closed vessel for 36 h (monitored by TLC). After reaction finished, the reaction solution was evaporated under reduced pressure and purified by column chromatography (PE/EA = 10:1) to afford the pure product **5a** (391 mg, 72%) as a white foam.



6a **2a** **7a**, 750 mg, 68%, >20:1 dr

A solution of **6a** (2 mmol), $Sc(OTf)_3$ (14.8 mg, 0.2 mmol), 4 Å MS (200 mg) and $Rh_2(OAc)_4$ (2.2 mg, 0.1 mmol) were dissolved in toluene (10 mL). A solution of diazo compound **2a** (3 mmol) in toluene (10 mL) was then added to the reaction mixture for a period of 1 h by using syringe pump and stirred at the indicated temperature in a closed vessel for 24 h(monitored by TLC). After reaction finished, the reaction solution was evaporated under reduced pressure and purified by column chromatography (PE/EA = 10:1) to afford the pure product **7a** (750 g, 68%) as a white foam.

Synthesis of compounds 8, 9 and 10:



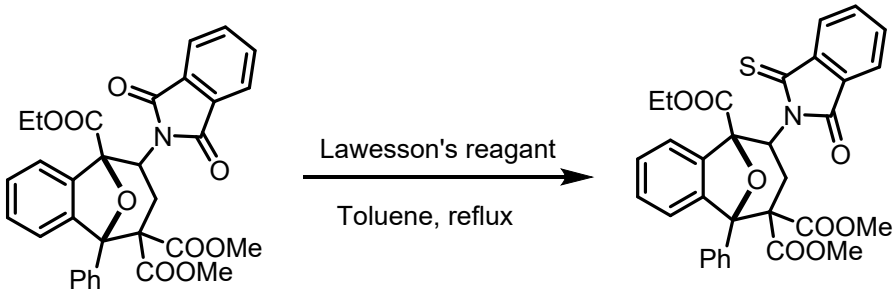
3a **8**, 72%, >20:1 dr

LiCl (21.2 mg, 0.5 mmol) was added to a solution of **3a** (50 mg, 0.1 mmol) in DMSO:H_2O 10:1 (0.5 mL, 0.2 M) at room temperature and the resulting mixture was then heated for 5 hours to 140 °C. The mixture was cooled to room temperature, quenched with sat. aq. NH_4Cl (5 mL) and extracted with DCM (3×10 mL). The combined org. extracts were washed with brine (10 mL) and dried over $MgSO_4$. The drying agent was filtered off and the solvent was evaporated. The residue was purified by column chromatography (PE/EA = 5:1) to afford the pure product **8** (36.5 mg, 72%) as a white foam.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.82 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.75 – 7.68 (m, 3H), 7.40 (td, *J* = 7.7, 1.4 Hz, 1H), 7.34 – 7.28 (m, 4H), 7.16 – 7.09 (m, 2H), 6.87 (dd, *J* = 7.7, 1.2 Hz, 1H), 6.03 (dd, *J* = 12.8, 5.8 Hz, 1H), 4.16 (qd, *J* = 7.2, 2.6 Hz, 2H), 3.56 (t, *J* = 12.8 Hz, 1H), 3.51 (s, 3H), 3.24 (s, 1H), 2.94 (dd, *J* = 12.8, 5.8 Hz, 1H), 1.23 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 172.6, 169.4, 168.8, 150.7, 141.0, 139.5, 138.7, 134.2, 131.9, 131.2, 129.8, 128.8, 128.3, 128.1, 127.9, 126.7, 125.6, 123.6, 63.2, 61.8, 51.8, 29.1, 13.9.

HRMS (ESI+) Calcd for $C_{30}H_{26}NO_7$ $(M+H)^+$ requires m/z 512.1704, found m/z 512.1698.



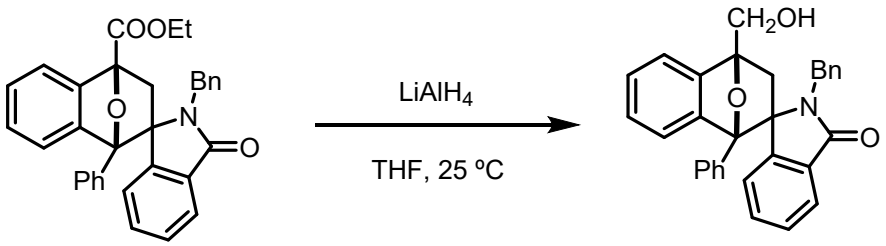
3a **9**, 63%, >20:1 dr

A mixture of compound **3a** (56.9 mg, 0.1 mmol) and Lawesson's reagant (80.8 mg, 0.2 mmol) in toluene was stirred in an oil bath at 110 °C for 6 h. The solvent was then evaporated, and the residue was purified by column chromatography (PE/EA = 5:1) to afford the pure product **9** (37.8 mg, 63%) as a red foam.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.97 (dd, *J* = 5.8, 2.7 Hz, 1H), 7.87 (d, *J* = 7.5 Hz, 2H), 7.74 (dd, *J* = 5.7, 2.8 Hz, 1H), 7.71 – 7.65 (m, 2H), 7.45 (dd, *J* = 5.8, 2.7 Hz, 1H), 7.40 (t, *J* = 7.7 Hz, 2H), 7.34 – 7.28 (m, 4H), 5.39 (dd, *J* = 6.4, 3.1 Hz, 1H), 4.27 (qd, *J* = 7.0, 1.1 Hz, 2H), 3.67 (s, 3H), 3.22 (s, 3H), 3.12 (dd, *J* = 14.9, 3.2 Hz, 1H), 2.14 (dd, *J* = 14.9, 6.5 Hz, 1H), 1.24 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 197.9, 170.4, 169.2, 169.1, 168.4, 143.0, 142.5, 141.1, 137.0, 134.1, 133.4, 129.0, 128.8, 128.1, 127.8, 127.3, 125.7, 125.4, 124.1, 122.8, 119.6, 89.5, 86.3, 62.0, 61.6, 52.8, 52.4, 51.4, 33.5, 14.1.

HRMS (ESI+) Calcd for $C_{32}H_{28}NO_8S$ $(M+H)^+$ requires m/z 586.153, found m/z 586.1525.



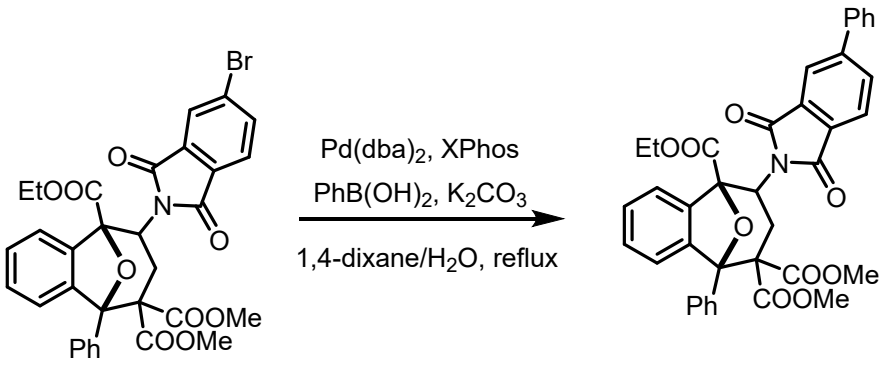
7a **10**, 56%, >20:1 dr

7a (50.1 mg, 0.1 mmol) was dissolved in abs. THF (2 mL, 0.05M) in a sealed tube. The solution was cooled to 0 °C and $LiAlH_4$ (5.4 mg, 0.6 mmol) was added. The solution was warmed to room temperature and stirred for 2 h at the same temperature. Upon completion of the reaction, aq. NH_4Cl solution (5 mL) was added and the mixture was extracted with EtOAc (3×5 mL). The organic layers were dried over Na_2SO_4, filtered, and concentrated in vacuo. The residue was purified by column chromatography (PE/EA = 5:1) to afford the pure product **10** (26 mg, 56%) as a white foam.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.88 (d, *J* = 7.5 Hz, 1H), 7.47 – 7.36 (m, 3H), 7.33 – 7.26 (m, 6H), 7.26 – 7.16 (m, 4H), 6.97 (d, *J* = 7.5 Hz, 2H), 6.87 (d, *J* = 7.5 Hz, 1H), 5.29 (d, *J* = 7.8 Hz, 1H), 4.85 (d, *J* = 15.8 Hz, 1H), 4.55 (d, *J* = 15.8 Hz, 1H), 4.34 (dd, *J* = 12.4, 3.7 Hz, 1H), 4.24 (d, *J* = 12.4 Hz, 1H), 2.61 (d, *J* = 12.9 Hz, 1H), 2.16 (s, 1H), 1.89 (d, *J* = 12.9 Hz, 1H).

^{13}C NMR (100 MHz, Chloroform-*d*) δ 169.0, 146.5, 146.2, 145.8, 138.9, 134.5, 132.6, 131.3, 128.7, 128.7, 128.4, 128.3, 127.9, 127.3, 126.9, 124.8, 123.4, 122.7, 122.7, 119.0, 95.1, 88.5, 61.7, 44.1, 42.3.

HRMS (ESI+) Calcd for $C_{31}H_{26}NO_3$ $(M+H)^+$ requires m/z 460.1947, found m/z 460.1911.



3d **3aa**, 86%, 10:1 dr

3d (64.7 mg, 0.1 mmol), phenylboronic acid (36.6 mg, 0.3 mmol), $Pd_2(dba)_3$ (4.6 mg, 0.015 mmol, 5 mol%), XPhos (4.8 mg, 0.02 mmol, 20 mol%) and K_2CO_3 (33.0 mg 0.2 mmol) were dissolved in a solvent mixture of degassed 1,4-dioxane/H_2O (1:1, v/v, 2 mL, 0.05 M) in a sealed tube under Aratmosphere. The reaction was stirred at 110°C for 5 h and then cooled to room temperature. Upon completion of the reaction, aq. NH_4Cl solution (5 mL) was added and the mixture was extracted with EtOAc (3 ×5 mL). The organic layers were dried over Na_2SO_4, filtered, and concentrated in vacuo. The residue was purified by column chromatography (PE/EA = 3:1) to afford the pure product **3aa** (55.1 mg, 86%) as a white foam.

^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.05 (s, 1H), 7.90 (q, *J* = 3.4 Hz, 2H), 7.84 (d, *J* = 7.9 Hz, 2H), 7.64 (d, *J* = 7.5 Hz, 2H), 7.52 – 7.44 (m, 4H), 7.39 (t, *J* = 7.7 Hz, 2H), 7.35 – 7.29 (m, 4H), 4.76 (d, *J* = 4.5 Hz, 1H), 4.31 (q, *J* = 7.1 Hz, 2H), 3.69 (s, 3H), 3.22 (s, 3H), 3.05 (d, *J* = 15.1 Hz, 1H), 2.11 (dd, *J* = 15.2, 6.2 Hz, 1H), 1.29 (t, *J* = 7.1 Hz, 3H).

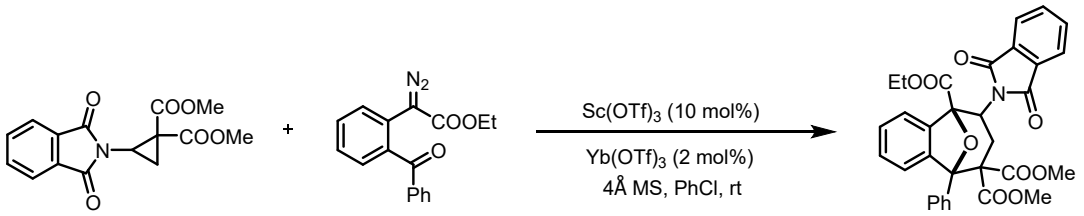
^{13}C NMR (150 MHz, Chloroform-*d*) δ 170.5, 169.2, 168.6, 147.5, 143.0, 142.0, 141.2, 139.4, 132.7, 129.3, 129.0, 128.89, 128.87, 128.1, 127.5, 127.3, 125.8, 125.4, 123.7, 121.8, 119.6, 89.7, 86.1, 62.0, 60.9, 52.9, 52.5, 48.8, 33.3, 14.2.

HRMS (ESI+) Calcd for $C_{38}H_{32}NO_9$ $(M+H)^+$ requires m/z 646.2072, found m/z 646.2070.

6. Control experiments

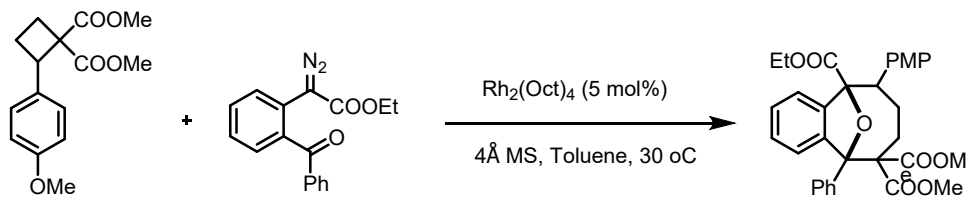
1a **2a** **3a**, ND

In a flame-dried 10 mL Schlenk tube equipped with a magnetic stir bar was charged sequentially with **1a** (0.1 mmol), 4 Å MS (60 mg) and $Rh_2(OAc)_4$ (2.2 mg, 0.005 mmol) were dissolved in PhCl (1 mL), A solution of diazo compound **2a** (0.15 mmol) in PhCl (1 mL) was then added to the reaction mixture for a period of 1 h by using syringe pump (monitored by TLC). Followed by the General Procedure E, no cyclized compound was detected.



1a **2a** **3a**, 53%, 8:1 dr

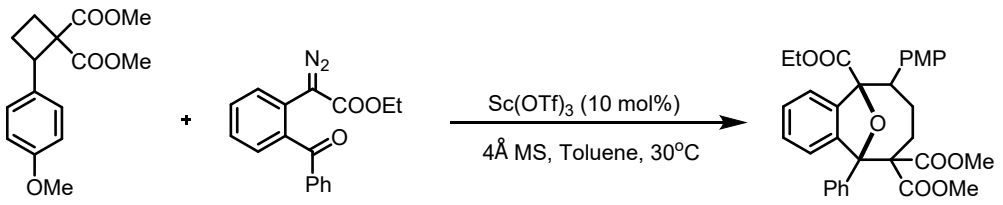
In a flame-dried 10 mL Schlenk tube equipped with a magnetic stir bar was charged sequentially with **1a** (0.1 mmol), $Sc(OTf)_3$ (4.9 mg, 0.01 mmol), $Yb(OTf)_3$ (1.2 mg, 0.002 mmol) and 4 Å MS (60 mg) were dissolved in PhCl (1 mL), A solution of diazo compound **2a** (0.15 mmol) in PhCl (1 mL) was then added to the reaction mixture for a period of 1 h by using syringe pump (monitored by TLC). Followed by the General Procedure E to afford the pure product **3a** in 53% yield (54.6 mg, 8:1 dr).



4a **2a** **5a,** ND

In a flame-dried 10 mL Schlenk tube equipped with a magnetic stir bar was charged

sequentially with **4a** (0.1 mmol), 4 Å MS (60 mg) and $Rh_2(Oct)_4$ (3.9 mg, 0.005 mmol) were dissolved in toluene (1 mL), A solution of diazo compound **2a** (0.15 mmol) in toluene (1 mL) was then added to the reaction mixture for a period of 1 h by using syringe pump (monitored by TLC). Followed by the General Procedure F, no cyclized compound was detected.

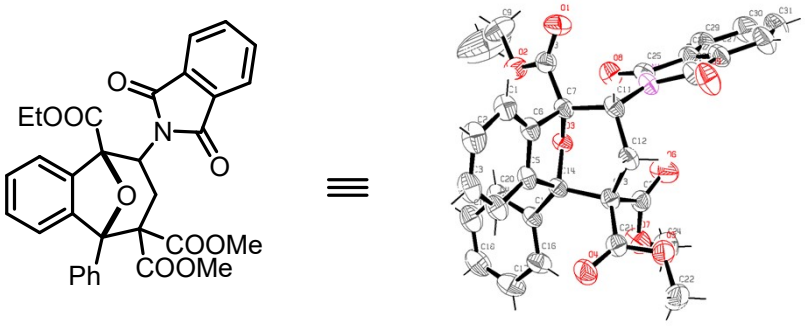


4a **2a** **5a,** ND

In a flame-dried 10 mL Schlenk tube equipped with a magnetic stir bar was charged sequentially with **4a** (0.1 mmol), 4 Å MS (60 mg) and $Sc(OTf)_3$ (4.9 mg, 0.01 mmol) were dissolved in toluene (1 mL), A solution of diazo compound **2a** (0.15 mmol) in toluene (1 mL) was then added to the reaction mixture for a period of 1 h by using syringe pump (monitored by TLC). Followed by the General Procedure F, no cyclized compound was detected.

7. Crystal data and structural refinement of compounds 3a, 5a and 7a

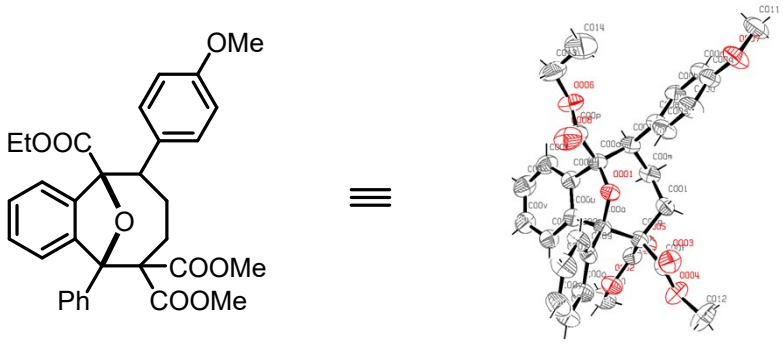
Single crystal of compound **3a, 5a** and **7a** was obtained by slow evaporation from CH_2Cl_2 solution.



3a

Table 1. Crystal data and structure refinement for 3a.

Identification code	**3a**	
CCDC	2381159	
Empirical formula	$C_{32}H_{27}NO_9$	
Formula weight	569.54	
Temperature	295.00 K	
Wavelength	1.54178 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 7.9838(4) Å	a= 74.965(3)°.
	b = 11.1916(6) Å	b= 77.534(3)°.
	c = 16.8046(9) Å	g =80.853(3)°.
Volume	1407.46(13) $Å^3$	
Z	2	
Density (calculated)	1.344 Mg/m^3	
Absorption coefficient	0.825 mm^{-1}	
F(000)	596	
Crystal size	0.3 x 0.2 x 0.1 mm^3	
Theta range for data collection	2.769 to 68.601°.	
Index ranges	-9<=h<=9, -13<=k<=13, -20<=l<=20	
Reflections collected	55636	
Independent reflections	5127 [R(int) = 0.0667]	
Completeness to theta = 67.679°	98.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7531 and 0.6203	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5127 / 0 / 383	
Goodness-of-fit on F^2	1.096	
Final R indices [I>2sigma(I)]	R1 = 0.0496, wR2 = 0.1129	
R indices (all data)	R1 = 0.0627, wR2 = 0.1165	
Extinction coefficient	0.0103(7)	
Largest diff. peak and hole	0.358 and -0.185 e.$Å^{-3}$	

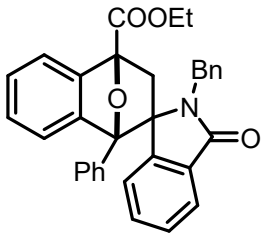


5a

Table 1. Crystal data and structure refinement for 5a.

Identification code	**5a**	
CCDC	2381160	
Empirical formula	$C_{32}H_{32}O_8$	
Formula weight	544.57	
Temperature	287.00 K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	P 1 21/c 1	
Unit cell dimensions	a = 8.3859(8) Å	a= 90°.
	b = 16.3267(14) Å	b= 93.005(4)°.
	c = 20.5864(19) Å	g = 90°.
Volume	2814.7(4) $Å^3$	
Z	4	
Density (calculated)	1.285 Mg/m^3	
Absorption coefficient	0.757 mm^{-1}	
F(000)	1152	
Crystal size	0.25 x 0.22 x 0.18 mm^3	
Theta range for data collection	3.457 to 68.271°.	
Index ranges	-10<=h<=9, -18<=k<=19, -24<=l<=24	
Reflections collected	50087	

Independent reflections	5063 [R(int) = 0.0887]
Completeness to theta = 67.679°	98.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7531 and 0.5527
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5063 / 0 / 366
Goodness-of-fit on F^2	1.061
Final R indices [I>2sigma(I)]	R1 = 0.0516, wR2 = 0.1343
R indices (all data)	R1 = 0.0606, wR2 = 0.1423
Extinction coefficient	0.0034(4)
Largest diff. peak and hole	0.270 and -0.205 e.Å^{-3}



7a

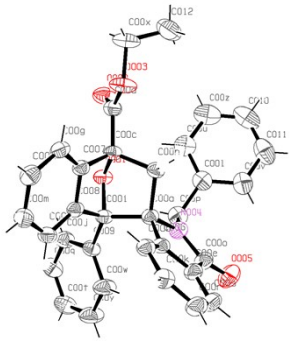


Table 1. Crystal data and structure refinement for 7a.

Identification code	**7a**	
CCDC	2381161	
Empirical formula	$C_{33}H_{27}NO_4$	
Formula weight	501.55	
Temperature	180.00 K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	P 1 21/c 1	
Unit cell dimensions	a = 12.3025(5) Å	a= 90°.
	b = 16.4509(7) Å	b= 114.328(2)°.
	c = 13.7775(6) Å	g = 90°.

Volume	2540.78(19) $Å^3$
Z	4
Density (calculated)	1.311 Mg/m^3
Absorption coefficient	0.689 mm^{-1}
F(000)	1056
Crystal size	0.18 x 0.15 x 0.12 mm^3
Theta range for data collection	3.943 to 68.564°.
Index ranges	-14<=h<=14, -19<=k<=19, -16<=l<=16
Reflections collected	55829
Independent reflections	4615 [R(int) = 0.0599]
Completeness to theta = 67.679°	99.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7531 and 0.6159
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4615 / 0 / 344
Goodness-of-fit on F^2	1.167
Final R indices [I>2sigma(I)]	R1 = 0.0555, wR2 = 0.1113
R indices (all data)	R1 = 0.0622, wR2 = 0.1143
Extinction coefficient	n/a
Largest diff. peak and hole	0.204 and -0.283 $e.Å^{-3}$

8. References

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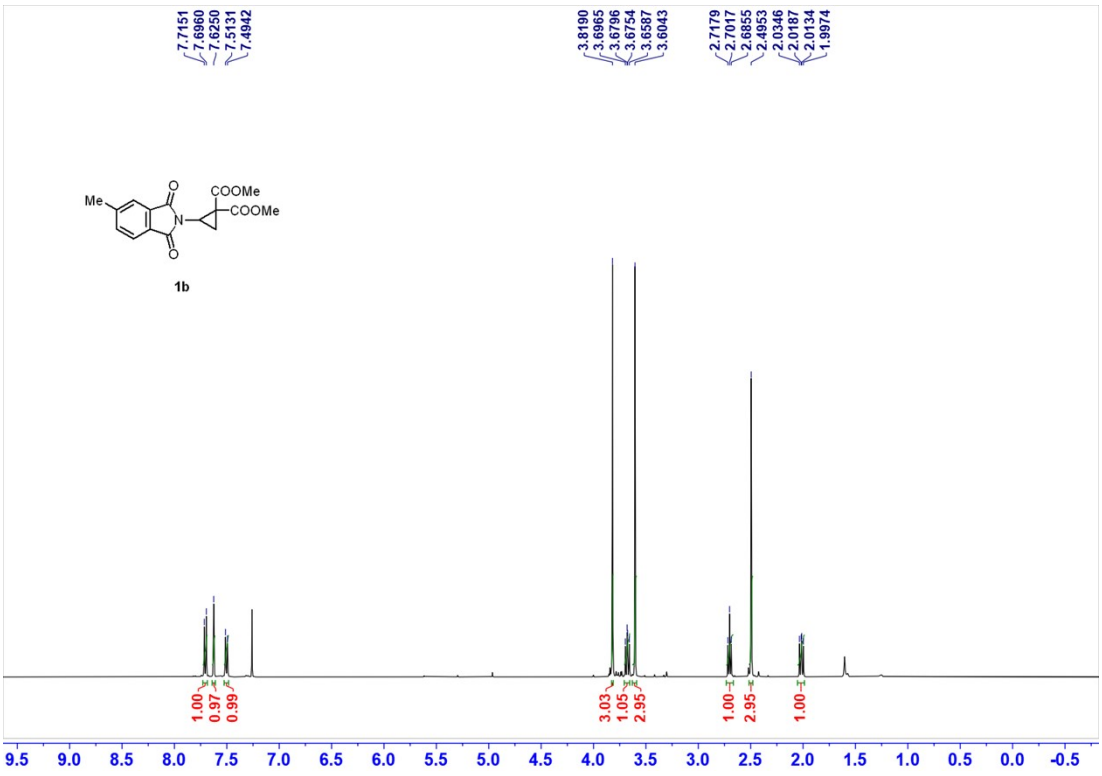
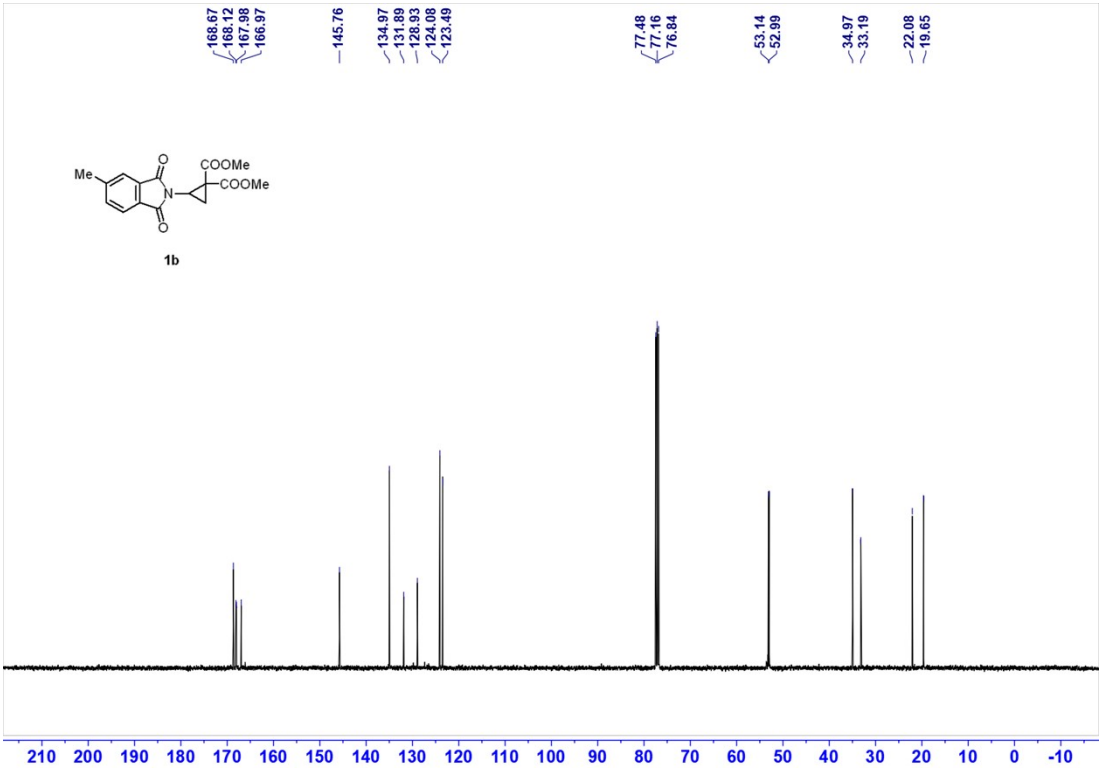
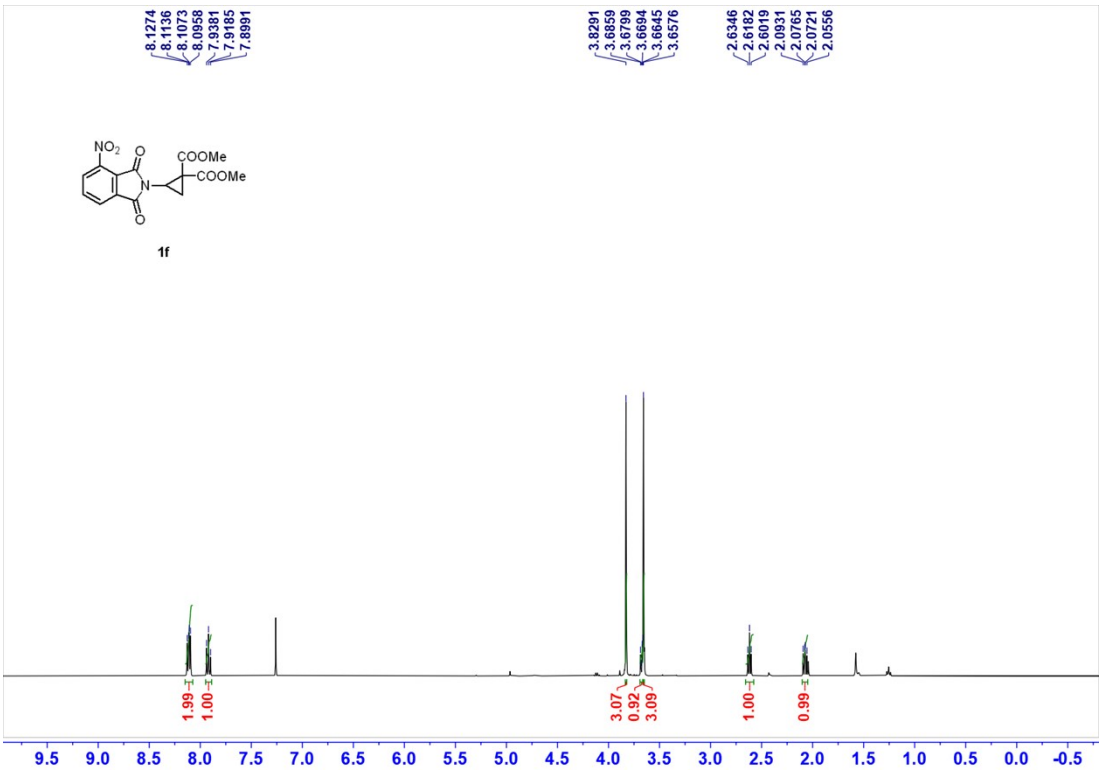
3. A. Suneja, H. J. Loui, and C. Schneider, Cooperative Catalysis for the Highly Diastereo-and Enantioselective [4+3]-Cycloannulation of ortho-Quinone Methides and Carbonyl Ylides, *Angew. Chem. Int. Ed.,* 2020, **59**, 5536–5540.

4. H. Luo, J. Yan, Z. Chen, Y. Wei, B. Chen, Y. Liu,. A Practical Method for the Synthesis of Donor-Acceptor Cyclobutanes by $Cu(OAc)_2$ or $FeCl_3$ Catalyzed [2+2] Cycloaddition, *ChemistrySelect* 2020, **5**, 4074.

9. Copies of the NMR Spectra



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COOMe
N
O
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1.00
3.07
0.92
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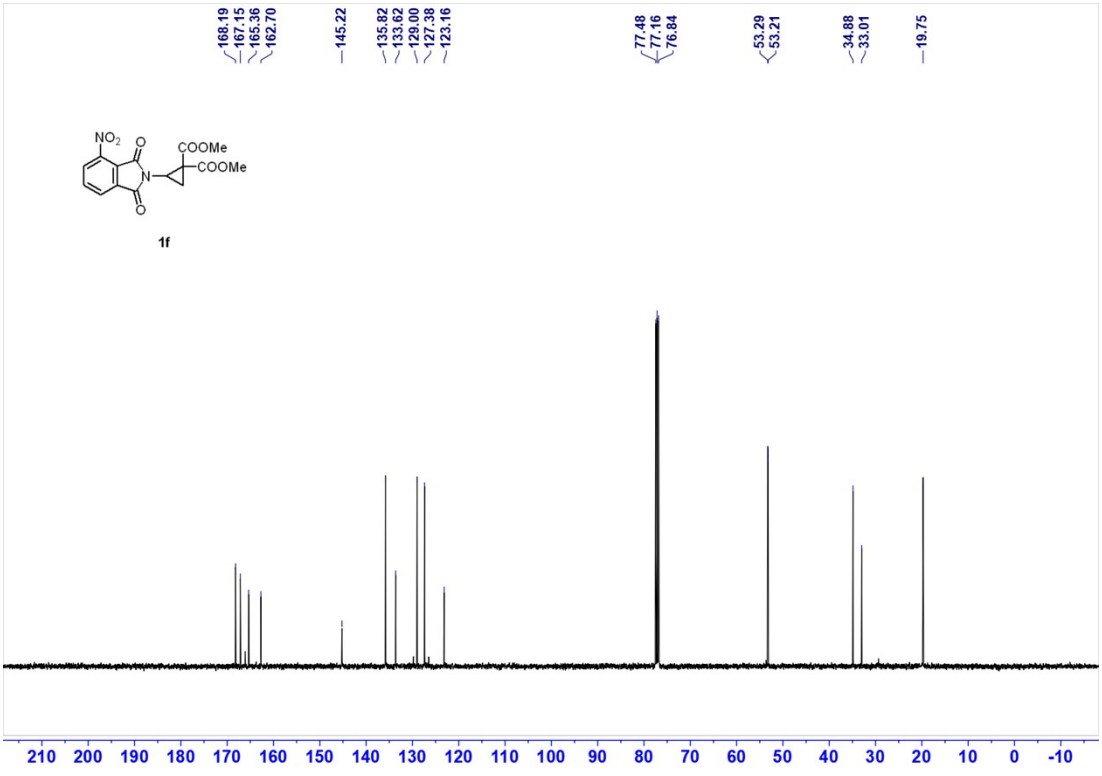
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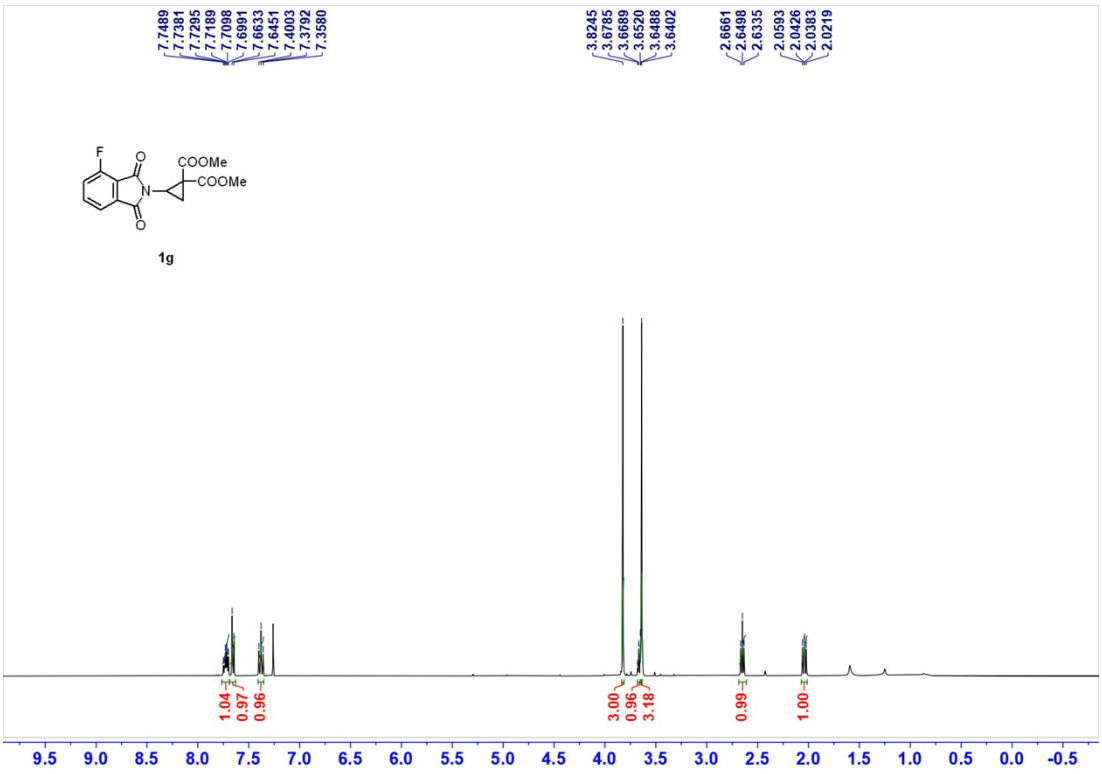
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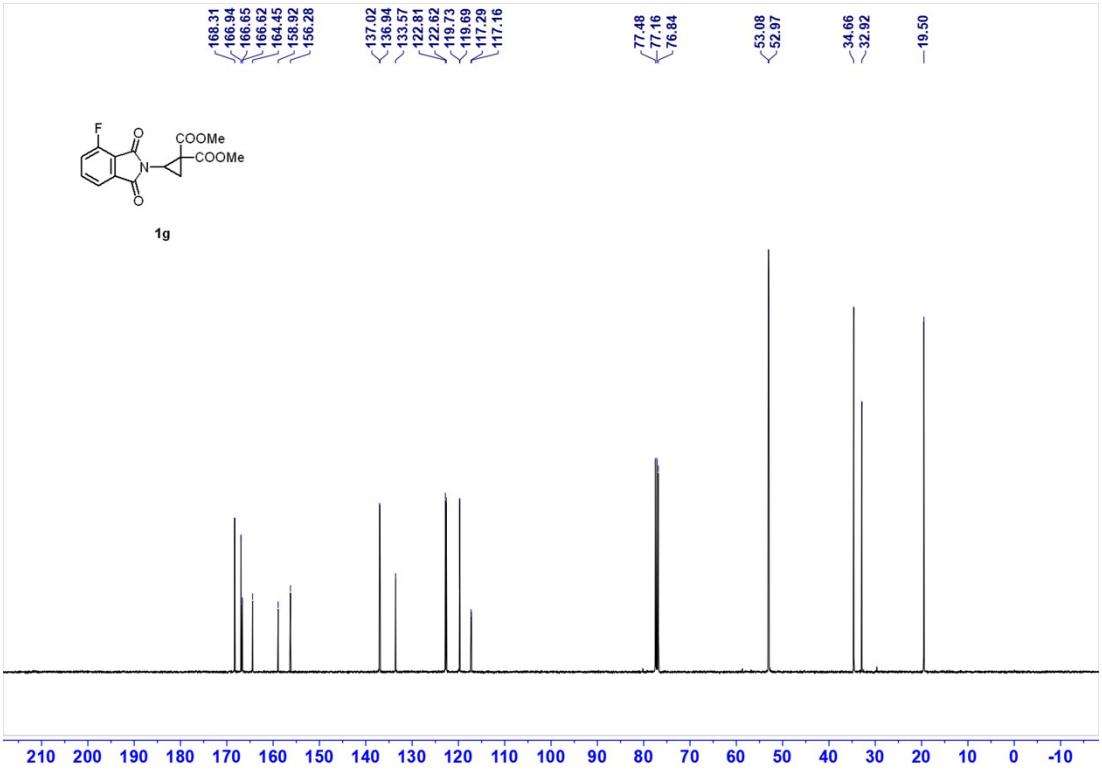
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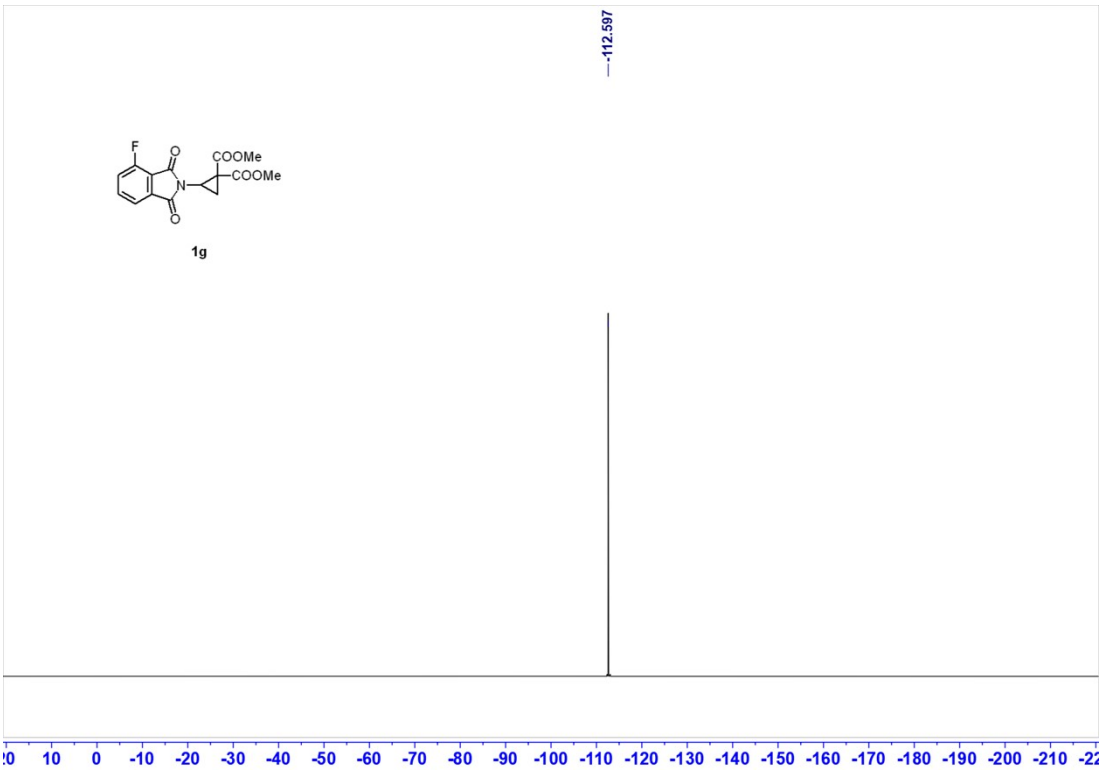
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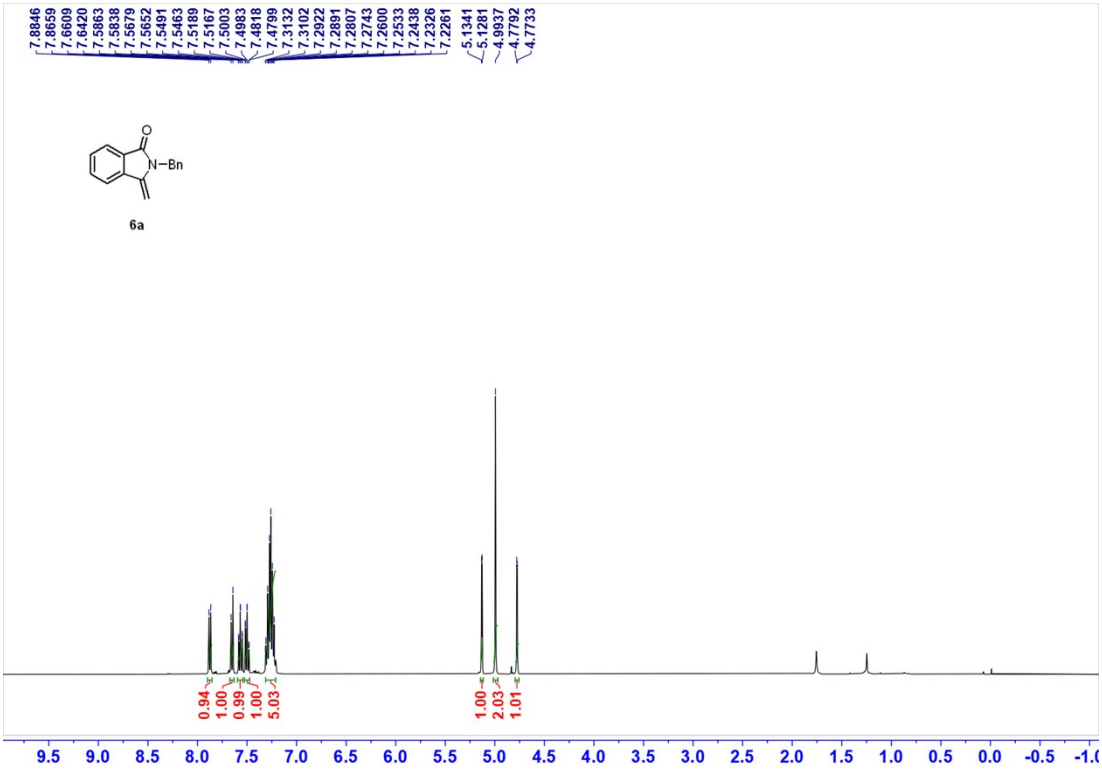
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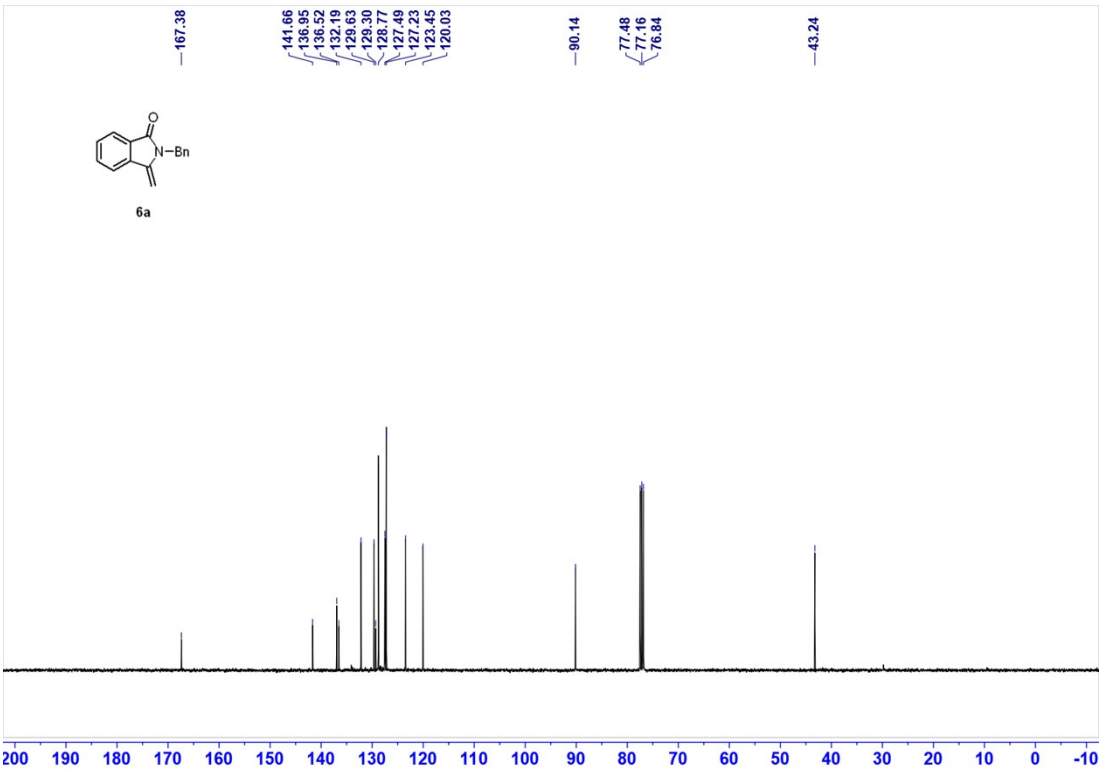
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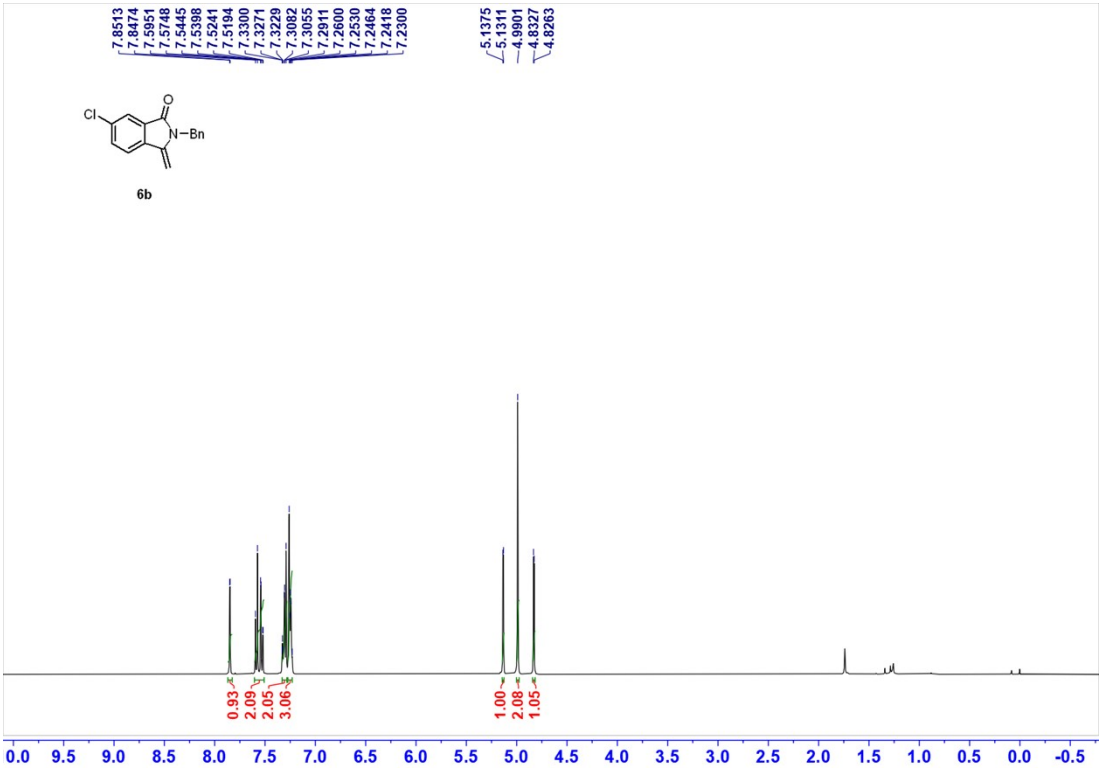
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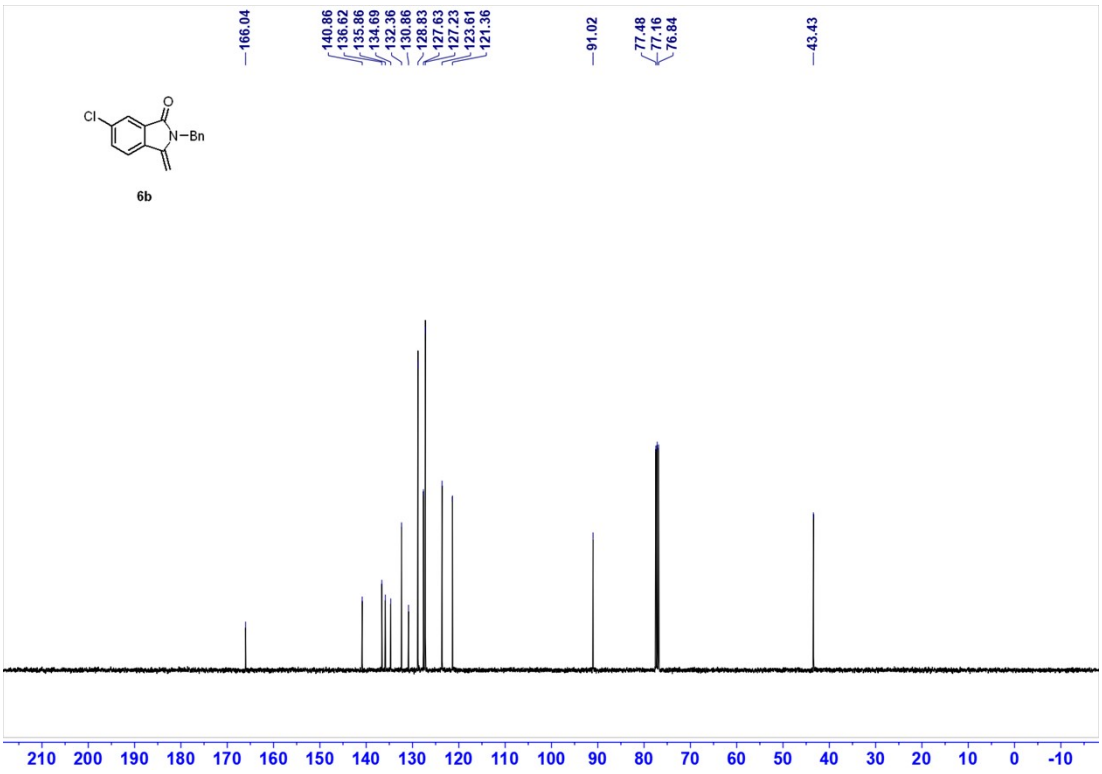
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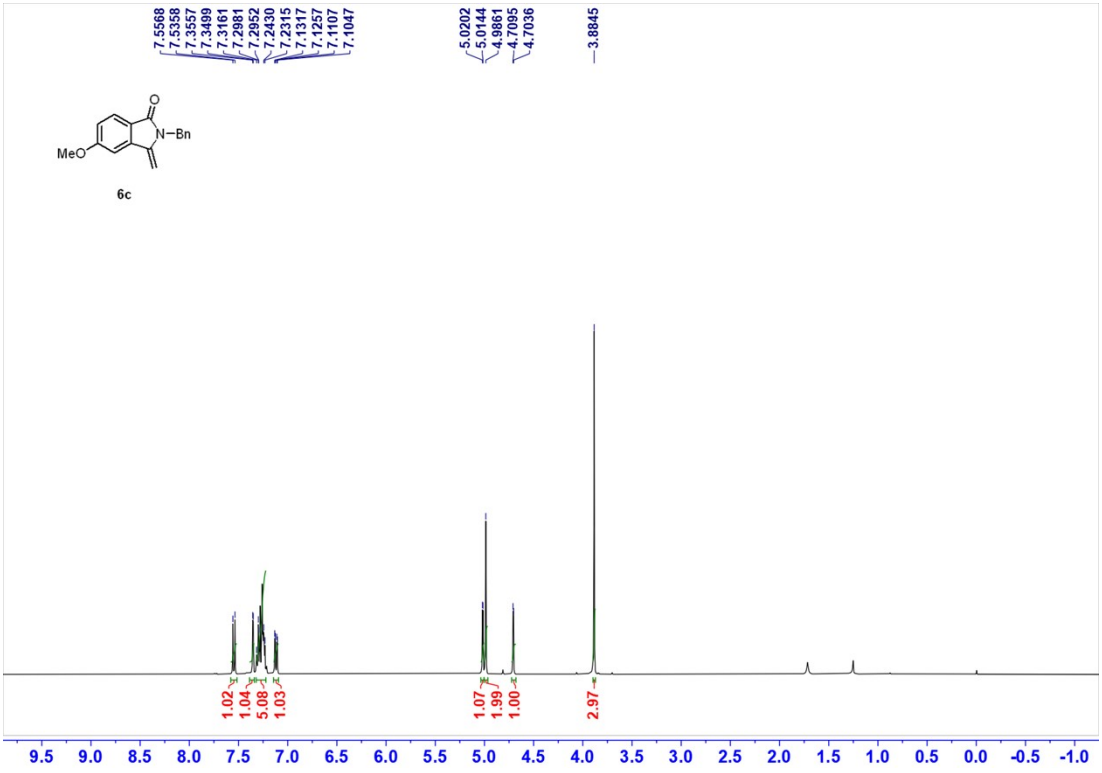
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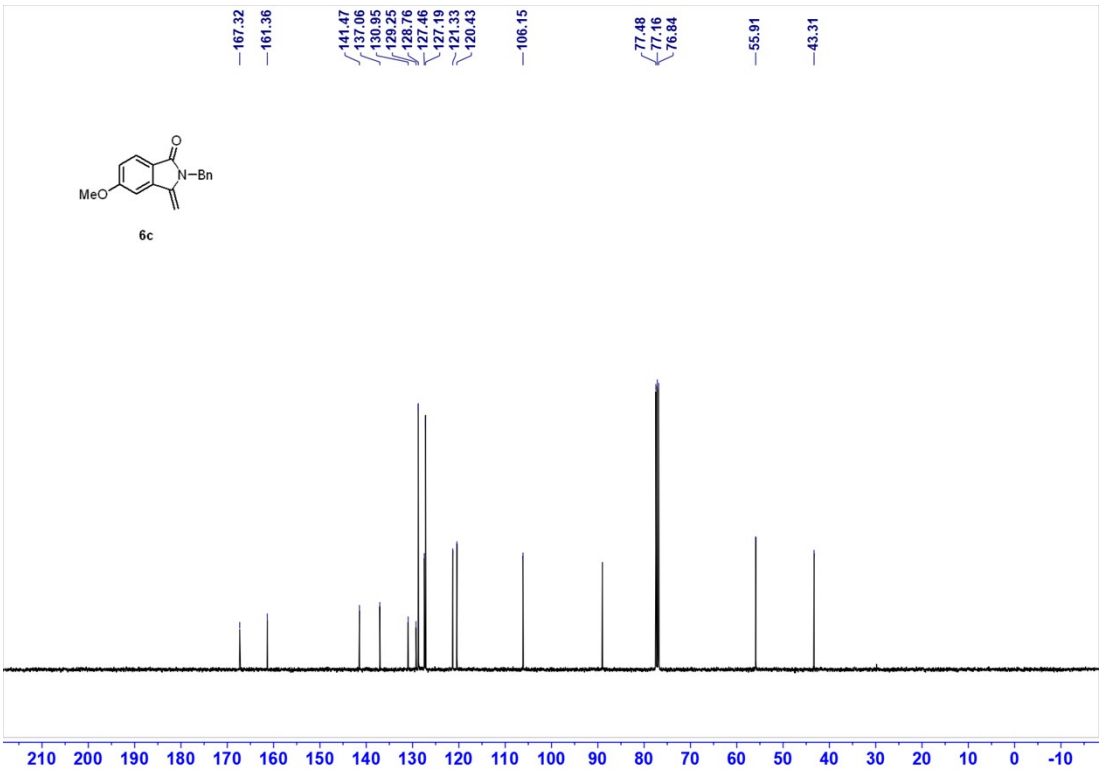
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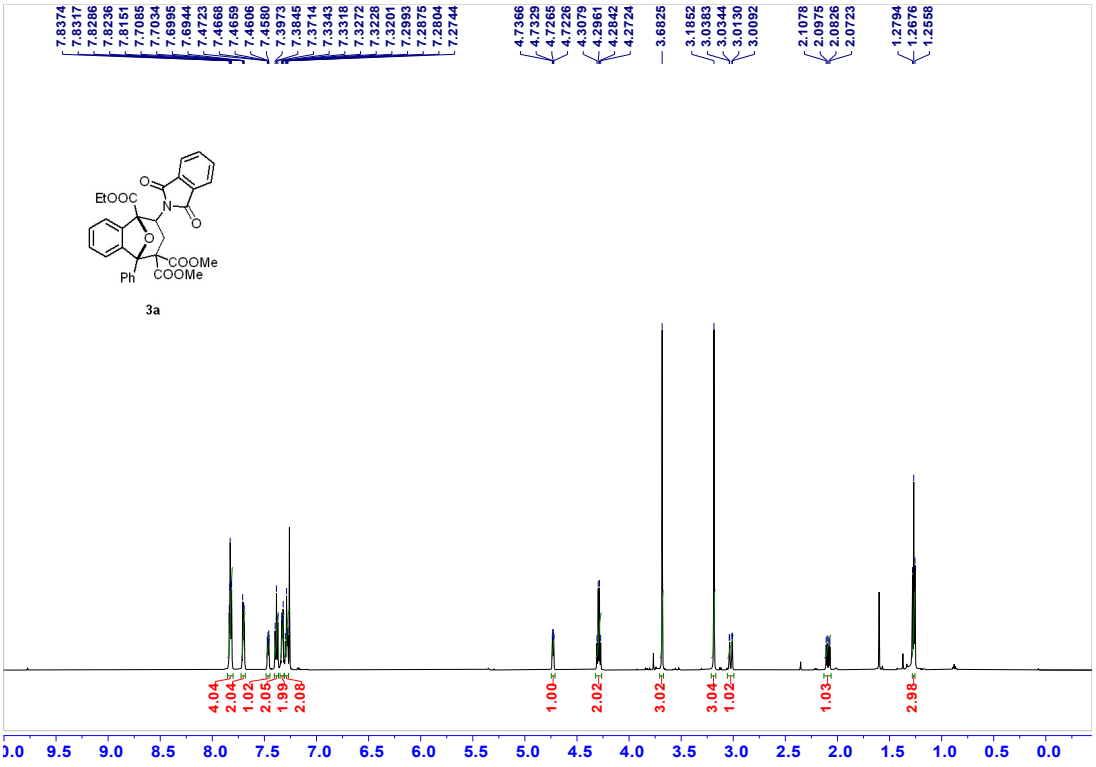
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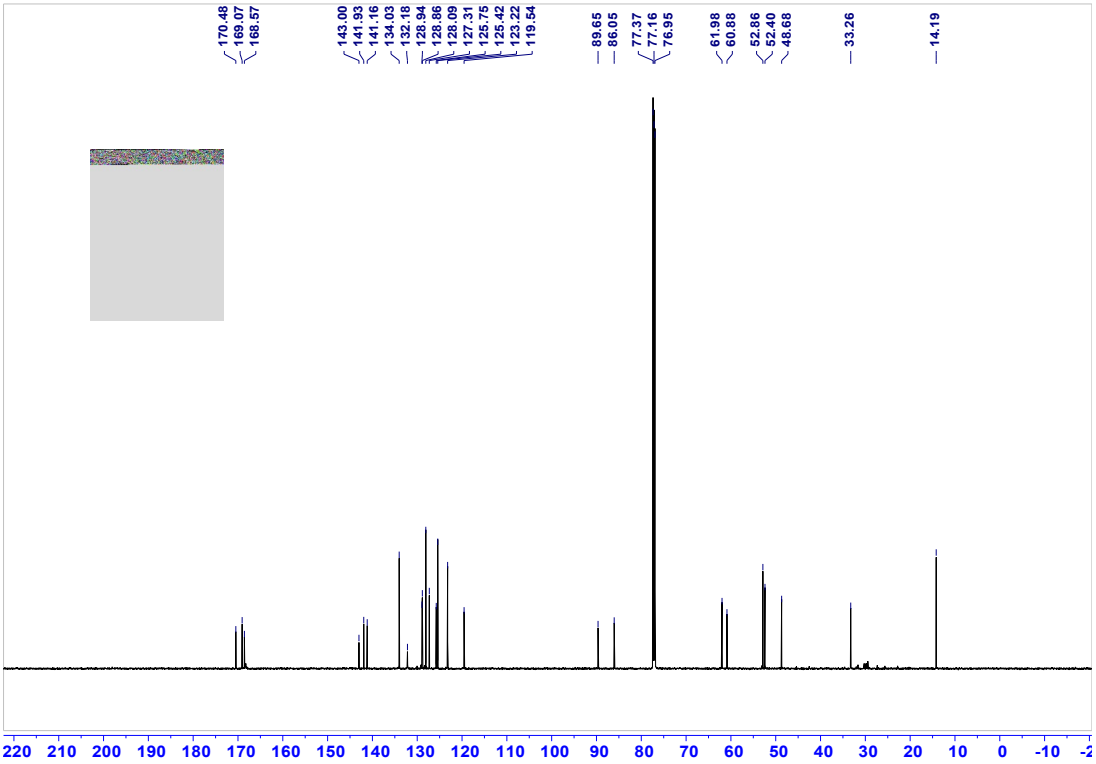
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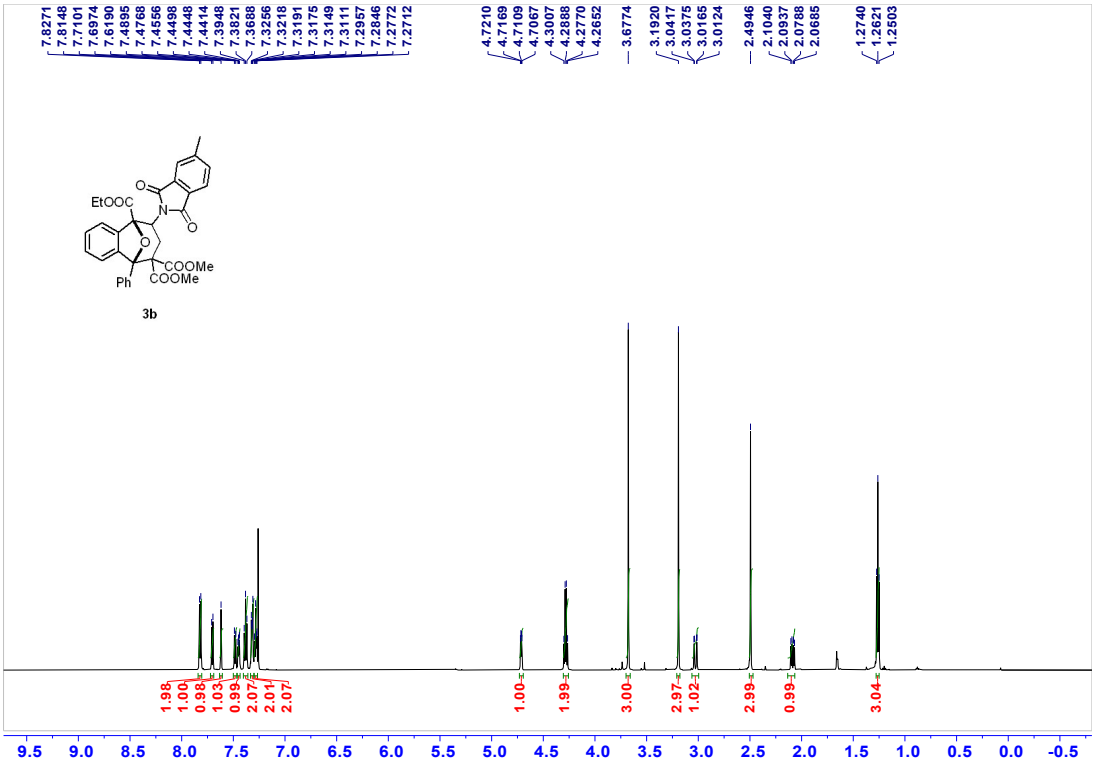
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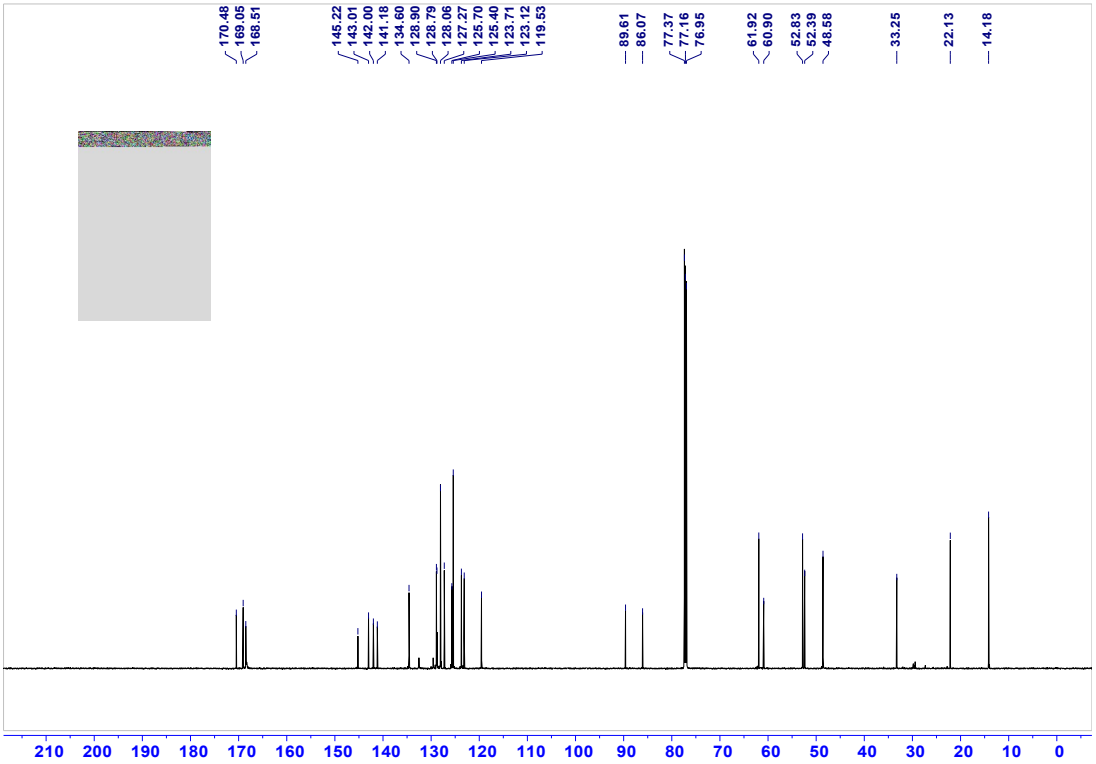
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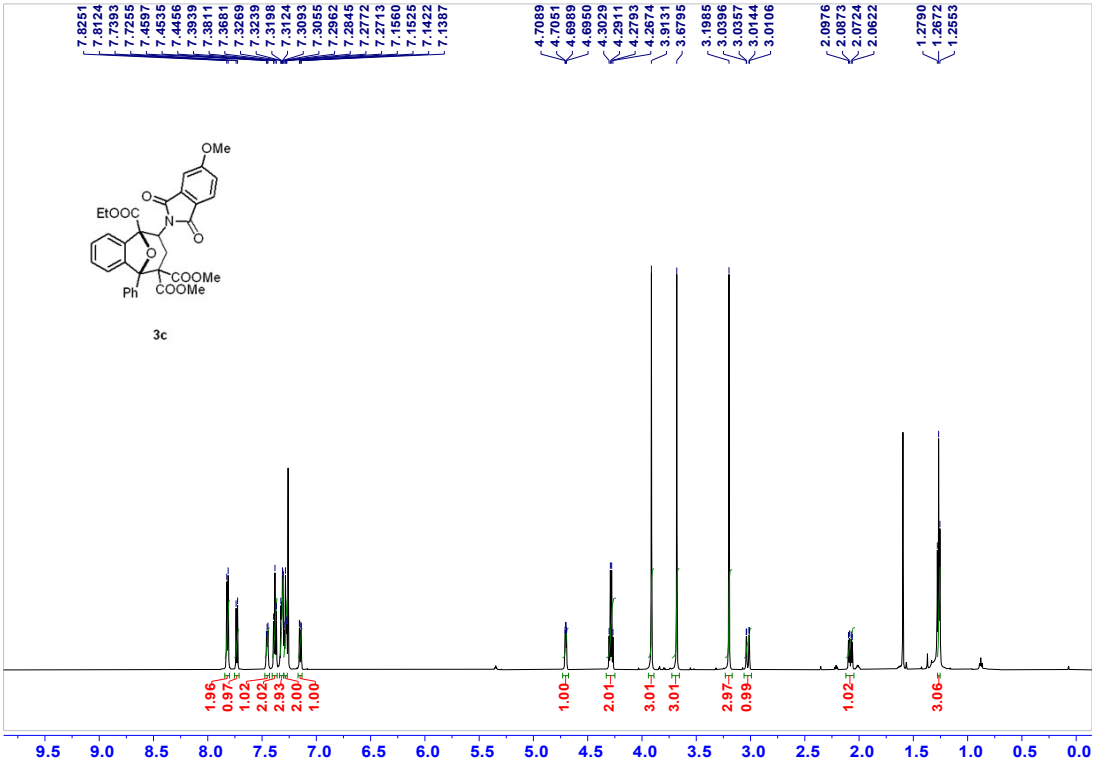
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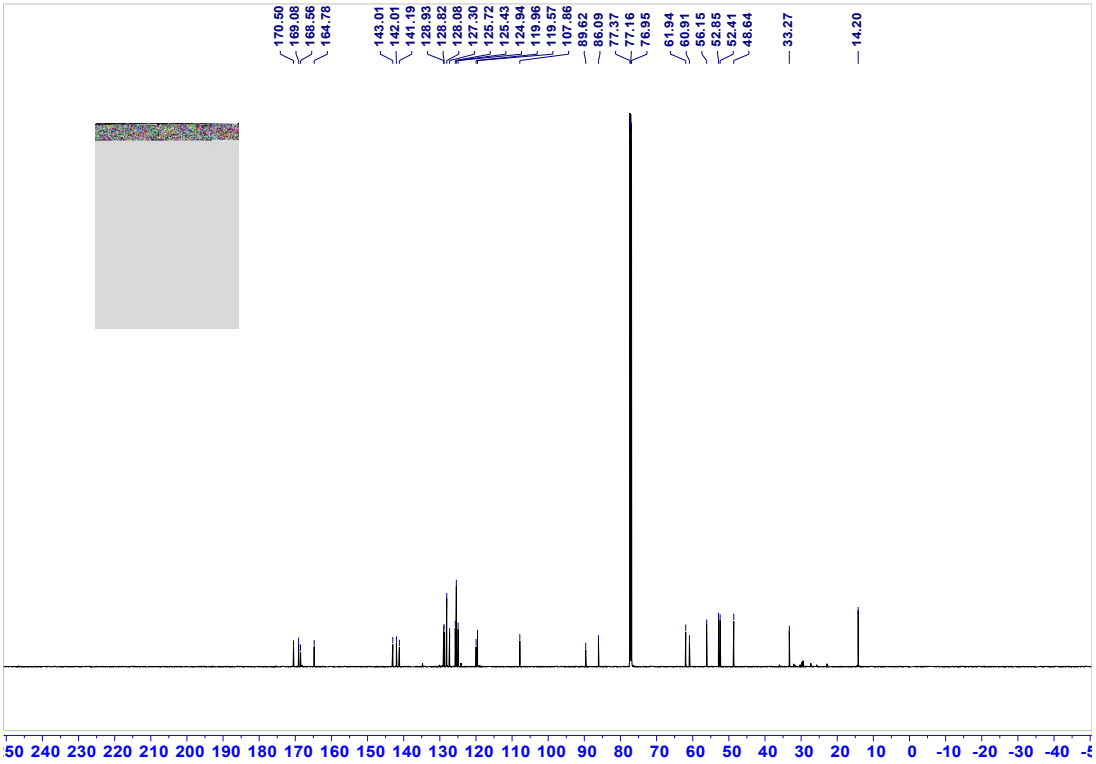
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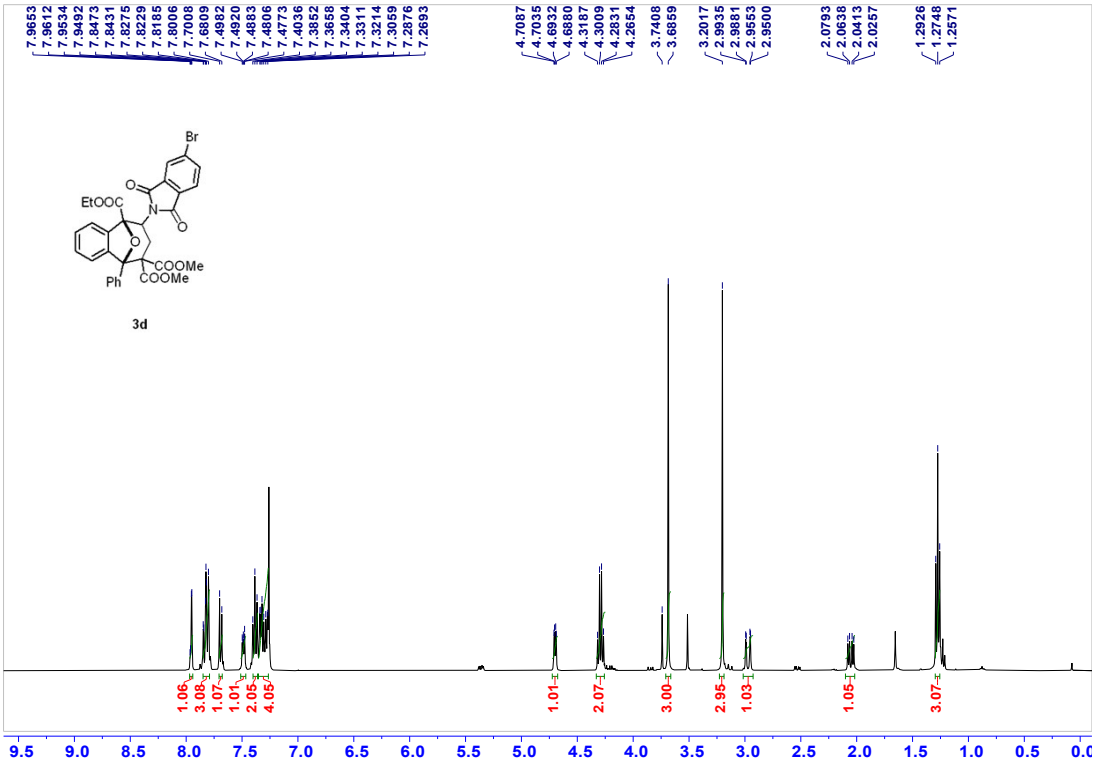
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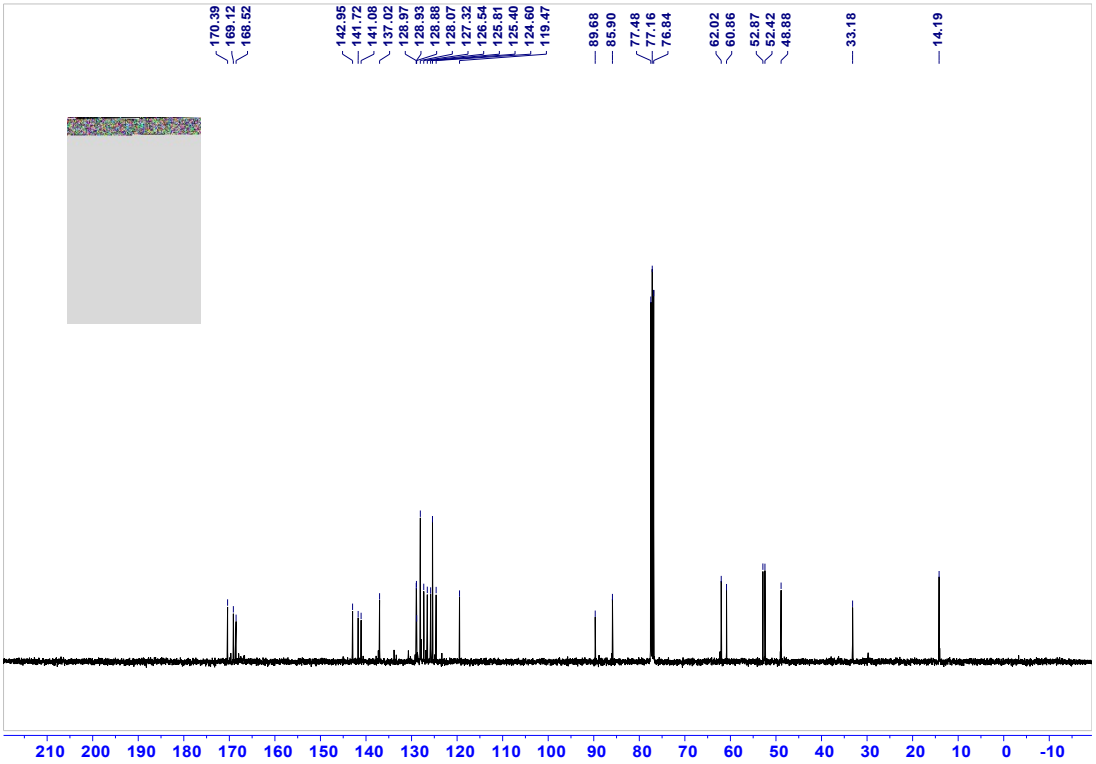
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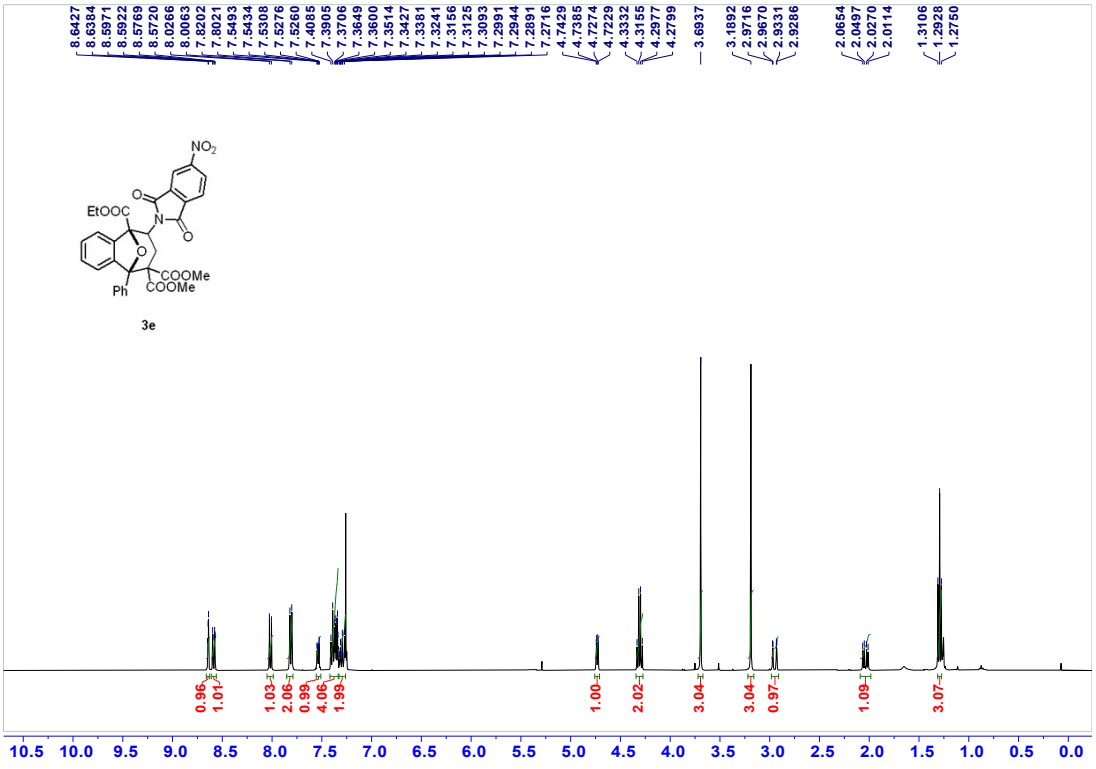
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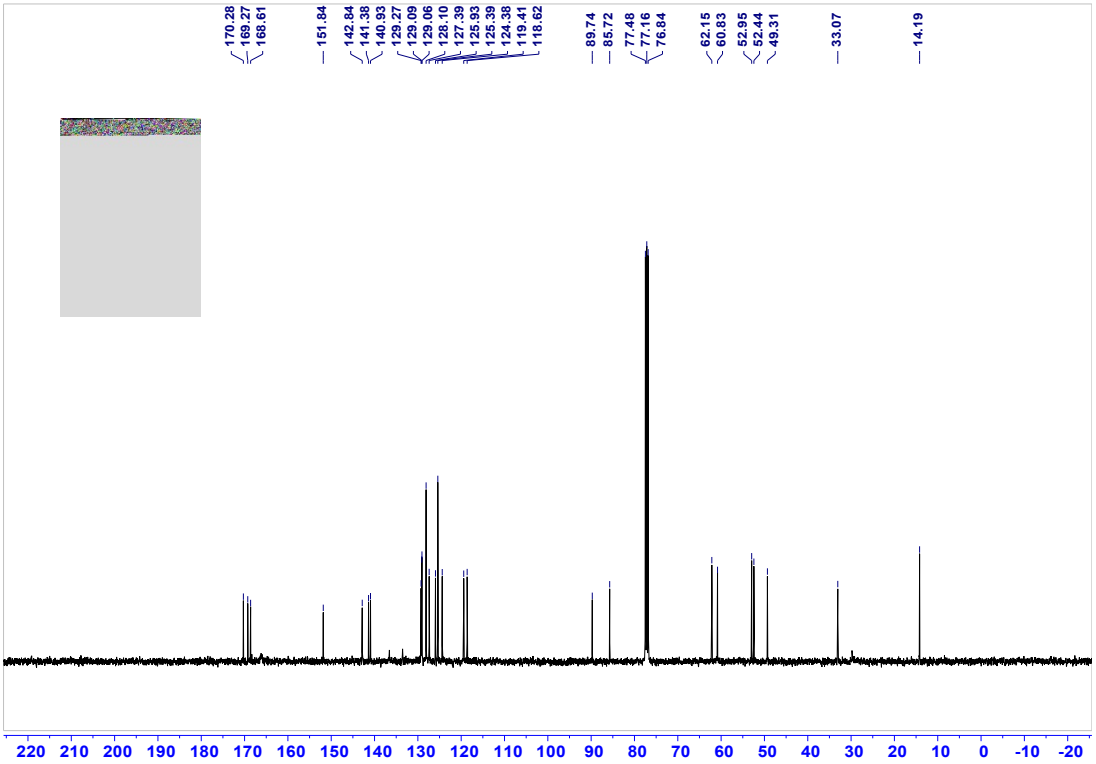
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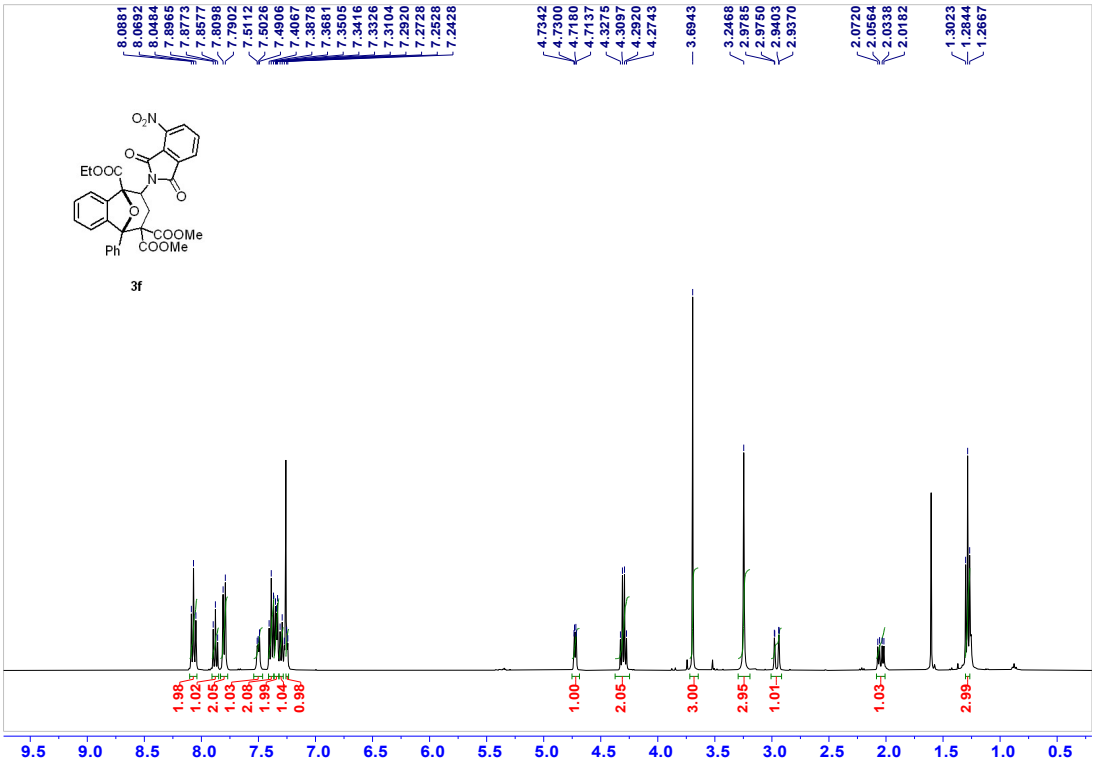
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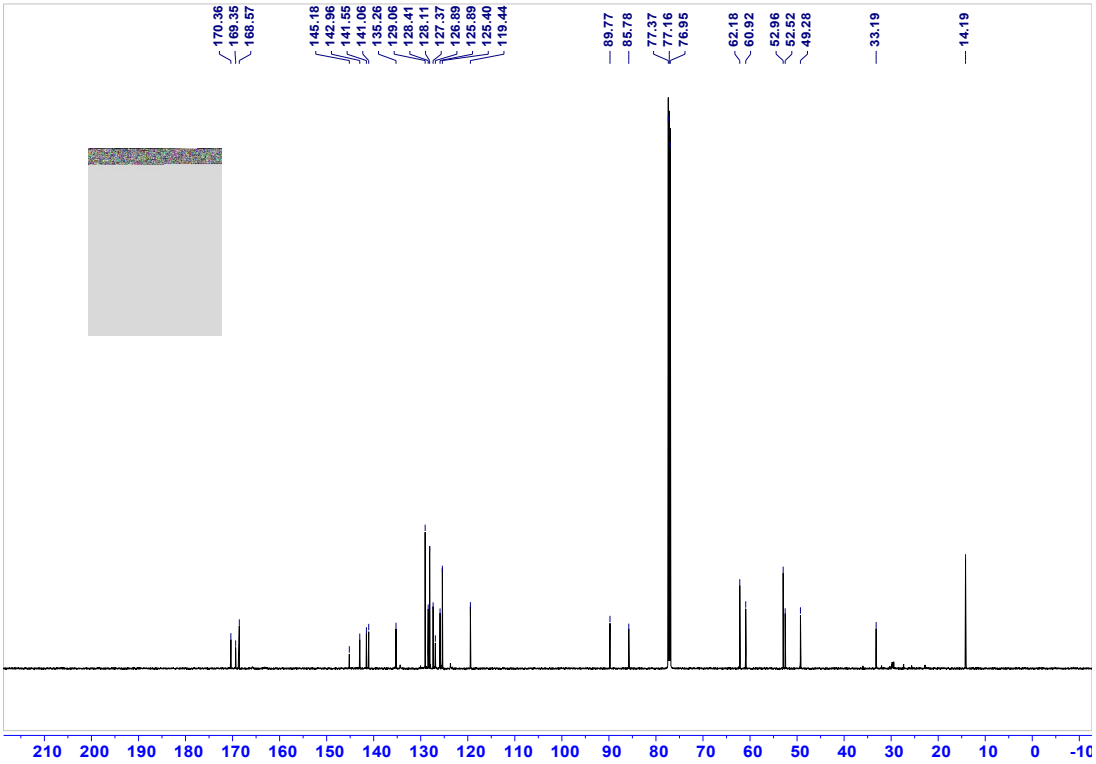
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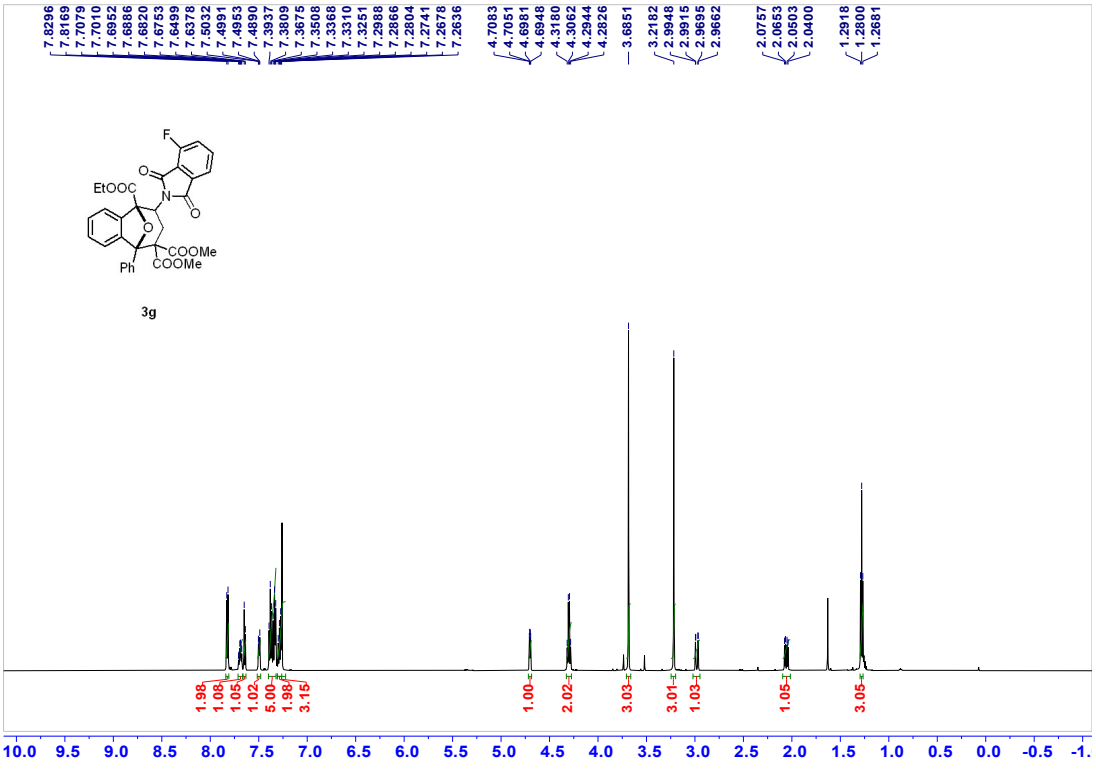
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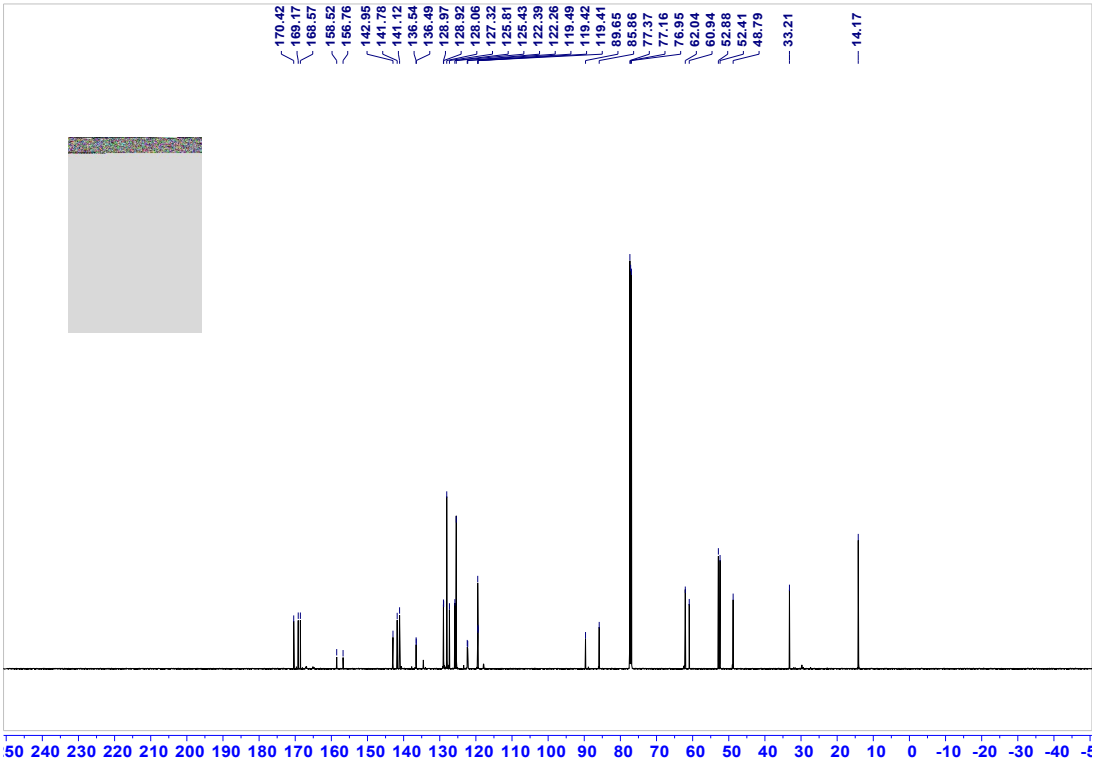
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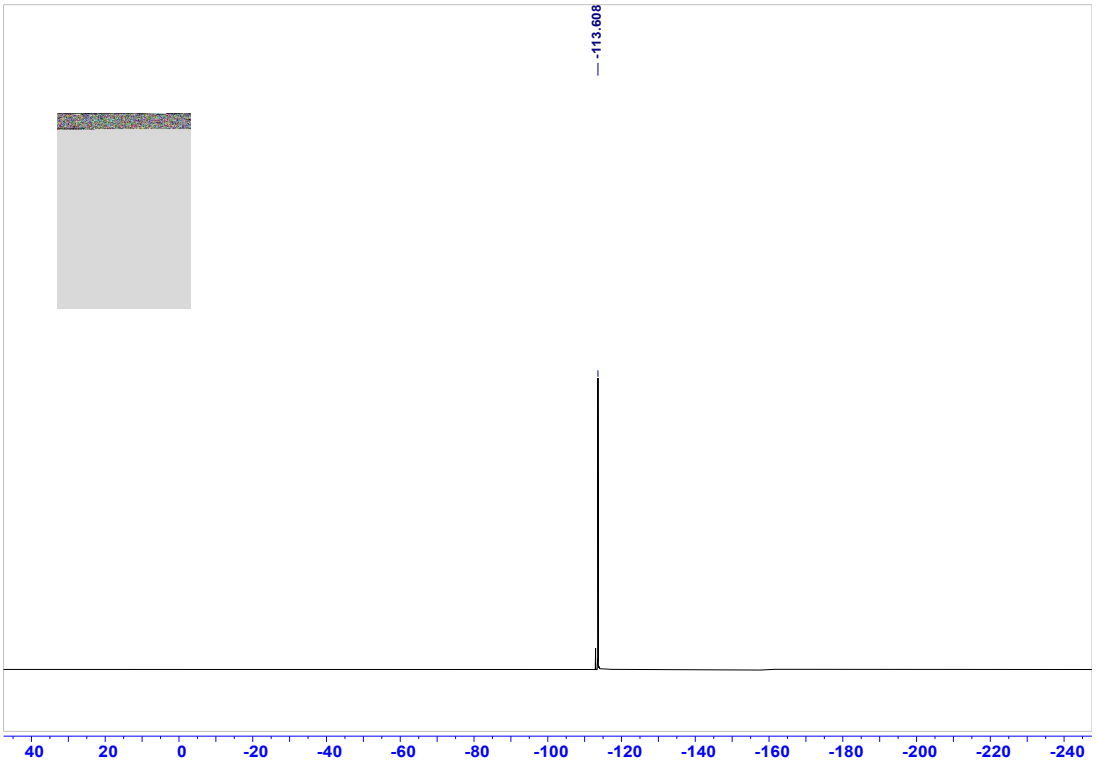
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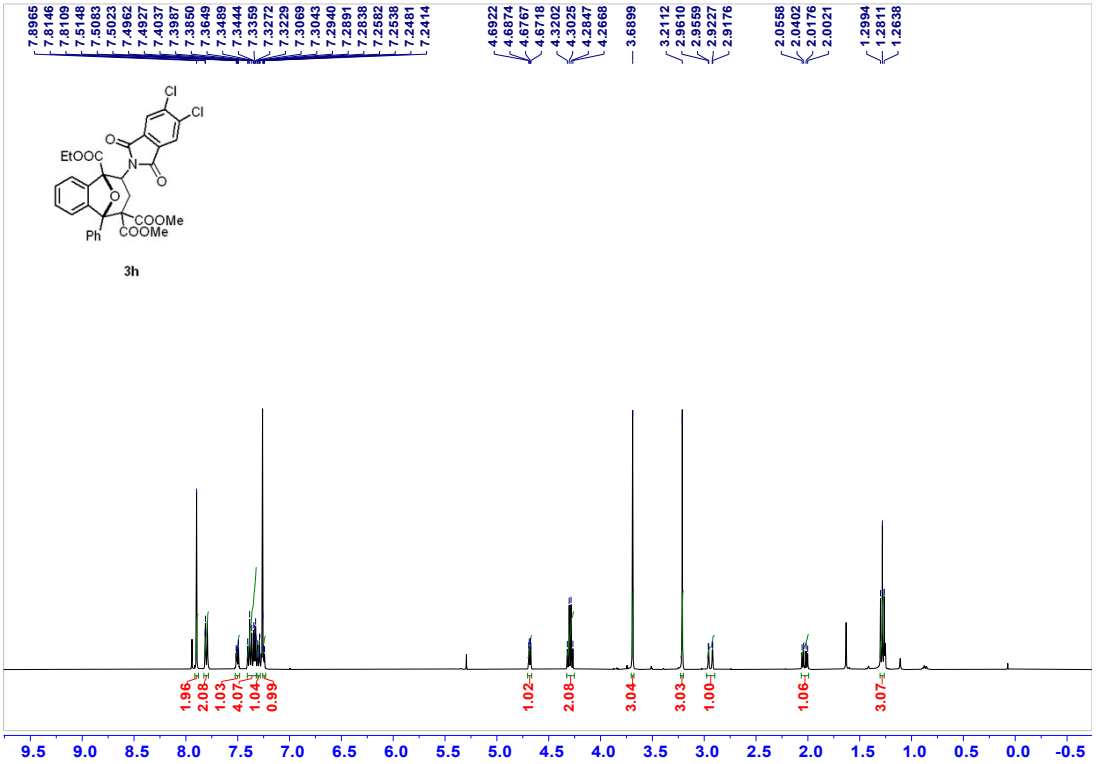
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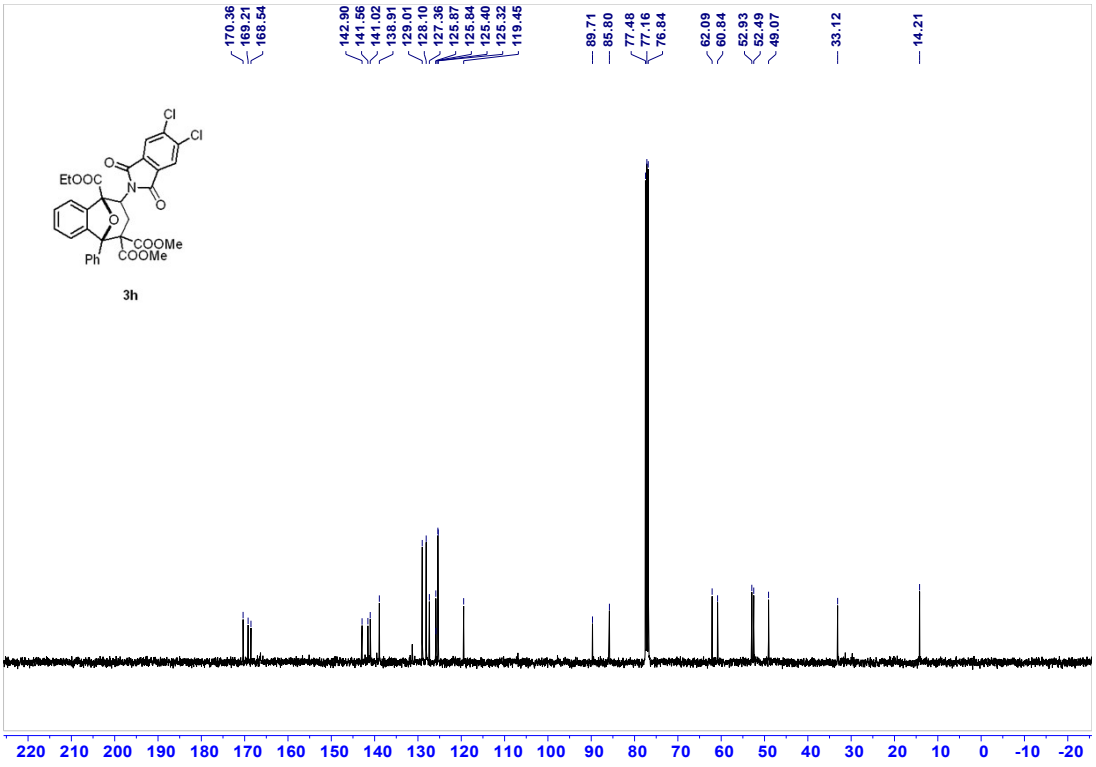
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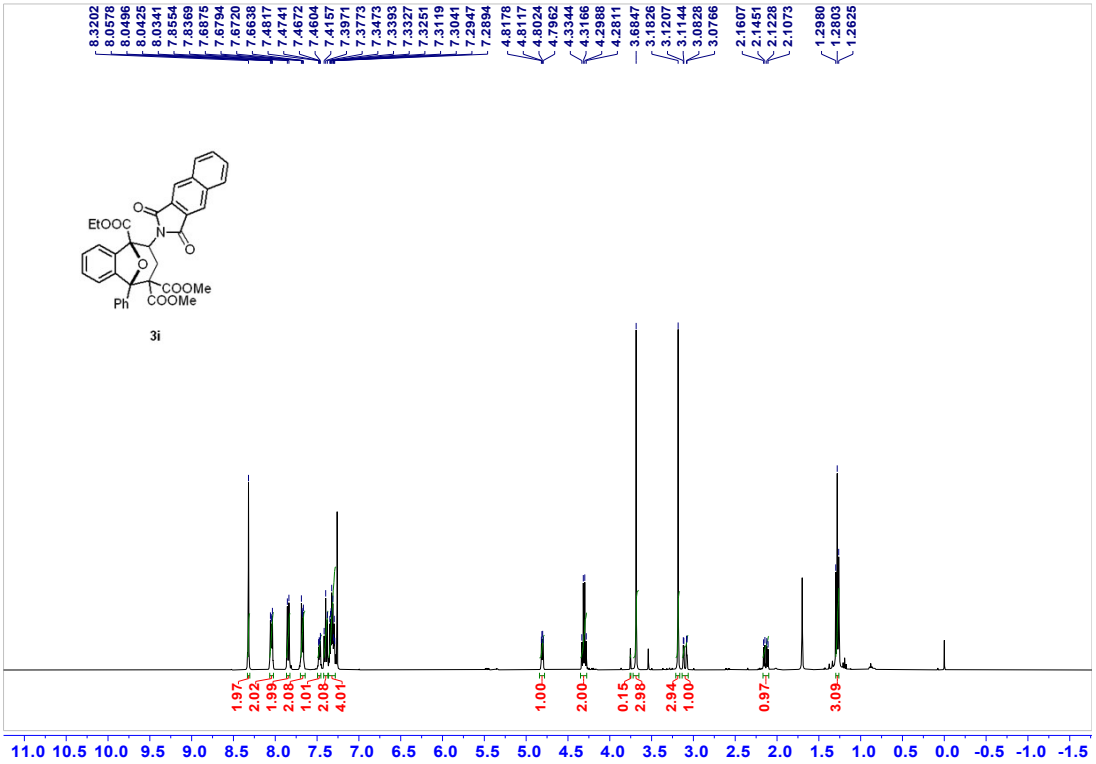
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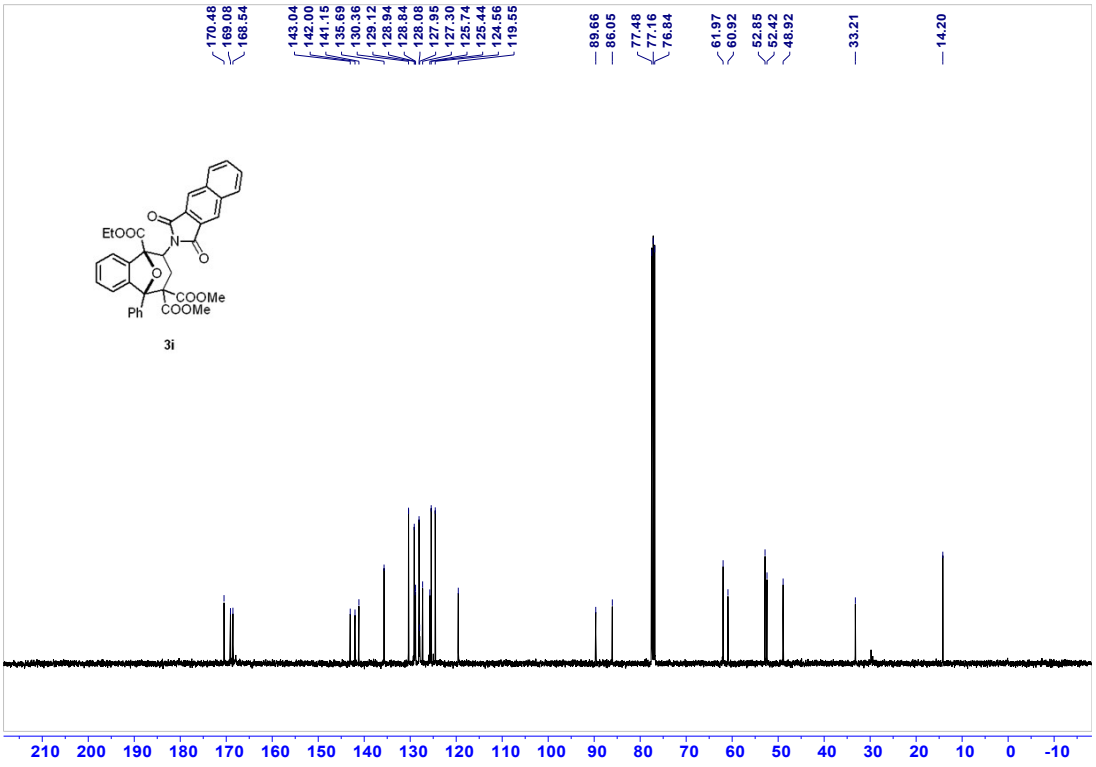
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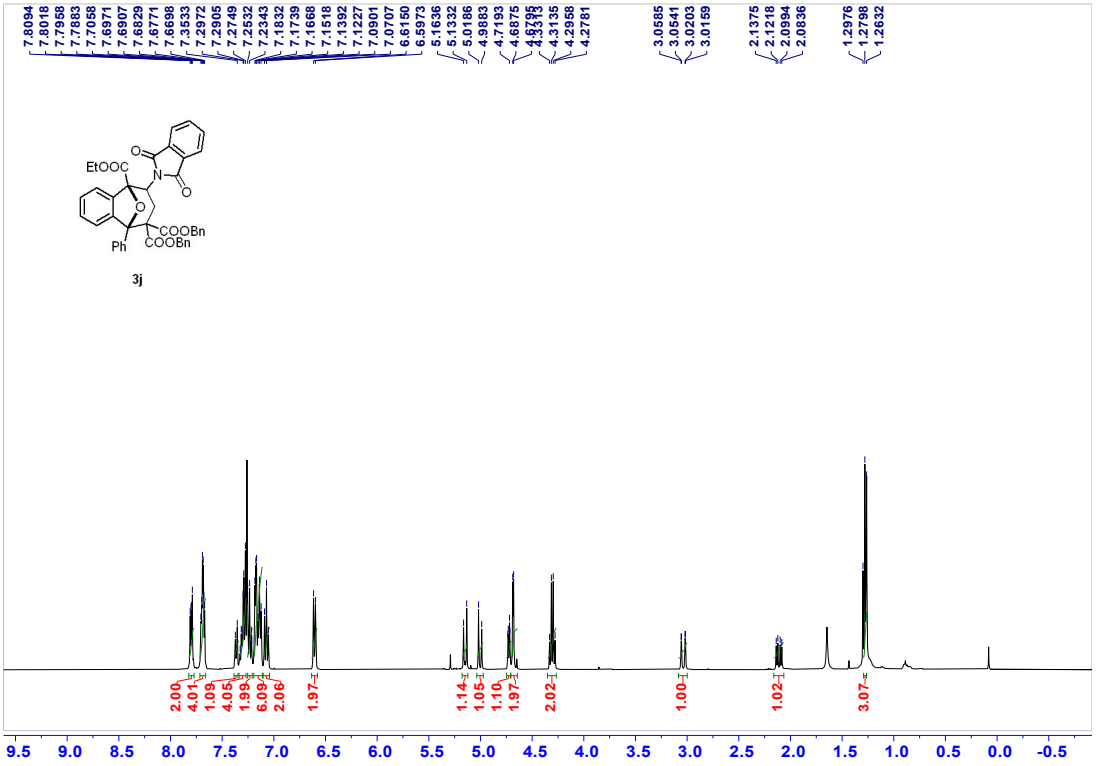
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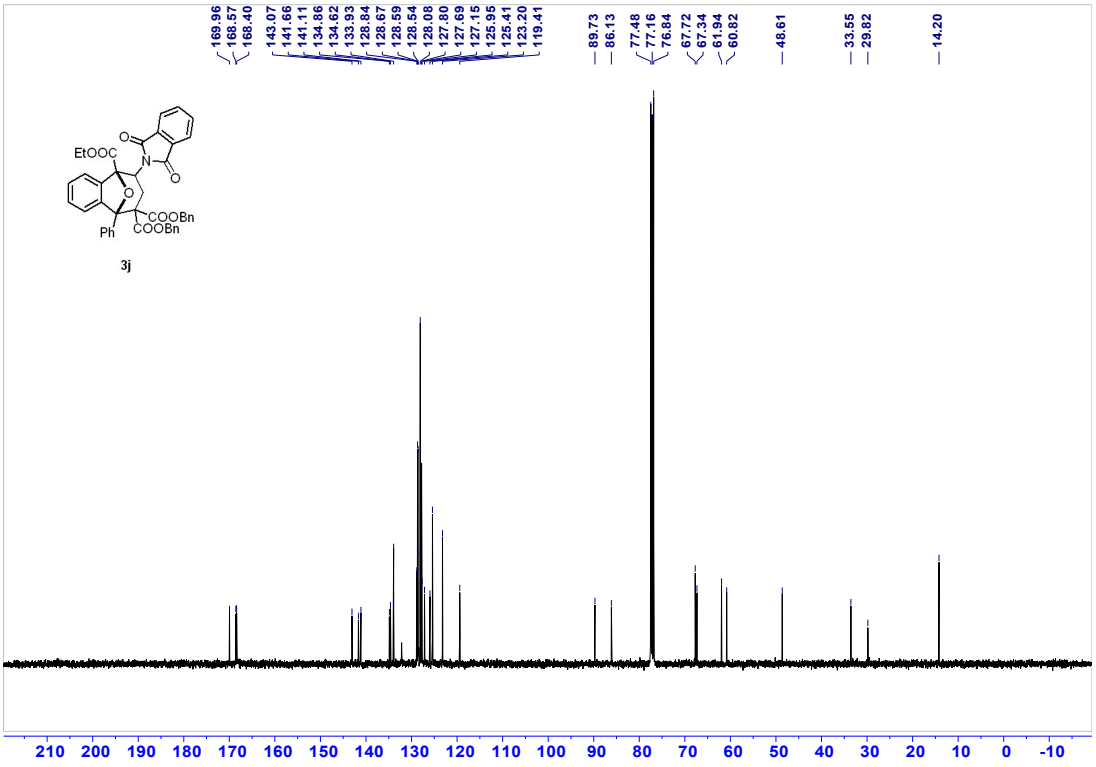
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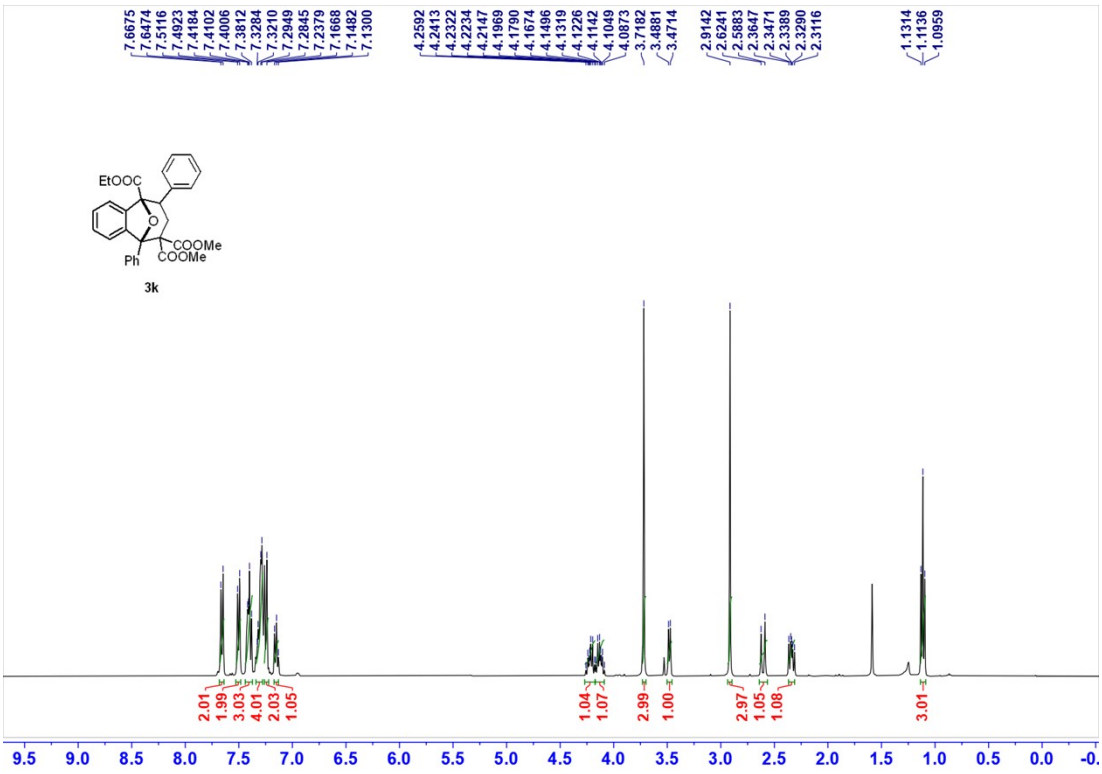
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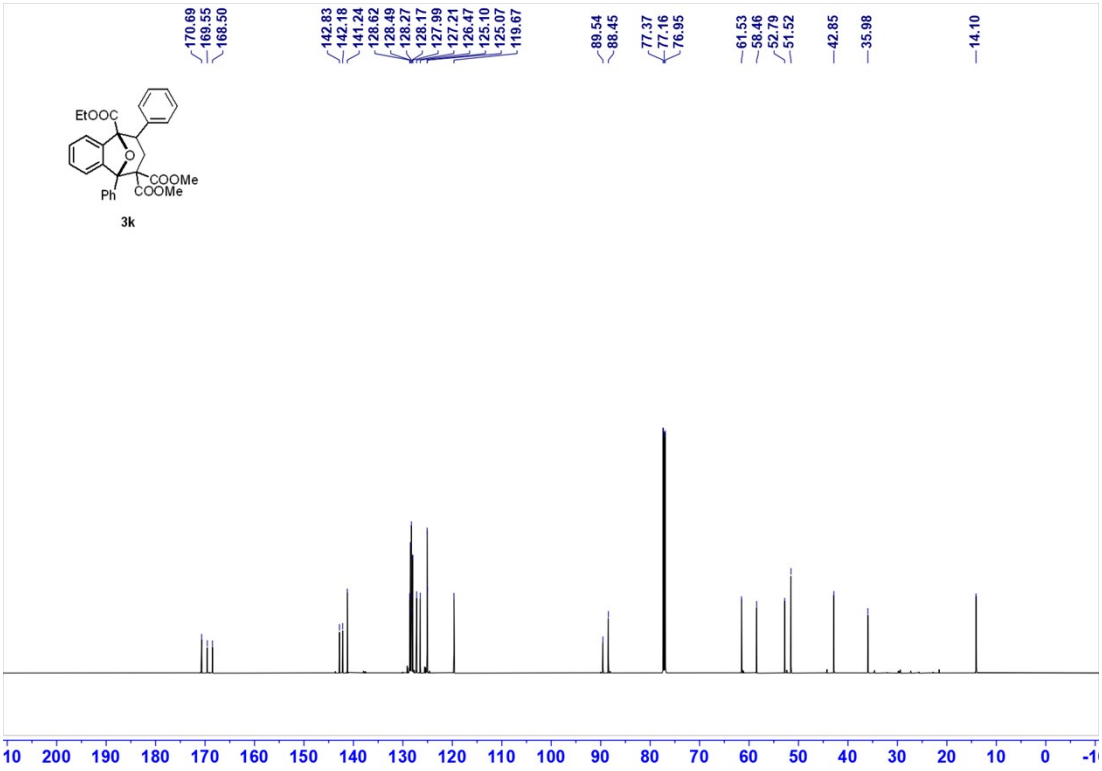
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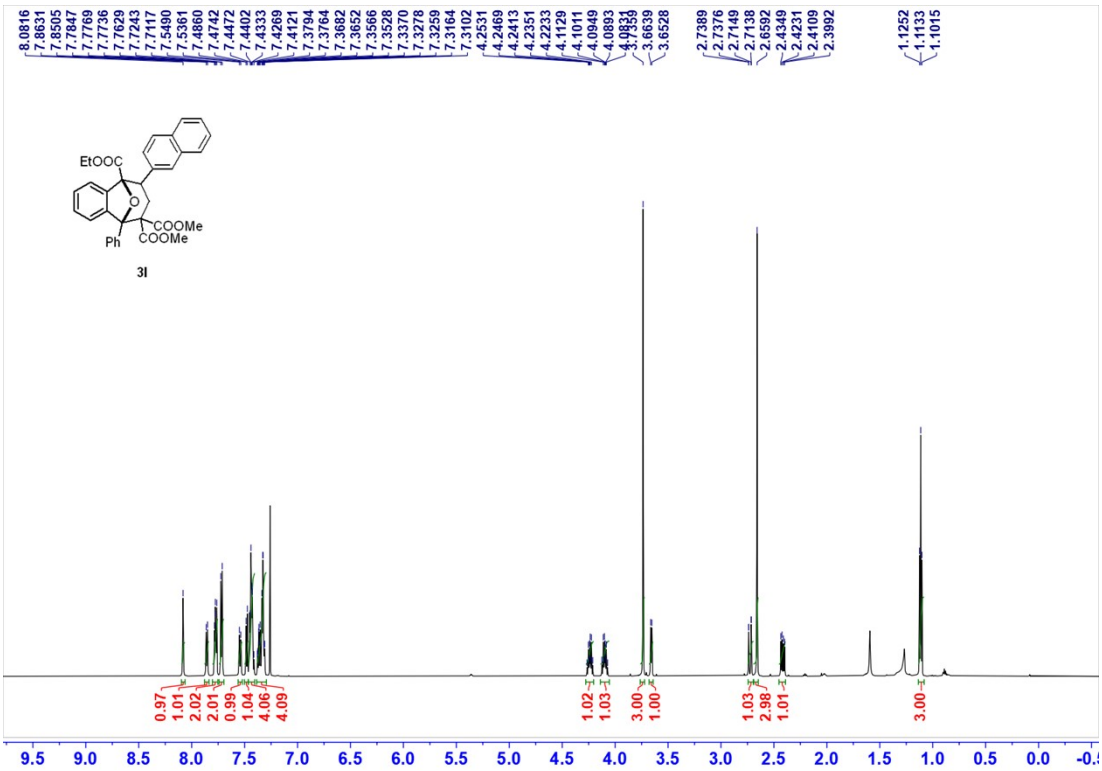
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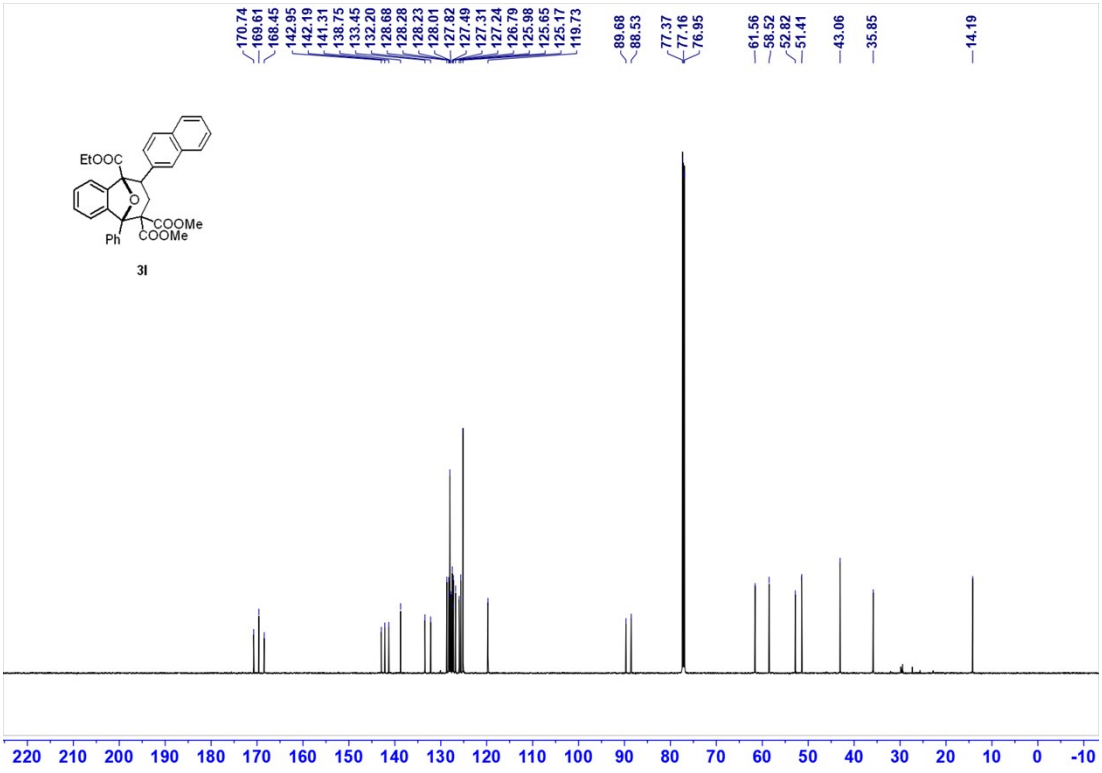
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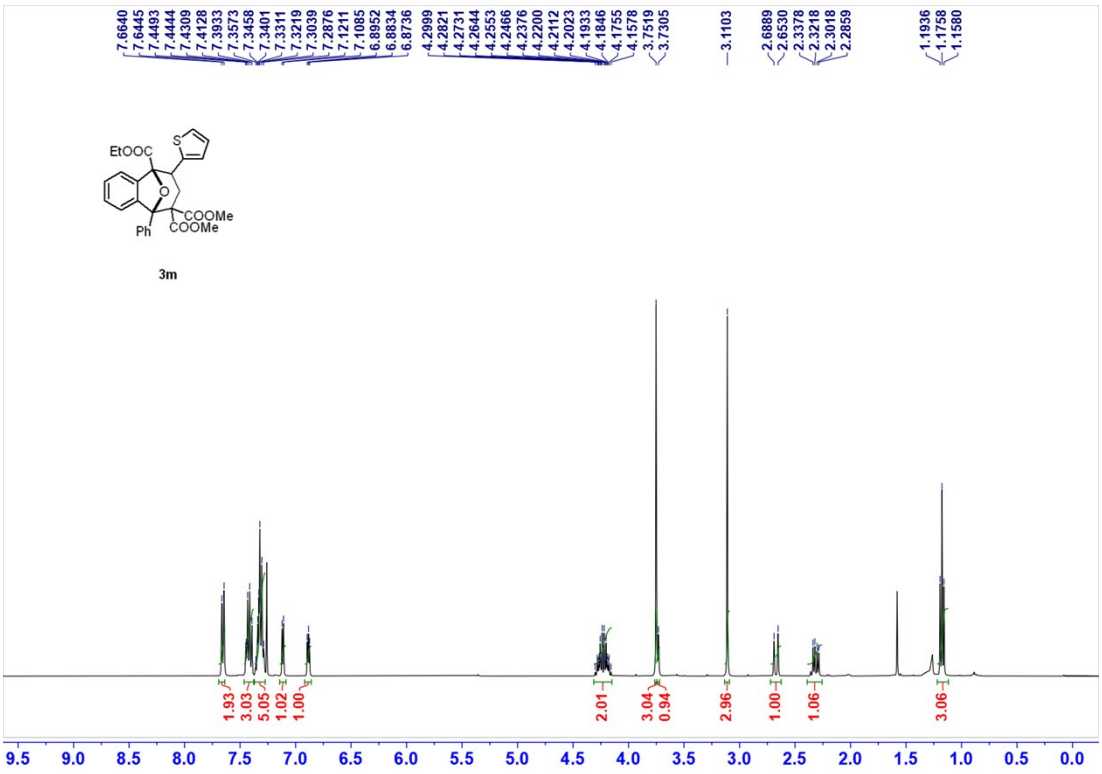
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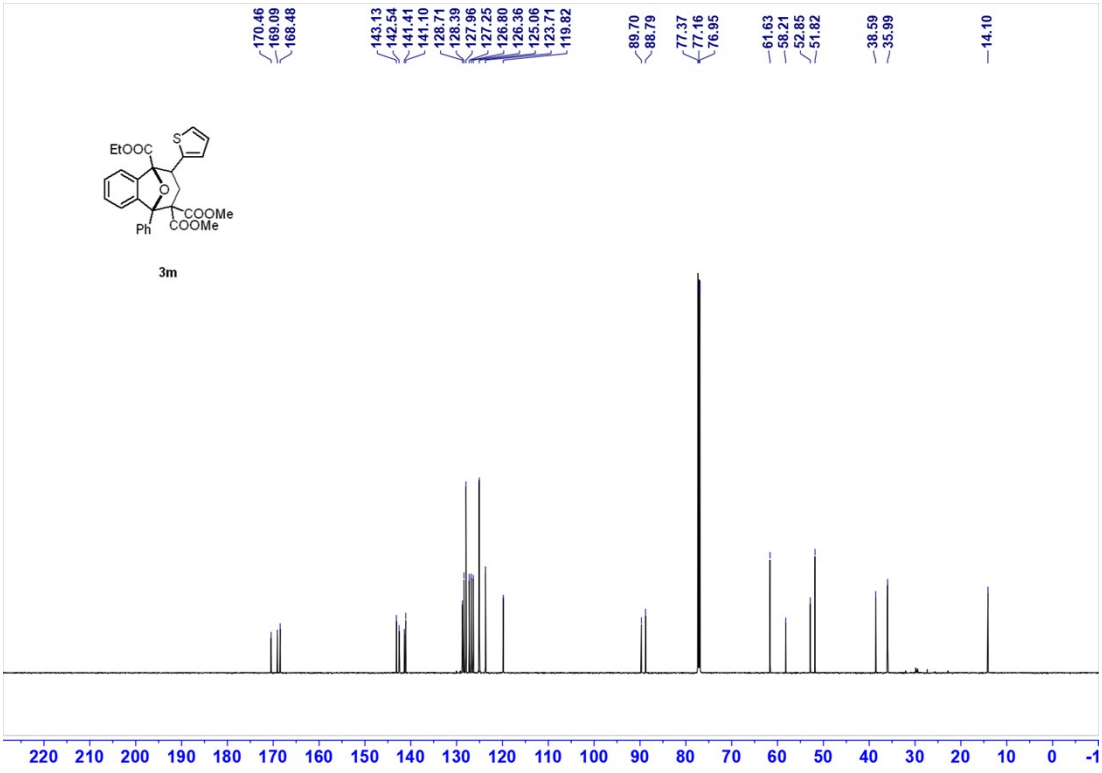
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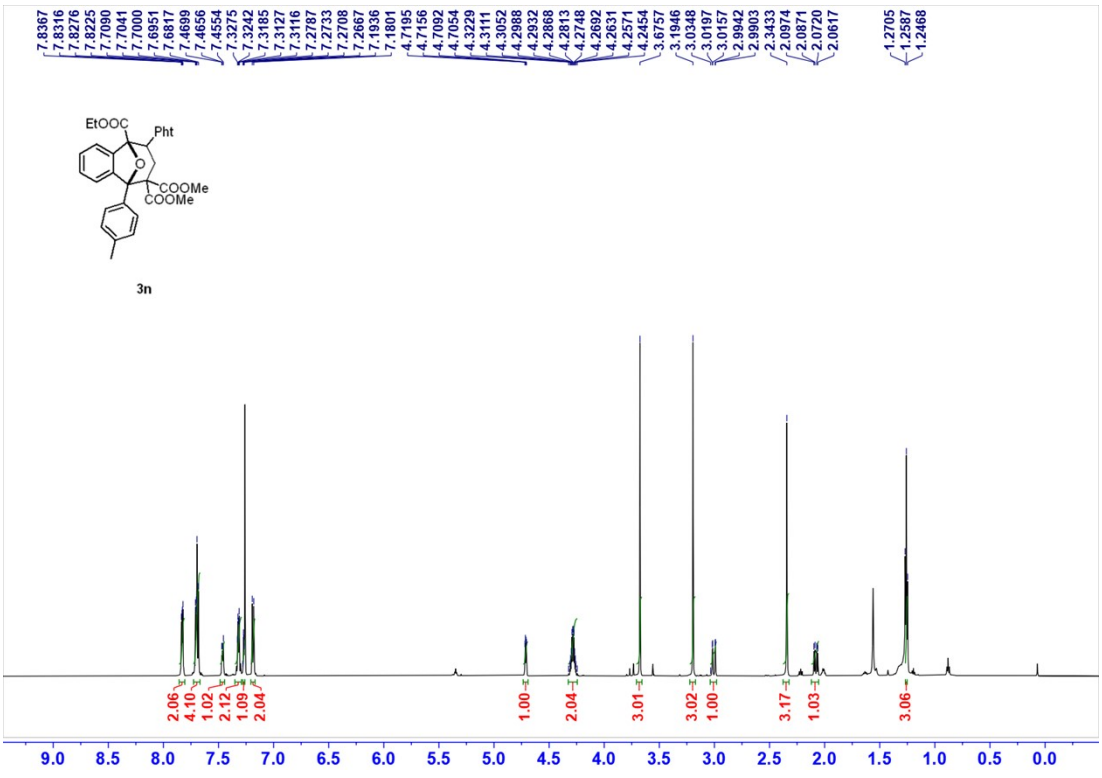
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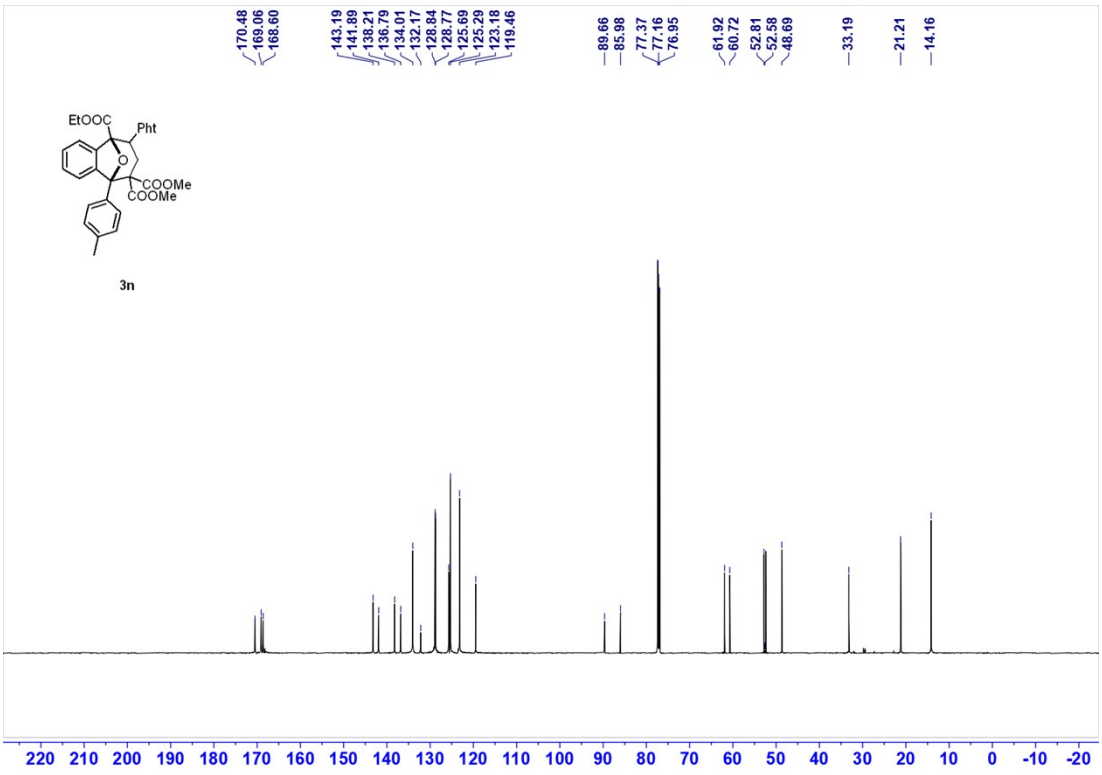
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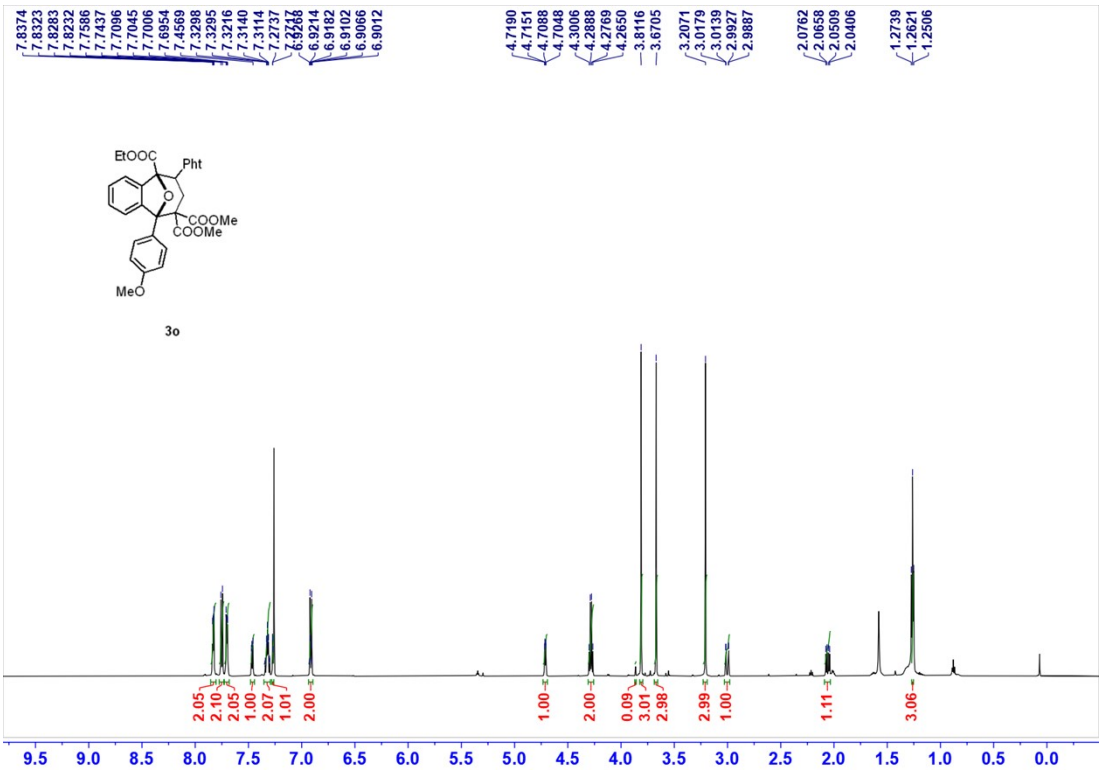
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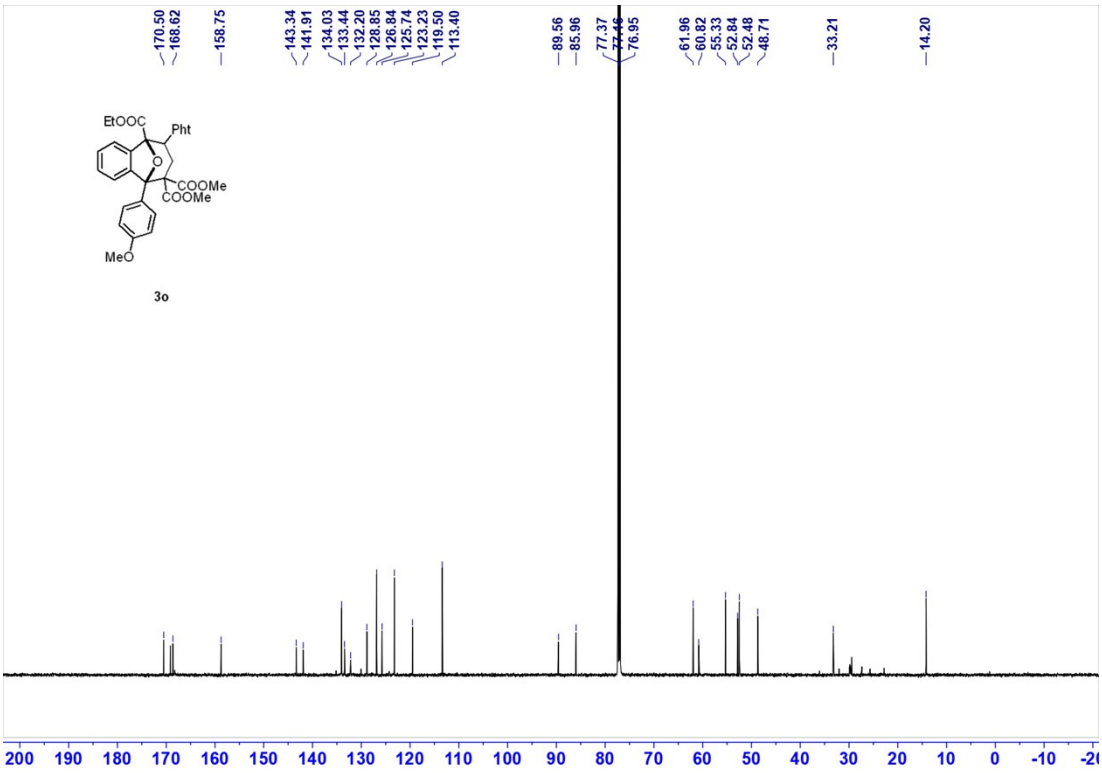
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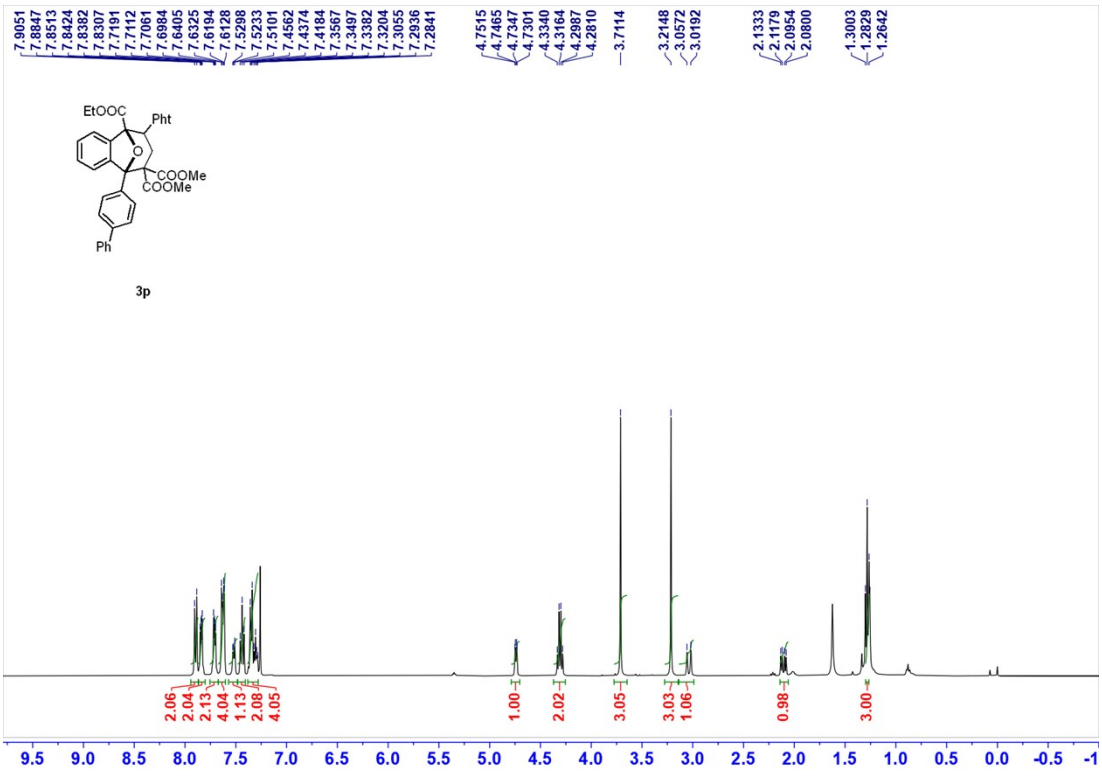
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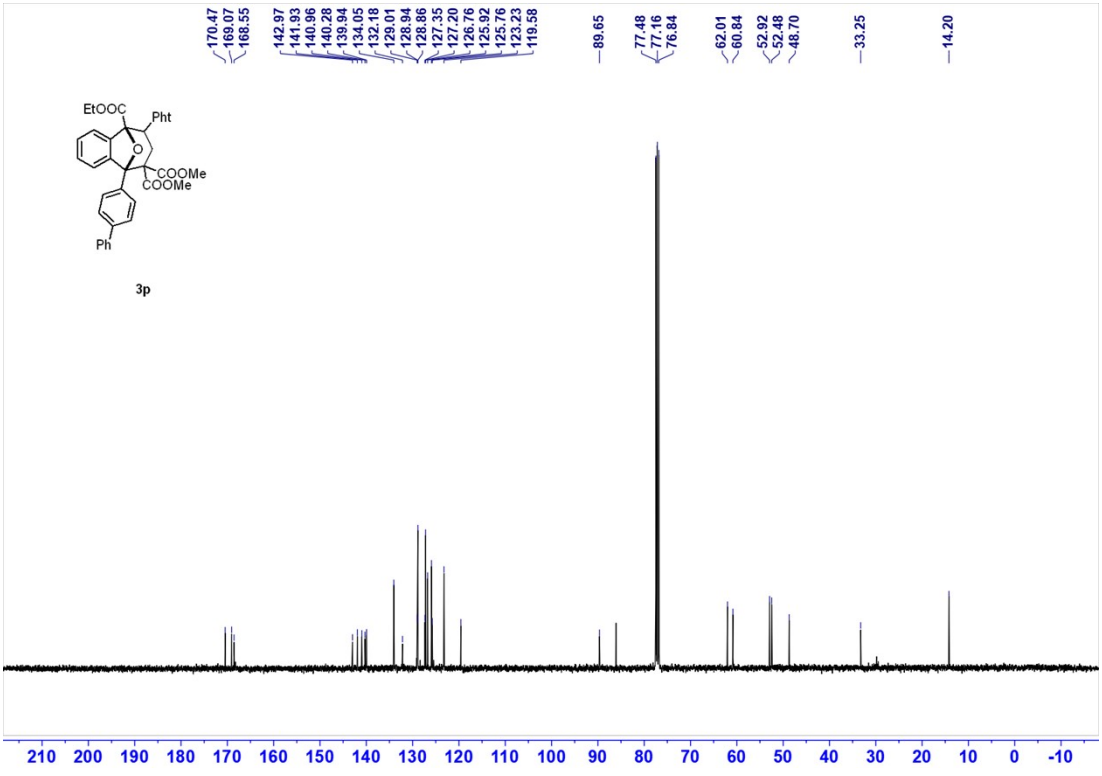
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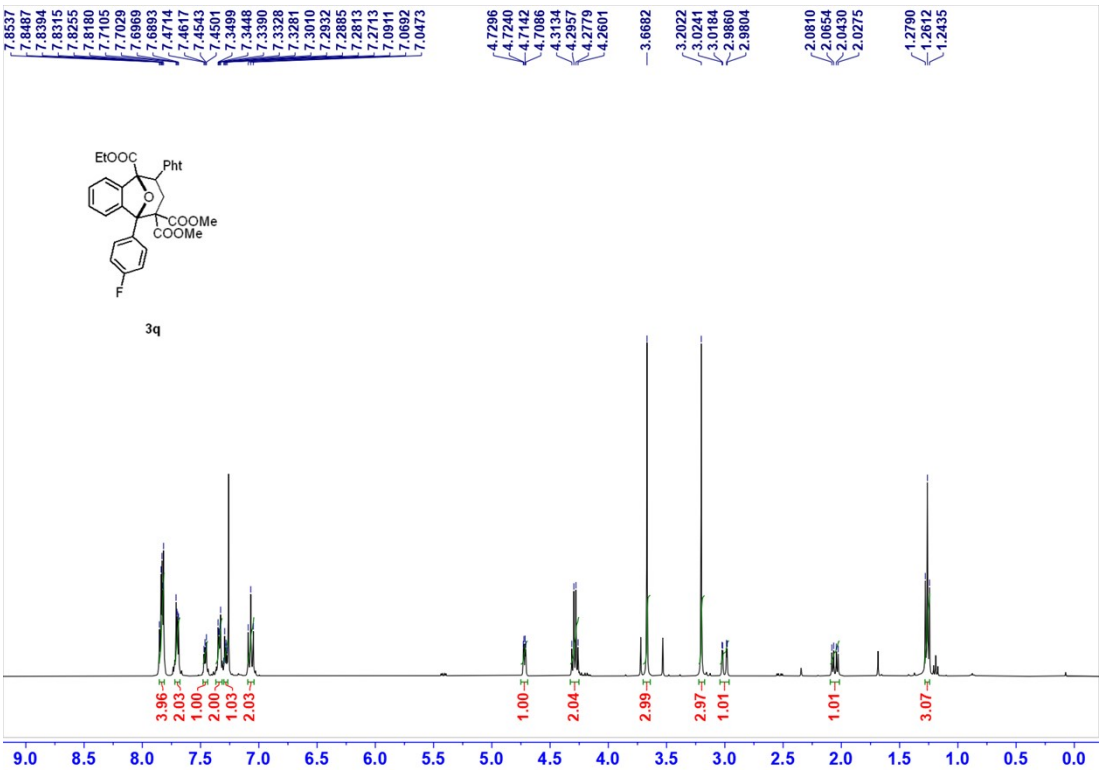
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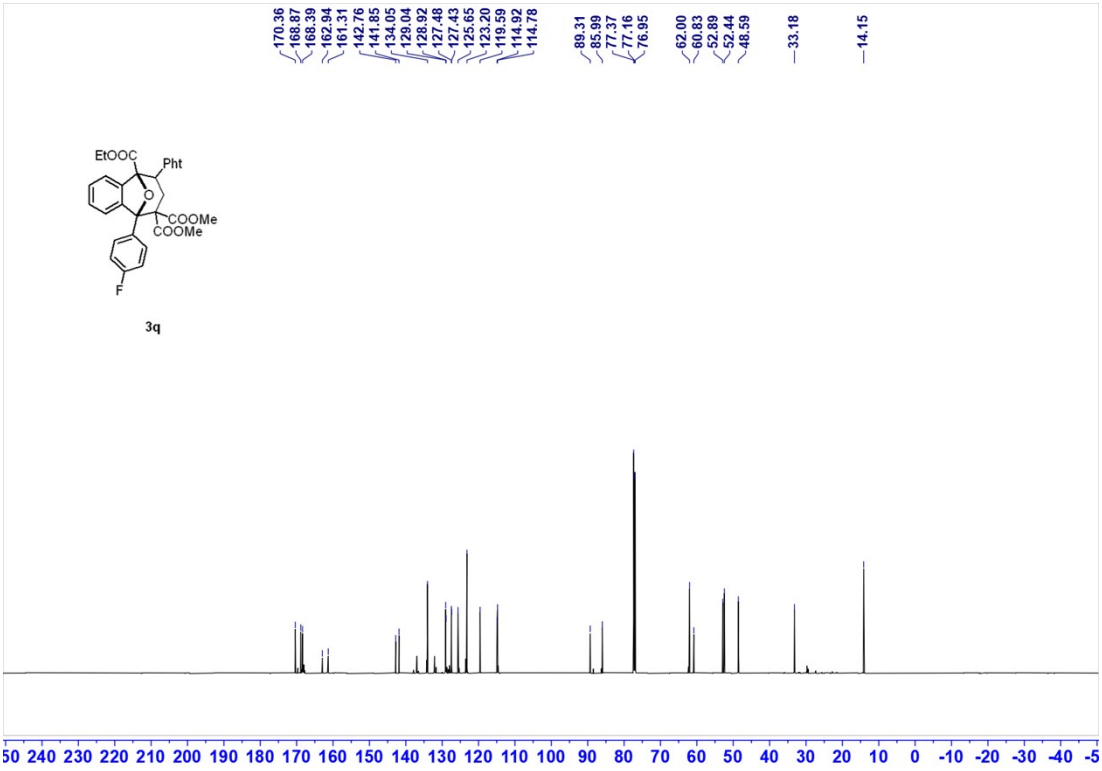
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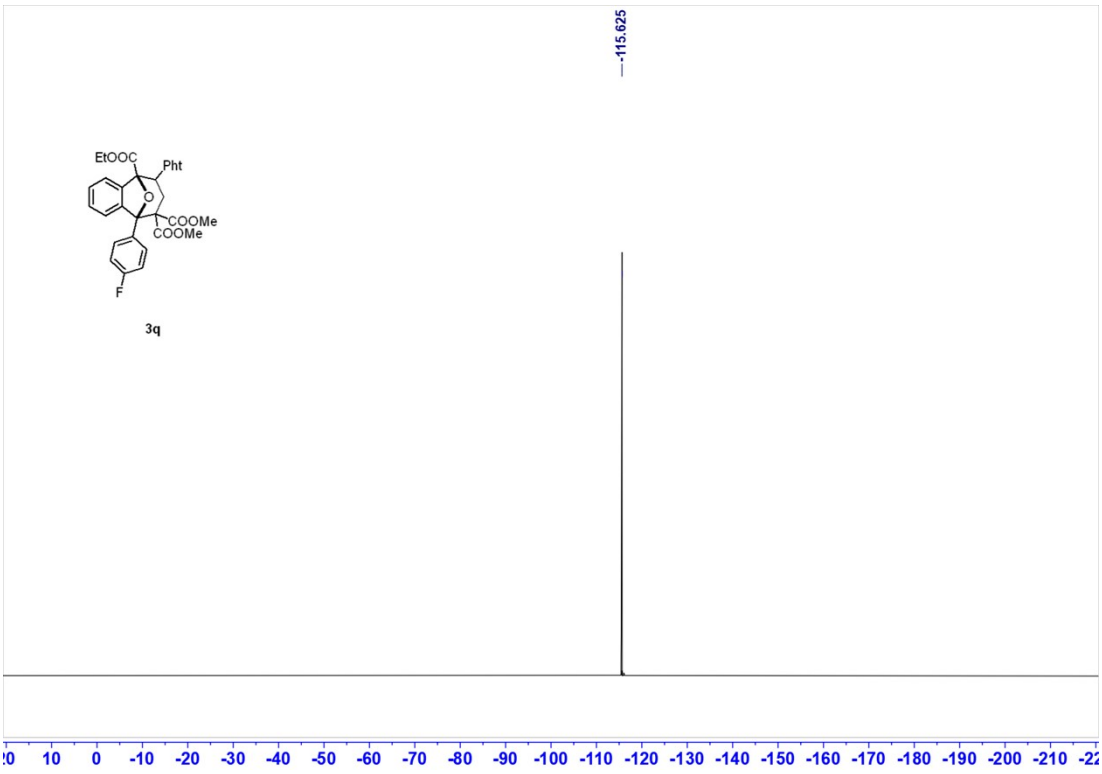
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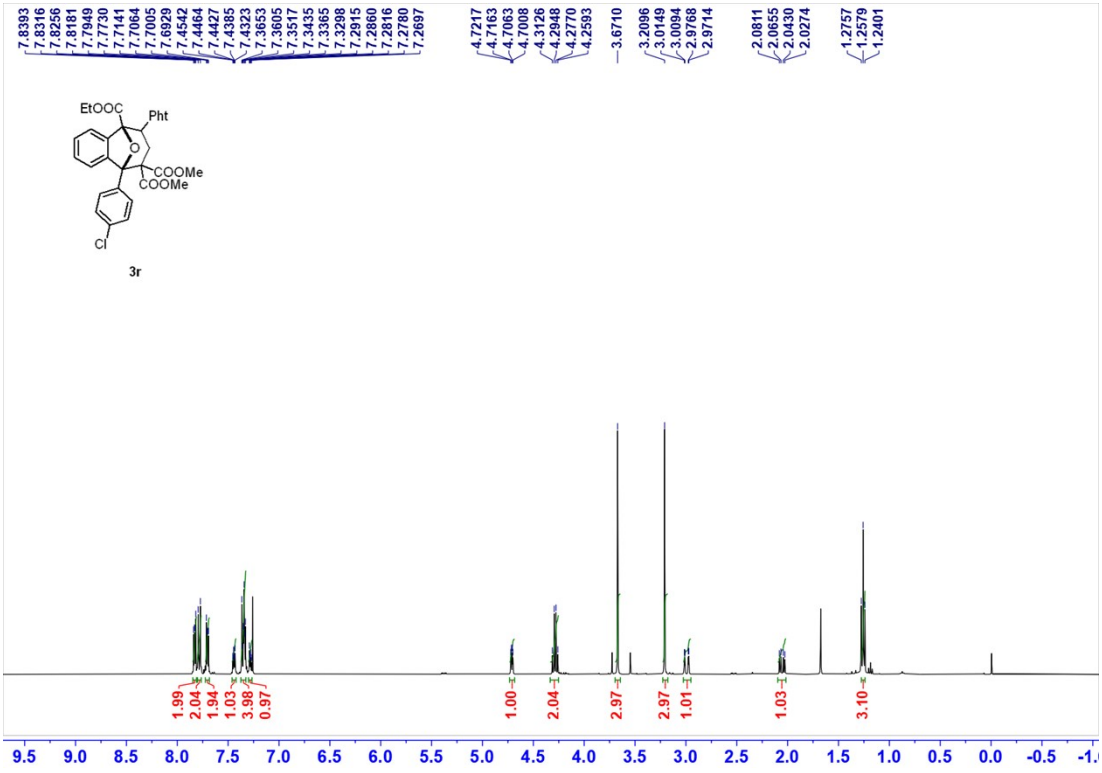
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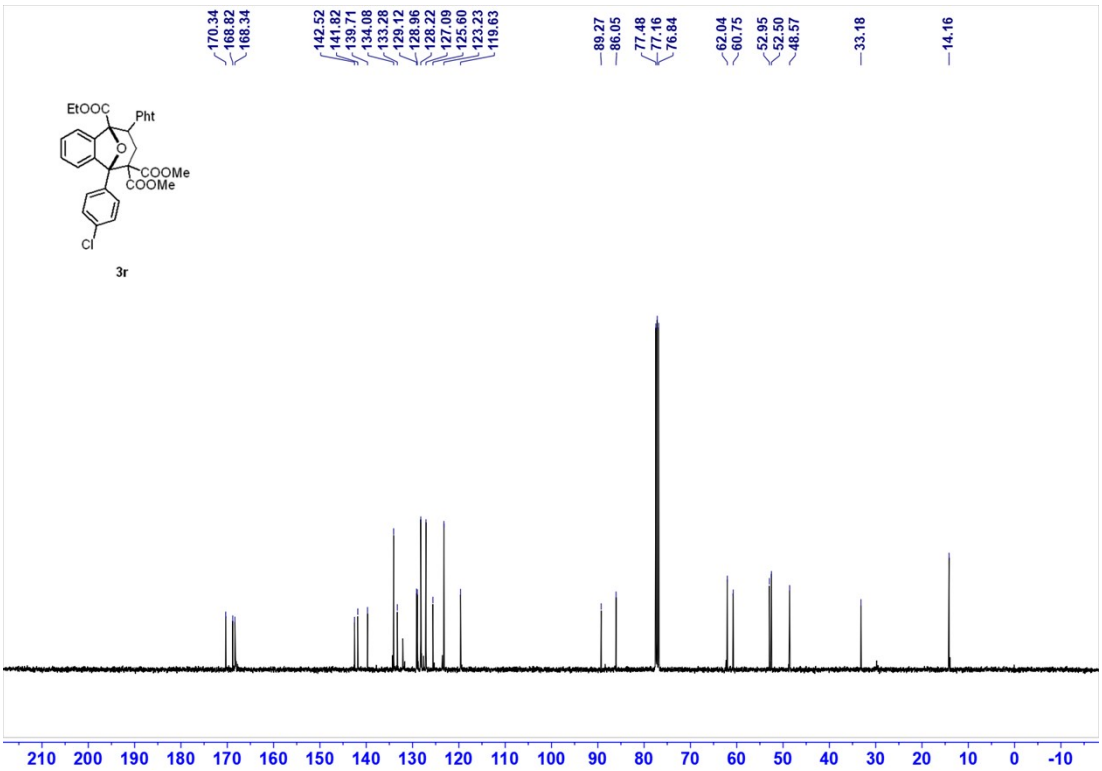
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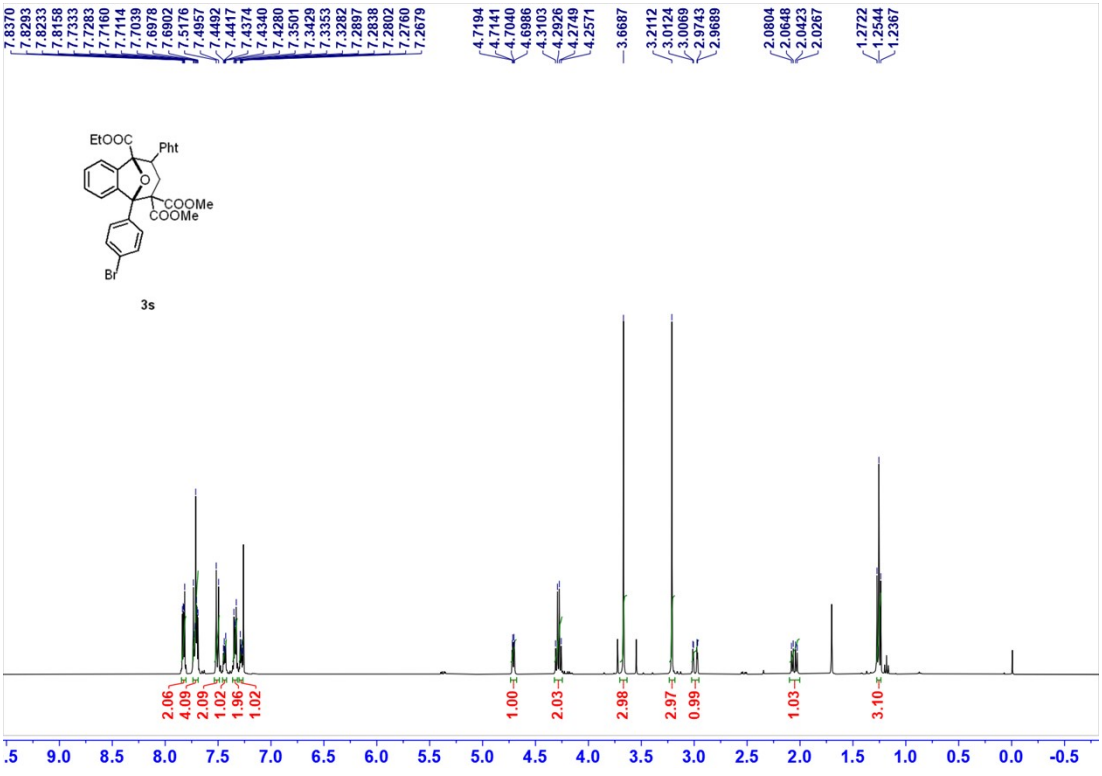
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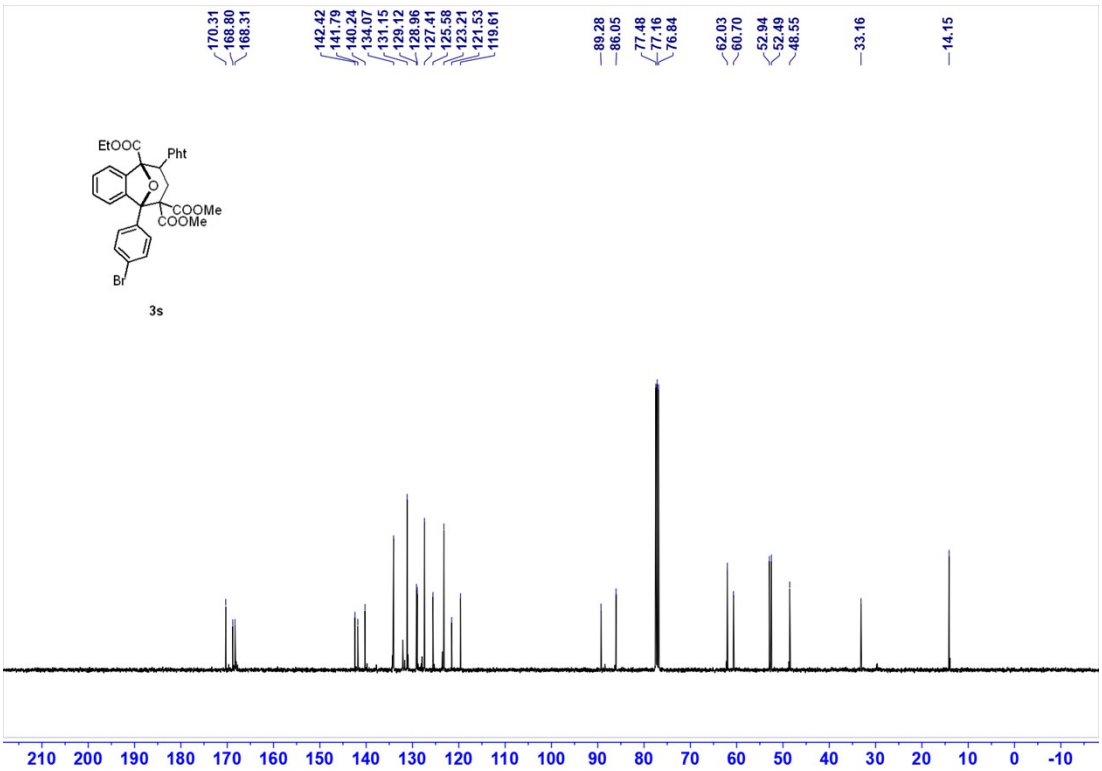
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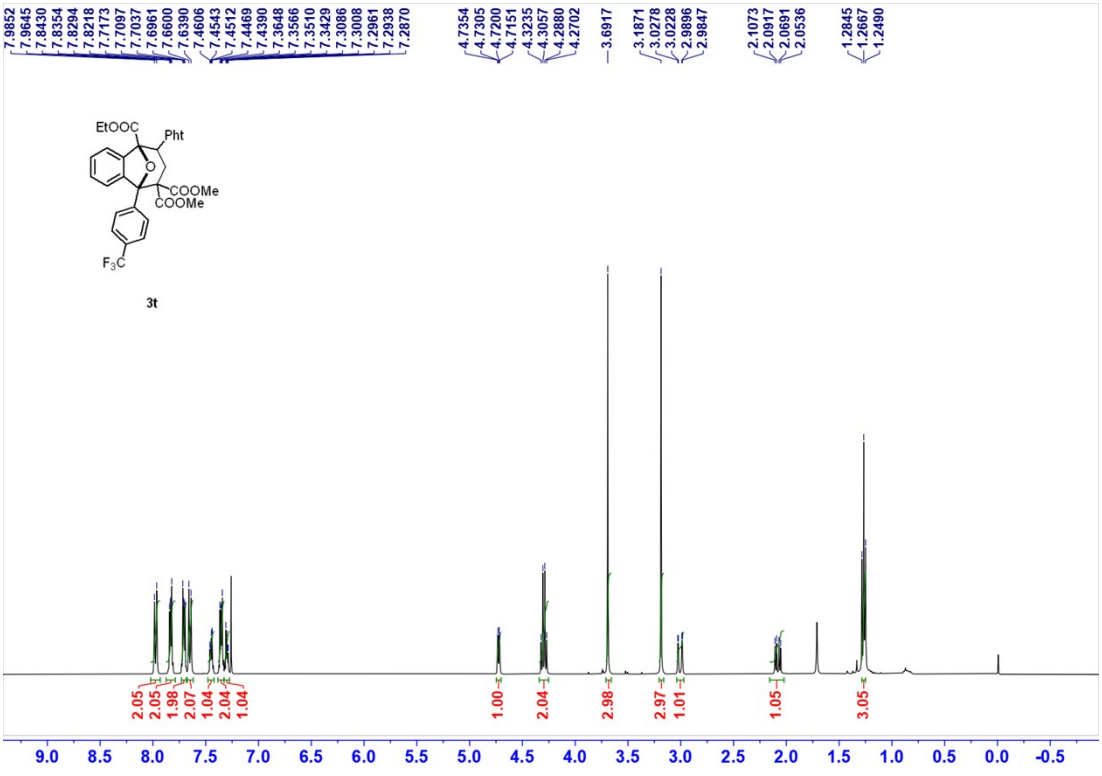
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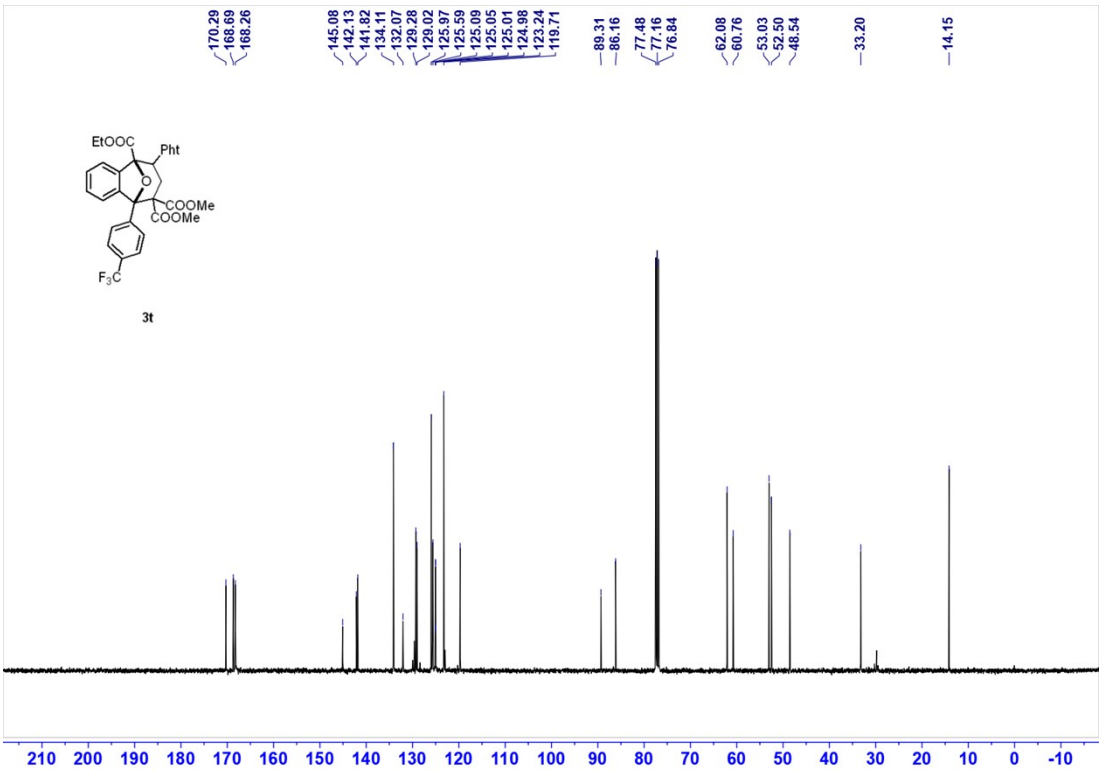
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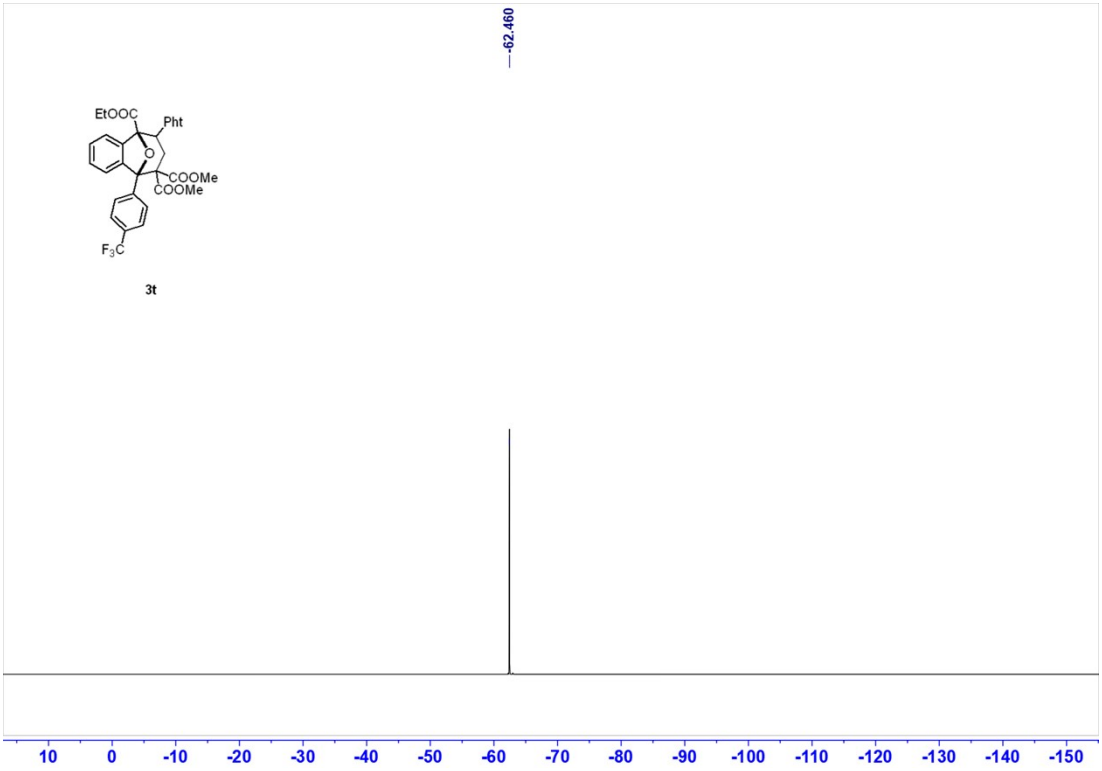
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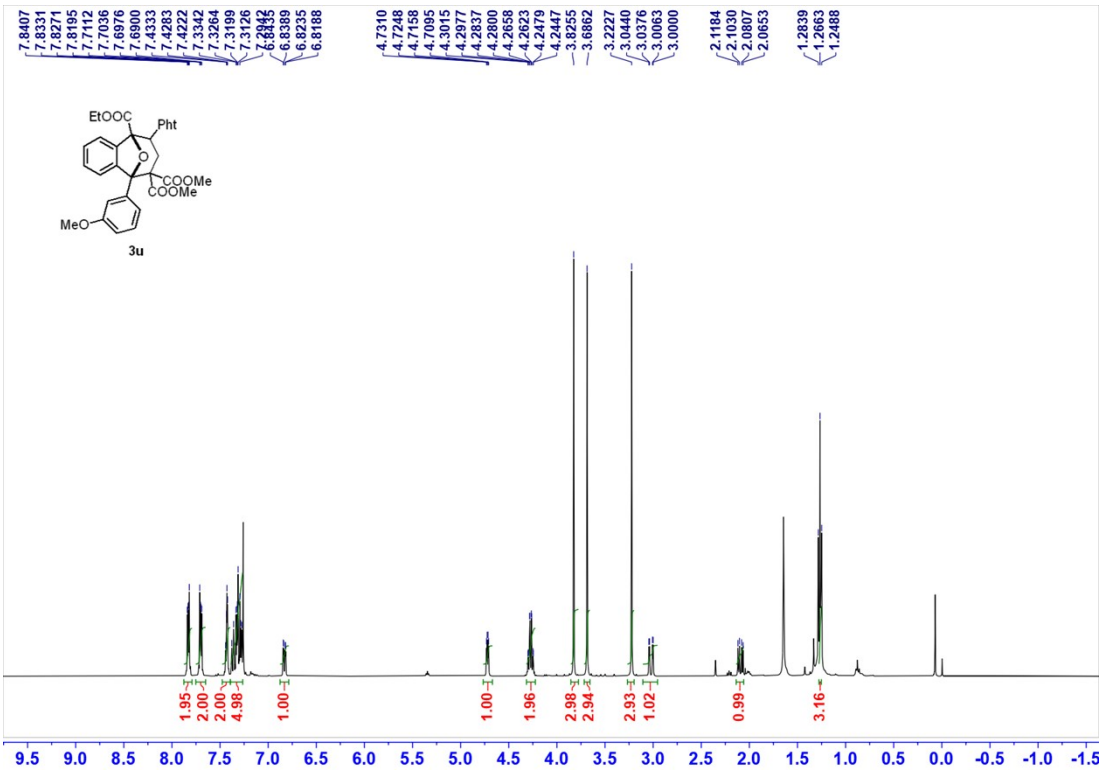
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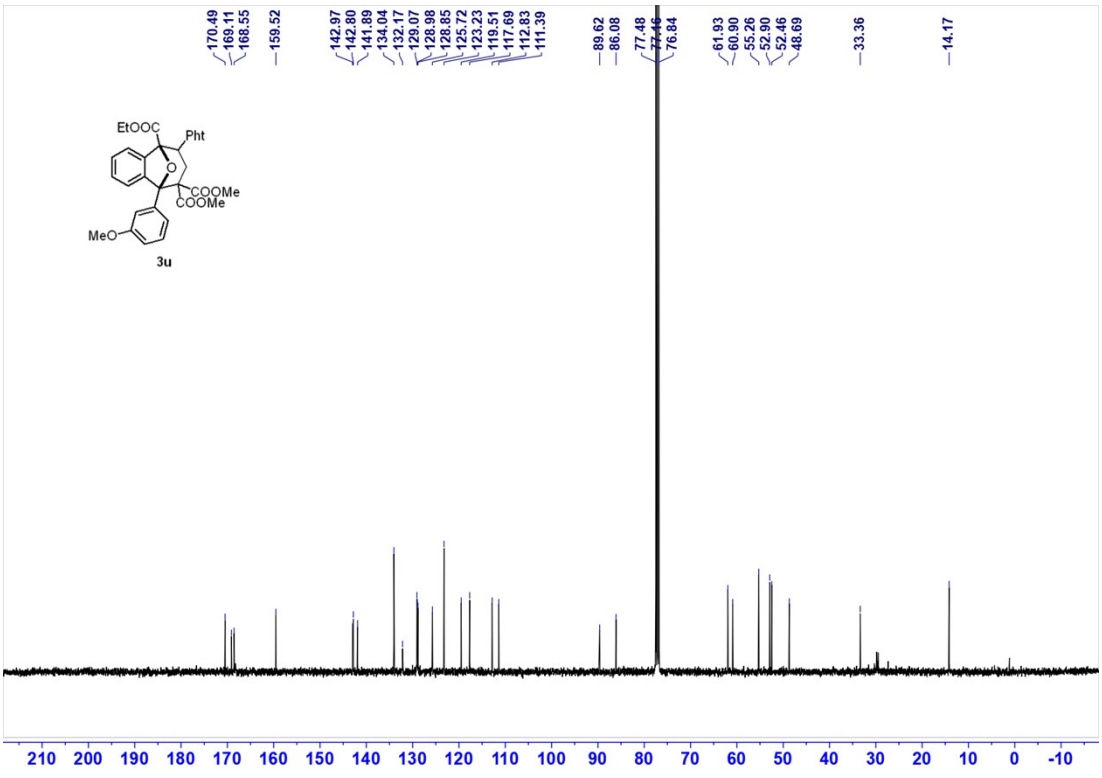
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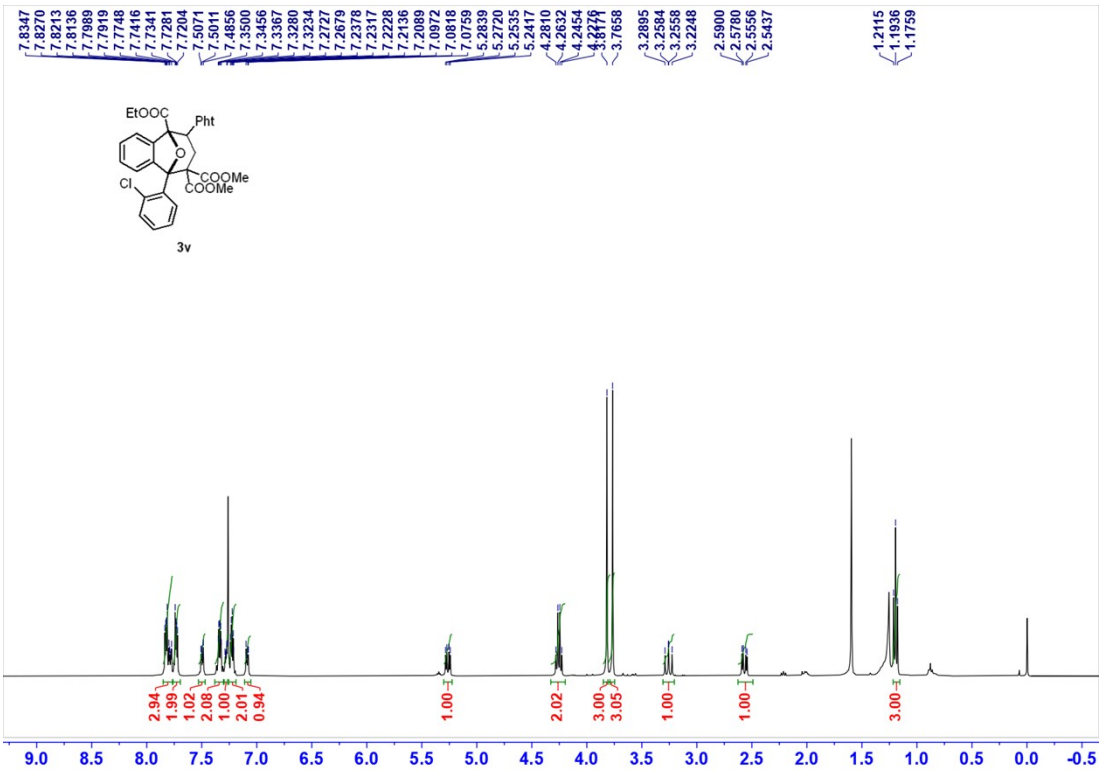
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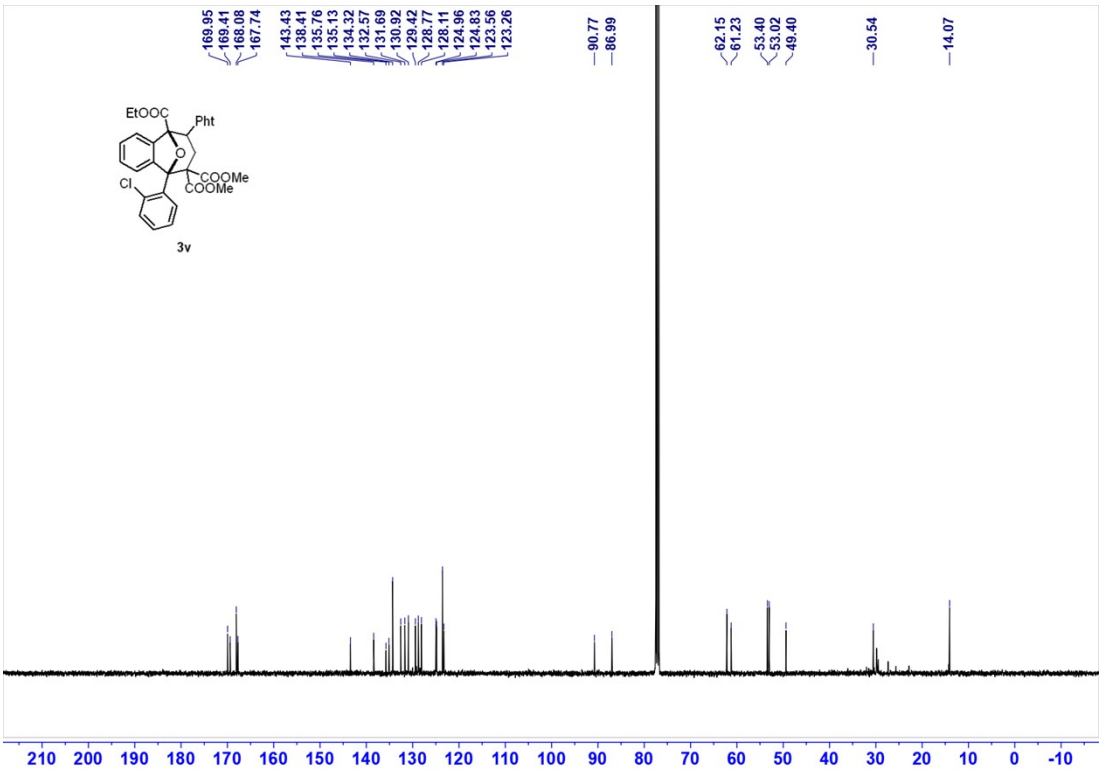
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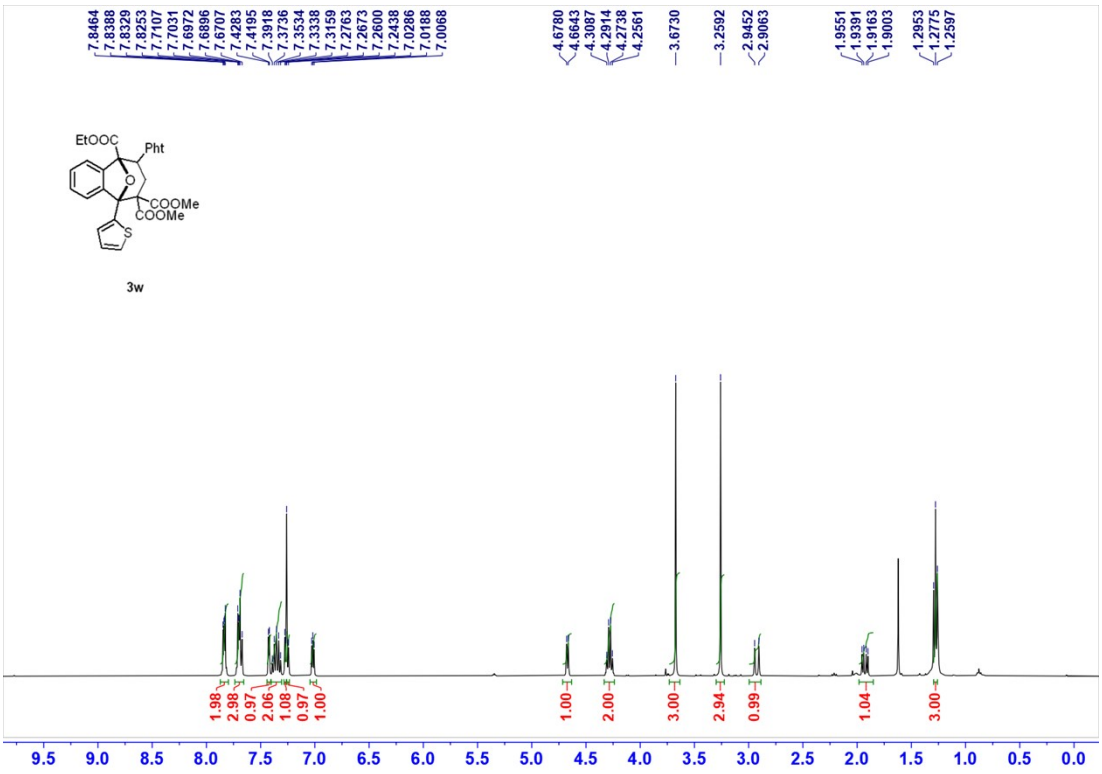
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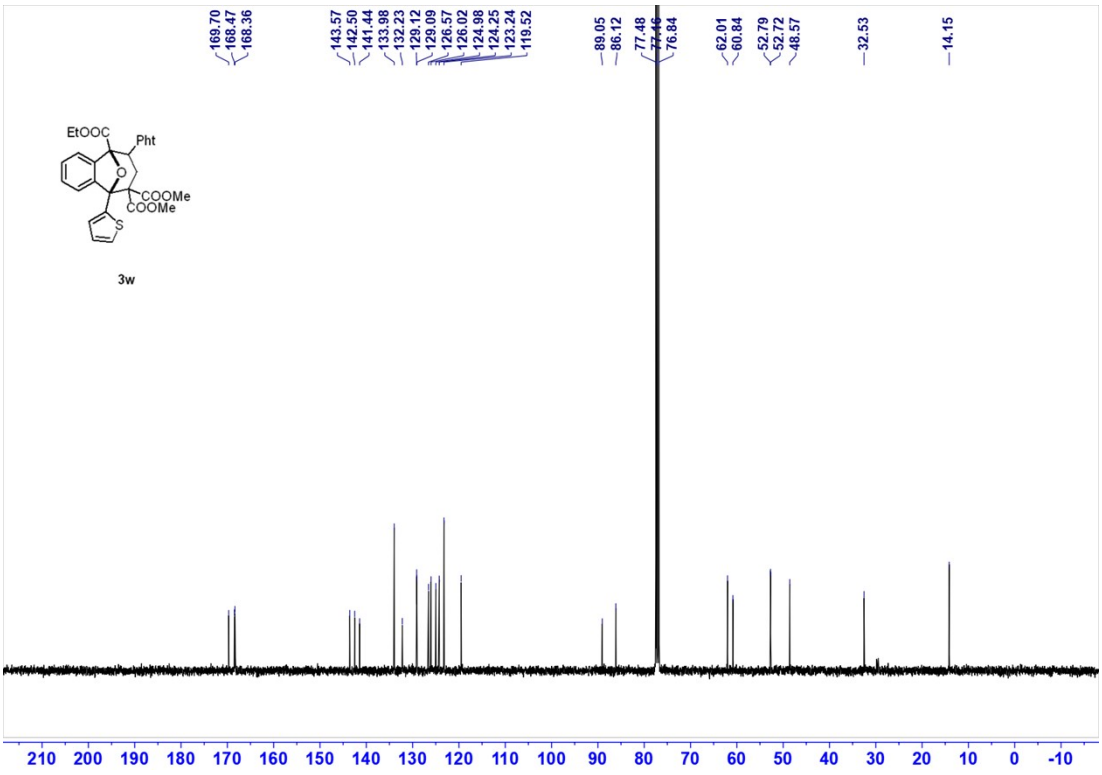
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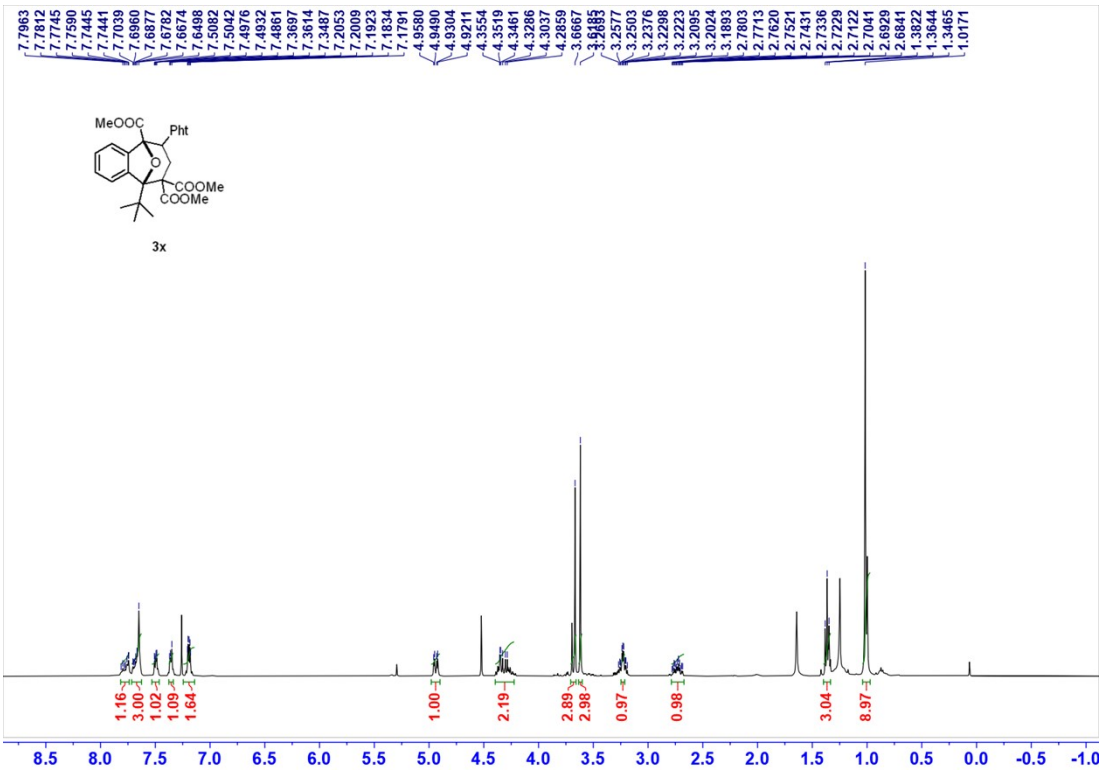
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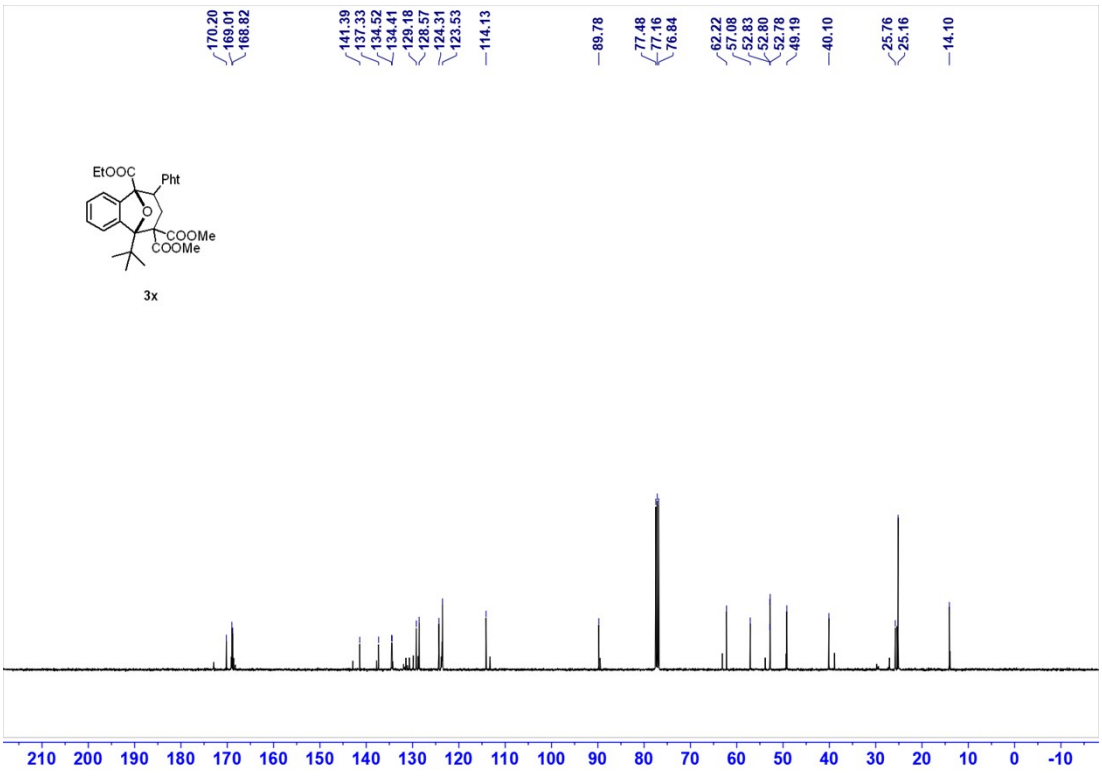
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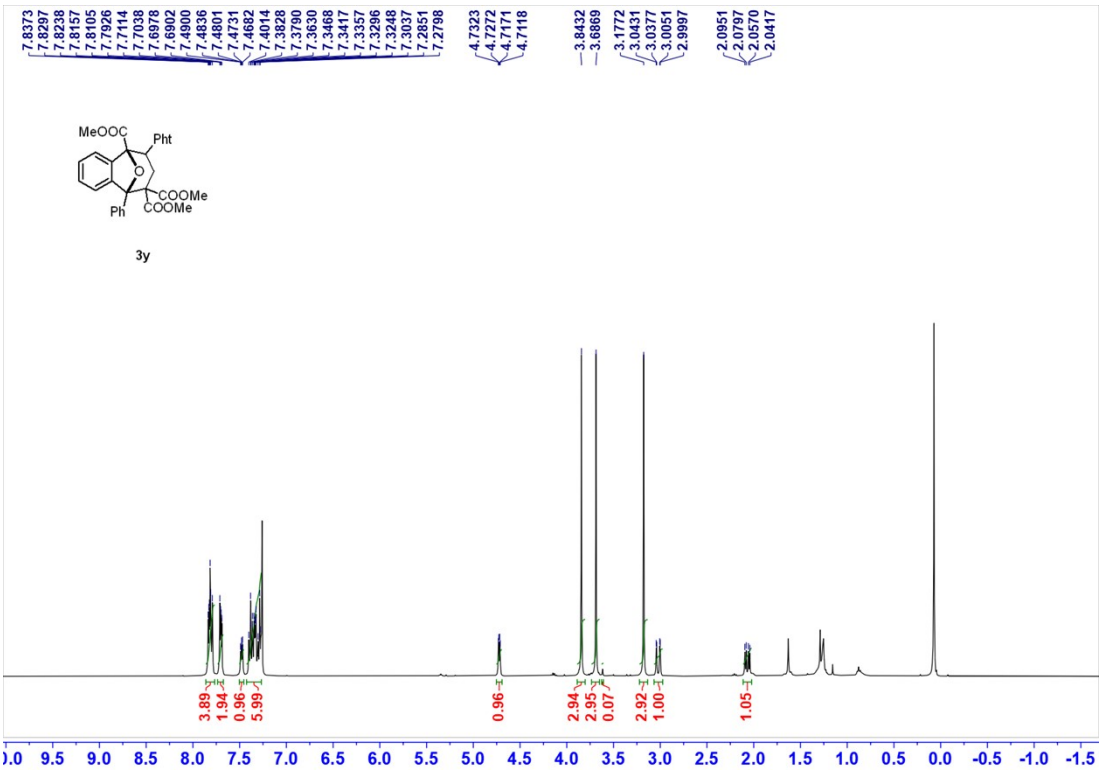
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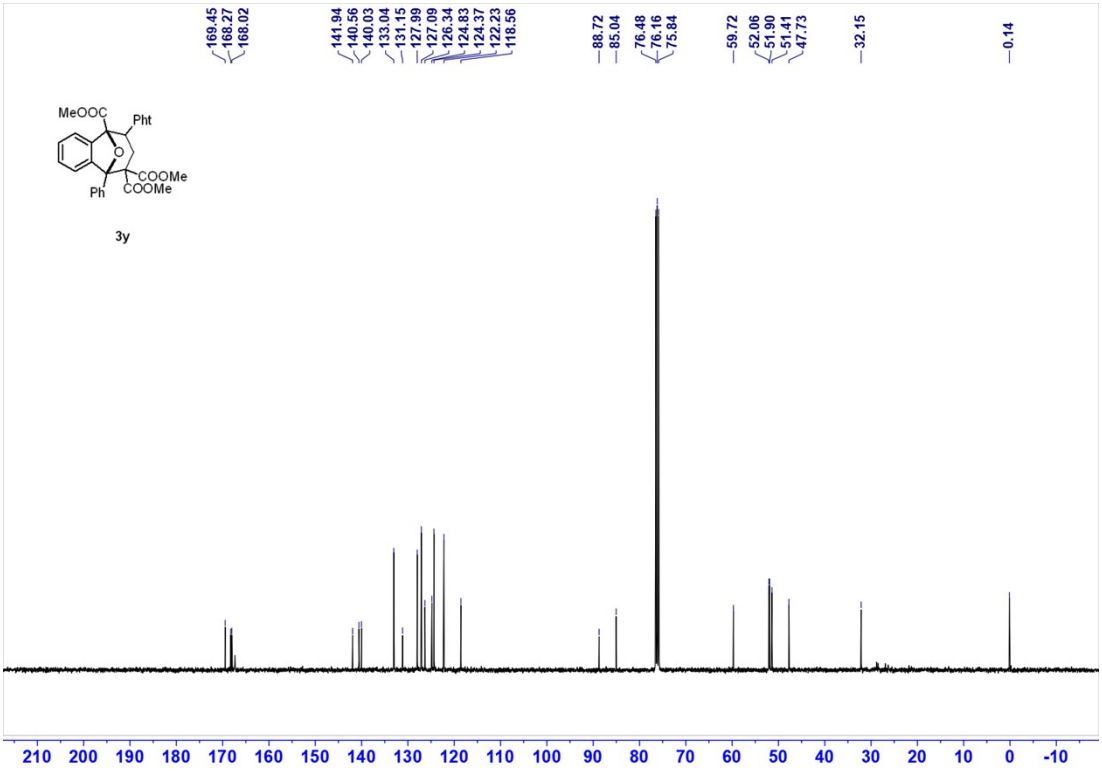
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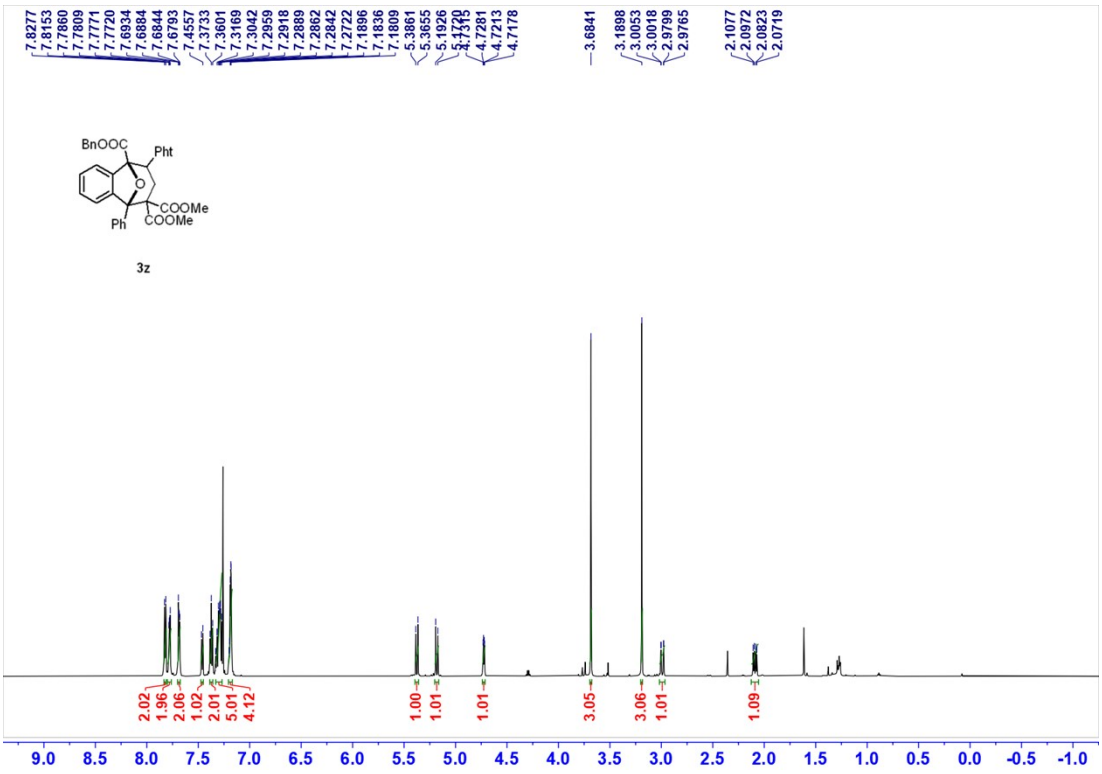
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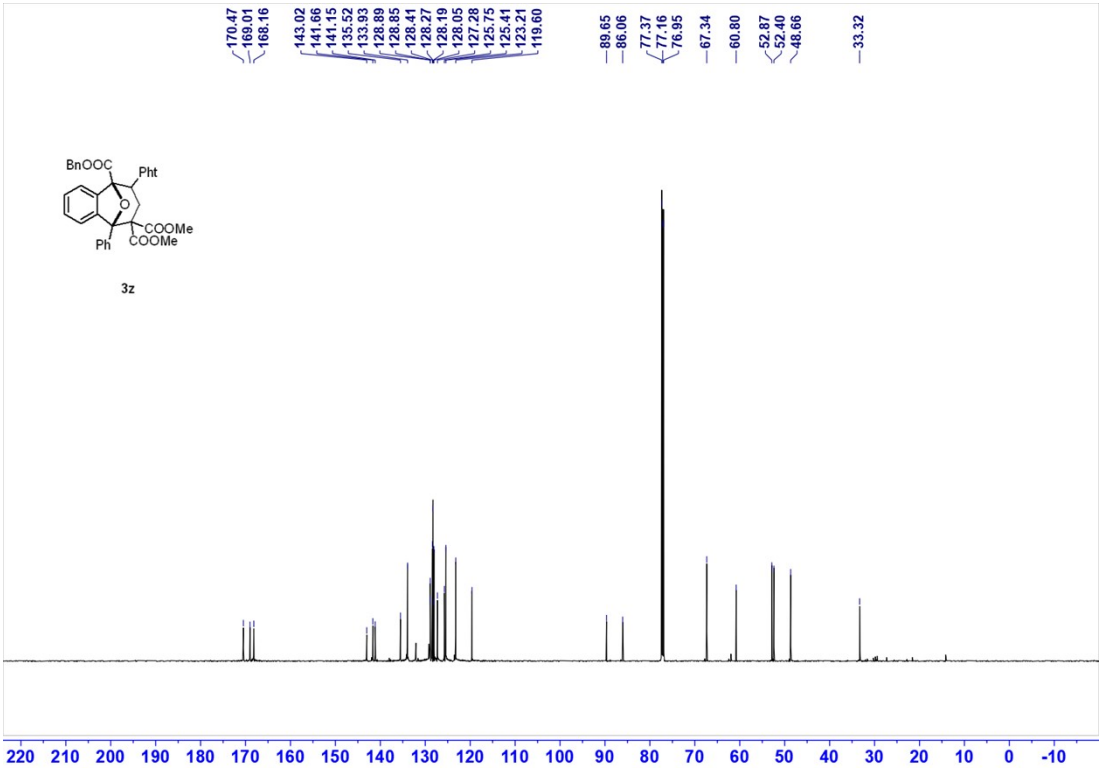
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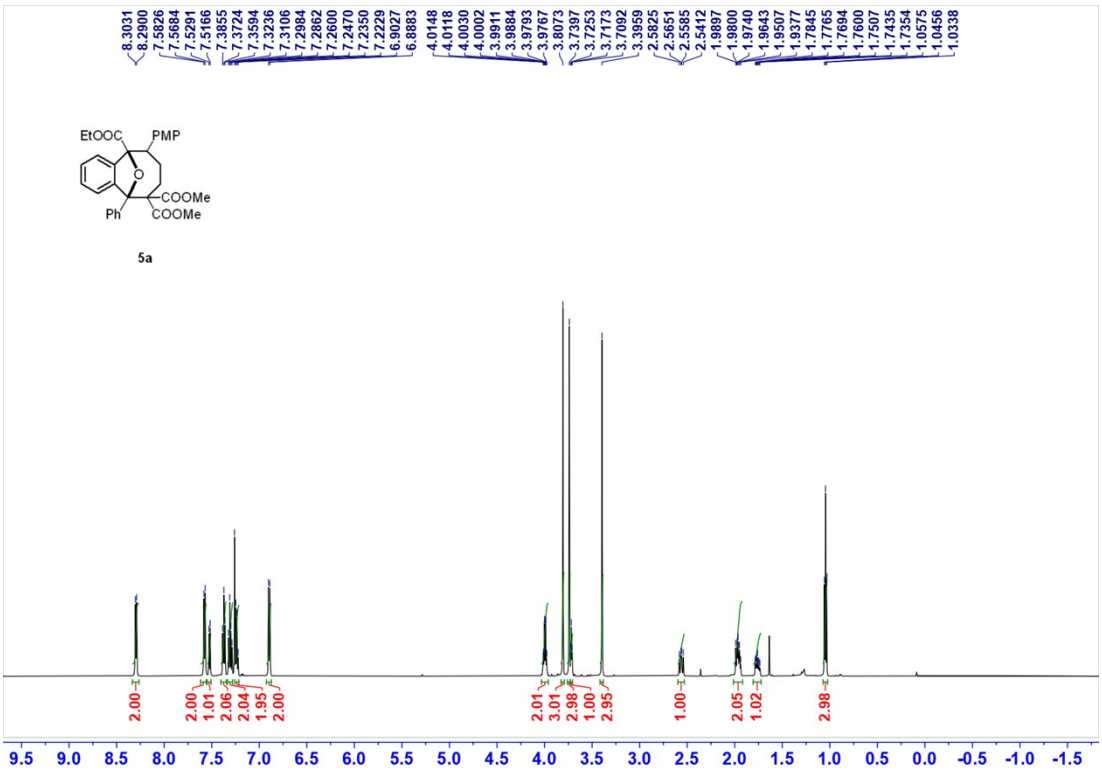
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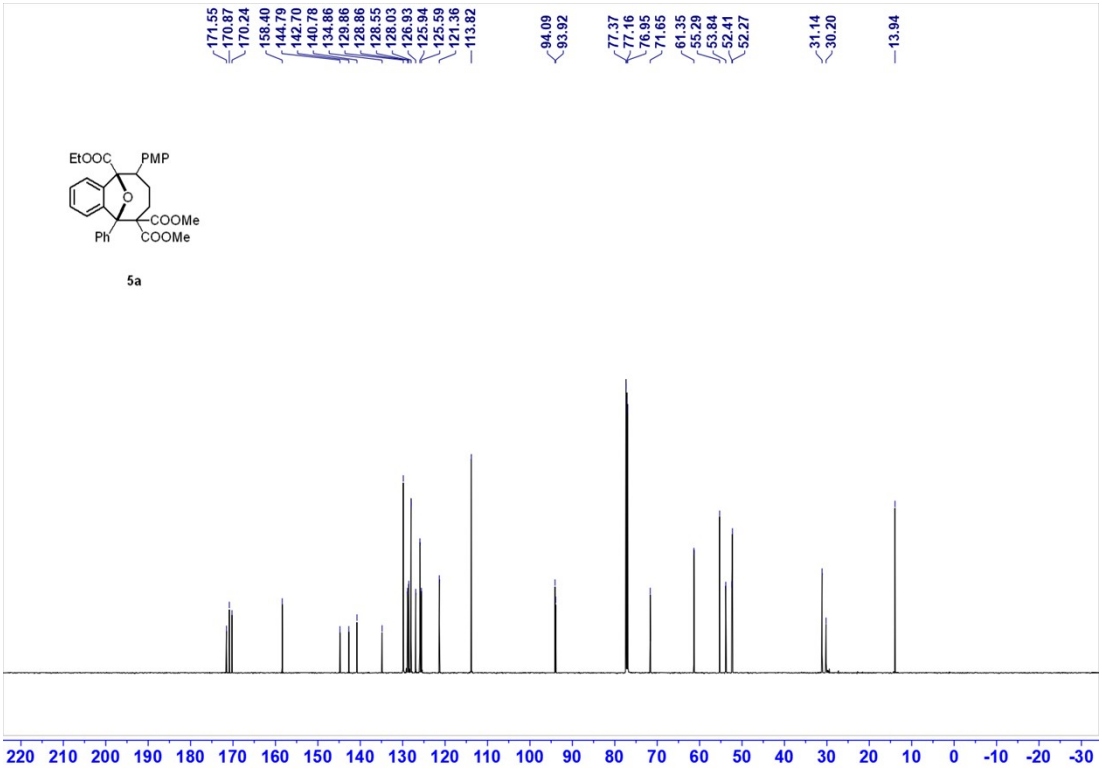
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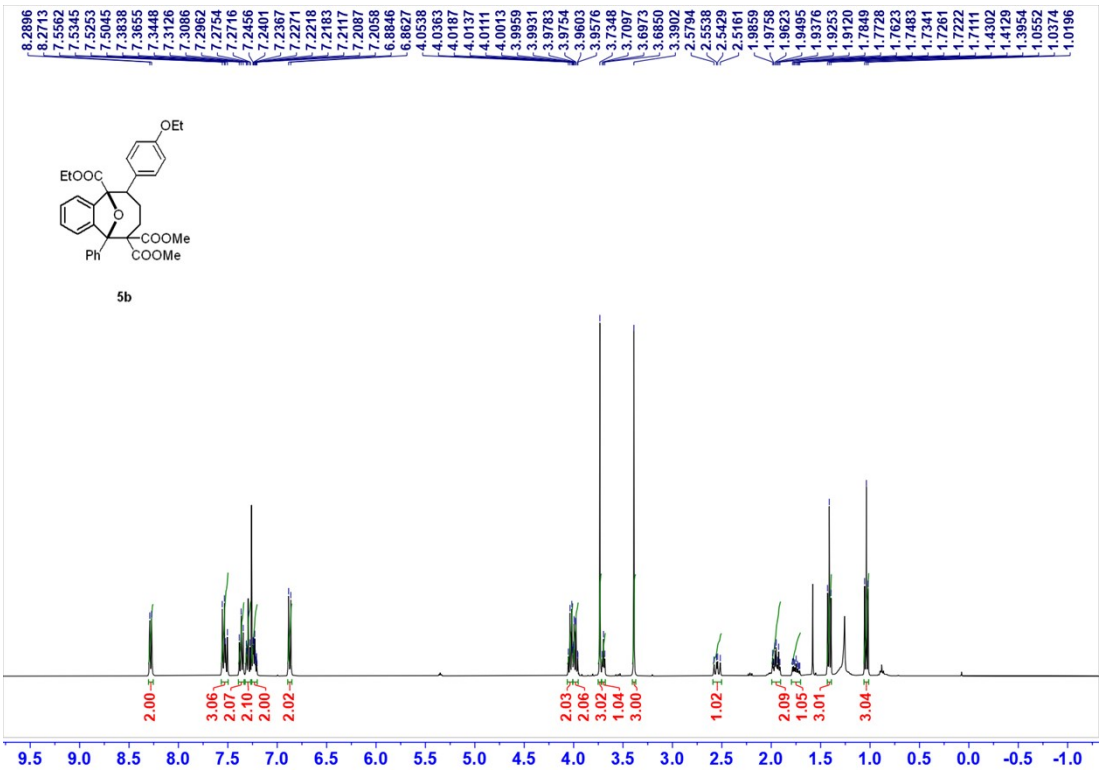
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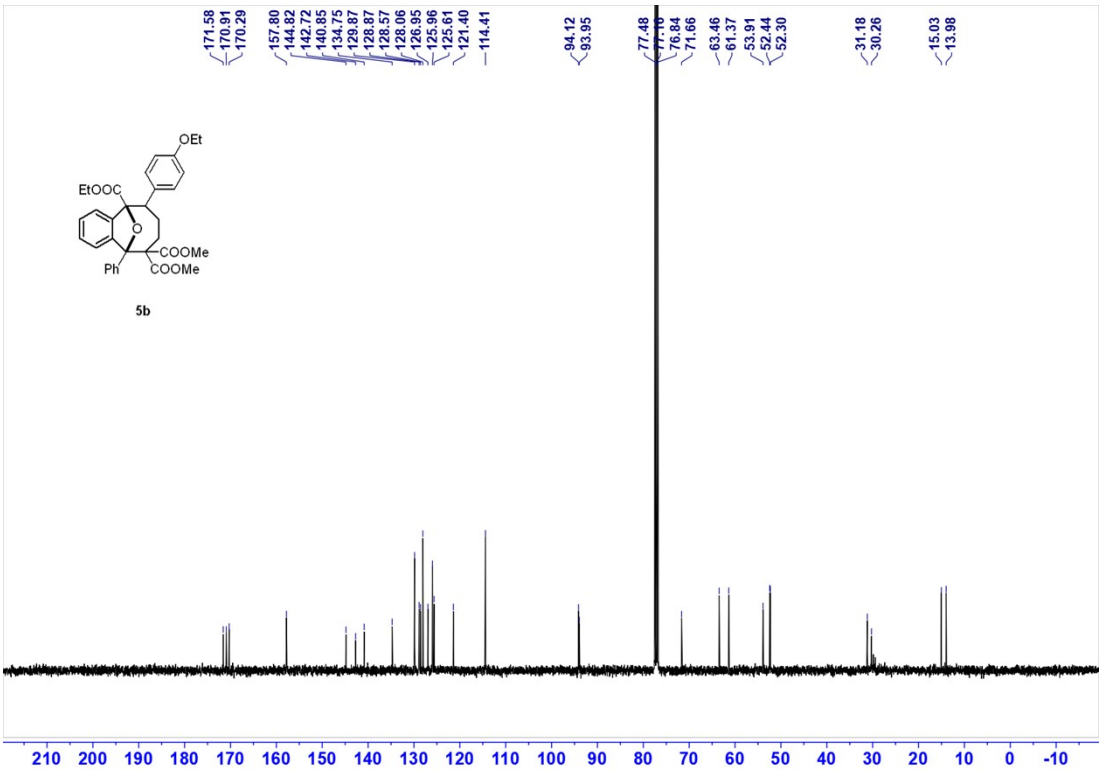
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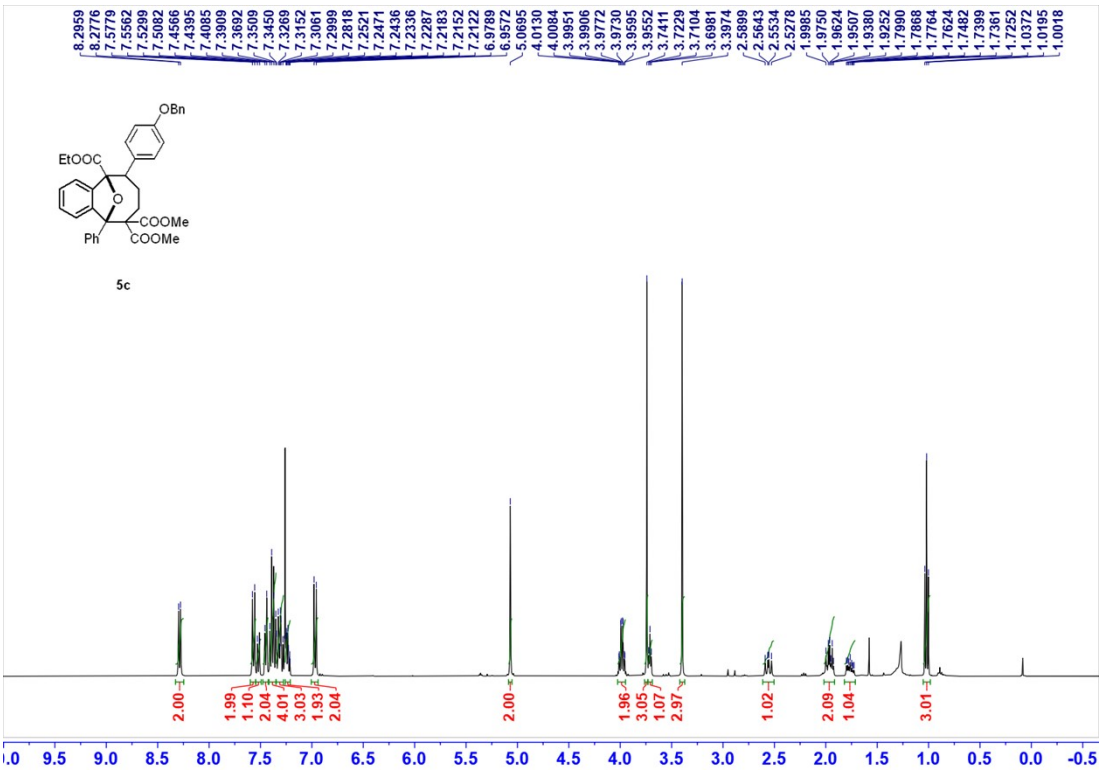
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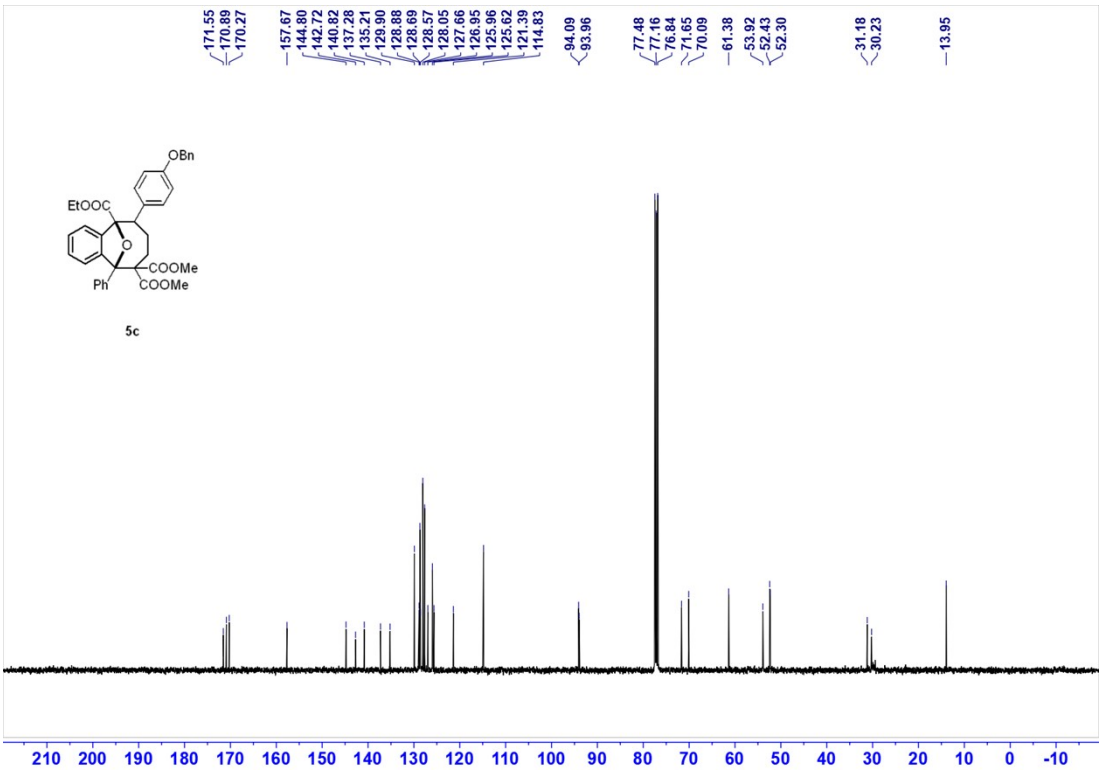
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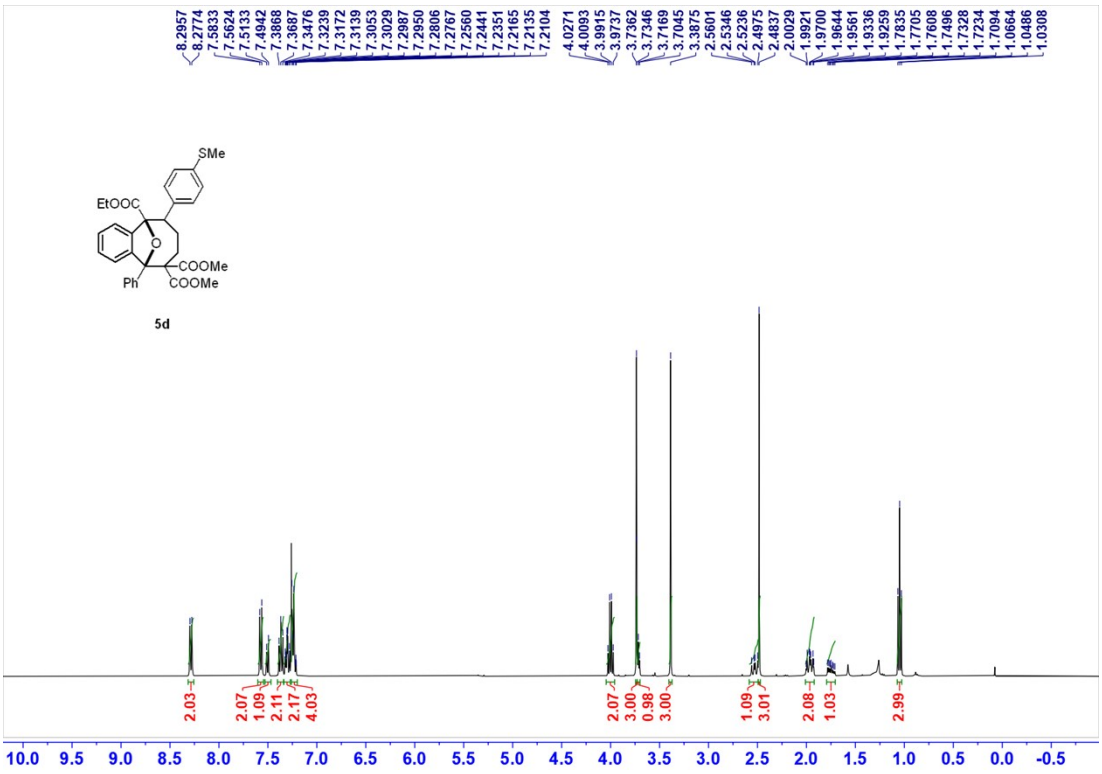
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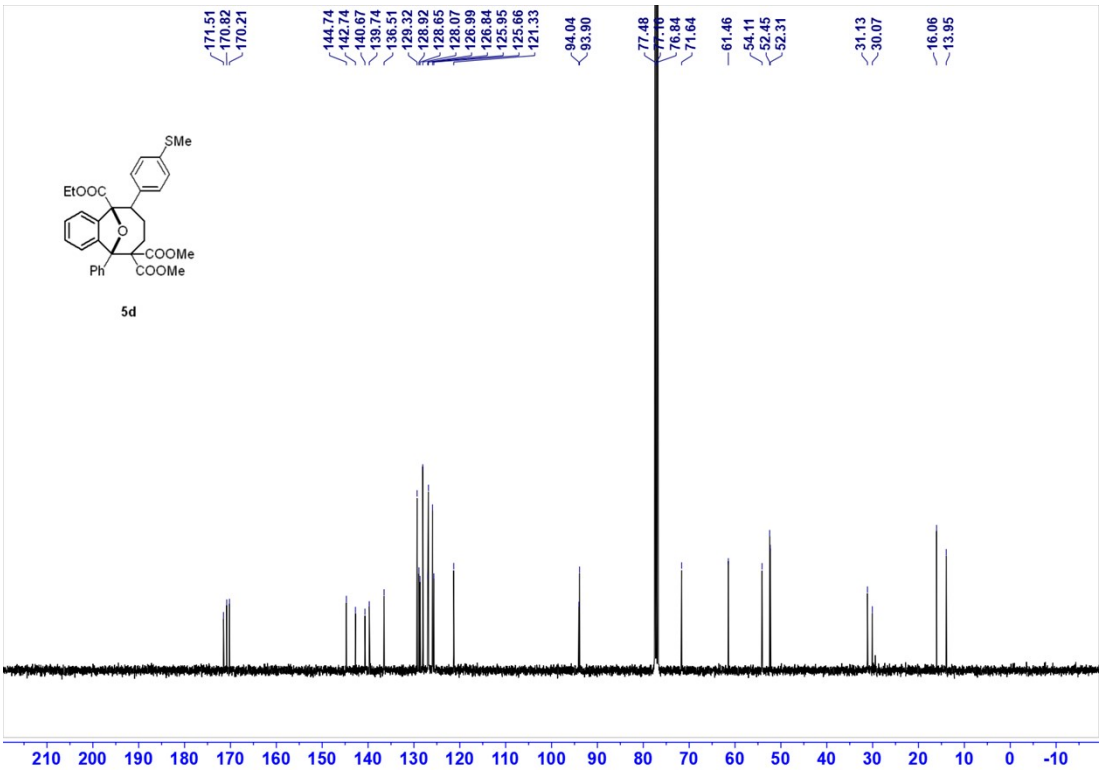
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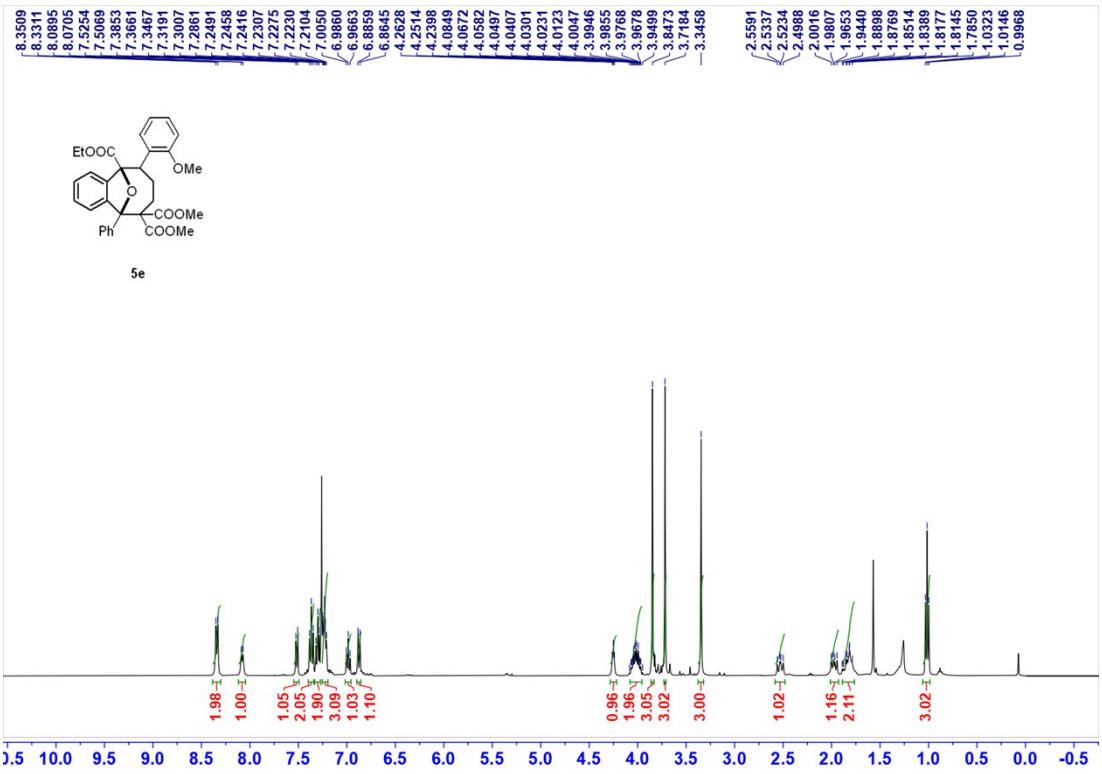
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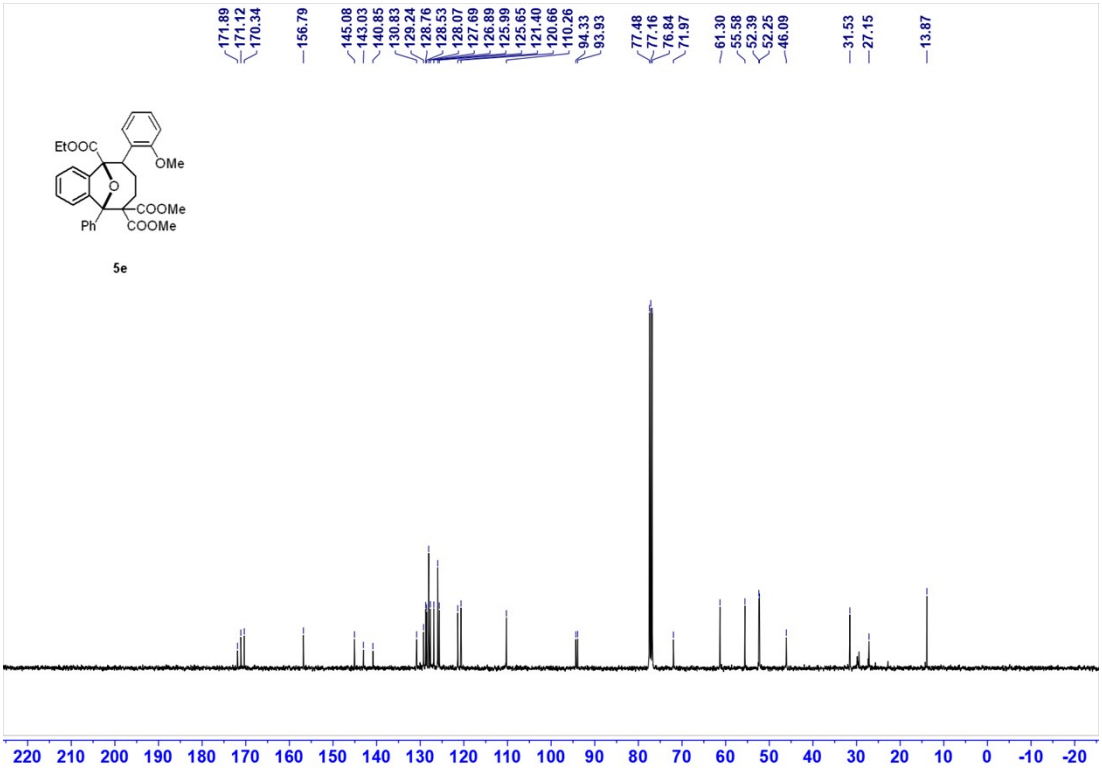
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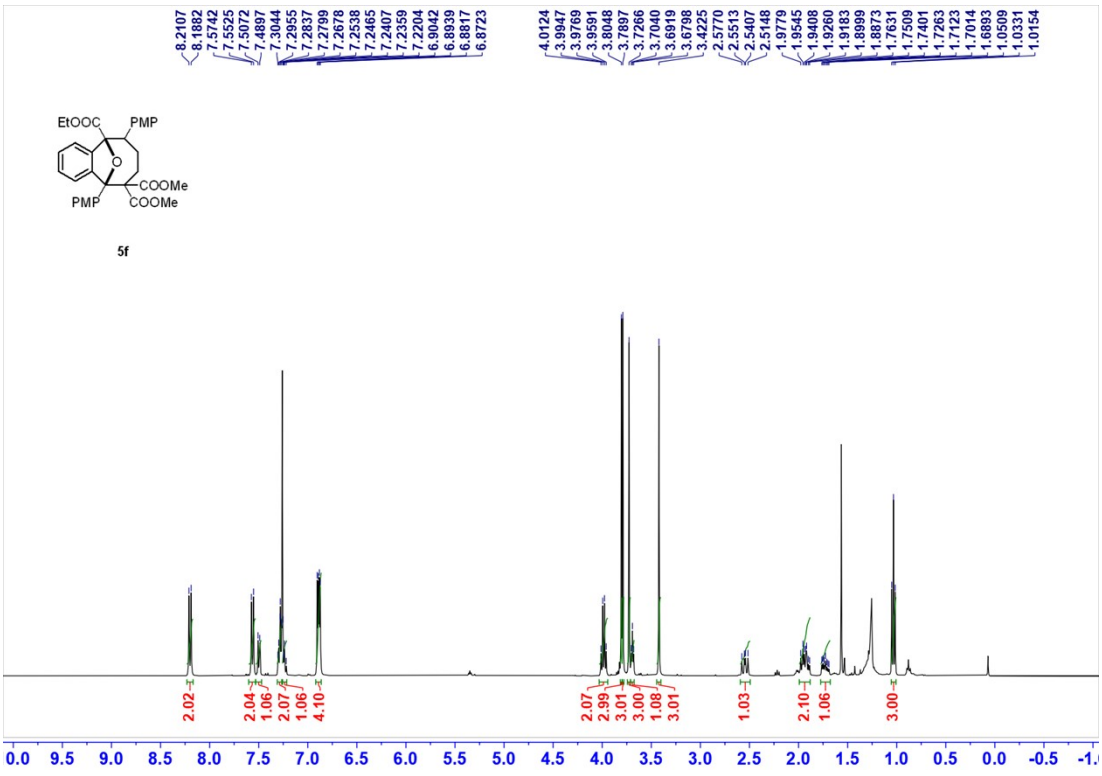
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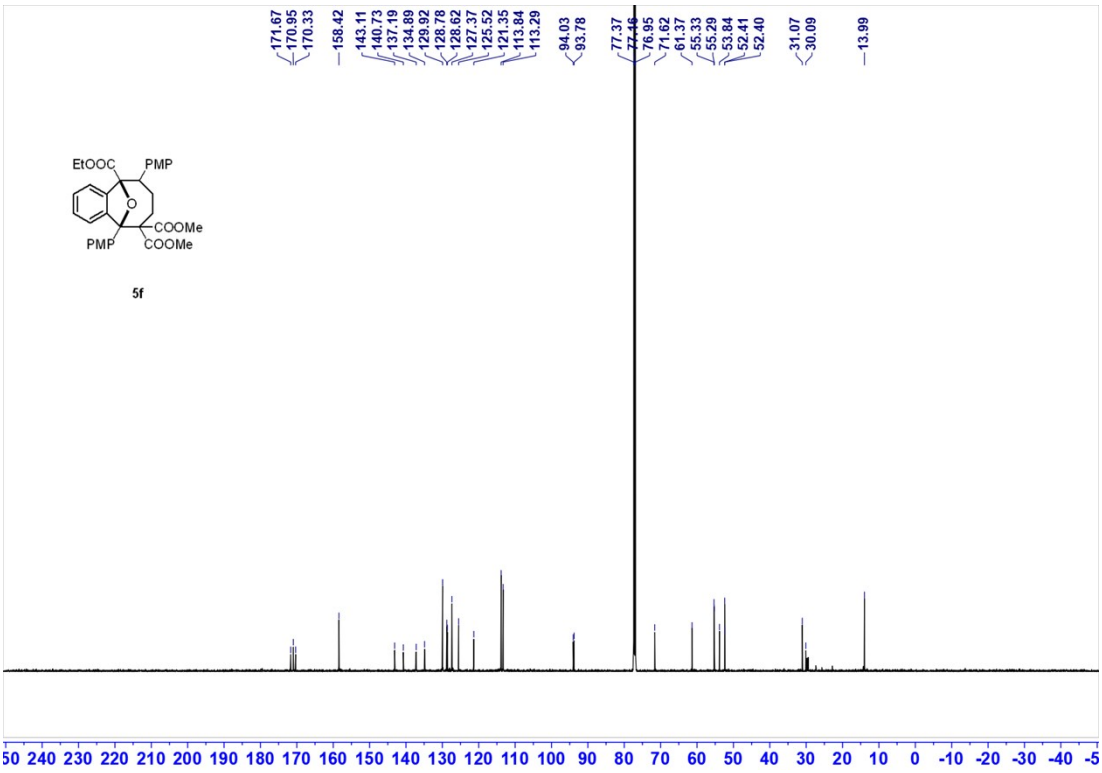
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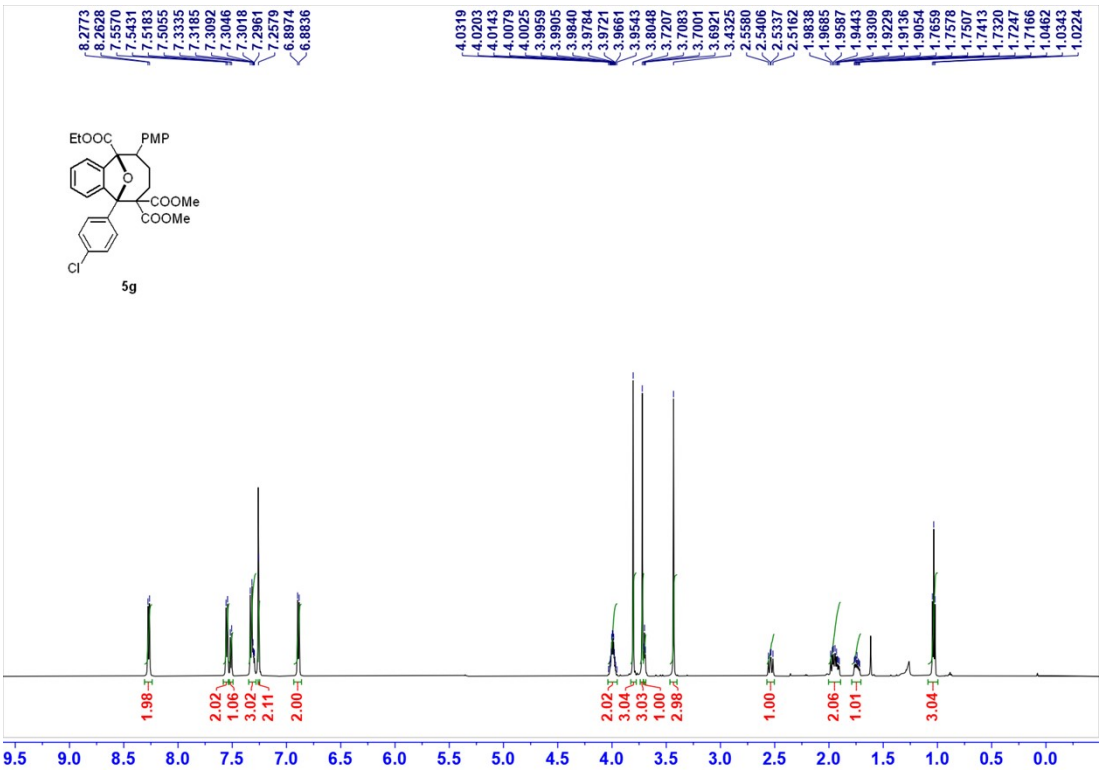
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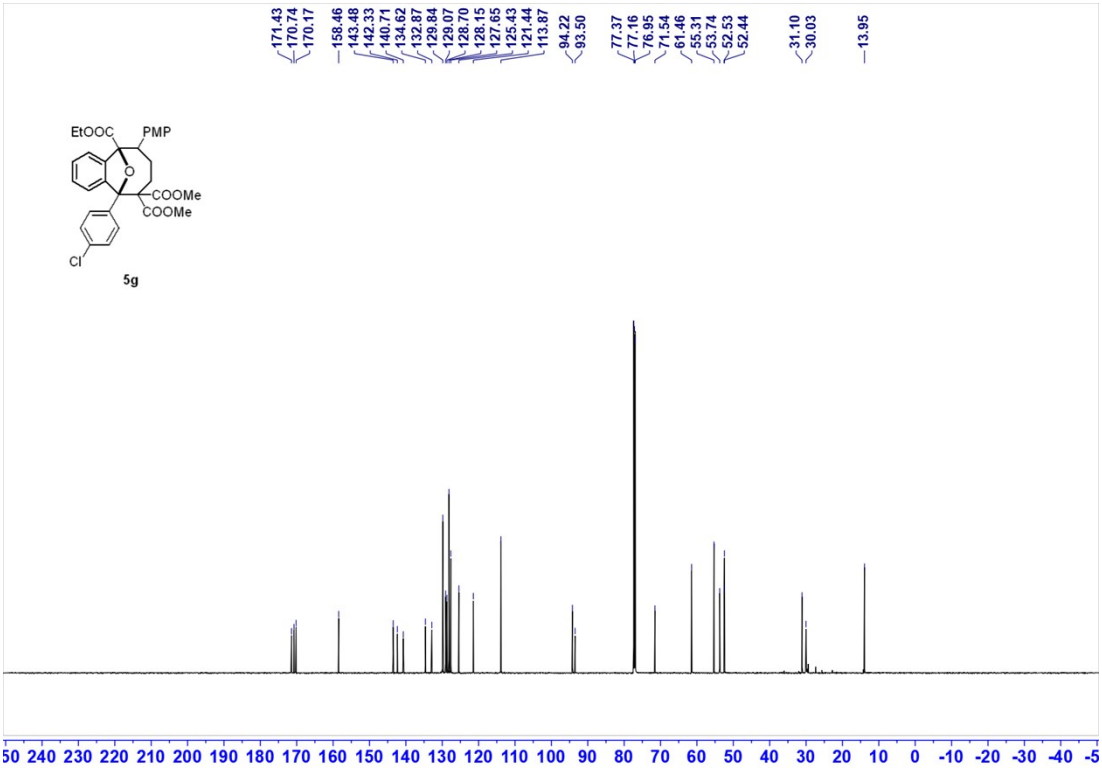
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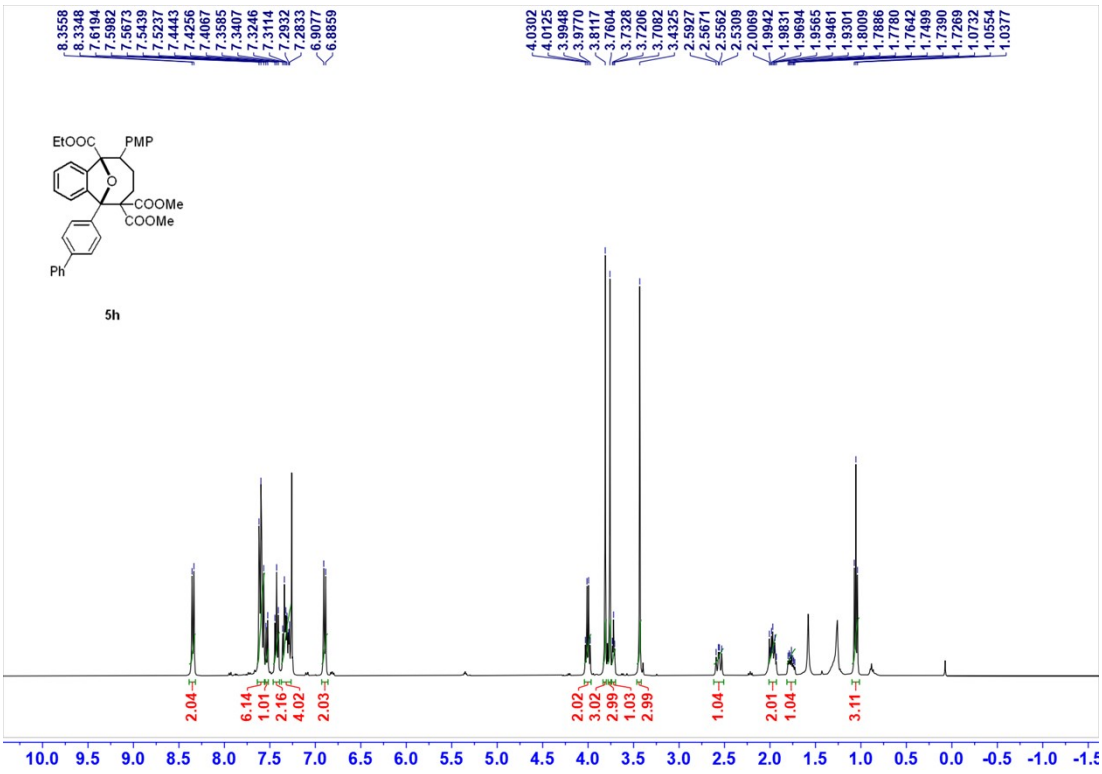
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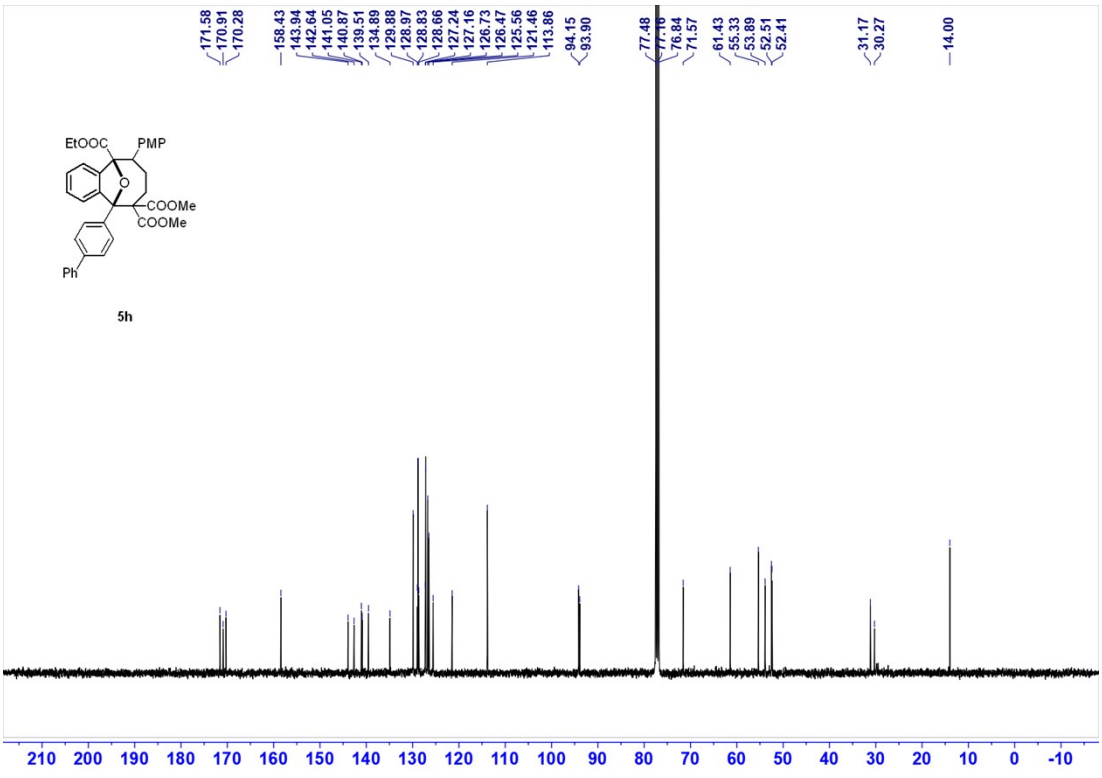
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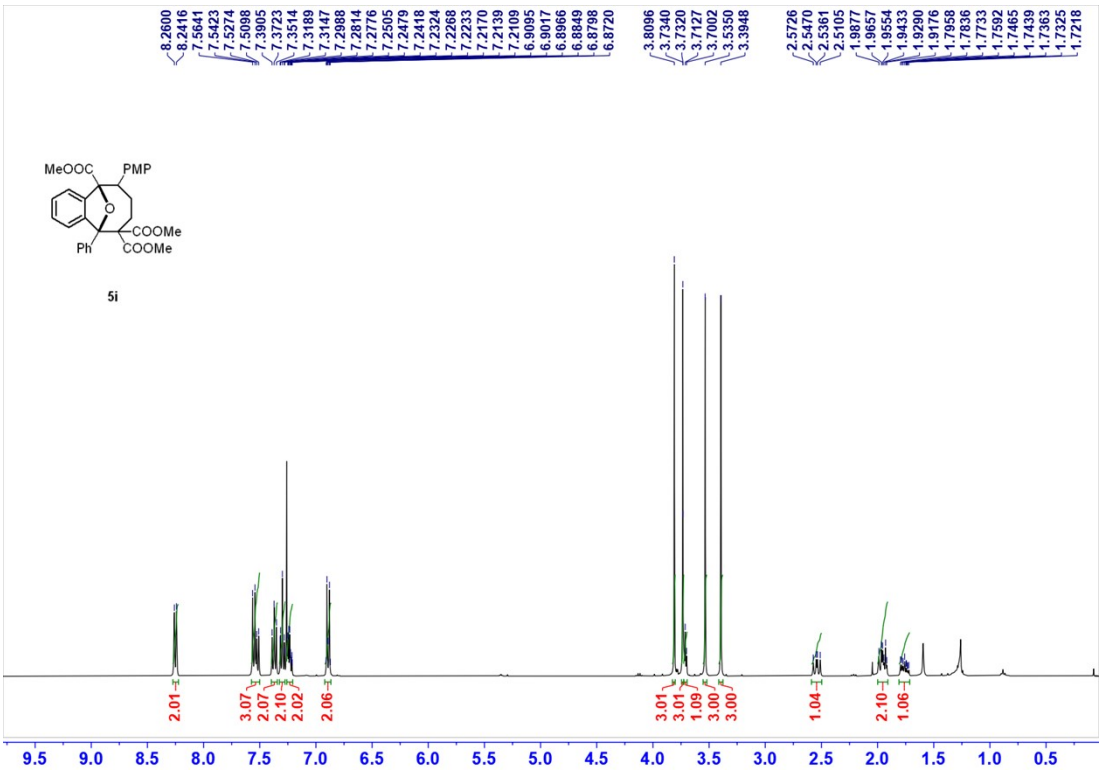
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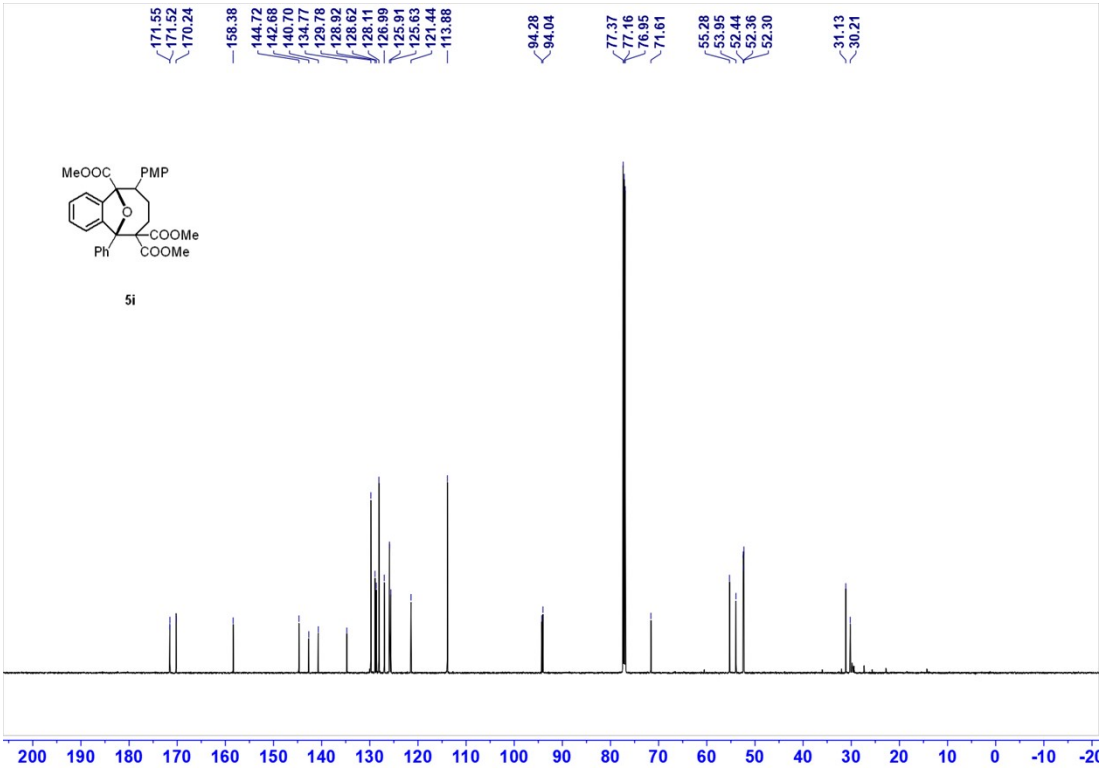
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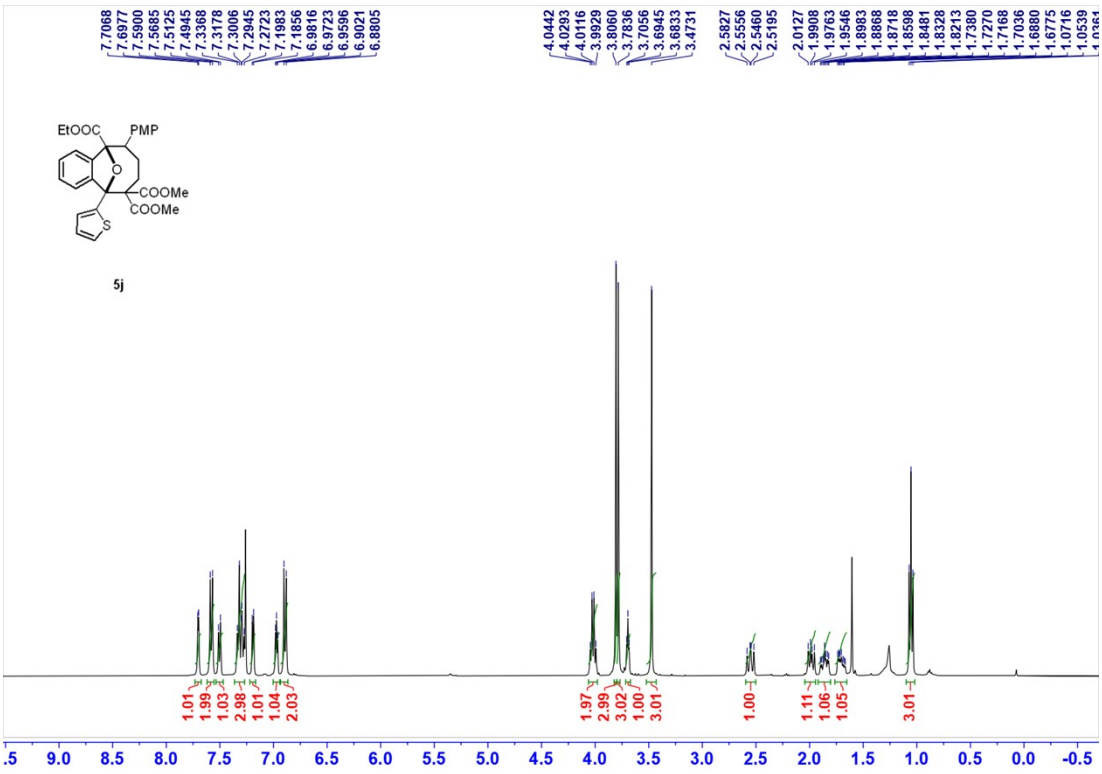
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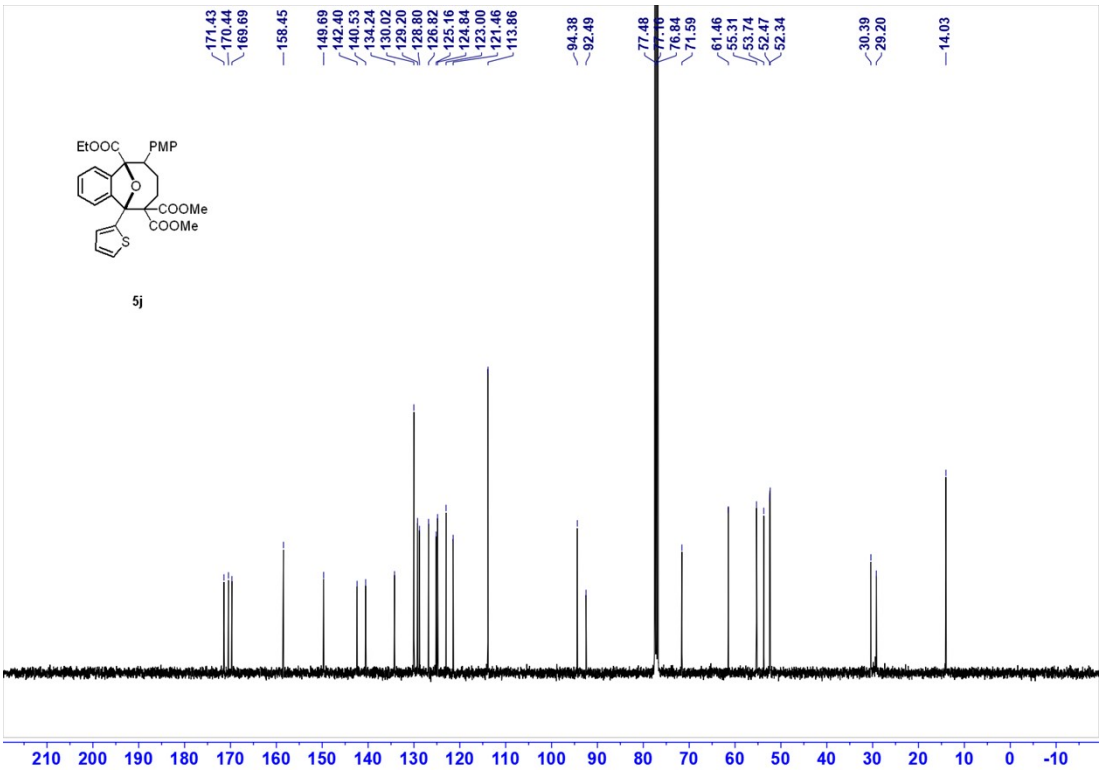
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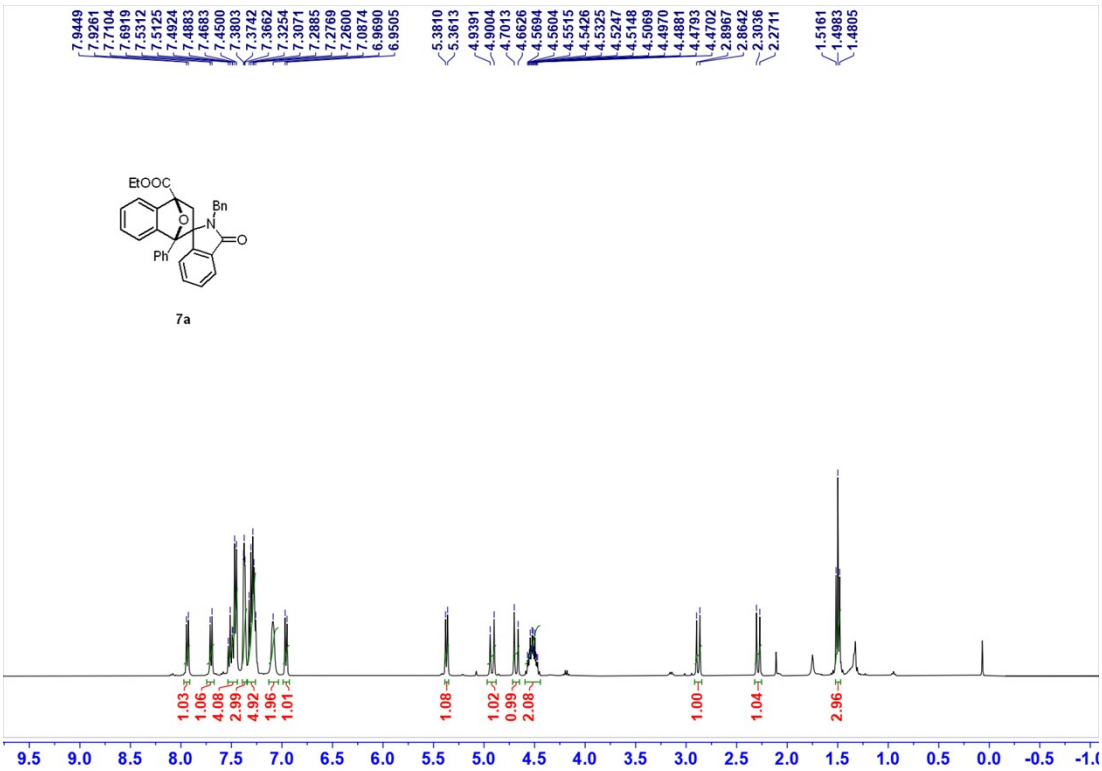
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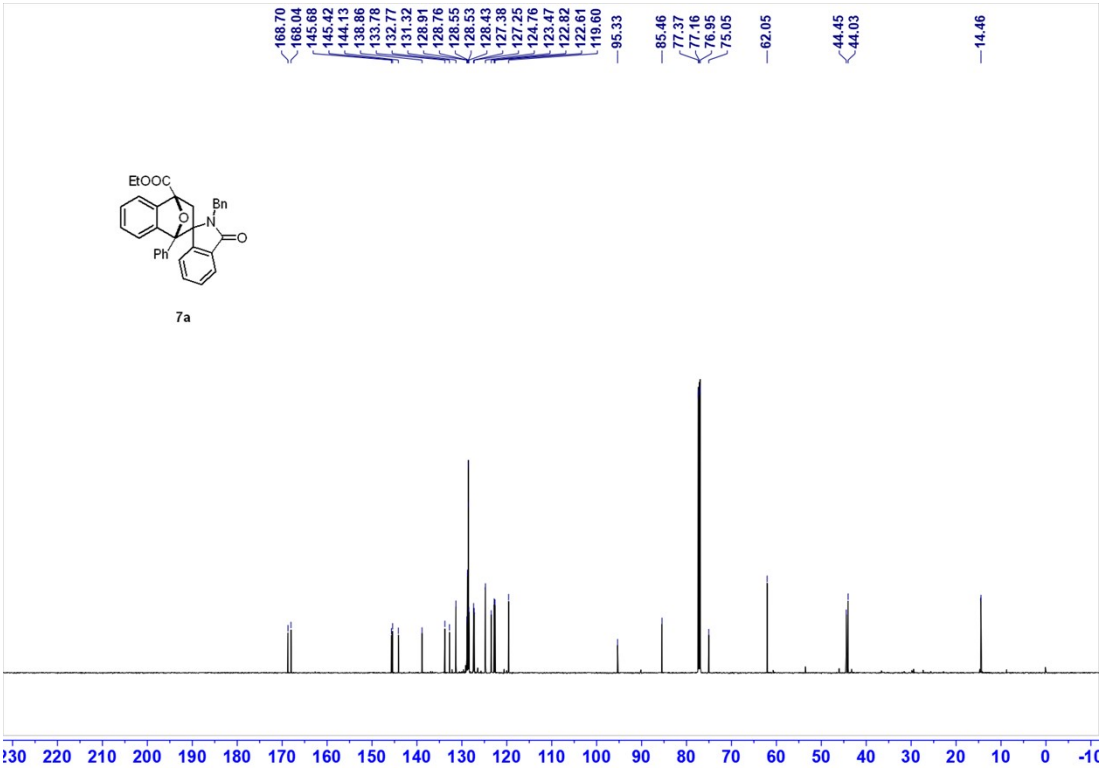
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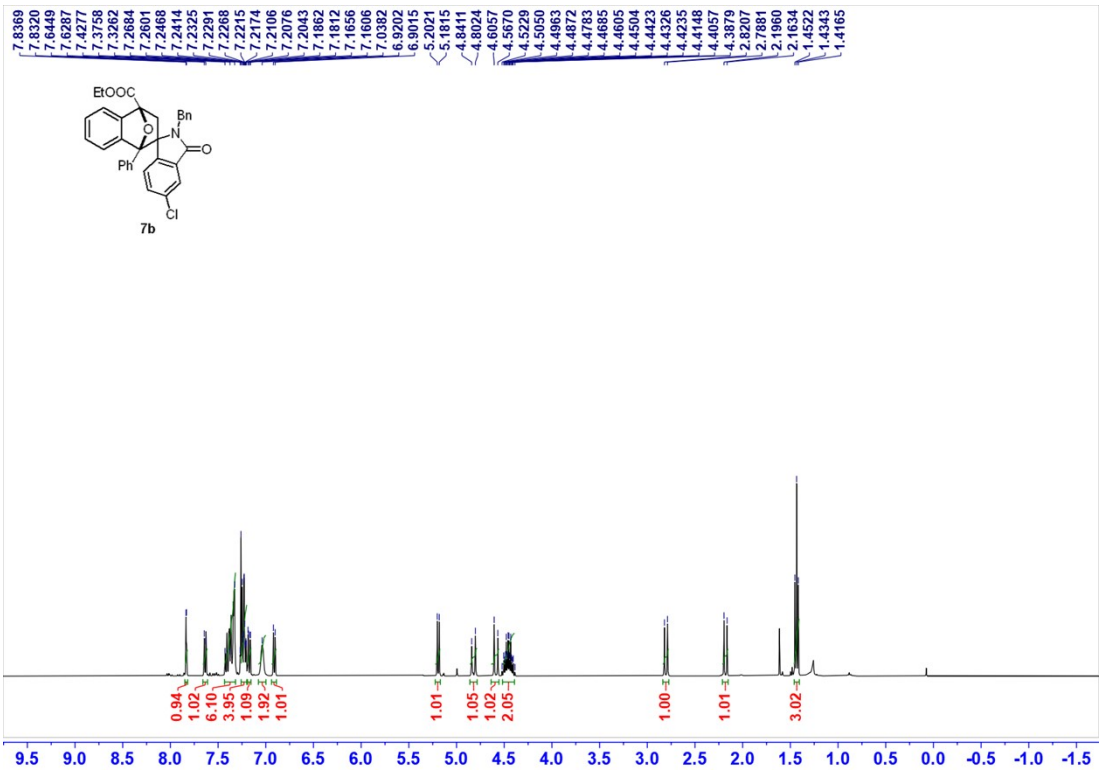
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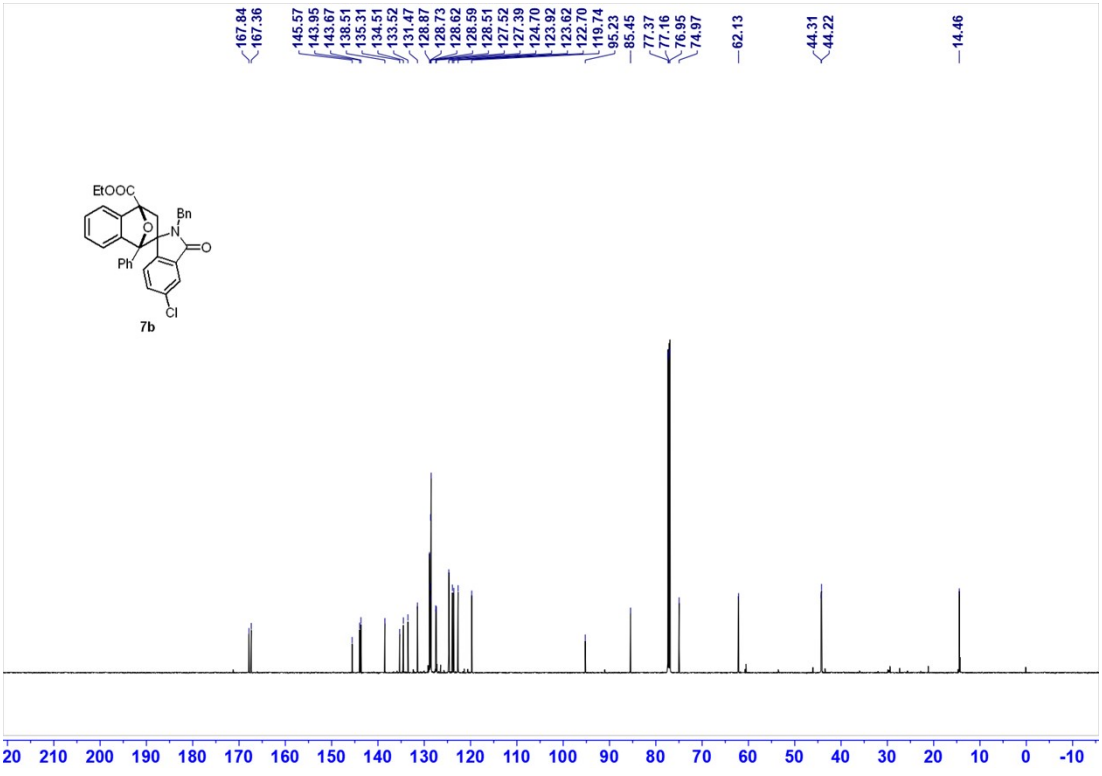
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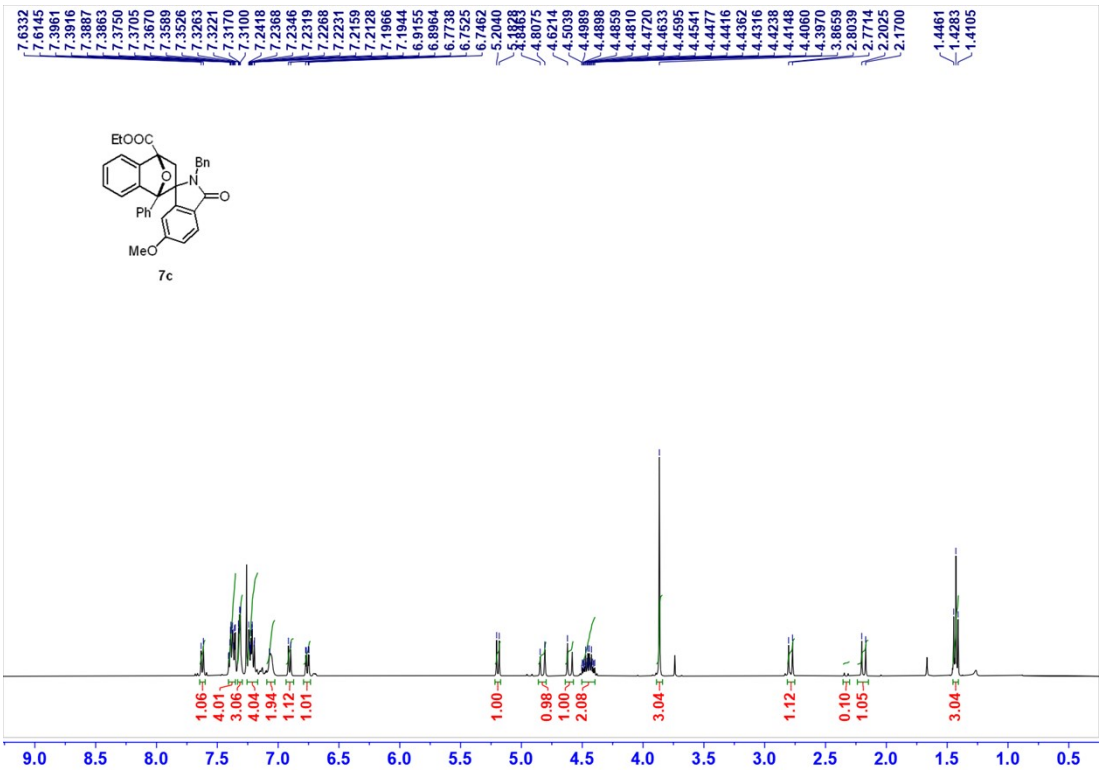
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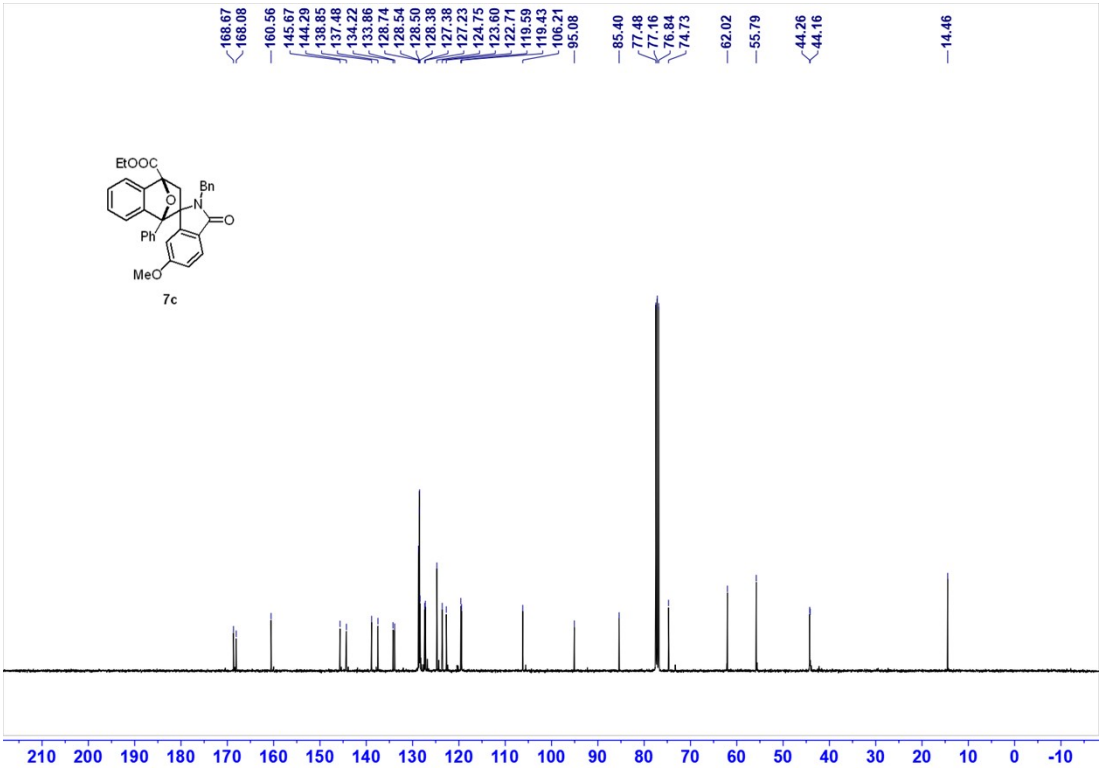
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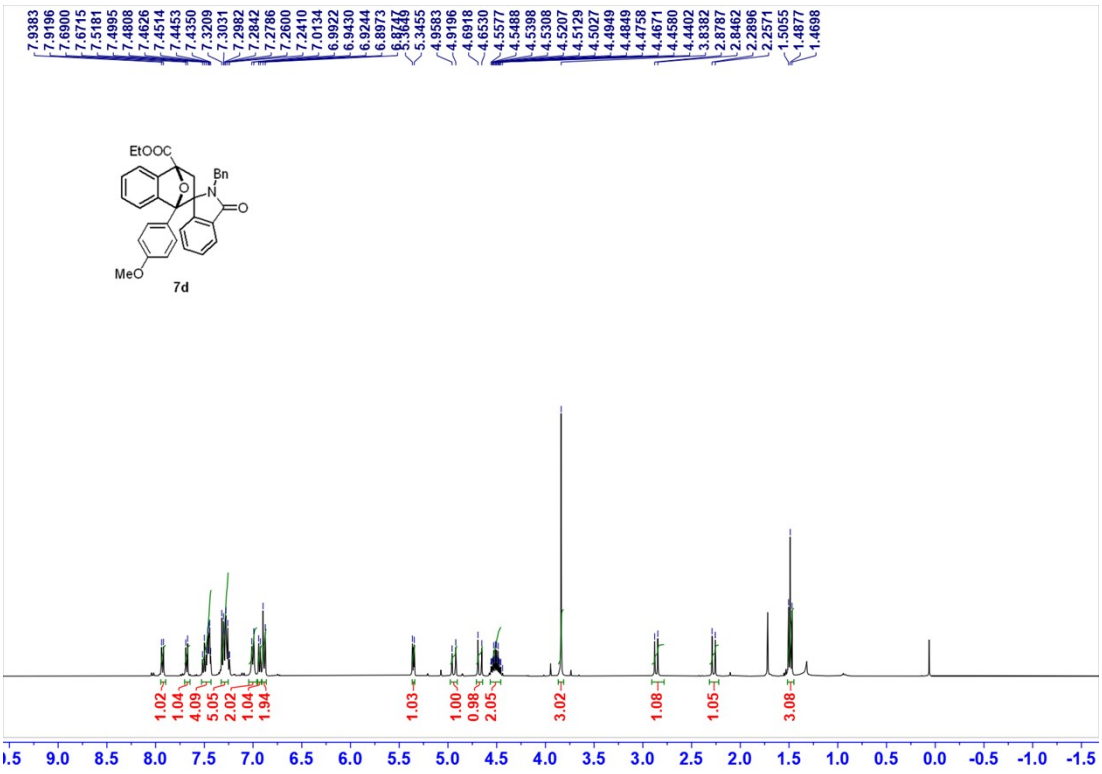
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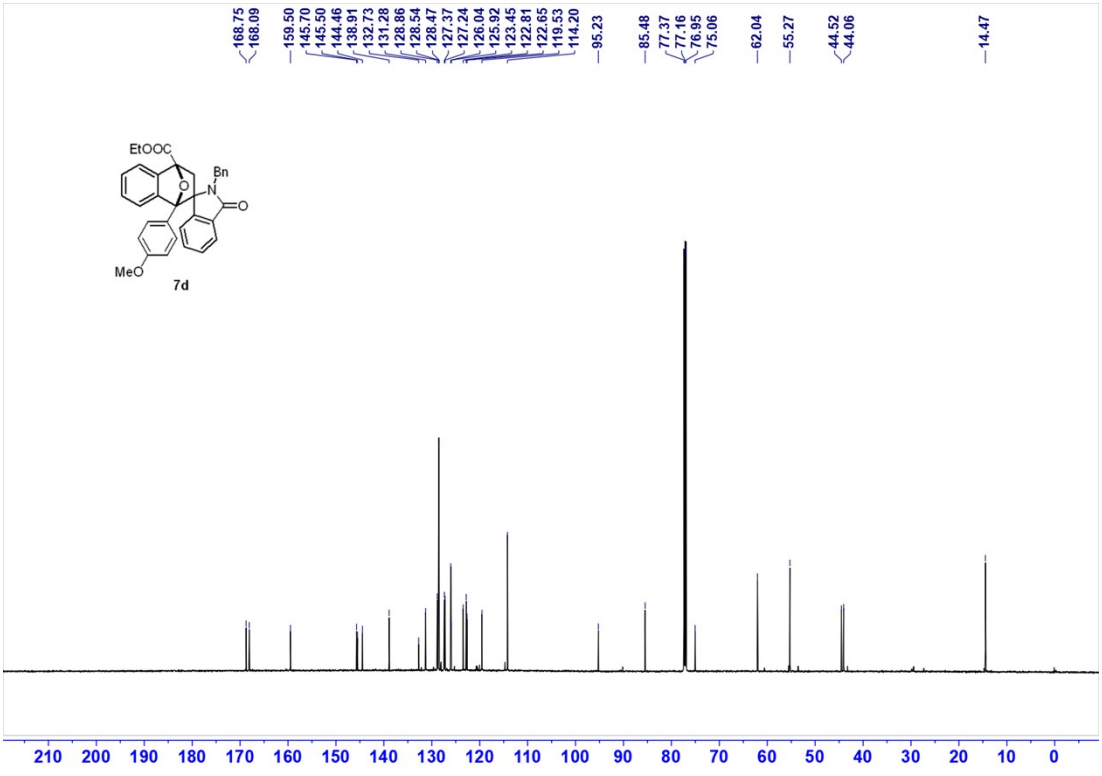
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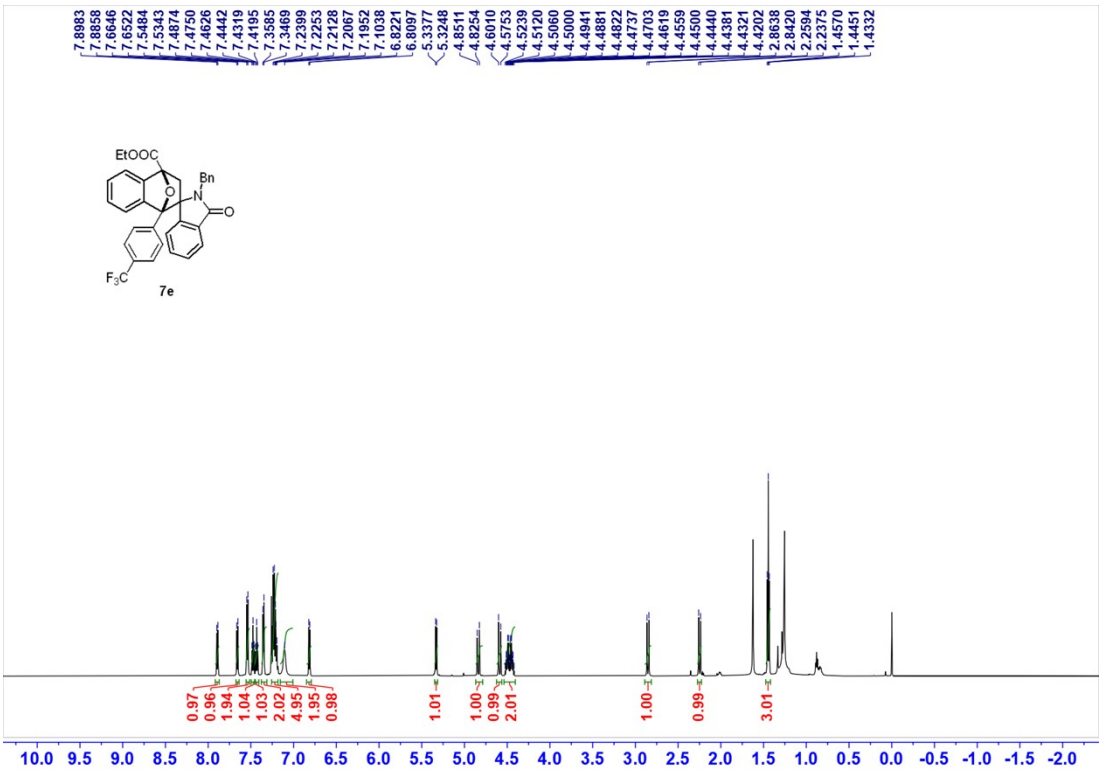
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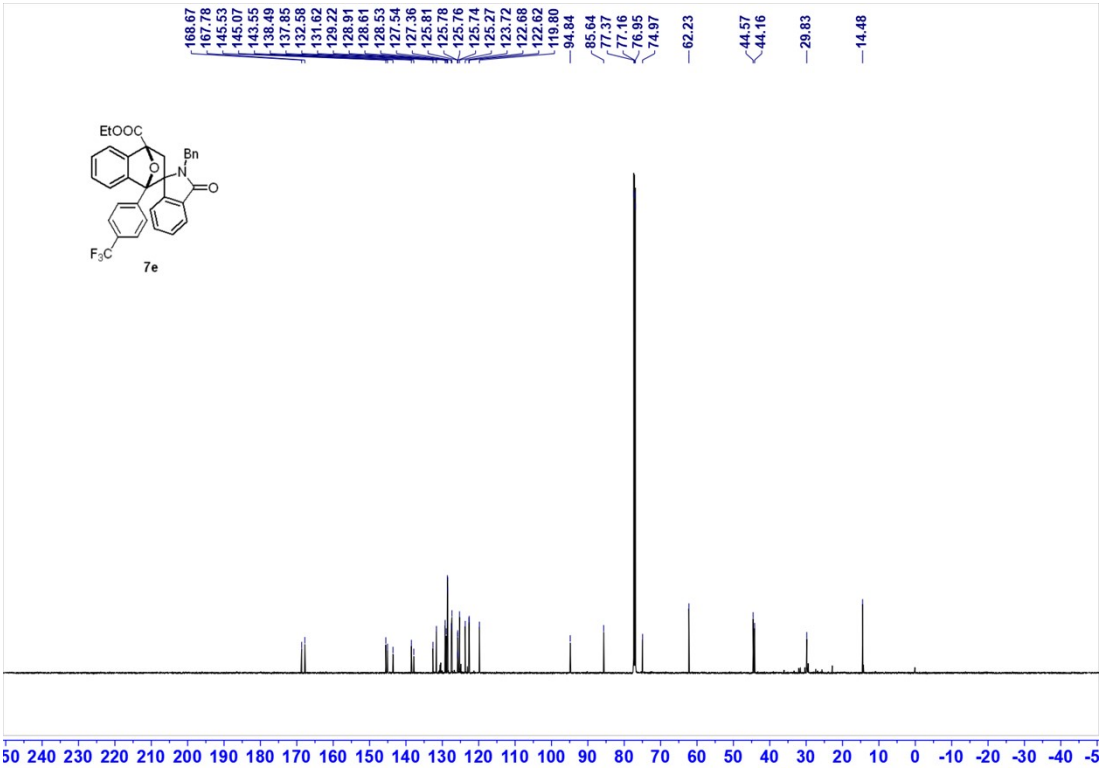
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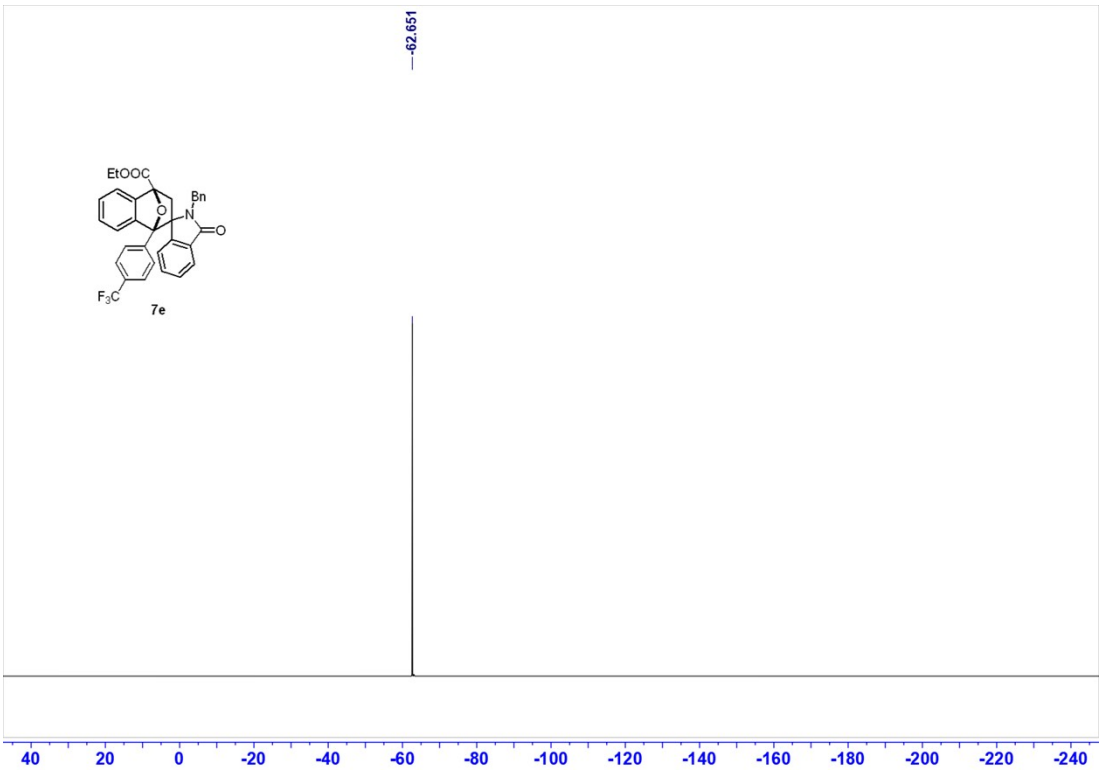
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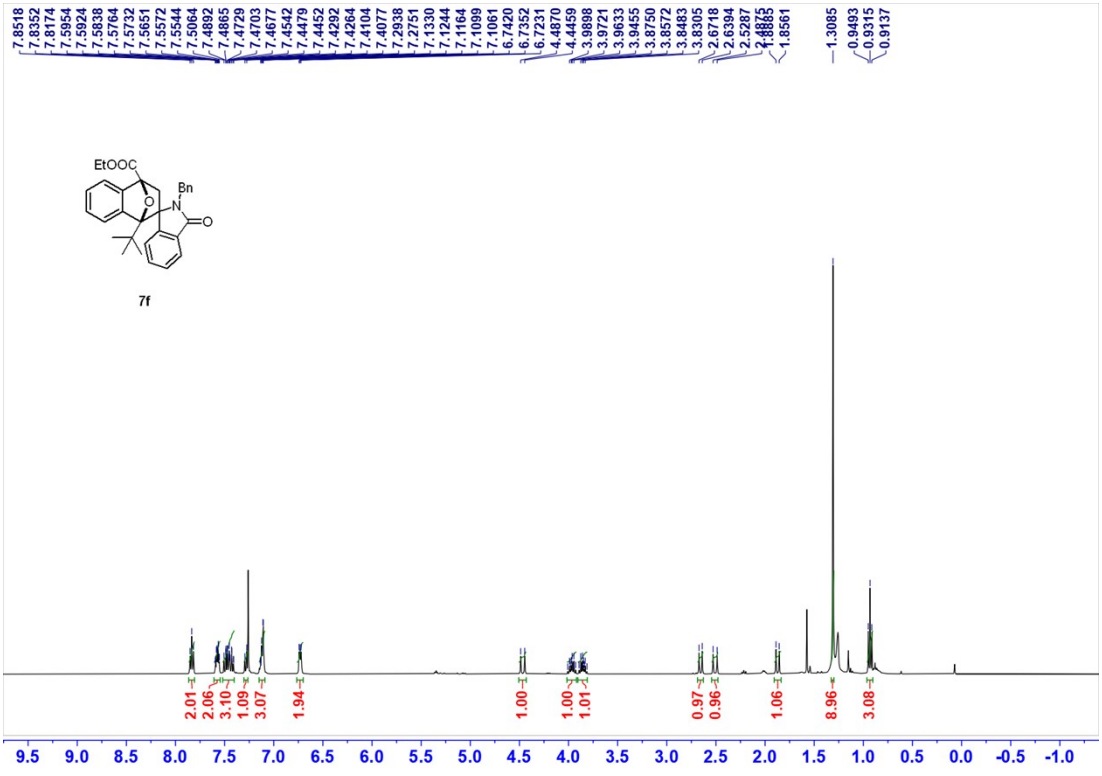
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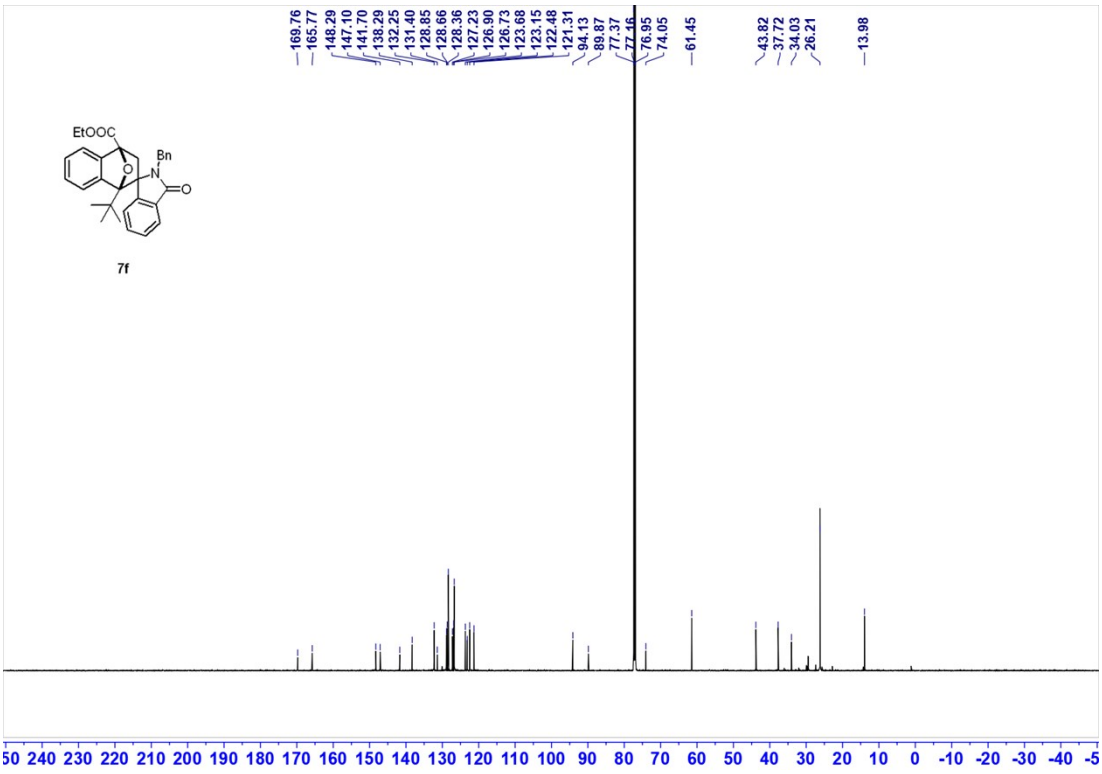
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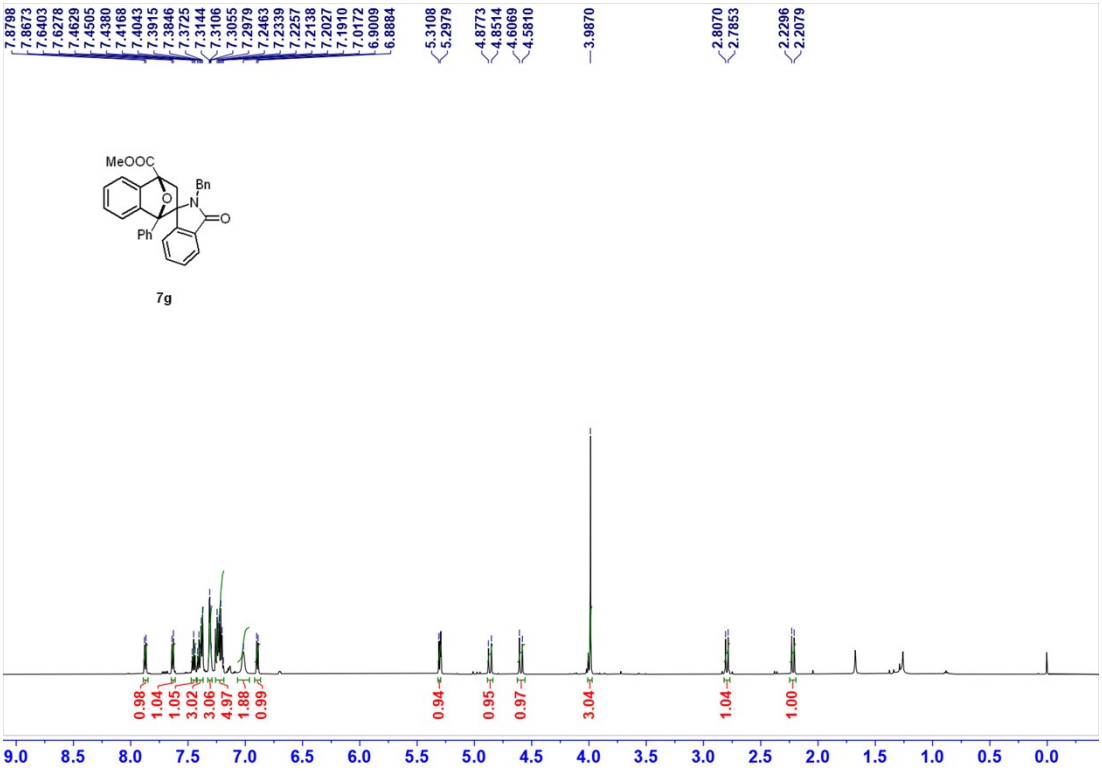
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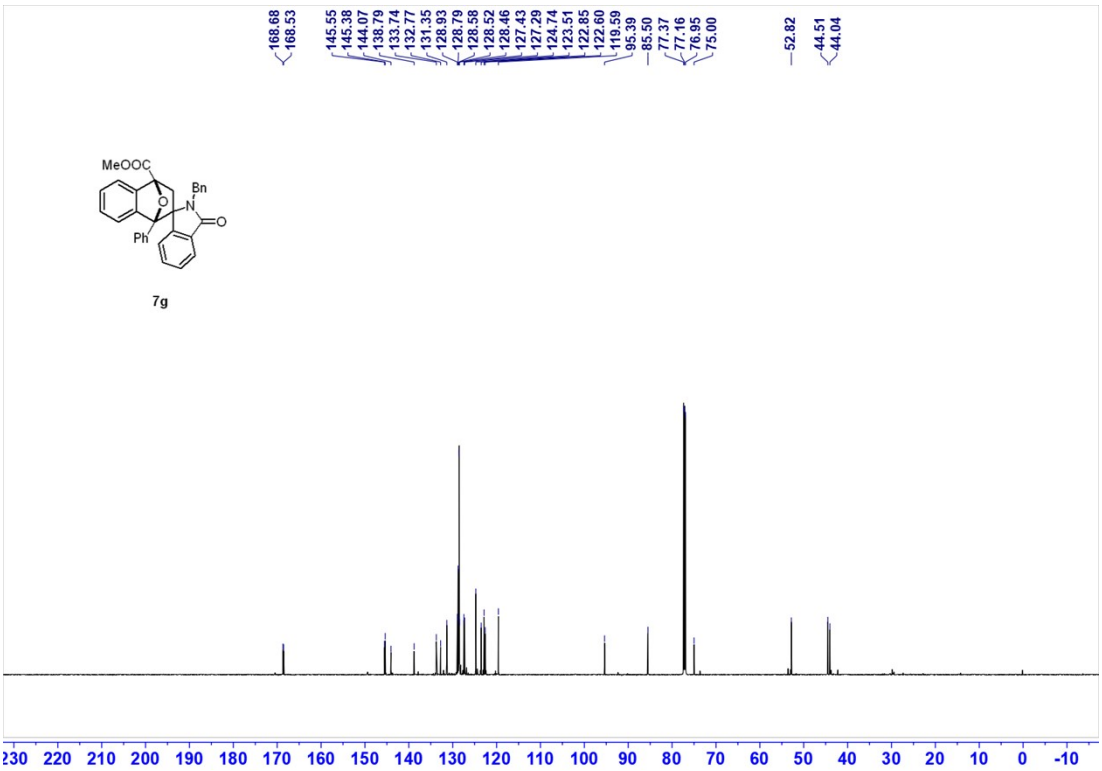
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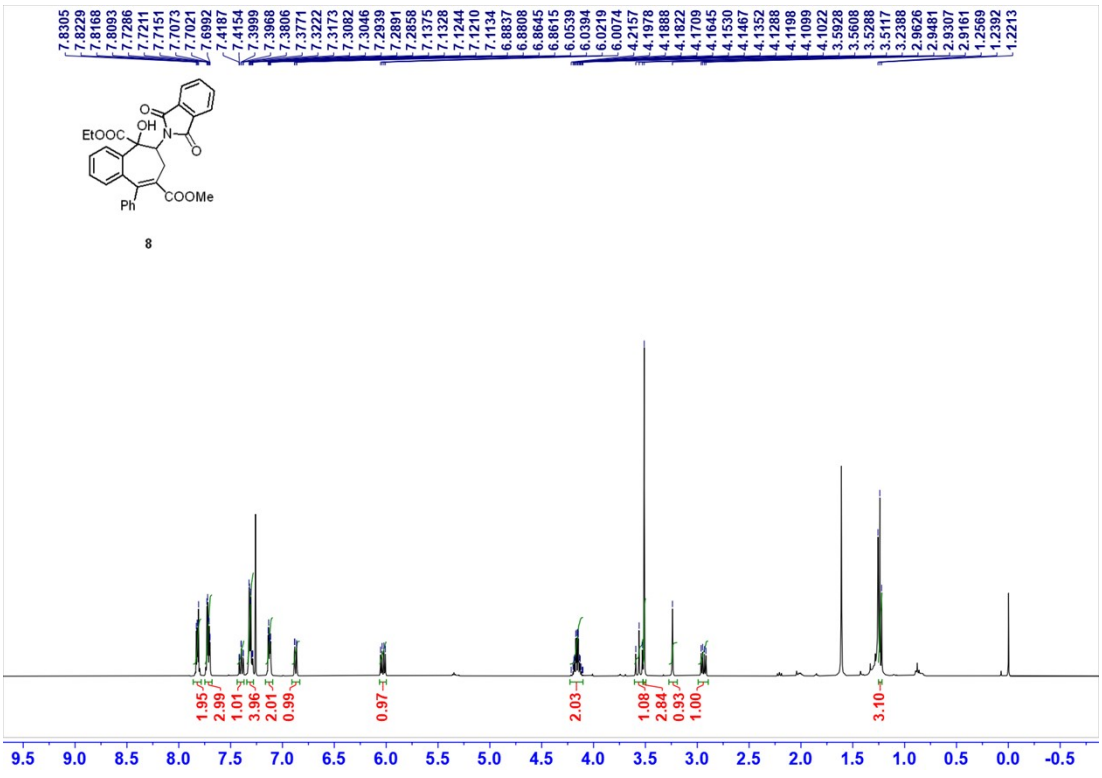
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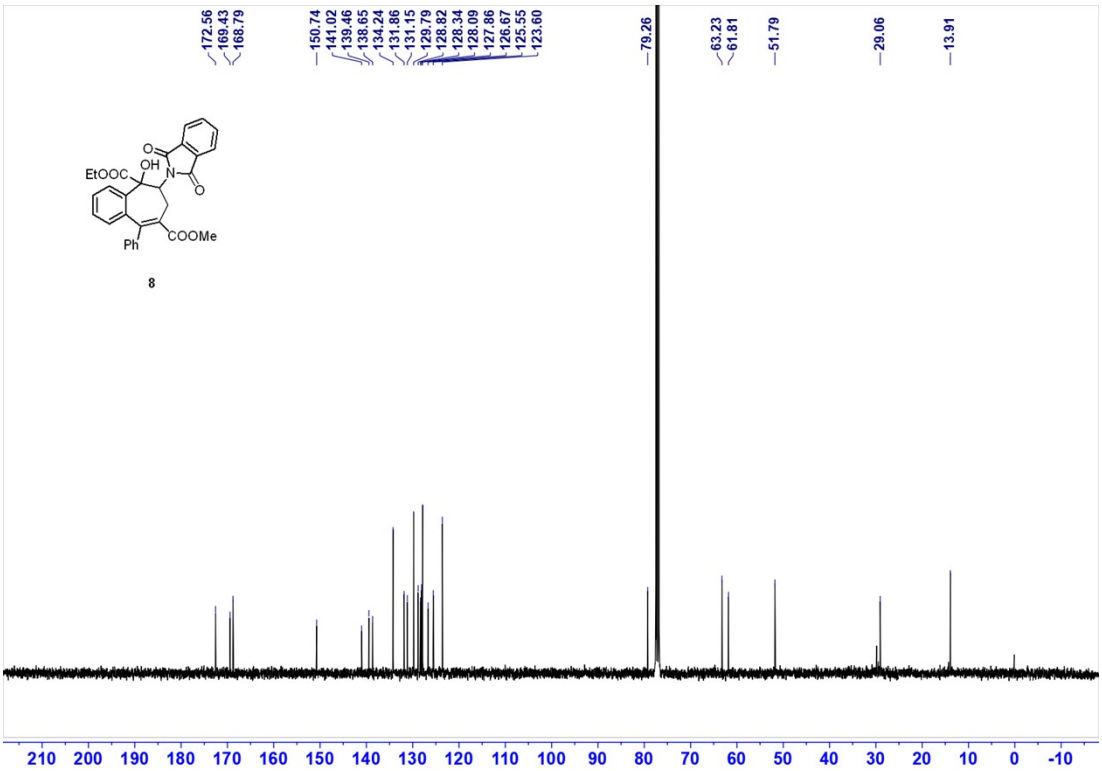
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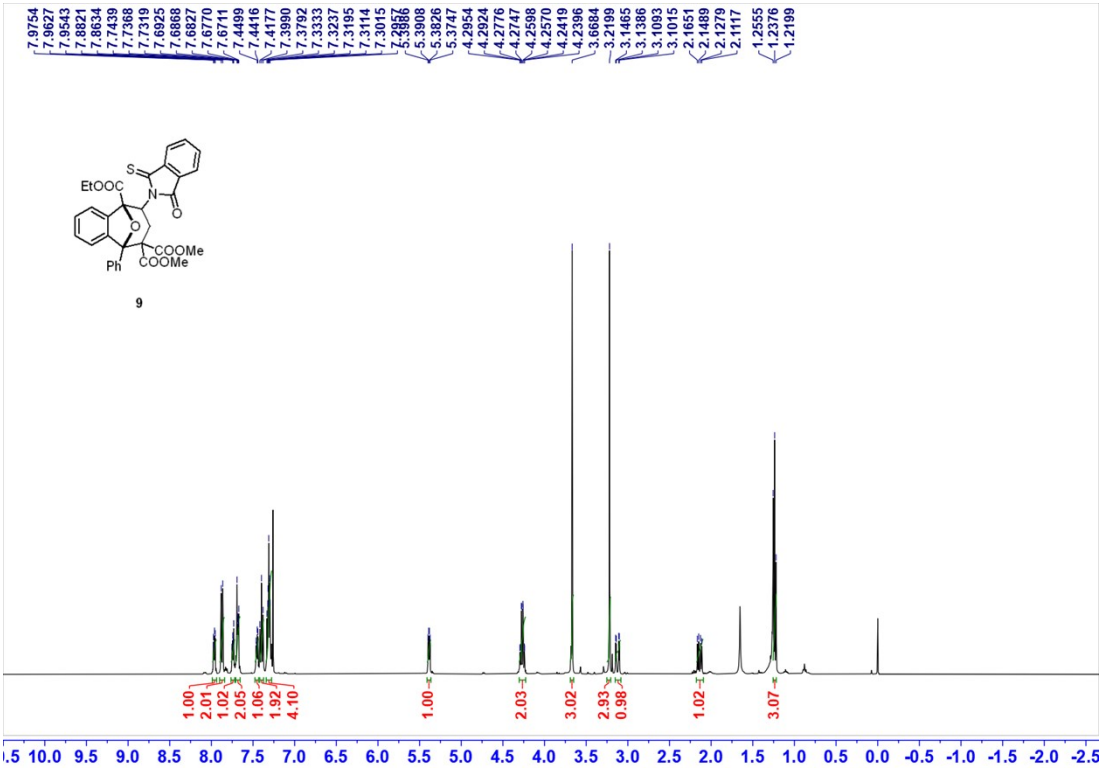
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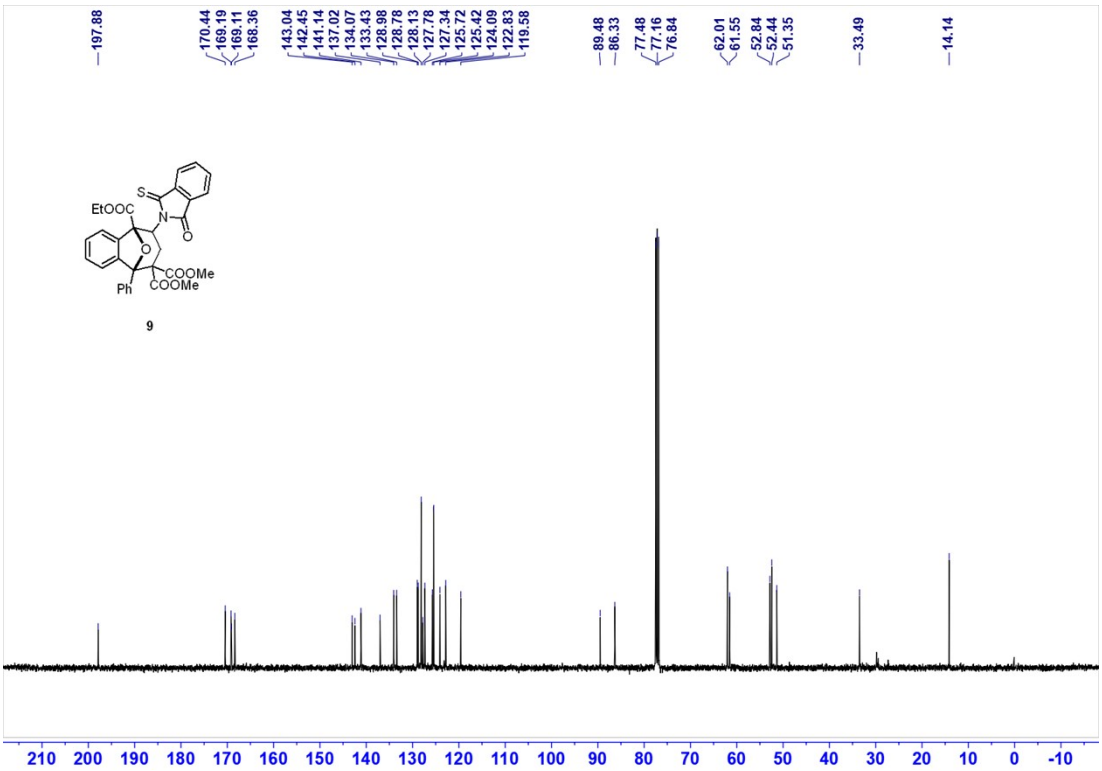
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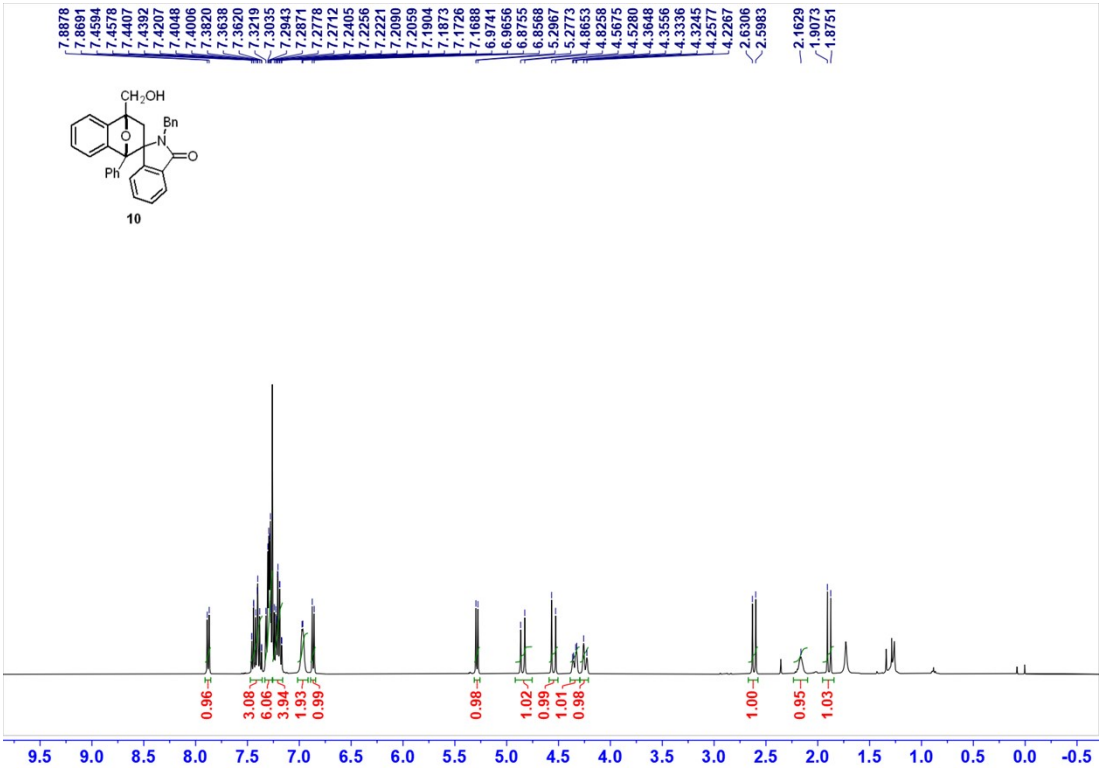
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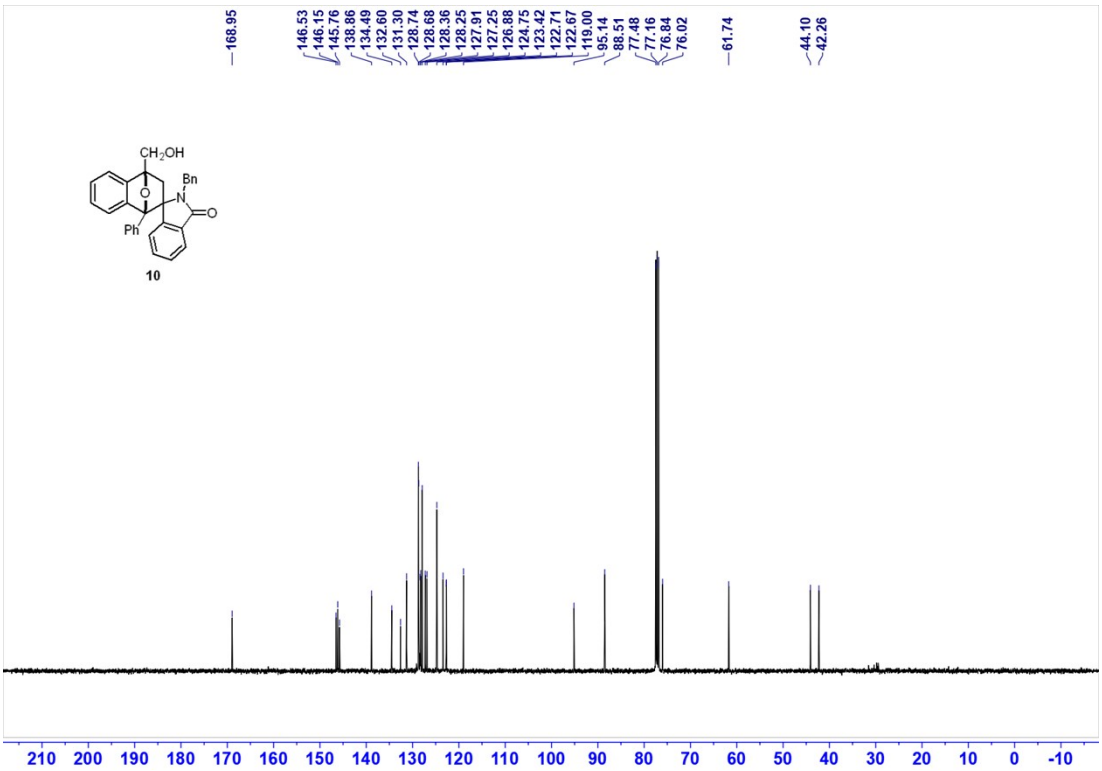
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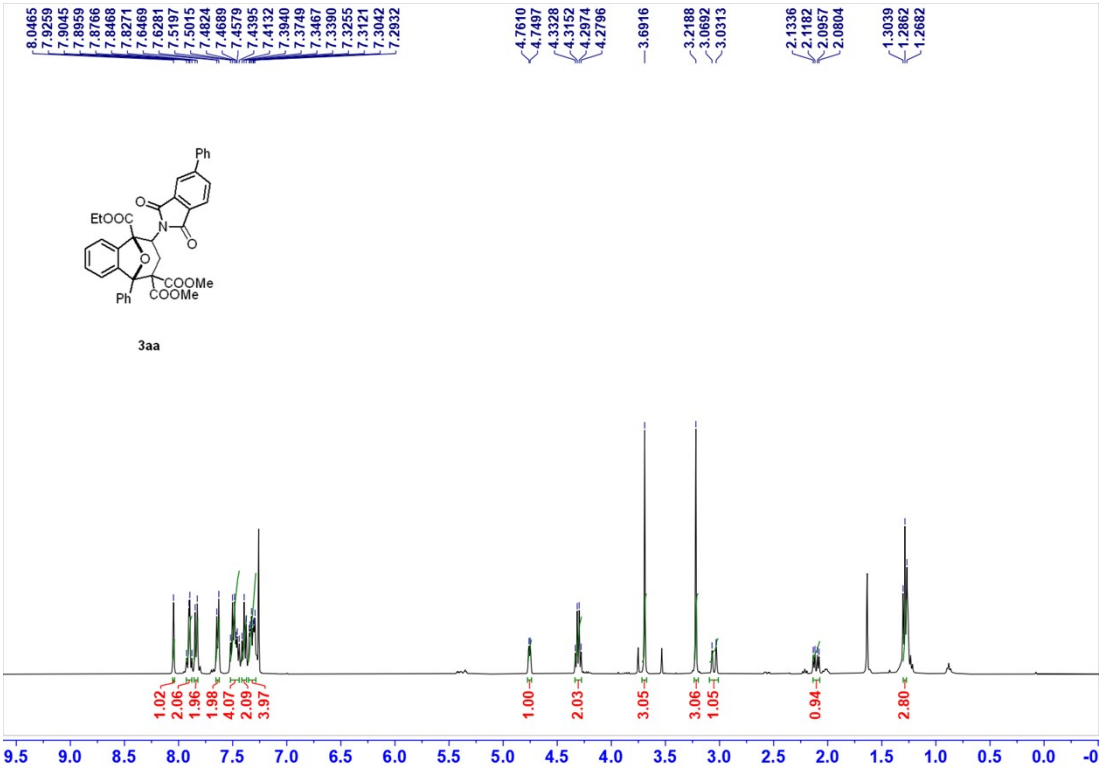
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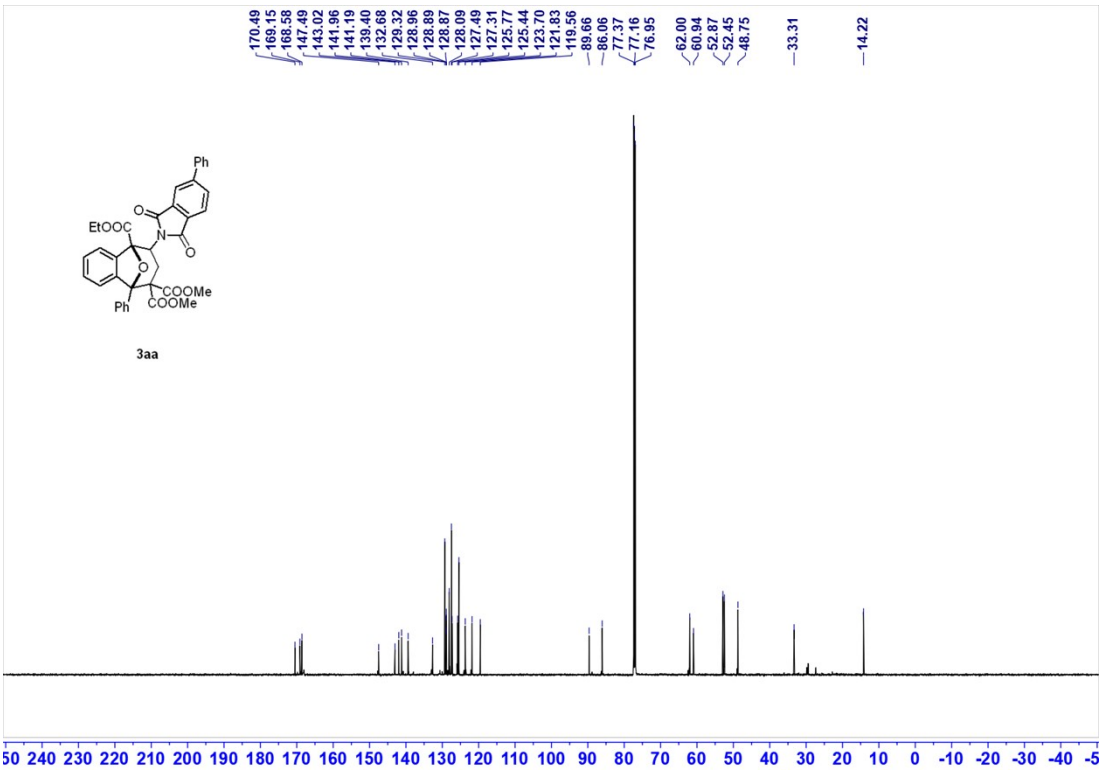
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