

Cooperative Photoredox and *N*-Heterocyclic Carbene-Catalyzed Formal C-H Acylation of Cyclopropanes via Deconstruction- Reconstruction Strategy

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Table of Contents

1. General information	S2
2. Emission spectrum of photoreactor lamp	S2
3. Synthesis of substrates	S2
4. Conditions Optimization	S3
5. Control experiments	S5
6. Stern-Volmer fluorescence quenching experiments	S6
7. Determination of quantum yield	S7
8. Characterization data of products	S8
9. ¹H, ¹³C, and ¹⁹F NMR spectra	S17
10. Reference	S44

1. General information

All reagents were purchased and used directly without further purification. All reactions were monitored by thin-layer chromatography (TLC), and column chromatography was performed on 200–300 mesh of silica gel purchased from Qing Dao Hai Yang Chemical Industry. ¹H, ¹³C and ¹⁹F NMR spectra were recorded on a Bruker Ascend 400 MHz spectrometer operating at 400 MHz, 101 MHz and 376 MHz, respectively. All NMR spectra were recorded in CDCl₃ at room temperature (20 ± 2 °C). Proton chemical shifts δ were given in ppm relative to tetramethylsilane (0.00 ppm) in CDCl₃. Spectra were calibrated relative to solvent's residual proton and carbon chemical shift:

CHCl_3 ($\delta = 7.26$ ppm for ^1H NMR and $\delta = 77.16$ ppm for ^{13}C NMR). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, brs = broad singlet, m = multiplet), coupling constants (Hz) and integration. High-resolution mass spectra (HRMS) were acquired on Thermo Q-Exactive instrument (quadrupole mass analyzer) using electrospray ionization mode (ESI). Fluorescence spectra were obtained with Edinburgh Instruments Ltd. FS5 spectrophotometer.

2. Emission spectrum of photoreactor lamp

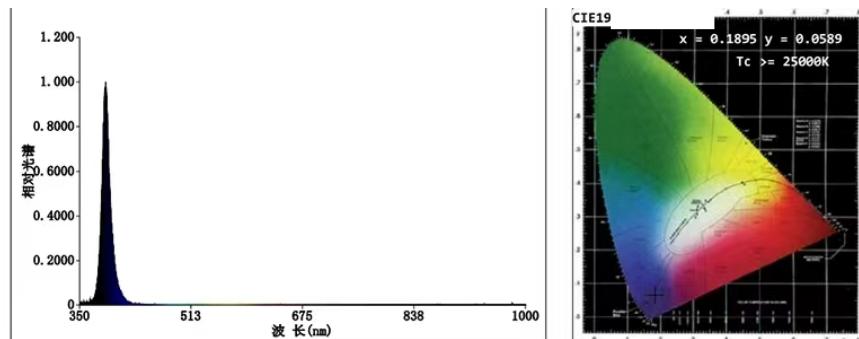


Figure S1. The emission spectrum of our lamp (blue LEDs 440 nm).

3. Synthesis of substrates

Starting material cyclopropane **1a-1f** and **1h-1s** were synthesized according to the literature¹, **1g²**, **1t³**, **1u⁴**, **1v⁵** and **1w³** were synthesized according to the literature.

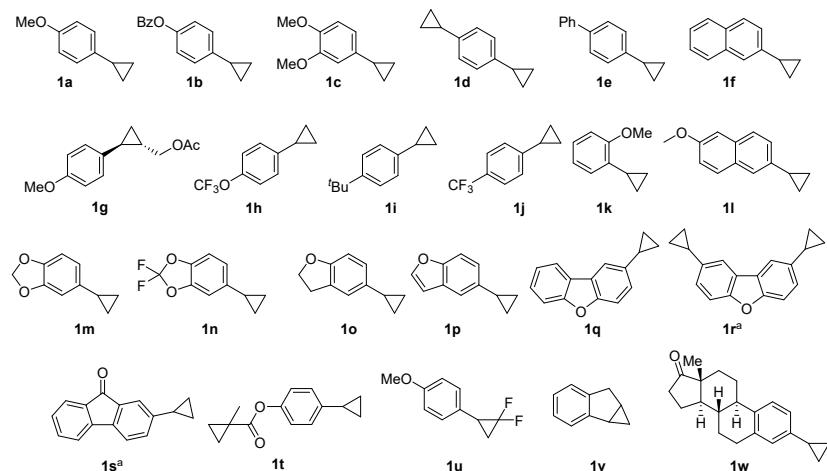


Figure S2. The synthesis of substrates cyclopropane **1**. ^a the yield of α -acylated cyclopropane **3** determined by ^1H NMR is 10-20% under the standard reaction conditions.

Starting material acyl imidazole **2** were synthesized according to the literature⁶.

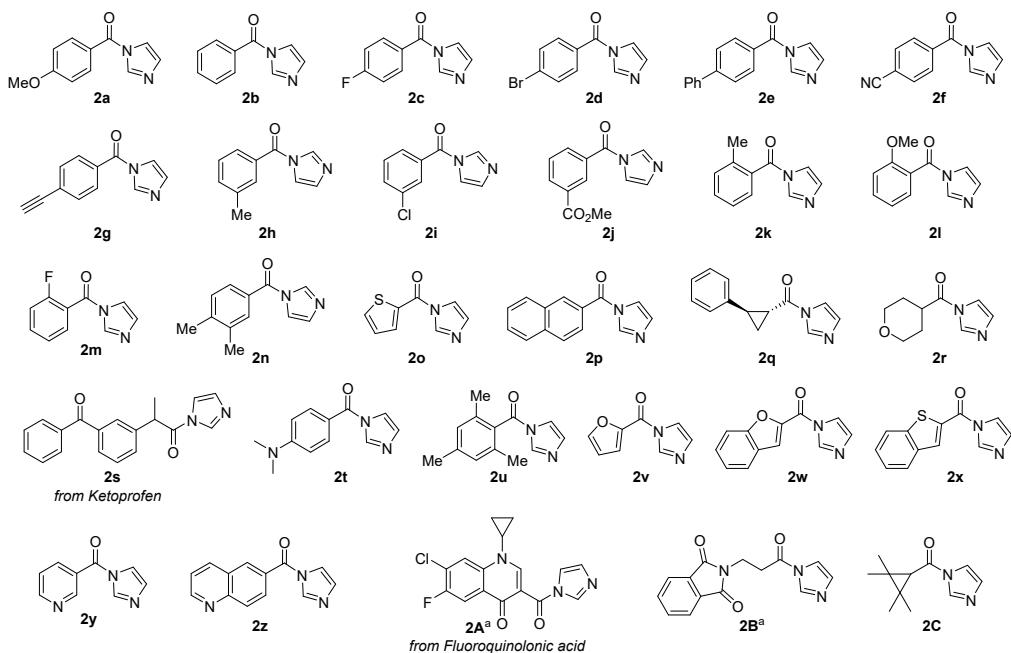


Figure S3. The synthesis of acyl imidazole **2**. ^a the yield of α -acylated cyclopropane **3** determined by ^1H NMR is 15-30% under the standard reaction conditions.

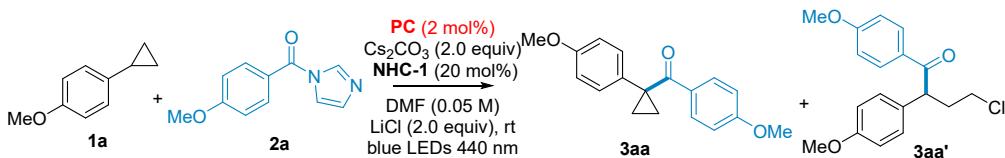
4. Conditions Optimization

Table S1. Optimization of the solvents^[a]

Entry	Solvent (0.05 M)	Yield 3aa [%]	Yield 3aa' [%]
1	MeCN	21	20
2	DMSO	42	0
3	DMF	57	0
4	dioxane	23	23
5	DMA	43	0
6	NMP	30	0
7	CF ₃ CH(OH)	0	28

^[a] All reactions were performed by using **1a** (0.1 mmol), **2a** (2.0 equiv), **PC-1** (2 mol%), **NHC-1** (20 mol%), Cs₂CO₃ (2.0 equiv), LiCl (2.0 equiv), solvent (0.05 M) under blue LEDs (440 nm, 20 W), stirred at room temperature and in Ar for 48 h; yield was determined by ^1H NMR.

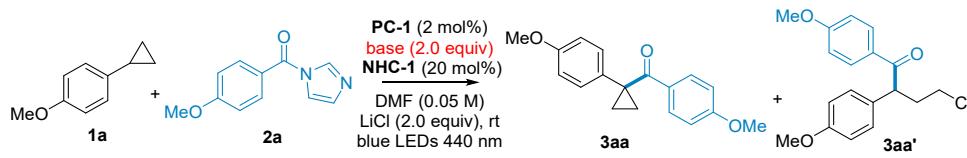
Table S2. Optimization of the photocatalysts^[a]



Entry	PC (X mol%)	Yield 3aa [%]	Yield 3aa' [%]
1	PC-1 (2 mol%)	57	0
2	Thioxanthen-9-one (5 mol%)	0	0
3	PC-2 (5 mol%)	32	0
4	3DPAFIPN (5 mol%)	22	0
5	PC-3 (5 mol%)	11	0
6	(Mes-Acr-Me)ClO ₄ (2 mol%)	0	0
7	(Mes-Acr-Ph) PF ₆ (2 mol%)	0	0

[a] All reactions were performed by using **1a** (0.1 mmol), **2a** (2.0 equiv), **PC** (2 mol% or 5 mol%), **NHC-1** (20 mol%), Cs₂CO₃ (2.0 equiv), LiCl (2.0 equiv), solvent (0.05 M) under blue LEDs (440 nm, 20 W), stirred at room temperature and in Ar for 48 h; yield was determined by ¹H NMR.

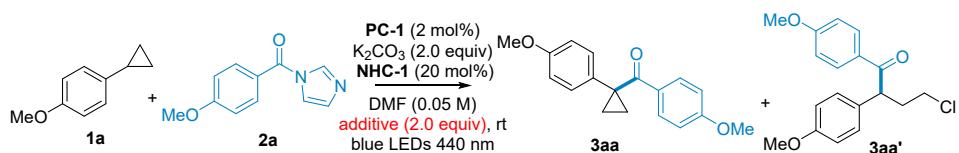
Table S3. Optimization of the bases [a]



Entry	base (2.0 equiv)	Yield 3aa [%]	Yield 3aa' [%]
1	Cs ₂ CO ₃	57	0
2	K ₂ CO ₃	78	0
3	Na ₂ CO ₃	37	0
4	NaHCO ₃	37	0
5	Na ₂ HPO ₄	28	33
6	K ₃ PO ₄	29	22

[a] All reactions were performed by using **1a** (0.1 mmol), **2a** (2.0 equiv), **PC-1** (2 mol%), **NHC-1** (20 mol%), base (2.0 equiv), LiCl (2.0 equiv), solvent (0.05 M) under blue LEDs (440 nm, 20 W), stirred at room temperature and in Ar for 48 h; yield was determined by ¹H NMR.

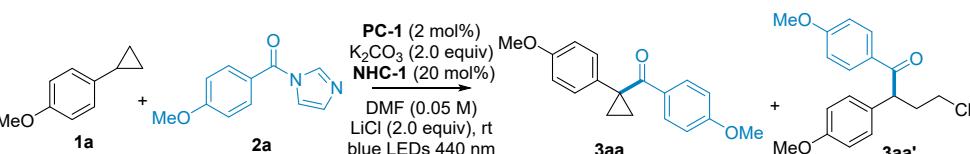
Table S4. Optimization of the additives [a]



Entry	additives (2.0 equiv)	Yield 3aa [%]	Yield 3aa' [%]
1	LiCl	78	0
2	KCl	15	0
3	NaCl	25	0
4	Me ₄ NCl	50	0

[a] All reactions were performed by using **1a** (0.1 mmol), **2a** (2.0 equiv), **PC** (2 mol% or 5 mol%), **NHC-1** (20 mol%), K₂CO₃ (2.0 equiv), additive (2.0 equiv), solvent (0.05 M) under blue LEDs (440 nm, 20 W), stirred at room temperature and in Ar for 48 h; a yield was determined by ¹H NMR.

Table S5. Optimization of other parameters [a]



Entry	Variation from “standard conditions”	Yield 3aa [%]	Yield 3aa' [%]
1	None	78	0
2	DMF (0.025 M) instead of DMF (0.05 M)	50	0
3	DMF (0.1 M) instead of DMF (0.05 M)	85 (79) ^b	0
4	NHC-1 (10 mol%) instead of NHC-1 (20 mol%)	71	0
5	LiCl (0.5 equiv) instead of LiCl (2.0 equiv)	42	33
6	K ₂ CO ₃ (0.2 equiv) instead of K ₂ CO ₃ (2.0 equiv)	50	26
7	K ₂ CO ₃ (1.0 equiv) instead of K ₂ CO ₃ (2.0 equiv)	59	12
8	24 h instead of 48 h	59	0
9	36 h instead of 48 h	71	0
10	Without NHC-1	0	0
11	Without PC-1	0	0
12	In dark	0	0

[a] All reactions were performed by using **1a** (2.0 equiv), **2a** (0.1 mmol), **PC-1** (2 mol%), **NHC-1** (20 mol%), K₂CO₃ (2.0 equiv), LiCl (2.0 equiv), DMF (0.05 M) under blue LEDs (440 nm, 20 W), stirred at room temperature and in Ar for 48 h; ^a yield was determined by ¹H NMR. ^b yield was isolated.

5. Control experiments

1a (2.0 equiv), **2a** (0.1 mmol), **PC-1** (2 mol%), **NHC-1** (20 mol%), K₂CO₃ (2.0 equiv), LiCl (2.0 equiv) and additives including TEMPO and BHT were sequentially added in a 25 mL reaction vial. The reaction system added DMF (0.1 M) was sealed under Ar and stirred under the irradiation of blue LEDs (440 nm, 20 W) at room temperature for 48 h. After reaction, corresponding

yields were determined by ^1H NMR of the reaction solution. Additionally, the adduct **A** and **B** were successfully detected by high-resolution mass spectrometry (HRMS)

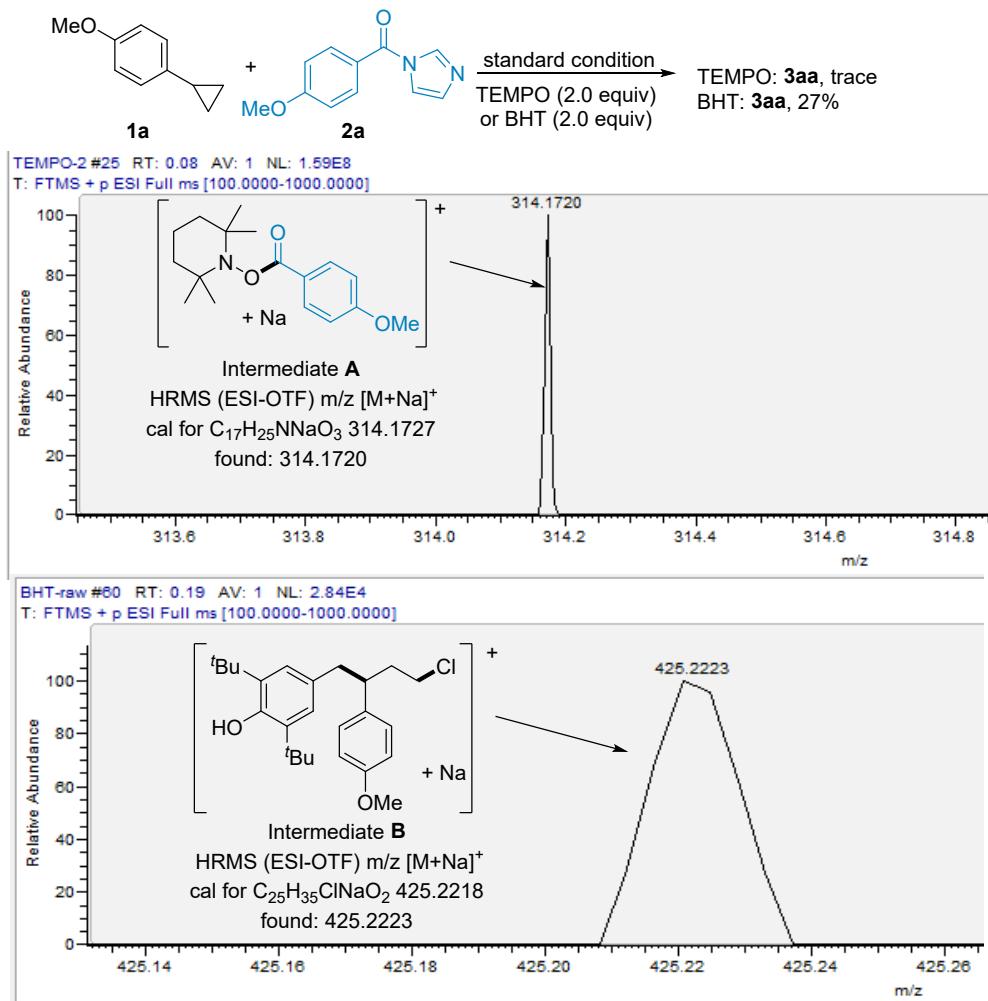


Figure S4. The mixture of reaction with TEMPO and BHT were detected by HRMS

6. Stern-Volmer fluorescence quenching experiments

Emission intensities were recorded using Edinburgh Instruments Ltd. FS5 spectrophotometer. All Stern-Volmer fluorescence quenching experiments were excited at 375 nm and the emission intensity was collected at 450 nm. The fluorescence emission spectrum was measured from 300 nm to 800 nm. A freshly prepared solution of 5×10^{-5} M solution of **PC-1** in dry and degassed CH_3CN was added the appropriate amount of a quencher (**1a** and **9**) in a screw-top quartz cuvette at room temperature (Figure S6).

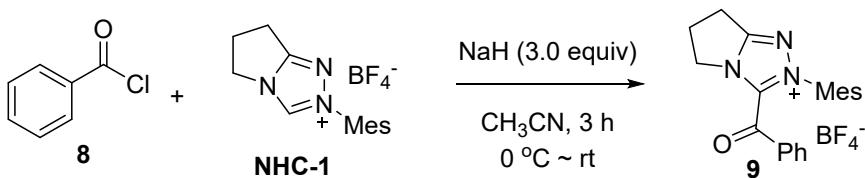


Figure S5. The synthesis of compound **9**

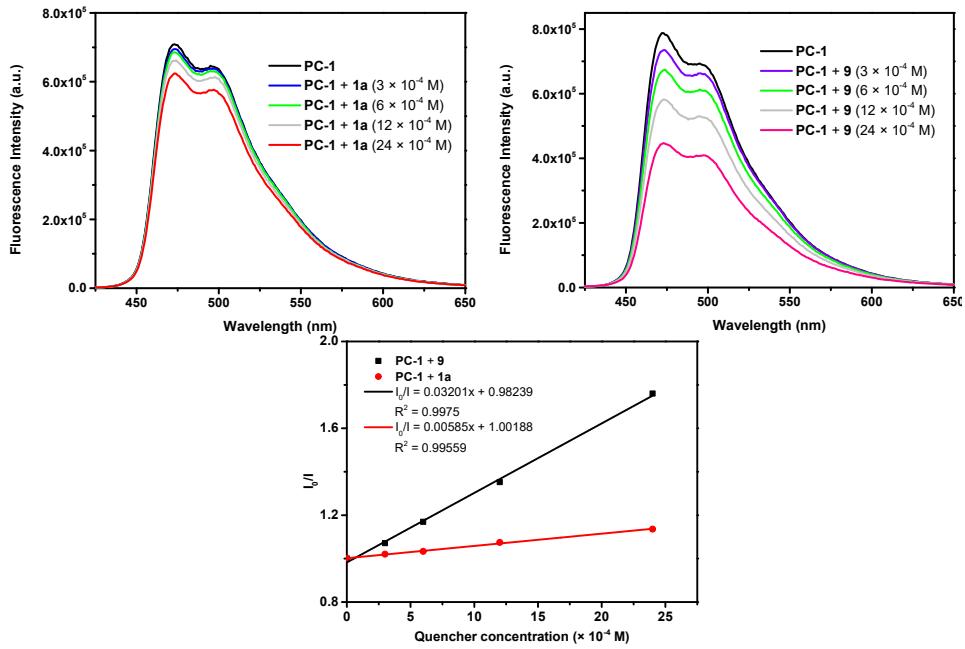


Figure S6. Fluorescence quenching experiments.

7. Determination of quantum yield

Following a literature procedure of Yoon,⁷ the photon flux of the single blue LED (20 W, $\lambda = 440$ nm) was determined by standard ferrioxalate actinometry. The photon flux of the spectrophotometer was determined by standard ferrioxalate actinometry. A 0.15 M solution of ferrioxalate was prepared by dissolving 1.473 g of potassium ferrioxalate hydrate in 20 mL of 0.05 M H₂SO₄. A buffered solution of phenanthroline was prepared by dissolving 10 mg of phenanthroline and 2.25 g of sodium acetate in 10 mL of 0.5 M H₂SO₄. Both solutions were stored in the dark. To determine the photon flux of the spectrophotometer, 2.0 mL of the ferrioxalate solution was placed in a cuvette and irradiated for 90.0 seconds at $\lambda = 440$ nm with an emission slit width at 10.0 nm. After irradiation, 0.35 mL of the phenanthroline solution was added to the cuvette. The solution was then allowed to rest for 1 h to allow the ferrous ions to completely coordinate to the phenanthroline. The absorbance of the solution was measured at 510 nm. A non-irradiated sample was also prepared and the absorbance at 510 nm measured. Conversion was calculated using eq 1.

$$n_{Fe(II)} = \frac{V \cdot \Delta A_{510\text{ nm}}}{l \cdot \epsilon} \quad (1)$$

Where V is the total volume (0.00235 L) of the solution after addition of phenanthroline, ΔA is the difference in absorbance at 510 nm between the irradiated and non-irradiated solutions, l is the path length (1.000 cm), and ϵ is the molar absorptivity at 510 nm (11,100 L mol⁻¹ cm⁻¹). The photon flux can be calculated using eq 2.

$$\text{photon flux} = \frac{n_{Fe(II)}}{\phi \cdot t \cdot f} \quad (2)$$

Where Φ is the quantum yield for the ferrioxalate actinometer (1.01 for a 0.15 M solution at $\lambda = 436$ nm), t is the time (90.0 s), and f is the fraction of light absorbed at $\lambda = 440$ nm (0.99713, vide infra). The photon flux was calculated (average of three experiments) to be 1.9×10^{-9} einstein s⁻¹.

$$f = 1 - 10^{-A_{440\text{ nm}}} \quad (3)$$

Sample calculation:

$$n_{Fe(II)} = 0.00235 \times 0.8144 / 11100 = 1.7242 \times 10^{-7} \text{ mol}$$

$$\text{photon flux} = 1.7242 \times 10^{-7} / (1.01 \times 90 \times 0.99713) = 1.9023 \times 10^{-9} \text{ einstein s}^{-1}$$

Determination of the Reaction Quantum Yield

In glove box, a 25 ml seal bottle was charged with 1-anisoylcyclopropane **1a** (0.1 mmol) and *N*-anisoylimidazole **2a** (2.0 equiv), **PC-1** (2 mol%), **NHC-1** (20 mol%), K₂CO₃ (2.0 equiv), LiCl (2.0 equiv), anhydrous DMF (0.05 M). The reaction was capped and removed from the glovebox, and the vial was stirred and irradiated with blue LEDs (440 nm) for 2 h. After completion of the reaction, the reaction was quenched by water, extracted by ethyl acetate and concentrated under in vacuum, the yields were determined by crude ¹H NMR analysis using CH₂Br₂ (3.5 μL, 0.10 mmol) as an internal standard. The quantum yield of the reaction can be calculated using equation 4.

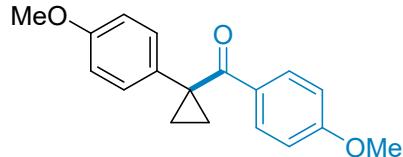
$$\Phi = \frac{n_{product}}{\Phi_q \cdot t \cdot f_R} \quad (4)$$

where Φ_q is the photon flux, t is the irradiation time (2 h). The fraction of light absorbed (f_R) of the reaction was determined by measuring the absorbance of PC-1 in DMF, which was same concentration with the reaction system ($f_R = 0.9903$).

$$\Phi = 5 \times 10^{-6} \text{ mol} / (1.9023 \times 10^{-9} \times 2 \times 3600 \times 0.9903) = 0.36$$

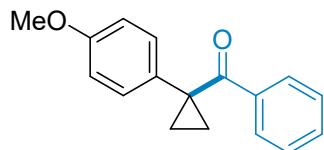
8. Characterization data of products

(4-methoxyphenyl)(1-(4-methoxyphenyl)cyclopropyl)methanone (**3aa**)



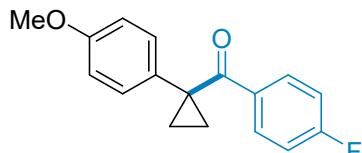
Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 40/1, v/v) afforded the title compound as a pale yellow oil (22.3 mg, 79% yield); ¹H NMR (400 MHz, CDCl₃) δ 7.75 – 7.68 (m, 2H), 7.09 – 7.03 (m, 2H), 6.73 – 6.64 (m, 4H), 3.70 (s, 3H), 3.67 (s, 3H), 1.49 (q, *J* = 4.2 Hz, 2H), 1.17 (q, *J* = 4.2 Hz, 2H); ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 198.6, 162.6, 158.2, 133.4, 131.9, 129.6, 128.8, 114.0, 113.2, 55.3, 55.2, 34.1, 15.6. HRMS (ESI-TOF) *m/z* [M + Na]⁺ calcd for C₁₈H₁₈NaO₃ 305.1149, found 305.1138.

*(1-(4-methoxyphenyl)cyclopropyl)(phenyl)methanone (3ab)*⁸



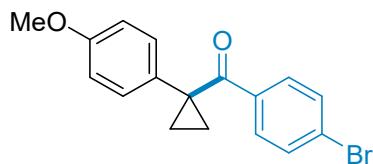
Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 50/1, v/v) afforded the title compound as a pale yellow oil (15.6 mg, 62% yield); ¹H NMR (400 MHz, CDCl₃) δ 7.67 (d, *J* = 7.8 Hz, 2H), 7.29 (t, *J* = 7.3 Hz, 1H), 7.19 (t, *J* = 7.6 Hz, 2H), 7.06 (d, *J* = 8.6 Hz, 2H), 6.69 (d, *J* = 8.6 Hz, 2H), 3.66 (s, 3H), 1.56 (q, *J* = 4.2 Hz, 2H), 1.22 (q, *J* = 4.2 Hz, 2H); ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 200.8, 158.4, 137.3, 133.1, 131.9, 129.5, 129.4, 128.1, 114.1, 55.3, 34.7, 16.3.

*(4-fluorophenyl)(1-(4-methoxyphenyl)cyclopropyl)methanone (3ac)*⁹



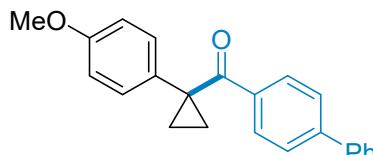
Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 50/1, v/v) afforded the title compound as a pale yellow oil (17.0 mg, 63% yield); ¹H NMR (400 MHz, CDCl₃) δ 7.72 – 7.67 (m, 2H), 7.08 – 7.01 (m, 2H), 6.93 – 6.82 (m, 2H), 6.74 – 6.66 (m, 2H), 3.68 (s, 3H), 1.55 (q, *J* = 4.1 Hz, 2H), 1.22 (q, *J* = 4.2 Hz, 2H); ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 199.1, 164.9 (d, *J*_{C-F} = 253.5 Hz), 158.5, 133.5, 132.9, 132.1 (d, *J*_{C-F} = 9.1 Hz), 129.3, 115.2 (d, *J*_{C-F} = 21.8 Hz), 114.3, 55.4, 34.6, 16.3; ¹⁹F NMR (376 MHz, CDCl₃) δ -106.72.

(4-bromophenyl)(1-(4-methoxyphenyl)cyclopropyl)methanone (3ad)



Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 50/1, v/v) afforded the title compound as a pale yellow oil (14.6 mg, 44% yield); ¹H NMR (400 MHz, CDCl₃) δ 7.55 – 7.49 (m, 2H), 7.35 – 7.30 (m, 2H), 7.09 – 7.00 (m, 2H), 6.74 – 6.66 (m, 2H), 3.68 (s, 3H), 1.56 (q, *J* = 4.1 Hz, 2H), 1.23 (q, *J* = 4.2 Hz, 2H); ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 199.7, 158.4, 136.0, 132.5, 131.2, 130.9, 129.3, 126.8, 114.2, 55.2, 34.6, 16.5. HRMS (ESI-TOF) *m/z* [M + Na]⁺ calcd for C₁₇H₁₅⁷⁹BrNaO₂ 353.0148, found 353.0142.

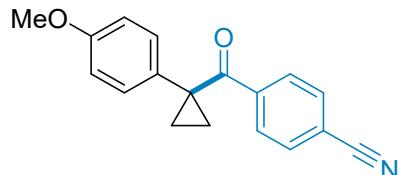
*[1,1'-biphenyl]-4-yl(1-(4-methoxyphenyl)cyclopropyl)methanone (3ae)*⁹



Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 50/1, v/v) afforded the title compound as a pale yellow oil (22.6 mg, 69% yield); ¹H NMR (400 MHz, CDCl₃)

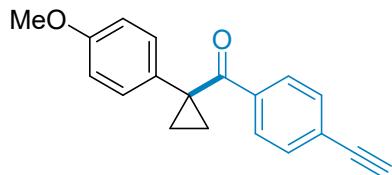
δ 7.77 – 7.70 (m, 2H), 7.49 – 7.44 (m, 2H), 7.41 (d, J = 8.4 Hz, 2H), 7.36 – 7.31 (m, 2H), 7.27 (d, J = 7.2 Hz, 1H), 7.13 – 7.07 (m, 2H), 6.69 – 6.73 (m, 2H), 3.66 (s, 3H), 1.57 (q, J = 4.1 Hz, 2H), 1.23 (q, J = 4.2 Hz, 2H); $^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 200.1, 158.4, 144.6, 140.1, 135.9, 133.1, 130.2, 129.4, 128.96, 128.1, 127.3, 126.7, 114.2, 55.3, 34.7, 16.2.

(4-(1-(4-methoxyphenyl)cyclopropane-1-carbonyl)benzonitrile (3af)⁸



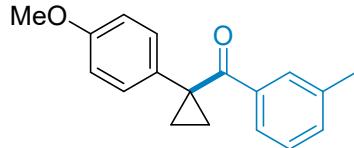
Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 50/1, v/v) afforded the title compound as a pale yellow oil (12.5 mg, 45% yield); ^1H NMR (400 MHz, CDCl_3) δ 7.65 (d, J = 8.0 Hz, 2H), 7.47 (d, J = 7.8 Hz, 2H), 7.03 (d, J = 8.3 Hz, 2H), 6.70 (d, J = 8.2 Hz, 2H), 3.68 (s, 3H), 1.63 (q, J = 4.2 Hz, 2H), 1.29 (q, J = 4.1 Hz, 2H); $^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 200.3, 158.8, 141.3, 131.9, 129.9, 129.5, 118.2, 114.9, 114.4, 55.4, 35.2, 17.5.

(4-ethynylphenyl)(1-(4-methoxyphenyl)cyclopropyl)methanone (3ag)



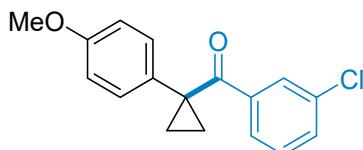
Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 40/1, v/v) afforded the title compound as a pale yellow oil (9.1 mg, 33% yield); ^1H NMR (400 MHz, CDCl_3) δ 7.66 (d, J = 8.3 Hz, 2H), 7.37 (d, J = 8.3 Hz, 2H), 7.11 (d, J = 8.7 Hz, 2H), 6.77 (d, J = 8.7 Hz, 2H), 3.75 (s, 3H), 3.15 (s, 1H), 1.65 (q, J = 4.1 Hz, 2H), 1.31 (q, J = 4.1 Hz, 2H); $^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 200.3, 158.5, 137.3, 132.7, 131.8, 129.5, 129.3, 125.6, 114.2, 83.1, 79.7, 55.3, 34.8, 16.6. HRMS (ESI-TOF) m/z [M + Na]⁺ calcd for $\text{C}_{19}\text{H}_{16}\text{NaO}_2$ 299.1043, found 299.1039.

(1-(4-methoxyphenyl)cyclopropyl)(m-tolyl)methanone (3ah)



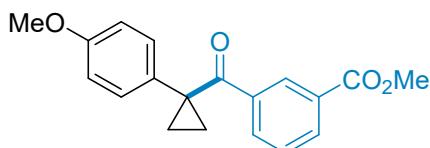
Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 50/1, v/v) afforded the title compound as a pale yellow oil (21.5 mg, 81% yield); ^1H NMR (400 MHz, CDCl_3) δ 7.57 (s, 1H), 7.52 (d, J = 7.7 Hz, 1H), 7.19 – 7.11 (m, 4H), 6.80 – 6.74 (m, 2H), 3.74 (s, 3H), 2.29 (s, 3H), 1.62 (d, J = 3.2 Hz, 2H), 1.28 (q, J = 3.5 Hz, 2H); $^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 201.0, 158.4, 137.8, 137.3, 133.2, 132.7, 129.9, 129.5, 127.8, 126.8, 114.1, 55.3, 34.8, 21.4, 16.2. HRMS (ESI-TOF) m/z [M + Na]⁺ calcd for $\text{C}_{18}\text{H}_{18}\text{NaO}_2$ 289.1199, found 289.1190.

(3-chlorophenyl)(1-(4-methoxyphenyl)cyclopropyl)methanone (3ai)



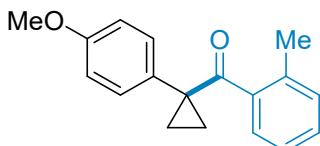
Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 50/1, v/v) afforded the title compound as a pale yellow oil (17.5 mg, 61% yield); ^1H NMR (400 MHz, CDCl_3) δ 7.63 (t, J = 1.8 Hz, 1H), 7.48 (dt, J = 7.8, 1.1 Hz, 1H), 7.27 – 7.24 (m, 1H), 7.10 (t, J = 7.9 Hz, 1H), 7.07 – 7.00 (m, 2H), 6.73 – 6.65 (m, 2H), 3.67 (s, 3H), 1.57 (q, J = 4.1 Hz, 2H), 1.24 (q, J = 4.1 Hz, 2H); $^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 199.7, 158.6, 139.1, 134.3, 132.5, 131.8, 129.6, 129.4, 129.3, 127.5, 114.3, 55.4, 34.9, 16.6. HRMS (ESI-TOF) m/z [M + Na] $^+$ calcd for $\text{C}_{17}\text{H}_{15}\text{ClNaO}_2$ 309.0653, found 309.0648.

methyl 3-(1-(4-methoxyphenyl)cyclopropane-1-carbonyl)benzoate (3aj)



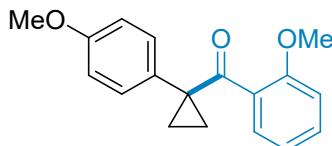
Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 40/1, v/v) afforded the title compound as a pale yellow oil (10.5 mg, 34% yield); ^1H NMR (400 MHz, CDCl_3) δ 8.31 (s, 1H), 7.95 (d, J = 7.8 Hz, 1H), 7.79 (d, J = 7.8 Hz, 1H), 7.26 (t, J = 7.8 Hz, 1H), 7.12 – 7.04 (m, 2H), 6.74 – 6.61 (m, 2H), 3.82 (s, 3H), 3.66 (s, 3H), 1.60 (q, J = 3.1 Hz, 2H), 1.27 (q, J = 2.9 Hz, 2H); $^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 200.3, 166.4, 158.6, 137.8, 133.5, 132.64, 132.56, 130.6, 130.2, 129.8, 128.3, 114.3, 55.3, 52.4, 35.0, 16.6. HRMS (ESI-TOF) m/z [M + Na] $^+$ calcd for $\text{C}_{19}\text{H}_{18}\text{NaO}_4$ 333.1098, found 333.1091.

(1-(4-methoxyphenyl)cyclopropyl)(o-tolyl)methanone (3al)



Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 50/1, v/v) afforded the title compound as a pale yellow oil (11.4 mg, 43% yield); ^1H NMR (400 MHz, CDCl_3) δ 7.10 (d, J = 7.8 Hz, 2H), 7.06 (d, J = 8.5 Hz, 2H), 7.00 (d, J = 7.5 Hz, 1H), 6.94 (t, J = 7.4 Hz, 1H), 6.64 (d, J = 8.5 Hz, 2H), 3.65 (s, 3H), 2.25 (s, 3H), 1.65 (q, J = 3.9 Hz, 2H), 1.26 (q, J = 3.5 Hz, 2H); $^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 207.2, 158.5, 139.8, 135.1, 132.4, 131.2, 130.7, 129.3, 126.98, 124.9, 113.8, 55.3, 37.0, 20.0, 18.3. HRMS (ESI-TOF) m/z [M + Na] $^+$ calcd for $\text{C}_{18}\text{H}_{18}\text{NaO}_2$ 289.1199, found 289.1193.

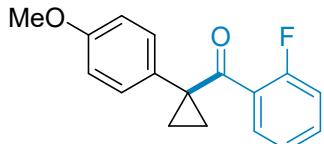
(2-methoxyphenyl)(1-(4-methoxyphenyl)cyclopropyl)methanone (3al)



Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 30/1, v/v)

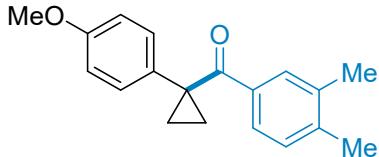
afforded the title compound as a pale yellow oil (13.3 mg, 47% yield); ^1H NMR (400 MHz, CDCl_3) δ 7.15 – 7.09 (m, 1H), 7.08 – 7.01 (m, 3H), 6.76 (t, J = 7.5 Hz, 1H), 6.63 – 6.55 (m, 3H), 3.64 (s, 3H), 3.59 (s, 3H), 1.65 (q, J = 3.5 Hz, 2H), 1.21 (q, J = 3.7 Hz, 2H); $^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 206.4, 158.3, 155.7, 132.7, 131.4, 130.9, 130.7, 127.9, 120.2, 113.6, 110.7, 55.3, 37.4, 19.1, 19.0. HRMS (ESI-TOF) m/z [M + Na] $^+$ calcd for $\text{C}_{18}\text{H}_{18}\text{NaO}_3$ 305.1149, found 305.1142.

(2-fluorophenyl)(1-(4-methoxyphenyl)cyclopropyl)methanone (3am)



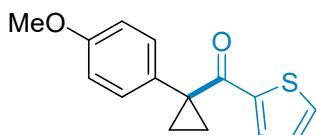
Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 50/1, v/v) afforded the title compound as a pale yellow oil (11.1 mg, 41% yield); ^1H NMR (400 MHz, CDCl_3) δ 7.23 (t, J = 7.3 Hz, 1H), 7.21 – 7.15 (m, 1H), 7.08 (d, J = 8.2 Hz, 2H), 6.95 (t, J = 7.5 Hz, 1H), 6.80 (t, J = 9.1 Hz, 1H), 6.63 (d, J = 8.2 Hz, 2H), 3.65 (s, 3H), 1.70 (q, J = 4.2 Hz, 2H), 1.27 (q, J = 4.1 Hz, 2H); $^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 202.2, 158.6 (d, $J_{\text{C}-\text{F}} = 251.5$ Hz), 158.4, 131.9 (d, $J_{\text{C}-\text{F}} = 8.3$ Hz), 131.7, 131.0, 129.1 (d, $J_{\text{C}-\text{F}} = 3.7$ Hz), 128.5 (d, $J_{\text{C}-\text{F}} = 16.6$ Hz), 123.8 (d, $J_{\text{C}-\text{F}} = 3.5$ Hz), 115.7 (d, $J_{\text{C}-\text{F}} = 21.9$ Hz), 113.5, 55.2, 37.2, 18.4.; ^{19}F NMR (376 MHz, CDCl_3) δ -106.72. HRMS (ESI-TOF) m/z [M + Na] $^+$ calcd for $\text{C}_{17}\text{H}_{15}\text{FNaO}_2$ 293.0949, found 293.0941.

(3,4-dimethylphenyl)(1-(4-methoxyphenyl)cyclopropyl)methanone (3an)



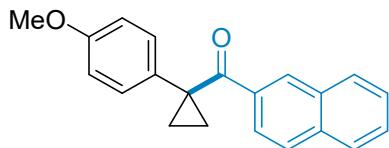
Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 50/1, v/v) afforded the title compound as a pale yellow oil (21.3 mg, 76% yield); ^1H NMR (400 MHz, CDCl_3) δ 7.50 (s, 1H), 7.41 (d, J = 7.8 Hz, 1H), 7.11 – 7.01 (m, 2H), 6.92 (d, J = 7.9 Hz, 1H), 6.72 – 6.64 (m, 2H), 3.66 (s, 3H), 2.13 (s, 3H), 2.11 (s, 3H), 1.51 (q, J = 4.1 Hz, 2H), 1.18 (q, J = 4.2 Hz, 2H); $^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 200.3, 158.3, 141.4, 136.4, 134.8, 133.5, 130.7, 129.2, 127.5, 114.1, 55.3, 34.5, 20.0, 19.8, 15.9. HRMS (ESI-TOF) m/z [M + Na] $^+$ calcd for $\text{C}_{19}\text{H}_{20}\text{NaO}_2$ 303.1356, found 303.1351.

(1-(4-methoxyphenyl)cyclopropyl)(thiophen-2-yl)methanone (3ao)



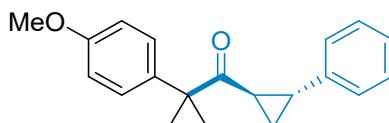
Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 50/1, v/v) afforded the title compound as a pale yellow oil (6.7 mg, 26% yield); ^1H NMR (400 MHz, CDCl_3) δ 7.37 (dd, J = 5.0, 1.2 Hz, 1H), 7.29 – 7.23 (m, 2H), 6.89 (dd, J = 3.9, 1.2 Hz, 1H), 6.85 – 6.75 (m, 3H), 3.76 (s, 3H), 1.66 (q, J = 3.8 Hz, 2H), 1.20 (q, J = 3.8 Hz, 2H); $^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 193.0, 159.3, 144.0, 133.9, 133.3, 132.9, 131.7, 127.7, 114.2, 55.5, 35.4, 19.1. HRMS (ESI-TOF) m/z [M + Na] $^+$ calcd for $\text{C}_{14}\text{H}_{15}\text{NaO}_2\text{S}$ 281.0607, found 281.0602.

(1-(4-methoxyphenyl)cyclopropyl)(naphthalen-2-yl)methanone (3ap)⁸



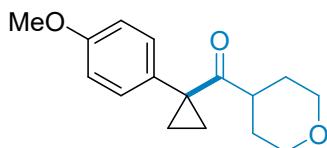
Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 40/1, v/v) afforded the title compound as a pale yellow oil (18.7 mg, 62% yield); ¹H NMR (400 MHz, CDCl₃) δ 8.20 (s, 1H), 7.79 – 7.67 (m, 3H), 7.63 (d, J = 8.6 Hz, 1H), 7.47 – 7.34 (m, 2H), 7.12 (d, J = 8.5 Hz, 2H), 6.68 (d, J = 8.4 Hz, 2H), 3.64 (s, 3H), 1.63 (q, J = 4.2 Hz, 2H), 1.28 (q, J = 4.3 Hz, 2H); ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 200.7, 158.4, 135.0, 134.6, 133.2, 132.4, 131.1, 129.6, 129.4, 128.1, 127.8, 127.7, 126.5, 125.5, 114.2, 55.3, 34.7, 16.4.

*(1-(4-methoxyphenyl)cyclopropyl)((1*R*,2*R*)-2-phenylcyclopropyl)methanone (3aq)*



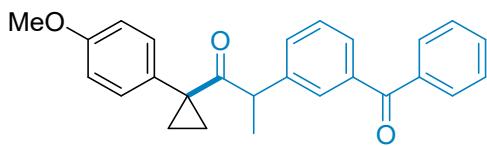
Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 50/1, v/v) afforded the title compound as a pale yellow oil (22.5 mg, 77% yield); ¹H NMR (400 MHz, CDCl₃) δ 7.28 (d, J = 8.6 Hz, 2H), 7.20 (t, J = 7.2 Hz, 2H), 7.16 (d, J = 7.1 Hz, 1H), 6.93 (d, J = 7.3 Hz, 2H), 6.82 (d, J = 8.6 Hz, 2H), 3.79 (s, 3H), 2.45 (ddd, J = 9.6, 6.5, 4.3 Hz, 1H), 2.12 (dt, J = 8.6, 4.7 Hz, 1H), 1.62 (q, J = 5.5, 4.9 Hz, 3H), 1.24 – 1.10 (m, 3H); ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 209.0, 158.9, 140.6, 132.8, 132.0, 128.4, 126.5, 126.4, 114.1, 55.4, 36.9, 29.9, 29.8, 19.8, 19.1, 18.7. HRMS (ESI-TOF) m/z [M + Na]⁺ calcd for C₂₀H₂₀NaO₂ 315.1356, found 315.1349.

*(1-(4-methoxyphenyl)cyclopropyl)(tetrahydro-2*H*-pyran-4-yl)methanone (3ar)*



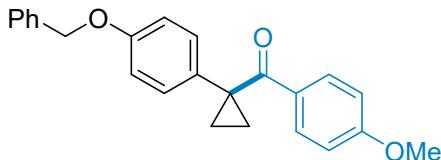
Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 50/1, v/v) afforded the title compound as a pale yellow oil (18.5 mg, 71% yield); ¹H NMR (400 MHz, CDCl₃) δ 7.20 (dd, J = 9.1, 2.5 Hz, 2H), 6.84 – 6.78 (m, 2H), 3.81 – 3.76 (m, 2H), 3.76 (s, 3H), 3.10 (td, J = 11.7, 2.2 Hz, 2H), 2.74 – 2.66 (m, 1H), 1.59 – 1.55 (m, 2H), 1.47 (q, J = 3.5 Hz, 2H), 1.44 – 1.40 (m, 2H), 1.04 (q, J = 3.6 Hz, 2H); ¹³C{¹H} NMR (101 MHz, CDCl₃) δ 212.1, 159.0, 132.6, 131.9, 114.1, 67.2, 55.4, 44.3, 35.9, 28.7, 18.6. HRMS (ESI-TOF) m/z [M + Na]⁺ calcd for C₁₆H₂₀NaO₃ 283.1305, found 283.1298.

2-(3-benzoylphenyl)-1-(1-(4-methoxyphenyl)cyclopropyl)propan-1-one (3as)



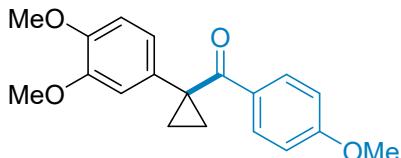
Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 40/1, v/v) afforded the title compound as a pale yellow oil (18.4 mg, 48% yield); ^1H NMR (400 MHz, CDCl_3) δ 7.74 (d, J = 7.5 Hz, 2H), 7.64 – 7.55 (m, 2H), 7.48 (t, J = 7.4 Hz, 2H), 7.32 (t, J = 7.7 Hz, 1H), 7.20 – 7.08 (m, 2H), 6.97 (d, J = 8.2 Hz, 2H), 6.71 (d, J = 8.2 Hz, 2H), 4.04 (q, J = 7.0 Hz, 1H), 3.75 (s, 3H), 1.67 – 1.61 (m, 1H), 1.54 – 1.45 (m, 1H), 1.27 (d, J = 6.8 Hz, 3H), 1.14 – 1.01 (m, 2H); $^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 210.9, 196.5, 159.0, 141.3, 137.9, 137.7, 132.6, 132.5, 132.3, 131.8, 130.10, 130.05, 128.5, 128.5, 128.4, 113.8, 55.4, 49.7, 36.5, 20.0, 19.8, 19.6. HRMS (ESI-TOF) m/z [M + Na] $^+$ calcd for $\text{C}_{26}\text{H}_{24}\text{NaO}_3$ 407.1618, found 407.1609.

(1-(4-(benzyloxy)phenyl)cyclopropyl)(4-methoxyphenyl)methanone (3ba)



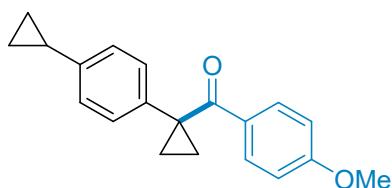
Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 30/1, v/v) afforded the title compound as a pale yellow oil (19.7 mg, 55% yield); ^1H NMR (400 MHz, CDCl_3) δ 7.80 (d, J = 8.5 Hz, 2H), 7.41 – 7.32 (m, 5H), 7.15 (d, J = 8.3 Hz, 2H), 6.86 (d, J = 8.3 Hz, 2H), 6.77 (d, J = 8.5 Hz, 2H), 5.00 (s, 2H), 3.79 (s, 3H), 1.58 (q, J = 4.4 Hz, 2H), 1.26 (q, J = 4.4 Hz, 2H); $^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 198.7, 162.7, 157.5, 137.0, 133.9, 132.0, 129.7, 129.0, 128.7, 128.1, 127.6, 115.1, 113.3, 70.1, 55.5, 34.2, 15.7. HRMS (ESI-TOF) m/z [M + Na] $^+$ calcd for $\text{C}_{24}\text{H}_{22}\text{NaO}_2$ 381.1462, found 381.1456.

(1-(3,4-dimethoxyphenyl)cyclopropyl)(4-methoxyphenyl)methanone (3ca)



Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 30/1, v/v) afforded the title compound as a pale yellow oil (16.8 mg, 54% yield); ^1H NMR (400 MHz, CDCl_3) δ 7.83 – 7.77 (m, 2H), 6.82 – 6.74 (m, 5H), 6.71 (d, J = 2.1 Hz, 1H), 3.83 (s, 3H), 3.79 (s, 3H), 3.77 (s, 3H), 1.57 (q, J = 4.2 Hz, 2H), 1.26 (q, J = 4.2 Hz, 2H); $^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 198.7, 162.7, 149.1, 147.9, 134.0, 131.9, 129.8, 119.7, 113.4, 111.8, 111.3, 56.0, 56.0, 55.5, 34.7, 15.7. HRMS (ESI-TOF) m/z [M + Na] $^+$ calcd for $\text{C}_{21}\text{H}_{18}\text{NaO}_2$ 335.1254, found 335.1246.

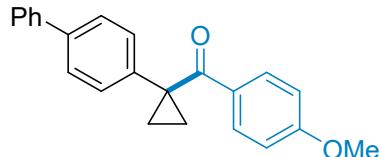
(1-(4-cyclopropylphenyl)cyclopropyl)(4-methoxyphenyl)methanone (3da)



Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 40/1, v/v) afforded the title compound as a pale yellow oil (12.0 mg, 41% yield); ^1H NMR (400 MHz, CDCl_3) δ 7.81 (d, J = 8.5 Hz, 2H), 7.08 (d, J = 7.9 Hz, 2H), 6.94 (d, J = 7.9 Hz, 2H), 6.76 (d, J = 8.6 Hz, 2H), 3.79 (s, 3H), 1.82 (tt, J = 8.7, 5.1 Hz, 1H), 1.56 (q, J = 4.3 Hz, 2H), 1.27 (q, J = 4.3 Hz, 2H),

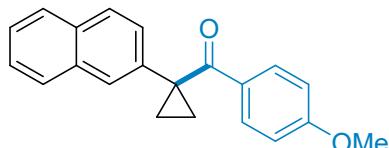
0.95 – 0.85 (m, 2H), 0.69 – 0.58 (m, 2H); $^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 198.5, 162.8, 142.3, 138.5, 132.1, 129.7, 127.5, 126.0, 113.4, 55.5, 34.4, 15.6, 15.1, 9.3. HRMS (ESI-TOF) m/z [M + Na] $^+$ calcd for $\text{C}_{20}\text{H}_{20}\text{NaO}_2$ 315.1356, found 315.1350.

(1-[1,1'-biphenyl]-4-yl)cyclopropyl)(4-methoxyphenyl)methanone (3ea)



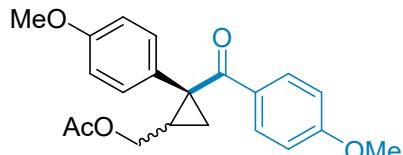
Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 40/1, v/v) afforded the title compound as a pale yellow oil (10.5 mg, 32% yield); ^1H NMR (400 MHz, CDCl_3) δ 7.87 – 7.64 (m, 2H), 7.51 – 7.44 (m, 2H), 7.45 – 7.37 (m, 2H), 7.36 – 7.30 (m, 2H), 7.27 – 7.22 (m, 1H), 7.21 – 7.20 (m, 1H), 7.19 – 7.18 (m, 1H), 6.78 – 6.62 (m, 2H), 3.72 (s, 3H), 1.56 (q, J = 4.3 Hz, 2H), 1.28 (q, J = 4.4 Hz, 2H); $^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 198.2, 162.9, 140.7, 140.6, 139.3, 132.1, 129.6, 128.9, 127.9, 127.42, 127.4, 127.0, 113.5, 55.5, 34.5, 16.0. HRMS (ESI-TOF) m/z [M + Na] $^+$ calcd for $\text{C}_{23}\text{H}_{20}\text{NaO}_2$ 351.1356, found 351.1352.

(4-methoxyphenyl)(1-(naphthalen-2-yl)cyclopropyl)methanone (3fa)



Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 30/1, v/v) afforded the mixture compound of **3fa** and arylation product as a pale yellow oil [7.2 mg, 24% yield, (**3fa**, 5.4 mg, 18% yield determined by ^1H NMR), (arylation product, 1.8 mg, 6% yield determined by ^1H NMR)]; ^1H NMR (400 MHz, CDCl_3) δ 7.88 – 7.83 (m, 2H), 7.76 (d, J = 7.6 Hz, 2H), 7.72 – 7.67 (m, 2H), 7.47 – 7.41 (m, 2H), 7.31 (dd, J = 8.5, 1.8 Hz, 1H), 6.76 – 6.68 (m, 2H), 3.74 (s, 3H), 1.69 (q, J = 4.3 Hz, 2H), 1.42 (q, J = 4.3 Hz, 2H); $^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 198.3, 197.0, 163.9, 162.8, 140.4, 139.2, 137.3, 134.0, 133.6, 132.9, 132.2, 132.1, 131.2, 129.6, 129.5, 128.5, 127.8, 127.7, 126.6, 126.47, 126.45, 126.4, 126.1, 125.8, 125.8, 125.7, 125.4, 113.8, 113.4, 55.7, 55.4, 35.0, 16.0, 15.7, 9.4. HRMS (ESI-TOF) m/z [M + Na] $^+$ calcd for $\text{C}_{21}\text{H}_{18}\text{NaO}_2$ 315.1199, found 315.1192.

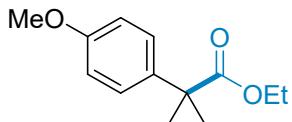
(2-(4-methoxybenzoyl)-2-(4-methoxyphenyl)cyclopropyl)methyl acetate (3ga)



Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 20/1, v/v) afforded the title compound as a pale yellow oil (10.6 mg, 30% yield, dr = 78:22); ^1H NMR (400 MHz, CDCl_3) (mixture of diastereoisomers) δ [7.85 (d, J = 8.8 Hz) + 7.78 (d, J = 8.8 Hz), 2H], [7.21 (d, J = 8.6 Hz) + 7.17 (d, J = 8.7 Hz), 2H], 6.82 – 6.71 (m, 4H), [4.13 (dd, J = 11.9, 6.7 Hz), 3.82 (dd, J = 11.7, 7.0 Hz), 1H], [3.97 (dd, J = 11.8, 7.9 Hz) + 3.63 (dd, J = 11.7, 7.9 Hz), 1H], [3.79 (s) + 3.78 (s), 3H], [3.75 (s) + 3.74 (s), 3H], 2.51 – 2.37 (m, 1H), [1.99 (s) + 1.90 (s), 3H], [1.81 (t, J =

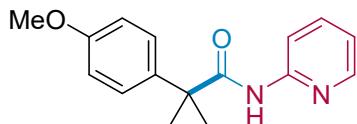
5.7 Hz) + 1.64 (t, J = 5.7 Hz), 2H), [1.44 (dd, J = 9.1, 5.1 Hz) + 1.09 (dd, J = 8.8, 4.7 Hz), 2H]; $^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) (mixture of diastereoisomers) δ 197.5, 196.3, 171.0, 170.9, 163.0, 162.8, 158.9, 158.5, 133.1, 132.3, 132.0, 130.9, 129.9, 129.4, 128.9, 128.2, 114.3, 114.2, 113.4, 113.3, 64.6, 64.4, 55.48, 55.46, 55.4, 55.3, 39.3, 39.0, 24.6, 22.2, 21.0, 20.9, 18.8, 17.7. HRMS (ESI-TOF) m/z [M + Na] $^+$ calcd for $\text{C}_{21}\text{H}_{22}\text{NaO}_5$ 377.1360, found 377.1360.

ethyl 1-(4-methoxyphenyl)cyclopropane-1-carboxylate (6)



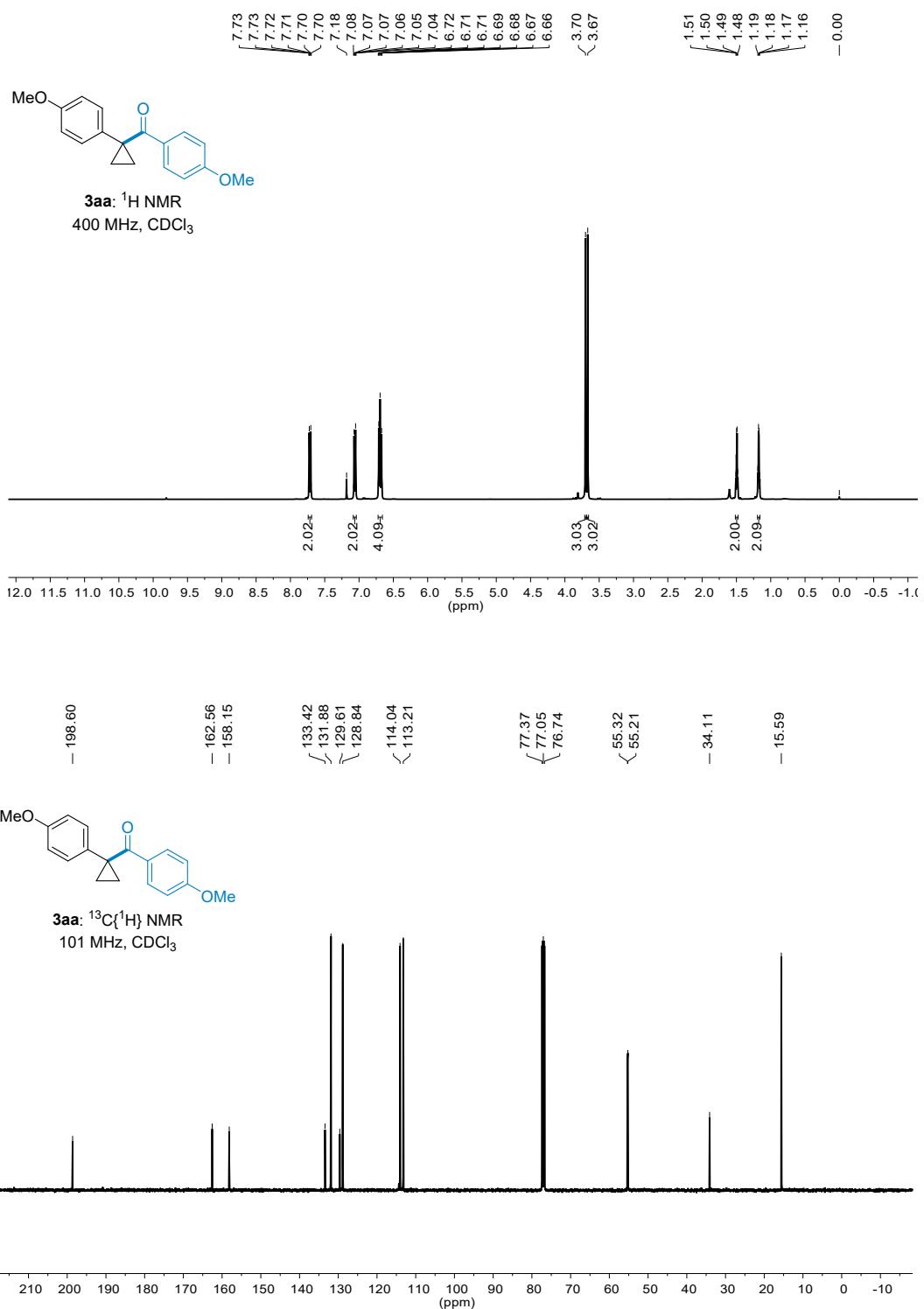
Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 80/1, v/v) afforded the title compound as a pale yellow oil (16.3 mg, 74% yield); ^1H NMR (400 MHz, CDCl_3) δ 7.32 – 7.23 (m, 2H), 6.88 – 6.83 (m, 2H), 4.10 (q, J = 7.1 Hz, 2H), 3.82 (s, 3H), 1.59 – 1.55 (m, 2H), 1.21 – 1.13 (m, 5H); $^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 174.9, 158.7, 132.1, 131.7, 113.6, 61.0, 55.4, 28.5, 16.7, 14.3. HRMS (ESI-TOF) m/z [M + Na] $^+$ calcd for $\text{C}_{13}\text{H}_{16}\text{NaO}_3$ 243.0992, found 243.0985.

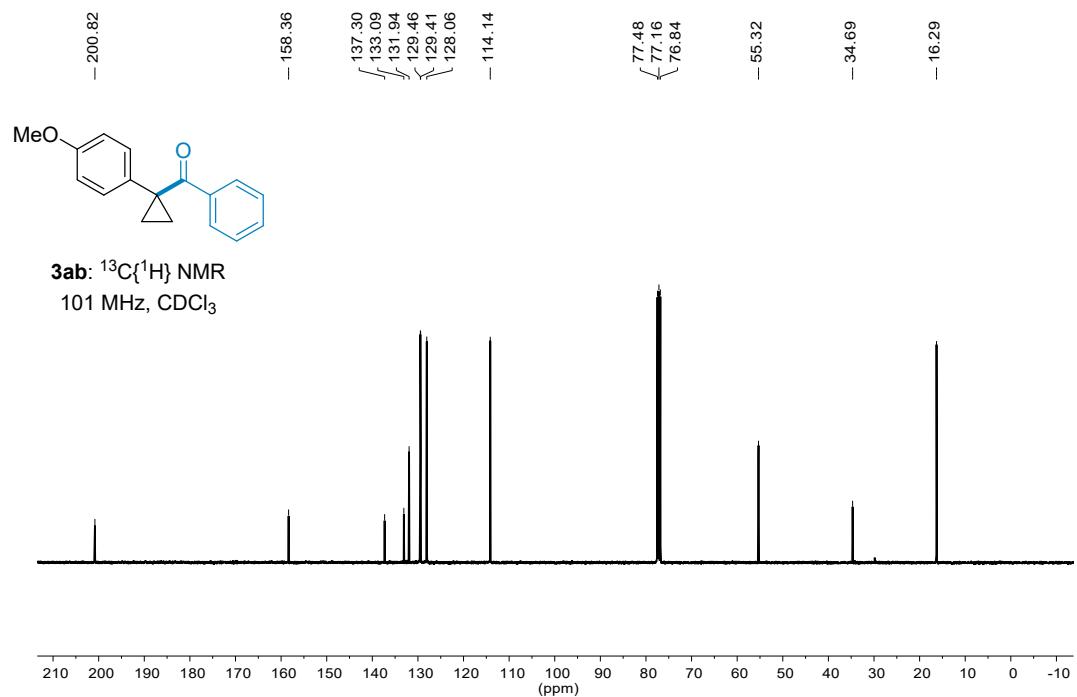
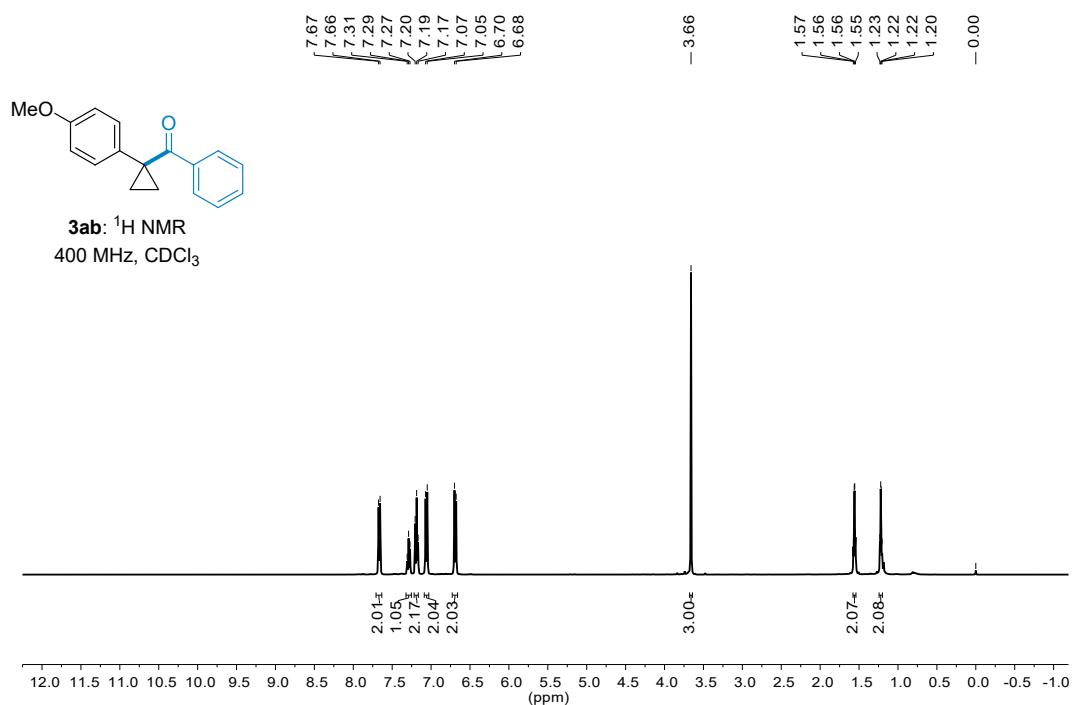
1-(4-methoxyphenyl)-N-(pyridin-2-yl)cyclopropane-1-carboxamide (7)

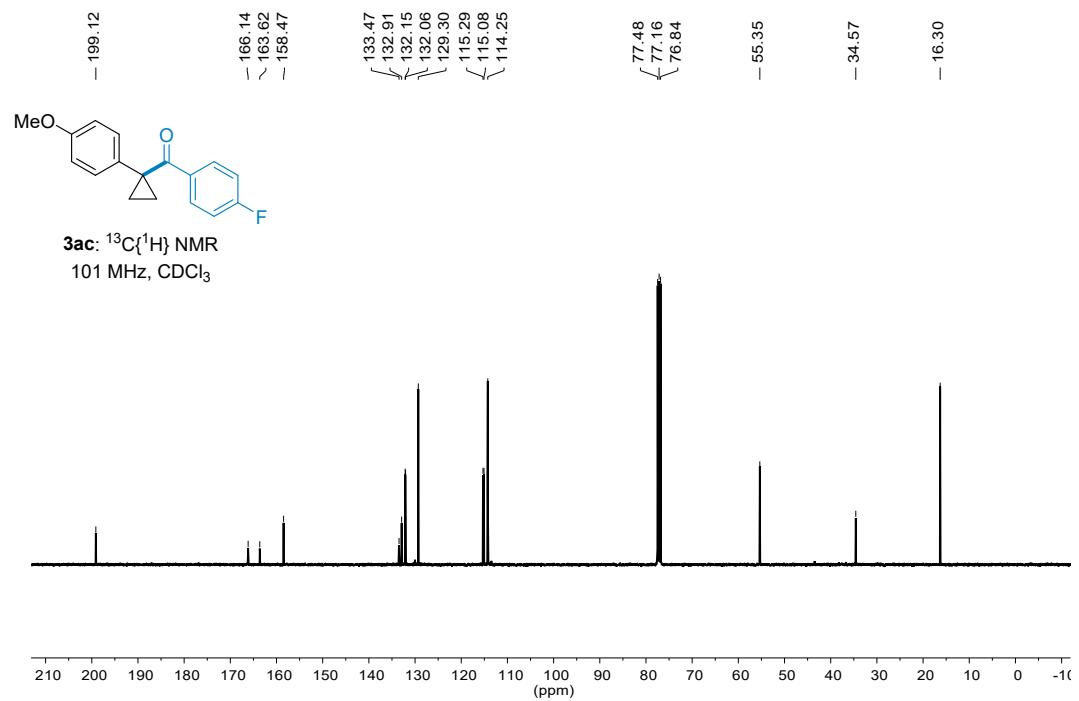
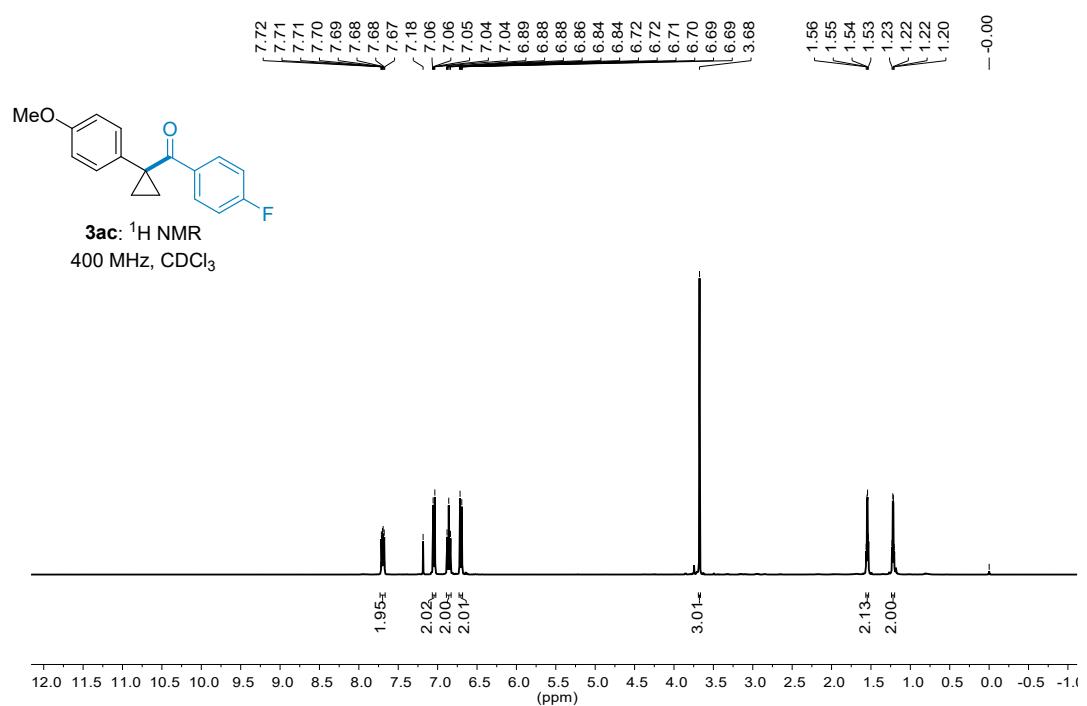


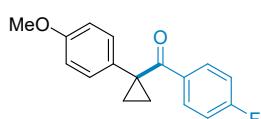
Purification by column chromatography on silica gel (eluting with pentane/ethyl acetate = 4/1, v/v) afforded the title compound as a pale yellow oil (12.1 mg, 45% yield); ^1H NMR (400 MHz, CDCl_3) δ 8.19 (d, J = 8.4 Hz, 1H), 8.16 (d, J = 5.0 Hz, 1H), 7.79 (brs, 1H), 7.65 (t, J = 7.9 Hz, 1H), 7.40 (d, J = 8.5 Hz, 2H), 6.97 – 6.73 (m, 3H), 3.84 (s, 3H), 1.70 (q, J = 3.8 Hz, 2H), 1.16 (q, J = 3.8 Hz, 2H); $^{13}\text{C}\{\text{H}\}$ NMR (101 MHz, CDCl_3) δ 172.2, 158.8, 150.5, 146.9, 137.3, 131.4, 129.8, 118.7, 114.0, 112.8, 54.5, 29.7, 16.1. HRMS (ESI-TOF) m/z [M + Na] $^+$ calcd for $\text{C}_{16}\text{H}_{16}\text{N}_2\text{NaO}_2$ 291.1104, found 291.1097.

9. ^1H , ^{13}C , and ^{19}F NMR spectra



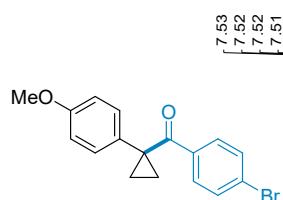
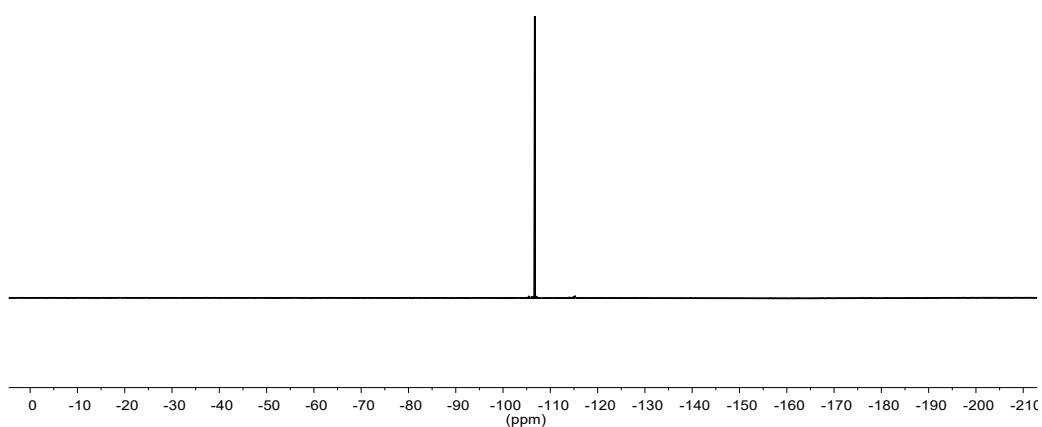






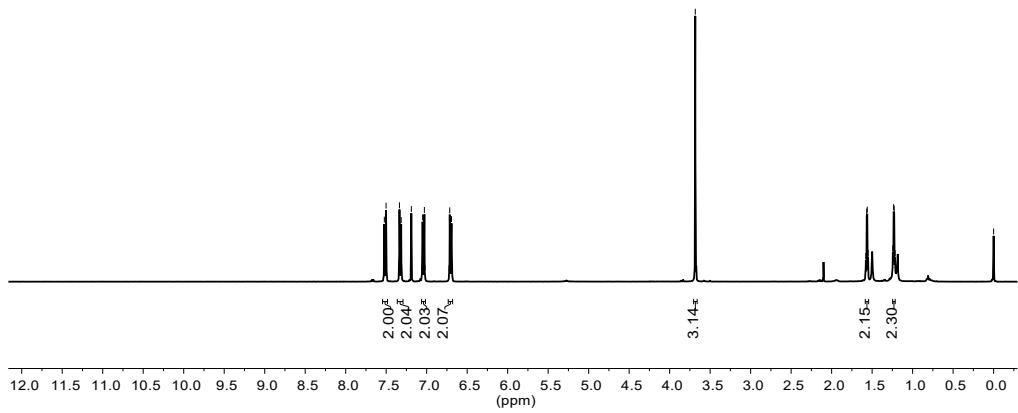
3ac: ^1H NMR

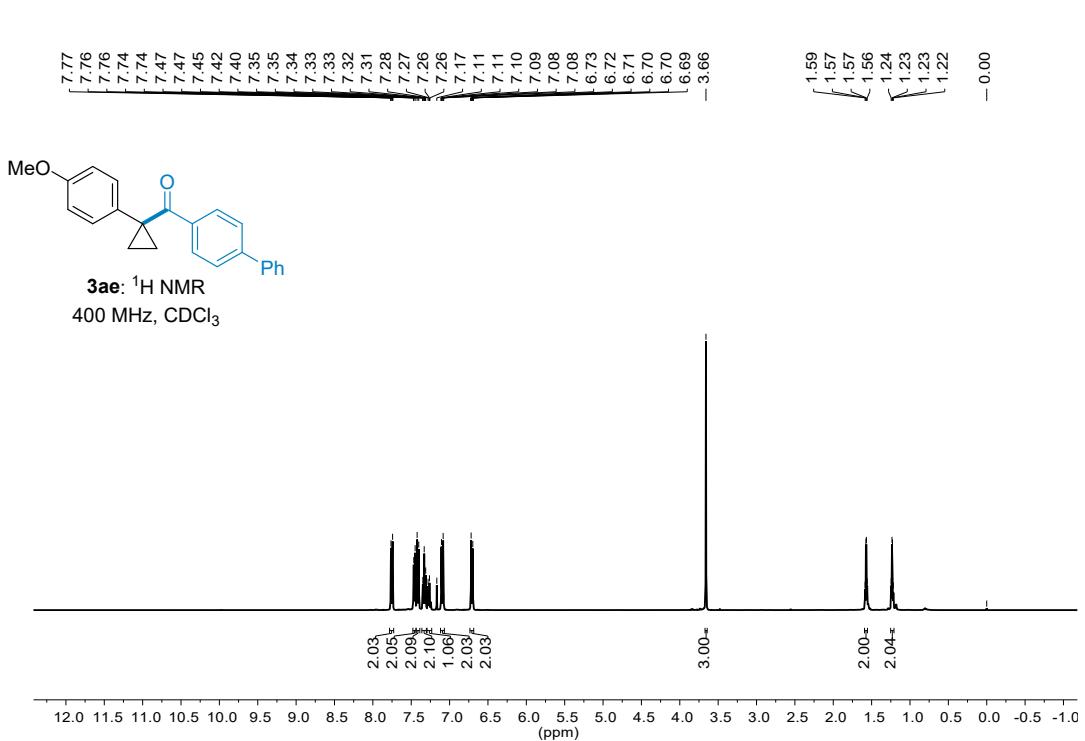
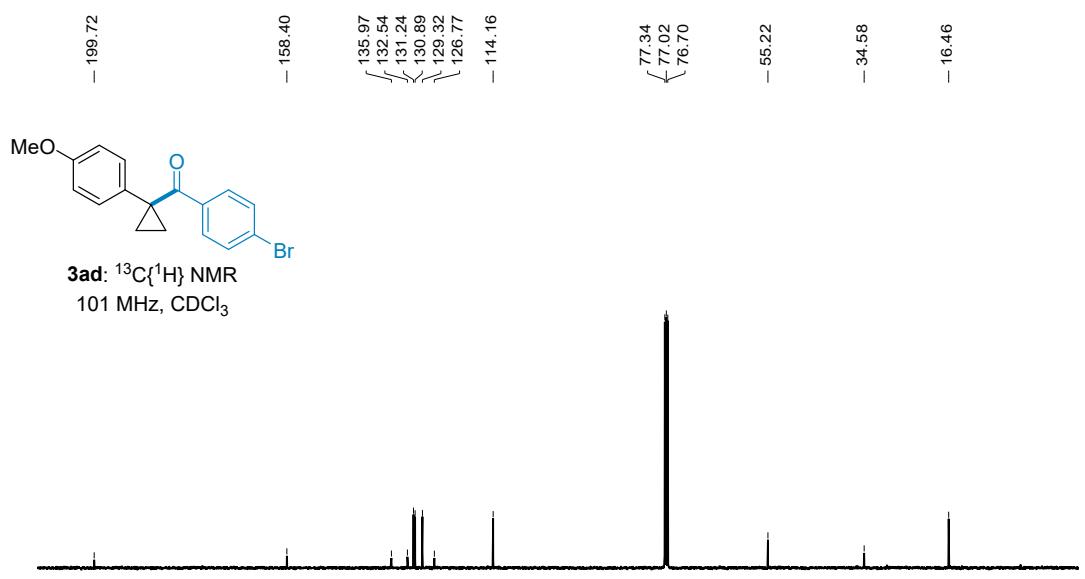
376 MHz, CDCl_3

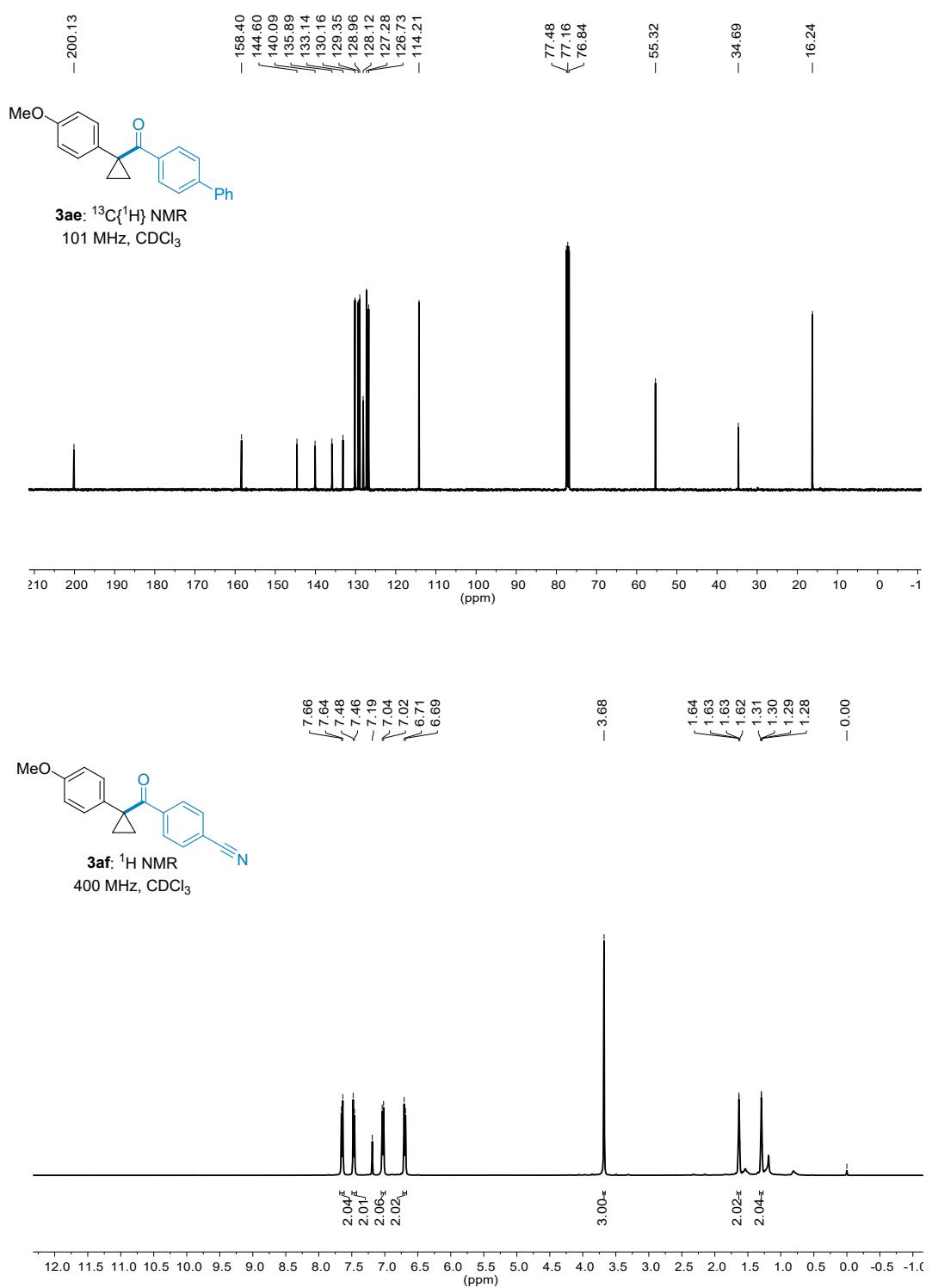


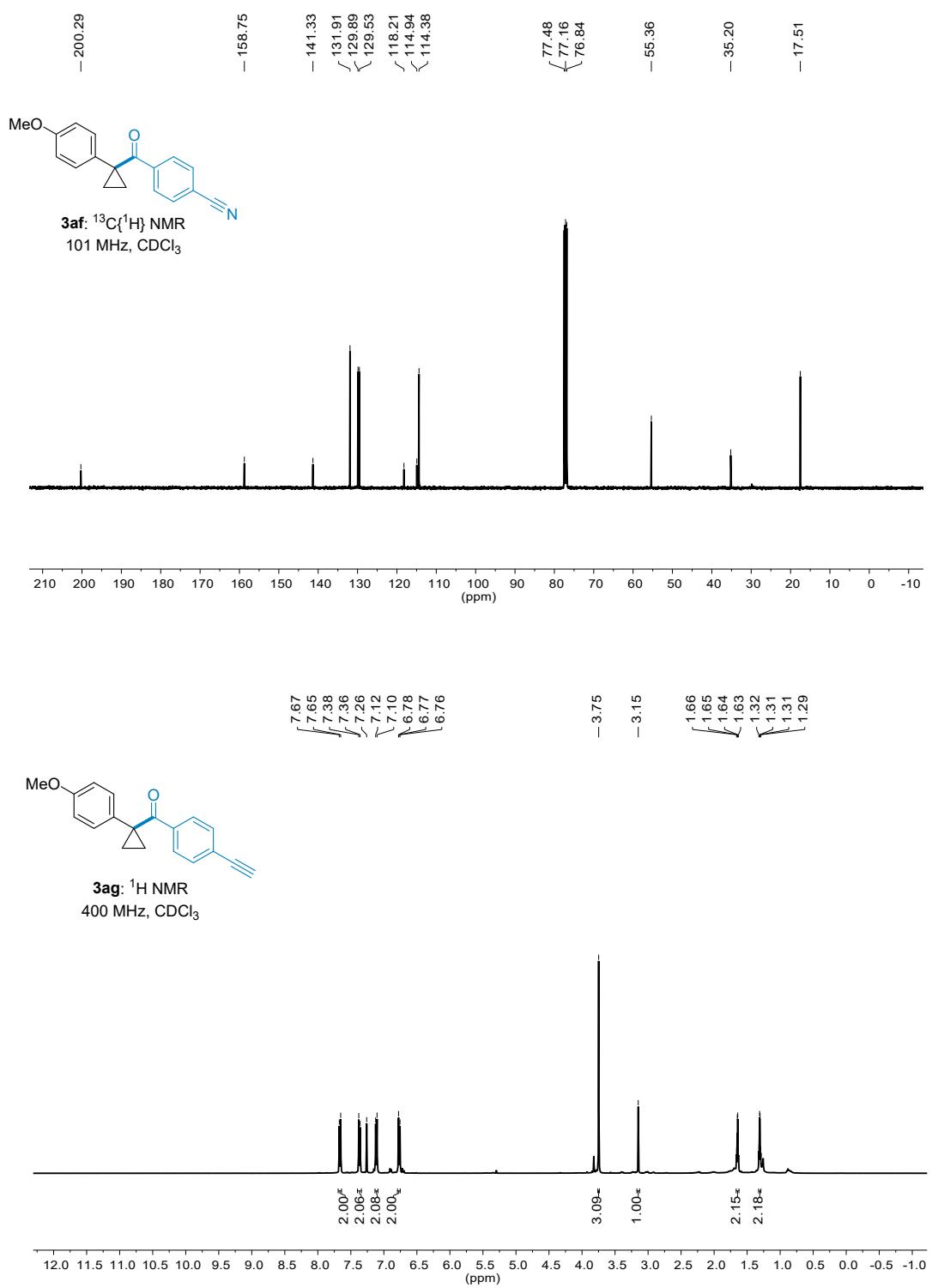
3ad: ^1H NMR

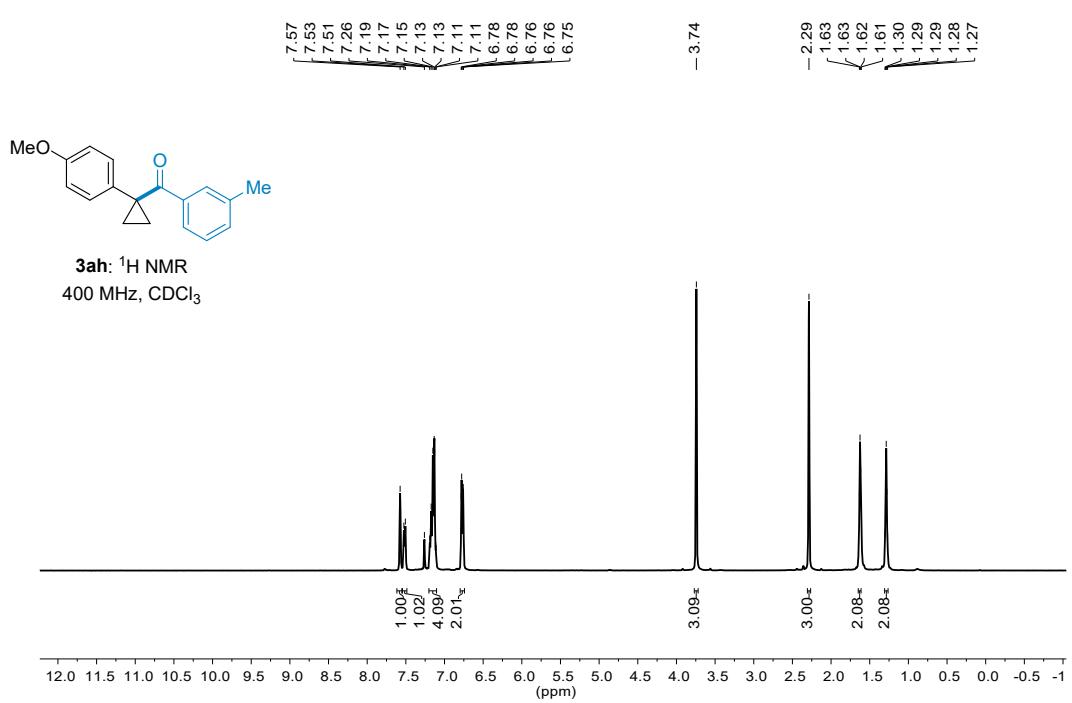
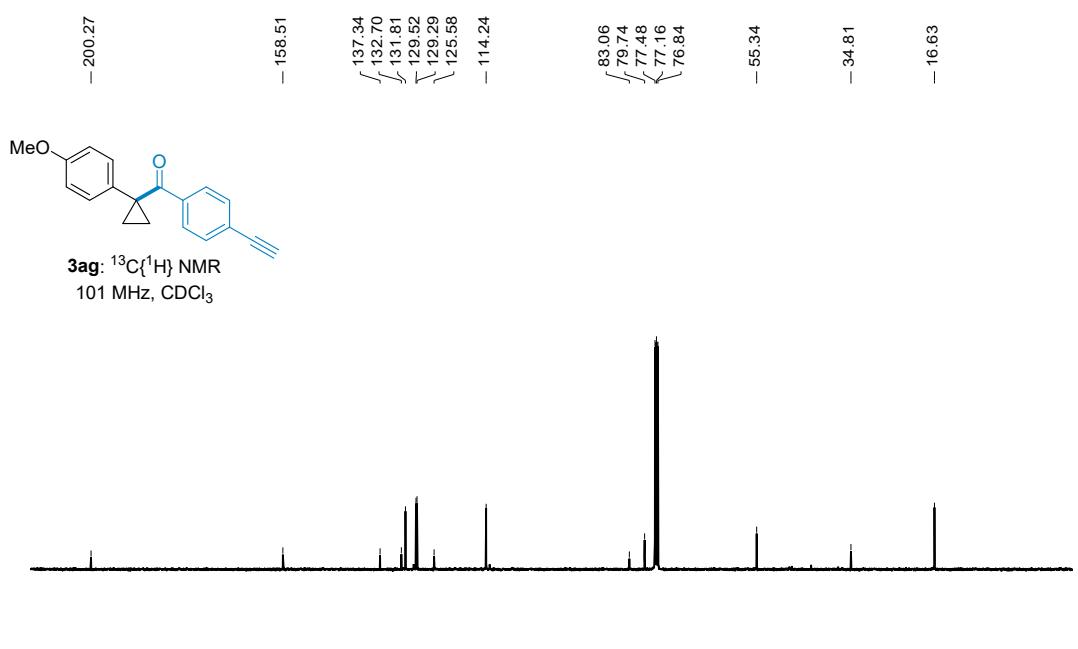
400 MHz, CDCl_3

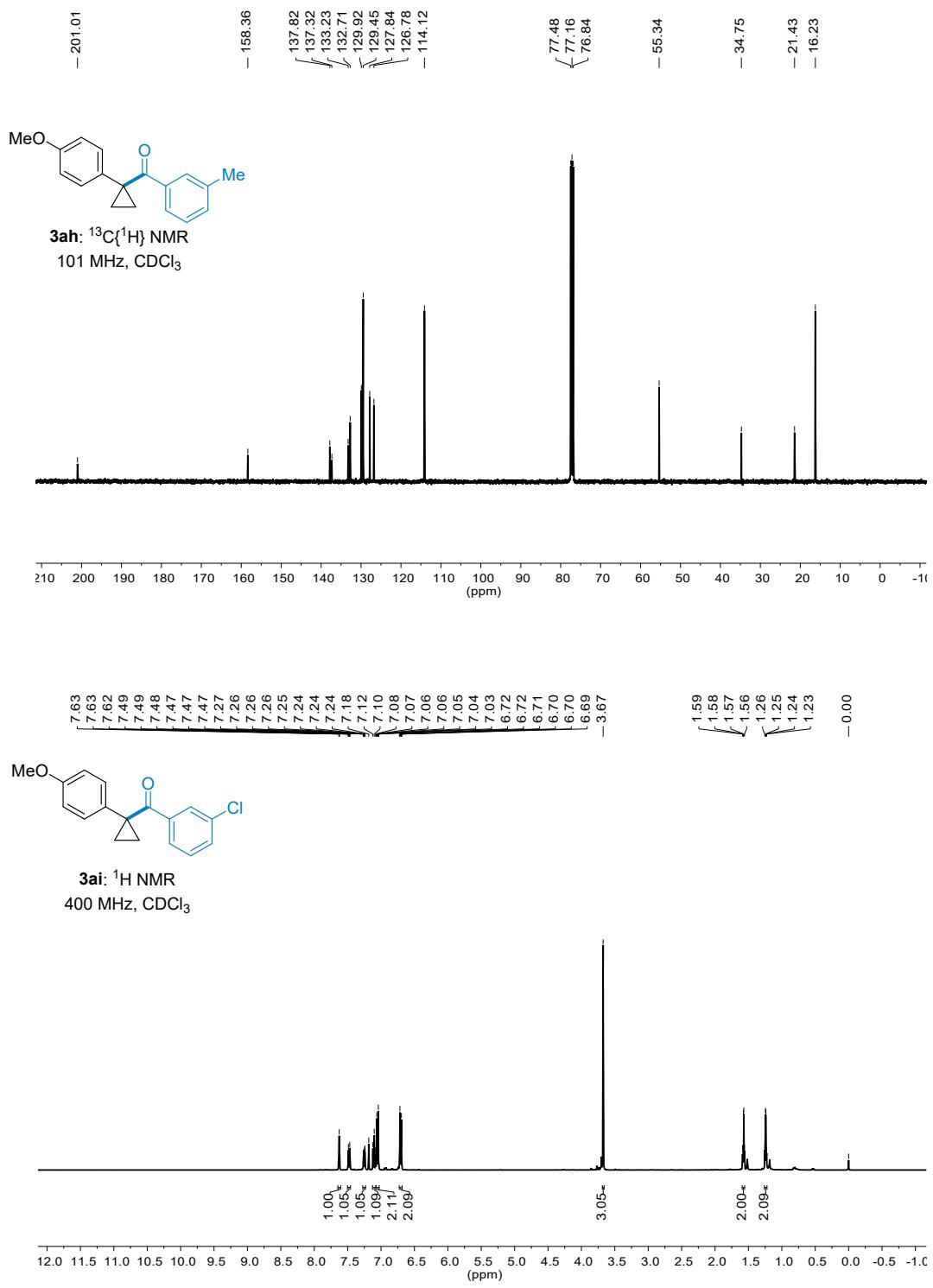


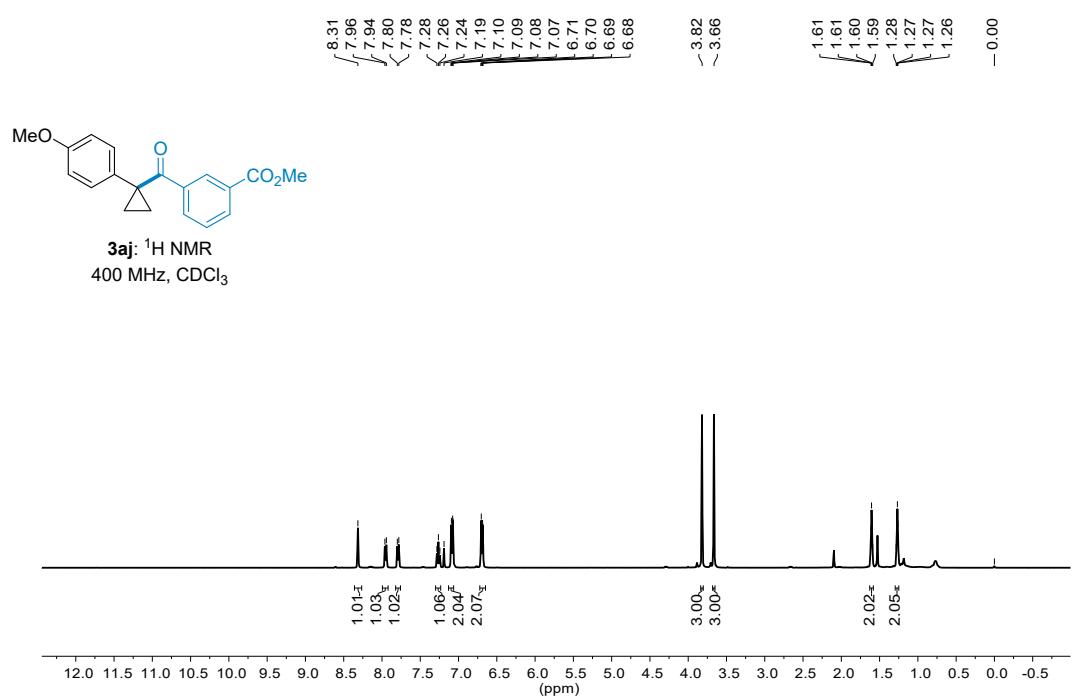
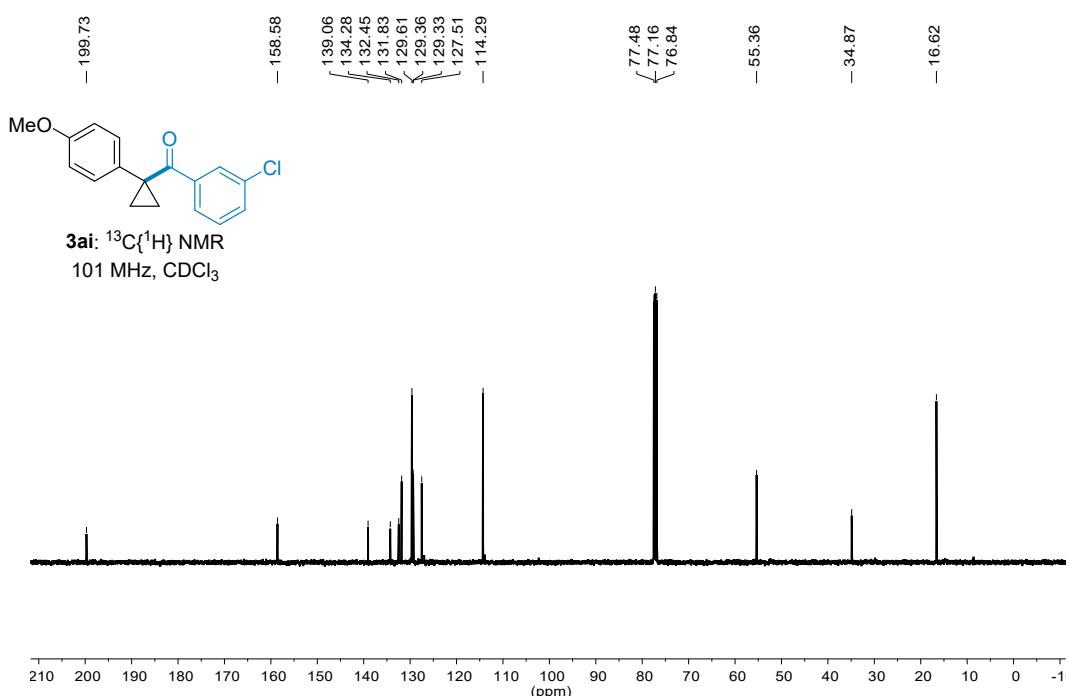


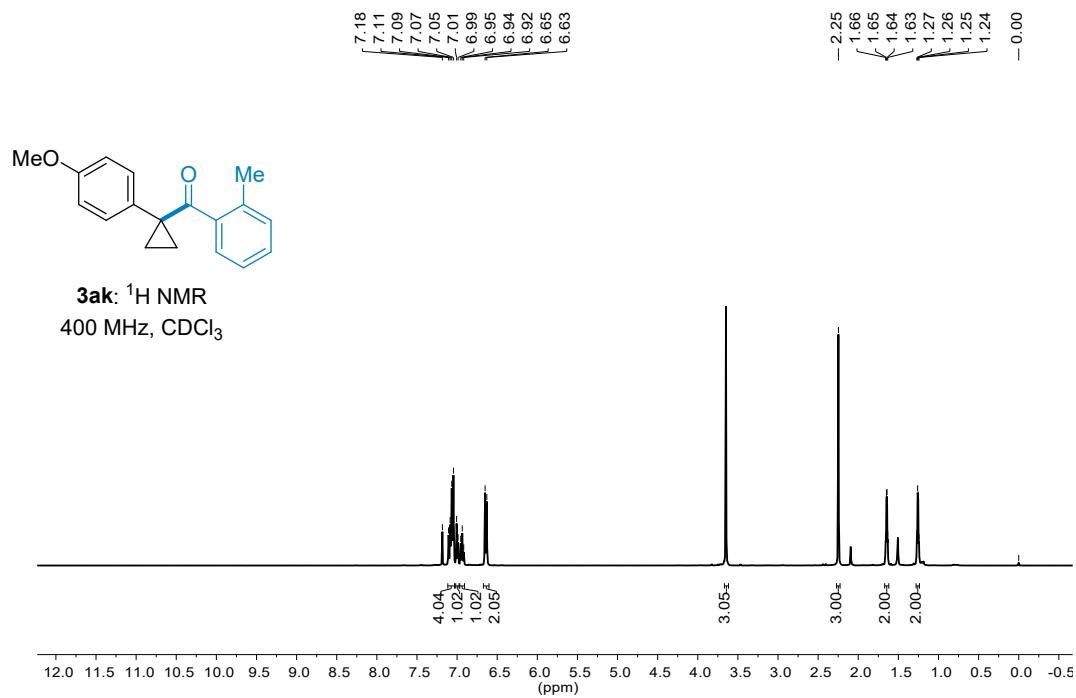
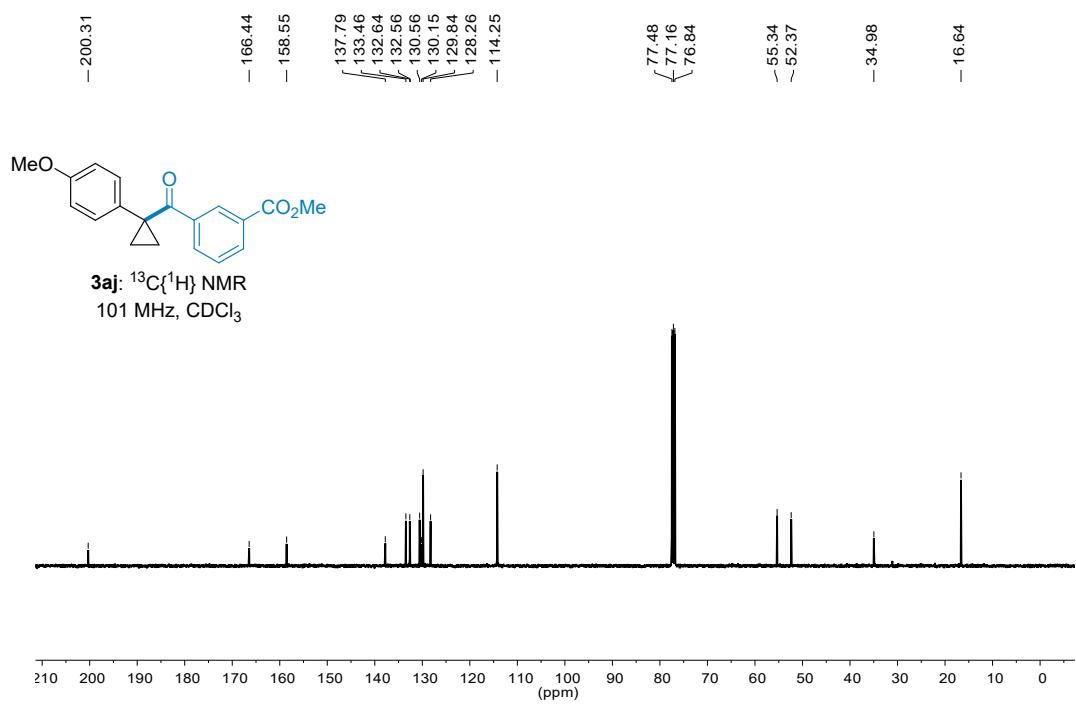


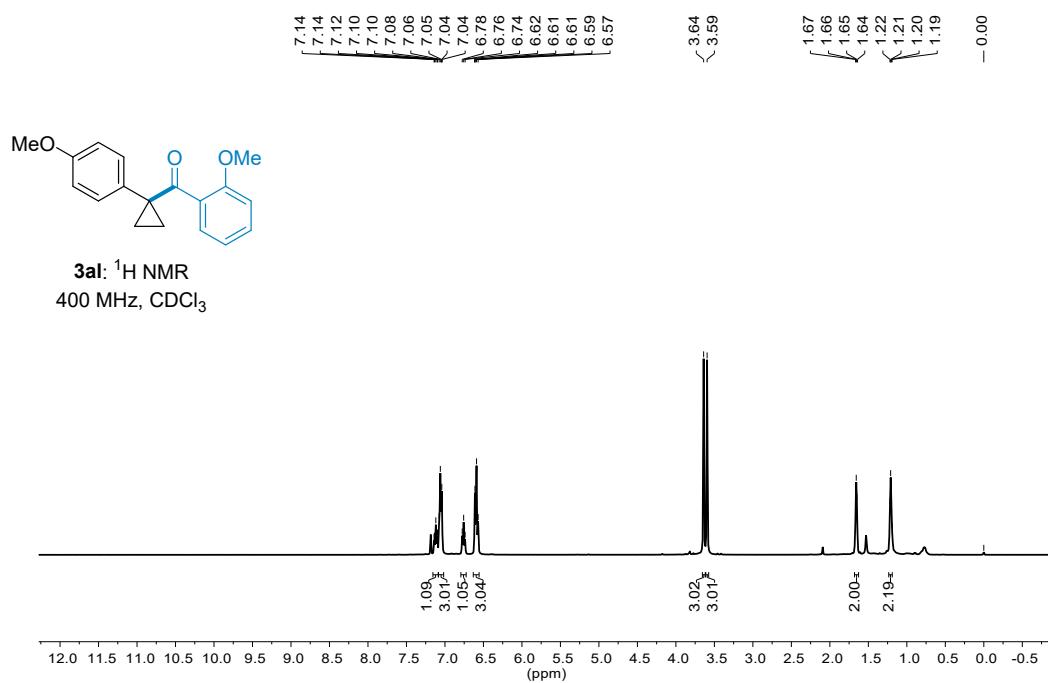
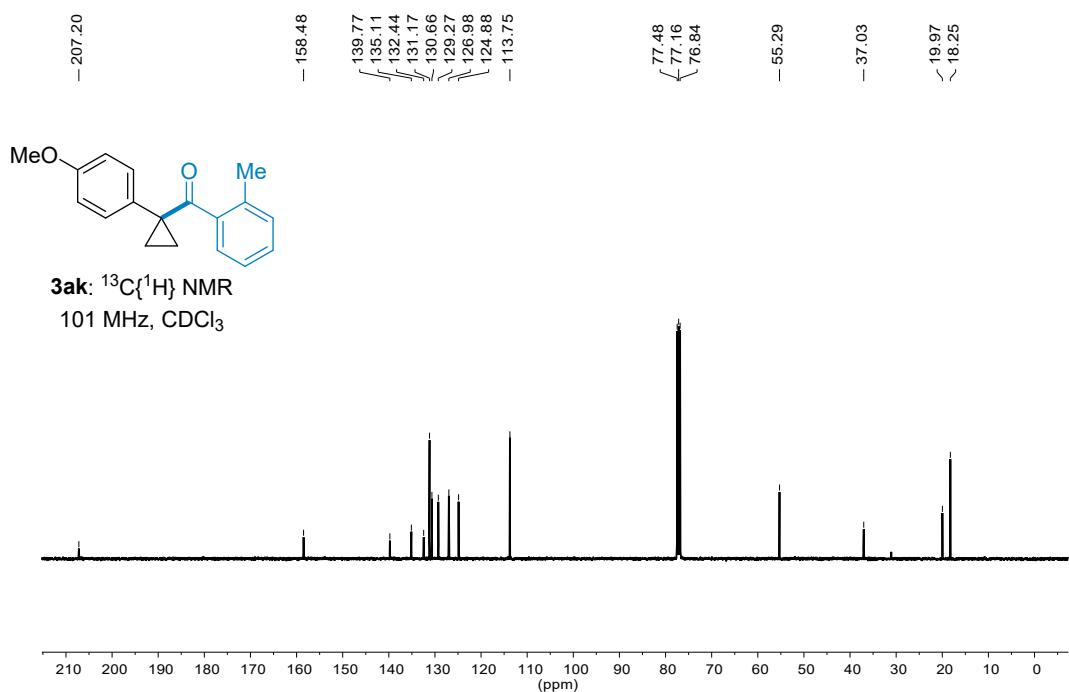


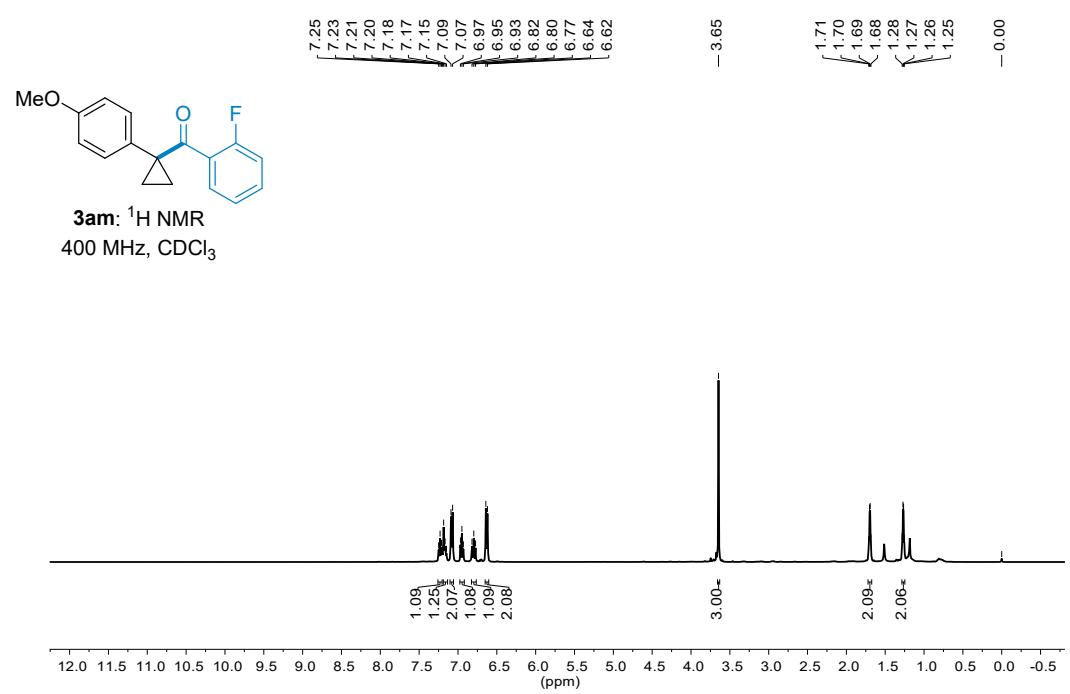
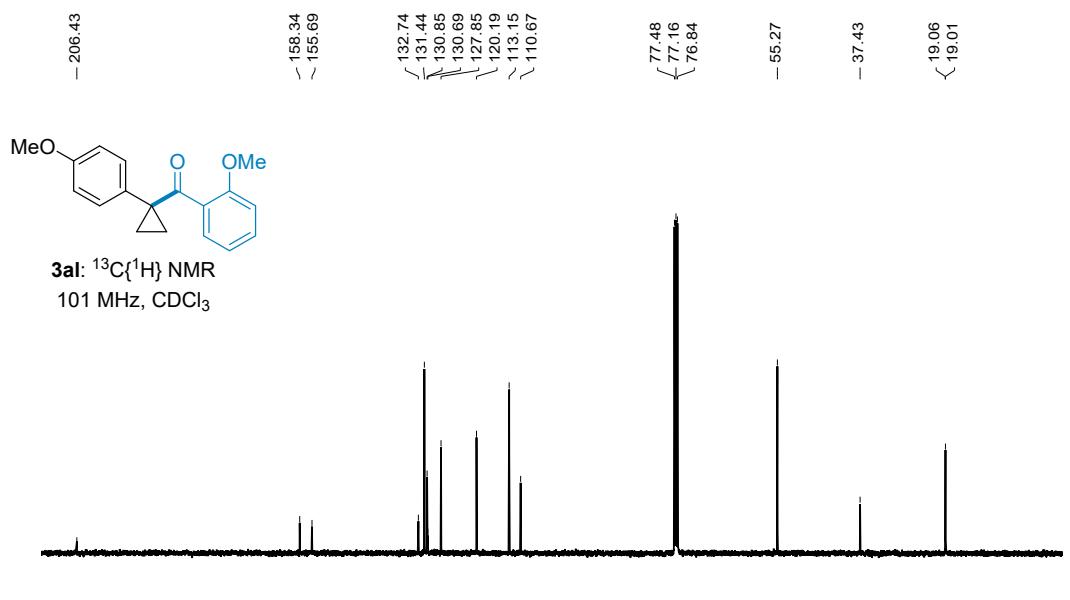


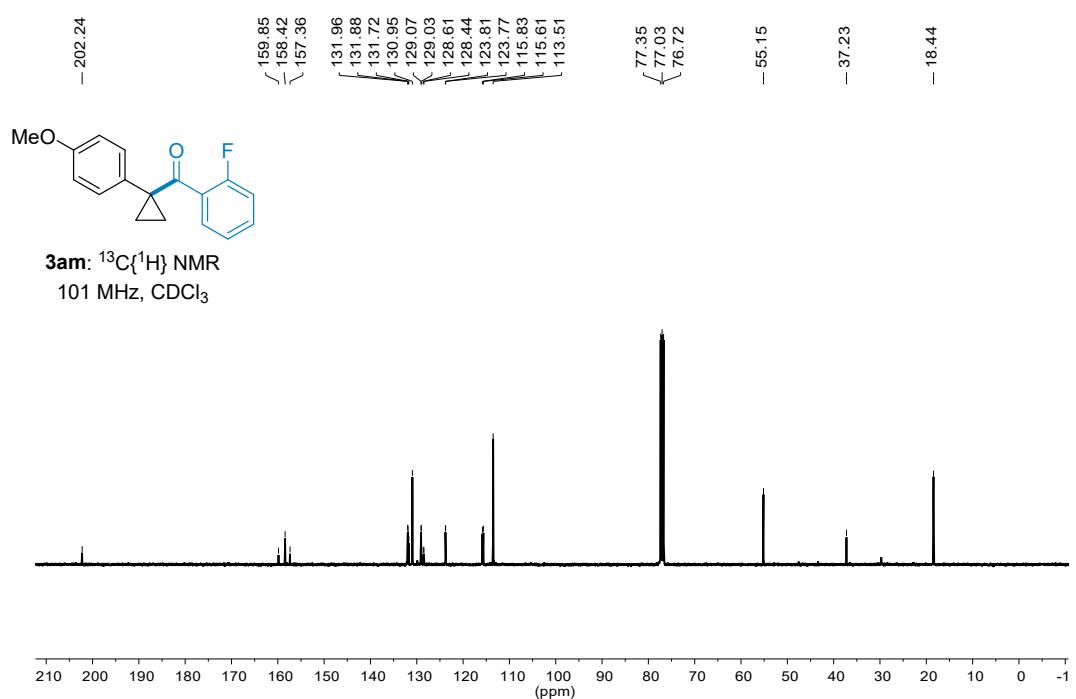


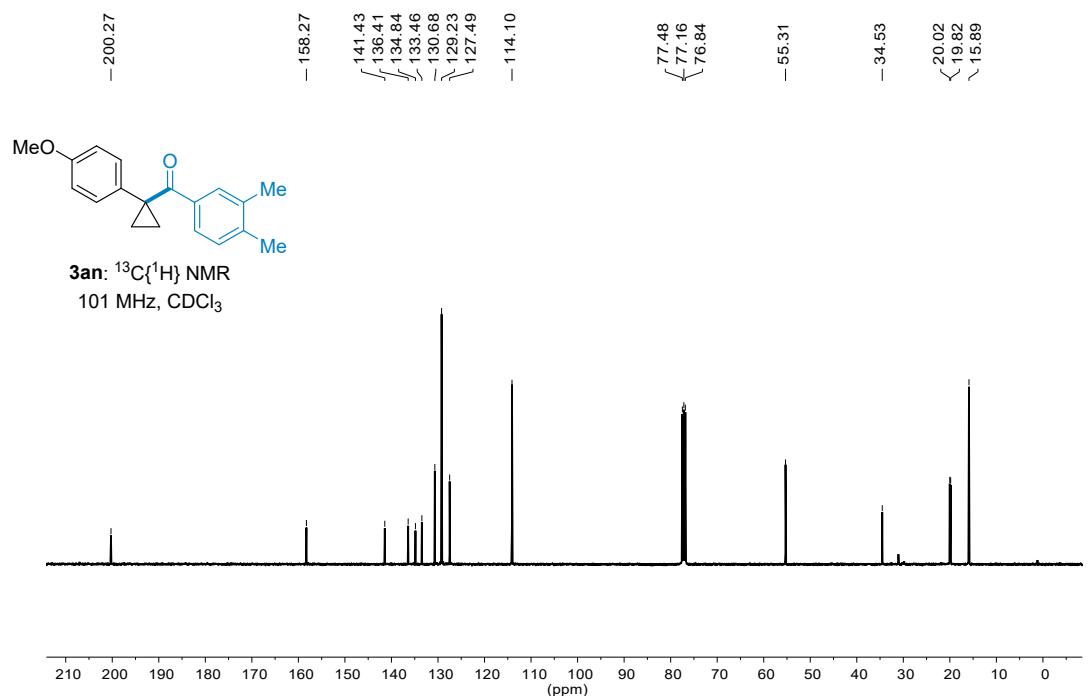
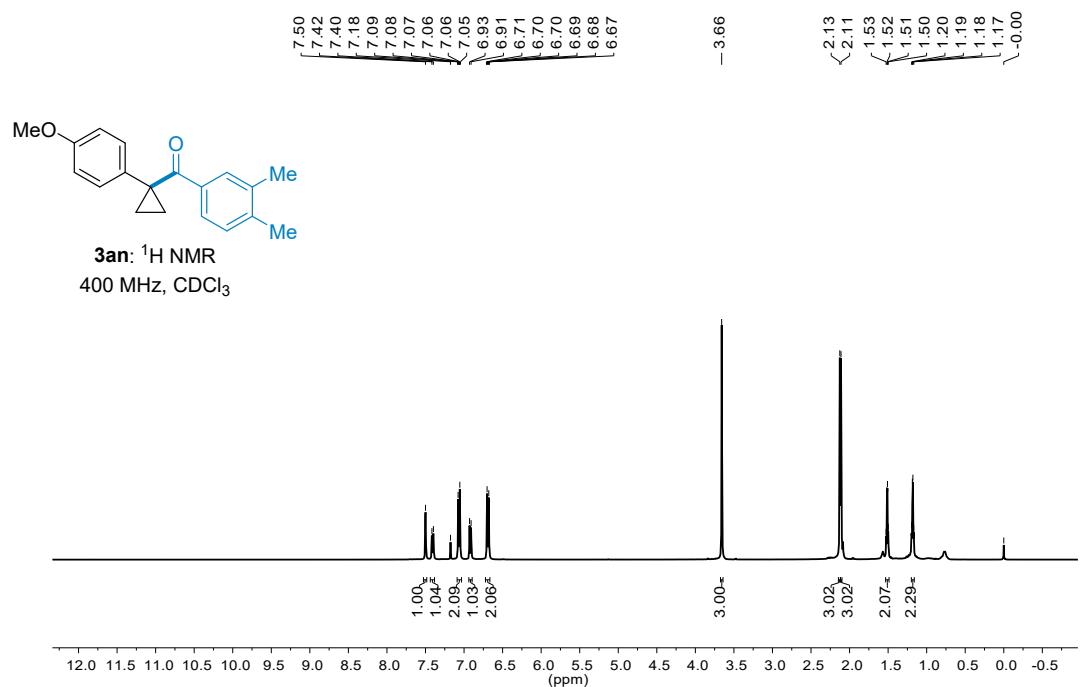


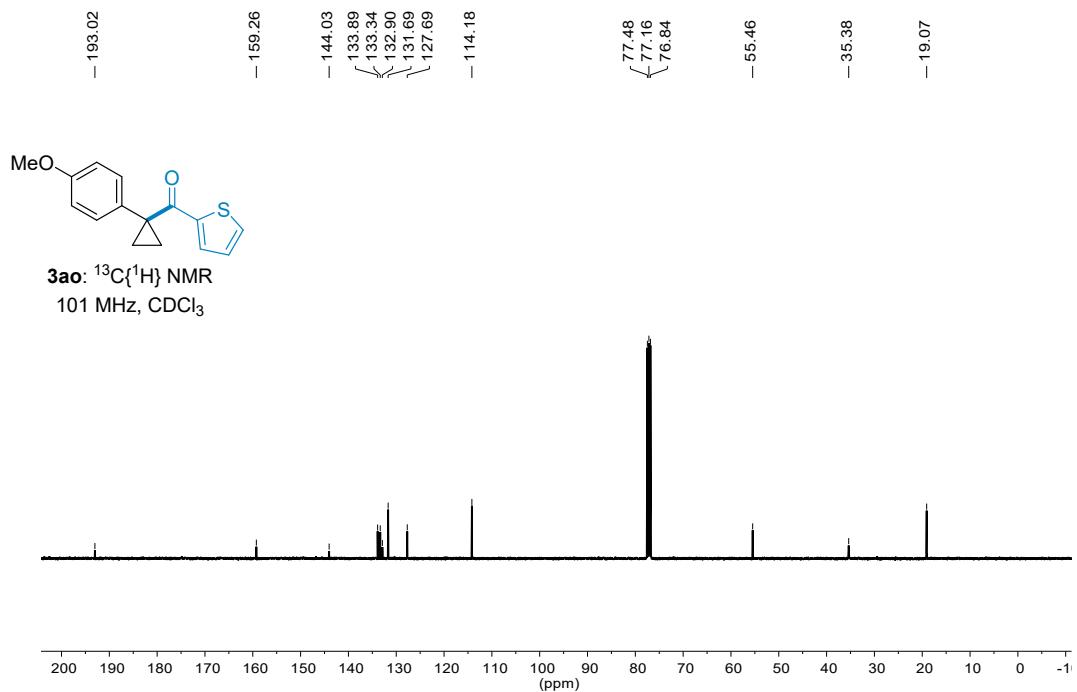
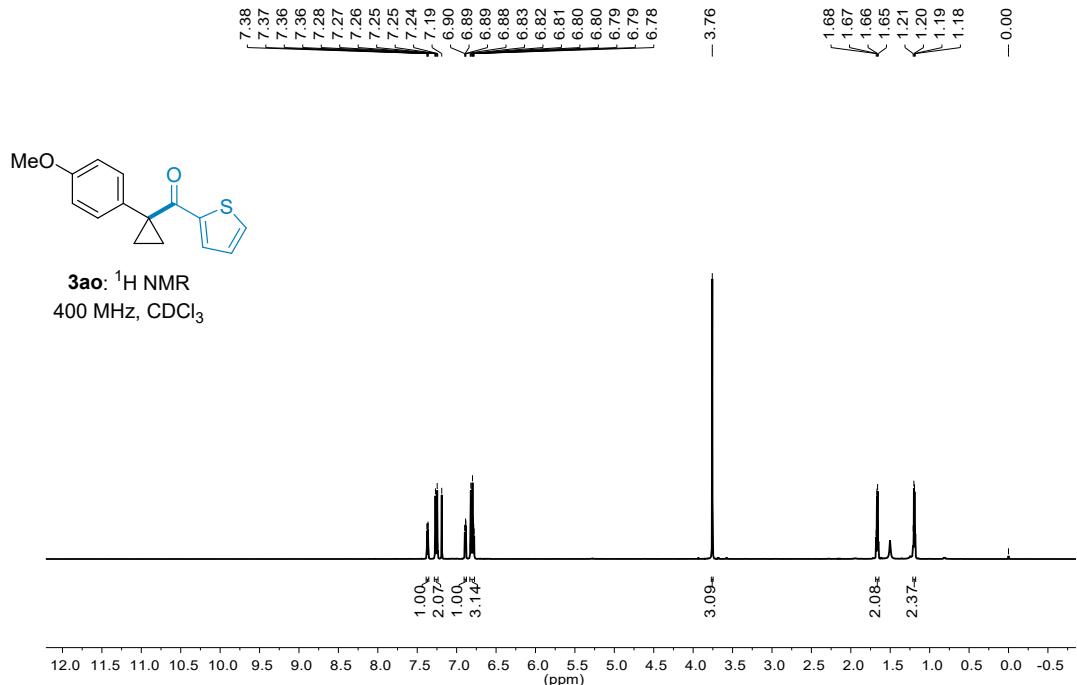


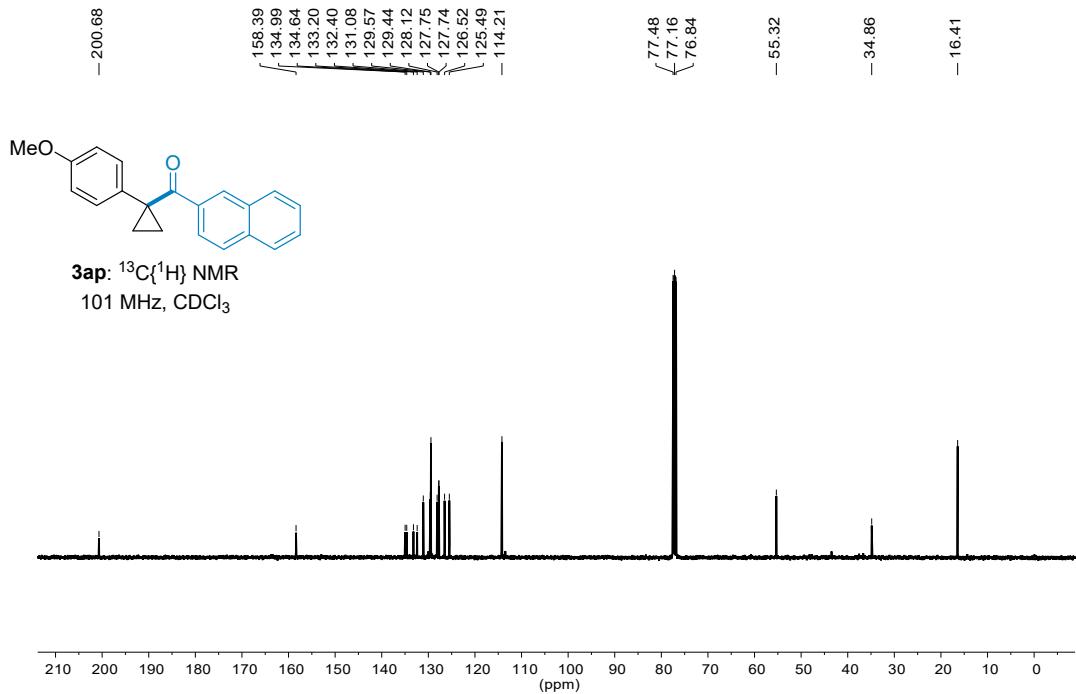
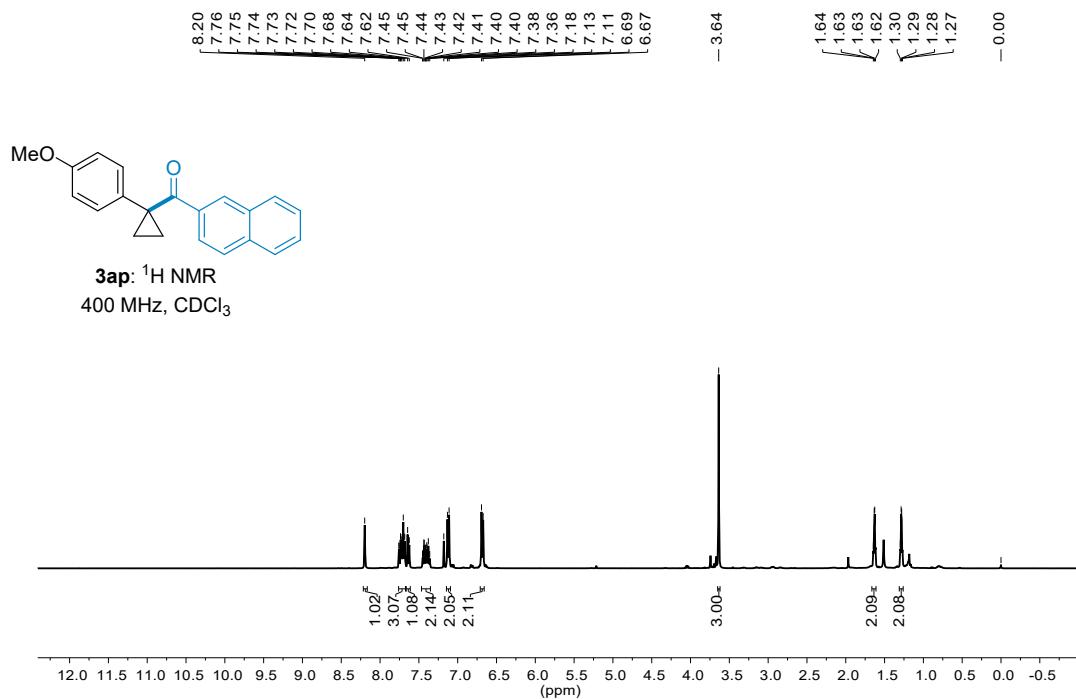


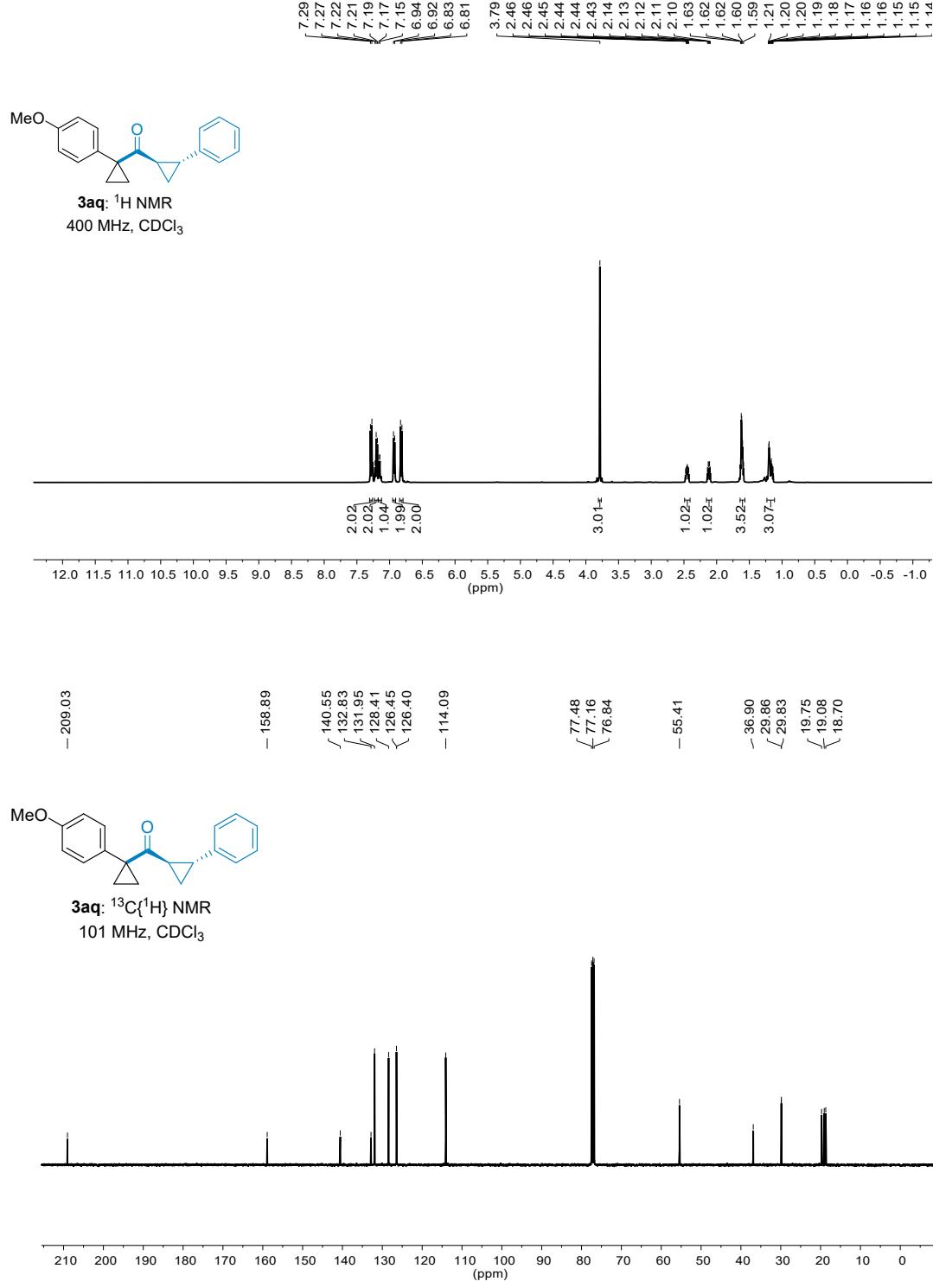


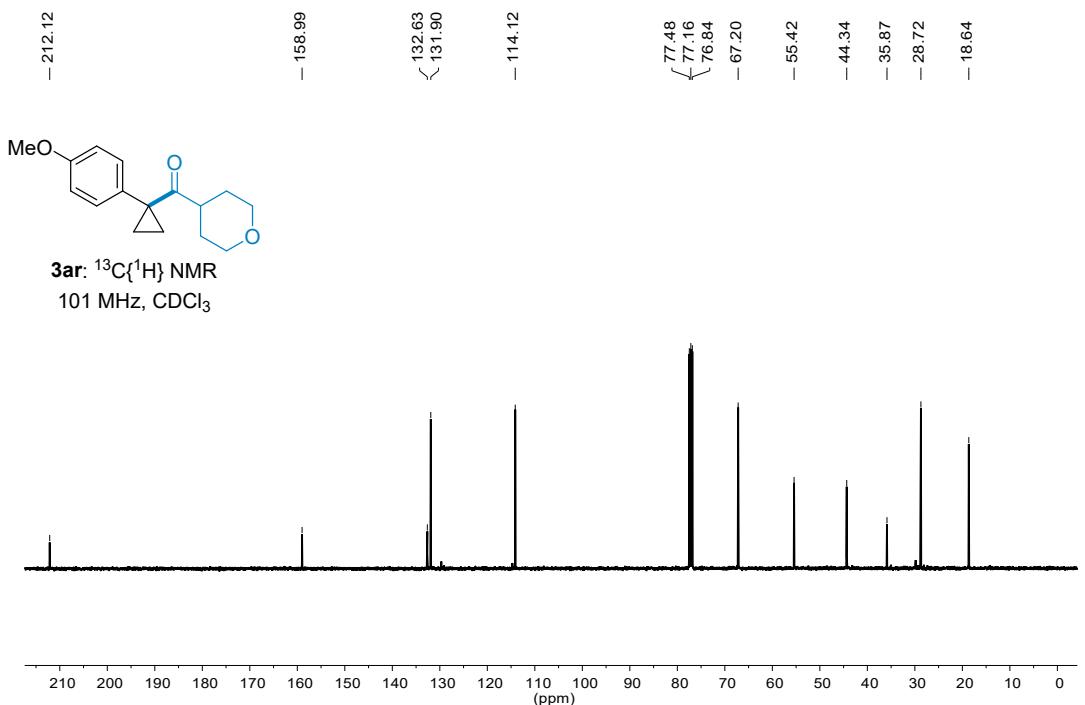
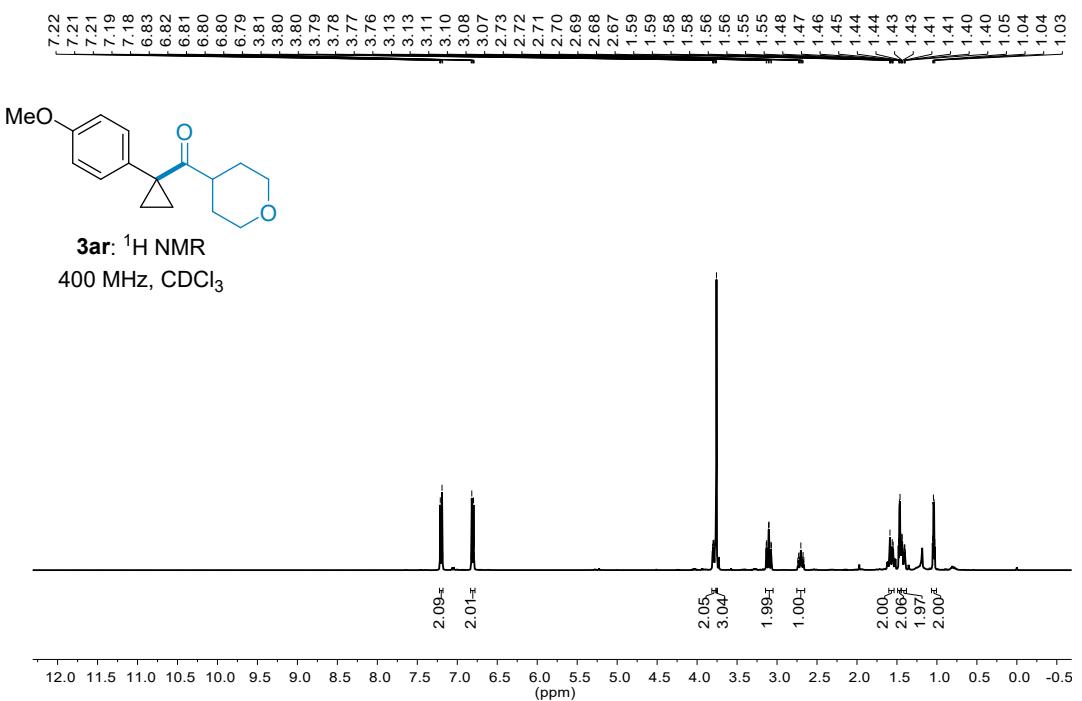


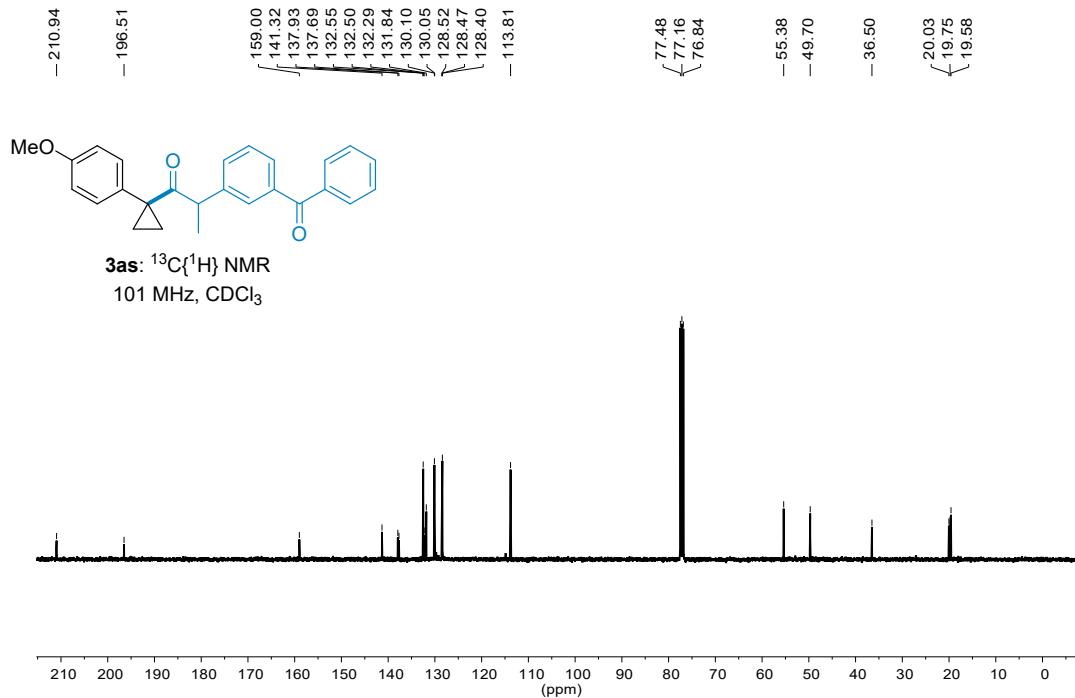
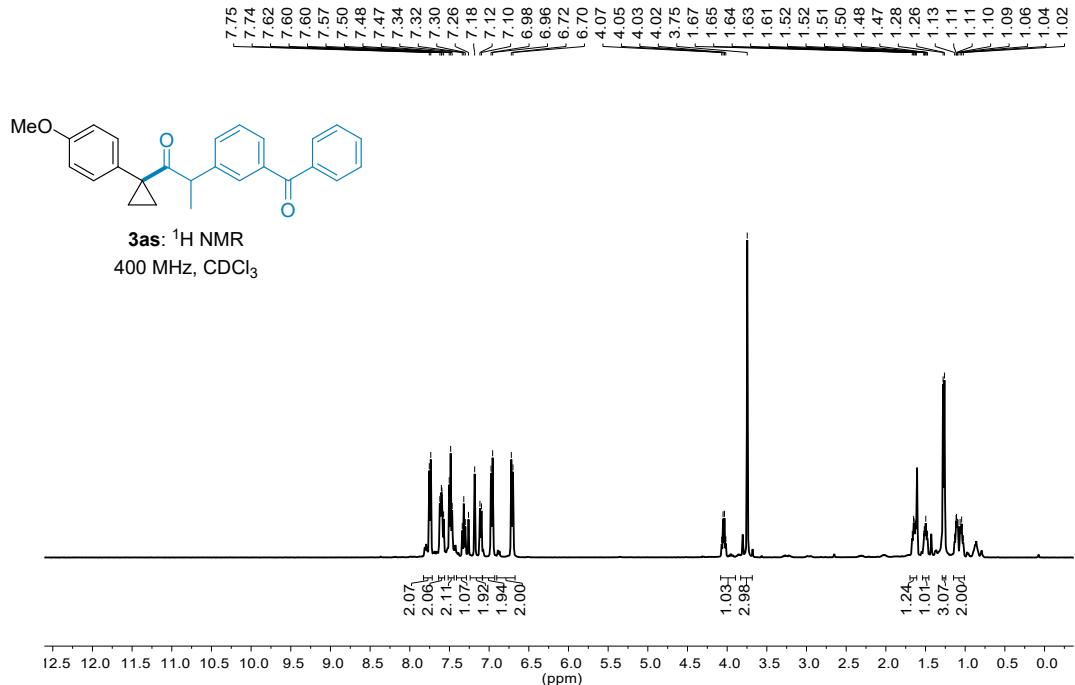


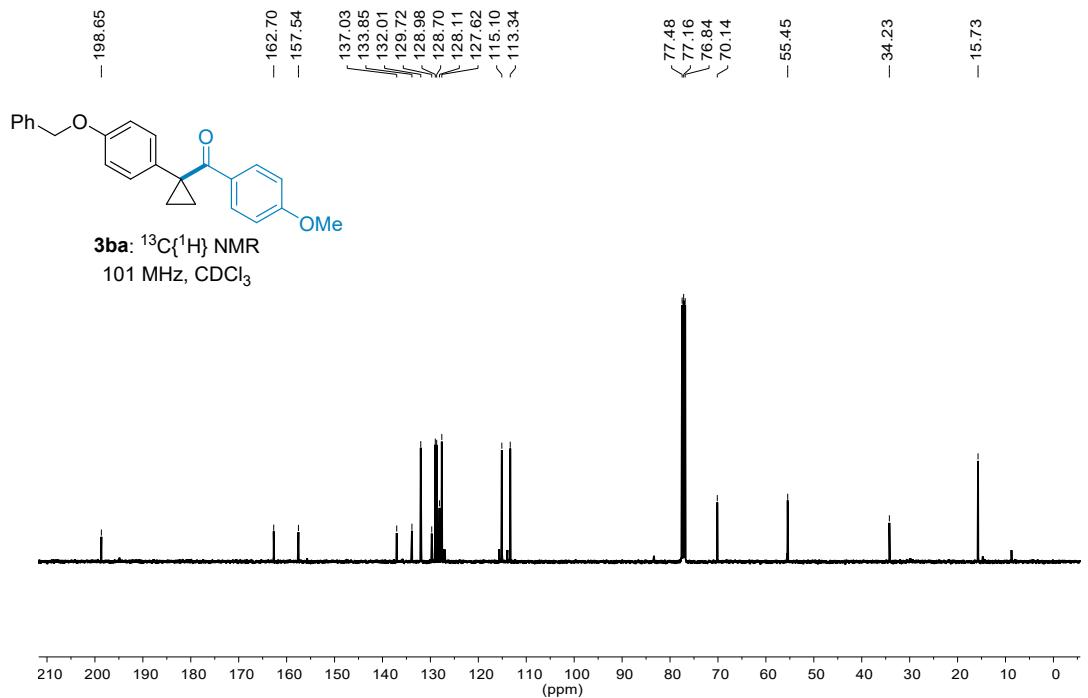
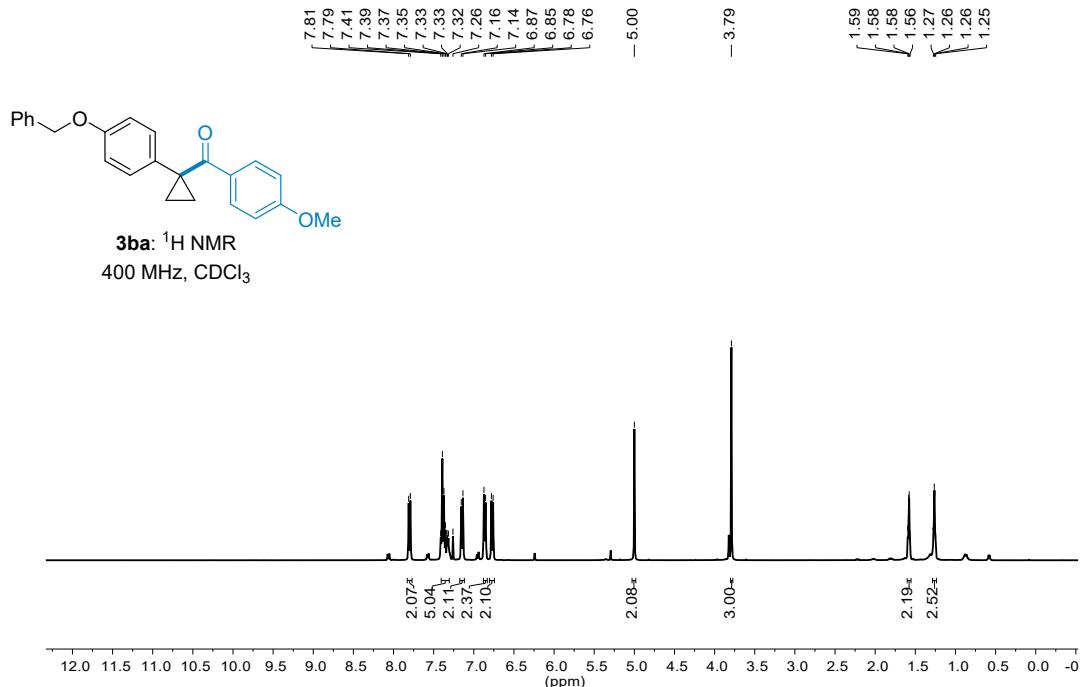


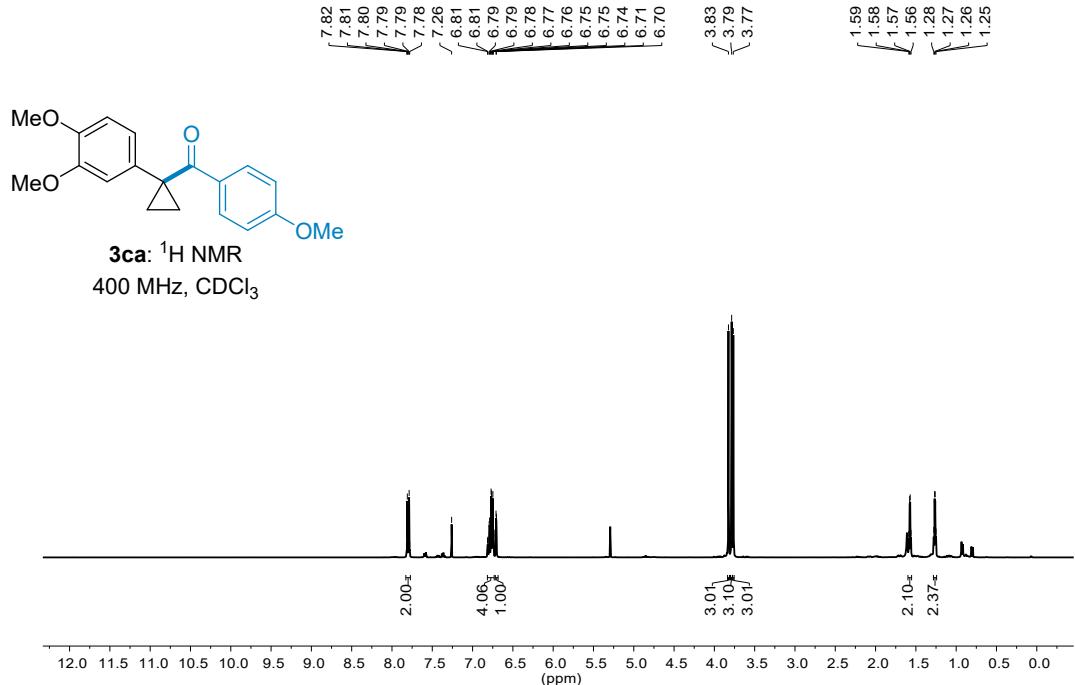


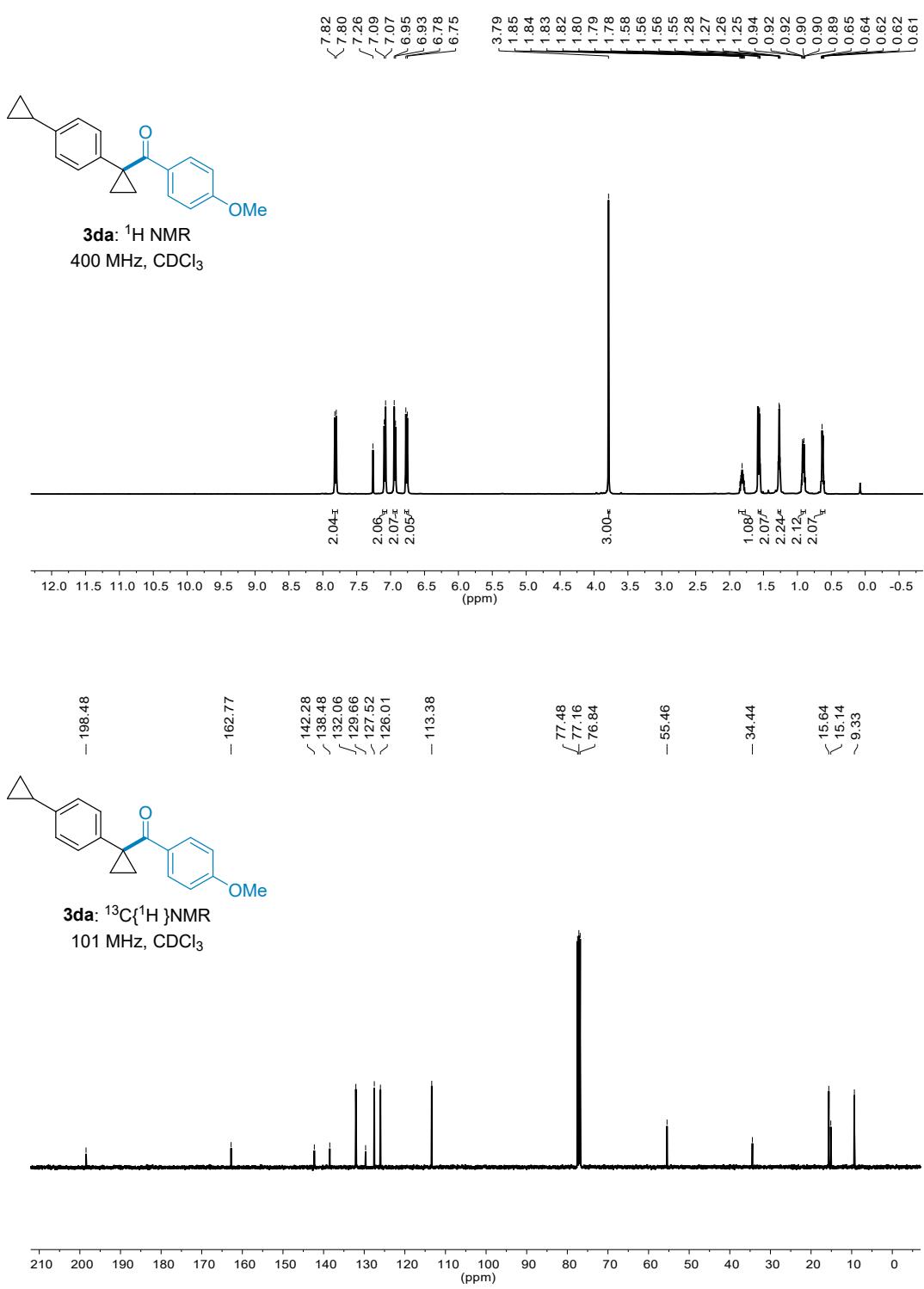


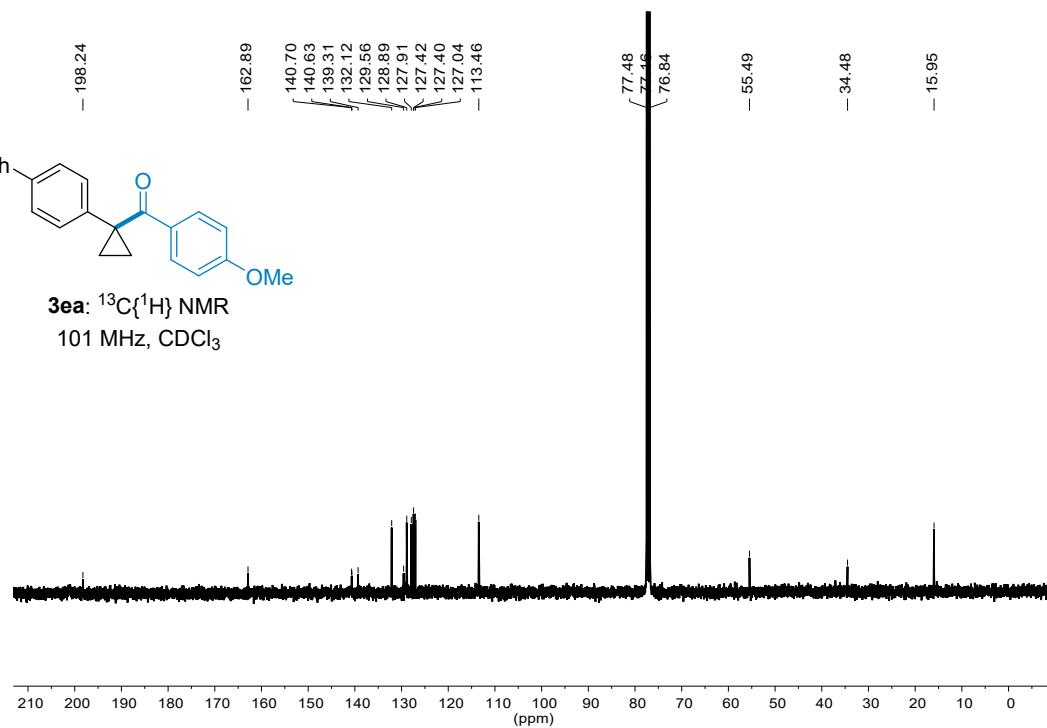
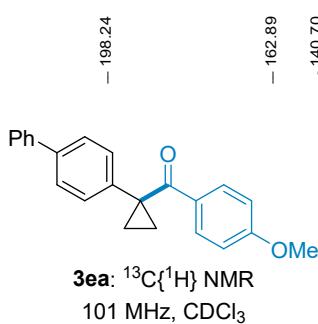
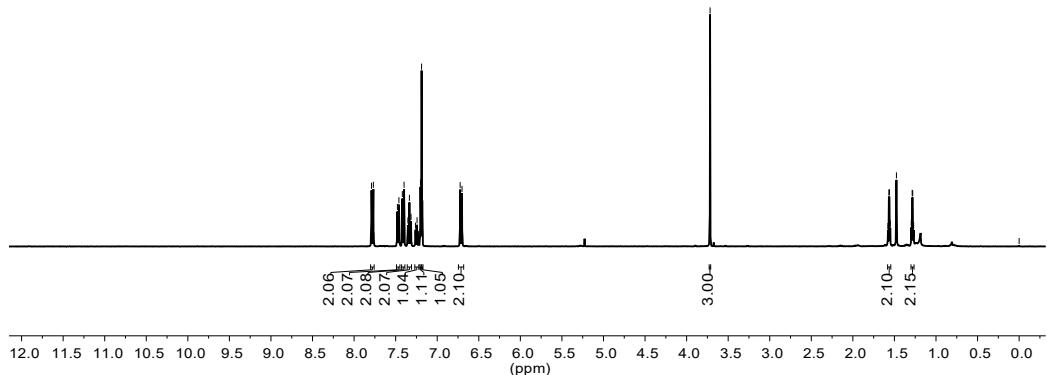
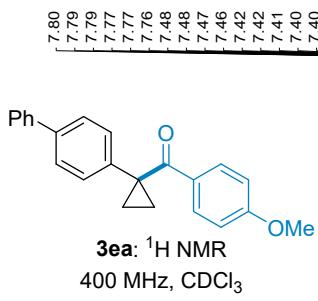


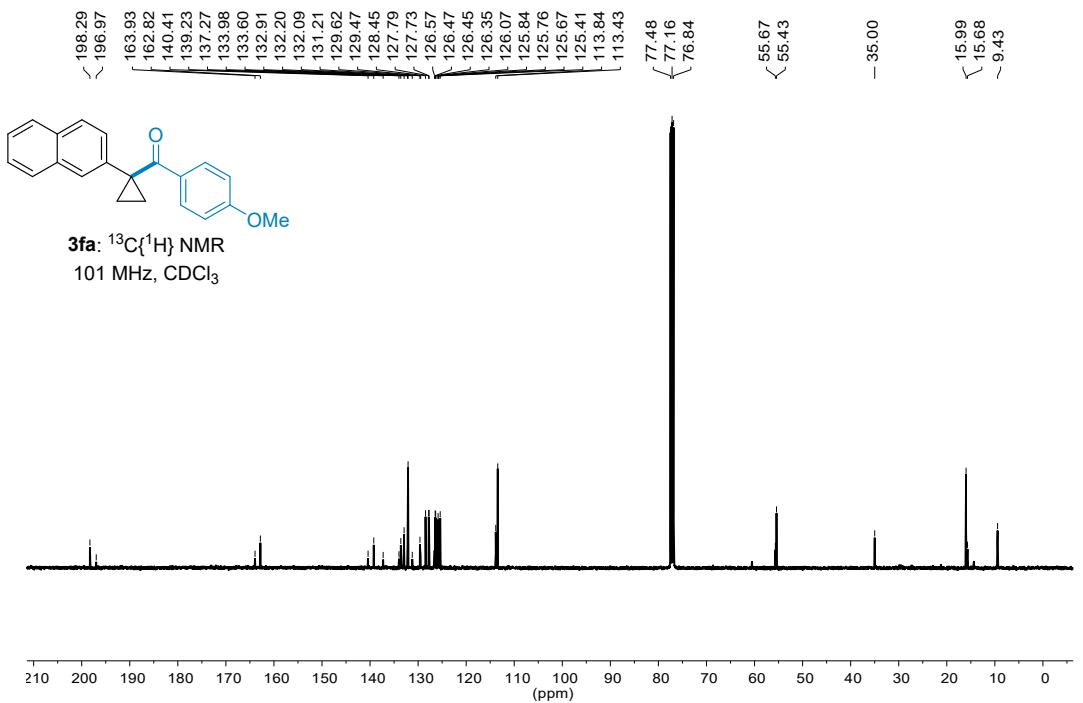
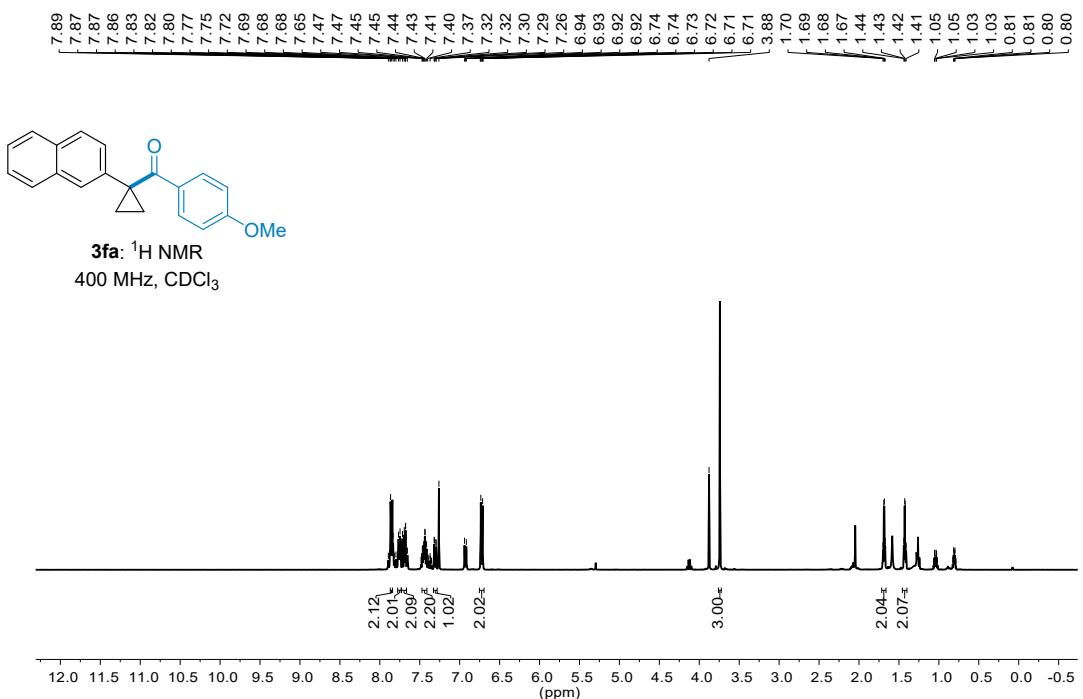


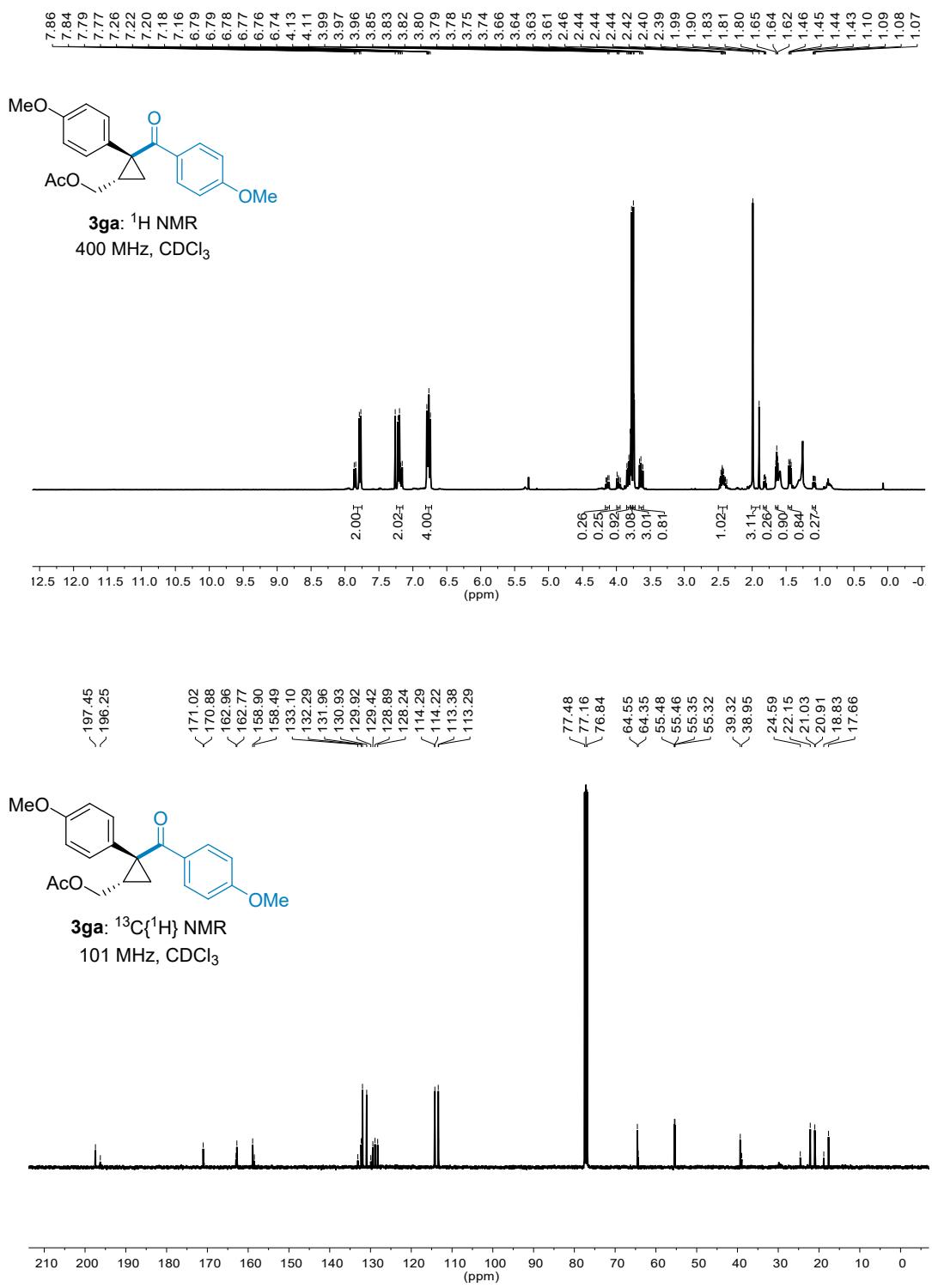


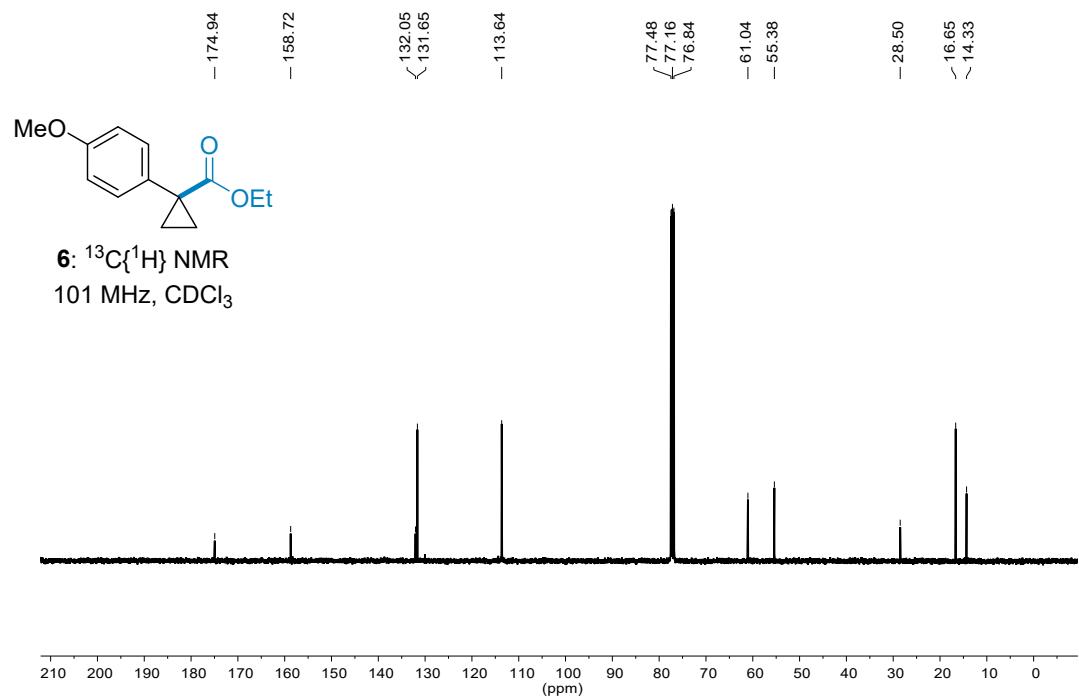
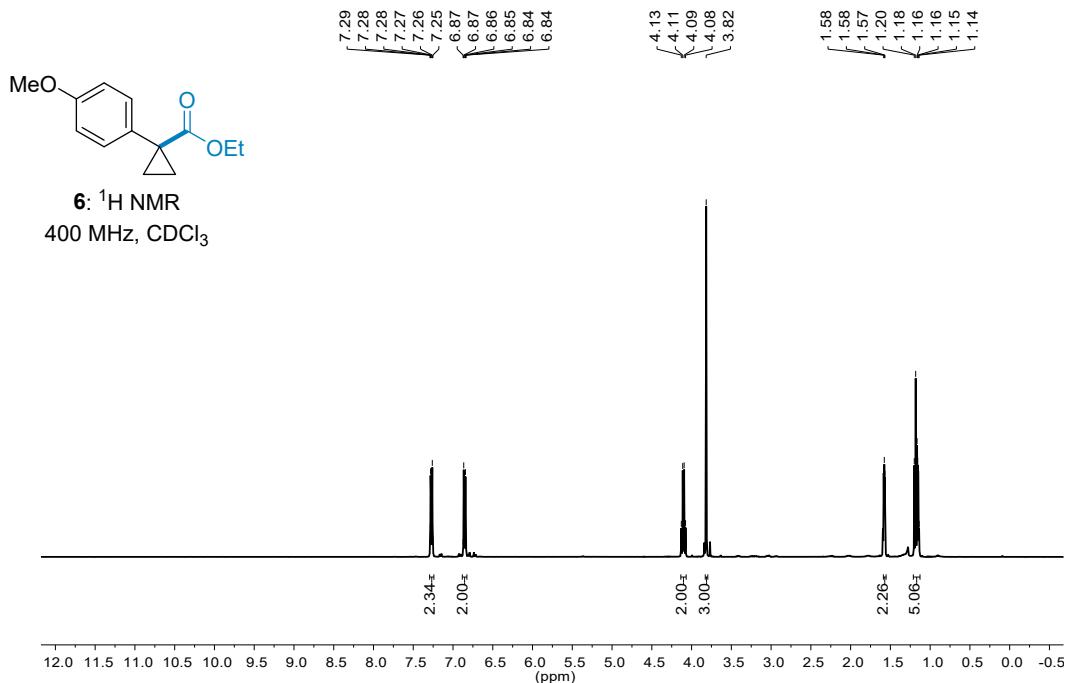


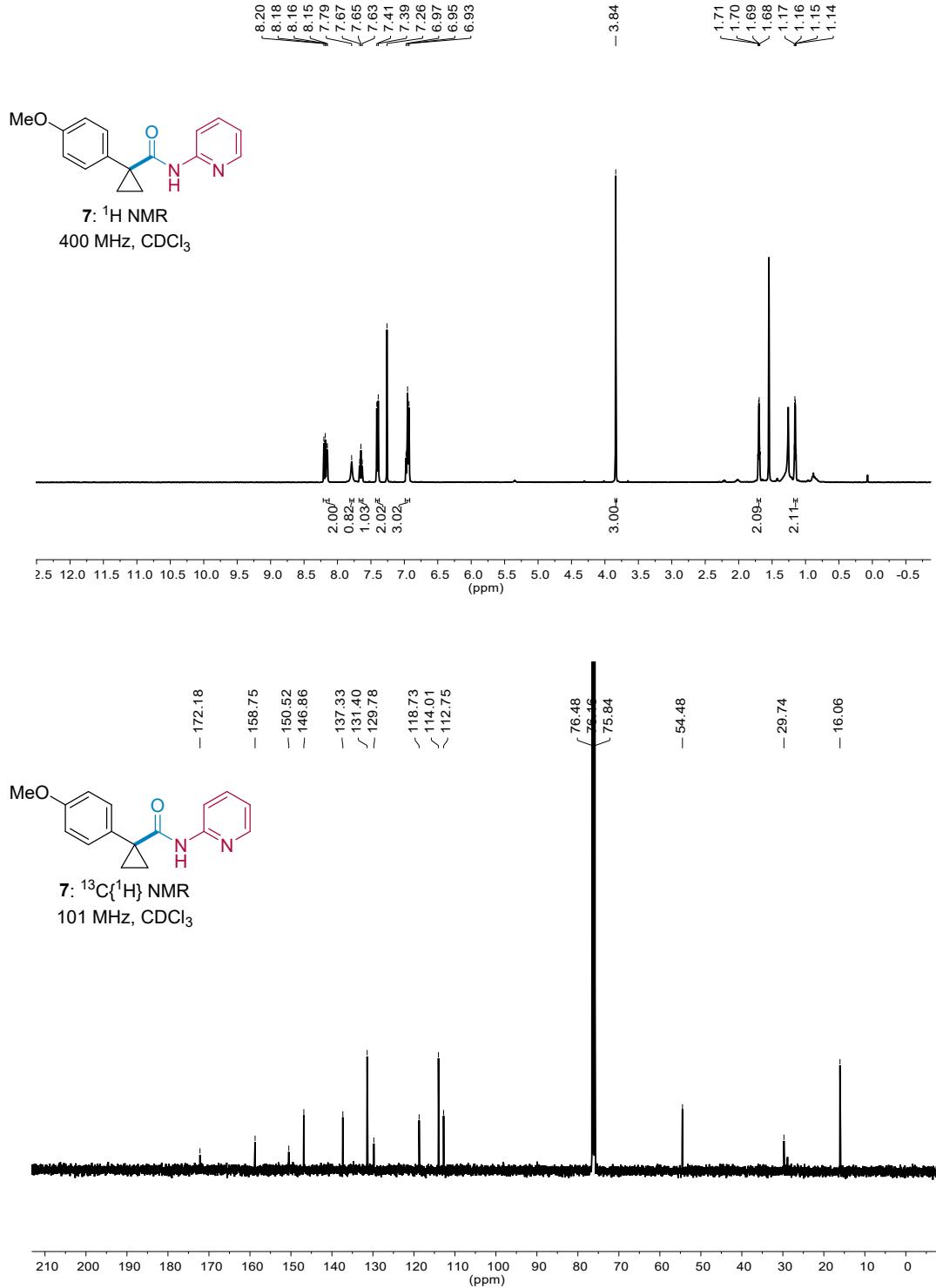












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