

Supporting information

Visible-Light Promoted Oxidative Cyclization of Cinnamic Acid Derivatives using Xanthone as the Photocatalyst

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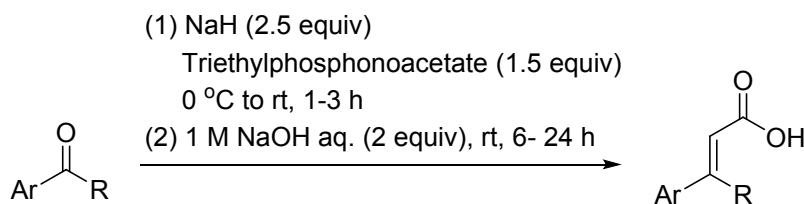
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1. General

Commercial reagents and solvents were obtained from the commercial providers and used without further purification. The products were purified using a commercial flash chromatography system or a regular glass column. TLC was developed on silica gel 60 F254 glass plates. ^1H NMR (400 MHz or 500MHz) and ^{13}C NMR (101 MHz or 126MHz) spectra were recorded on a Bruker NMR apparatus. The chemical shifts are reported in δ (ppm) values (^1H and ^{13}C NMR relative to CHCl_3 , δ 7.26 ppm for ^1H NMR and δ 77.0 ppm for ^{13}C NMR). Or alternatively, ^1H NMR chemical shifts were referenced to tetramethylsilane signal (0 ppm). Multiplicities are recorded by s (singlet), d (doublet), t (triplet), q (quartet), p (pentet), h (hextet), m (multiplet) and br (broad). Coupling constants (J), are reported in Hertz (Hz). GC analyses were performed using a Shimadzu GC-2010 ultra gas chromatography–mass spectrometry instrument equipped with a Shimadzu AOC-20s autosampler.

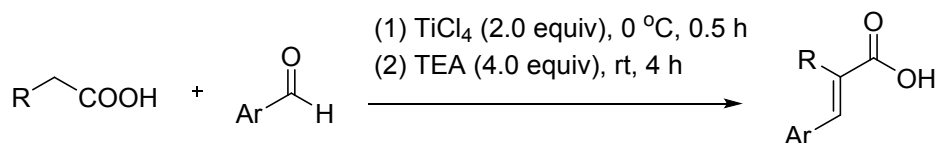
2. Preparation of starting materials - cinnamic acid derivatives



General procedure A.^[1] To a mixture solution of NaH (2.5 equiv, 60% dispersion in mineral) in dry THF, triethylphosphonoacetate (1.5 equiv) was added dropwise before cooled the reaction mixture in an ice bath. After 30 min, the corresponding aldehyde or ketone (1.0 equiv) was added to the reaction mixture before agitated for 1-3 hours at room temperature. After the reaction was completed, the reaction mixture was quenched with AcOH and water. THF was evaporated under reduced pressure, and the residue was extracted with dichloromethane. The organic layer was dried by solvent-switch with ethyl acetate. The crude product was purified by the column chromatography on silica gel using petroleum ether and ethyl acetate (v/v, 10:1) as eluent yielding compound **1**.

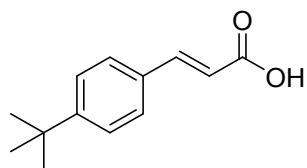
Dissolve compound **1** in ethanol and 1M NaOH aq. (2.0 equiv), stir the solution for 6 to 24 h at room temperature. After the reaction was completed, adjust the pH of the reaction mixture with

2M HCl aq. to 1-2, then filter the mixture and wash the wet cake with water. Compound **2** was obtained after dry in vacuum at 45 to 50 degrees centigrade. If necessary, purified compound **2** with the column chromatography on silica gel using petroleum ether and ethyl acetate as eluent.

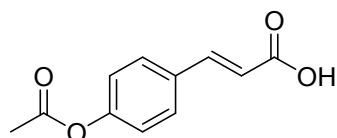


General procedure B.^[2] To a stirred solution of benzaldehyde (1.5 equiv), the corresponding acids (1.0 equiv) in dry THF was added TiCl_4 (2.0 equiv) in an ice bath. The mixture was vigorously stirred for 0.5 h, followed by adding dropwise Et_3N (4.0 equiv). After being kept for an additional 4 h at room temperature, the reaction was quenched by adding water dropwise in an ice bath, and the obtained mixture was extracted with CH_2Cl_2 . The organic layer was concentrated to dryness under reduced pressure. The obtained residue was purified by flash chromatography using petroleum ether and ethyl acetate as eluent (v/v, 5:1) as the eluent to give the target product **2**.

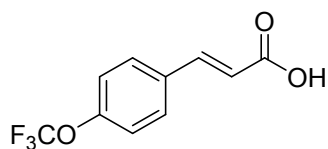
Compounds **1a**, **1b**, **1c**, **1d**, **1e**, **1i**, **1k**, **1l**, **1m**, **1o**, **1p**, **1q**, **1s**, **1t**, **1ae**, **1an**, **1ao** are commercially available, used as obtained.



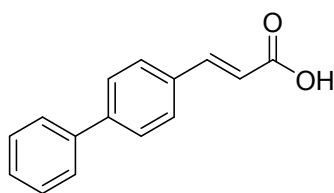
(E)-3-(4-(tert-butyl)phenyl)acrylic acid (**1f**).^[3] Prepared according to General Procedure A, white solid, 78% yield, >99% of (E) isomer. ^1H NMR (400 MHz, d_6 -DMSO): δ 12.38 (s, 1H), 7.60 (d, $J = 8.0$ Hz, 2H), 7.56 (d, $J = 16.2$ Hz, 1H), 7.42 (d, $J = 8.0$ Hz, 2H), 6.47 (d, $J = 16.0$ Hz, 1H), 1.27 (s, 9H). ^{13}C NMR (101 MHz, d_6 -DMSO): δ 167.71, 153.11, 143.83, 131.52, 128.05, 125.73, 118.30, 34.59, 30.92.



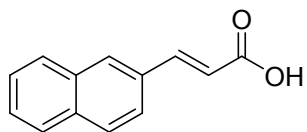
(E)-3-(4-acetoxyphenyl)acrylic acid (**1g**).^[4] Prepared according to General Procedure A, white solid, 63% yield, >99% of (E) isomer. ¹H NMR (400 MHz, *d6*-DMSO): δ 7.74 (d, *J* = 7.2 Hz, 2H), 7.59 (d, *J* = 16.2 Hz, 1H), 7.17 (d, *J* = 7.2 Hz, 2H), 6.51 (d, *J* = 16.2 Hz, 1H), 2.28 (s, 3H). ¹³C NMR (101 MHz, *d6*-DMSO): δ 169.07, 167.57, 151.82, 142.99, 131.93, 129.46, 122.38, 119.29, 20.89.



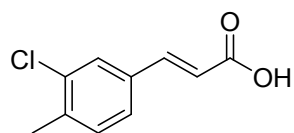
(E)-3-(4-(trifluoromethoxy) phenyl) acrylic acid (**1h**).^[5] Prepared according to General Procedure A, white solid, 79% yield, >99% of (E) isomer. ¹H NMR (600 MHz, *d6*-DMSO): δ 12.53 (s, 1H), 7.83 (d, *J* = 8.7 Hz, 2H), 7.61 (d, *J* = 16.0 Hz, 1H), 7.38 (d, *J* = 8.2 Hz, 2H), 6.57 (d, *J* = 16.1 Hz, 1H). ¹⁹F NMR (565 MHz, *d6*-DMSO): δ -56.81. ¹³C NMR (151 MHz, *d6*-DMSO): δ 167.38, 149.31, 142.16, 133.61, 130.15, 121.25, 120.57, 120.00 (q, *J* = 257.2 Hz).



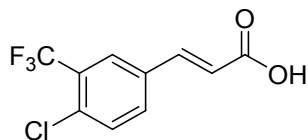
(E)-3-([1,1'-biphenyl]-4-yl)acrylic acid (**1j**).^[6] Prepared according to General Procedure A, white solid, 75% yield, >99% of (E) isomer. ¹H NMR (400 MHz, *d6*-DMSO): δ 7.79-7.70 (m, 6H), 7.64 (d, *J* = 15.9 Hz, 1H), 7.47 (t, *J* = 7.2 Hz, 2H), 7.39 (t, *J* = 7.5 Hz, 1H), 6.59 (d, *J* = 15.9 Hz, 1H). ¹³C NMR (101 MHz, *d6*-DMSO): δ 167.71, 143.31, 141.68, 139.26, 133.43, 129.05, 128.87, 127.95, 127.09, 126.71, 119.38.



(E)-3-(naphthalen-2-yl)acrylic acid (**1r**).^[7] Prepared according to General Procedure A, white solid, 73% yield, >99% of (E) isomer. ¹H NMR (600 MHz, *d*₆-DMSO): δ 12.50 (s, 1H), 8.15 (s, 1H), 7.92-7.85 (m, 4H), 7.77 (d, *J* = 15.9 Hz, 1H), 7.54 (d, *J* = 3.6 Hz, 2H), 6.68 (d, *J* = 16.0 Hz, 1H). ¹³C NMR (151 MHz, *d*₆-DMSO): δ 167.68, 143.95, 133.72, 132.92, 131.89, 129.68, 128.53, 128.47, 127.69, 127.25, 126.74, 123.95, 119.61.

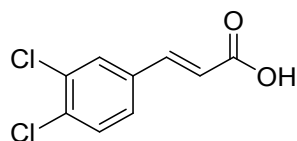


(E)-3-(3-chloro-4-methylphenyl) acrylic acid (**1u**). Prepared according to General Procedure A, white solid, 71% yield, >99% of (E) isomer. ¹H NMR (400 MHz, *d*₆-DMSO): δ 12.45 (s, 1H), 7.77 (s, 1H), 7.58-7.51 (m, 2H), 7.38 (d, *J* = 7.8 Hz, 1H), 6.56 (d, *J* = 16.1 Hz, 1H), 2.34 (s, 3H). ¹³C NMR (101 MHz, *d*₆-DMSO): δ 167.47, 142.36, 137.54, 134.03, 133.87, 131.62, 128.37, 126.72, 119.92, 19.58.

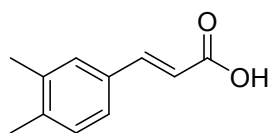


(E)-3-(4-chloro-3-(trifluoromethyl)phenyl)acrylic acid (**1v**).^[8] Prepared according to General Procedure A, white solid, 73% yield, >99% of (E) isomer. ¹H NMR (600 MHz, *d*₆-DMSO): δ

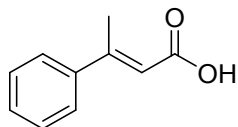
12.58 (s, 1H), 8.13 (s, 1H), 8.01 (d, $J = 8.1$ Hz, 1H), 7.72 (d, $J = 8.3$ Hz, 1H), 7.65 (d, $J = 16.1$ Hz, 1H), 6.70 (d, $J = 16.1$ Hz, 1H). ^{19}F NMR (565 MHz, d_6 -DMSO): δ -61.40. ^{13}C NMR (151 MHz, d_6 -DMSO): δ 167.63, 141.48, 134.55, 133.39, 132.53, 132.26, 128.08 (q, $J = 5.2$ Hz), 127.66 (q, $J = 31.2$ Hz), 123.09 (q, $J = 273.7$ Hz), 122.52.



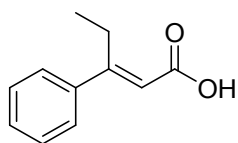
(E)-3-(3,4-dichlorophenyl)acrylic acid (**1w**).^[7] Prepared according to General Procedure A, white solid, 83% yield, >99% of (E) isomer. ^1H NMR (600 MHz, d_6 -DMSO): δ 12.54 (s, 1H), 8.01 (d, $J = 1.9$ Hz, 1H), 7.69 (dd, $J = 8.4, 1.9$ Hz, 1H), 7.64 (d, $J = 8.4$ Hz, 1H), 7.56 (d, $J = 16.0$ Hz, 1H), 6.64 (d, $J = 16.0$ Hz, 1H). ^{13}C NMR (151 MHz, d_6 -DMSO): δ 167.23, 141.22, 135.13, 132.38, 131.73, 130.92, 129.96, 128.10, 121.58.



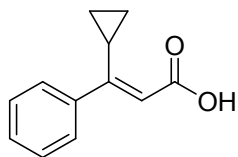
(E)-3-(3,4-dimethylphenyl)acrylic acid (**1x**).^[9] Prepared according to General Procedure A, white solid, 81% yield, >99% of (E) isomer. ^1H NMR (400 MHz, d_6 -DMSO): δ 12.33 (s, 1H), 7.51 (d, $J = 15.9$ Hz, 1H), 7.45 (s, 1H), 7.38 (d, $J = 7.6$ Hz, 1H), 7.16 (d, $J = 7.6$ Hz, 1H), 6.44 (d, $J = 15.9$ Hz, 1H), 2.22 (s, 6H). ^{13}C NMR (101 MHz, d_6 -DMSO): δ 167.78, 144.15, 139.03, 136.86, 131.83, 130.01, 129.14, 125.88, 117.87, 19.39, 19.28.



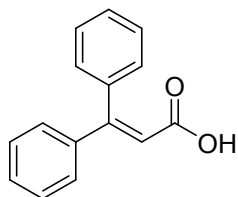
(E)-3-phenylbut-2-enoic acid (**1y**).^[1] Prepared according to General Procedure A, white solid, 66% yield, >99% of (E) isomer. ¹H NMR (400 MHz, *d6*-DMSO): δ 12.21 (s, 1H), 7.51-7.49 (m, 2H), 7.39-7.33 (m, 3H), 6.05 (s, 1H), 2.46 (s, 3H). ¹³C NMR (101 MHz, *d6*-DMSO): δ 167.60, 153.83, 141.49, 129.13, 128.66, 126.22, 117.57, 17.29.



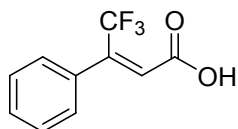
(E)-3-phenylpent-2-enoic acid (**1z**).^[1] Prepared according to General Procedure A, white solid, 63% yield, >99% of (E) isomer. ¹H NMR (400 MHz, *d6*-DMSO): δ 12.25 (s, 1H), 7.51-7.49 (m, 2H), 7.43-7.38 (m, 3H), 5.96 (s, 1H), 3.05 (q, *J* = 7.7 Hz, 2H), 0.95 (q, *J* = 7.4 Hz, 3H). ¹³C NMR (101 MHz, *d6*-DMSO): δ 167.22, 160.14, 140.28, 129.02, 128.74, 126.59, 117.30, 23.11, 13.55.



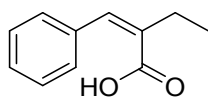
(E)-3-cyclopropyl-3-phenylacrylic acid (**1aa**).^[1] Prepared according to General Procedure A, white solid, 33% yield, >99% of (E) isomer. ¹H NMR (400 MHz, *d6*-DMSO): δ 12.22 (s, 1H), 7.33 (s, 3H), 7.16 (d, *J* = 6.9 Hz, 2H), 5.64 (s, 1H), 3.14-3.07 (m, 1H), 0.85-0.83 (m, 2H), 0.34 (m, 2H). ¹³C NMR (101 MHz, *d6*-DMSO): δ 167.54, 161.68, 138.35, 127.96, 127.87, 127.80, 119.25, 13.00, 6.51.



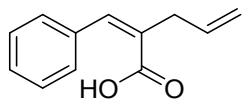
3,3-Diphenylacrylic acid (**1ac**).^[10] Prepared according to General Procedure A, white solid, 67% yield. ¹H NMR (400 MHz, CDCl₃): δ 11.52 (s, 1H), 7.37-7.20 (m, 10H), 6.31 (s, 1H). ¹³C NMR (151 MHz, CDCl₃): δ 171.15, 158.99, 140.81, 138.36, 129.70, 129.23, 128.50, 128.42, 128.37, 127.89, 116.43.



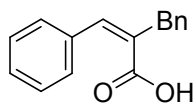
(*Z*)-4,4,4-trifluoro-3-phenylbut-2-enoic acid (**1ad**).^[11] Prepared according to General Procedure A, white solid, 72% yield, >99% of (*Z*) isomer. ¹H NMR (400 MHz, *d6*-DMSO): δ 13.14 (s, 1H), 7.45-7.43 (m, 3H), 7.32-7.30 (m, 2H), 6.84 (d, *J* = 1.0 Hz, 1H). ¹⁹F NMR (377 MHz, *d6*-DMSO): δ -65.71. ¹³C NMR (101 MHz, *d6*-DMSO): δ 165.46, 137.44 (q, *J* = 30.4 Hz), 130.81, 129.42, 128.67, 128.50, 127.65 (q, *J* = 5.4 Hz), 122.95 (q, *J* = 274.1 Hz).



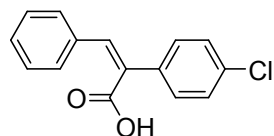
(*Z*)-2-benzylidenebutanoic acid (**1af**). Prepared according to General Procedure B, white solid, 78% yield, >99% of (*Z*) isomer. ¹H NMR (600 MHz, *d6*-DMSO): δ 12.51 (s, 1H), 7.55 (s, 1H), 7.46-7.39 (m, 4H), 7.38-7.34 (m, 1H), 2.44 (q, *J* = 7.4 Hz, 2H), 1.10 (t, *J* = 7.4 Hz, 3H). ¹³C NMR (151 MHz, *d6*-DMSO): δ 168.93, 137.34, 135.33, 134.99, 129.03, 128.63, 128.42, 20.35, 13.64.



(*Z*)-2-benzylidenepent-4-enoic acid (**1ag**). Prepared according to General Procedure B, white solid, 71% yield, >99% of (*Z*) isomer. ^1H NMR (600 MHz, d_6 -DMSO): δ 12.57 (s, 1H), 7.72 (s, 1H), 7.42 (q, $J = 7.9$ Hz, 4H), 7.36 (t, $J = 6.7$ Hz, 1H), 5.97-5.96 (m, 1H), 5.05-5.03 (m, 2H), 3.19 (d, $J = 5.4$ Hz, 2H). ^{13}C NMR (151 MHz, d_6 -DMSO): δ 168.76, 139.10, 135.79, 135.05, 130.53, 129.12, 128.71, 128.57, 115.43, 31.19.

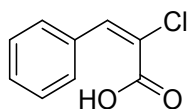


(*Z*)-2-benzyl-3-phenylacrylic acid (**1ai**). Prepared according to General Procedure B, white solid, 65% yield, >99% of (*Z*) isomer. ^1H NMR (600 MHz, CDCl_3): δ 12.3 (b, 1H), 8.05 (s, 1H), 7.40-7.39 (m, 2H), 7.37-7.34 (m, 3H), 7.30 (t, $J = 7.6$ Hz, 2H), 7.23-7.20 (m, 3H), 3.96 (s, 2H). ^{13}C NMR (101 MHz, CDCl_3): δ 173.48, 143.15, 139.06, 135.00, 129.77, 129.38, 129.15, 128.63, 128.59, 127.88, 126.20, 32.82.

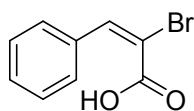


(*Z*)-2-(4-chlorophenyl)-3-phenylacrylic acid (**1aj**).^[12] Prepared according to General Procedure B, white solid, 65% yield, >99% of (*Z*) isomer. ^1H NMR (400 MHz, d_6 -DMSO): δ 12.86 (s, 1H), 7.80 (s, 1H), 7.43 (d, $J = 7.7$ Hz, 2H), 7.24-7.19 (m, 5H), 7.08 (d, $J = 6.9$ Hz, 2H). ^{13}C NMR (101

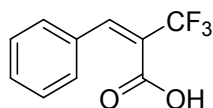
MHz, *d6*-DMSO): δ 168.03, 139.61, 135.19, 134.21, 132.36, 132.13, 131.58, 130.18, 129.20, 128.58, 128.43.



(E)-2-chloro-3-phenylacrylic acid (**1ak**).^[13] Prepared according to General Procedure B, white solid, 85% yield, >99% of (*Z*) isomer. ¹H NMR (600 MHz, *d6*-DMSO): δ 12.54 (s, 1H), 8.01 (d, *J* = 1.9 Hz, 1H), 7.72-7.60 (m, 2H), 7.56 (d, *J* = 16.0 Hz, 1H), 6.64 (d, *J* = 16.0 Hz, 1H). ¹³C NMR (151 MHz, *d6*-DMSO): δ 167.23, 141.22, 135.13, 132.38, 131.73, 130.92, 129.96, 128.10, 121.58.



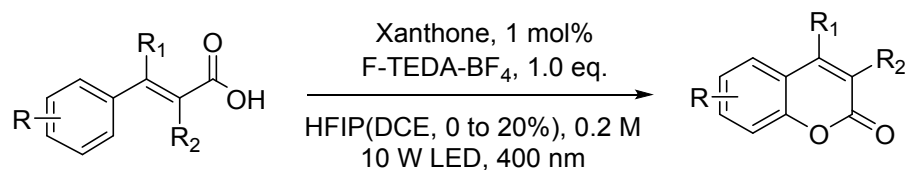
(E)-2-Bromo-3-phenylacrylic acid (**1al**).^[13] Prepared according to General Procedure B, white solid, 73% yield, >99% of (*Z*) isomer. ¹H NMR (600 MHz, *d6*-DMSO): δ 13.63 (s, 1H), 8.24 (s, 1H), 7.89 (dd, *J* = 7.3, 2.1 Hz, 2H), 7.49-7.44 (m, 3H). ¹³C NMR (151 MHz, *d6*-DMSO): δ 164.02, 140.02, 133.50, 130.19, 130.02, 128.52, 114.34.



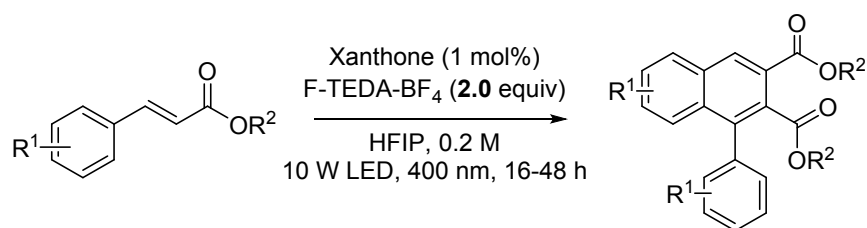
(E)-3-phenyl-2-(trifluoromethyl)acrylic acid (**1am**).^[2] Prepared according to General Procedure B, white solid, 71% yield, >99% of (*Z*) isomer.. ¹H NMR (400 MHz, *d6*-DMSO): δ 13.78 (s, 1H), 8.18 (s, 1H), 7.43 (s, 5H). ¹⁹F NMR (377 MHz, *d6*-DMSO): δ -56.99. ¹³C NMR (101 MHz, *d6*-

DMSO): δ 163.98, 148.07, 148.04, 132.61, 130.09, 129.12, 129.10, 128.39, 122.32 (q, $J = 30.8$ Hz), 122.22 (q, $J = 272.2$ Hz).

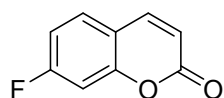
3. Oxidative cyclization of cinnamic acid derivatives



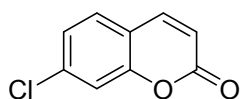
General procedure C. The specified α , β -unsaturated carbonyl compound (0.2 mmol, 1 equiv), F-TEDA-BF₄, photocatalyst Xanthone (1 mol %), and 1 mL HFIP (and DCE, 0 to 20%) were charged in the tube, then the mixture was agitated under blue LED irradiation (400 nm, 10 W) at room temperature. The reaction was monitored by GC-MS. After the reaction was completed, HFIP was removed under reduced pressure. The crude product was purified by flash chromatography using petroleum ether and ethyl acetate as eluent (v/v, 10:1).



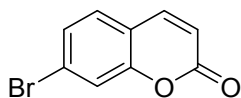
General procedure D. The specified cinnamic acid esters (0.2 mmol, 1 equiv), F-TEDA-BF₄ (2.0 equiv), photocatalyst Xanthone (1 mol %) and 1 mL HFIP were charged in the tube, then the mixture was agitated under blue LED irradiation (400 nm, 10 W) at room temperature. The reaction was monitored by GC-MS. After the reaction was completed, HFIP was removed under reduced pressure. The crude product was purified by flash chromatography using petroleum ether and ethyl acetate as eluent (v/v, 10:1).



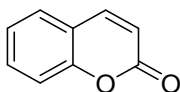
7-Fluoro-2H-chromen-2-one (**3a**).^[14] Prepared according to General Procedure C, white solid, 72% yield. ¹H NMR (600 MHz, CDCl₃): δ 7.68 (d, *J* = 9.5 Hz, 1H), 7.47 (m, 1H), 7.03-6.99 (m, 2H), 6.36 (d, *J* = 9.5 Hz, 1H). ¹⁹F NMR (377 MHz, CDCl₃): δ -105.11 (dd, *J* = 14.7, 8.5 Hz). ¹³C NMR (101 MHz, CDCl₃): δ 164.32 (d, *J* = 252.2 Hz), 160.07, 155.08 (d, *J* = 13.0 Hz), 142.81, 129.38 (d, *J* = 10.4 Hz), 115.43 (d, *J* = 2.9 Hz), 115.21 (d, *J* = 3.2 Hz), 112.40 (d, *J* = 22.6 Hz), 104.27 (d, *J* = 25.5 Hz).



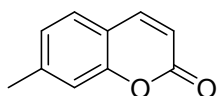
7-Chloro-2H-chromen-2-one (**3b**).^[15] Prepared according to General Procedure C, white solid, 73% yield. ¹H NMR (400 MHz, CDCl₃): δ 7.71 (d, *J* = 9.6 Hz, 1H), 7.45 (d, *J* = 7.2 Hz, 1H), 7.32-7.26 (m, 2H), 6.43 (d, *J* = 9.6 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃): δ 159.82, 154.22, 142.59, 137.67, 128.60, 124.93, 117.32, 117.07, 116.49.



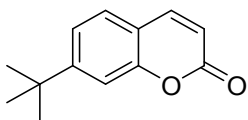
7-Bromo-2H-chromen-2-one (**3c**).^[1] Prepared according to General Procedure C, white solid, 71% yield. ¹H NMR (400 MHz, CDCl₃): δ 7.67 (d, *J* = 9.6 Hz, 1H), 7.46 (d, *J* = 1.4 Hz, 1H), 7.40-7.32 (m, 2H), 6.42 (d, *J* = 9.6 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃): δ 159.74, 154.16, 142.66, 128.73, 127.77, 125.67, 120.03, 117.65, 116.75.



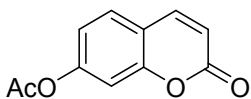
2H-chromen-2-one (**3d**).^[1] Prepared according to General Procedure C, white solid, 75% yield. ¹H NMR (400 MHz, CDCl₃): δ 7.73 (d, *J* = 9.6 Hz, 1H), 7.55-7.48 (m, 2H), 7.32-7.26 (m, 2H), 6.43 (d, *J* = 9.6 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃): δ 160.60, 153.86, 143.35, 131.68, 127.77, 124.29, 118.68, 116.65, 116.47.



7-Methyl-2H-chromen-2-one (**3e**).^[1] Prepared according to General Procedure C, white solid, 62% yield. ¹H NMR (400 MHz, CDCl₃): δ 7.67 (d, *J* = 9.6 Hz, 1H), 7.36 (d, *J* = 7.7 Hz, 1H), 7.12 (s, 1H), 7.09 (d, *J* = 7.7 Hz, 1H), 6.35 (d, *J* = 9.6 Hz, 1H), 2.44 (s, 3H). ¹³C NMR (101 MHz, CDCl₃): δ 161.04, 154.13, 143.33, 143.06, 127.48, 125.55, 117.00, 116.43, 115.40, 21.71.

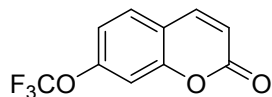


7-(Tert-butyl)-2H-chromen-2-one (**3f**).^[16] Prepared according to General Procedure C, white solid, 58% yield. ¹H NMR (600 MHz, CDCl₃): δ 7.67 (d, *J* = 9.4 Hz, 1H), 7.41 (d, *J* = 7.8 Hz, 1H), 7.33-7.31 (m, 2H), 6.36 (d, *J* = 9.4 Hz, 1H), 1.34 (s, 9H). ¹³C NMR (151 MHz, CDCl₃): δ 161.13, 156.33, 154.08, 143.14, 127.27, 121.91, 116.35, 115.68, 113.67, 31.02.

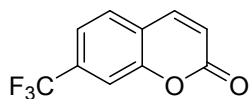


2-Oxo-2H-chromen-7-yl acetate (**3g**).^[17] Prepared according to General Procedure C, white solid, 69% yield. ¹H NMR (400 MHz, CDCl₃): δ 7.67 (d, *J* = 9.2 Hz, 1H), 7.46 (d, *J* = 7.2 Hz, 1H), 7.06

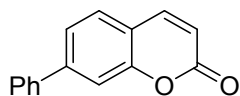
(s, 1H), 7.01 (d, $J = 7.2$ Hz, 1H), 6.34 (d, $J = 9.2$ Hz, 1H), 2.30 (s, 3H). ^{13}C NMR (151 MHz, CDCl_3): δ 168.61, 160.21, 154.46, 152.98, 142.82, 128.51, 118.29, 116.52, 115.85, 110.24, 20.98.



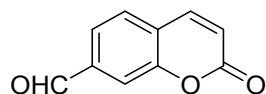
7-(Trifluoromethoxy)-2H-chromen-2-one (**3h**). Prepared according to General Procedure C, white solid, 73% yield. ^1H NMR (600 MHz, CDCl_3): δ 7.71 (d, $J = 9.4$ Hz, 1H), 7.52 (d, $J = 8.5$ Hz, 1H), 7.16 (s, 1H), 7.13 (d, $J = 8.5$ Hz, 1H), 6.41 (d, $J = 9.4$ Hz, 1H). ^{19}F NMR (565 MHz, CDCl_3): δ -57.85. ^{13}C NMR (151 MHz, CDCl_3): δ 159.77, 154.62, 151.19, 142.45, 129.12, 120.12 (q, $J = 259.1$ Hz), 117.21, 116.74, 116.62, 109.11. HRMS (EI): calculated for $\text{C}_{10}\text{H}_5\text{F}_3\text{O}_3$: 230.0191; found: 230.0190.



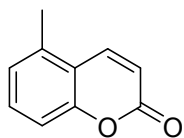
7-(Trifluoromethyl)-2H-chromen-2-one (**3i**).^[18] Prepared according to General Procedure C, white solid, 56% yield. ^1H NMR (600 MHz, CDCl_3): δ 7.76 (d, $J = 9.6$ Hz, 1H), 7.63 (d, $J = 8.1$ Hz, 1H), 7.56 (s, 1H), 7.52 (d, $J = 8.1$ Hz, 1H), 6.54 (d, $J = 9.6$ Hz, 1H). ^{19}F NMR (565 MHz, CDCl_3): δ -62.93. ^{13}C NMR (151 MHz, CDCl_3): δ 159.51, 153.62, 142.18, 133.39 (q, $J = 33.5$ Hz), 128.59, 123.09 (q, $J = 272.1$ Hz), 121.30, 120.97 (q, $J = 3.6$ Hz), 118.91, 114.25 (q, $J = 3.8$ Hz).



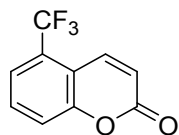
7-Phenyl-2H-chromen-2-one (**3j**).^[19] Prepared according to General Procedure C, white solid, 58% yield. ¹H NMR (400 MHz, CDCl₃): δ 7.73 (d, *J* = 9.3 Hz, 1H), 7.62 (d, *J* = 7.2 Hz, 2H), 7.54-7.40 (m, 6H), 6.43 (d, *J* = 9.3 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃): δ 160.85, 154.41, 144.98, 143.09, 139.08, 129.05, 128.50, 128.11, 127.18, 123.29, 117.73, 116.25, 114.99.



2-Oxo-2H-chromene-7-carbaldehyde (**3k**).^[20] Prepared according to General Procedure C, white solid, 44% yield. ¹H NMR (400 MHz, CDCl₃): δ 10.08 (s, 1H), 7.81-7.76 (m, 3H), 7.66 (d, *J* = 7.8 Hz, 1H), 6.57 (d, *J* = 9.6 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃): δ 190.58, 159.77, 154.16, 142.34, 138.31, 128.73, 124.51, 123.29, 119.45, 118.15.

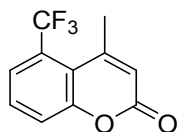


5-Methyl-2H-chromen-2-one (**3l**).^[21] Prepared according to General Procedure C, white solid, 58% yield. ¹H NMR (400 MHz, CDCl₃): δ 7.68 (d, *J* = 9.6 Hz, 1H), 7.35-7.26 (m, 2H), 7.14 (t, *J* = 7.3 Hz, 1H), 6.38 (d, *J* = 9.6 Hz, 1H), 2.41 (s, 3H). ¹³C NMR (101 MHz, CDCl₃): δ 160.84, 152.19, 143.70, 133.00, 126.07, 125.47, 123.85, 118.39, 116.06, 15.20.

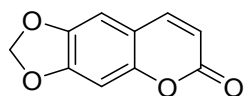


5-(Trifluoromethyl)-2H-chromen-2-one (**3m**). Prepared according to General Procedure C, white solid, 63% yield. ¹H NMR (400 MHz, CDCl₃): δ 8.01 (d, *J* = 9.7 Hz, 1H), 7.60-7.52 (m, 3H), 6.56

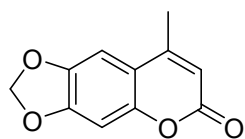
(d, $J = 9.6$ Hz, 1H). ^{19}F NMR (565 MHz, CDCl_3): δ -58.67. ^{13}C NMR (151 MHz, CDCl_3): δ 158.97, 154.78, 138.79 (d, $J = 2.2$ Hz), 131.07, 127.29 (q, $J = 33.5$ Hz), 123.25 (q, $J = 273.5$ Hz), 121.94 (q, $J = 3.8$ Hz), 121.09, 118.46, 115.77. HRMS (EI): calculated for $\text{C}_{10}\text{H}_5\text{F}_3\text{O}_2$: 214.0242; found: 214.0245.



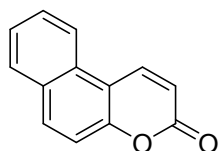
4-Methyl-5-(trifluoromethyl)-2H-chromen-2-one (**3n**). Prepared according to General Procedure C, white solid, 71% yield. ^1H NMR (600 MHz, CDCl_3): δ 7.81 (d, $J = 8.0$ Hz, 2H), 7.38 (t, $J = 7.8$ Hz, 1H), 6.36 (s, 1H), 2.47 (s, 3H). ^{19}F NMR (565 MHz, CDCl_3): δ -61.72. ^{13}C NMR (151 MHz, CDCl_3): δ 158.55, 151.64, 150.79, 128.92 (q, $J = 4.9$ Hz), 128.50, 123.47, 122.48 (q, $J = 273.3$ Hz), 120.85, 118.62 (q, $J = 31.7$ Hz), 115.87, 18.87. HRMS (EI): calculated for $\text{C}_{11}\text{H}_7\text{F}_3\text{O}_2$: 228.0398; found: 228.0397.



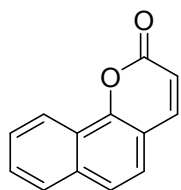
6H-[1,3]dioxolo[4,5-g]chromen-6-one (**3o**).^[22] Prepared according to General Procedure C, white solid, 46% yield. ^1H NMR (400 MHz, CDCl_3): δ 7.58 (d, $J = 9.5$ Hz, 1H), 6.83 (s, 2H), 6.28 (d, $J = 9.5$ Hz, 1H), 6.07 (s, 2H). ^{13}C NMR (101 MHz, CDCl_3): δ 161.20, 151.25, 144.88, 143.46, 113.37, 112.66, 105.00, 102.33, 98.38.



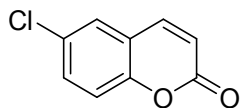
8-Methyl-6H-[1,3]dioxolo[4,5-g]chromen-6-one (**3p**).^[22] Prepared according to General Procedure C, white solid, 48% yield. ¹H NMR (600 MHz, CDCl₃): δ 6.94 (s, 1H), 6.80 (s, 1H), 6.15 (s, 1H), 6.06 (s, 2H), 2.35 (s, 3H). ¹³C NMR (151 MHz, CDCl₃): δ 161.20, 152.40, 150.91, 150.49, 144.86, 113.77, 112.16, 102.27, 102.05, 98.29, 19.10.



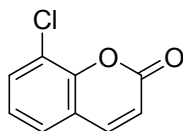
3H-benzo[f]chromen-3-one (**3q**).^[15] Prepared according to General Procedure C, white solid, 44% yield. ¹H NMR (600 MHz, CDCl₃): δ 8.49 (d, *J* = 9.4 Hz, 1H), 8.22 (d, *J* = 7.7 Hz, 1H), 7.98 (d, *J* = 7.7 Hz, 1H), 7.91 (d, *J* = 7.7 Hz, 1H), 7.69-7.57 (m, 2H), 7.46 (d, *J* = 8.0 Hz, 1H), 6.58 (d, *J* = 9.4 Hz, 1H). ¹³C NMR (151 MHz, CDCl₃): δ 160.90, 153.82, 139.09, 133.09, 130.22, 128.97, 128.95, 128.25, 126.02, 121.31, 117.03, 115.61, 112.93.



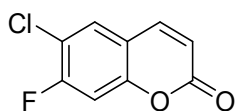
2H-benzo[g]chromen-2-one (**3r**).^[15] Prepared according to General Procedure C, white solid, 67% yield. ¹H NMR (600 MHz, CDCl₃): δ 8.51 (dt, *J* = 6.9, 2.2 Hz, 1H), 7.85 (d, *J* = 6.9 Hz, 1H), 7.80 (d, *J* = 9.2 Hz, 1H), 7.66 (d, *J* = 8.6 Hz, 1H), 7.62 (dt, *J* = 6.8, 2.6 Hz, 2H), 7.43 (d, *J* = 8.4 Hz, 1H), 6.50 (d, *J* = 9.4 Hz, 1H). ¹³C NMR (151 MHz, CDCl₃): δ 160.89, 151.22, 144.14, 134.75, 128.65, 127.73, 127.12, 124.38, 123.50, 122.97, 122.22, 115.85, 114.18.



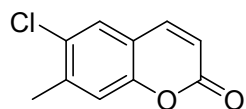
6-Chloro-2H-chromen-2-one (**3s**).^[15] Prepared according to General Procedure C, white solid, 56% yield. ¹H NMR (400 MHz, CDCl₃): δ 7.68 (d, *J* = 9.6 Hz, 1H), 7.48-7.47 (m, 2H), 7.28-7.26 (m, 1H), 6.49 (d, *J* = 9.6 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃): δ 159.89, 152.31, 142.13, 131.62, 129.55, 127.03, 119.72, 118.17, 117.72.



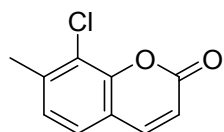
8-Chloro-2H-chromen-2-one (**3s'**).^[15] Prepared according to General Procedure C, white solid, 13% yield. **3s** / **3s'** = 1 / 0.18 in GCMS. ¹H NMR (400 MHz, CDCl₃): δ 7.70 (d, *J* = 9.6 Hz, 1H), 7.60 (d, *J* = 7.8 Hz, 1H), 7.40 (d, *J* = 7.8 Hz, 1H), 7.23 (t, *J* = 7.9 Hz, 1H), 6.47 (d, *J* = 9.6 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃): δ 159.51, 149.72, 143.08, 132.24, 126.33, 124.62, 121.77, 120.07, 117.35.



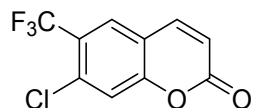
6-Chloro-7-fluoro-2H-chromen-2-one (**3t**). Prepared according to General Procedure C, white solid, 61% yield. ¹H NMR (400 MHz, CDCl₃): δ 7.63 (d, *J* = 9.6 Hz, 1H), 7.55 (d, *J* = 7.5 Hz, 1H), 7.14 (d, *J* = 8.9 Hz, 1H), 6.42 (d, *J* = 9.6 Hz, 1H). ¹⁹F NMR (377 MHz, CDCl₃) δ -106.77 (t, *J* = 8.5 Hz). ¹³C NMR (101 MHz, CDCl₃) δ 159.49, 159.47 (d, *J* = 256.7 Hz), 153.31 (d, *J* = 11.4 Hz), 141.76 (d, *J* = 1.3 Hz), 128.92 (d, *J* = 1.3 Hz), 117.68 (d, *J* = 19.1 Hz), 116.63 (d, *J* = 3.1 Hz), 116.08 (d, *J* = 3.4 Hz), 105.71 (d, *J* = 15.2 Hz). HRMS (EI): calculated for C₉H₄ClFO₂: 197.9884; found: 197.9888.



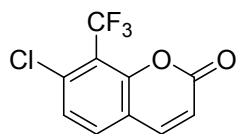
6-Chloro-7-methyl-2H-chromen-2-one (**3u**).^[18] Prepared according to General Procedure C, white solid, 47% yield. ¹H NMR (400 MHz, CDCl₃): δ 7.61 (d, *J* = 9.6 Hz, 1H), 7.45 (s, 1H), 7.19 (s, 1H), 6.39 (d, *J* = 9.6 Hz, 1H), 2.44 (s, 3H). ¹³C NMR (101 MHz, CDCl₃): δ 160.36, 152.31, 142.14, 140.69, 130.09, 127.23, 118.79, 117.75, 116.70, 20.63.



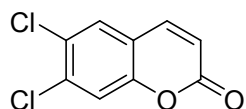
8-Chloro-7-methyl-2H-chromen-2-one (**3u'**). Prepared according to General Procedure C, white solid, 19% yield. **3u** / **3u'** = 1 / 0.44 (determined by GC-MS). ¹H NMR (400 MHz, CDCl₃): δ 7.68 (d, *J* = 9.5 Hz, 1H), 7.30 (d, *J* = 7.8 Hz, 1H), 7.18 (d, *J* = 7.8 Hz, 1H), 6.41 (d, *J* = 9.5 Hz, 1H), 2.50 (s, 3H). ¹³C NMR (101 MHz, CDCl₃): δ 159.92, 149.84, 143.19, 141.01, 126.17, 125.24, 121.60, 117.79, 115.93, 20.53. HRMS (EI): calculated for C₁₀H₇ClO₂: 194.0135; found: 194.0142.



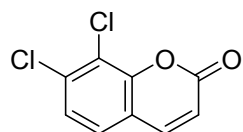
7-Chloro-6-(trifluoromethyl)-2H-chromen-2-one (**3v**). Prepared according to General Procedure C, white solid, 46% yield. ¹H NMR (600 MHz, CDCl₃): δ 7.83 (s, 1H), 7.72 (d, *J* = 9.6 Hz, 1H), 7.48 (s, 1H), 6.51 (d, *J* = 9.6 Hz, 1H). ¹⁹F NMR (565 MHz, CDCl₃): δ -62.15. ¹³C NMR (151 MHz, CDCl₃): δ 158.81, 155.75, 141.89, 135.52, 127.18 (q, *J* = 5.0 Hz), 124.91 (q, *J* = 32.3 Hz), 122.19 (q, *J* = 272.4 Hz), 119.99, 119.48, 118.01, 117.02.



7-Chloro-8-(trifluoromethyl)-2H-chromen-2-one (**3v'**). Prepared according to General Procedure C, white solid, 19% yield. **3v** / **3v'** = 1 / 0.41 in GCMS. ¹H NMR (600 MHz, CDCl₃): δ 7.71 (d, *J* = 9.6 Hz, 1H), 7.57 (d, *J* = 8.3 Hz, 1H), 7.41 (d, *J* = 8.3 Hz, 1H), 6.49 (d, *J* = 9.6 Hz, 1H). ¹⁹F NMR (565 MHz, CDCl₃): δ -55.43. ¹³C NMR (151 MHz, CDCl₃): δ 158.00, 152.64, 142.37, 136.78, 131.30, 127.44, 122.03 (q, *J* = 276.7 Hz), 118.25, 117.15, 116.92 (q, *J* = 31.6 Hz). HRMS (EI): calculated for C₁₀H₄ClF₃O₂: 247.9852; found: 247.9858.

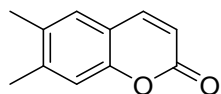


6,7-Dichloro-2H-chromen-2-one (**3w**).^[18] Prepared according to General Procedure C, white solid, 49% yield. ¹H NMR (600 MHz, CDCl₃) δ 7.62 (d, *J* = 9.6 Hz, 1H), 7.57 (s, 1H), 7.44 (s, 1H), 6.45 (d, *J* = 9.6 Hz, 1H). ¹³C NMR (151 MHz, CDCl₃): δ 159.28, 152.30, 141.54, 135.89, 128.45, 128.39, 118.80, 118.39, 117.75.

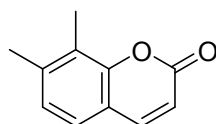


7,8-Dichloro-2H-chromen-2-one (**3w'**). Prepared according to General Procedure C, white solid, 21% yield. **3w** / **3w'** = 1 / 0.45 in GCMS. ¹H NMR (600 MHz, CDCl₃): δ 7.68 (d, *J* = 9.6 Hz, 1H), 7.39 (d, *J* = 8.4 Hz, 1H), 7.34 (d, *J* = 8.4 Hz, 1H), 6.46 (d, *J* = 9.6 Hz, 1H). ¹³C NMR (151 MHz,

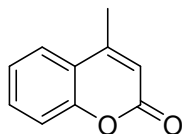
CDCl₃): δ 158.98, 150.77, 142.58, 136.71, 125.91, 125.65, 121.17, 118.28, 116.95. HRMS (EI): calculated for C₉H₄Cl₂O₂: 213.9588; found: 213.9593.



6,7-Dimethyl-2H-chromen-2-one (**3x**).^[18] Prepared according to General Procedure C, white solid, 33% yield. ¹H NMR (600 MHz, CDCl₃): δ 7.63 (d, *J* = 9.4 Hz, 1H), 7.21 (s, 1H), 7.11 (s, 1H), 6.33 (d, *J* = 9.4 Hz, 1H), 2.34 (s, 3H), 2.29 (s, 3H). ¹³C NMR (151 MHz, CDCl₃): δ 161.35, 152.45, 143.31, 141.90, 133.13, 127.87, 117.38, 116.58, 115.41, 20.31, 19.14.

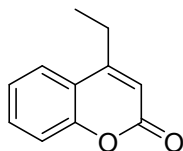


7, 8-Dimethyl-2H-chromen-2-one (**3x'**). Prepared according to General Procedure C, white solid, 18% yield. **3x** / **3x'** = 1 / 0.53 in GCMS. ¹H NMR (600 MHz, CDCl₃): δ 7.67 (d, *J* = 9.4 Hz, 1H), 7.22 (d, *J* = 7.9 Hz, 1H), 7.09 (d, *J* = 7.9 Hz, 1H), 6.35 (d, *J* = 9.4 Hz, 1H), 2.39 (s, 3H), 2.37 (s, 3H). ¹³C NMR (151 MHz, CDCl₃): δ 161.45, 152.24, 143.97, 141.67, 125.86, 124.66, 116.62, 114.95, 20.41, 11.36. HRMS (EI): calculated for C₁₁H₁₀O₂: 285.9491; found: 285.9485.

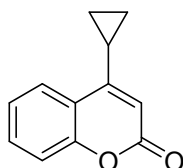


4-Methyl-2H-chromen-2-one (**3y**).^[1] Prepared according to General Procedure C, white solid, 68% yield. ¹H NMR (400 MHz, CDCl₃): δ 7.59 (d, *J* = 9.6 Hz, 1H), 7.50 (t, *J* = 7.6 Hz, 1H), 7.29 (d, *J*

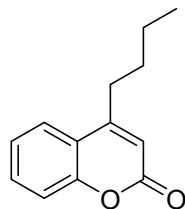
= 7.2 Hz, 2H), 6.26 (s, 1H), 2.42 (s, 3H). ¹³C NMR (101 MHz, CDCl₃): δ 160.57, 153.29, 152.27, 131.58, 124.44, 124.07, 119.76, 116.81, 114.86, 18.45.



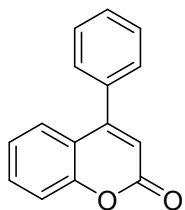
4-Ethyl-2H-chromen-2-one (**3z**).^[1] Prepared according to General Procedure C, white solid, 67% yield. ¹H NMR (400 MHz, CDCl₃): δ 7.64 (d, *J* = 9.6 Hz, 1H), 7.50 (dt, *J* = 7.7, 1.2 Hz, 1H), 7.32-7.26 (m, 2H), 6.28 (s, 1H), 2.84-2.78 (q, *J* = 7.2 Hz, 2H), 1.32 (t, *J* = 7.4 Hz, 3H). ¹³C NMR (151 MHz, CDCl₃): δ 161.02, 157.27, 153.48, 131.45, 124.07, 124.01, 119.18, 117.11, 112.88, 24.50, 11.93.



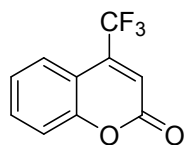
4-Cyclopropyl-2H-chromen-2-one (**3aa**).^[1] Prepared according to General Procedure C, white solid, 62% yield from **1aa** or **1aa'** in similar reaction proceed. ¹H NMR (600 MHz, CDCl₃): δ 7.92 (d, *J* = 7.8 Hz, 1H), 7.53 (t, *J* = 7.8 Hz, 1H), 7.33-7.30 (m, 2H), 6.05 (s, 1H), 2.13-2.08 (m, 1H), 1.19-1.13 (m, 2H), 0.85-0.83 (m, 2H). ¹³C NMR (151 MHz, CDCl₃): δ 161.36, 157.61, 153.35, 131.58, 124.61, 124.09, 120.14, 117.03, 110.29, 11.93, 7.99.



4-Butyl-2H-chromen-2-one (**3ab**).^[23] Prepared according to General Procedure C, white solid, 63% yield. ¹H NMR (600 MHz, CDCl₃): δ 7.61 (d, *J* = 7.7 Hz, 1H), 7.48 (t, *J* = 8.1 Hz, 1H), 7.30-7.25 (m, 2H), 6.24 (s, 1H), 2.74 (t, *J* = 7.6 Hz, 2H), 1.68-1.63 (m, 2H), 1.47-1.41 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 2H). ¹³C NMR (151 MHz, CDCl₃): δ 160.89, 156.21, 153.63, 131.46, 124.22, 124.04, 119.21, 117.16, 113.74, 31.32, 30.08, 22.43, 13.71.

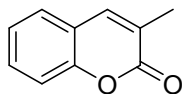


4-Phenyl-2H-chromen-2-one (**3ac**).^[22] Prepared according to General Procedure C, white solid, 58% yield. ¹H NMR (400 MHz, CDCl₃): δ 7.57-7.39 (m, 8H), 7.23 (t, *J* = 7.7 Hz, 1H), 6.37 (s, 1H). ¹³C NMR (101 MHz, CDCl₃): δ 160.69, 155.61, 154.11, 135.12, 131.87, 129.64, 128.82, 128.38, 126.95, 124.12, 118.90, 117.25, 115.11.

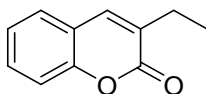


4-(Trifluoromethyl)-2H-chromen-2-one (**3ad**). Prepared according to General Procedure C, white solid, 88% yield. ¹H NMR (600 MHz, CDCl₃): δ 7.73 (d, *J* = 8.4 Hz, 1H), 7.62 (t, *J* = 7.9 Hz, 1H), 7.40 (d, *J* = 8.4 Hz, 1H), 7.36 (t, *J* = 7.9 Hz, 1H), 6.78 (s, 1H). ¹⁹F NMR (565 MHz, CDCl₃): δ -

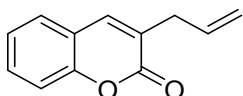
64.72 (s, 3F). ^{13}C NMR (151 MHz, CDCl_3): δ 158.76, 154.21, 141.43 (q, $J = 32.9$ Hz), 133.03, 125.26 (q, $J = 2.3$ Hz), 125.03, 121.34 (q, $J = 275.4$ Hz), 117.59, 116.01 (q, $J = 5.7$ Hz), 113.49.



3-Methyl-2H-chromen-2-one (**3ae**).^[24] Prepared according to General Procedure C, white solid, 68% yield. ^1H NMR (400 MHz, CDCl_3): δ 7.51 (s, 1H), 7.45-7.41 (m, 2H), 7.30-7.23 (m, 2H), 2.21 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ 162.07, 153.10, 139.12, 130.32, 126.85, 125.65, 124.15, 119.45, 116.27, 17.03.

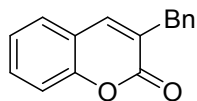


3-Ethyl-2H-chromen-2-one (**3af**).^[24] Prepared according to General Procedure C, white solid, 68% yield. ^1H NMR (600 MHz, CDCl_3): δ 7.46 (s, 1H), 7.43 (t, $J = 8.9$ Hz, 2H), 7.28 (d, $J = 8.2$ Hz, 1H), 7.23 (t, $J = 7.5$ Hz, 1H), 2.58 (q, $J = 7.4$ Hz, 2H), 1.24 (t, $J = 7.4$ Hz, 3H). ^{13}C NMR (151 MHz, CDCl_3): δ 161.74, 152.90, 137.43, 131.16, 130.36, 127.07, 124.15, 119.51, 116.27, 23.78, 12.18.

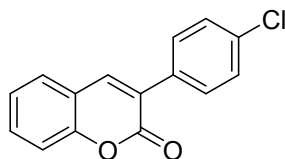


3-Allyl-2H-chromen-2-one (**3ag**).^[25] Prepared according to General Procedure C, white solid, 41% yield. ^1H NMR (400 MHz, CDCl_3): δ 7.51 (s, 1H), 7.47 (dd, $J = 15.8, 8.1$ Hz, 2H), 7.32 (d, $J = 8.2$ Hz, 1H), 7.26 (t, $J = 7.1$ Hz, 1H), 5.98 (dt, $J = 16.8, 7.0$ Hz, 1H), 5.25 (d, $J = 5.2$ Hz, 1H), 5.21 (s,

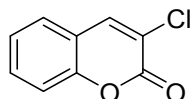
1H), 3.33 (d, $J = 6.5$ Hz, 2H). ^{13}C NMR (151 MHz, CDCl_3): δ 161.54, 153.04, 138.88, 133.72, 130.69, 127.94, 127.23, 124.26, 119.38, 118.15, 116.38, 34.48.



3-Benzyl-2H-chromen-2-one (**3ai**).^[26] Prepared according to General Procedure C, white solid, 65% yield. ^1H NMR (400 MHz, CDCl_3): δ 7.44 (t, $J = 7.5$ Hz, 1H), 7.36-7.26 (m, 8H), 7.20 (t, $J = 7.5$ Hz, 1H), 3.88 (s, 2H). ^{13}C NMR (101 MHz, CDCl_3): δ 161.61, 152.99, 139.20, 137.58, 130.72, 129.33, 128.71, 127.32, 126.78, 124.21, 119.34, 116.34, 36.49.

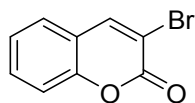


3-(4-Chlorophenyl)-2H-chromen-2-one (**3aj**).^[27] Prepared according to General Procedure C, white solid, 63% yield. ^1H NMR (600 MHz, CDCl_3): δ 7.81 (s, 1H), 7.66 (d, $J = 8.3$ Hz, 2H), 7.55-7.53 (m, 2H), 7.41 (d, $J = 8.3$ Hz, 2H), 7.36 (d, $J = 8.4$ Hz, 1H), 7.32 (d, $J = 7.5$ Hz, 1H). ^{13}C NMR (151 MHz, CDCl_3): δ 160.31, 153.46, 139.92, 134.85, 133.01, 131.64, 129.79, 128.63, 127.95, 127.04, 124.59, 119.41, 116.45.

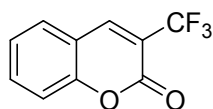


3-Chloro-2H-chromen-2-one (**3ak**).^[28] Prepared according to General Procedure C, white solid, 73% yield. ^1H NMR (600 MHz, CDCl_3): δ 7.89 (s, 1H), 7.56 (t, $J = 7.8$ Hz, 1H), 7.48 (d, $J = 7.7$

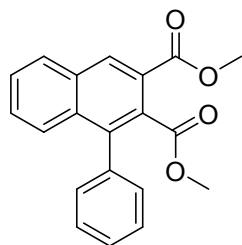
Hz, 1H), 7.34 (m, 2H). ^{13}C NMR (151 MHz, CDCl_3): δ 157.22, 152.54, 140.05, 131.84, 127.23, 125.01, 122.17, 118.69, 116.64.



3-Bromo-2H-chromen-2-one (**3al**).^[28] Prepared according to General Procedure C, white solid, 72% yield. ^1H NMR (600 MHz, CDCl_3): δ 8.08 (s, 1H), 7.54 (t, $J = 7.8$ Hz, 1H), 7.45 (d, $J = 7.5$ Hz, 1H), 7.29 (t, $J = 7.7$ Hz, 2H). ^{13}C NMR (151 MHz, CDCl_3): δ 156.96, 153.01, 144.36, 132.01, 127.09, 124.92, 119.17, 116.64, 111.65.

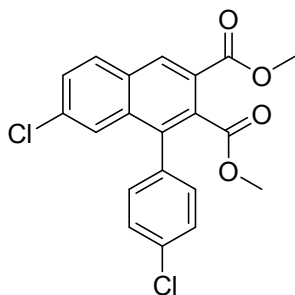


3-(Trifluoromethyl)-2H-chromen-2-one (**3am**).^[29] Prepared according to General Procedure C, white solid, 91% yield. ^1H NMR (400 MHz, CDCl_3): δ 8.17 (s, 1H), 7.69-7.62 (m, 2H), 7.39-7.35 (m, 2H). ^{19}F NMR (377 MHz, CDCl_3): δ -66.17 (s, 3F). ^{13}C NMR (101 MHz, CDCl_3): δ 155.87, 154.50, 143.43 (q, $J = 4.9$ Hz), 134.43, 129.51, 125.27, 121.30 (q, $J = 272.6$ Hz), 117.45 (q, $J = 33.2$ Hz), 116.83, 116.67.

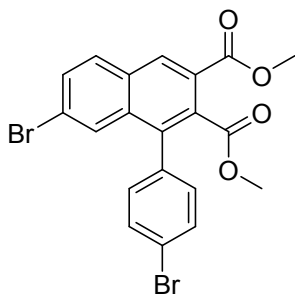


Dimethyl 1-phenylnaphthalene-2,3-dicarboxylate (**5a**).^[30] Prepared according to General Procedure D, white solid, 64% yield. ^1H NMR (400 MHz, CDCl_3): δ 8.61 (s, 1H), 7.98 (d, $J = 7.7$

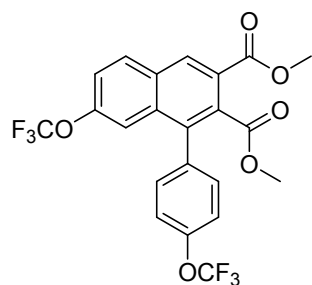
MHz), 7.58-7.56 (m, 2H), 7.52-7.45 (m, 4H), 7.37-7.35 (m, 2H), 3.96 (s, 3H), 3.60 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3): δ 169.15, 166.16, 138.58, 136.52, 133.99, 132.27, 131.35, 131.04, 130.10, 129.22, 128.90, 127.91, 127.39, 126.85, 124.32, 52.47, 51.99.



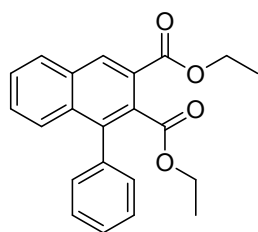
Dimethyl 7-chloro-1-(4-chlorophenyl)naphthalene-2,3-dicarboxylate (**5b**). Prepared according to General Procedure D, white solid, 55% yield. ^1H NMR (600 MHz, CDCl_3): δ 8.58 (s, 1H), 7.92 (d, $J = 8.5$ Hz, 1H), 7.52 (d, $J = 8.5$ Hz, 1H), 7.46 (s, 3H), 7.30 – 7.24 (m, 2H), 3.95 (s, 3H), 3.63 (s, 3H). ^{13}C NMR (151 MHz, CDCl_3): δ 168.69, 165.72, 136.50, 135.58, 134.56, 134.52, 134.18, 132.30, 131.48, 131.45, 130.90, 130.53, 128.68, 128.54, 125.49, 124.61, 52.71, 52.33.



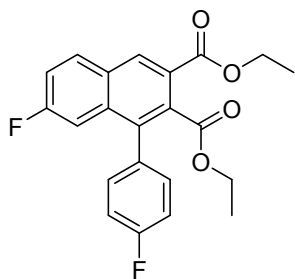
Dimethyl 7-bromo-1-(4-bromophenyl)naphthalene-2,3-dicarboxylate (**5c**). Prepared according to General Procedure D, white solid, 53% yield. ^1H NMR (600 MHz, CDCl_3): δ 8.58 (s, 1H), 7.85 (d, $J = 8.6$ Hz, 1H), 7.67 (d, $J = 8.6$ Hz, 1H), 7.66 – 7.58 (m, 3H), 7.21 (d, $J = 8.0$ Hz, 2H), 3.96 (s, 3H), 3.63 (s, 3H). ^{13}C NMR (151 MHz, CDCl_3): δ 168.66, 165.73, 136.43, 134.80, 134.64, 132.35, 132.23, 131.79, 131.57, 131.51, 131.25, 130.89, 130.75, 130.09, 128.77, 124.75, 124.15, 122.80, 52.74, 52.37.



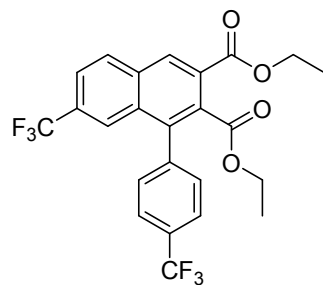
Dimethyl 7-(trifluoromethoxy)-1-(4-(trifluoromethoxy)phenyl)naphthalene-2,3-dicarboxylate (**5d**). Prepared according to General Procedure D, white solid, 44% yield. ^1H NMR (399 MHz, CDCl_3): δ 8.65 (s, 1H), 8.06 (d, $J = 9.0$ Hz, 1H), 7.51 – 7.46 (m, 1H), 7.40 – 7.33 (m, 4H), 7.30 (s, 1H), 3.97 (s, 3H), 3.61 (s, 3H). ^{19}F NMR (376 MHz, CDCl_3): δ -57.74 (s, 3F), -57.86 (s, 3F). ^{13}C NMR (100 MHz, CDCl_3): δ 168.6, 165.7, 149.4, 149.4, 137.0, 134.6, 134.3, 132.7, 131.7, 131.5, 130.6, 125.0, 121.8, 121.6, 121.5, 120.6, 119.2, 119.1, 117.0, 52.8, 52.3.



Diethyl 1-phenylnaphthalene-2,3-dicarboxylate (**5e**). Prepared according to General Procedure D, white solid, 40% yield. ^1H NMR (399 MHz, CDCl_3): δ 8.60 (s, 1H), 7.99 (d, $J = 7.9$ Hz, 1H), 7.63 – 7.42 (m, 6H), 7.36 (d, $J = 5.4$ Hz, 2H), 4.42 (q, $J = 7.0$ Hz, 2H), 4.05 (q, $J = 7.0$ Hz, 2H), 1.42 (t, $J = 7.1$ Hz, 3H), 0.98 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3): δ 168.7, 165.9, 138.5, 136.7, 134.0, 132.3, 131.3, 131.2, 130.3, 129.2, 128.8, 127.9, 127.4, 126.9, 124.9, 61.5, 61.1, 14.2, 13.6.



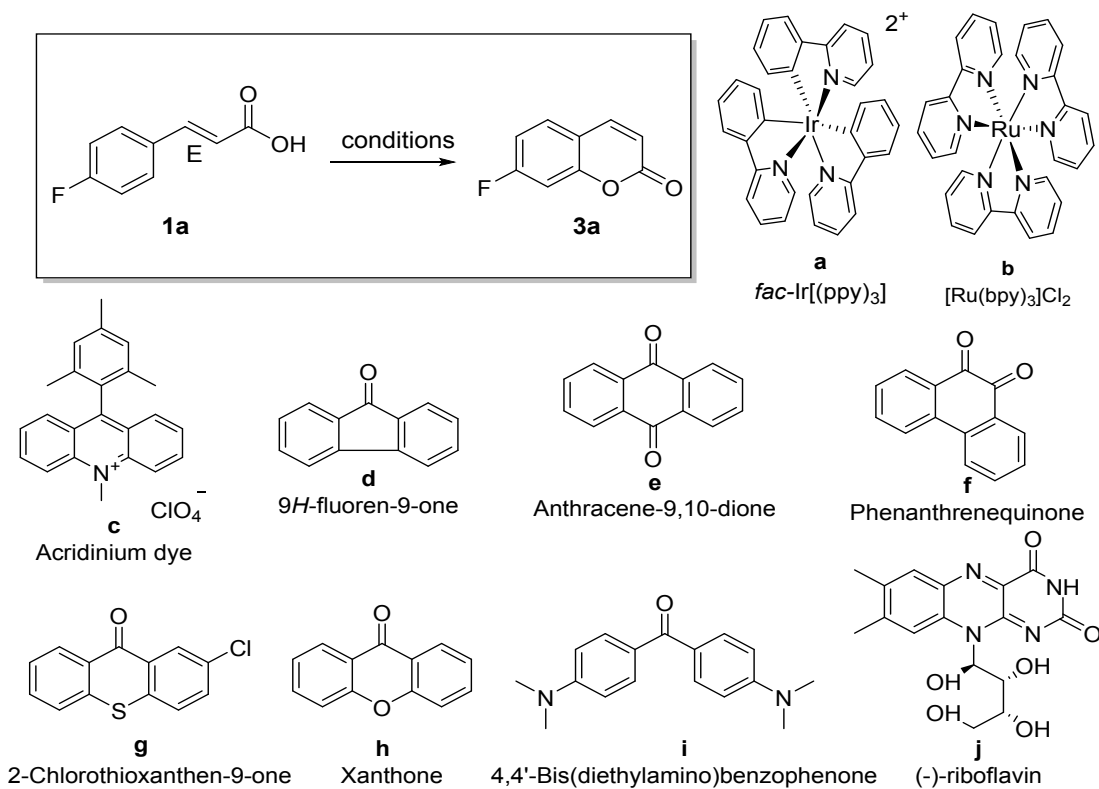
Diethyl 7-fluoro-1-(4-fluorophenyl)naphthalene-2,3-dicarboxylate (**5f**). Prepared according to General Procedure D, white solid, 56% yield. ^1H NMR (500 MHz, CDCl_3): δ 8.61 (s, 1H), 8.00 (dd, $J = 9.0, 5.7$ Hz, 1H), 7.38 – 7.30 (m, 3H), 7.20 – 7.15 (m, 2H), 7.11 (dd, $J = 10.6, 2.2$ Hz, 1H), 4.42 (q, $J = 7.1$ Hz, 2H), 4.09 (q, $J = 7.2$ Hz, 2H), 1.41 (t, $J = 7.1$ Hz, 3H), 1.03 (t, $J = 7.2$ Hz, 3H). ^{19}F NMR (376 MHz, CDCl_3): δ -108.02 – -108.12 (m, 1F), -113.40 – -113.50 (m, 1F). ^{13}C NMR (100 MHz, CDCl_3): δ 168.4, 165.5, 162.7 (d, $J = 247.5$ Hz), 162.5 (d, $J = 250.3$ Hz), 136.8, 136.7, 135.6, 135.5, 132.5, 132.1, 132.0, 131.4, 129.3, 124.3, 24.3, 118.0 (d, $J = 25.6$ Hz), 115.2 (d, $J = 21.5$ Hz), 110.4 (d, $J = 22.5$ Hz), 61.6, 61.3, 14.2, 13.7.



Diethyl 7-(trifluoromethyl)-1-(4-(trifluoromethyl)phenyl)naphthalene-2,3-dicarboxylate (**5g**). Prepared according to General Procedure D, white solid, 14% yield. ^1H NMR (500 MHz, CDCl_3): δ 8.68 (s, 1H), 8.16 (d, $J = 8.6$ Hz, 1H), 7.82 – 7.73 (m, 4H), 7.50 (d, $J = 8.0$ Hz, 2H), 4.44 (q, $J = 7.1$ Hz, 2H), 4.07 (q, $J = 7.1$ Hz, 2H), 1.43 (t, $J = 7.2$ Hz, 3H), 0.98 (t, $J = 7.1$ Hz, 3H). ^{19}F NMR (376 MHz, CDCl_3): δ -62.73 (s, 3F), -62.76 (s, 3F). ^{13}C NMR (100 MHz, CDCl_3): δ 167.8, 165.2, 139.5, 137.9, 133.6, 132.8, 132.6, 131.5, 131.4, 131.0, 130.8, 130.6, 130.4, 127.2, 125.3, 124.0, 123.9, 123.4, 77.3, 62.0, 61.5, 14.2, 13.5.

4. Additional information for optimization of conditions

Table S1. Screening solvents and photocatalysts of cyclization of **3a** with **1a**.^a



Entry	Photocatalyst	Solvent	F-TEDA-BF ₄	Yield(%) ^b
1	a , 1 mol%	HFIP, 0.2 M	1.5 equiv	20h, 2% ^c
2	b , 1 mol%	HFIP, 0.2 M	1.5 equiv	20h, 3% ^c
3	c , 1 mol%	HFIP, 0.2 M	1.5 equiv	20h, 57% ^c
4	d , 5 mol%	HFIP, 0.2 M	1.5 equiv	20h, 0% ^d
5	e , 5 mol%	HFIP, 0.2 M	1.5 equiv	20h, 30% ^c
6	f , 5 mol%	HFIP, 0.2 M	1.5 equiv	20h, 5% ^c
7	g , 5 mol%	HFIP, 0.2 M	1.5 equiv	20h, 68%
8	h , 5 mol%	HFIP, 0.2 M	1.5 equiv	20h, 70% 75% on 1 mmol scale
9	i , 5 mol%	HFIP, 0.2 M	1.5 equiv	20h, 14% ^c
10	j , 5 mol%	HFIP, 0.2 M	1.5 equiv	20h, 4% ^c
11	h , 5 mol%	HFIP/DCE(1/1), 0.2 M	1.5 equiv	40h, 65%
12	h , 5 mol%	HFIP/H ₂ O(1/1), 0.2 M	1.5 equiv	20h, 34% ^d
13	h , 5 mol%	HFIP/MeCN(1/1), 0.2 M	1.5 equiv	20h, 0% ^c

14	h , 5 mol%	MeOH/ACN(1/1), 0.2 M	1.5 equiv	20h, 0% ^e
15	h , 5 mol%	DMF, 0.2 M	1.5 equiv	20h, 0% ^d
16	h , 5 mol%	Dioxane, 0.2 M	1.5 equiv	20h, 0% ^d
17	h , 5 mol%	DCM, 0.2 M	1.5 equiv	44h, 54% ^c
18	h , 5 mol%	THF, 0.2 M	1.5 equiv	44h, 0% ^c
19	h , 5 mol%	ACN, 0.2 M	1.5 equiv	20h, 0%
20	a , 5 mol%	ACN, 0.2 M	1.5 equiv	20h, 0%
21	j , 5 mol%	ACN, 0.2 M	1.5 equiv	20h, 13%
22	h , 5 mol%	HFIP, 0.2 M	1.0 equiv	16h, 72%
23	h , 5 mol%	HFIP, 0.2 M	0.5 equiv	16h, 32%
24	h , 5 mol%	HFIP, 0.2 M	0.2 equiv	6h, 19%
				24h , 26%
25	h , 5 mol%	HFIP	-	24h, 9%
26	h , 5 mol%	HFIP, 0.1 M	1.0 equiv	16h, 69%
27	h , 1 mol%	HFIP	1.0 equiv	16h, 72%
28	h , 1 mol%	HFIP/DCE(9/1), 0.2 M	1.0 equiv	16h, 71%
29	-	HFIP	1.5 equiv	24h, 0% ^c
				Addition of 1 mol% h , 1h, 19% ^c
30	h , 1 mol%, no light	HFIP	1.5 equiv	24 h, 0% ^c
31	h , 1 mol%		1.5 equiv	24 h, 0% ^c
	520 nm LEDs	HFIP		
32	h , 1 mol%		1.5 equiv	24h, 15% ^c
	460 nm LEDs	HFIP		
33	h , 1 mol%		1.5 equiv	61%
	365 nm LEDs	HFIP		

^a Reaction conditions: **1a** (0.2 mmol, 1.0 equiv), catalyst, solvent, and F-TEDA-BF₄ as indicated, rt, 400 nm 10W LEDs, feeding operation in the glove box. ^b Yield of isolated product. ^c Conversion in GCMS. ^d Full conversion but a low isolated product. ^e All were esterification by-products.

Table S2. Screening of oxidants.^a

Entry	Solvent	Oxidant	Yield(%) ^b
1	ACN, 0.2 M	K ₂ S ₂ O ₈ , 1.5 equiv	20h, 0% ^c
2	HFIP, 0.2 M	K ₂ S ₂ O ₈ , 1.5 equiv	40 h, 40%
3	/	30% H ₂ O ₂ aq., 2.0 equiv	40 h, 15% ^c
4	HFIP, 0.2 M	F-TEDA-PF ₆	20 h, 56%
5	ACN, 0.2 M	<i>m</i> -CPBA	40 h, 0% ^c
6	HFIP, 0.2 M	O ₂	60 h, 54%
7	ACN, 0.2 M	O ₂	60 h, 0% ^c

^a Reaction conditions: **1a** (0.2 mmol, 1.0 equiv), **h** (1 mol%), solvent and oxidants as indicated, rt, 400 nm 10W LEDs. ^b Yield of isolated product. ^c Conversion in GCMS.

Table S3. E to Z Isomerization of (E)-3-(4-fluorophenyl) acrylic acid.^a

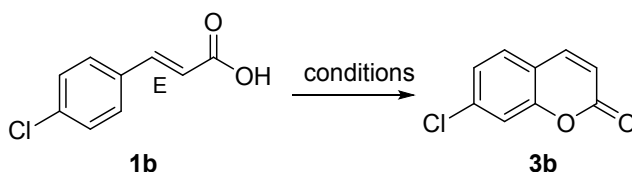
Entry	Photocatalyst	Additives	Solvent	Z/E Ratio ^b
1	a , 1 mol%	/	HFIP, 0.2 M	24:76
2	a , 1 mol%	/	ACN, 0.2 M	65:34
3	h , 5 mol%	/	HFIP, 0.2 M	2:98
4	h , 1 mol%	/	HFIP, 0.2 M	1:99
5	h , 1 mol%	/	ACN, 0.2 M	2:98
6	h , 1 mol%	/	DCM, 0.2 M	39:61
7	h , 1 mol%	/	THF, 0.2 M	10:90
8	h , 1 mol%	/	HFIP/DCE(9/1), 0.2 M	5:95
9	/	F-TEDA- BF ₄ /1.0equiv	HFIP/DCE(9/1), 0.2 M	6:94
10	j , 5 mol%	/	HFIP, 0.2 M	1:99
11	j , 5 mol%	/	ACN, 0.2 M	22:78

^a Reaction conditions: **1a** (0.2 mmol, 1.0 equiv), photocatalysts and solvents indicated, rt, 16 h, 400 nm 10W LEDs. ^b Z/E Ratio was detected by GC-MS.

Table S4. Kinetic study of cyclization of **3a** with **1a**.^a

Entry	Operations	Z/E Ratio ^b	Conversion ^c
1	Charge 2a , F-TEDA-BF ₄ (1.0 equiv), h (1 mol%) and HFIP/DCE (9/1, 0.2 M) into viral one portion	0h, 0:100	/
		1h, 0.05:1	19%
		2h, 0.22:1	40%
		4h, 0.40:1	72%
		8h, 0:0	100%

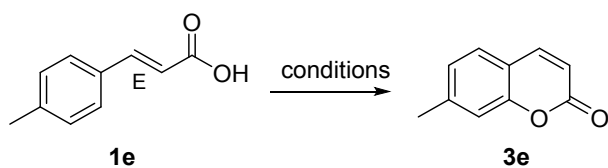
Reaction conditions: **1a** (1.0 equiv), F-TEDA-BF₄ (1.0 equiv), **h** (1 mol %), and HFIP/DCE (9/1, 0.2 M), rt, 400 nm, 10W. ^b Z/E ratio of remained **1a** was detected by GC-MS. ^c Conversion in GC-MS.

Table S5. Screen feeding sequence of cyclization of **3b** with **1b**.^a

Entry	Conditions	Yield(%) ^b
1	Addition of 1b , F-TEDA-BF ₄ (1.0 equiv), h (1 mol%) and HFIP/DCE (1/1, 0.2 M) in one portion	20 h, 73%
2	Addition of 1b , F-TEDA-BF ₄ (1.0 equiv), h (1 mol%) and HFIP (0.2 M) in one portion	20 h, 71%
3	Addition of 1b (0.2 mmol), h (5 mol%) and HFIP (0.2 M) in one portion, agitated for 10 h at 10W 400 nm UV light, then add 1.0 equiv F-TEDA-BF ₄	16 h, 74%

^a Reaction conditions as indicated. ^b Yield of isolated product.

Table S6. Verify the influence of DCE on the cyclization of **3e** with **1e**.^a



Entry	Photocatalyst	Solvent	F-TEDA- BF ₄	Yield(%) ^b	Esterification by-product
1	h , 5 mol%	HFIP, 0.2 M	1.0 equiv	24h, 100%	64% ^c
2	h , 5 mol%	HFIP, 0.1 M	1.0 equiv	24h, 100%	22% ^c
3	h , 1 mol%	HFIP/DCE (9/1) 0.2 M	1.0 equiv	24h, 100%	6% ^c
4	h , 5 mol%	HFIP/DCE (1/1) 0.2 M	1.0 equiv	40h, 60%	Trace

Reaction conditions: **1e** (1.0 equiv), F-TEDA-BF₄ (1.0 equiv), **h** (1 mol%), rt, 400 nm 10W LEDs and solvent as indicated. ^b Conversion in GC-MS. ^c Percentage of HFIP esterification by-product determined by GC-MS.

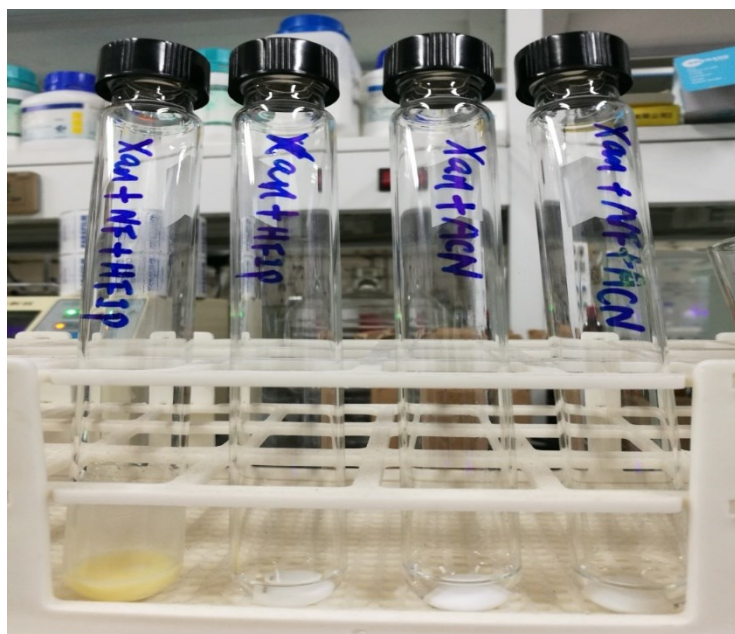


Figure S1. Color change of four vials (Xanthone/F-TEDA-BF₄/HFIP, Xanthone/HFIP, Xanthone/ACN and Xanthone/F-TEDA-BF₄/ACN subsequently).

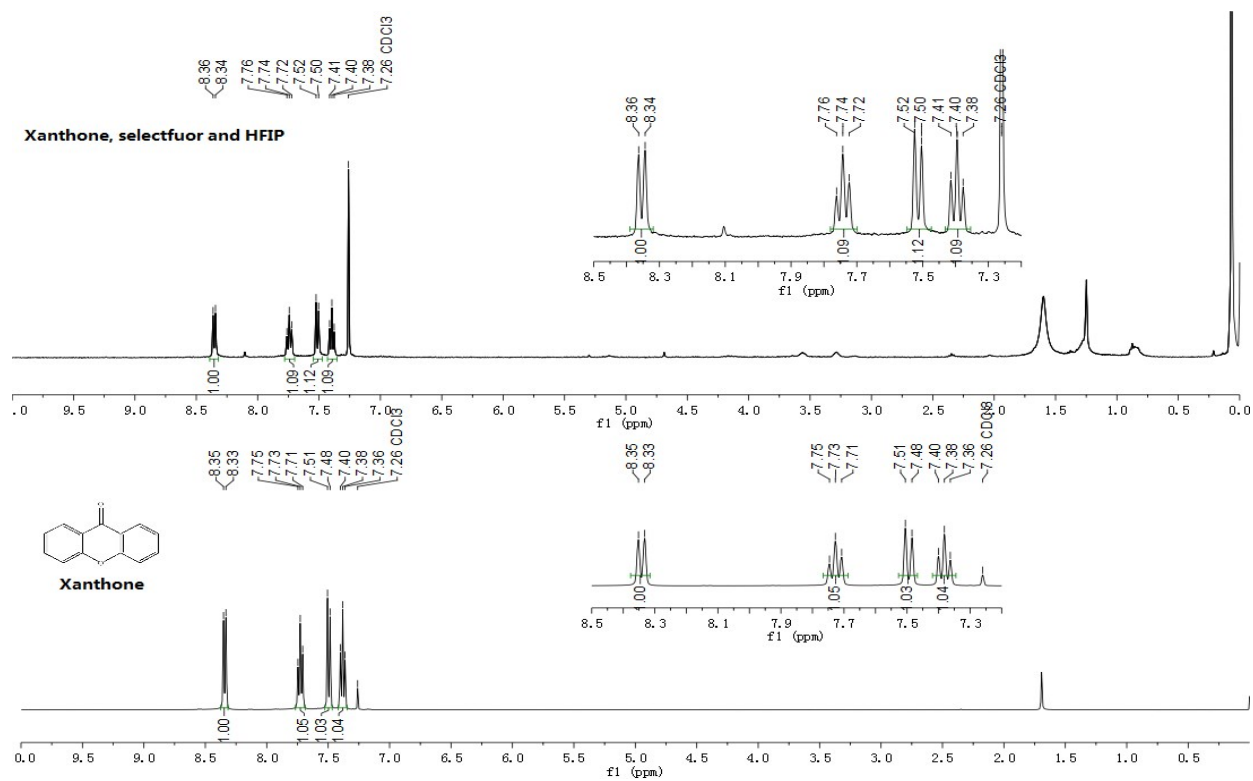
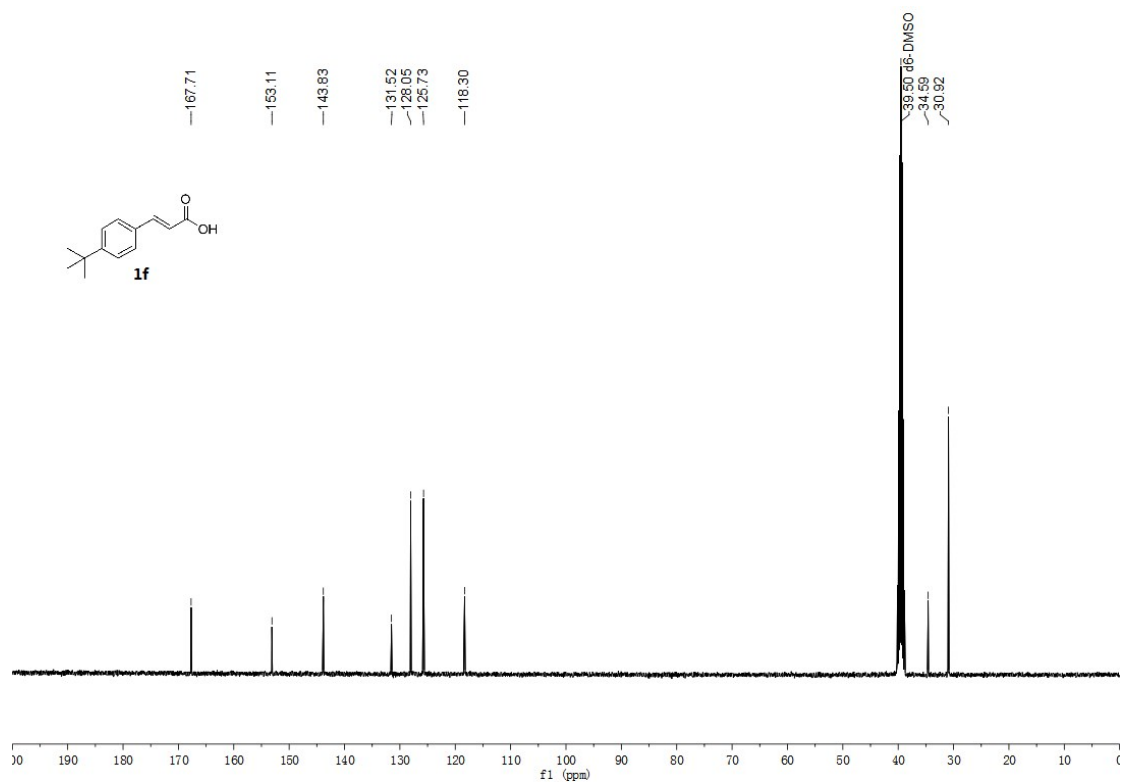
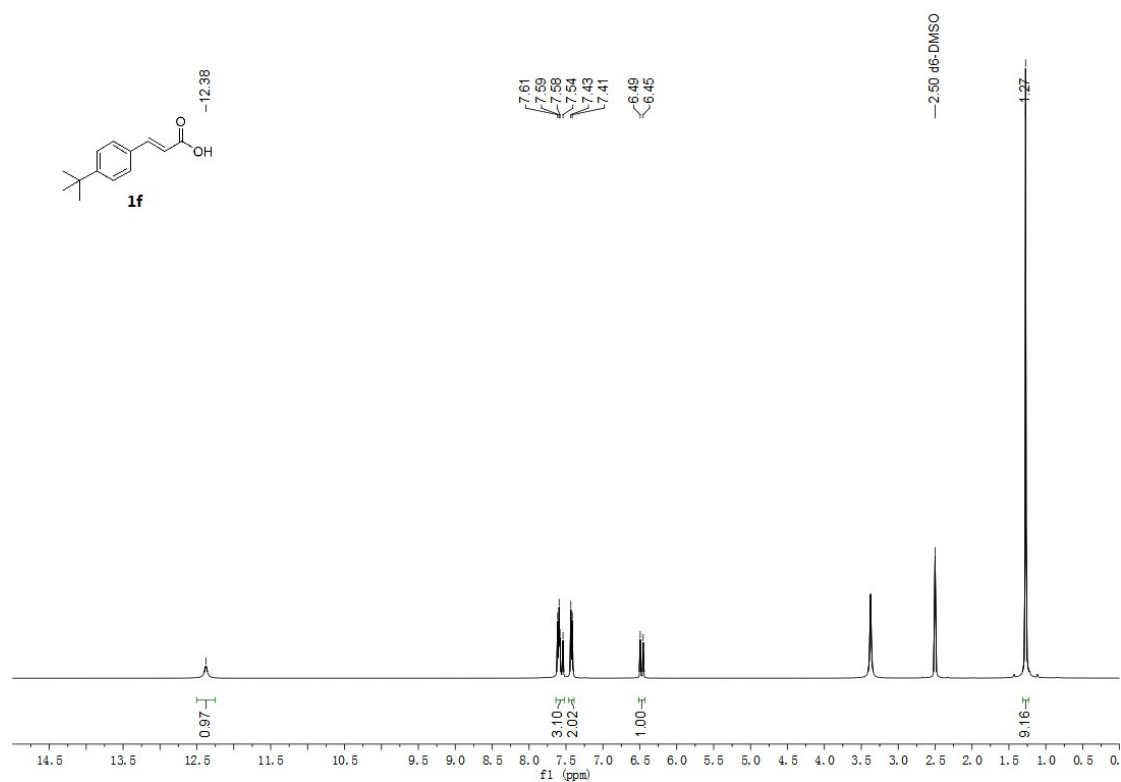
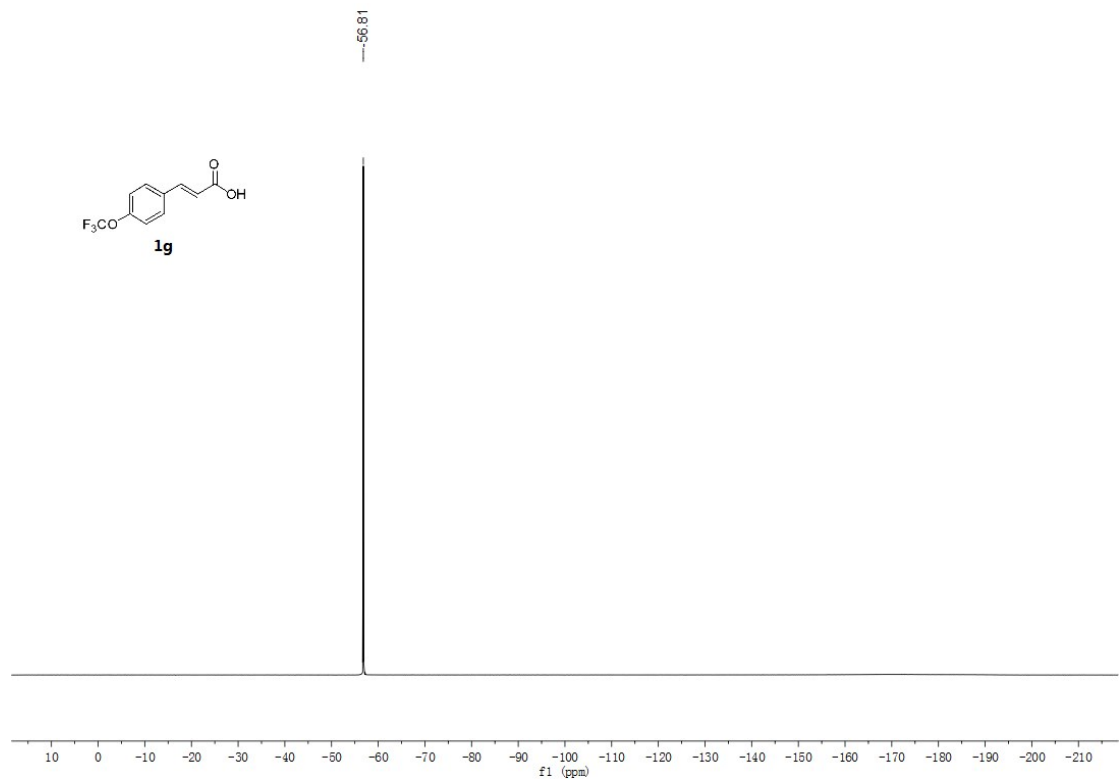
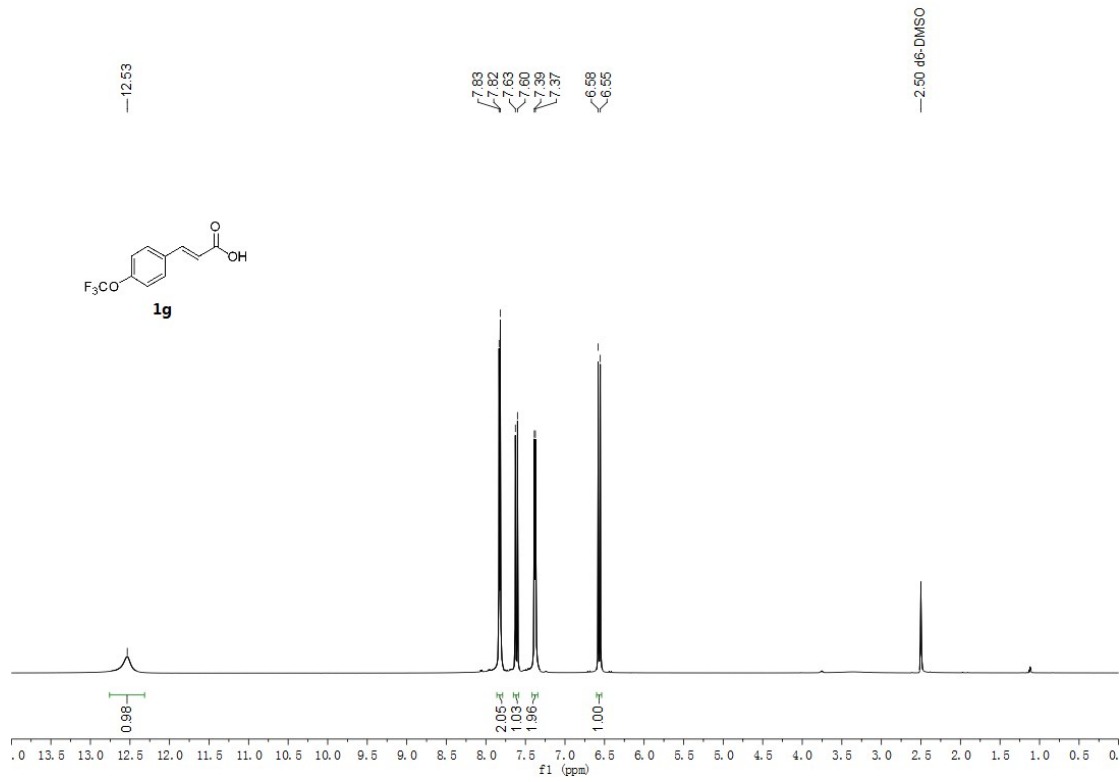
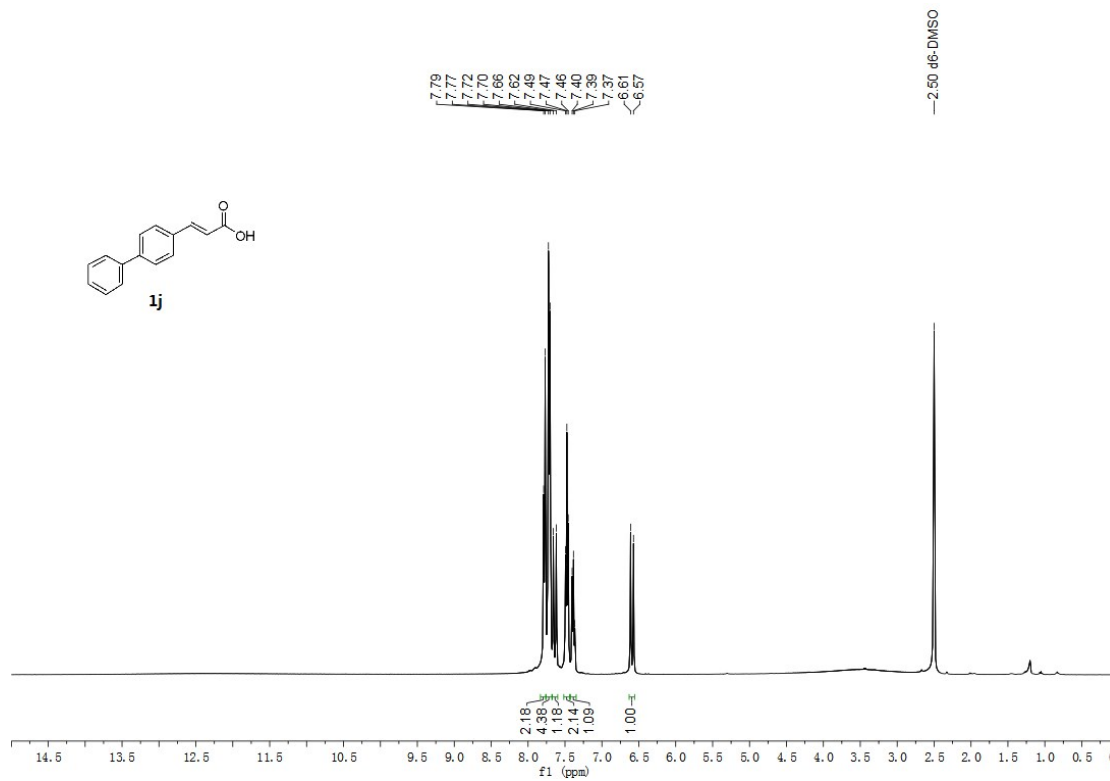
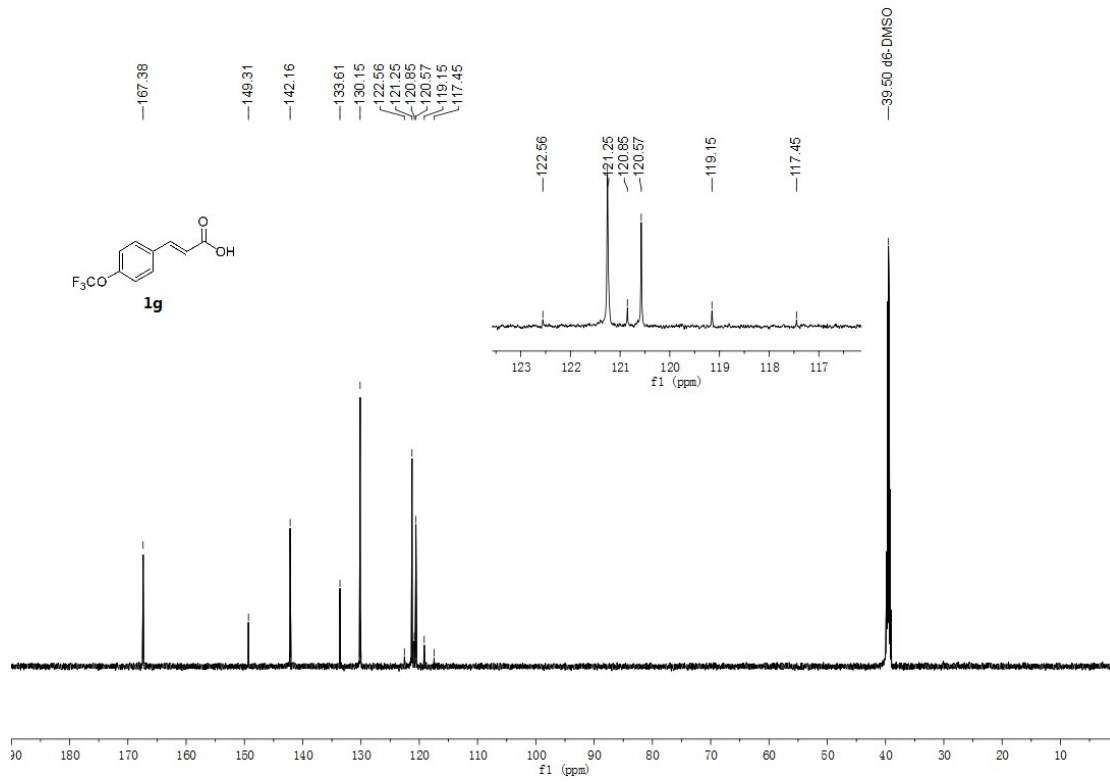


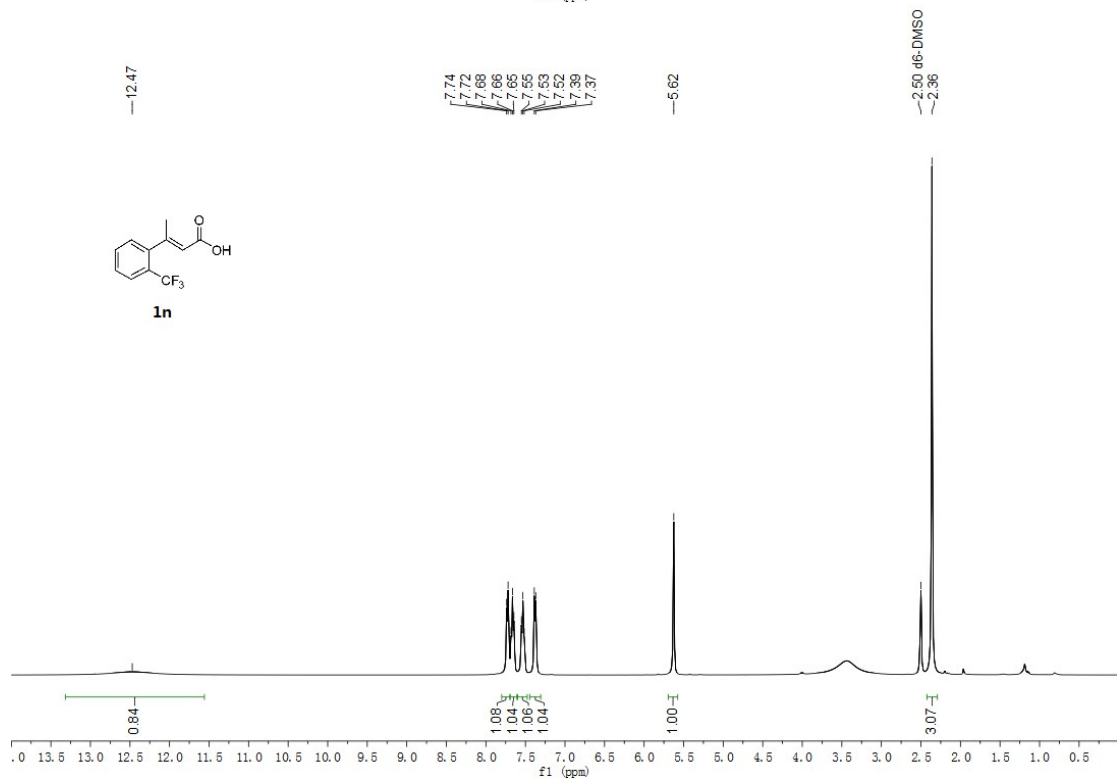
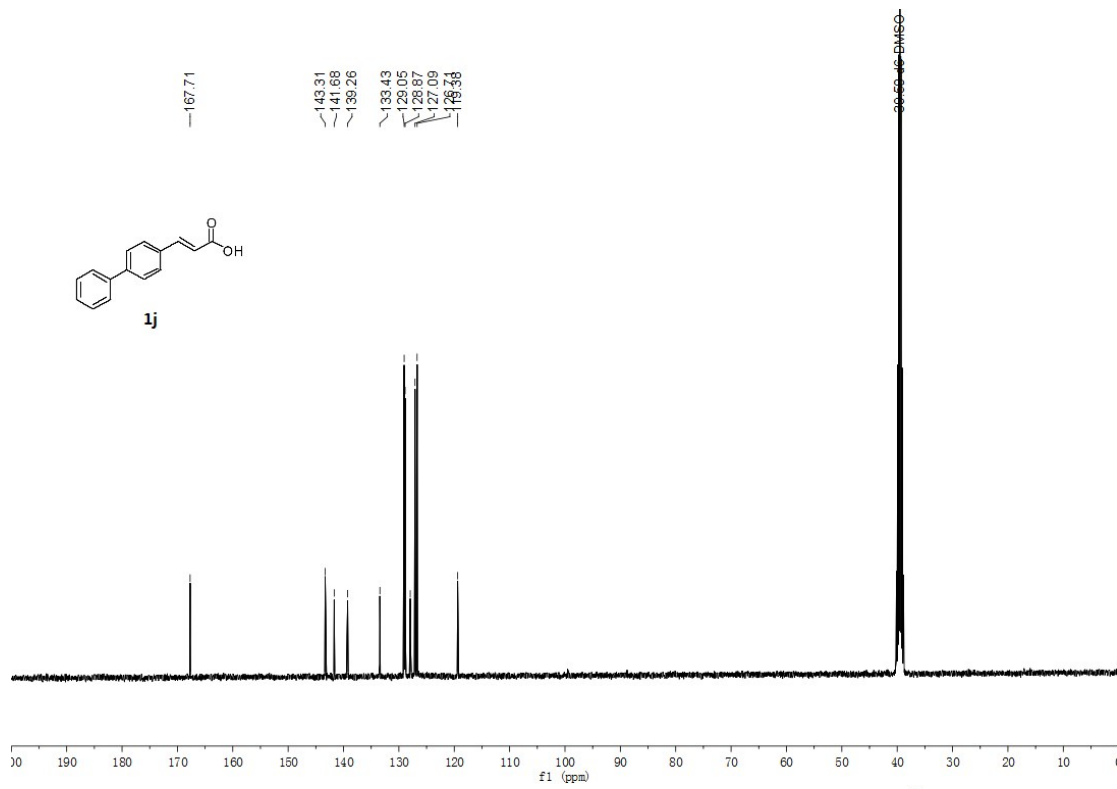
Figure S2. ¹H NMR spectrum of the crude mixture and pure xanthone.

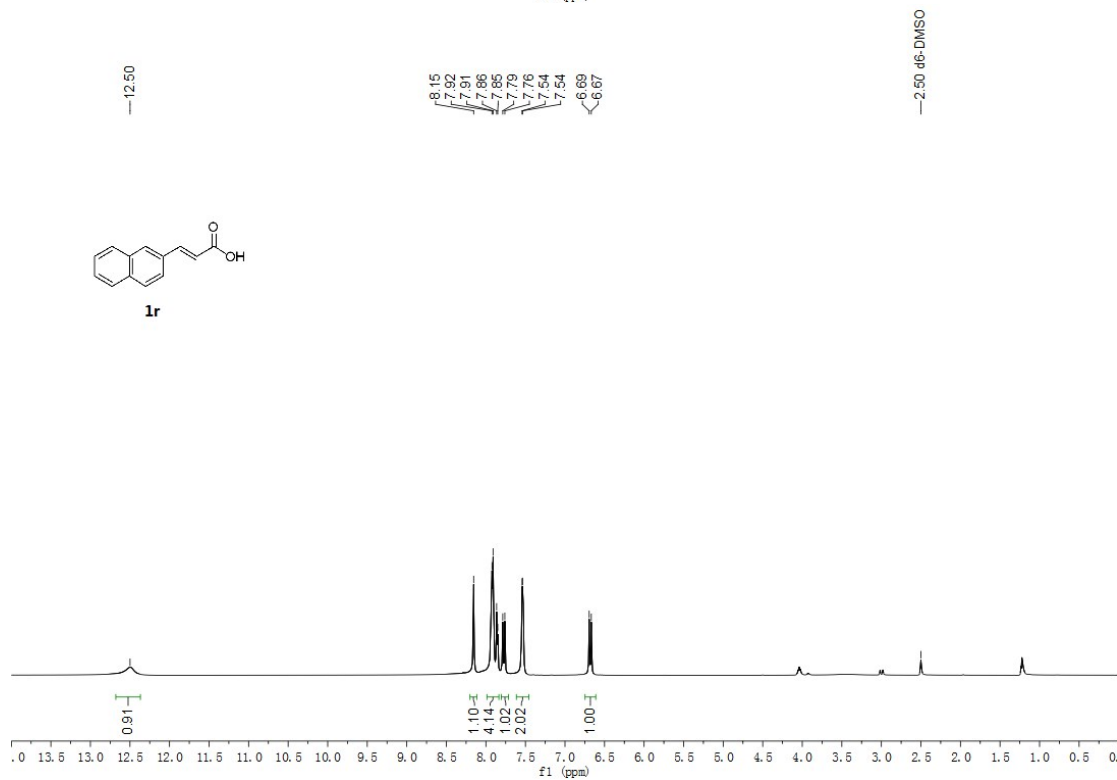
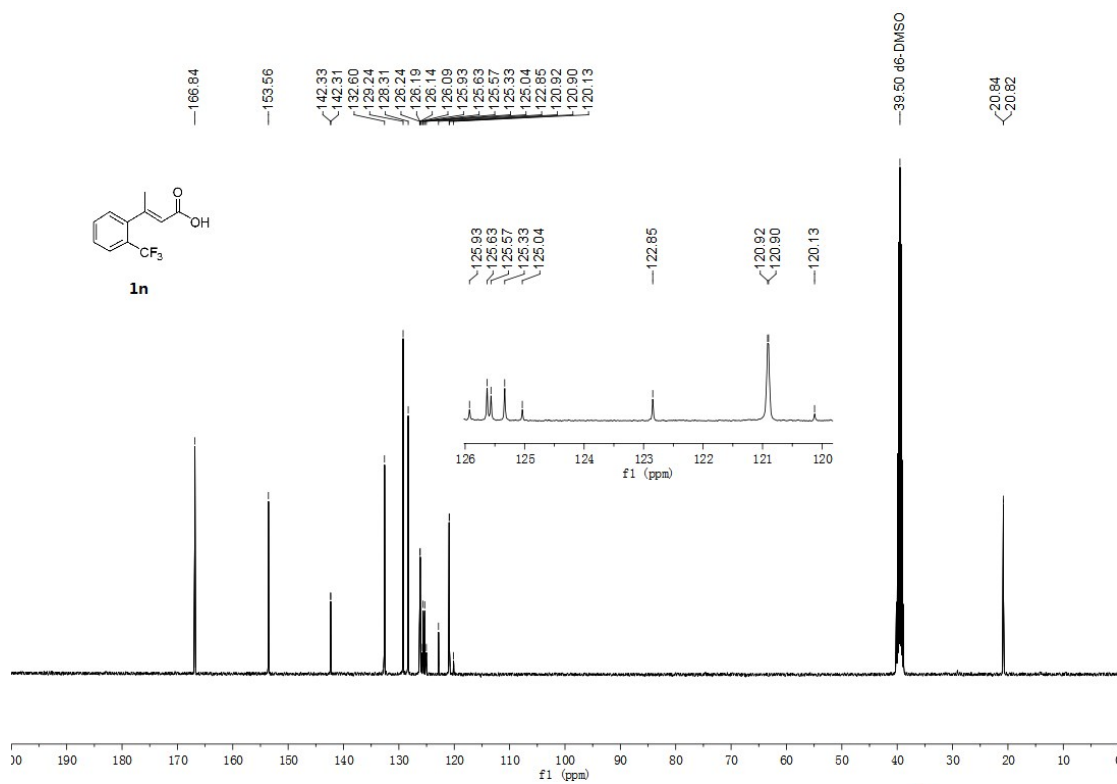
5. Copies of NMR spectra

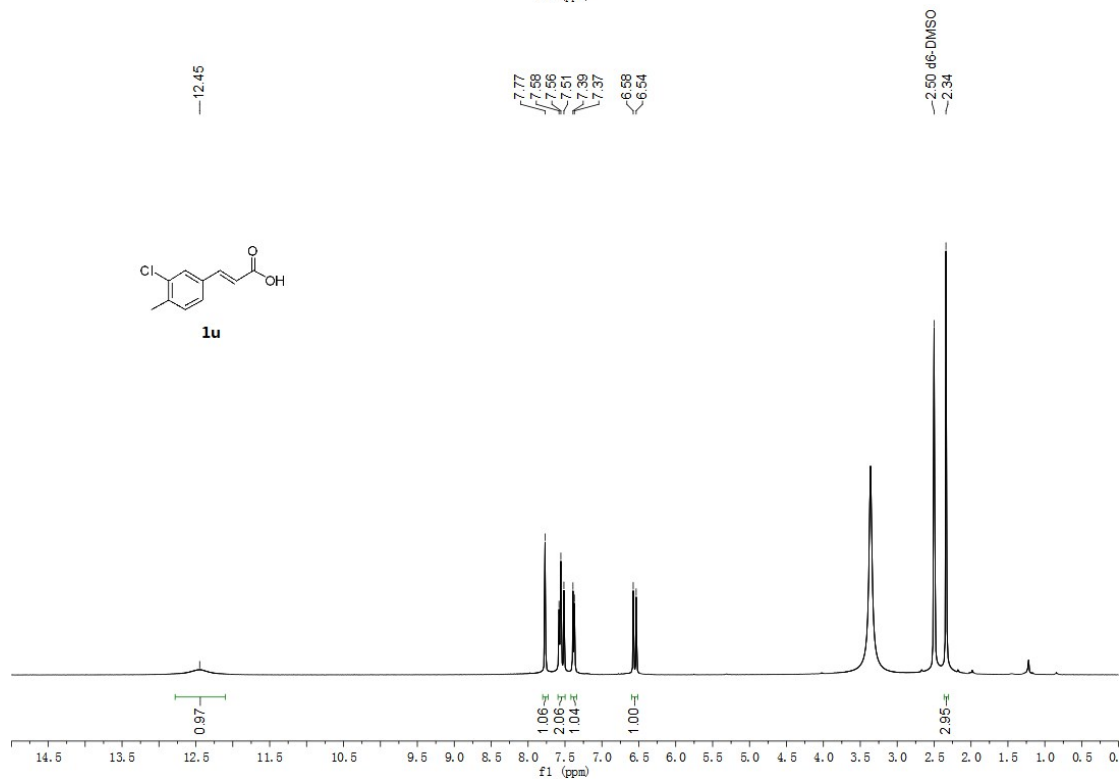
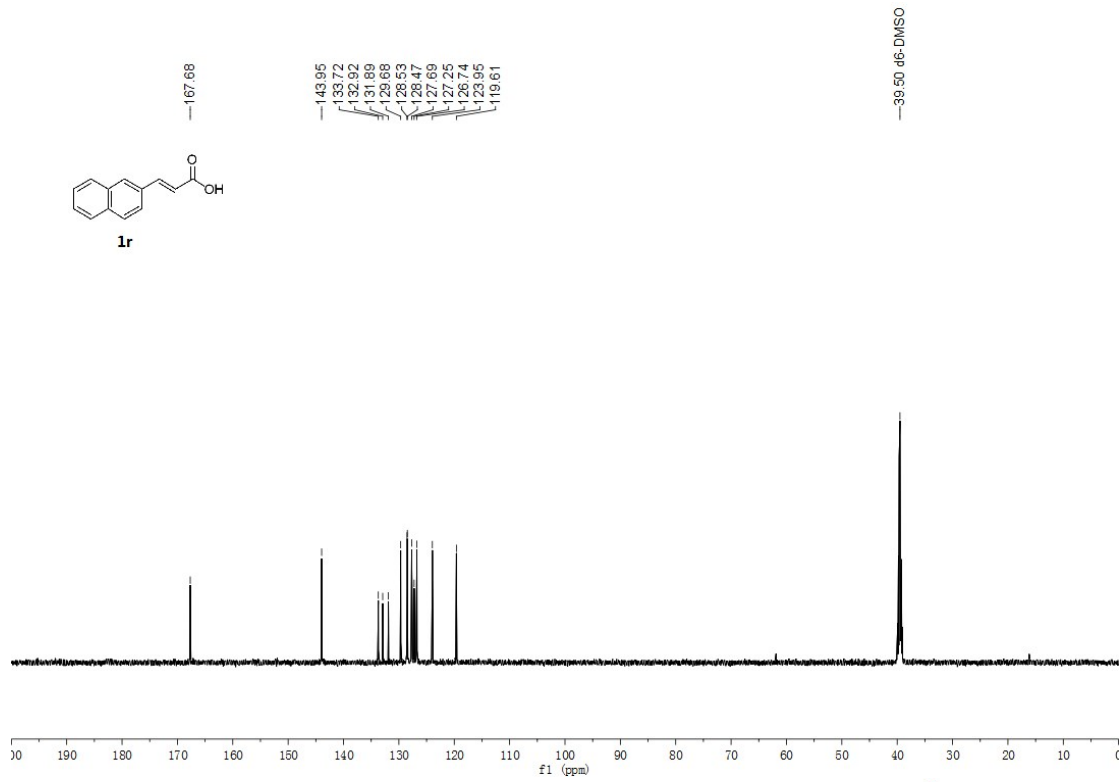


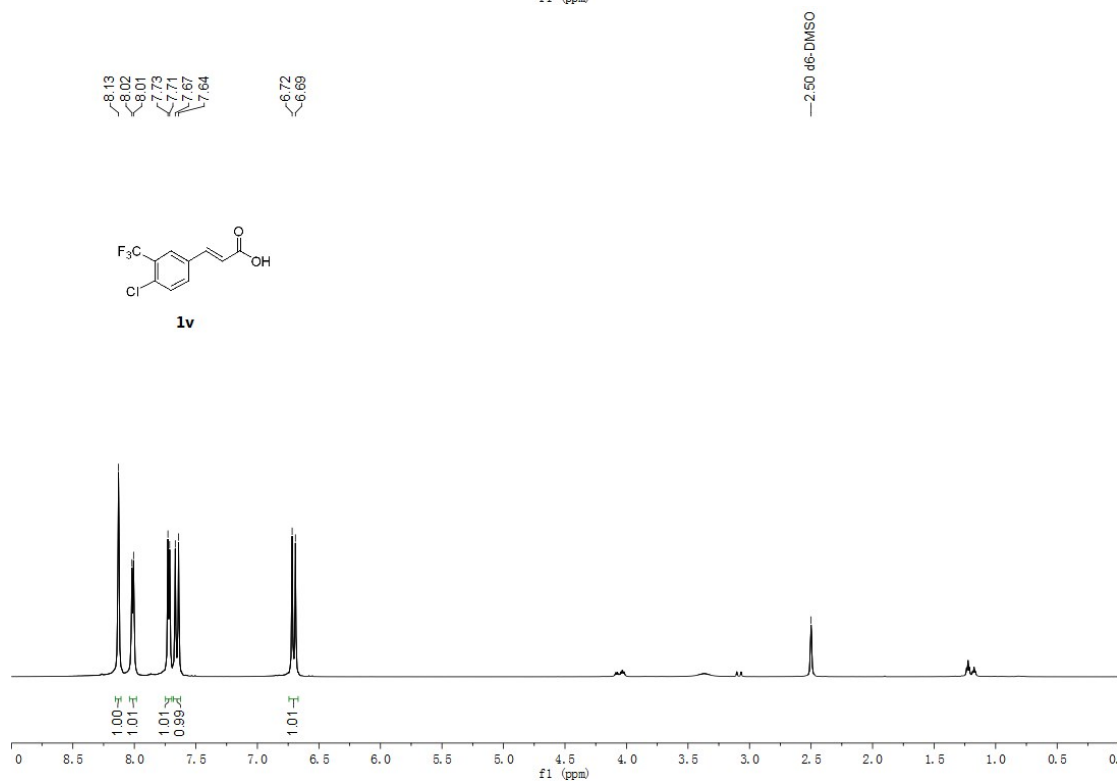
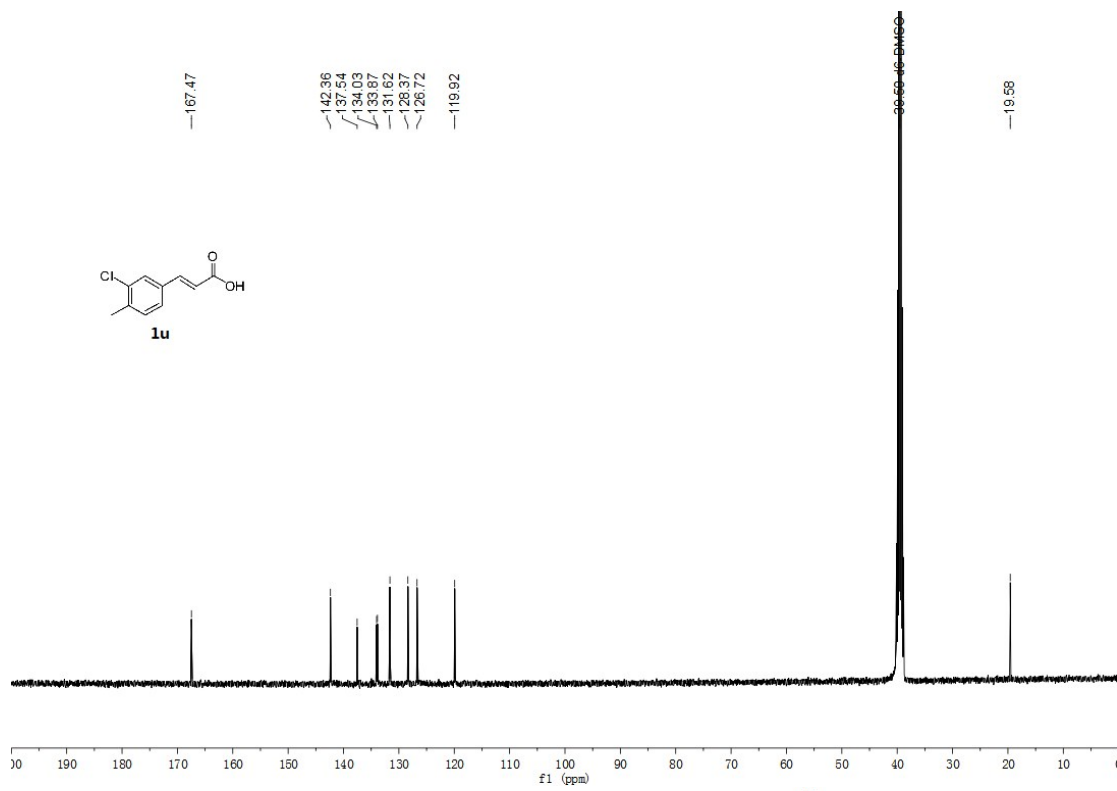


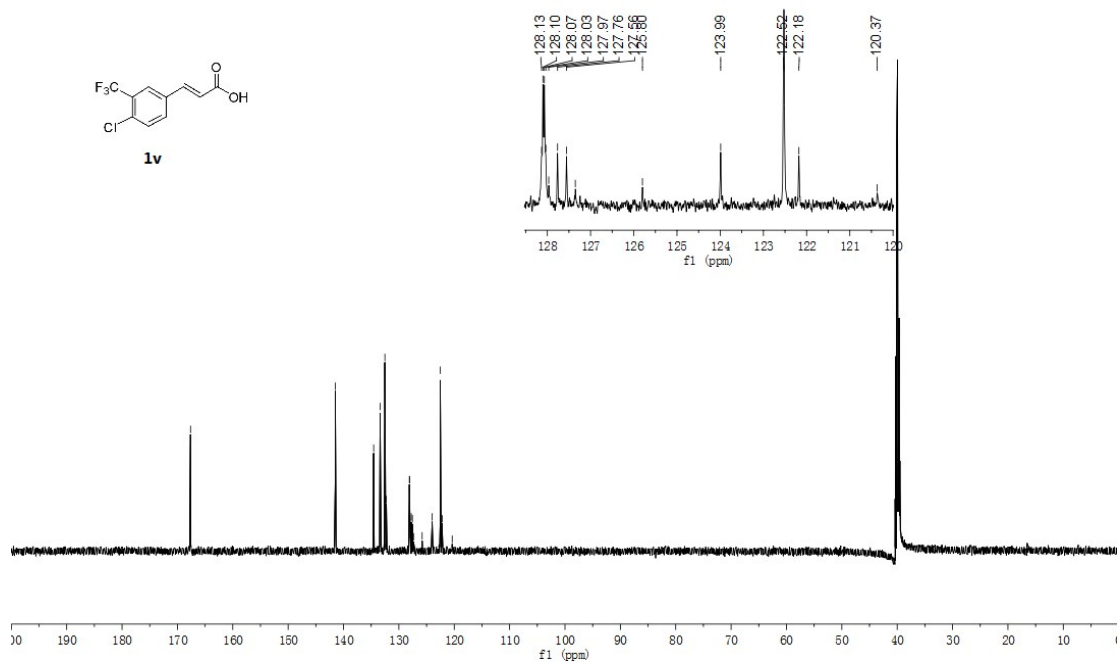
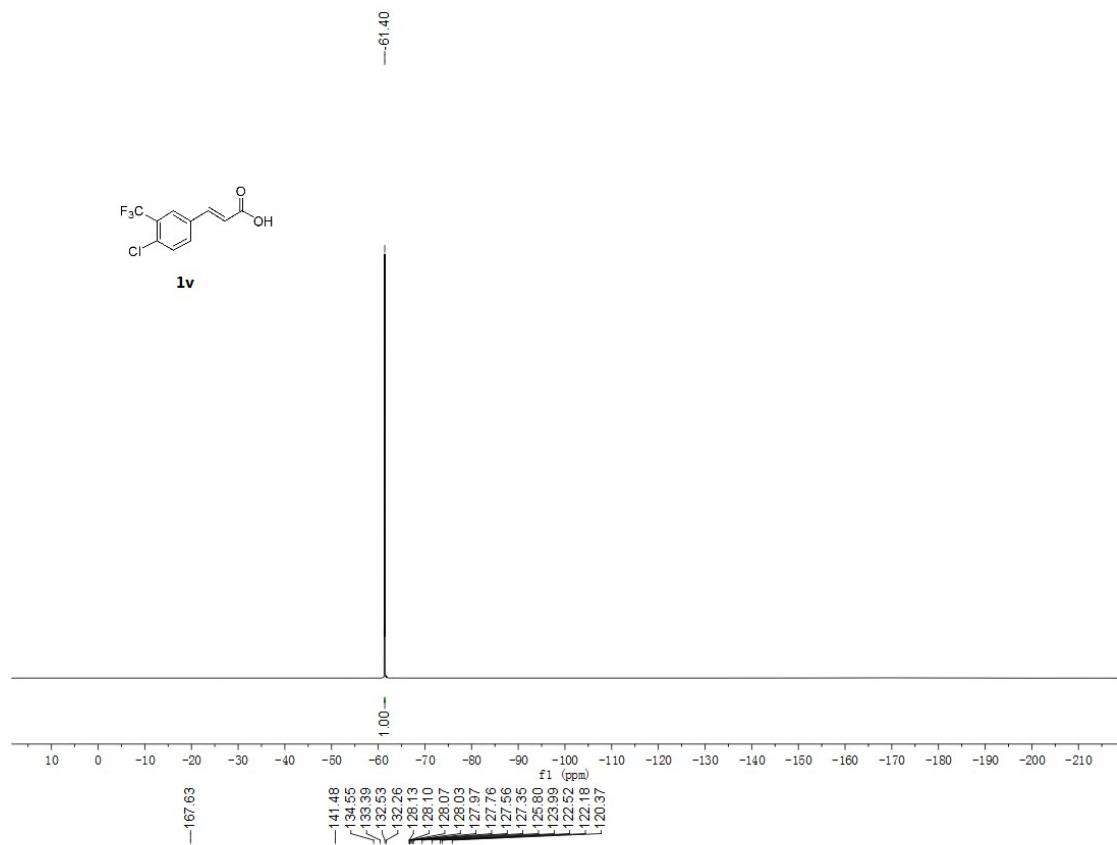


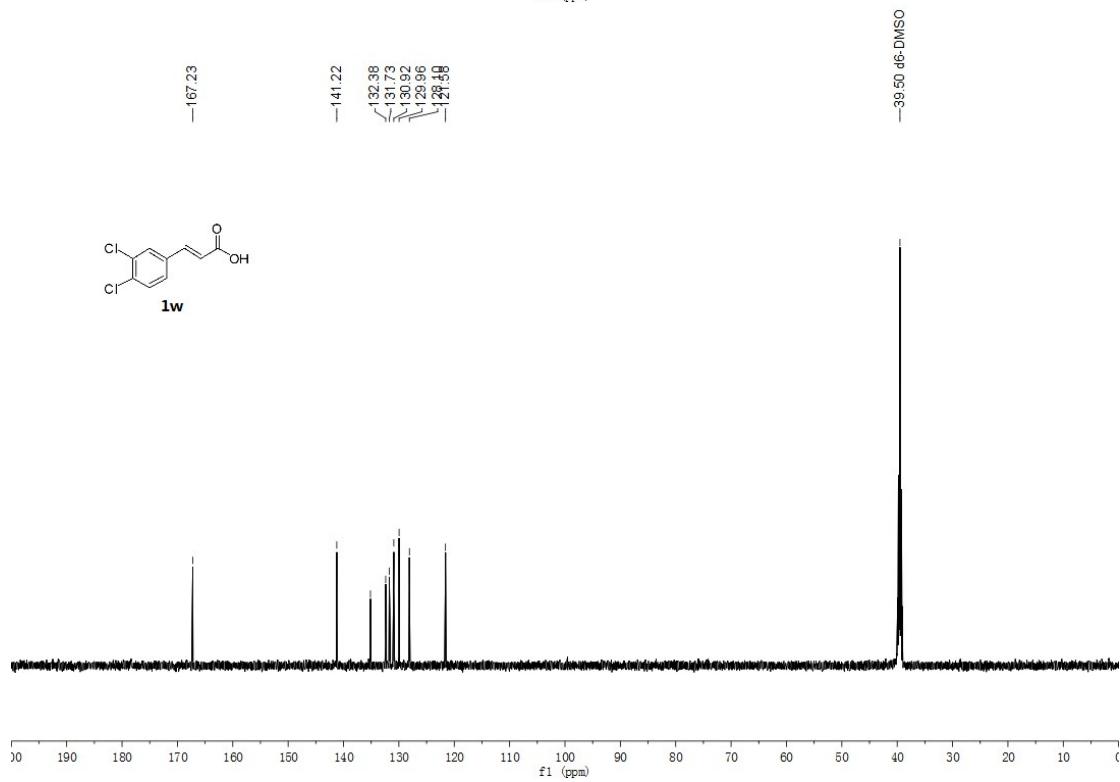
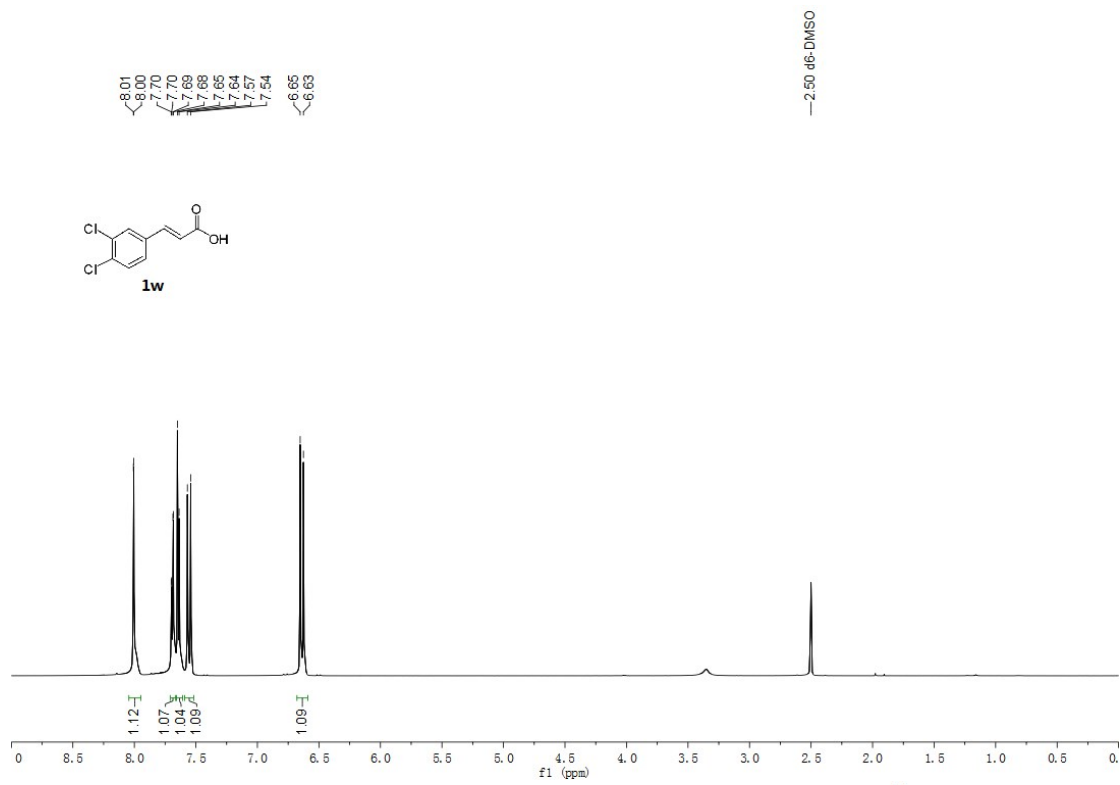


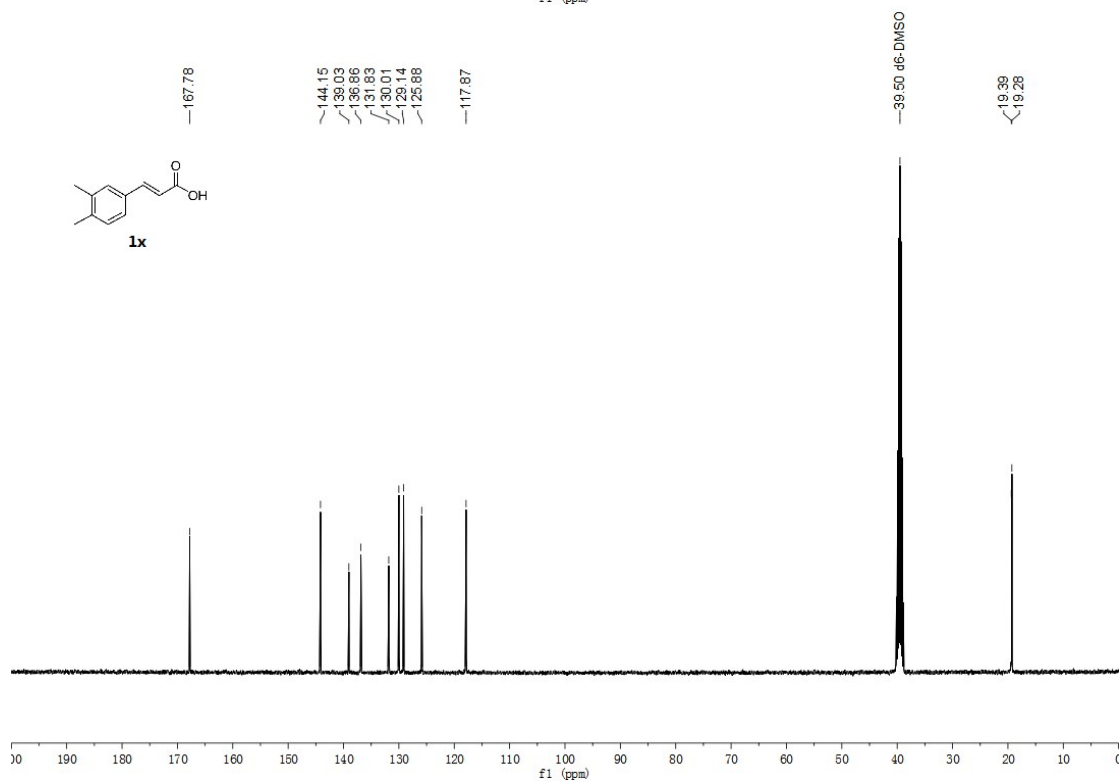
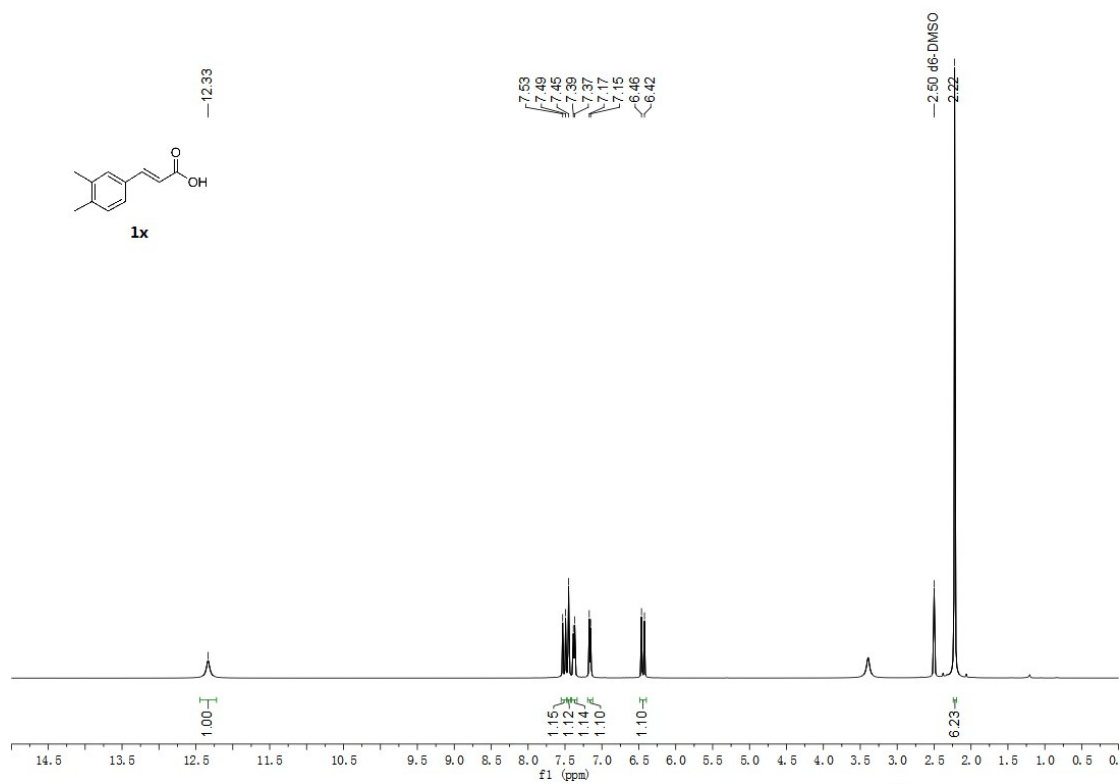


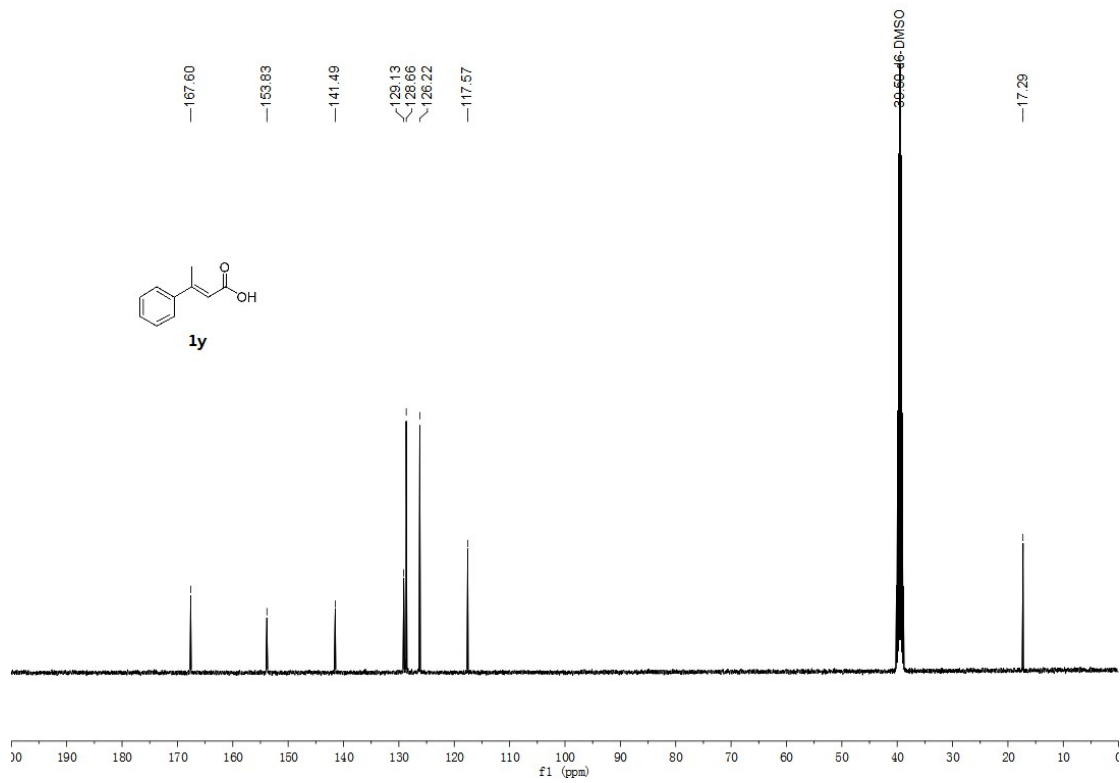
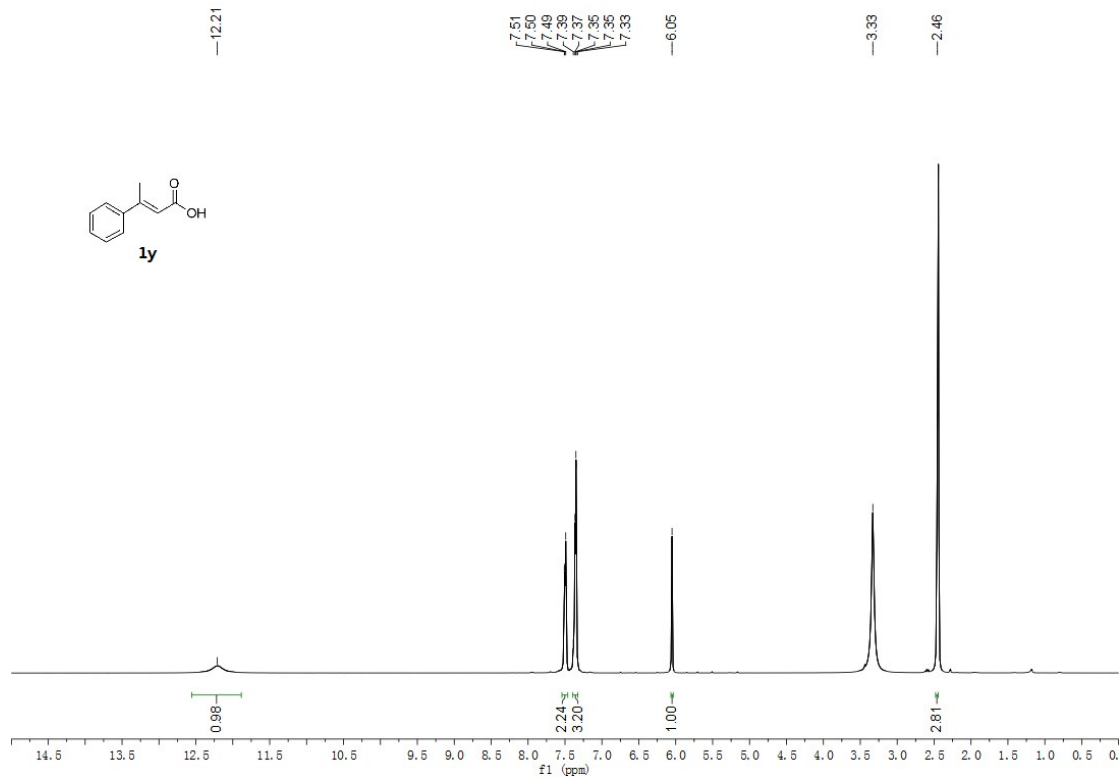


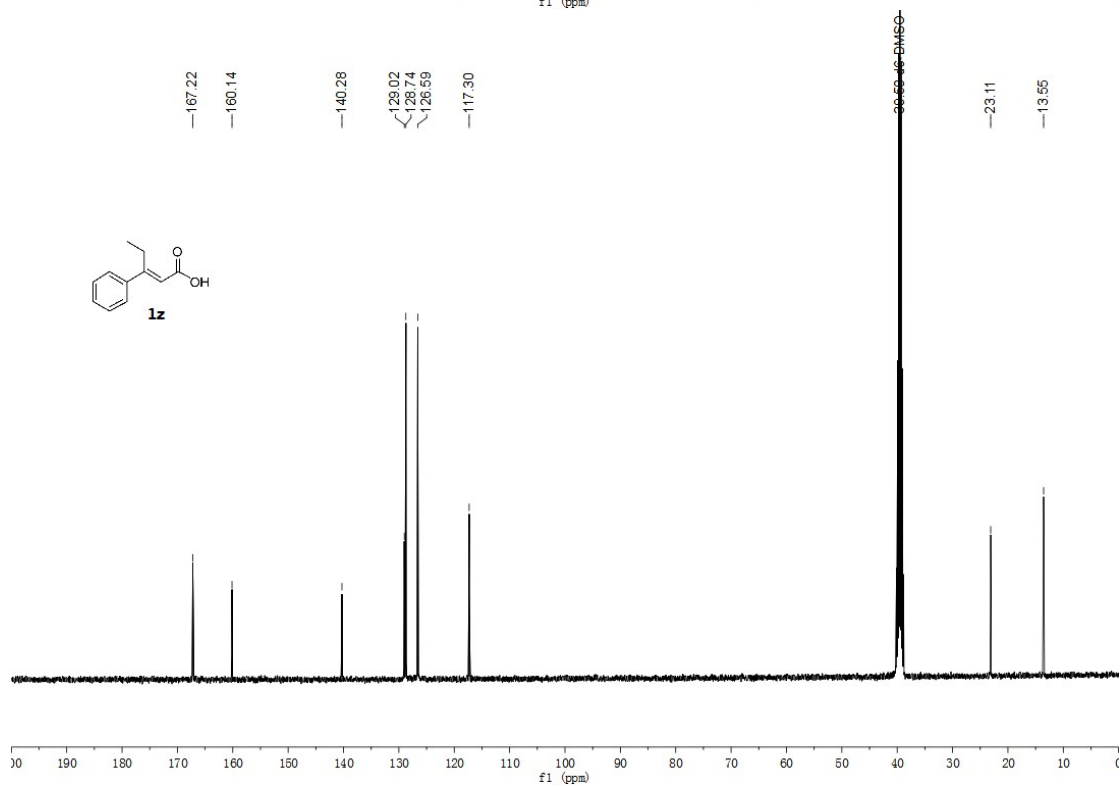
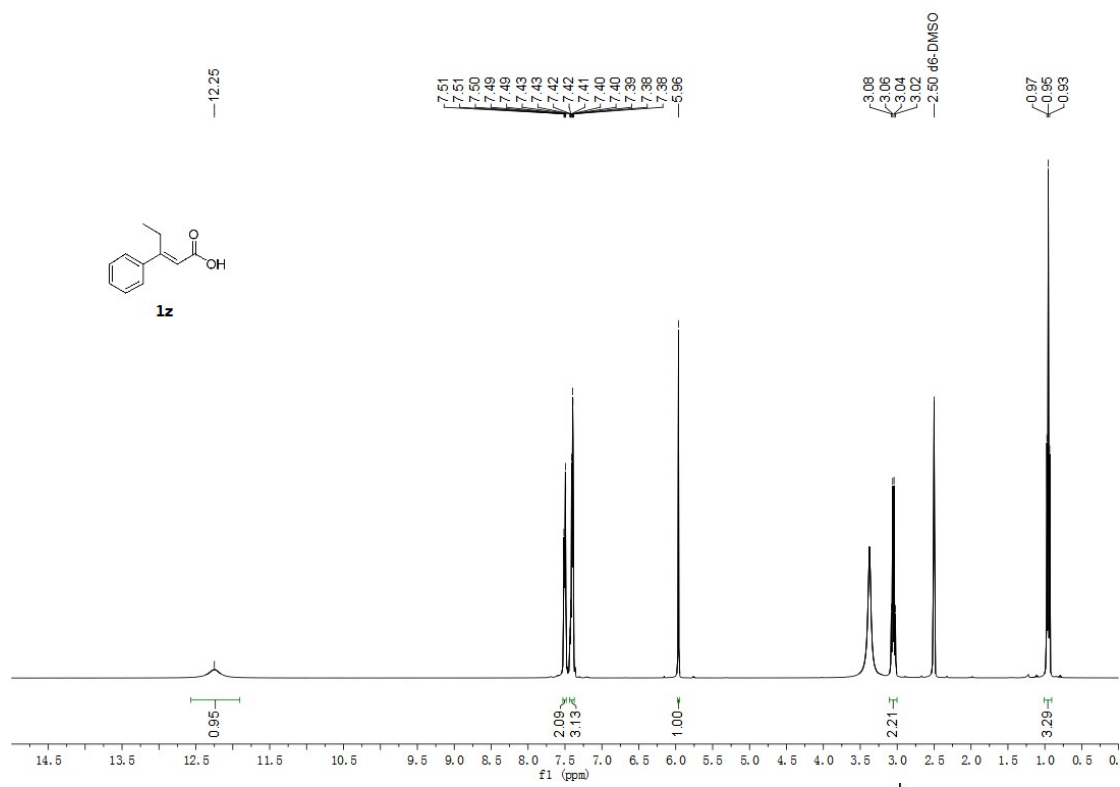


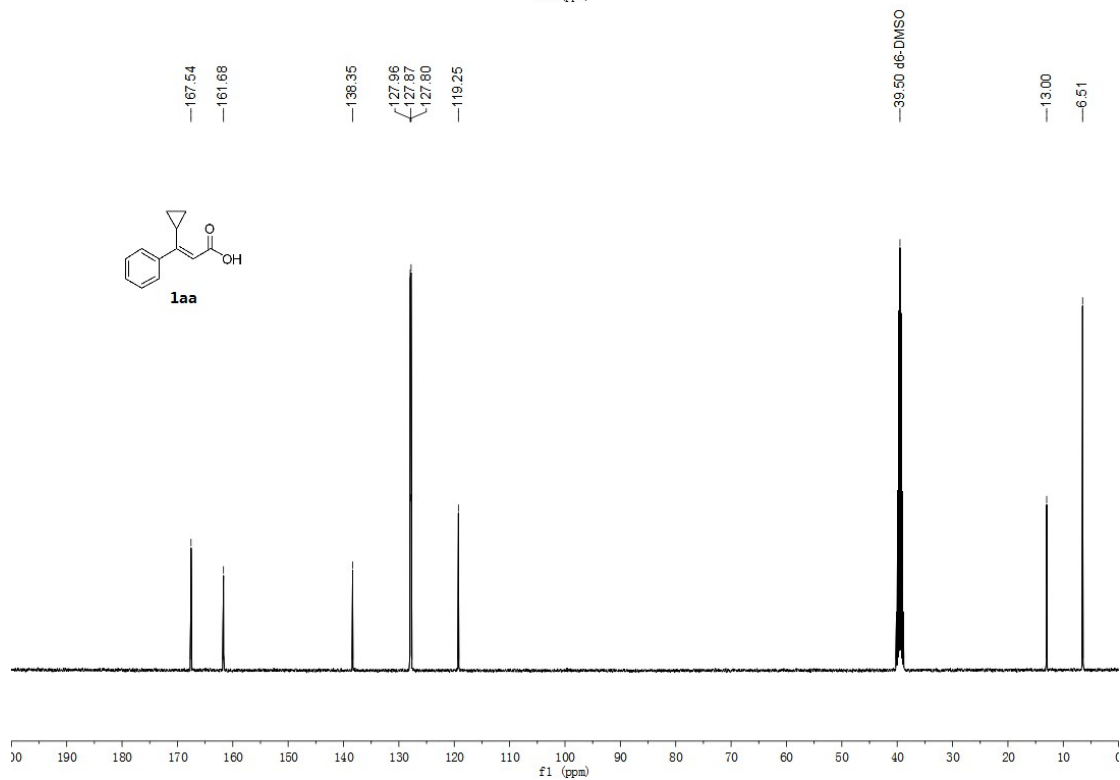
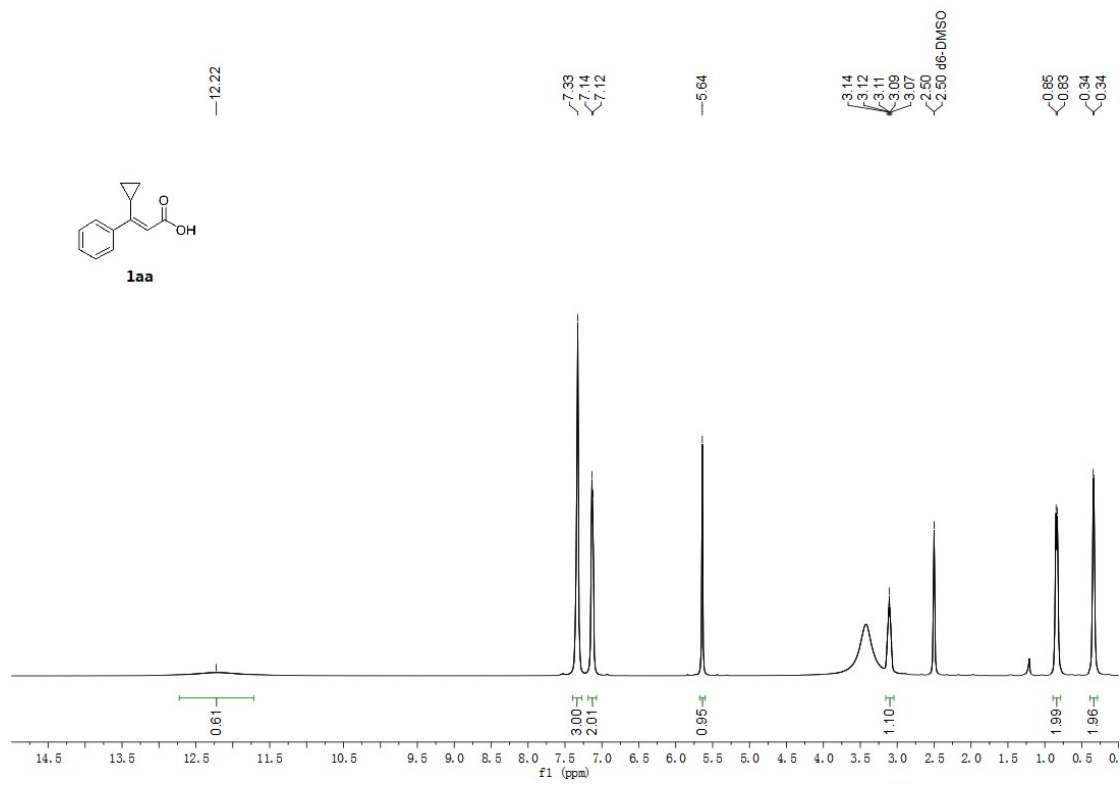


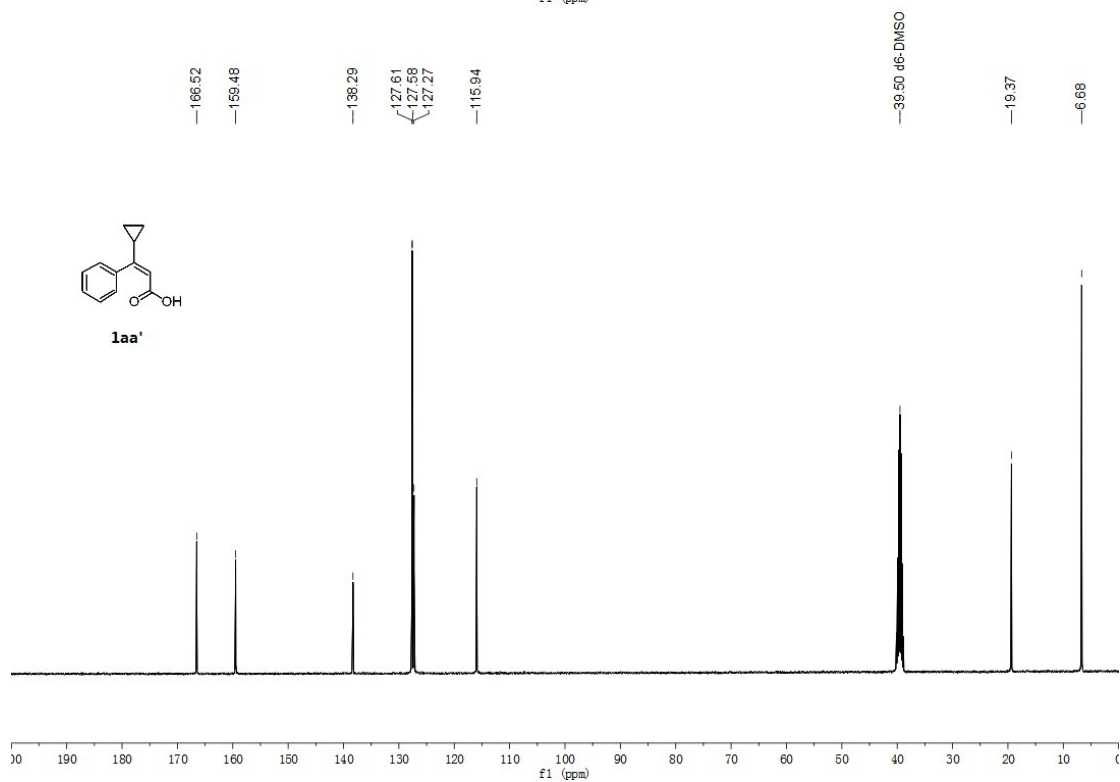
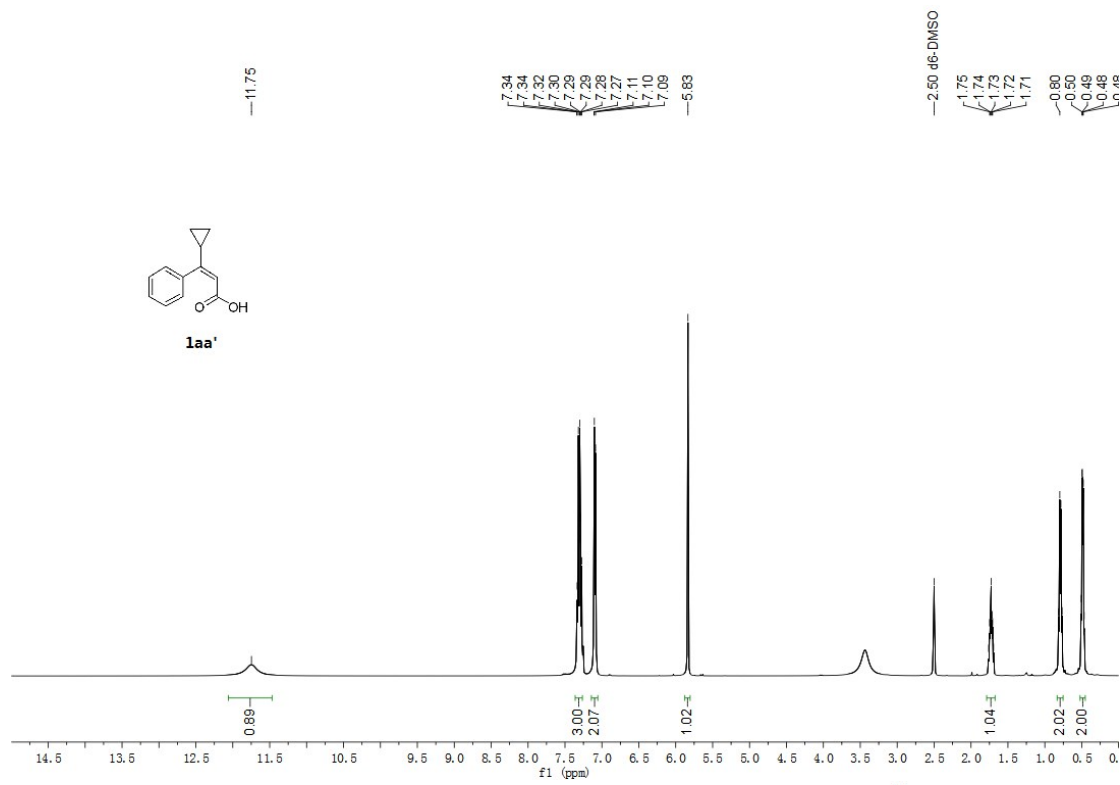


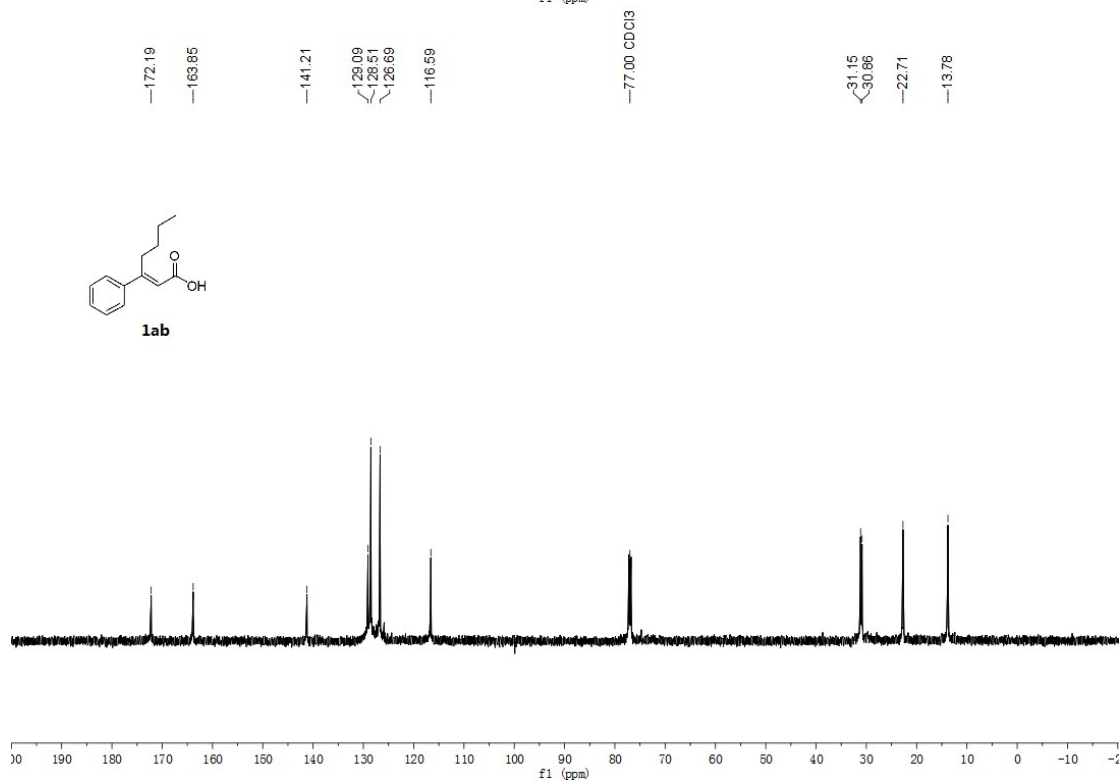
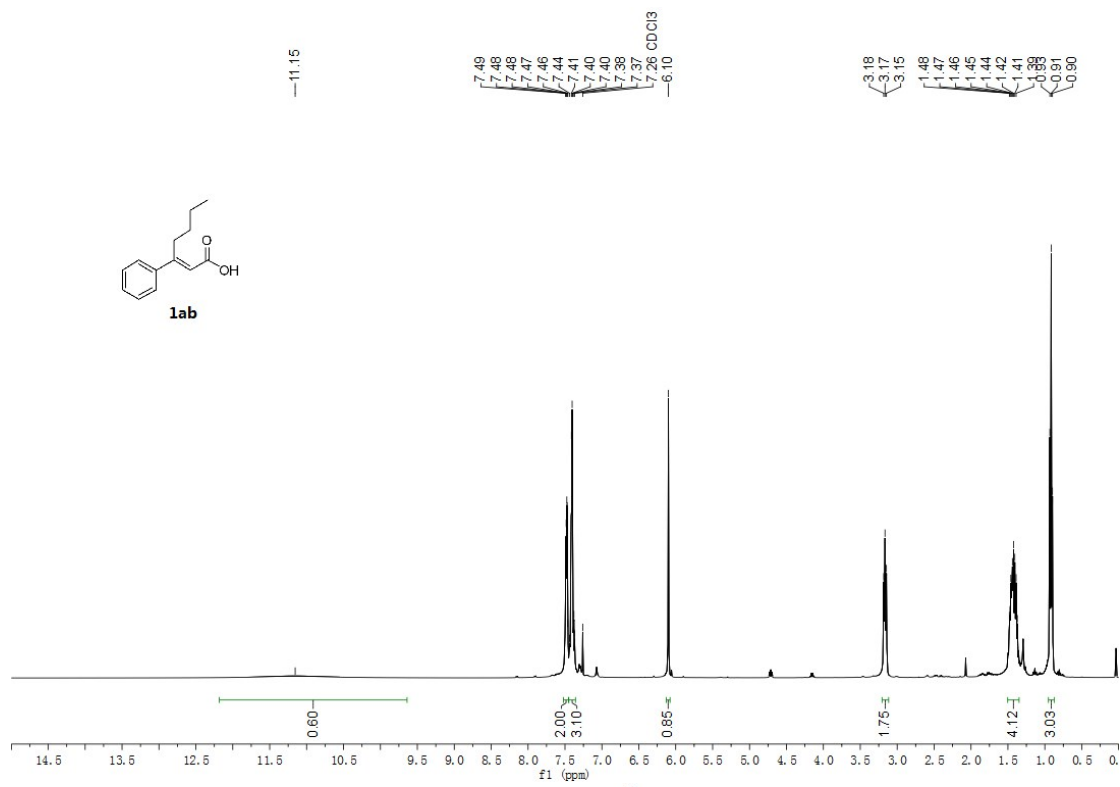


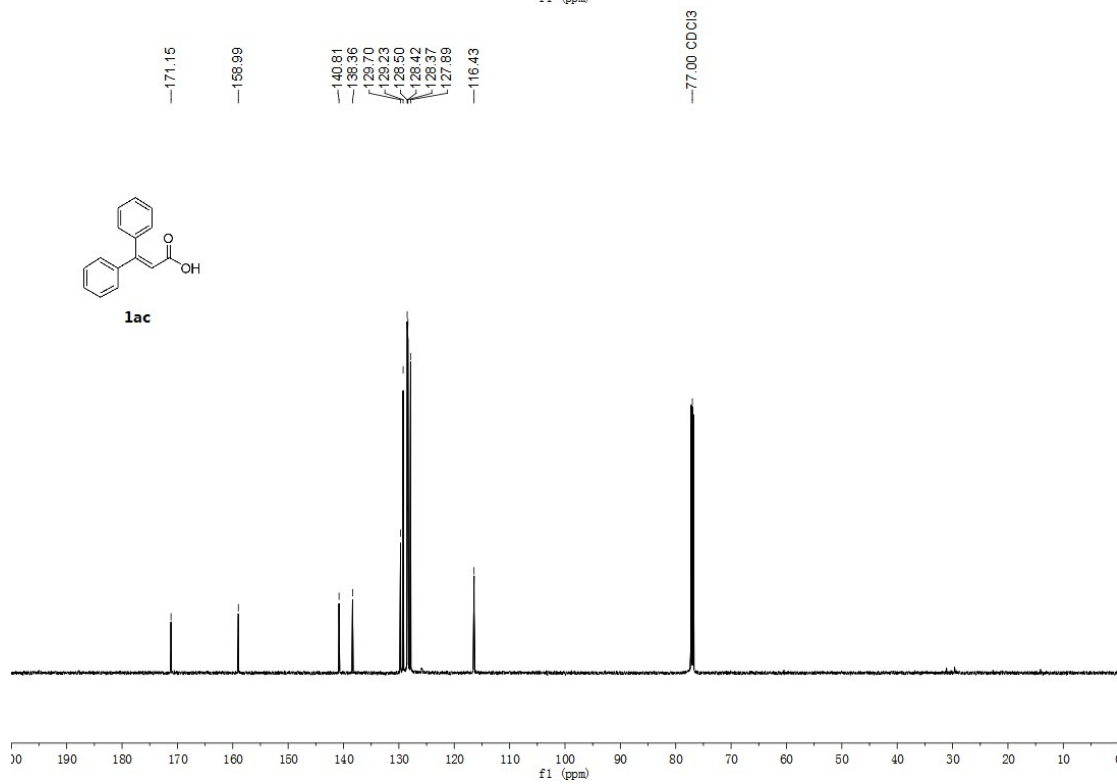
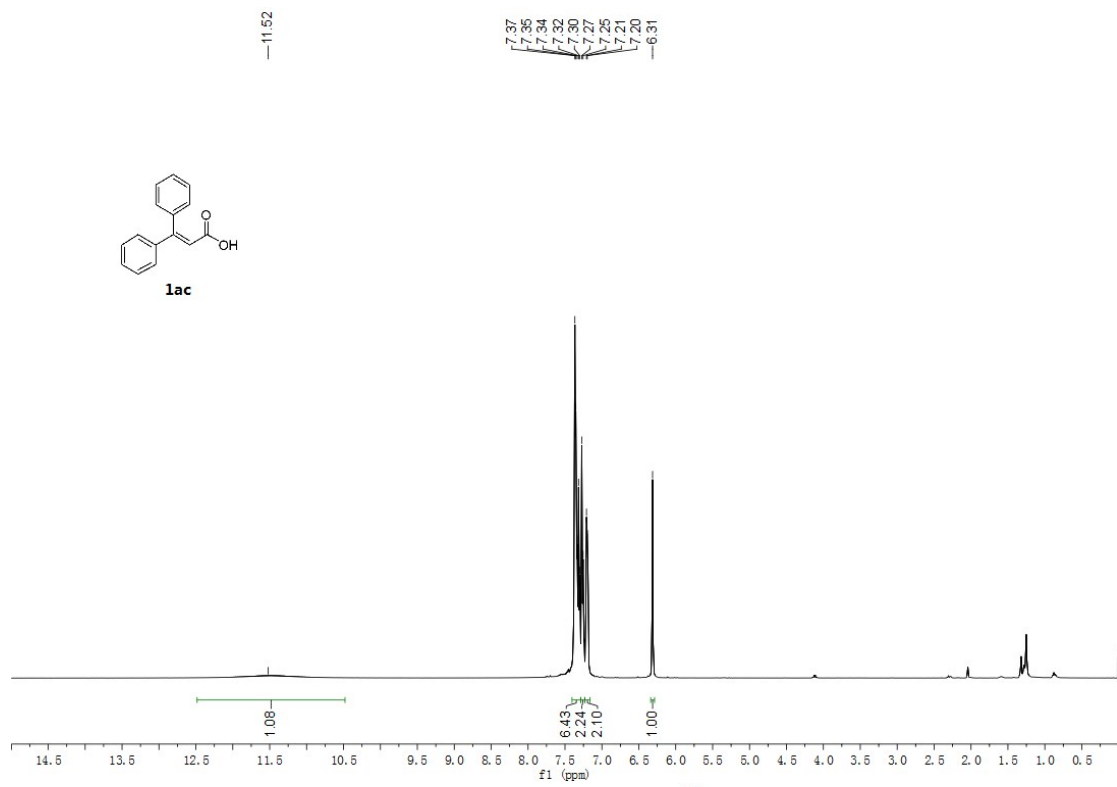


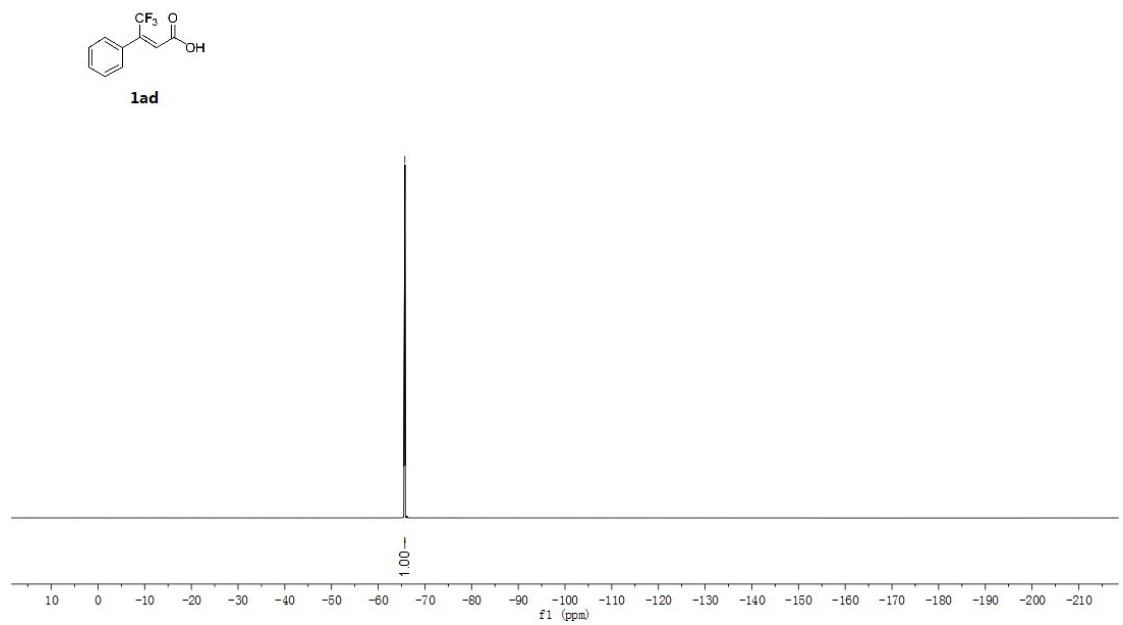
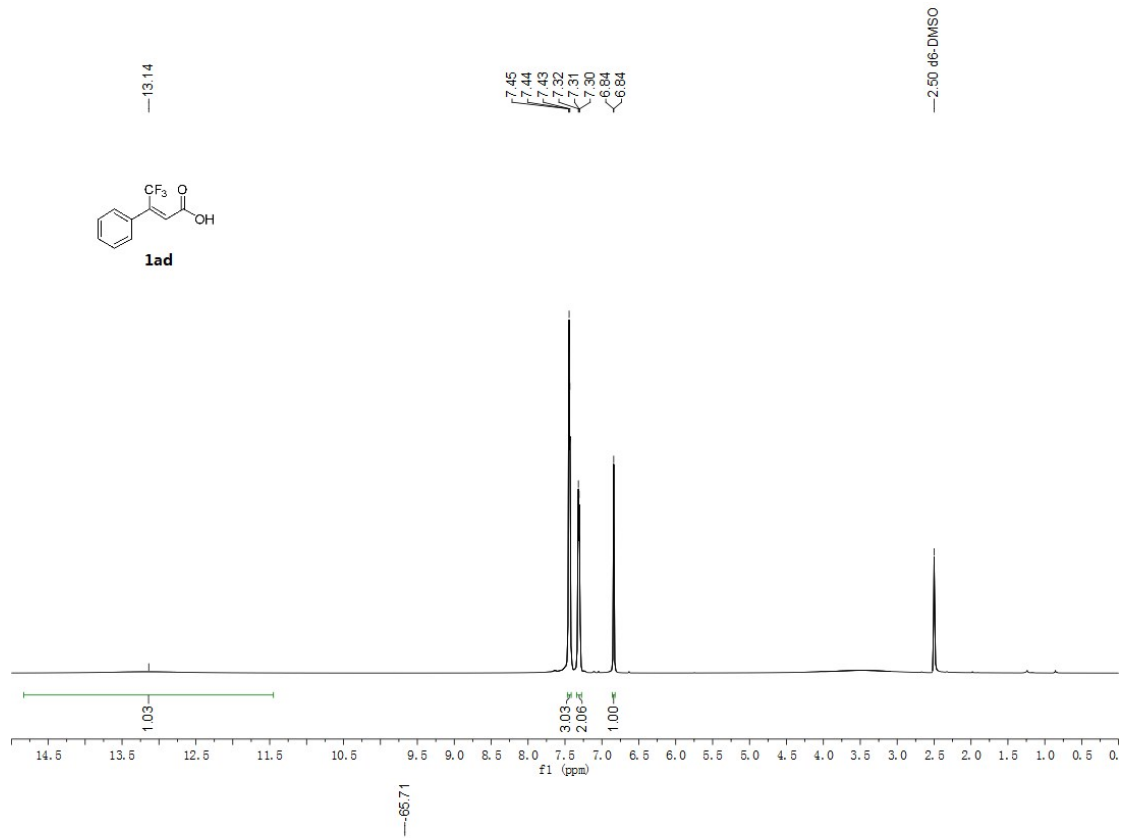


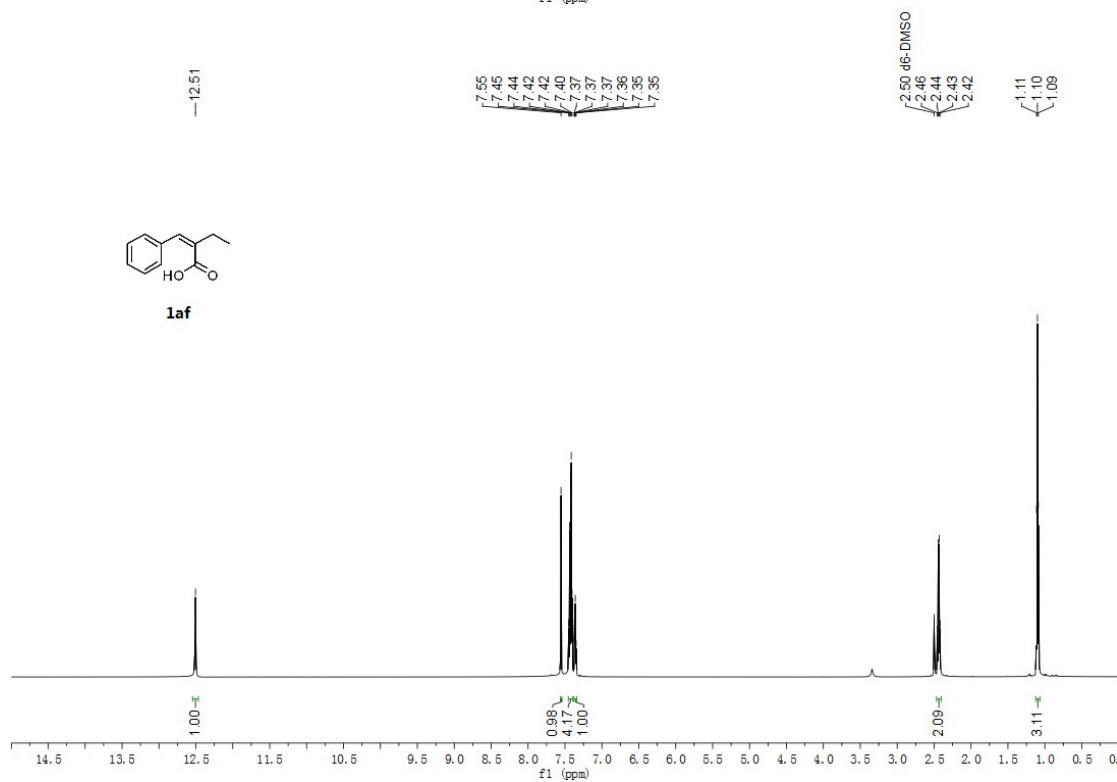
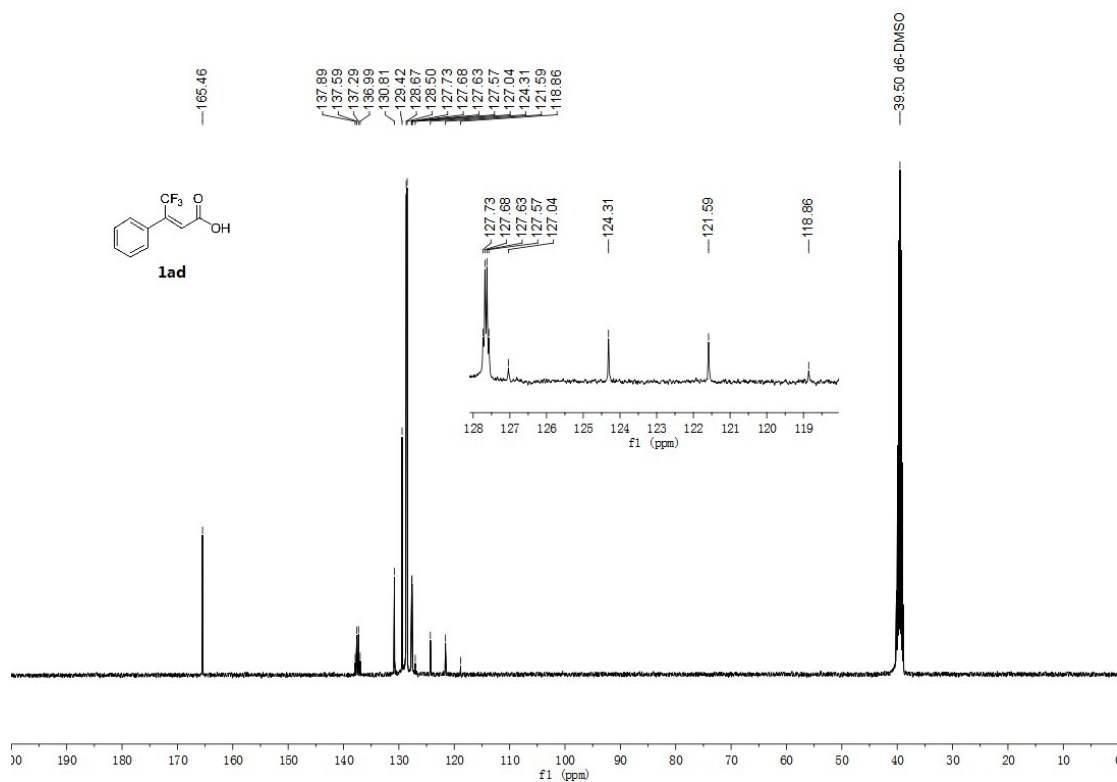


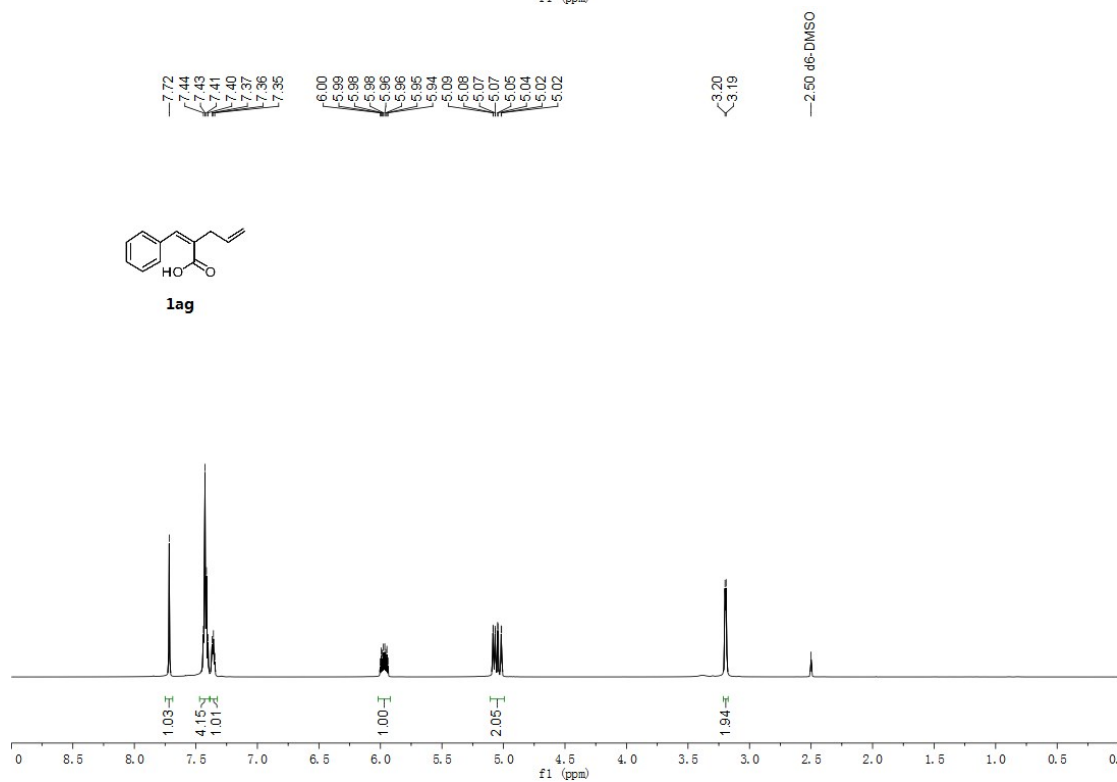
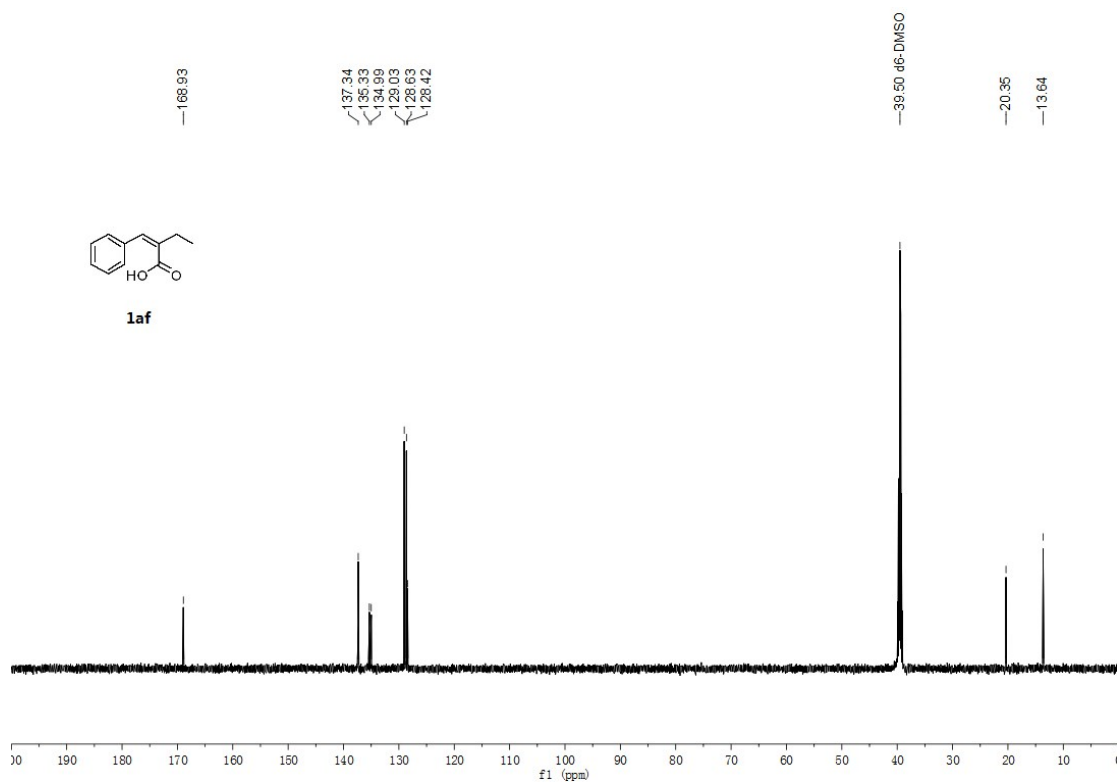


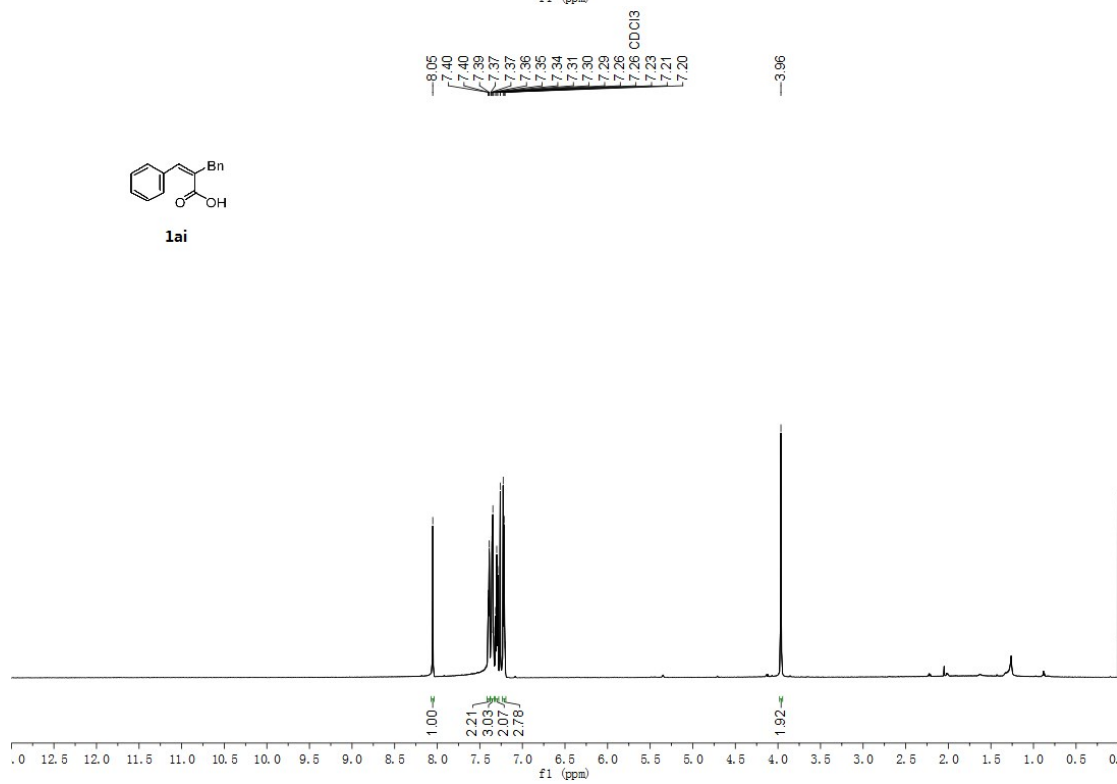
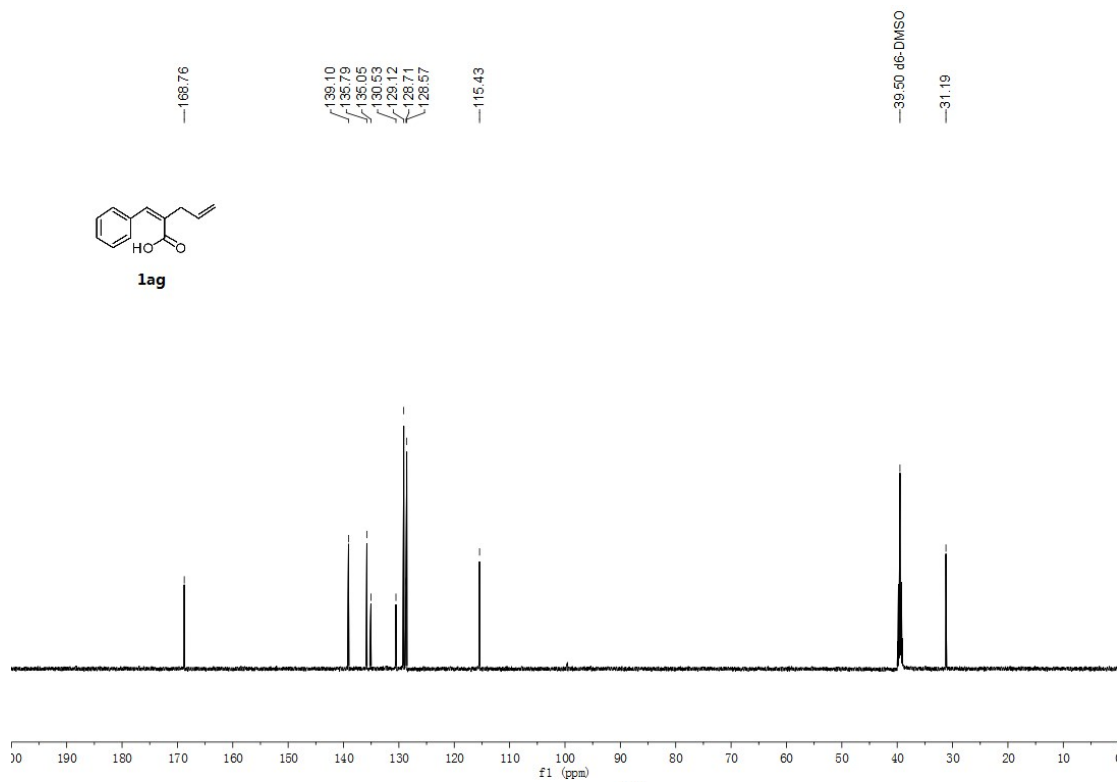


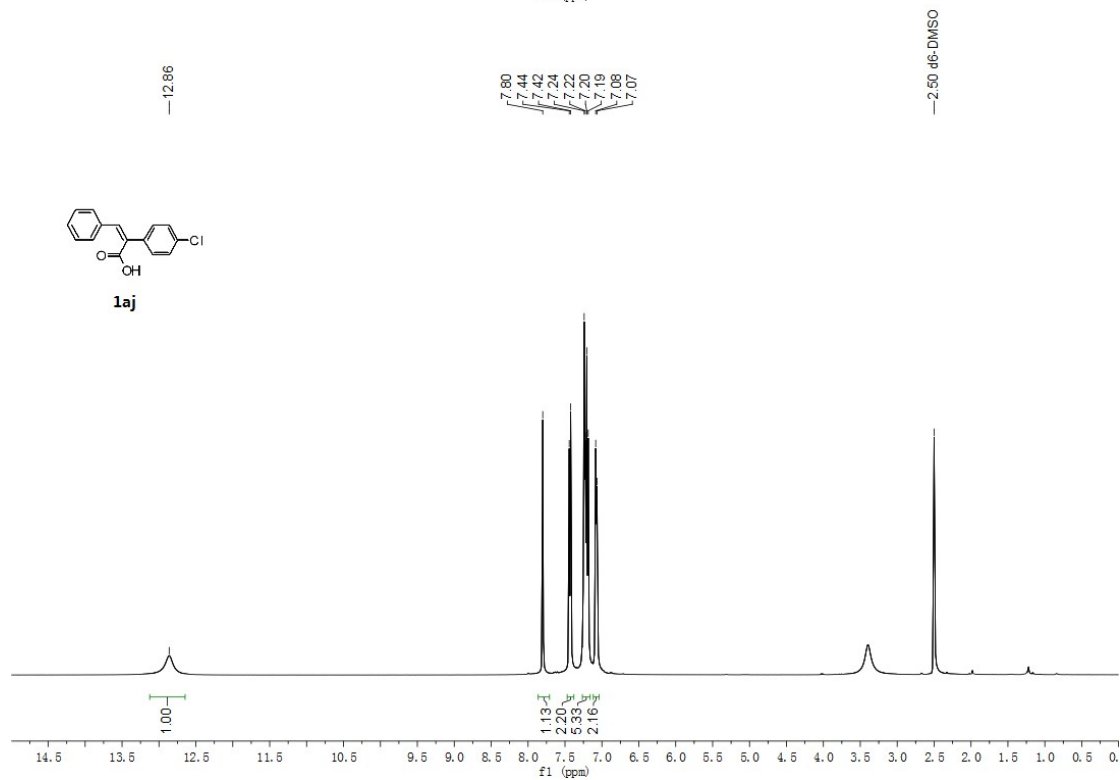
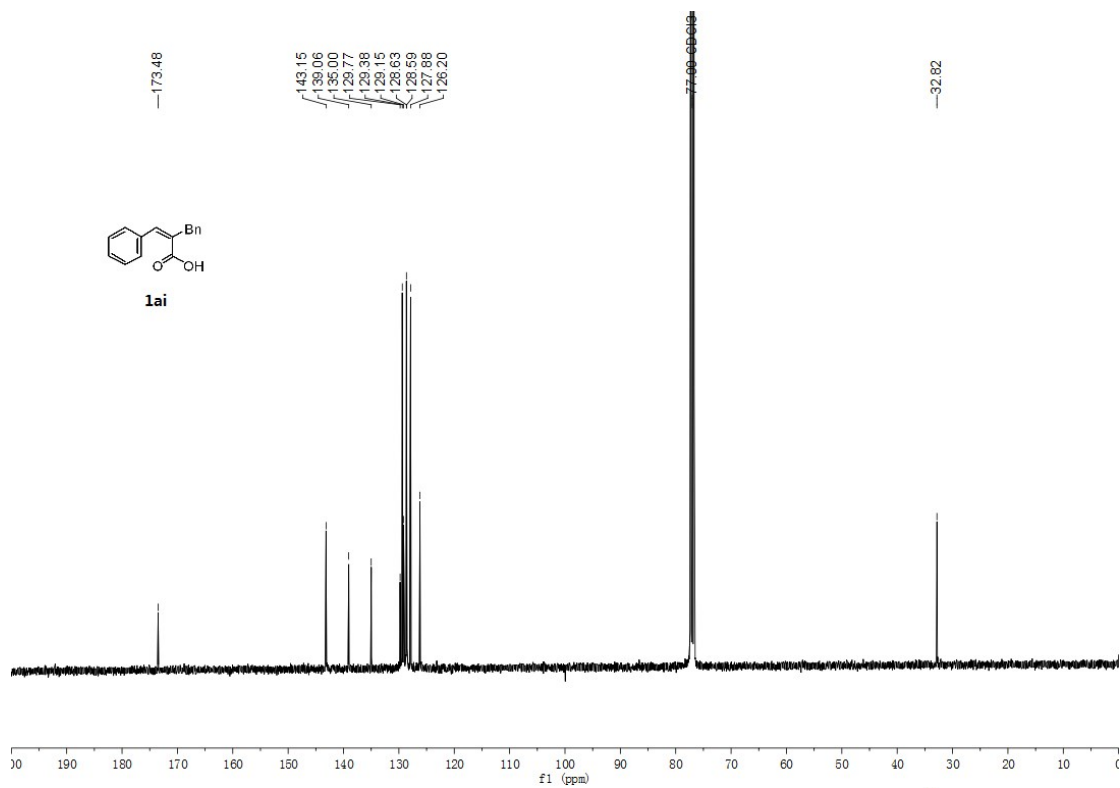


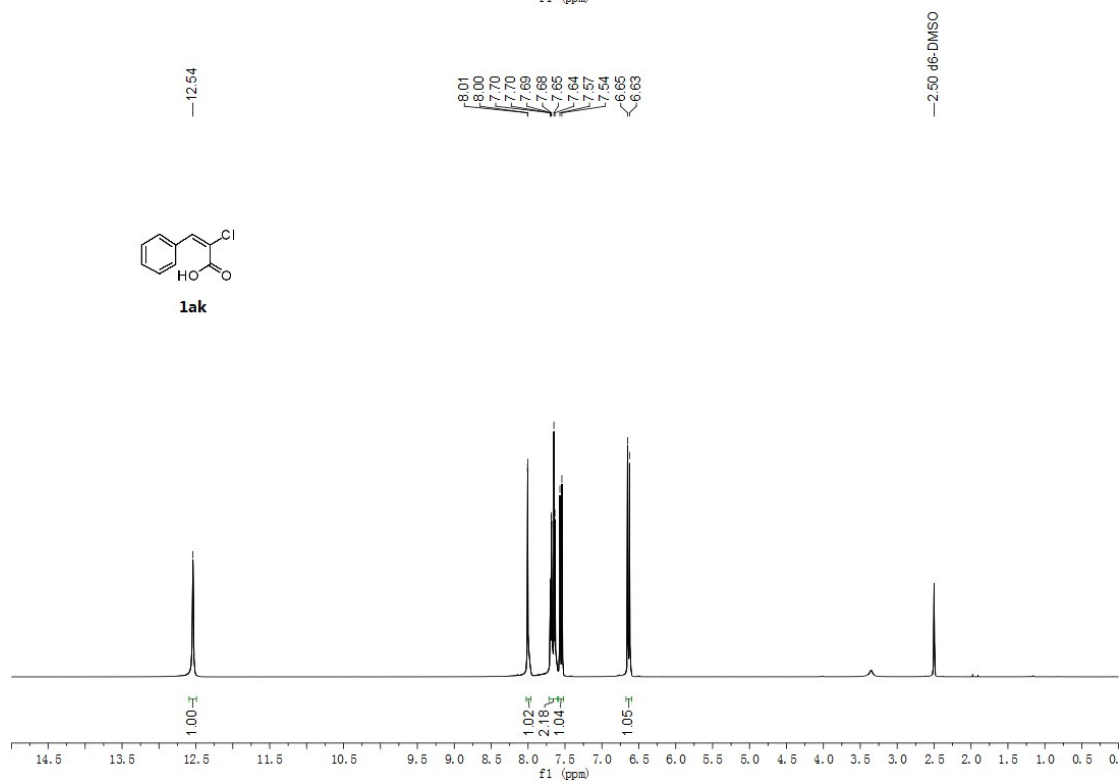
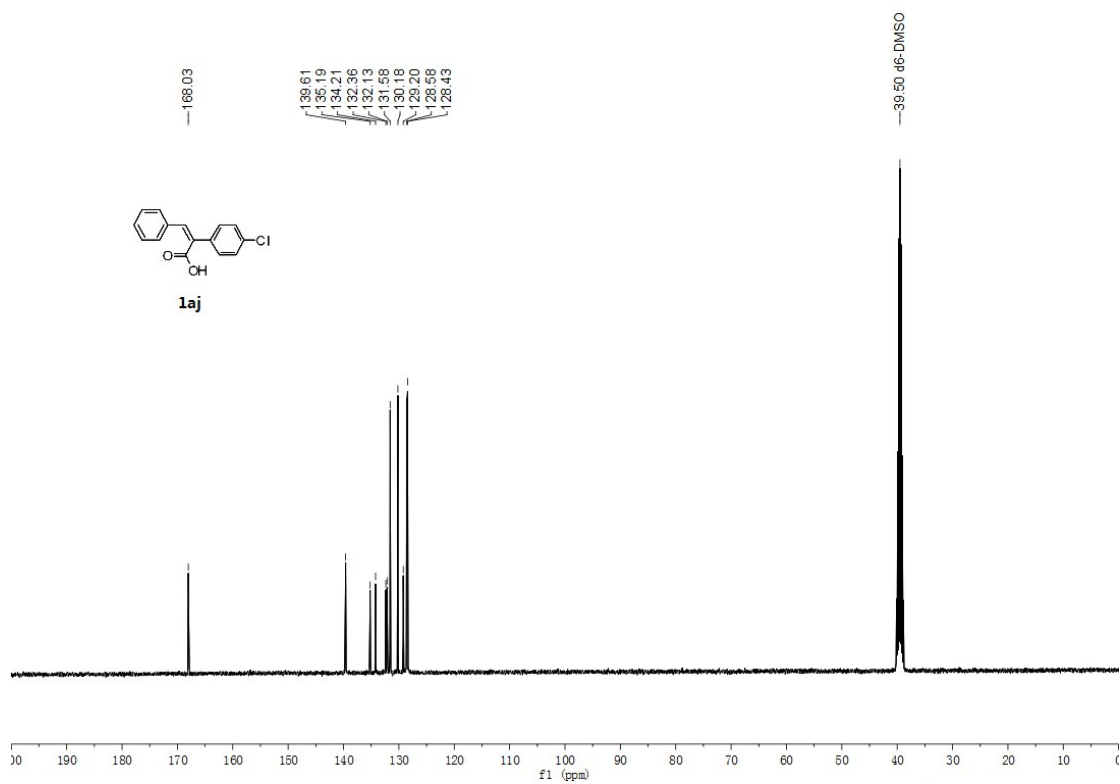


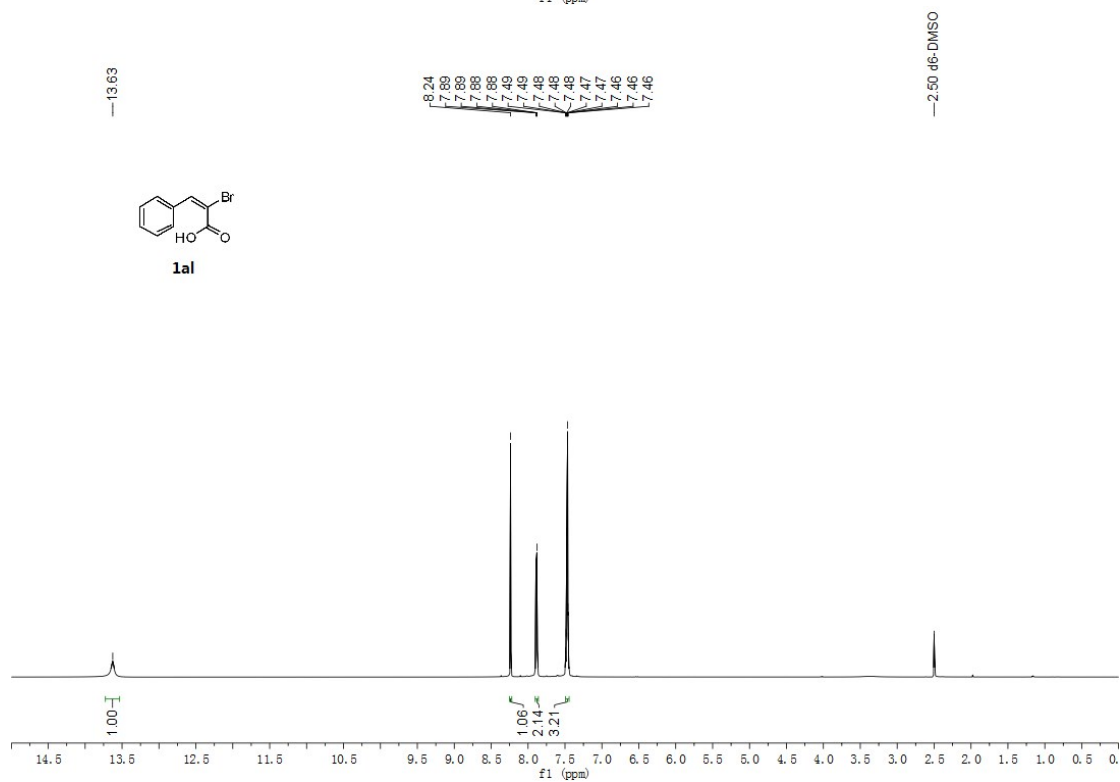
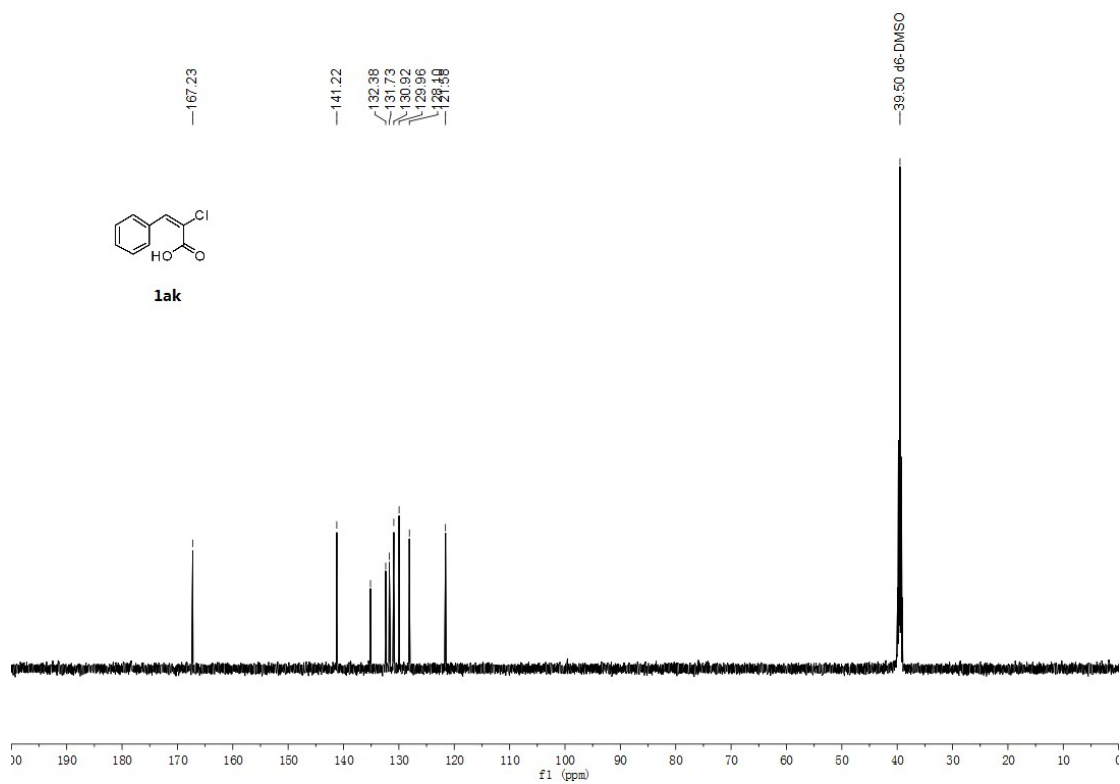


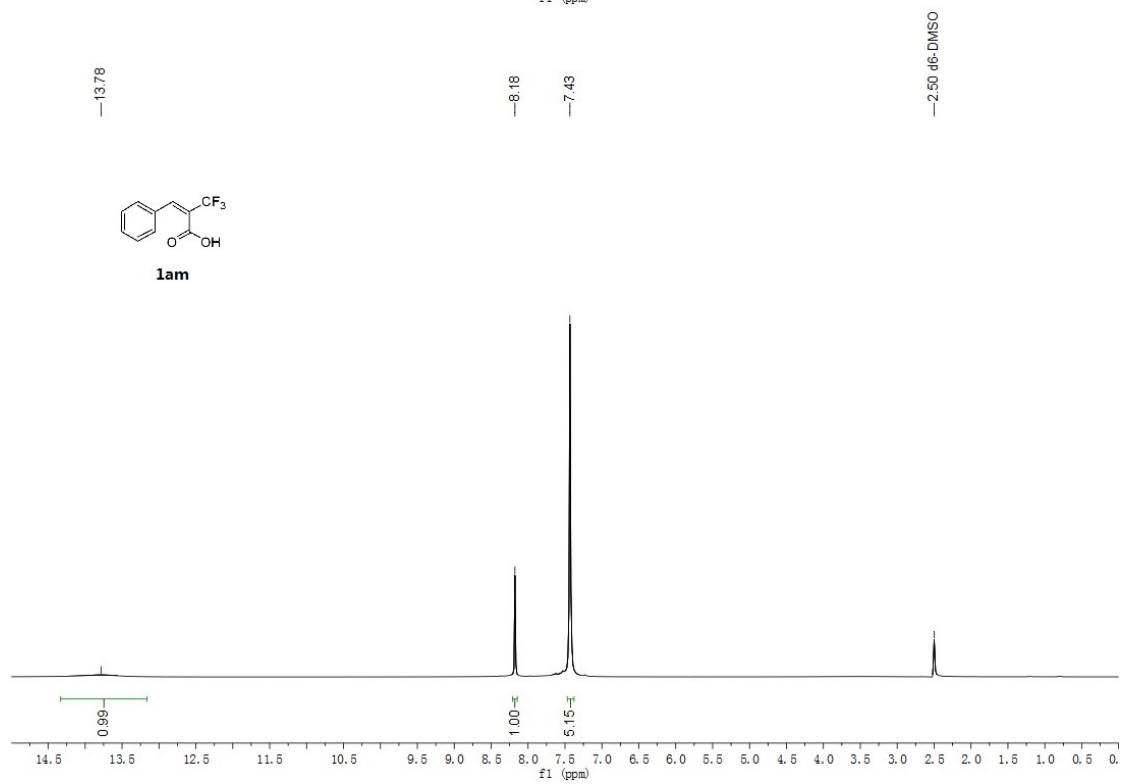
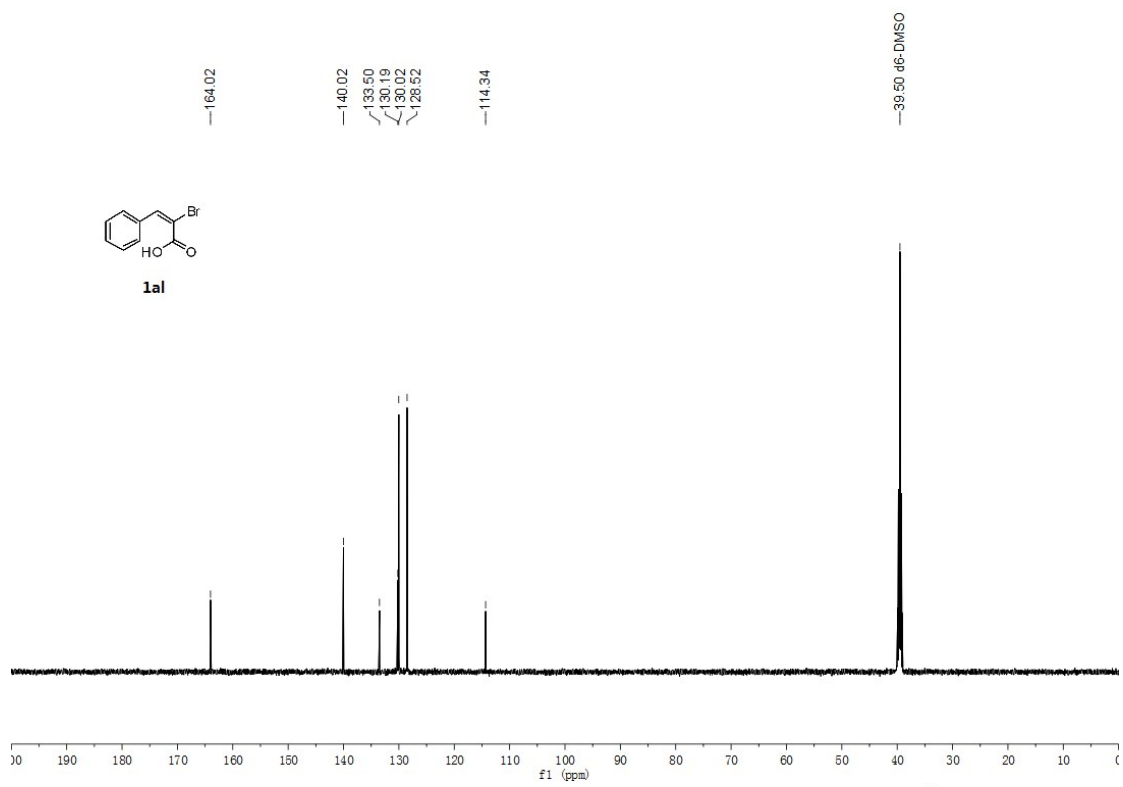


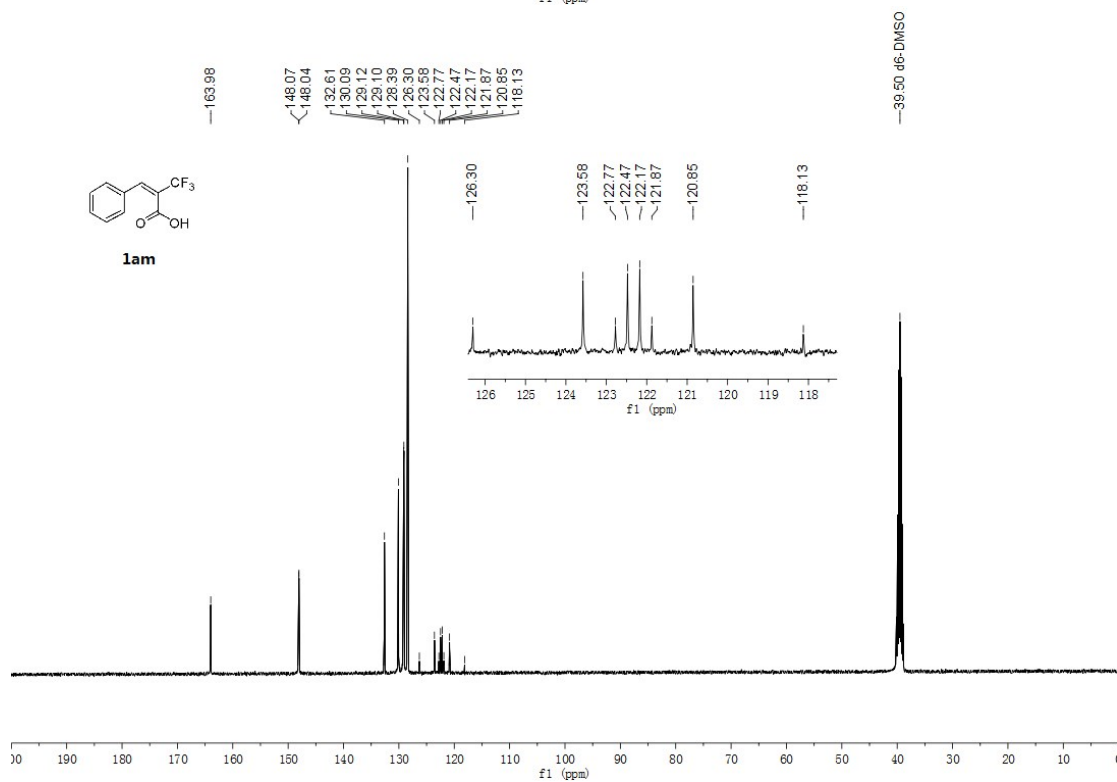
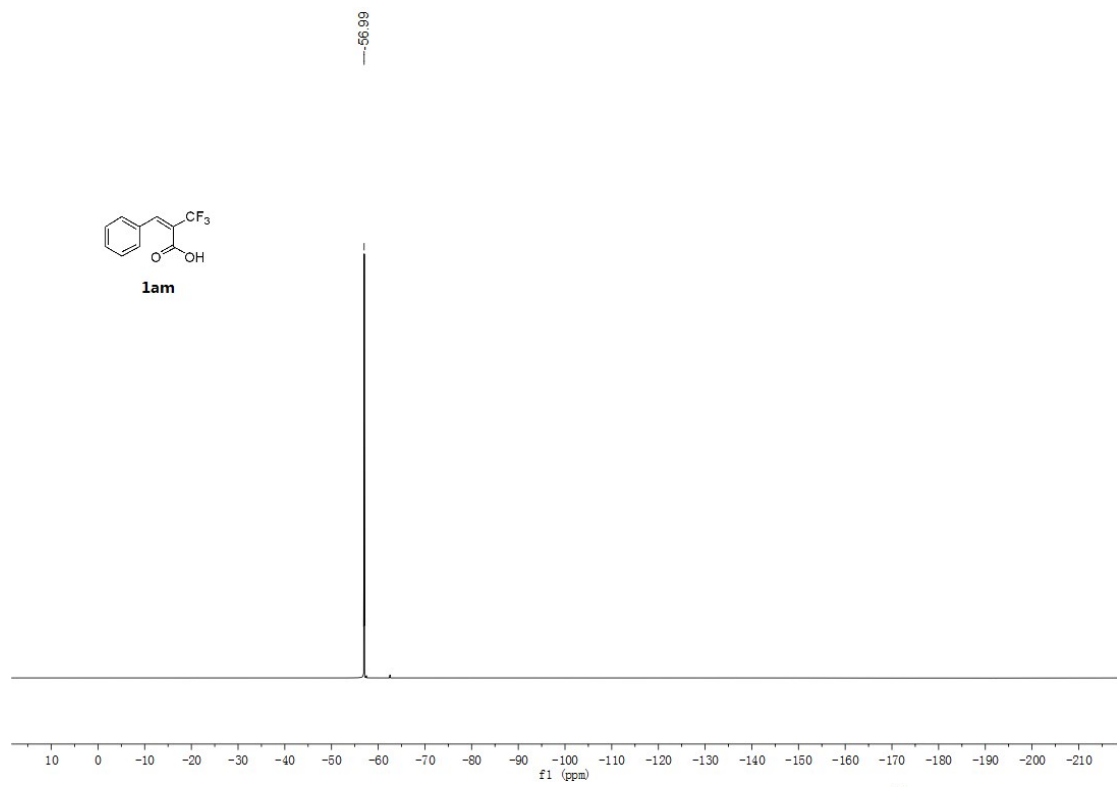


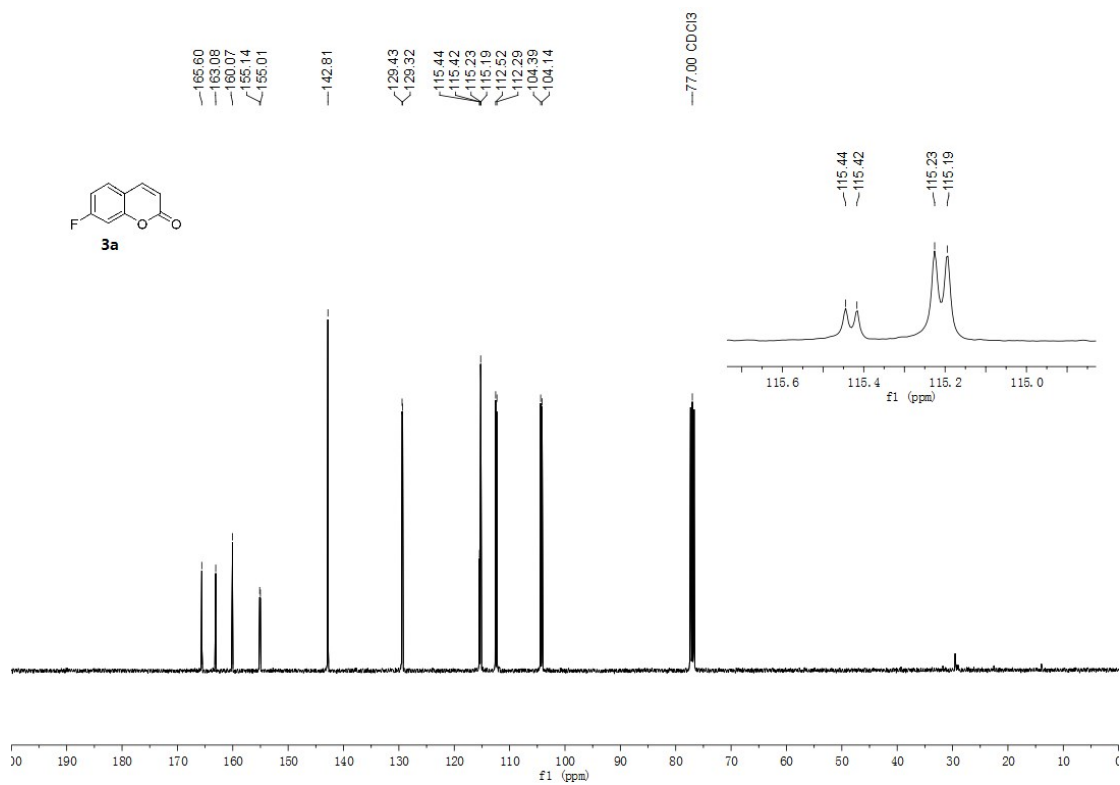
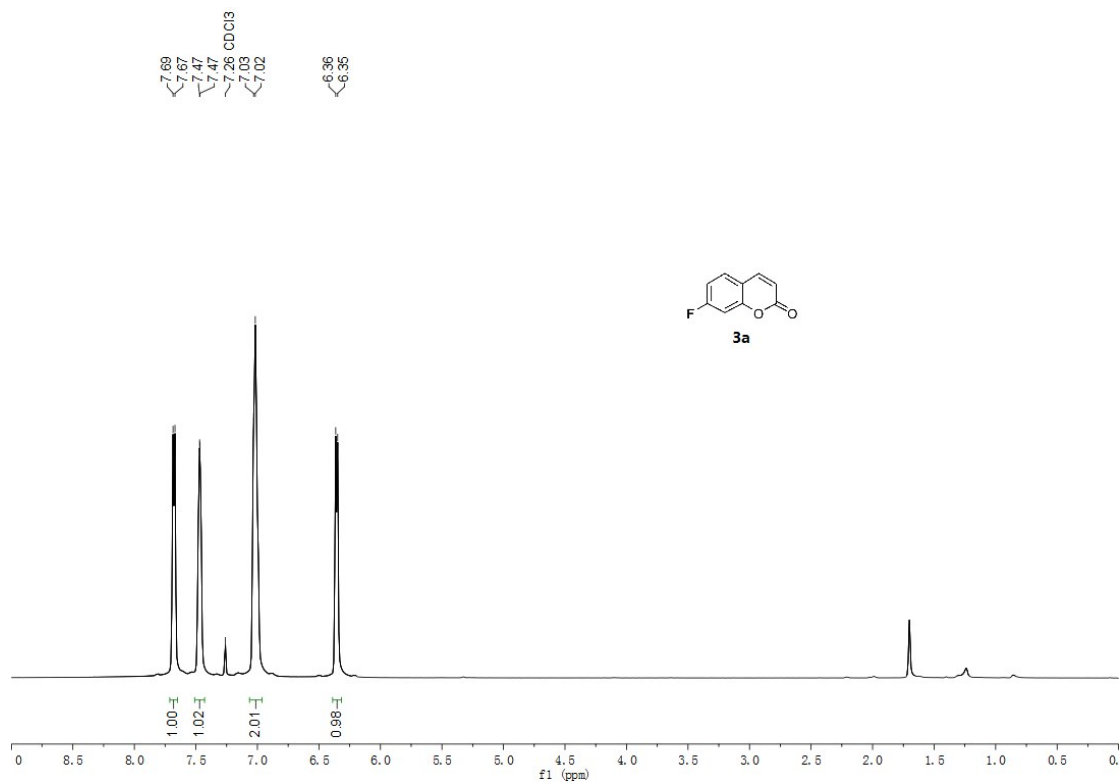


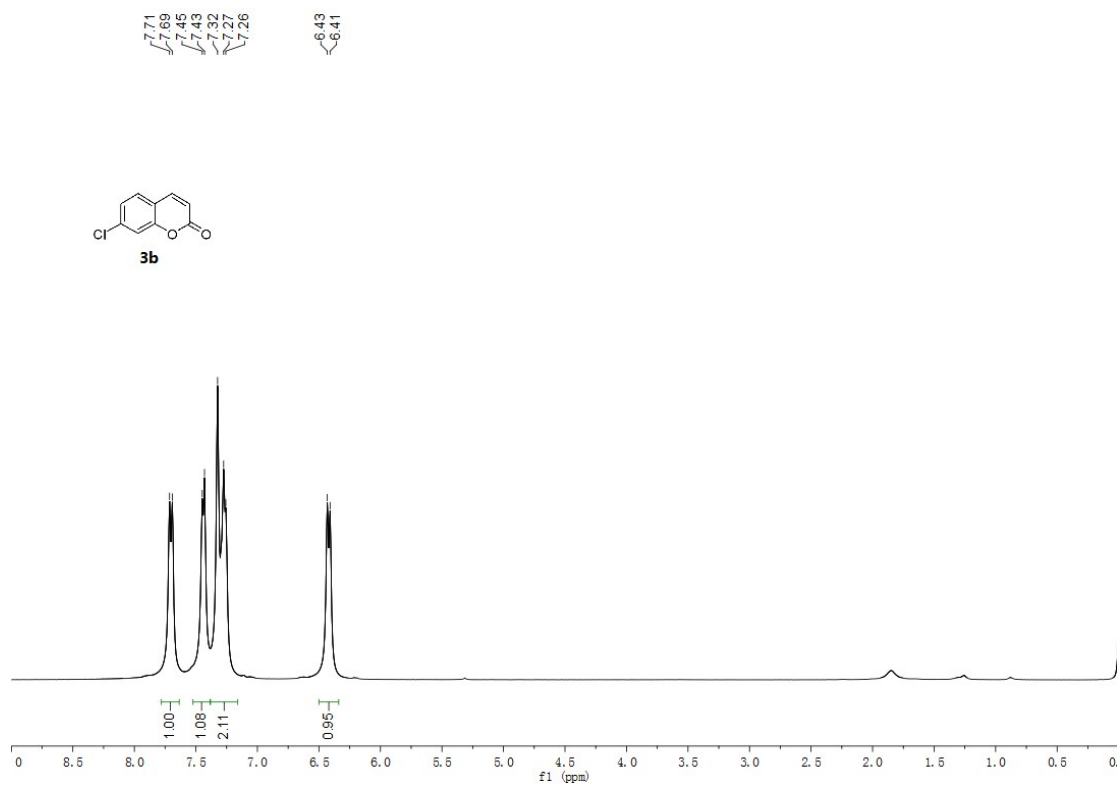
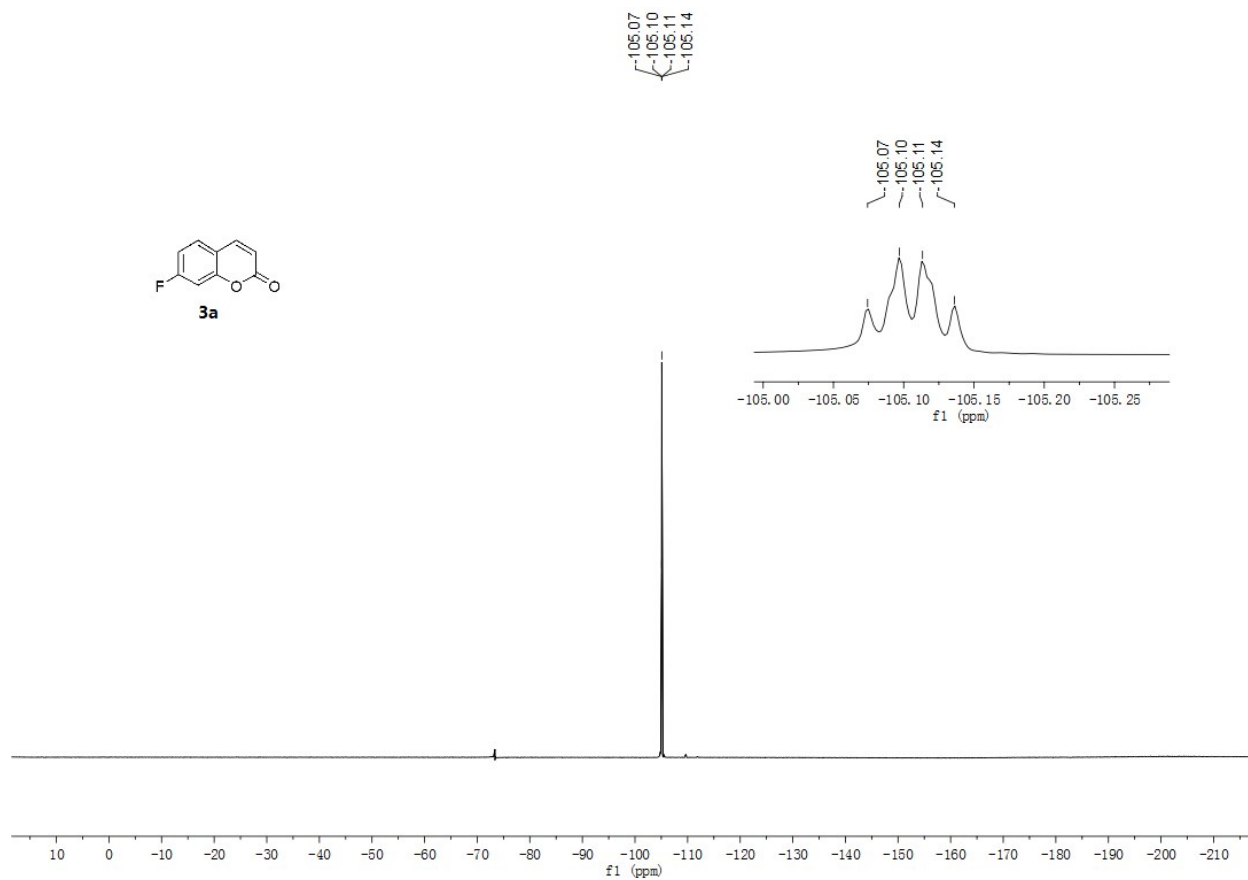


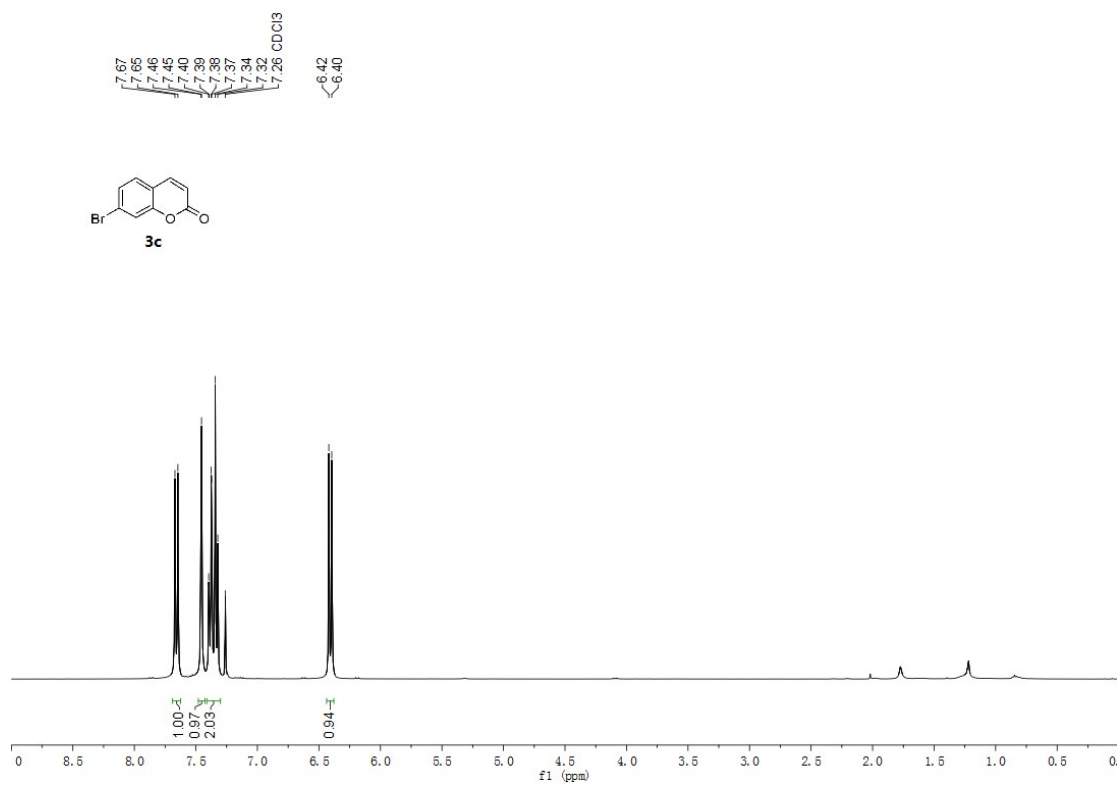
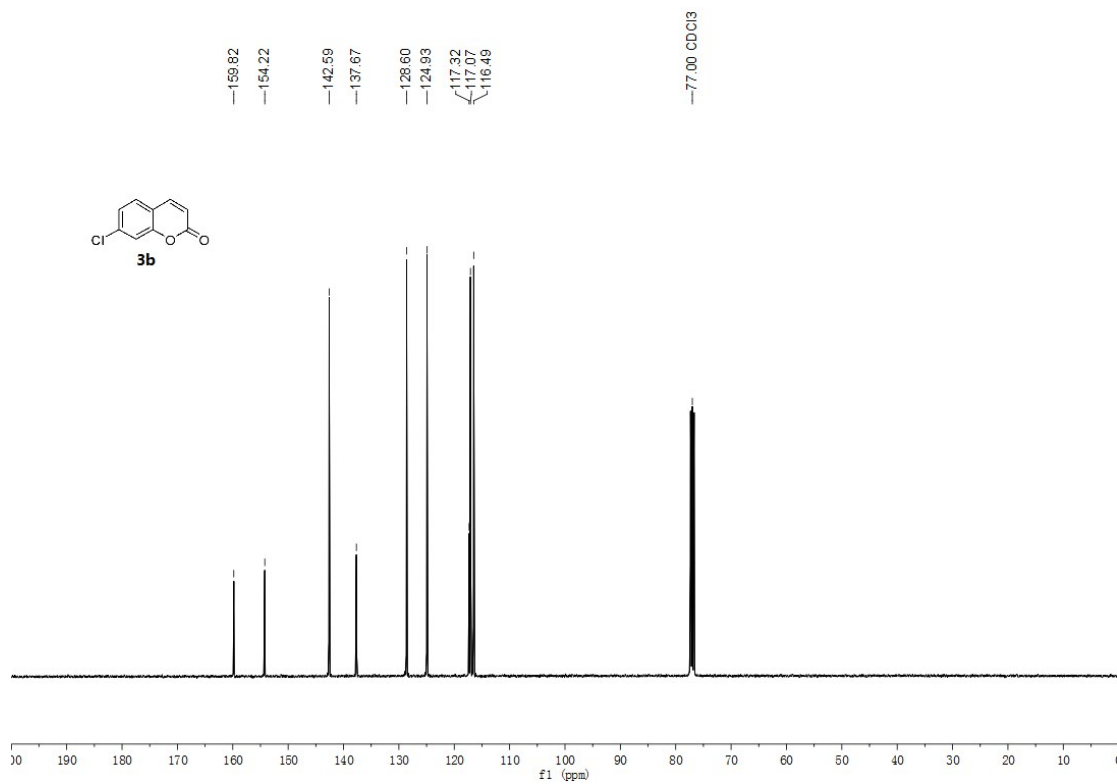


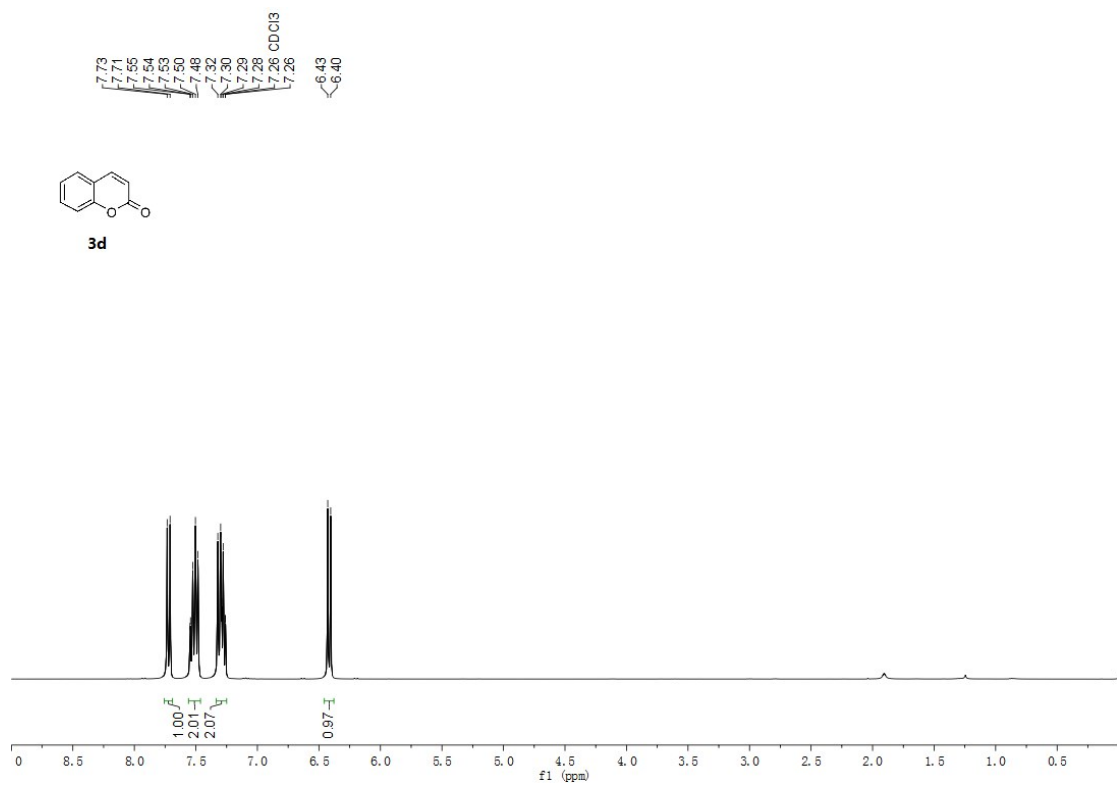
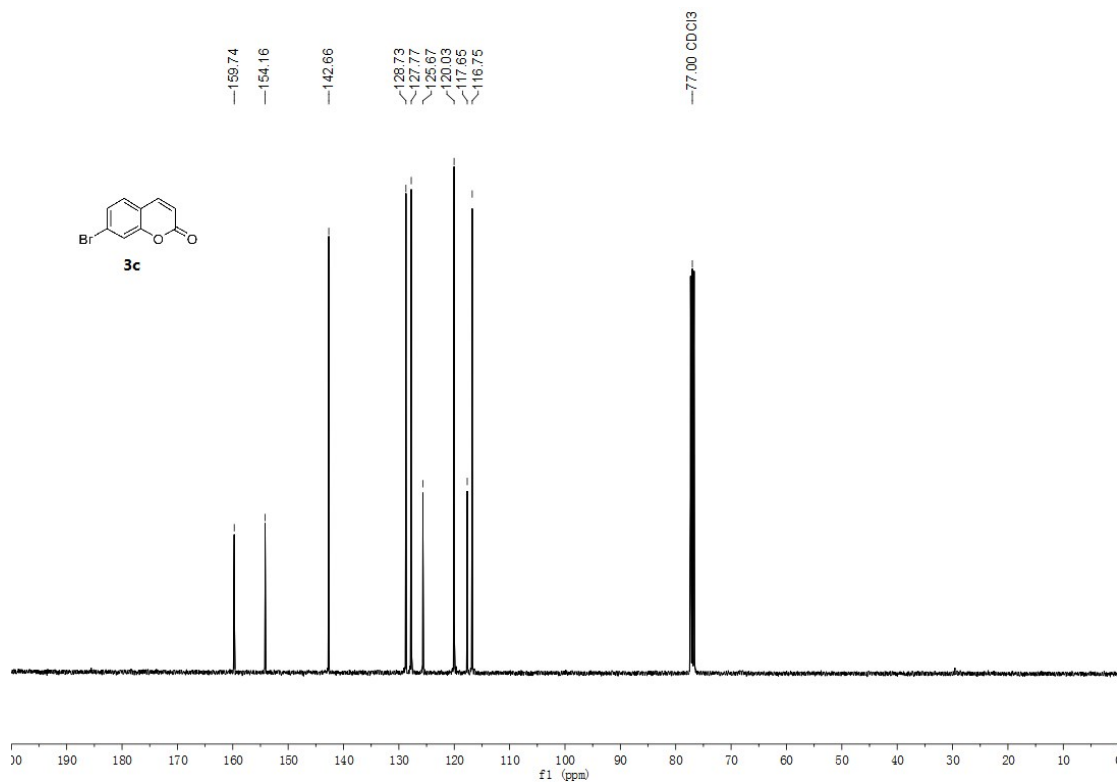


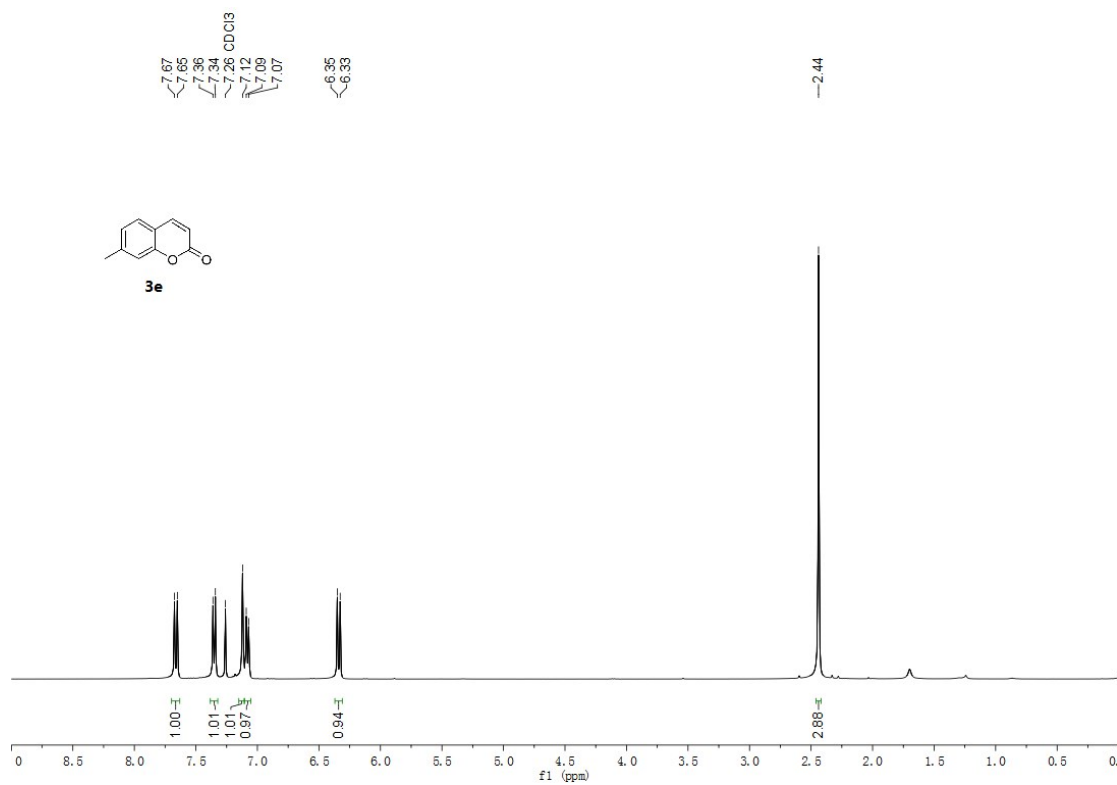
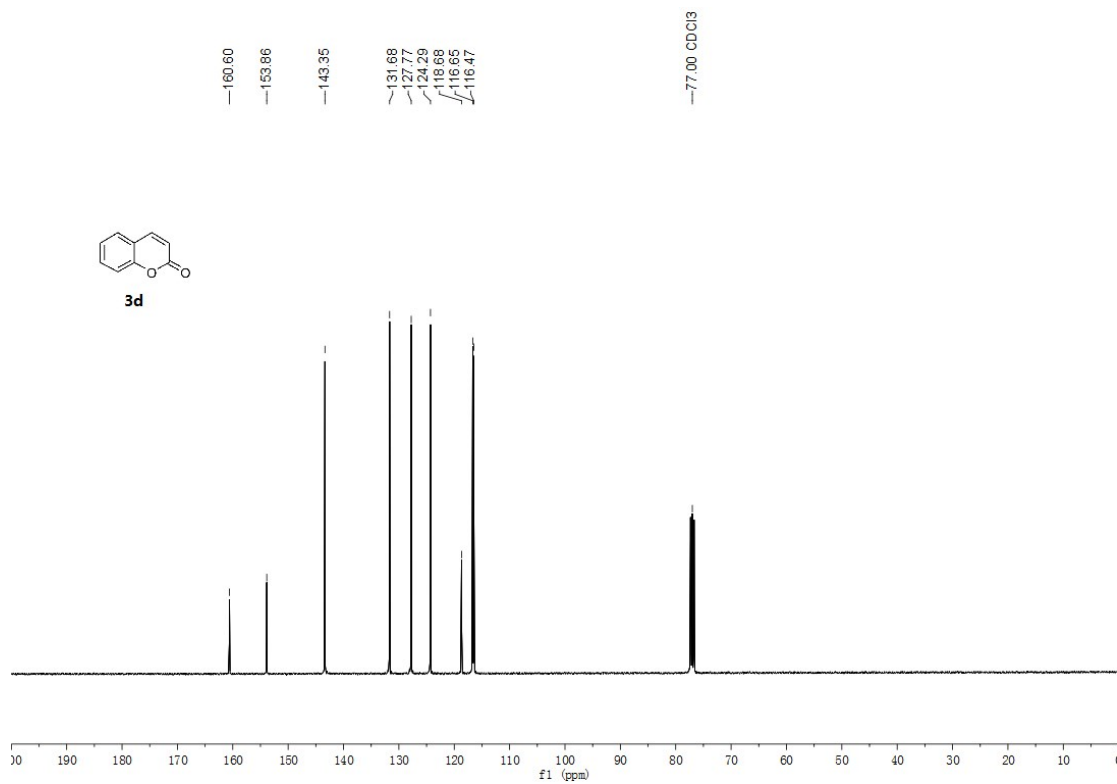


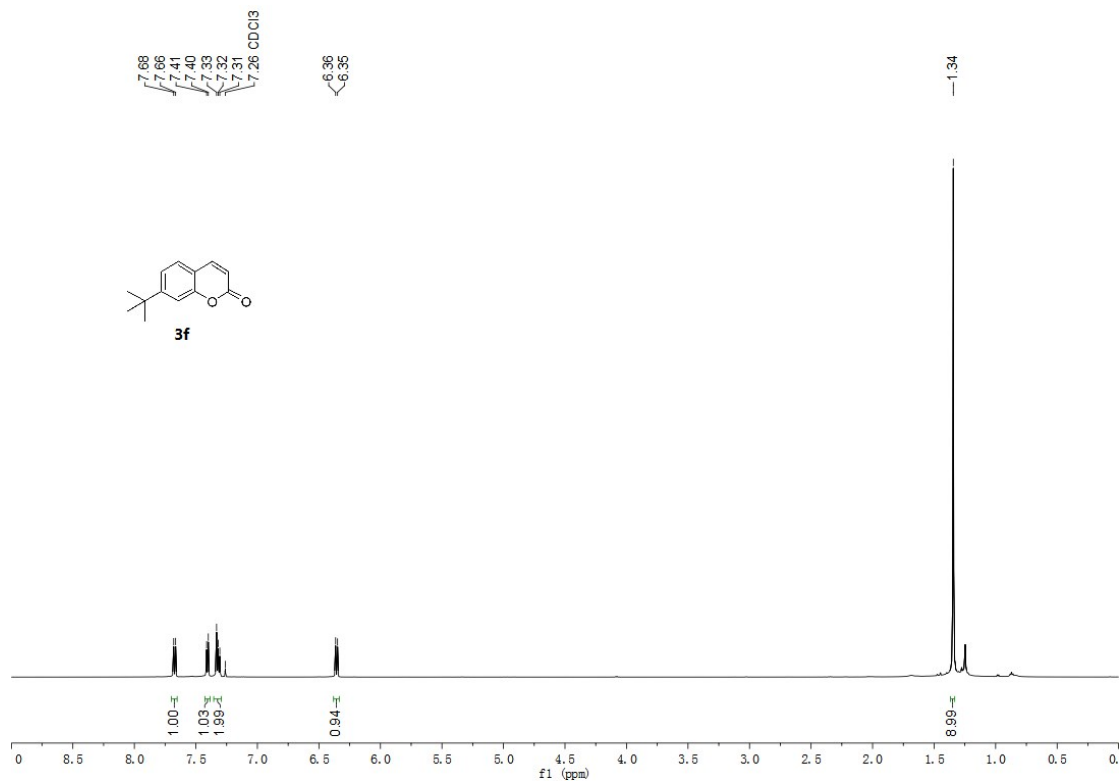
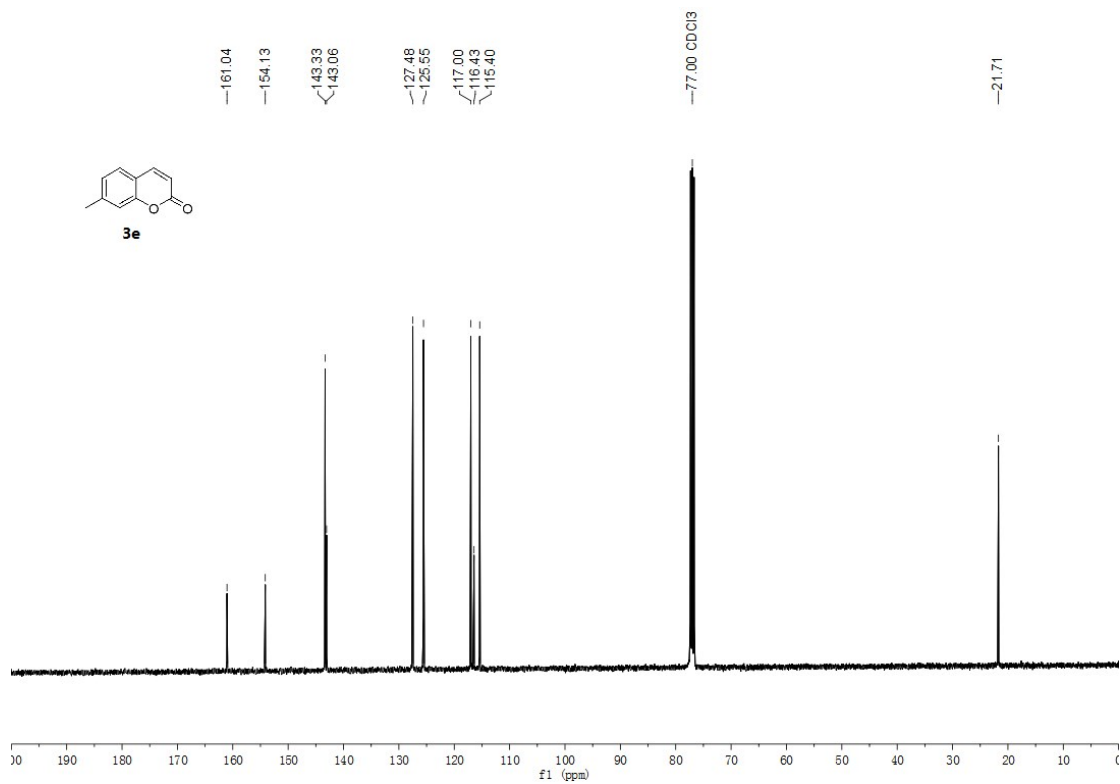


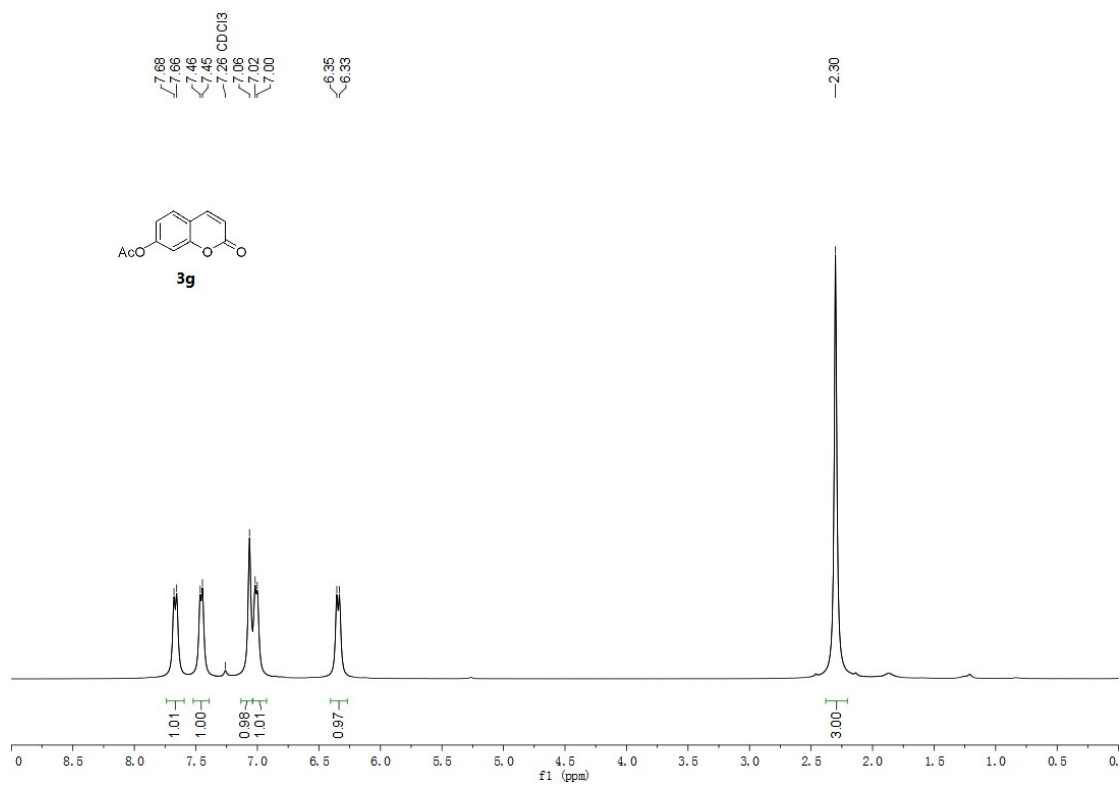
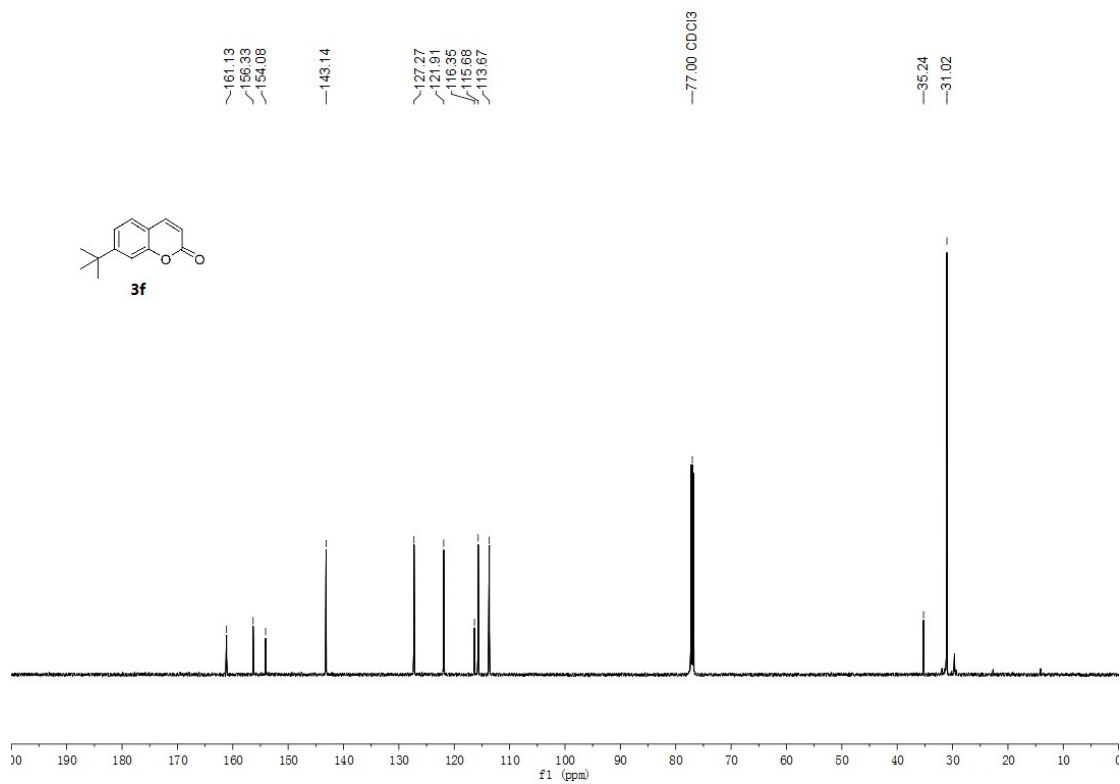


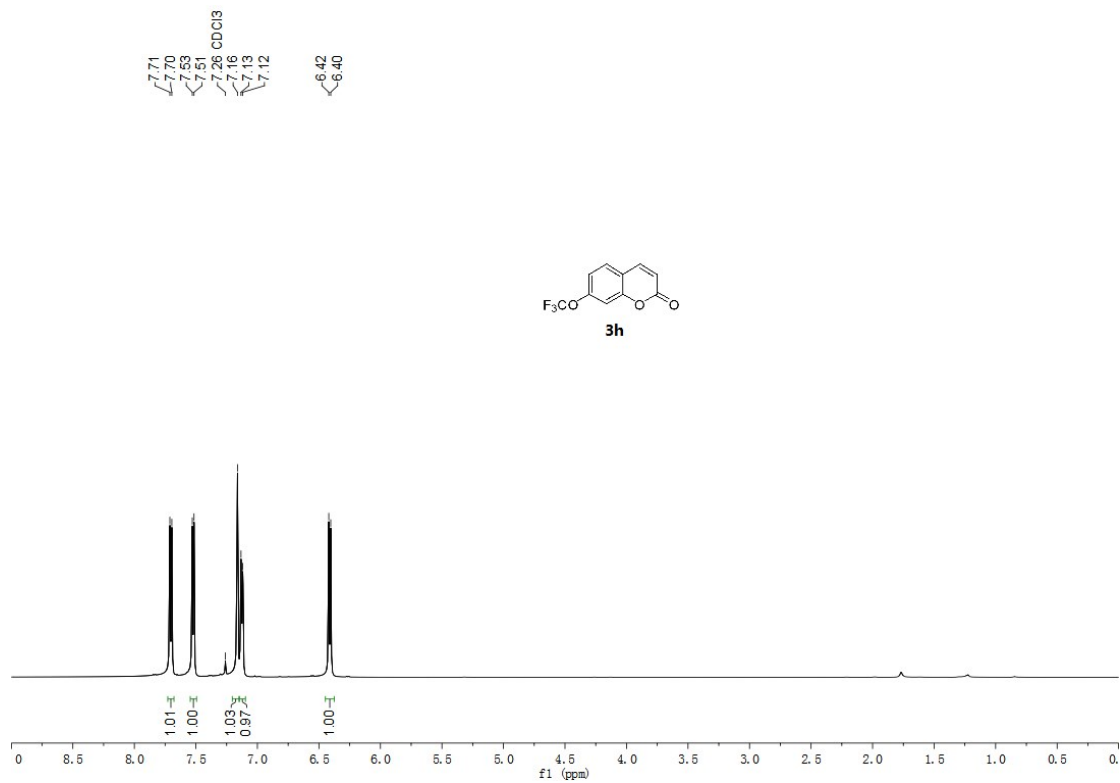
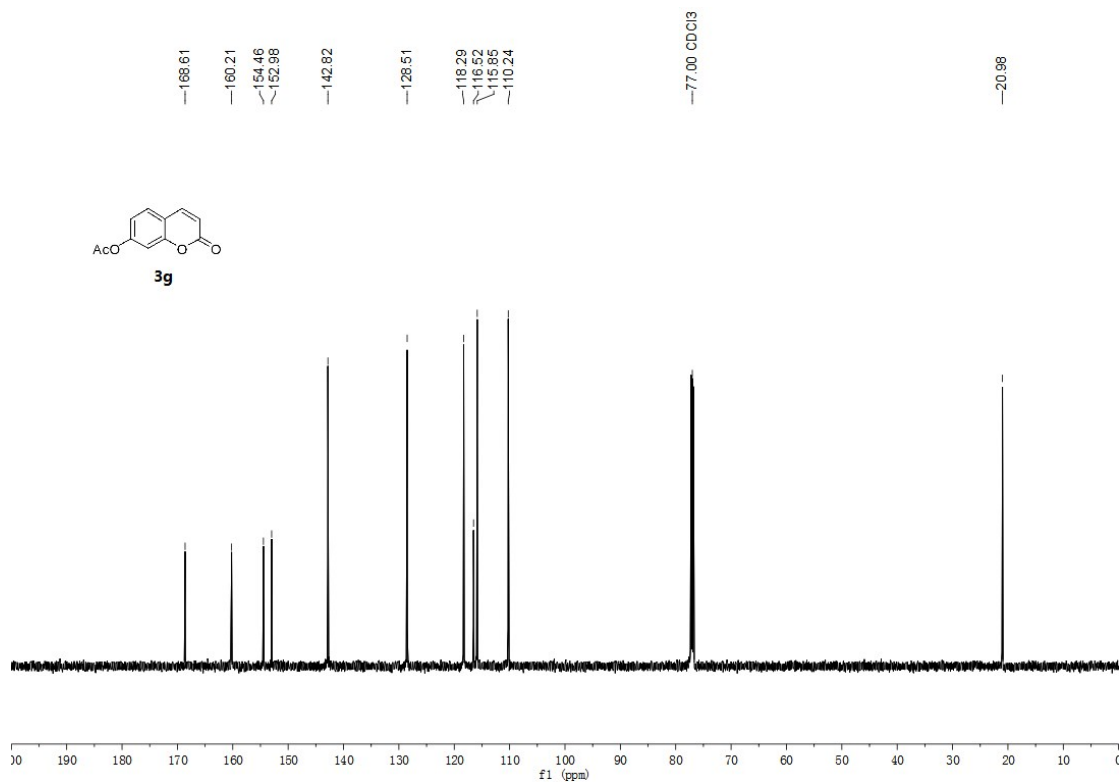


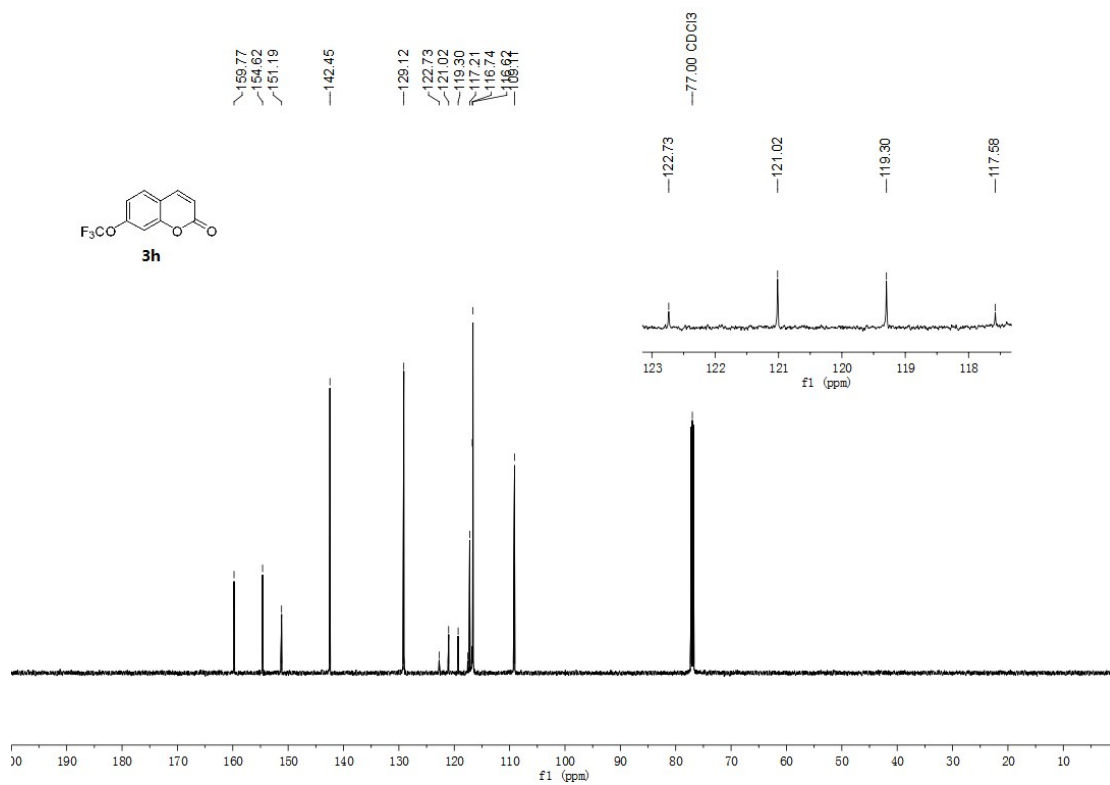
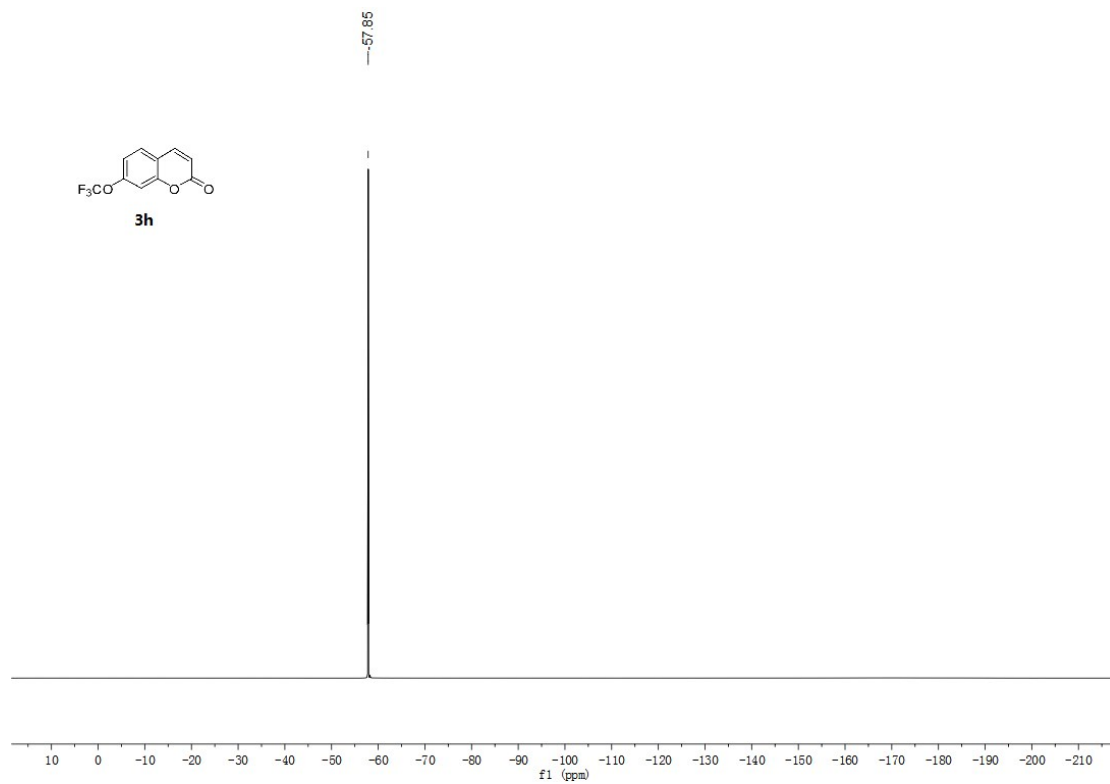


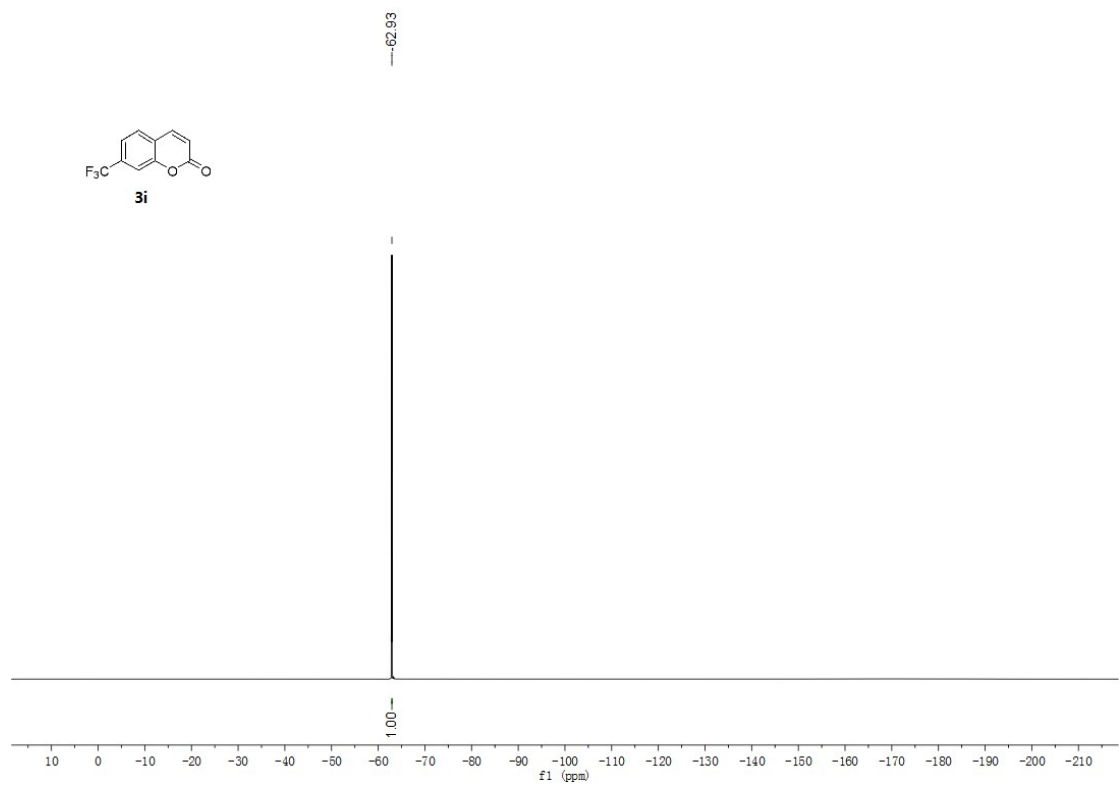
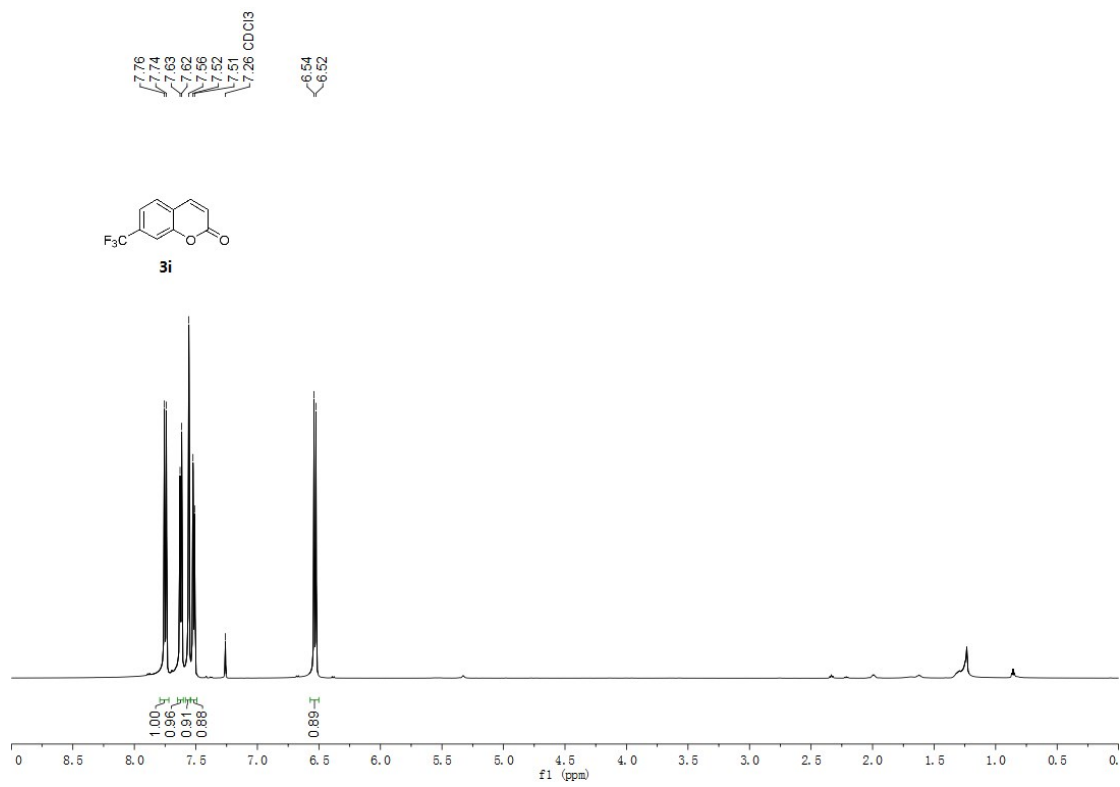


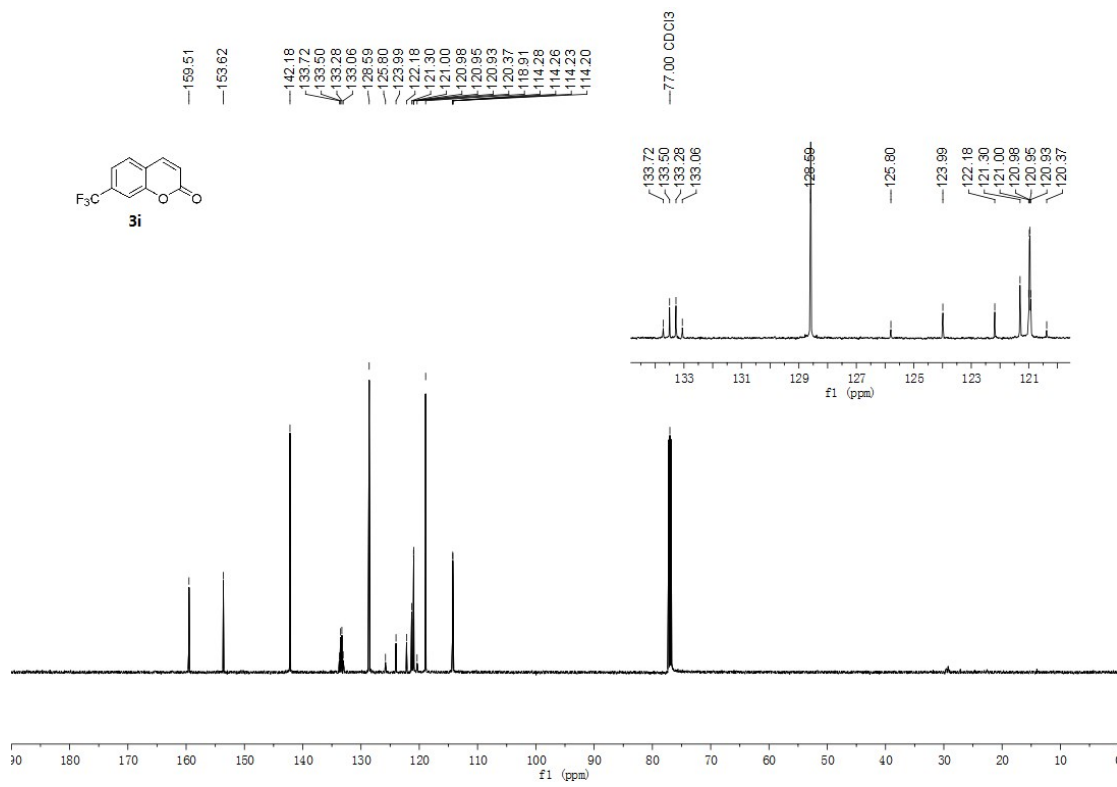


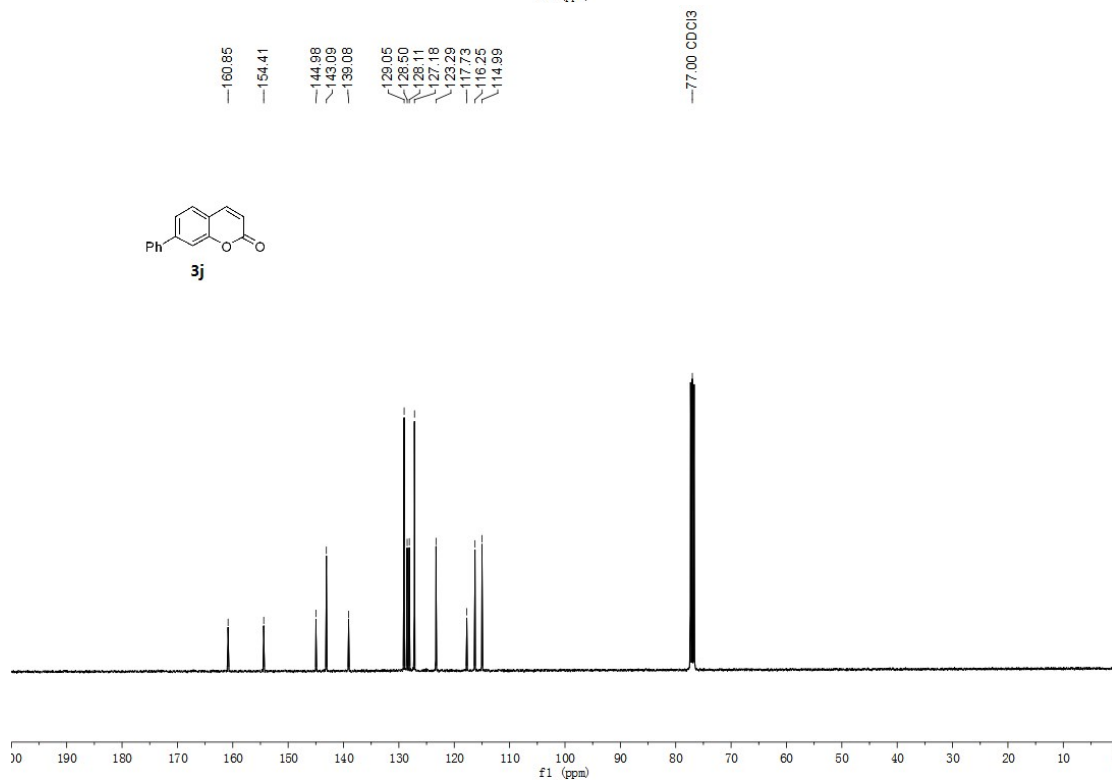
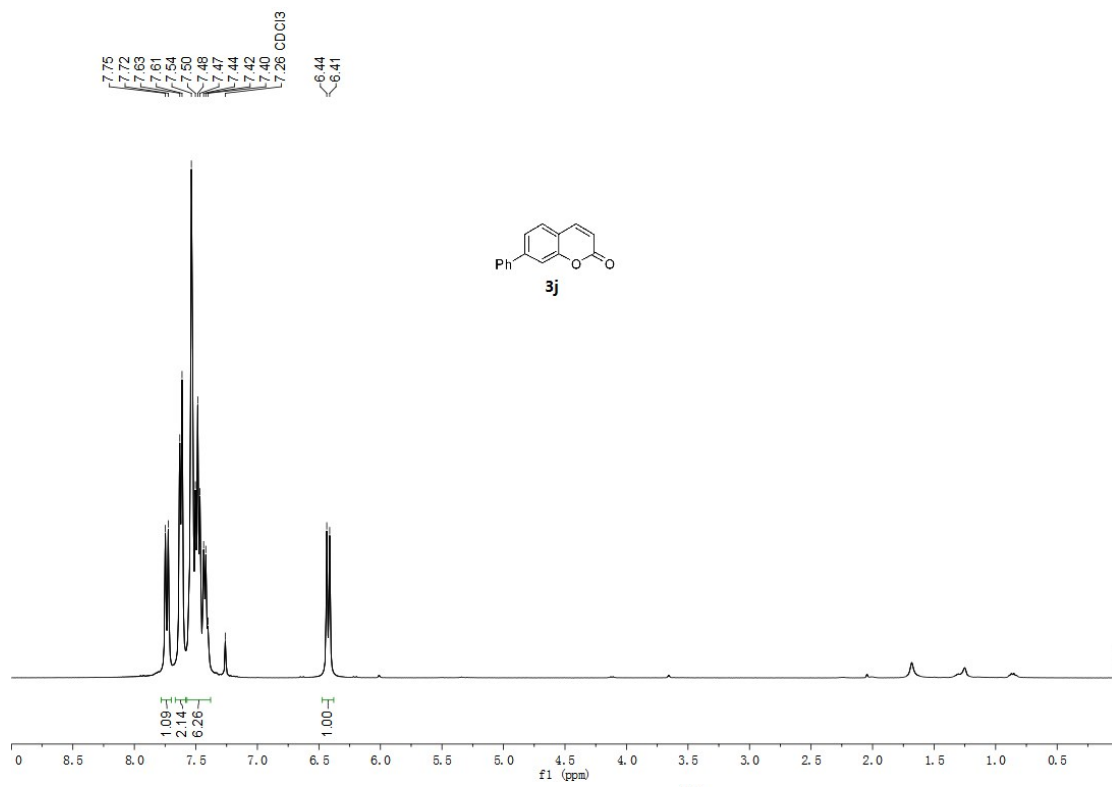


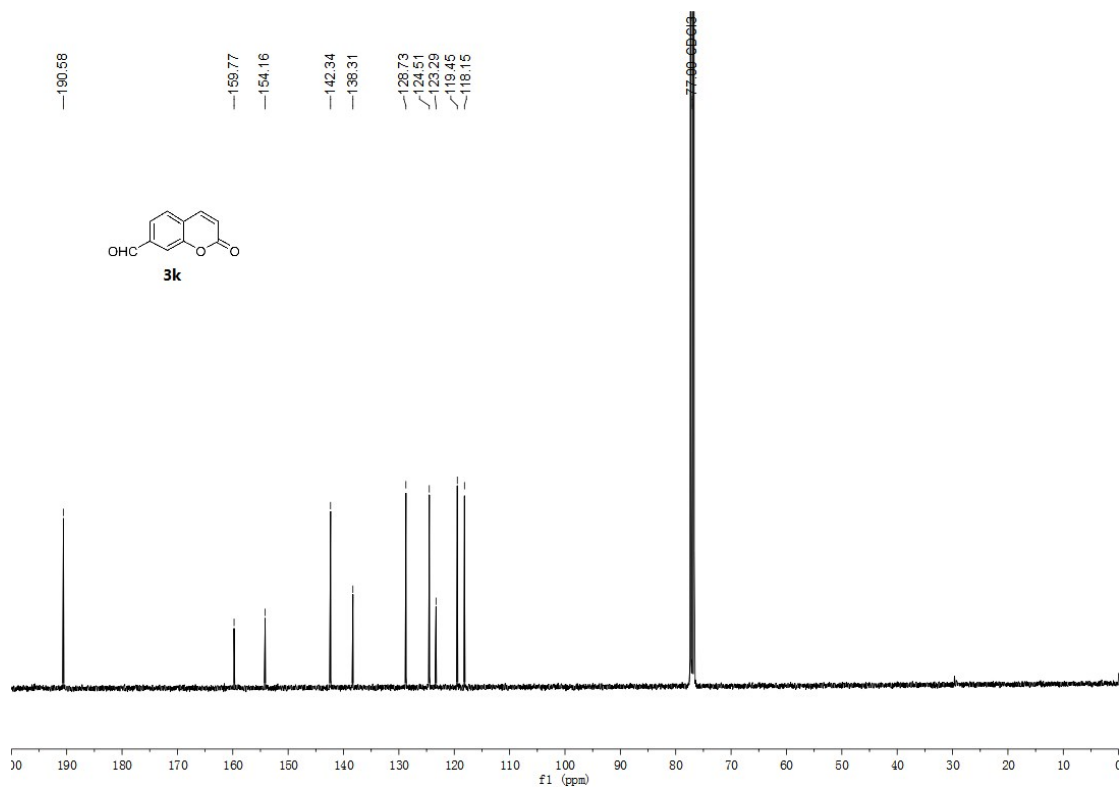
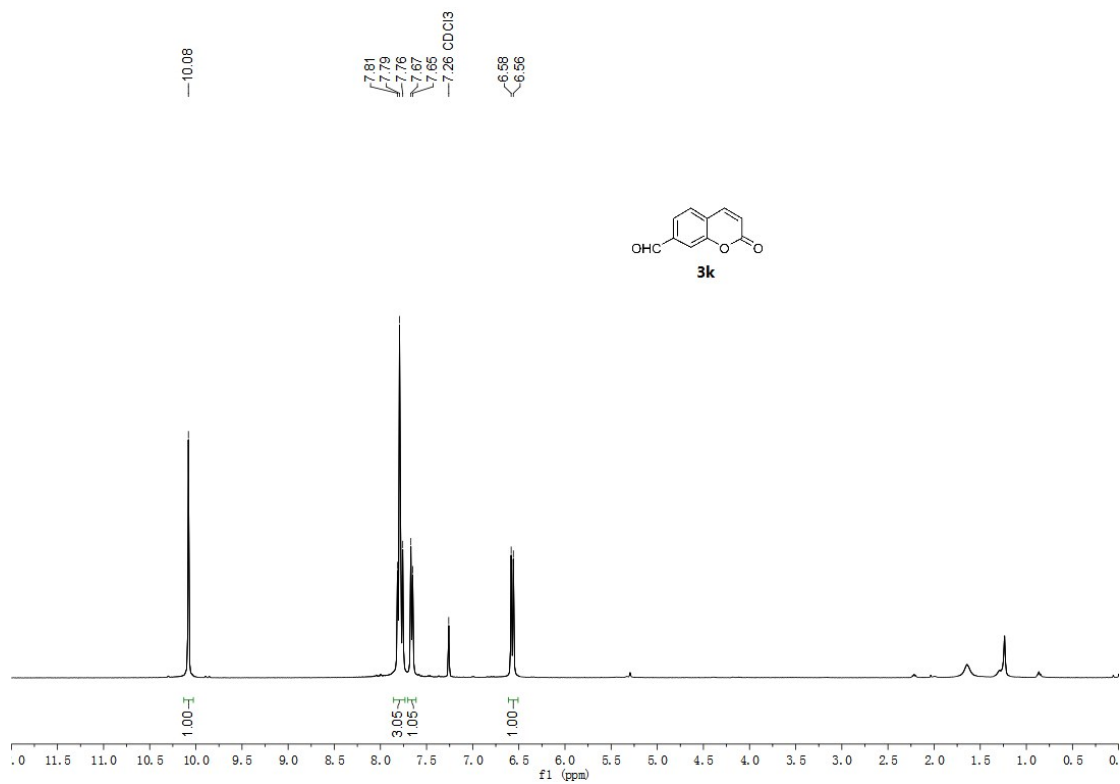


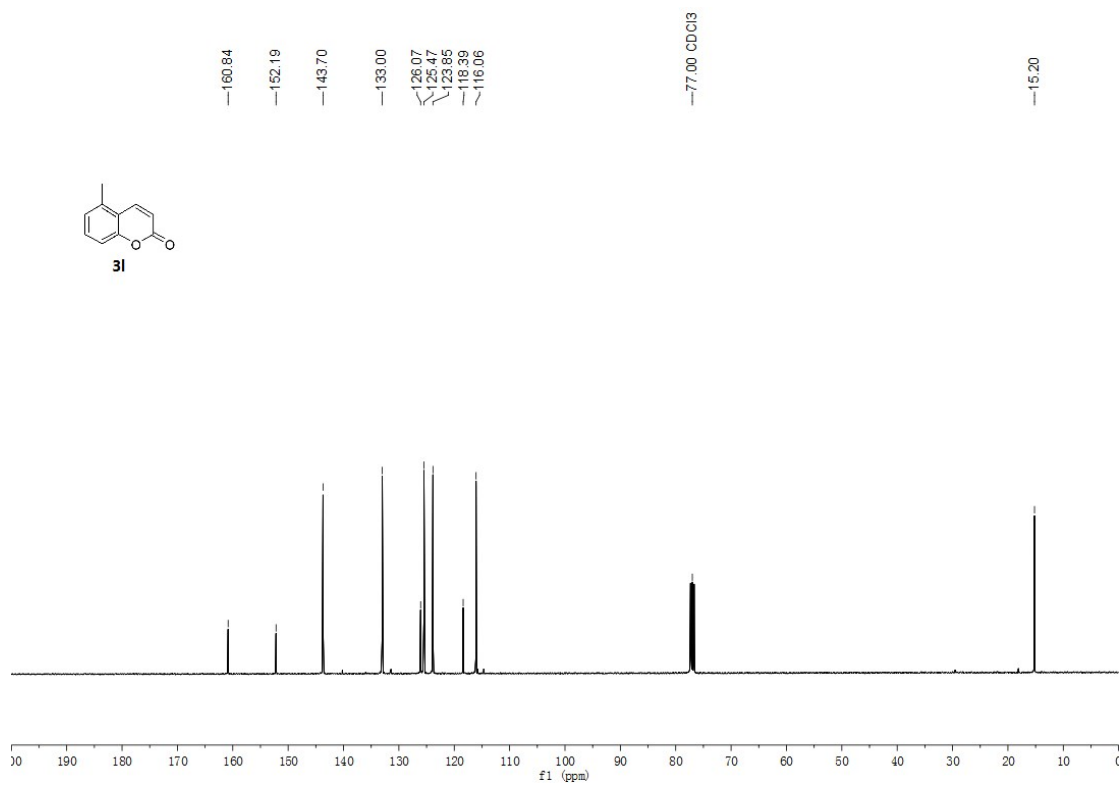
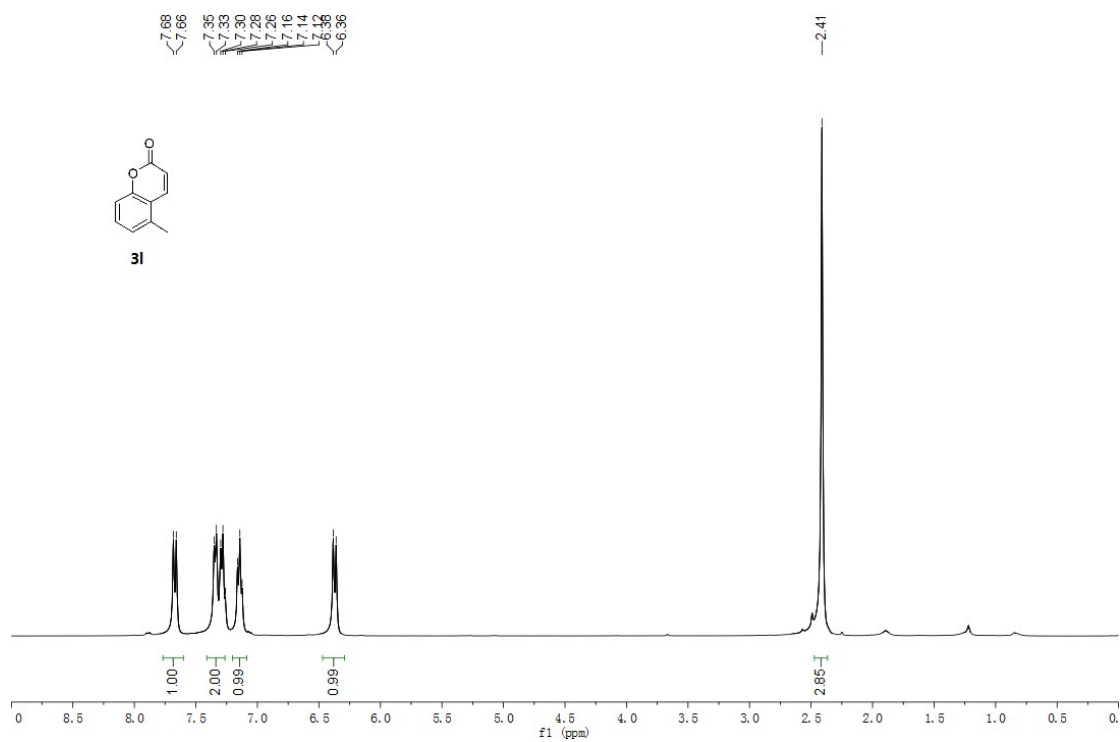


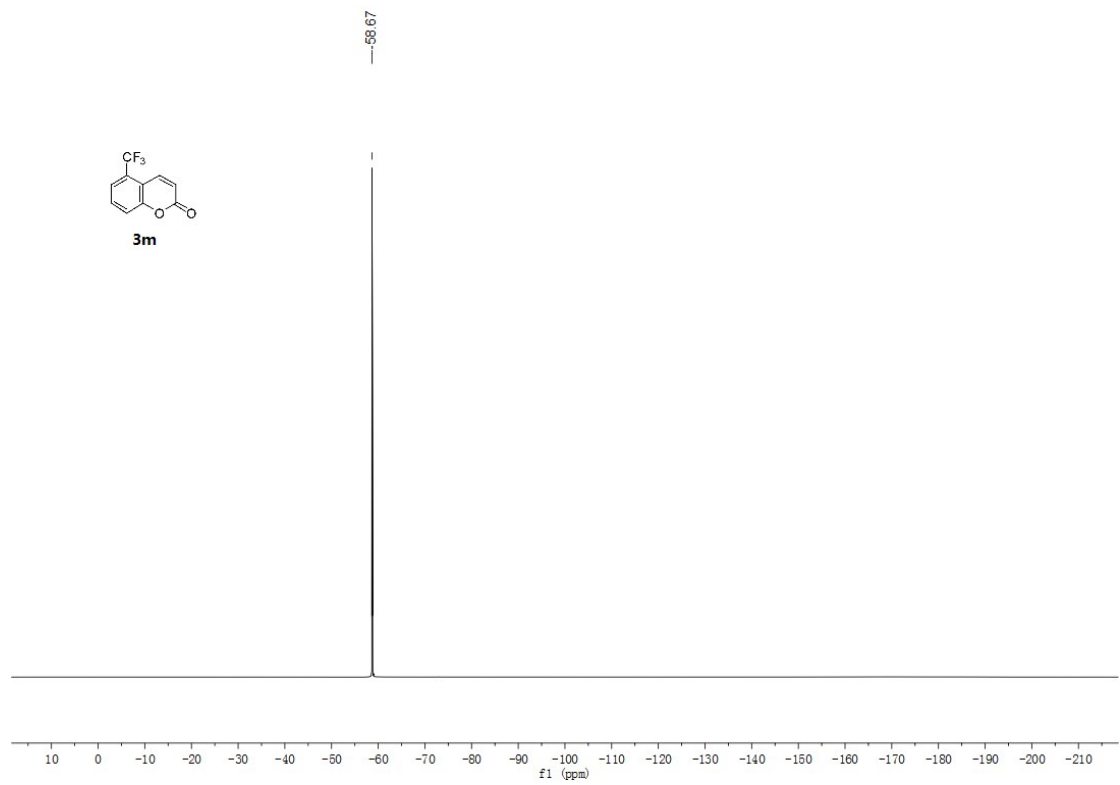
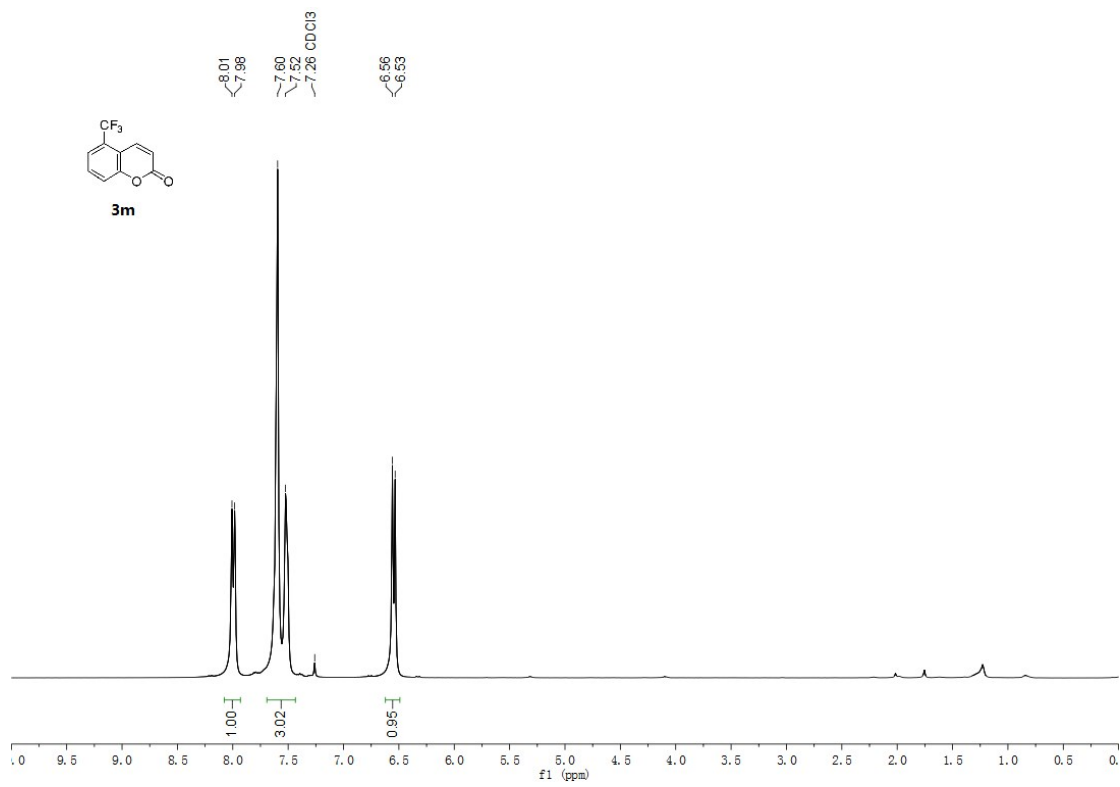


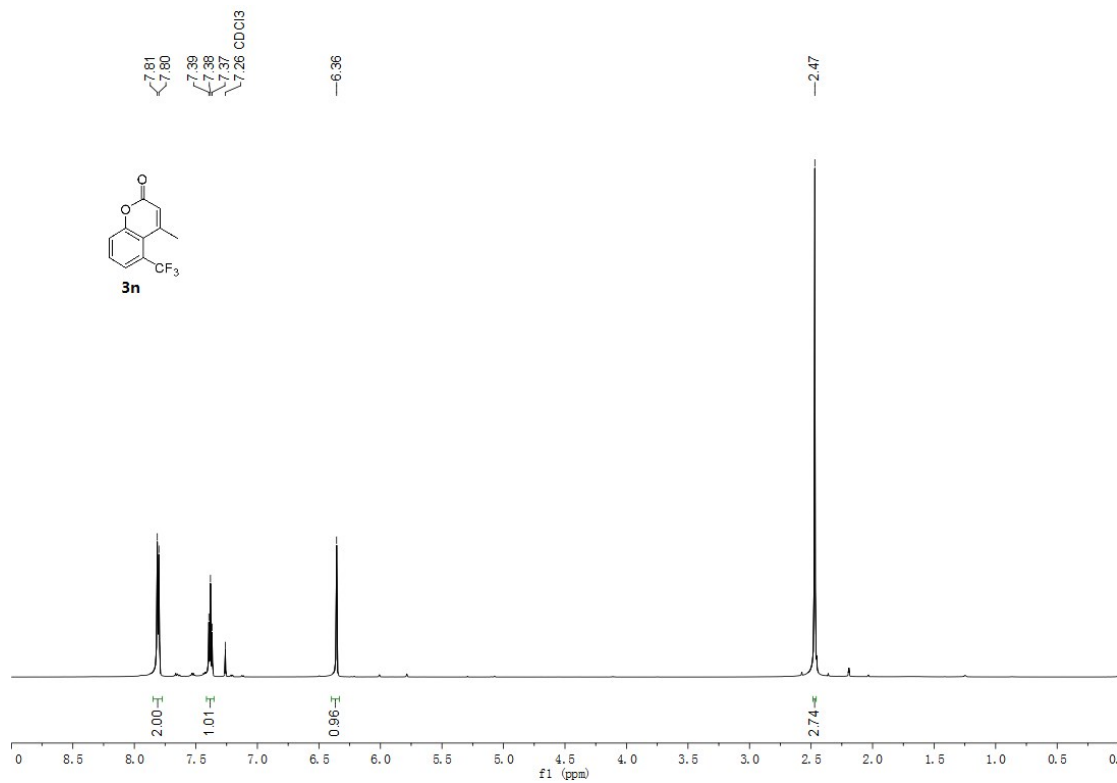
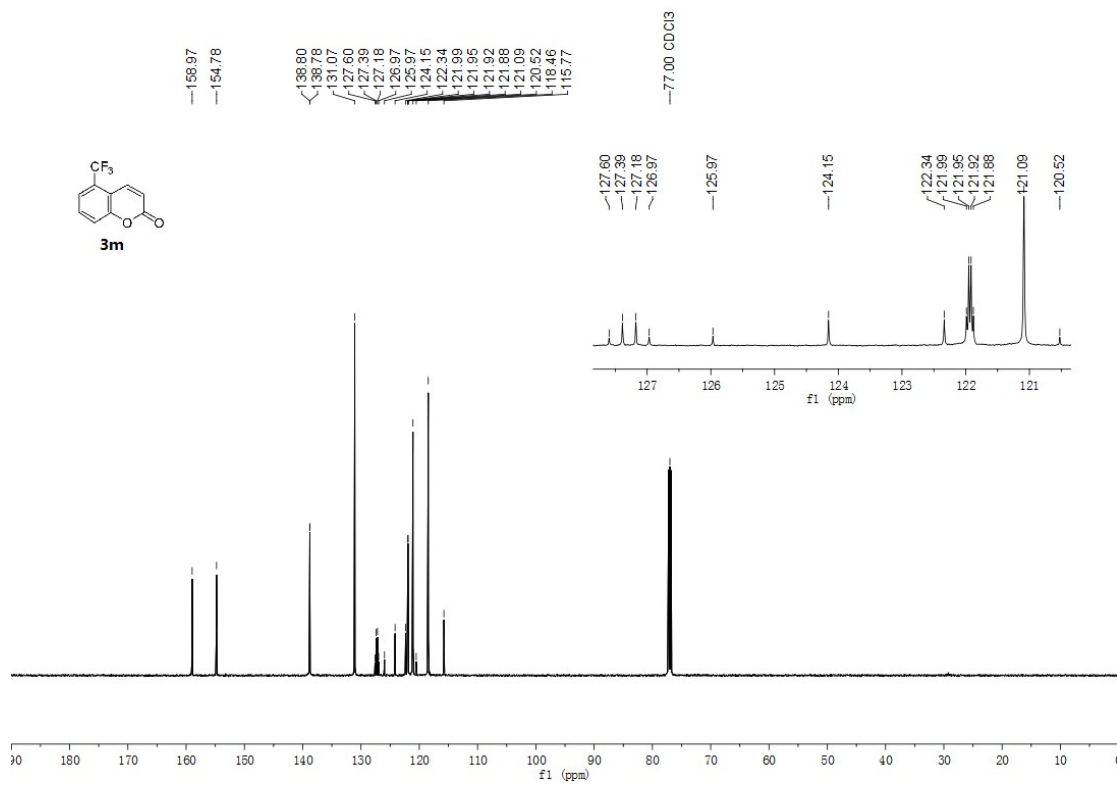


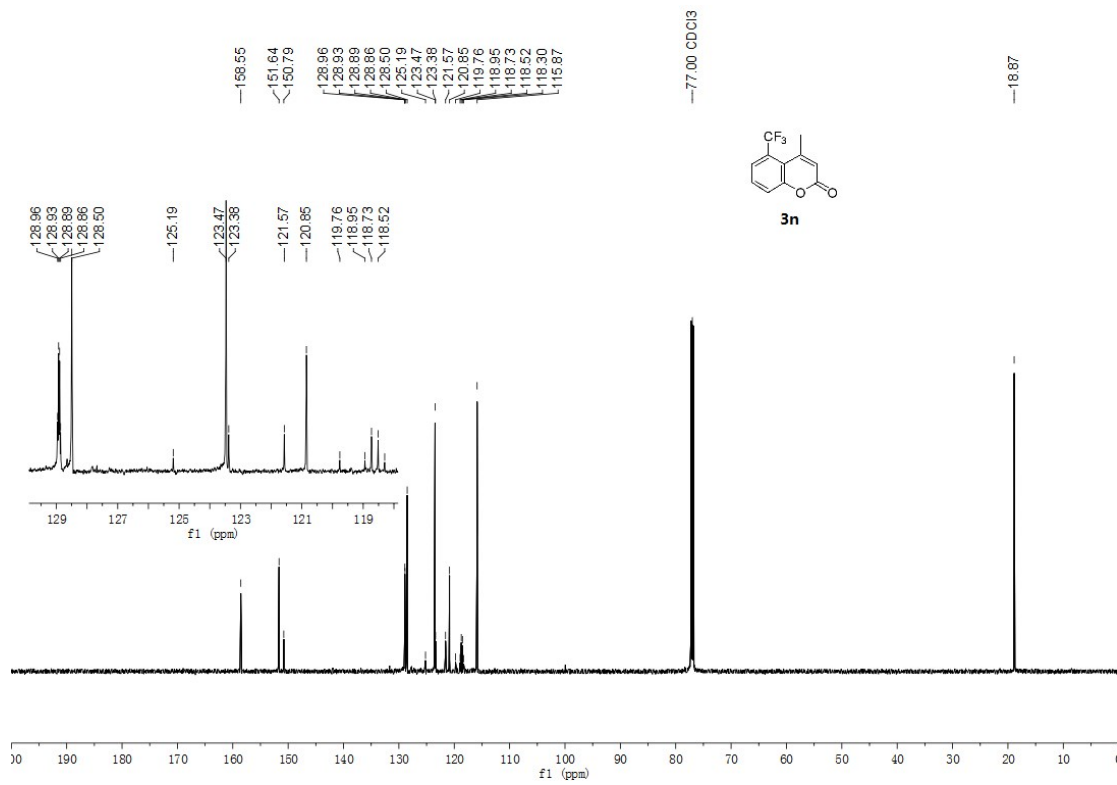
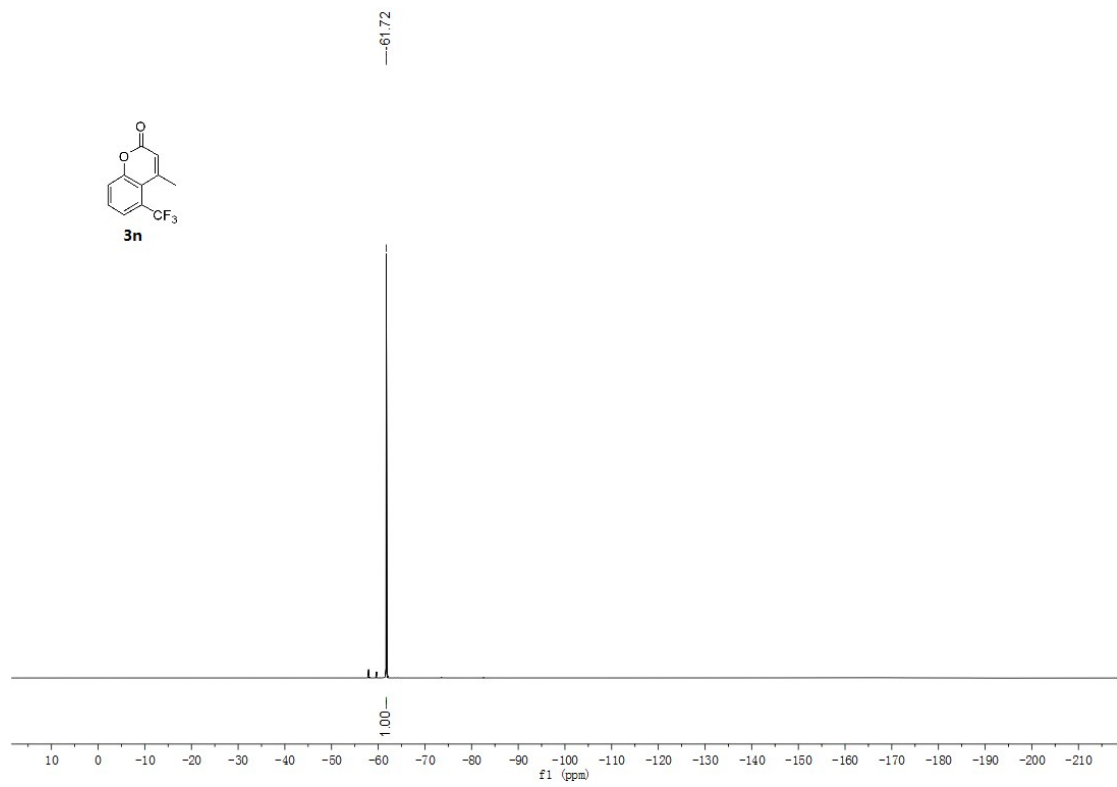


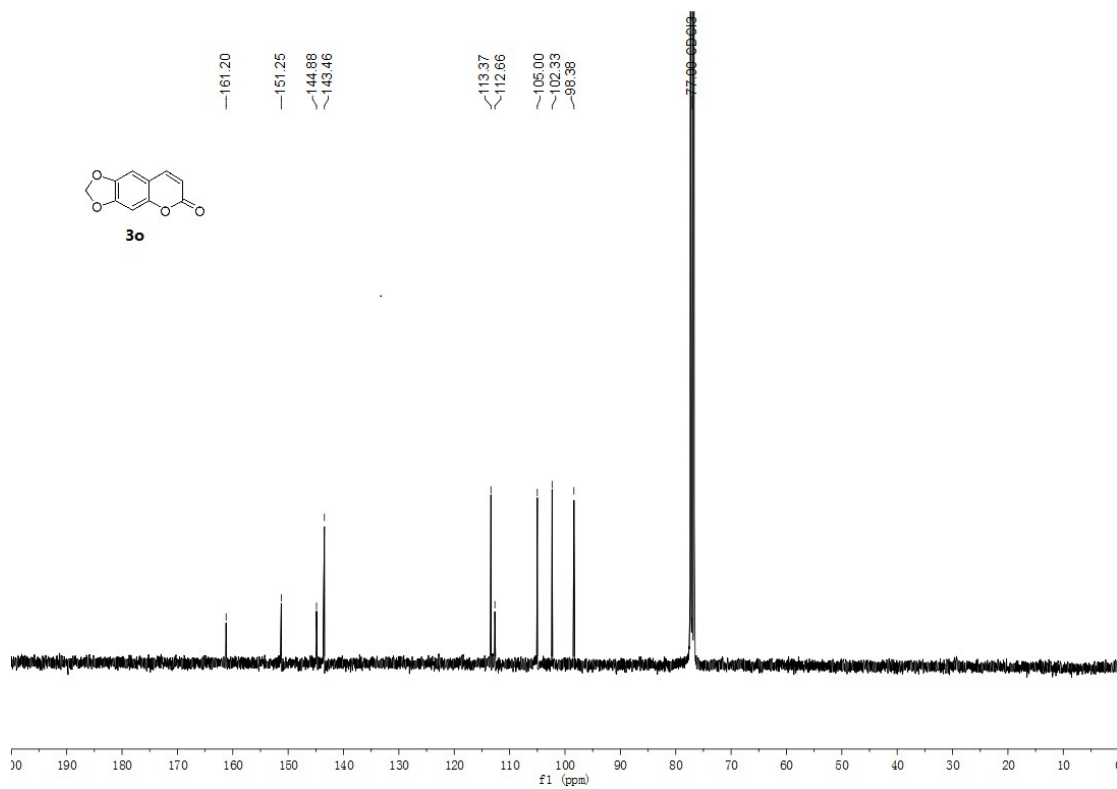
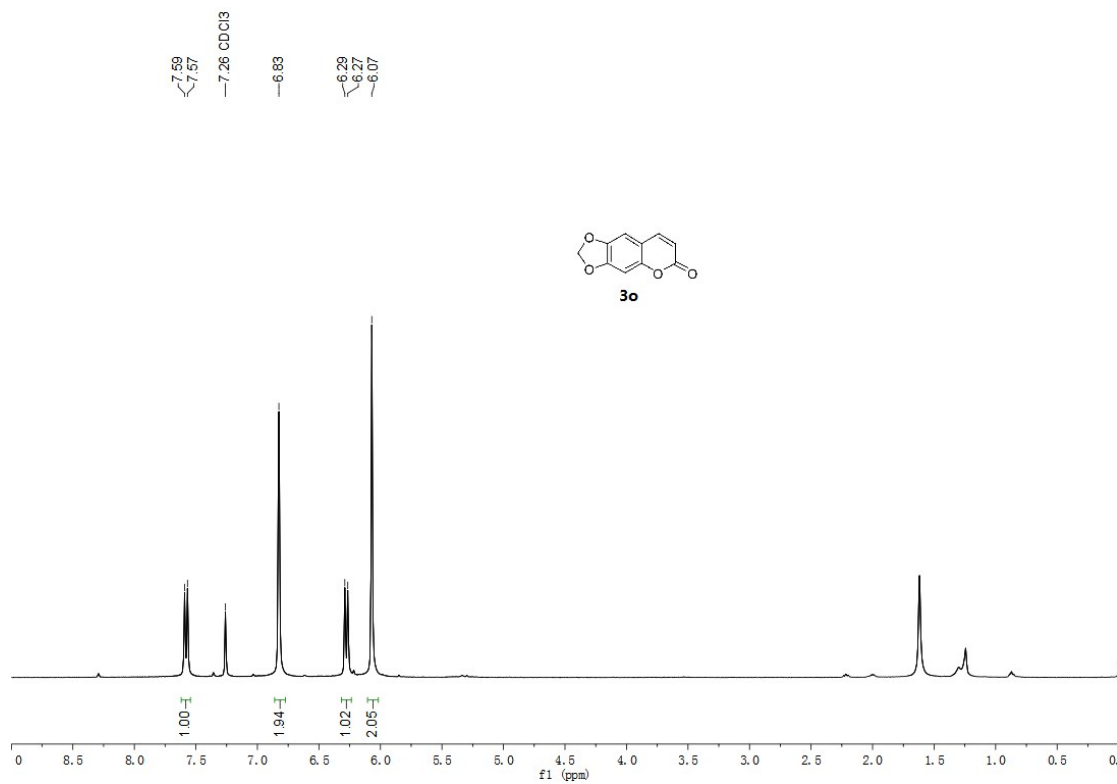


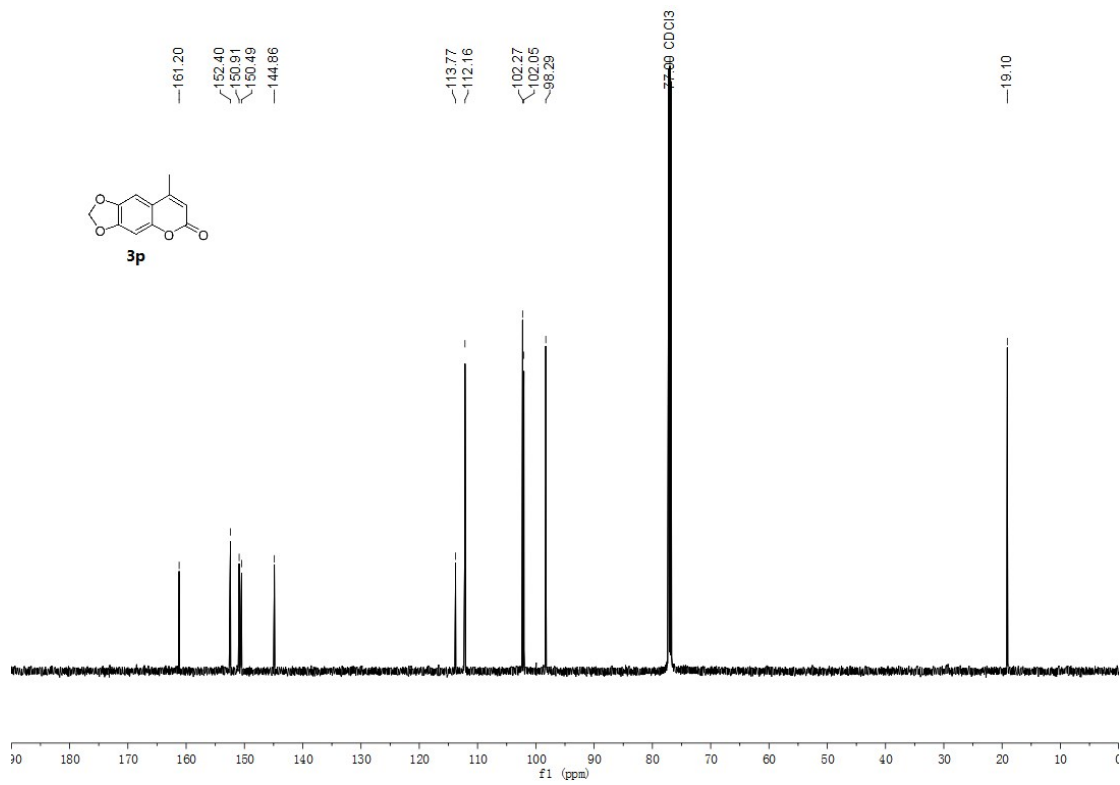
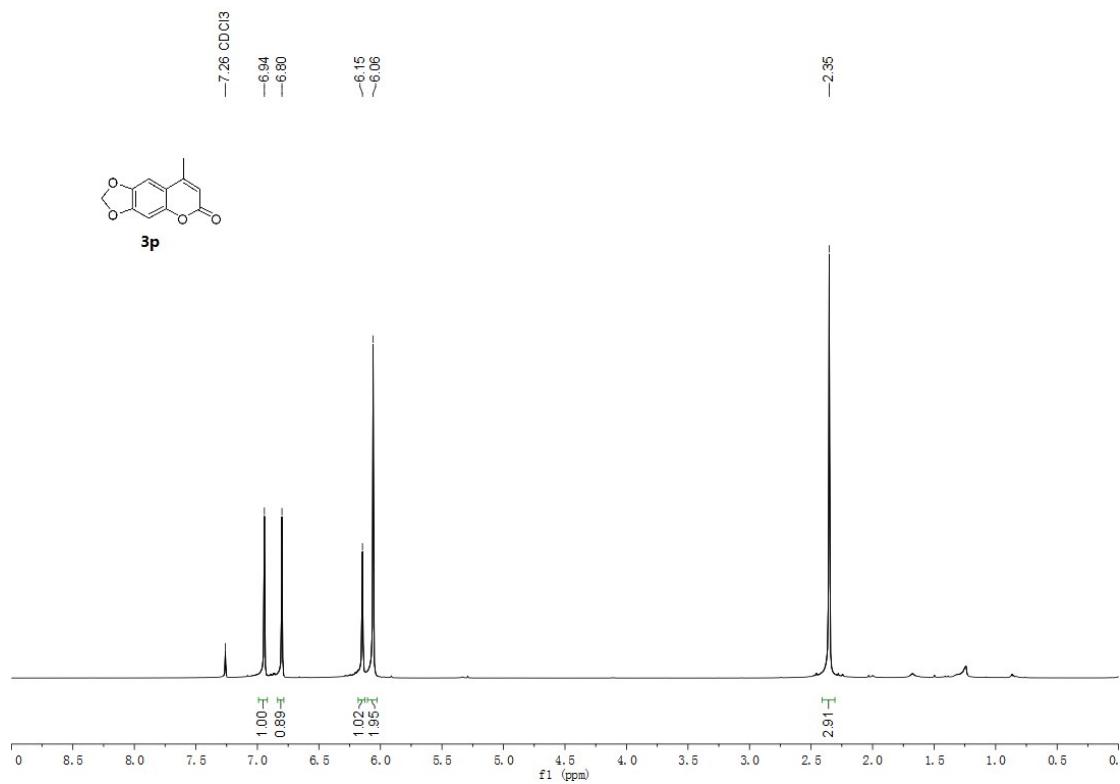


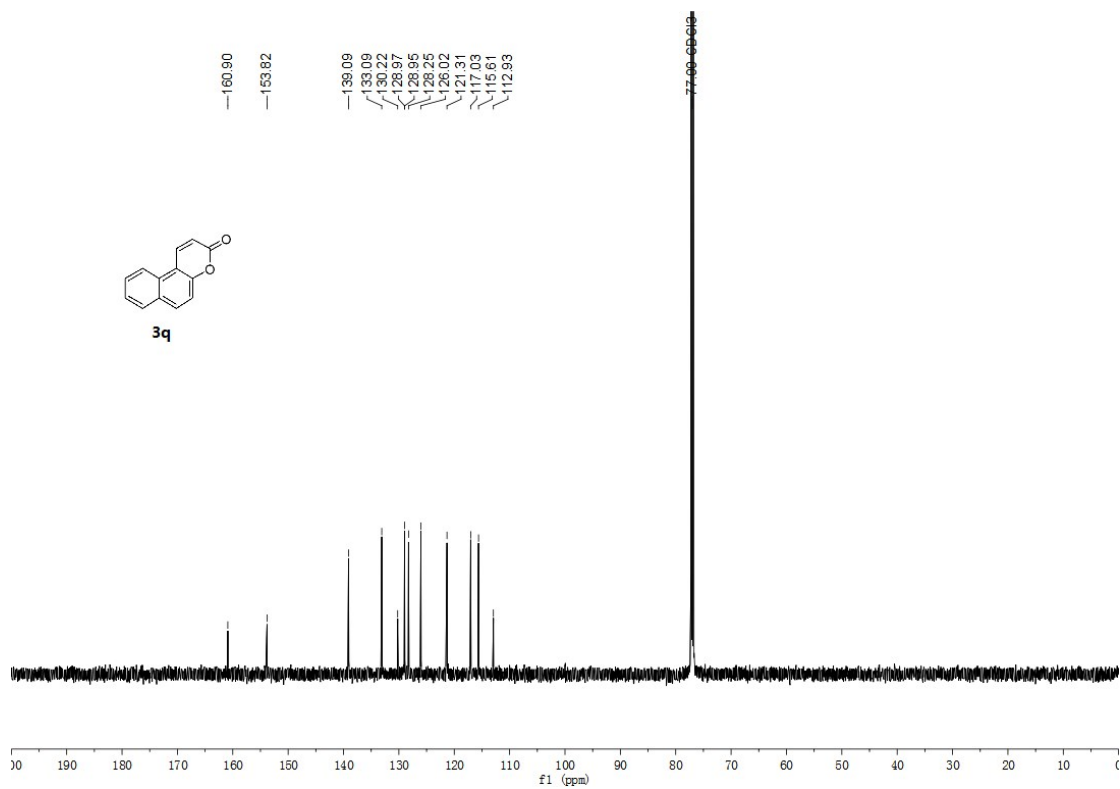
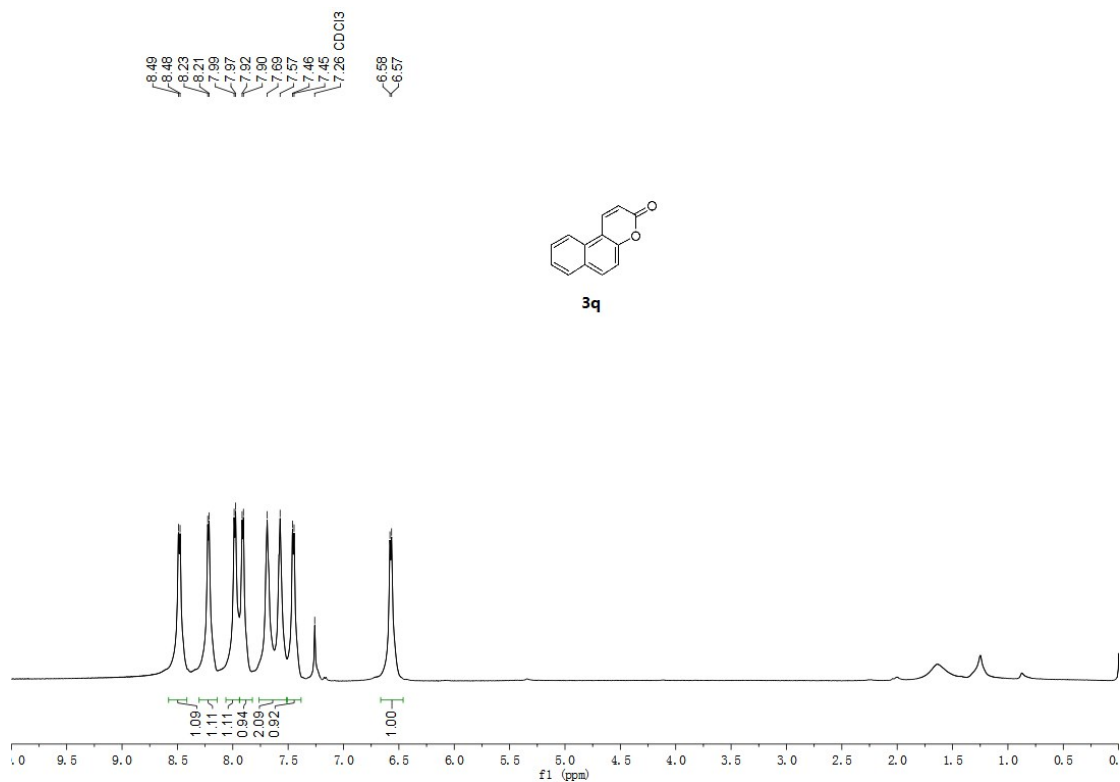


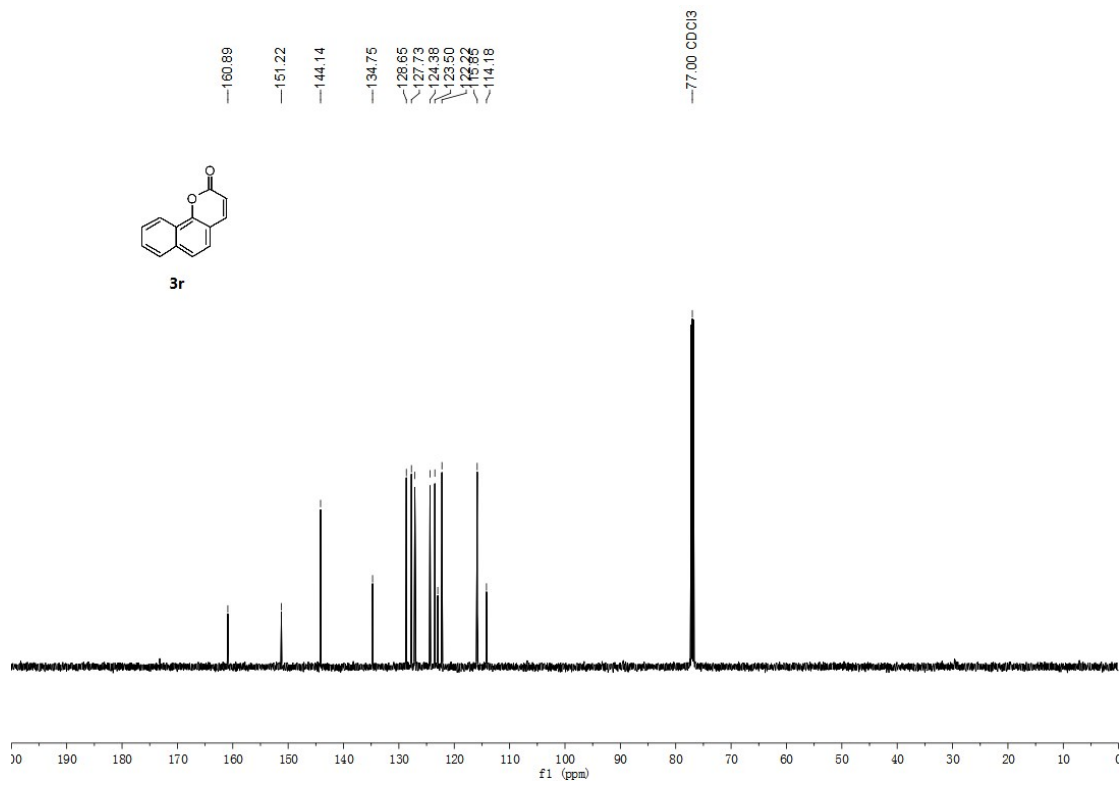
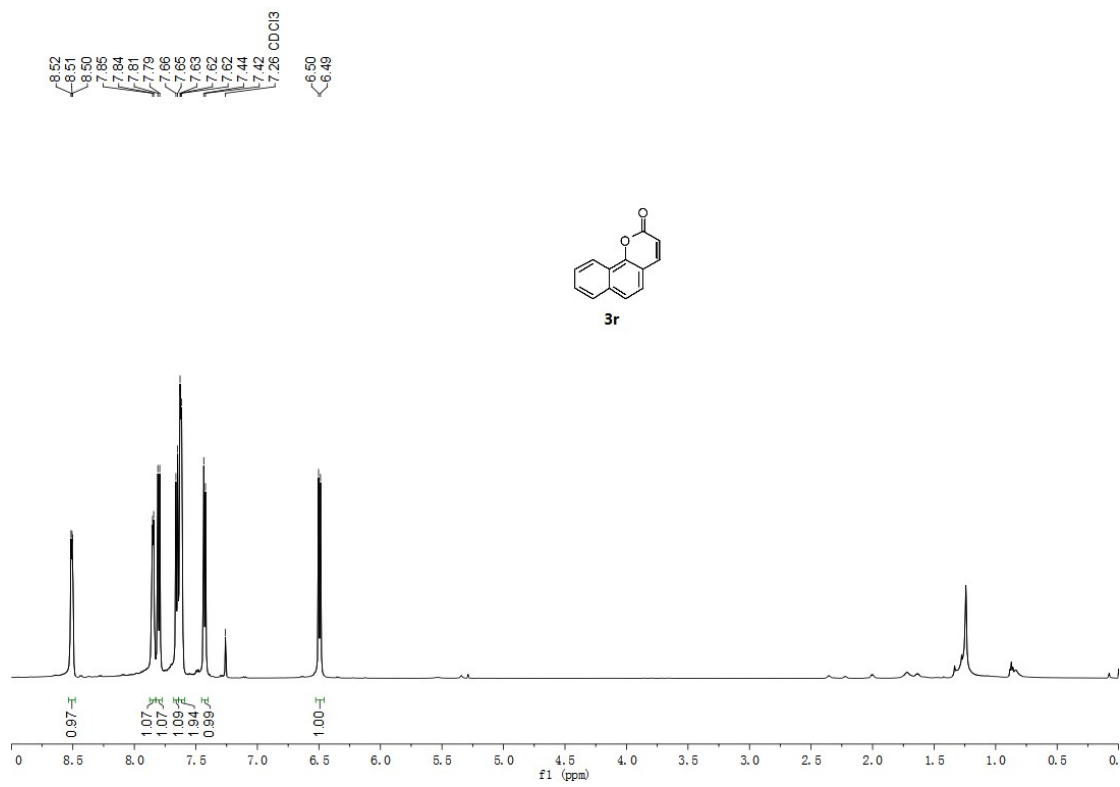


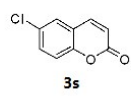




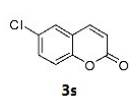
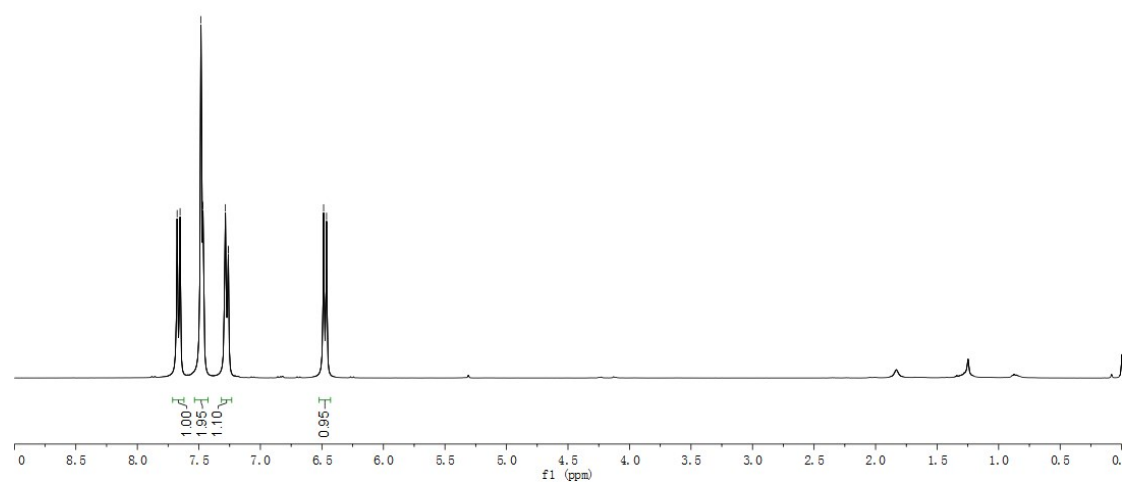




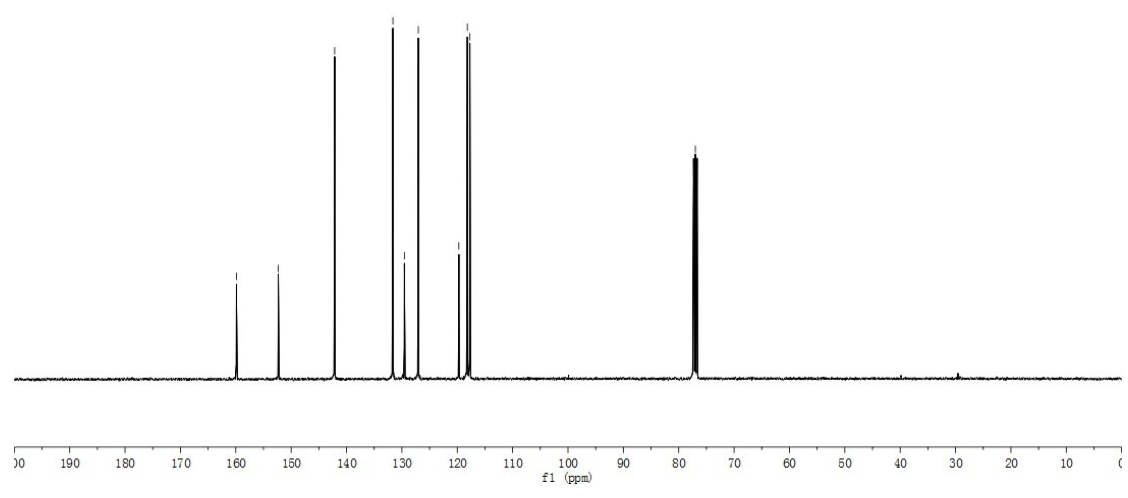


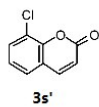
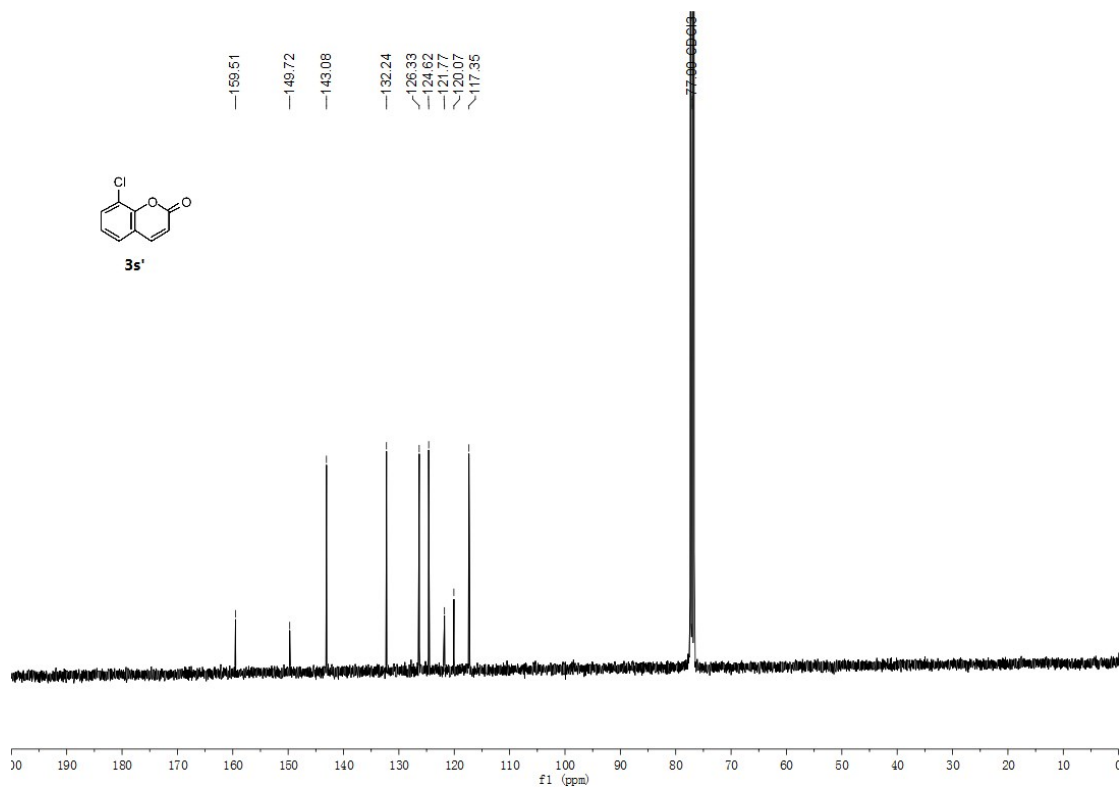
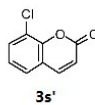
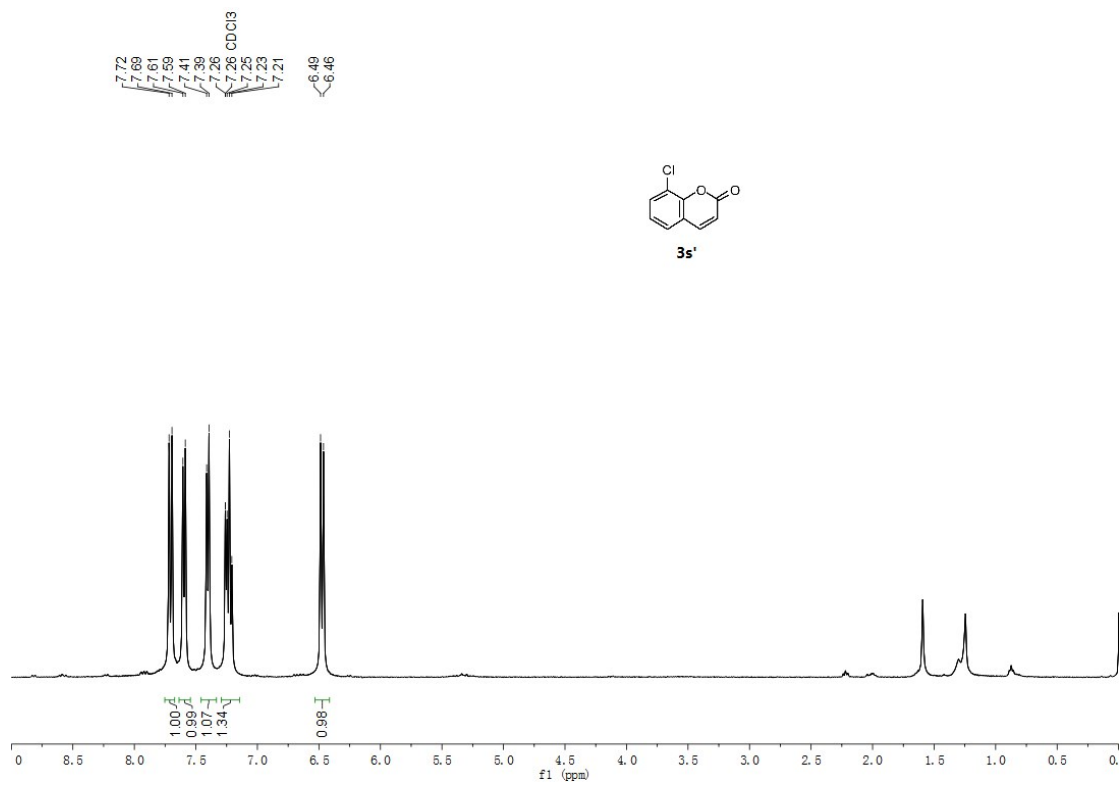


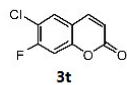
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 6.49
 6.46



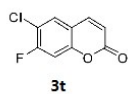
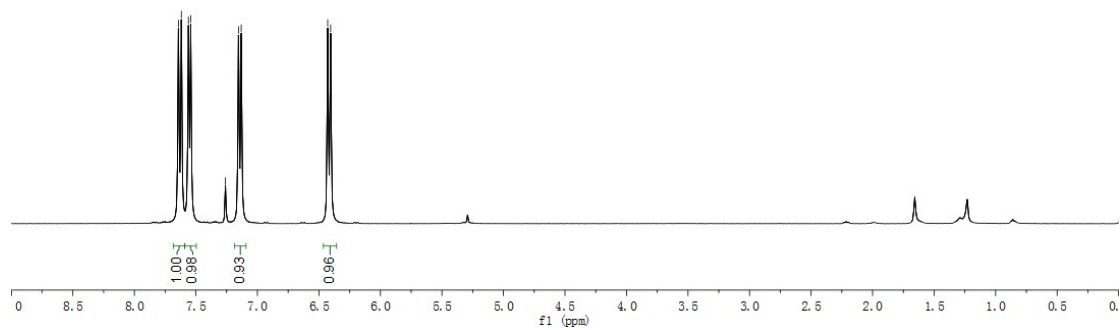
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 117.72
 77.00 CDCl3





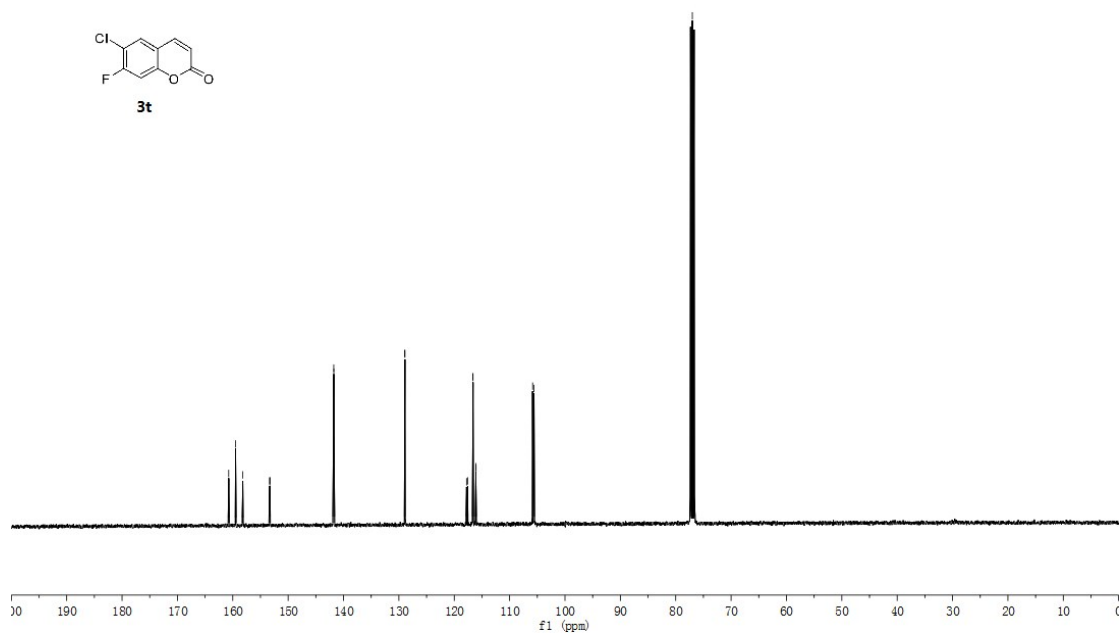


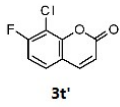
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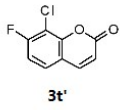
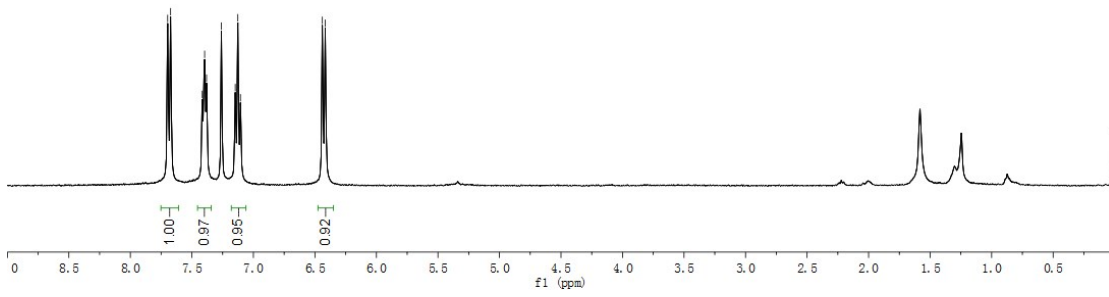
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 108.59

77.00 CDCl₃

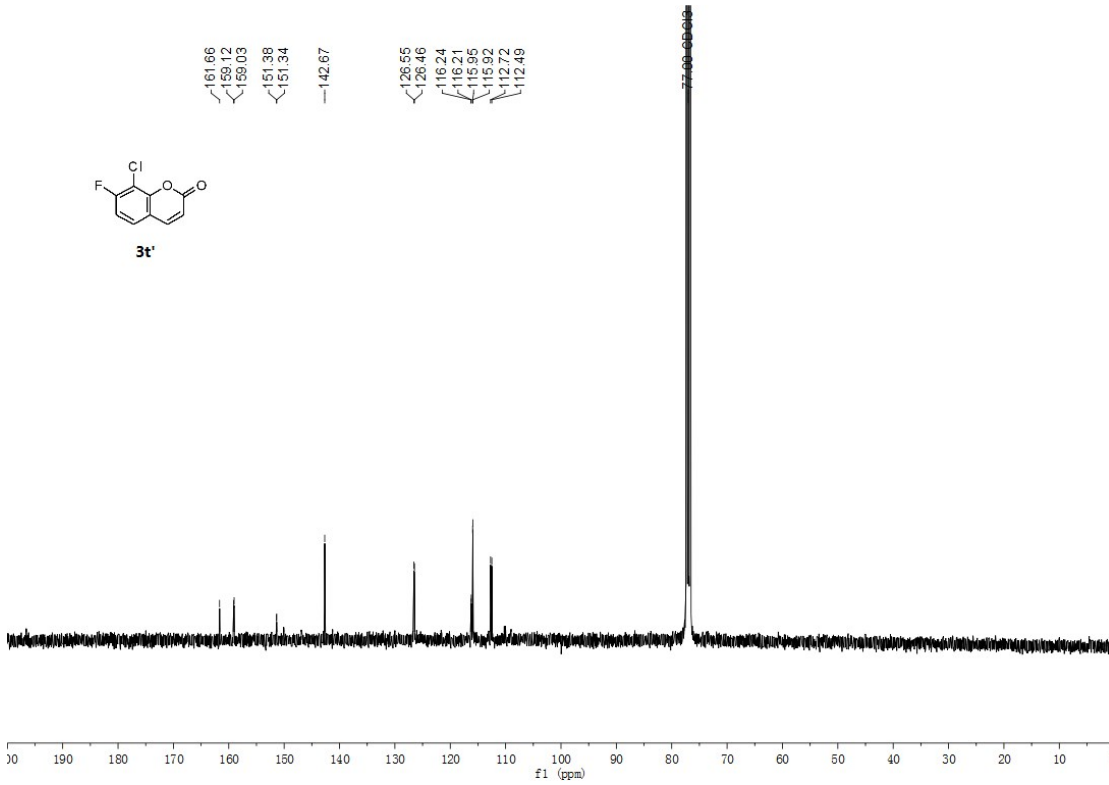


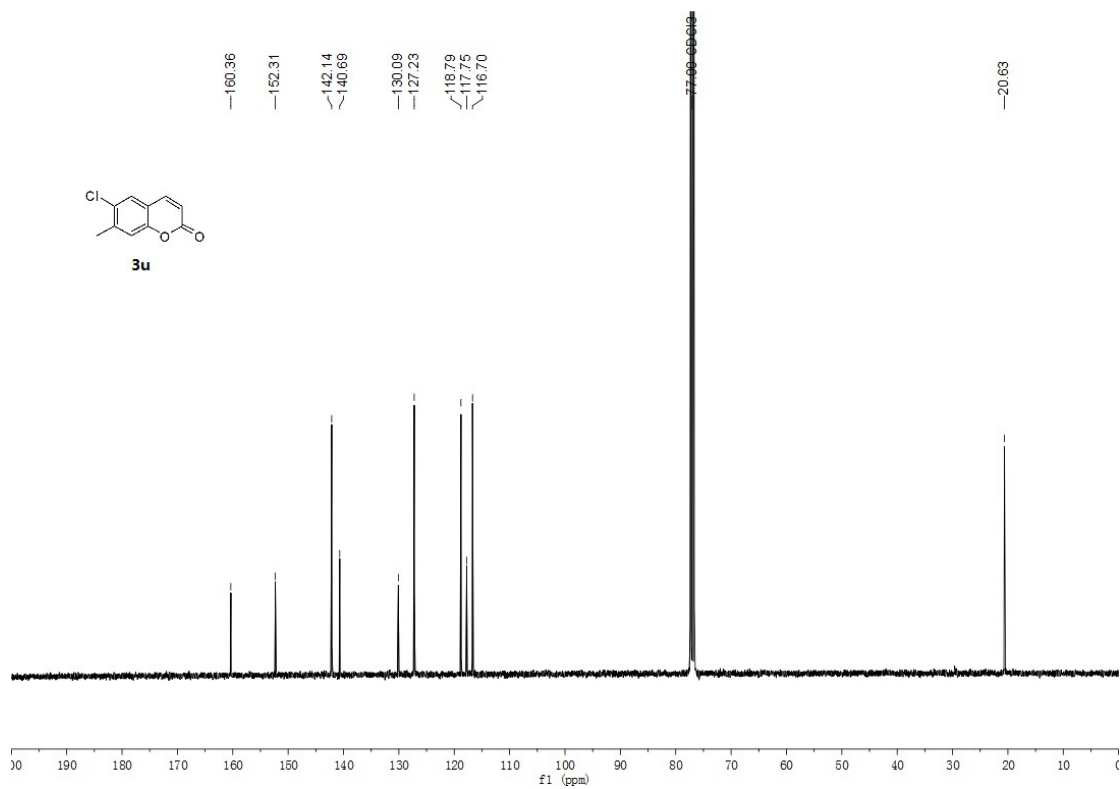
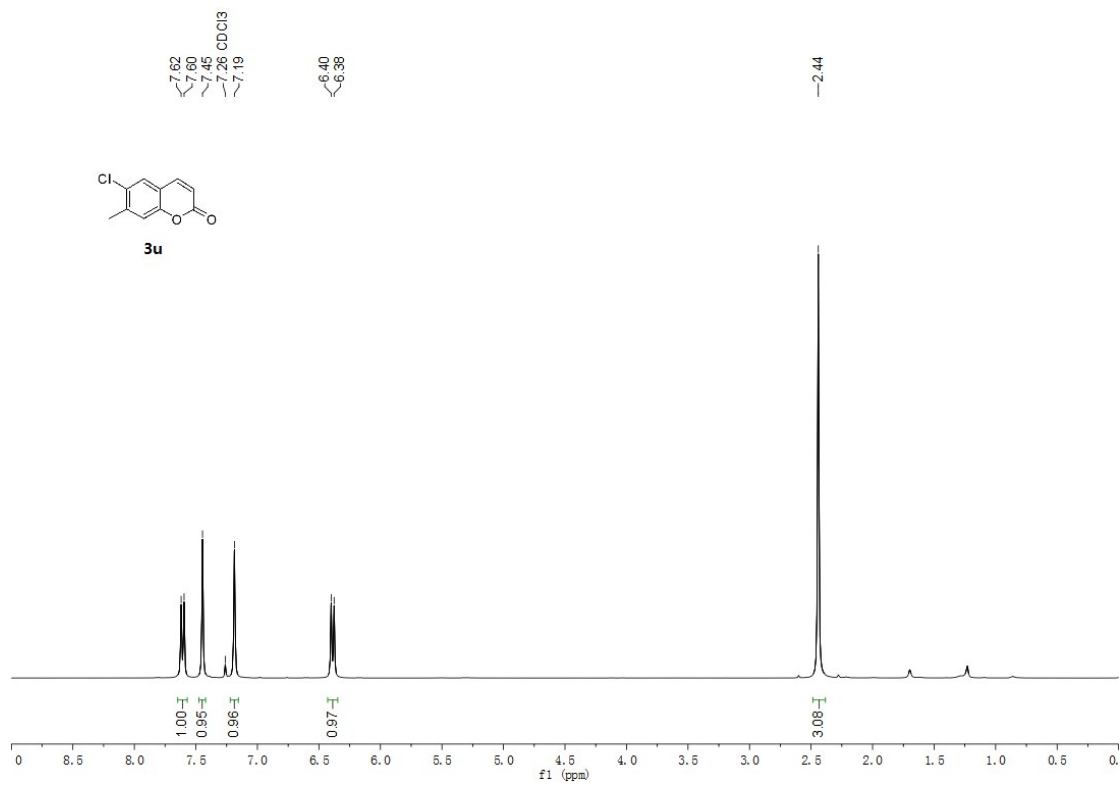


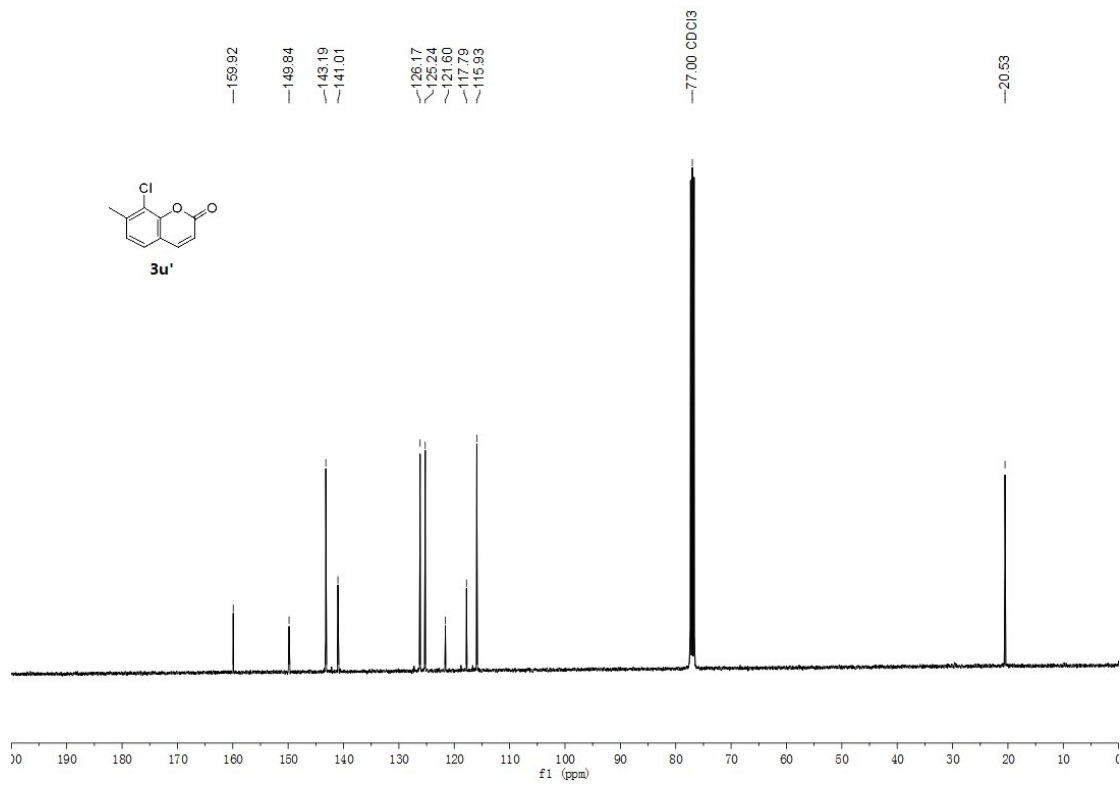
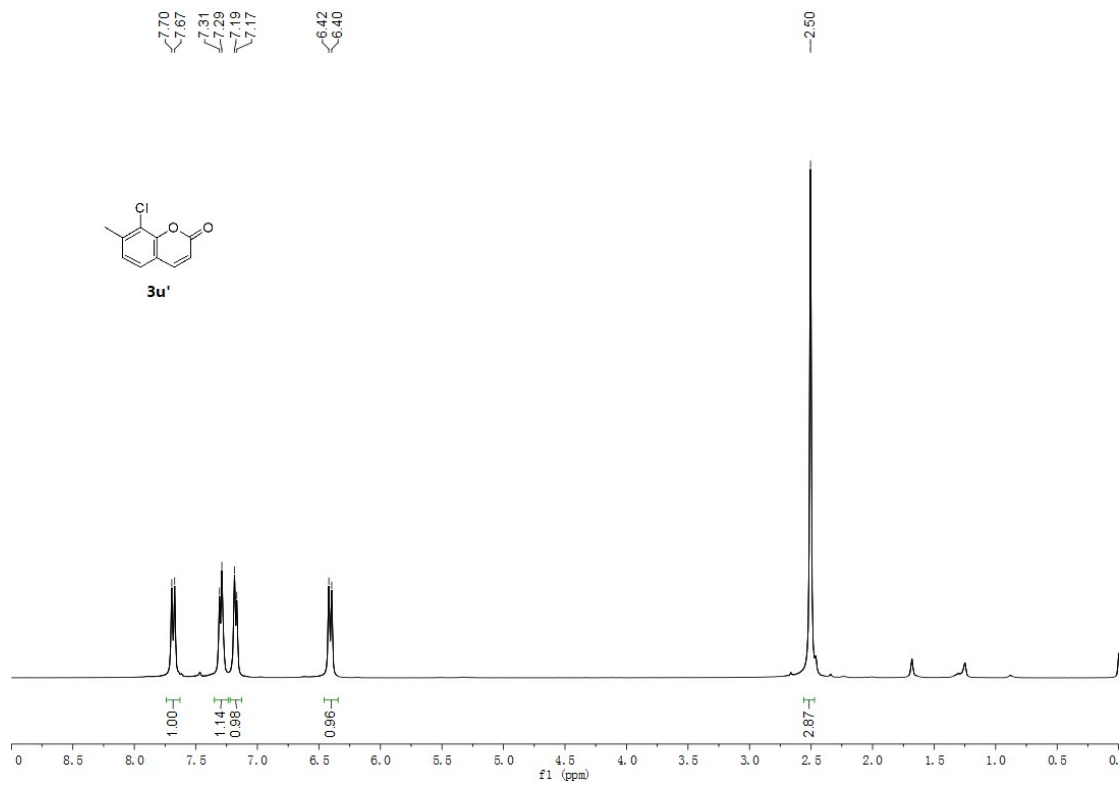
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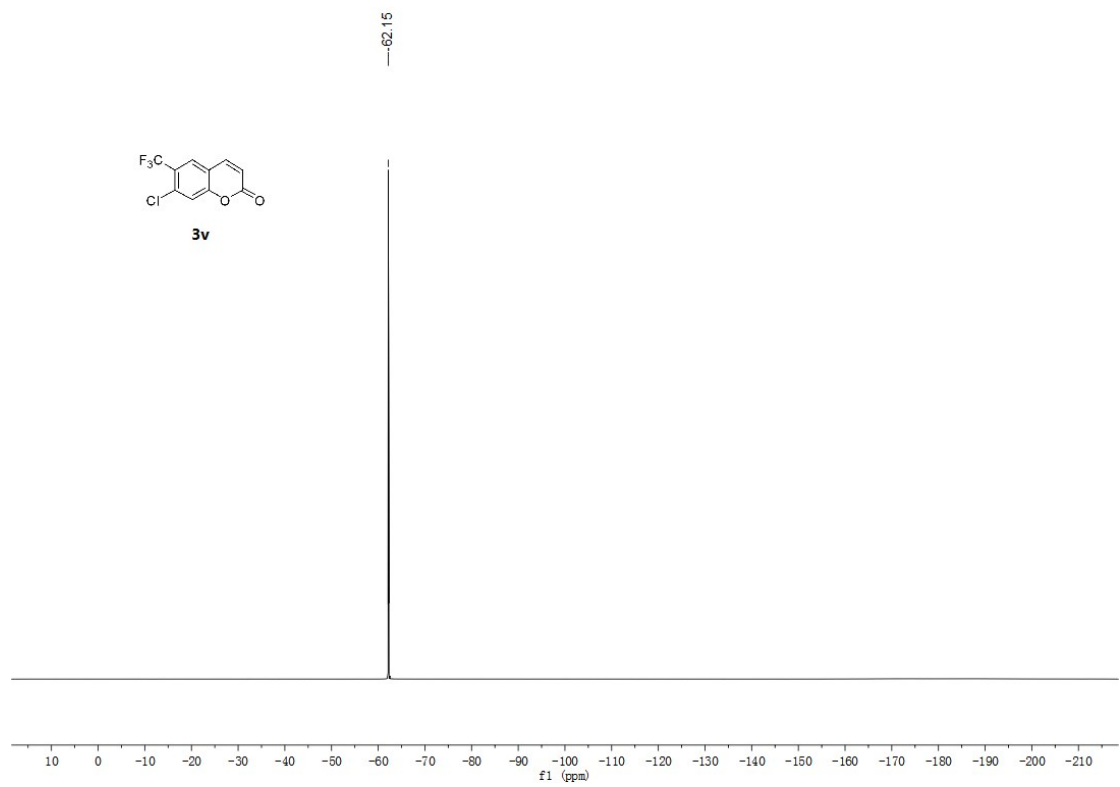
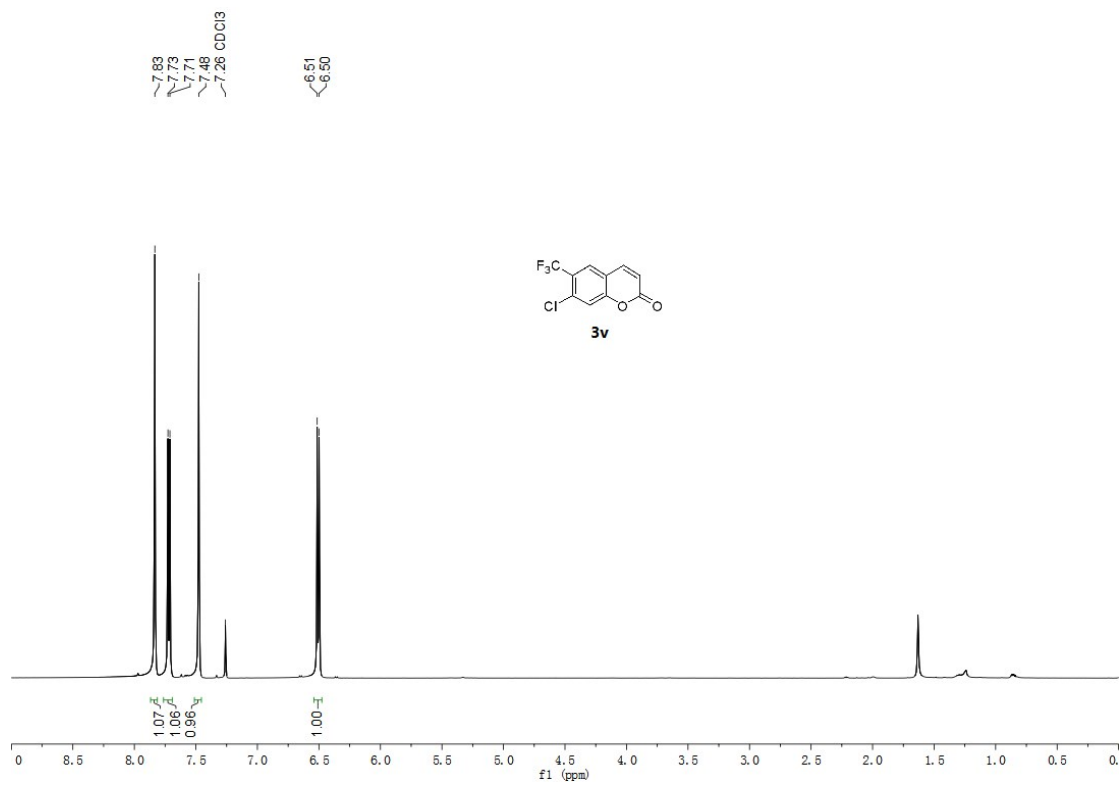


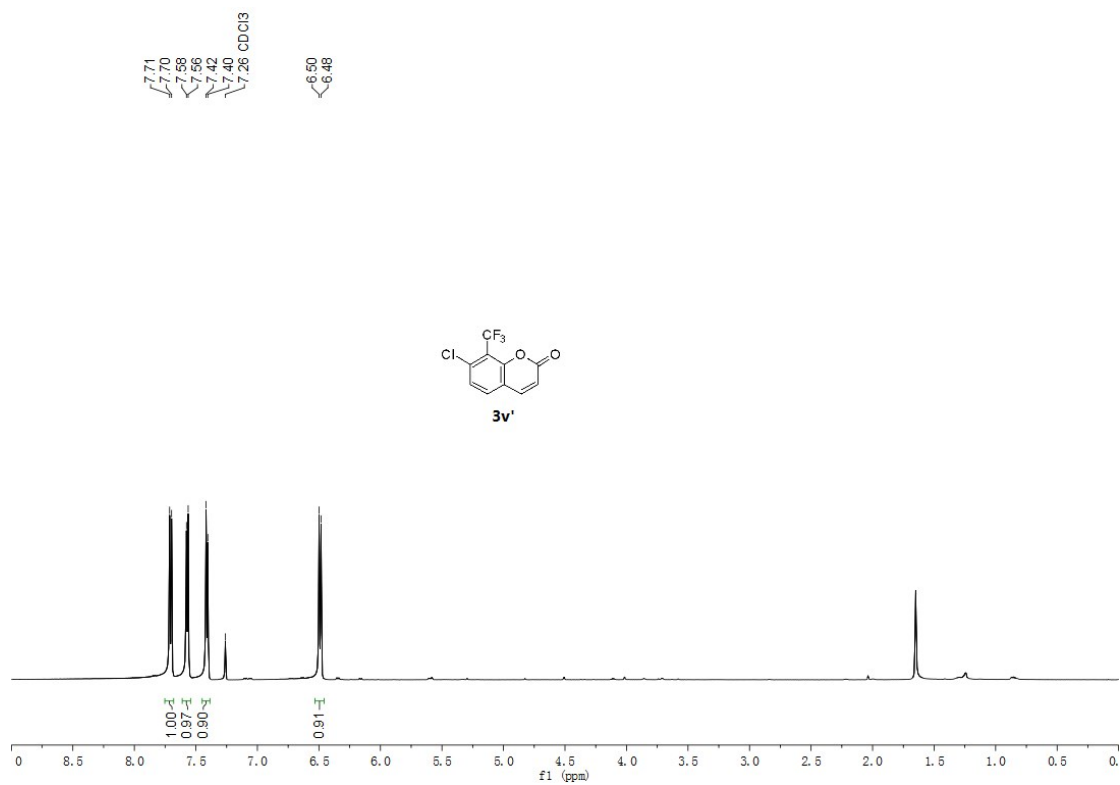
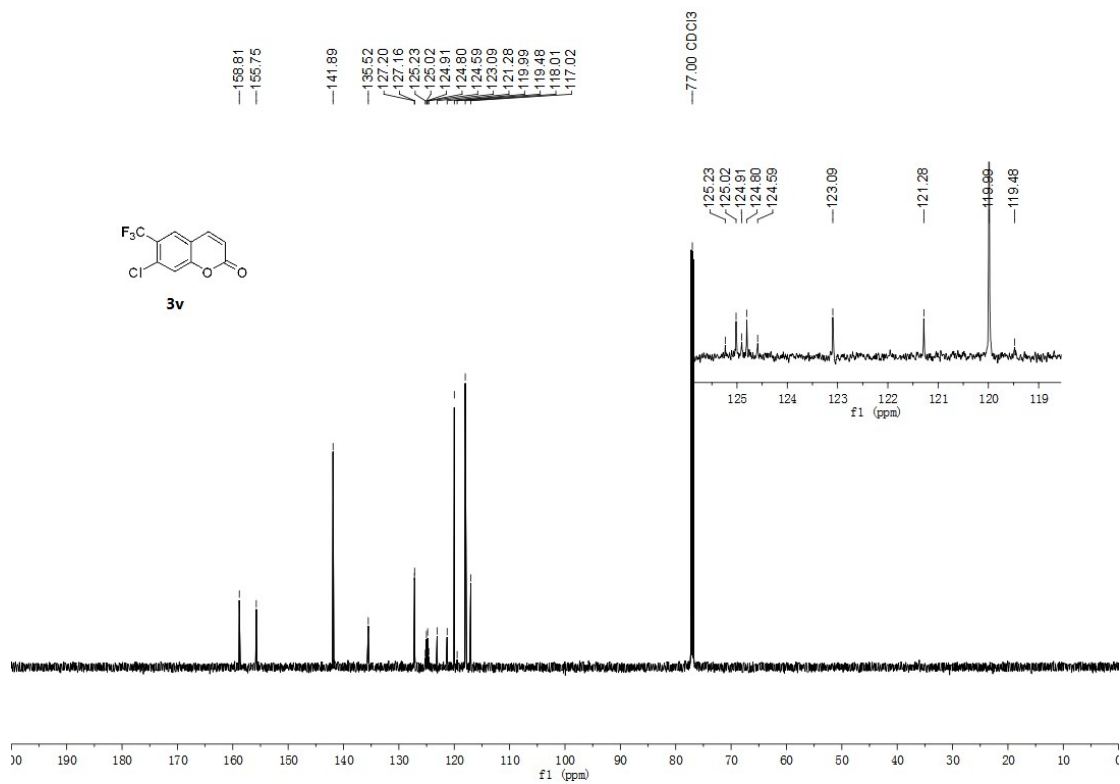
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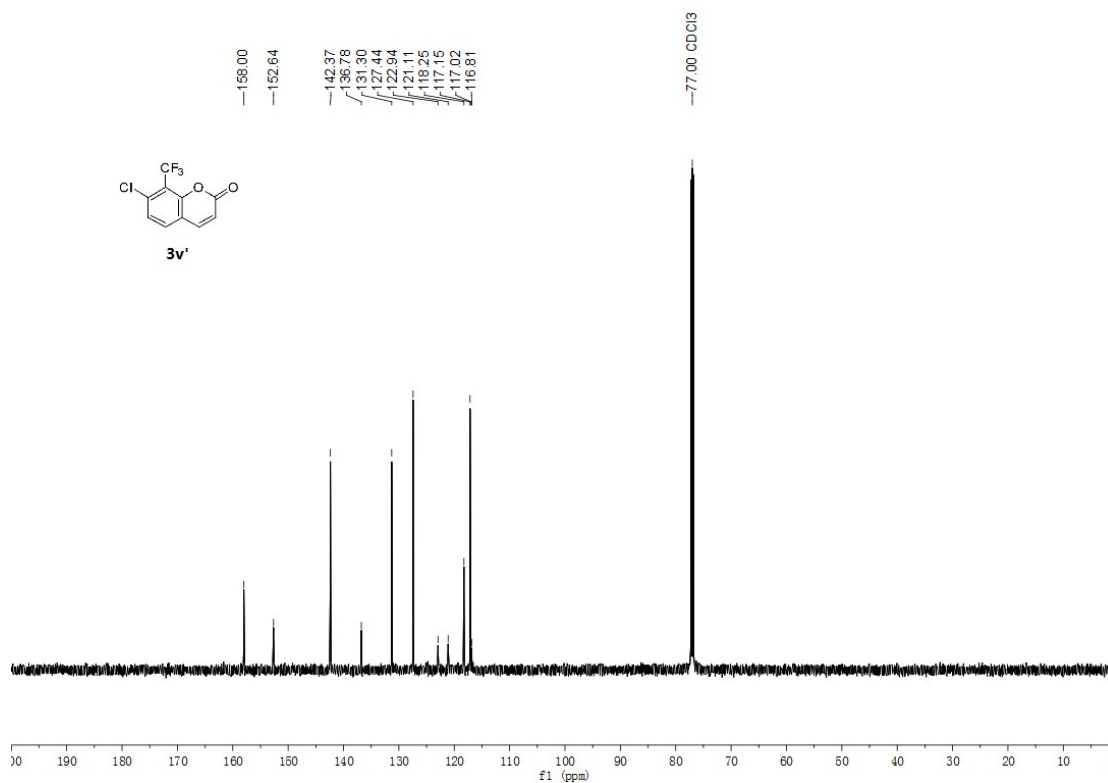
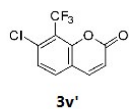
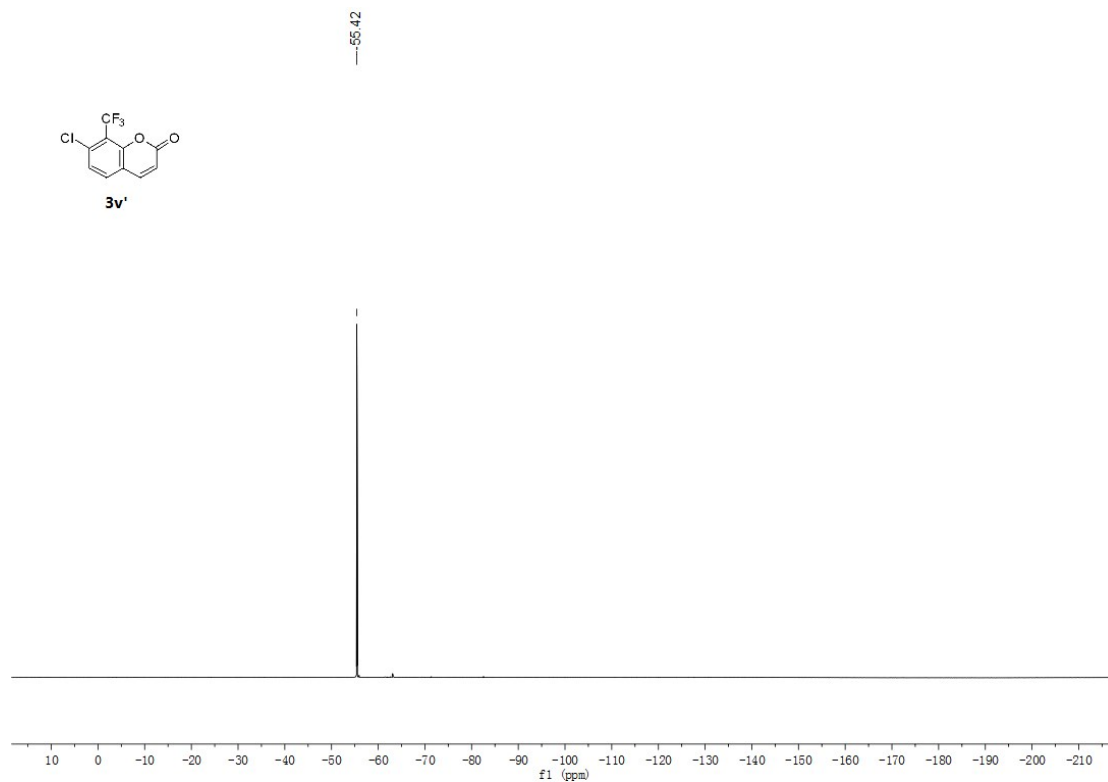
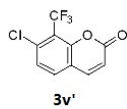


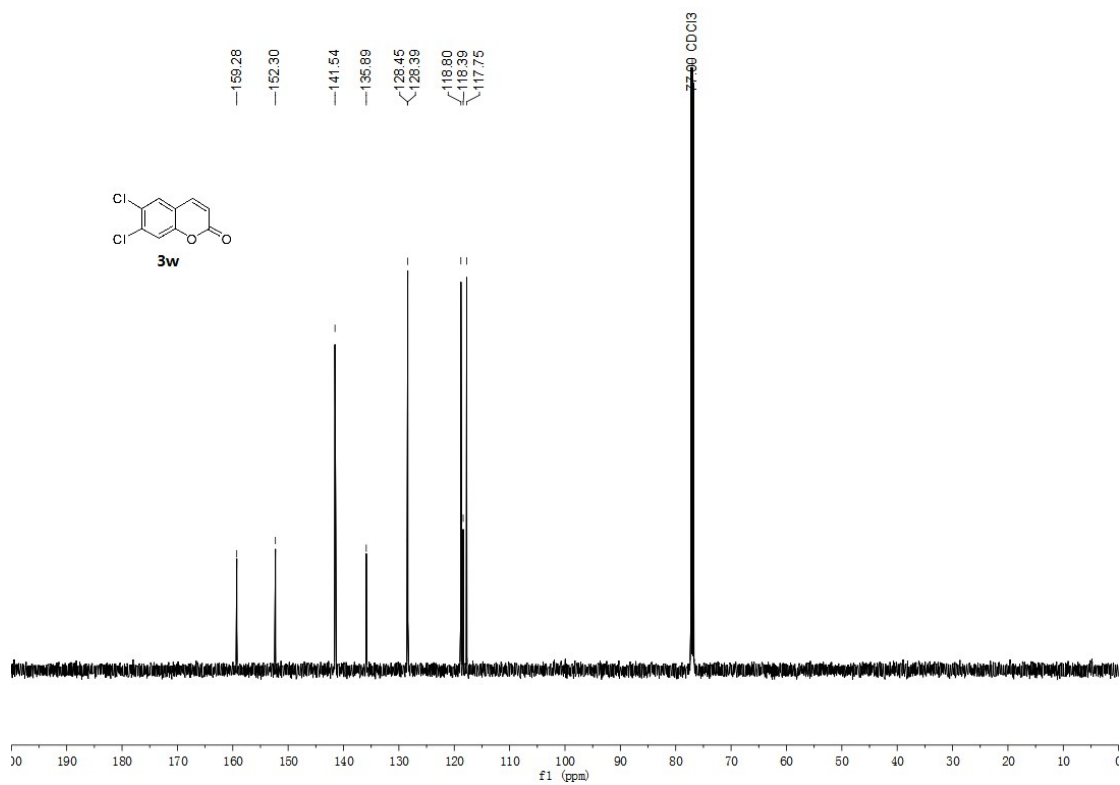
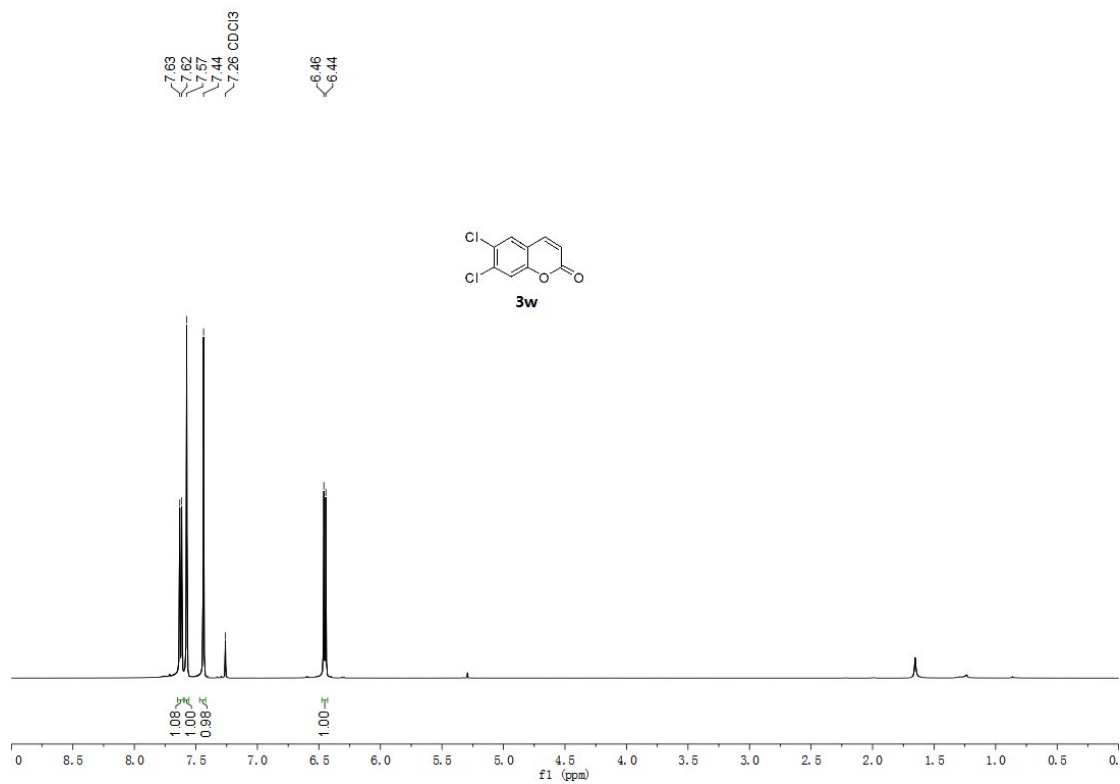


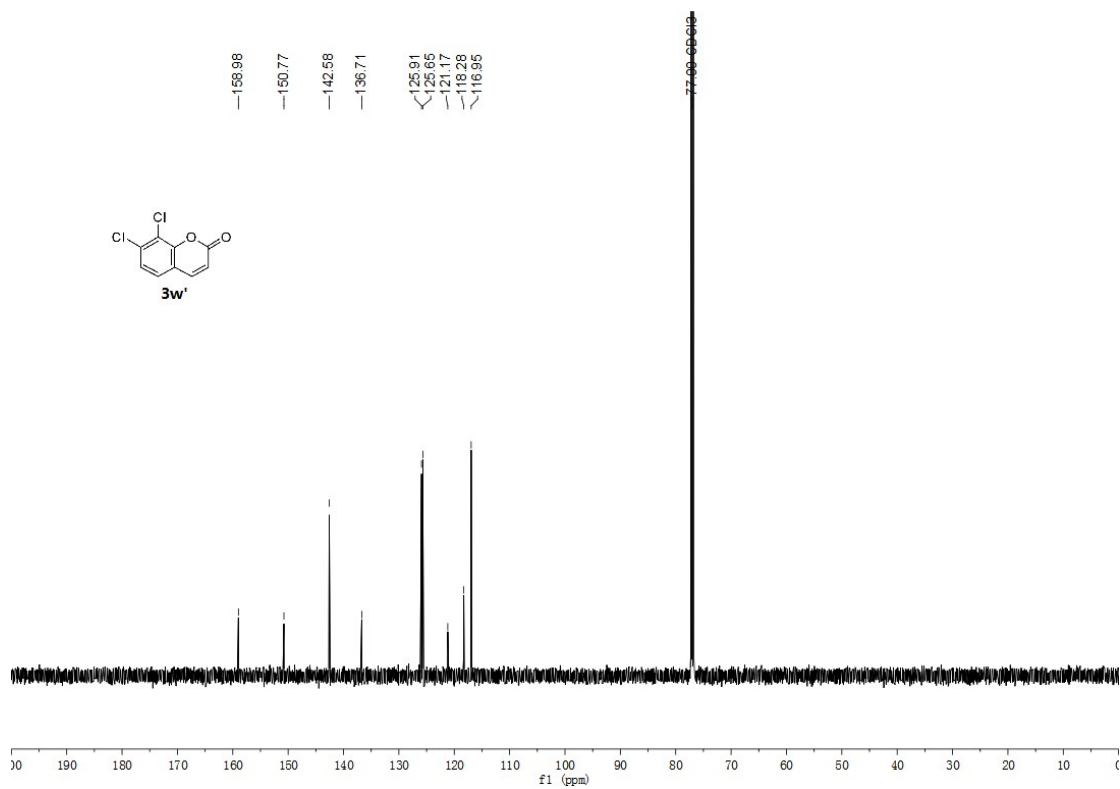
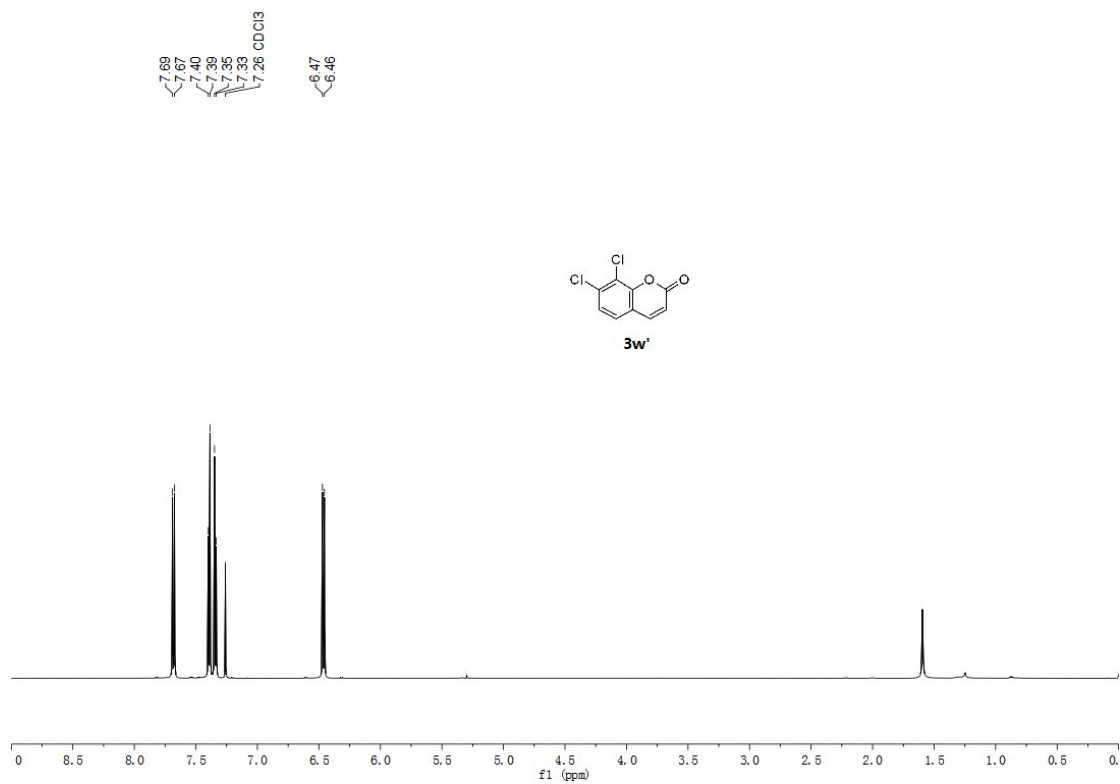


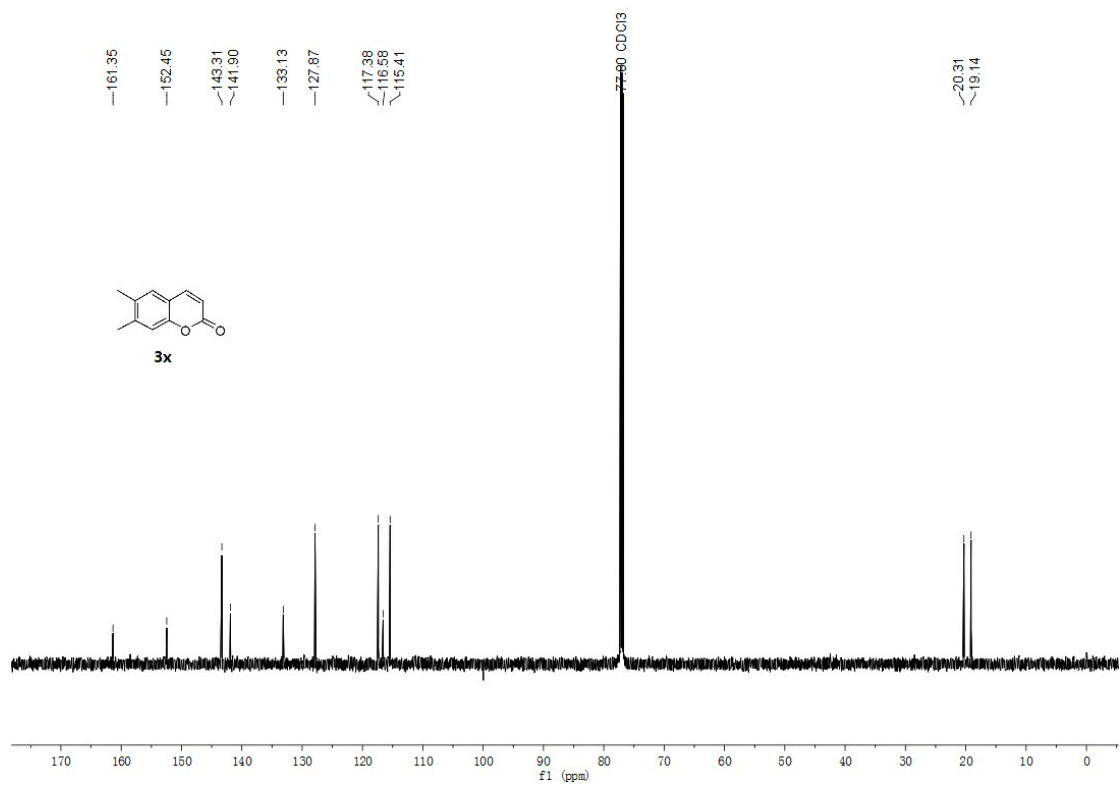
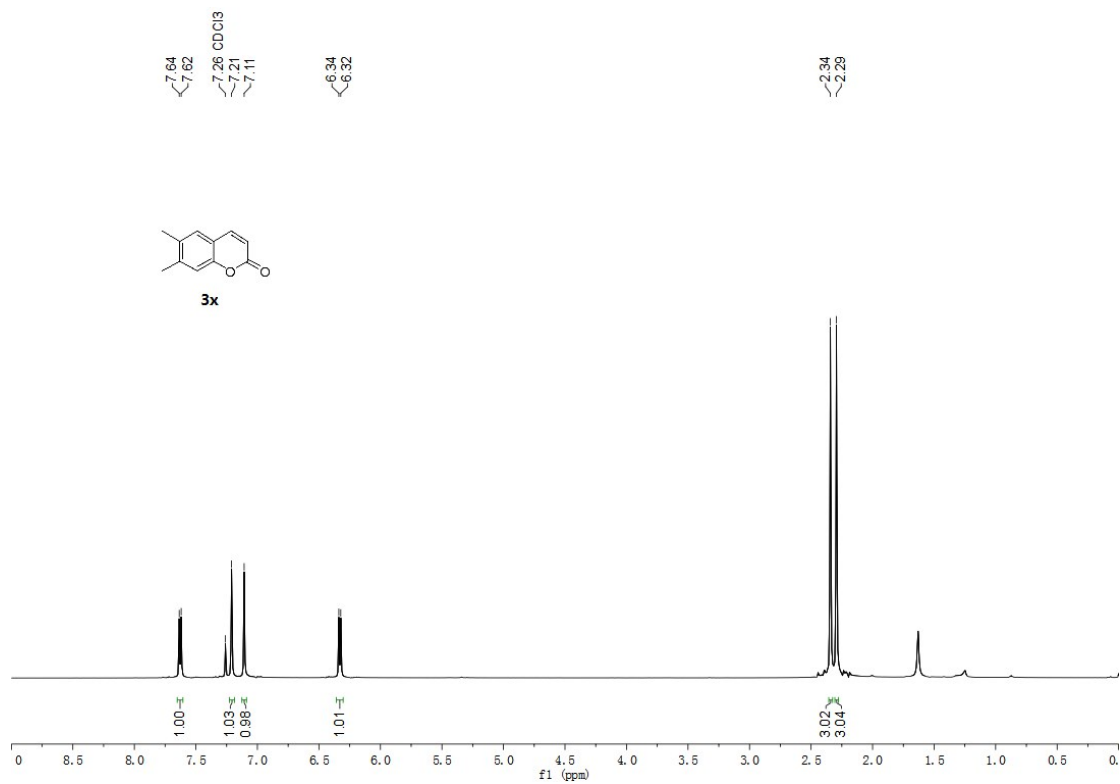


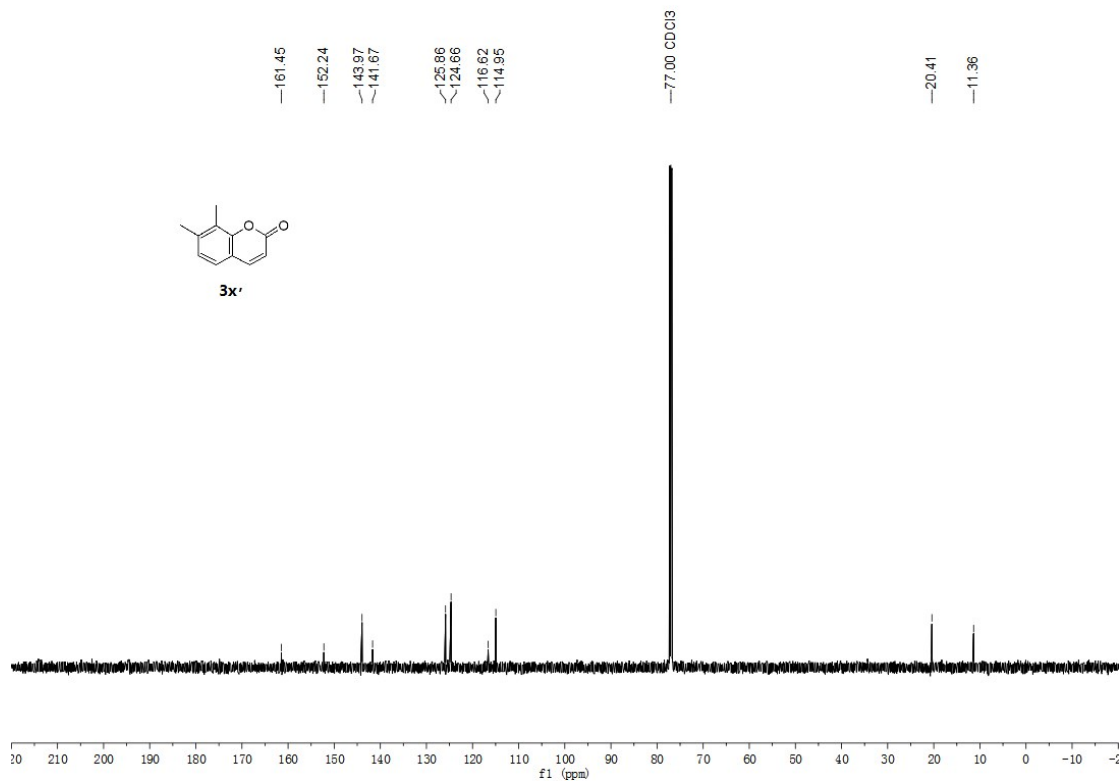
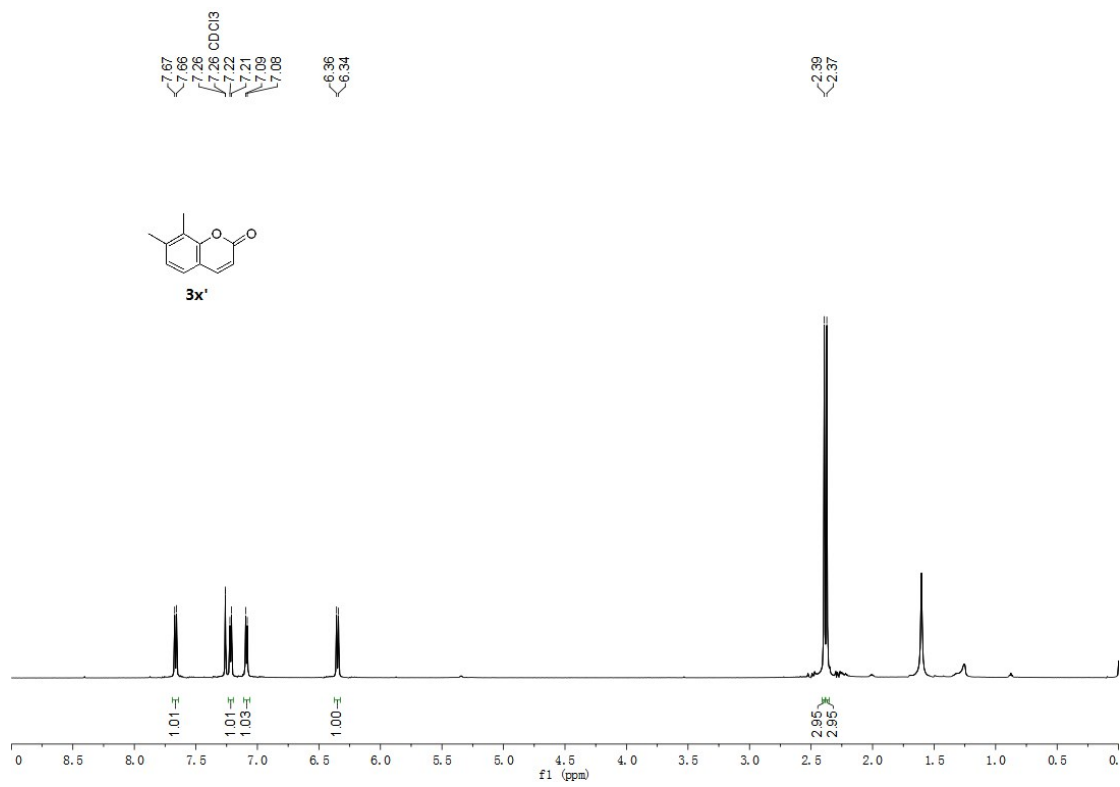


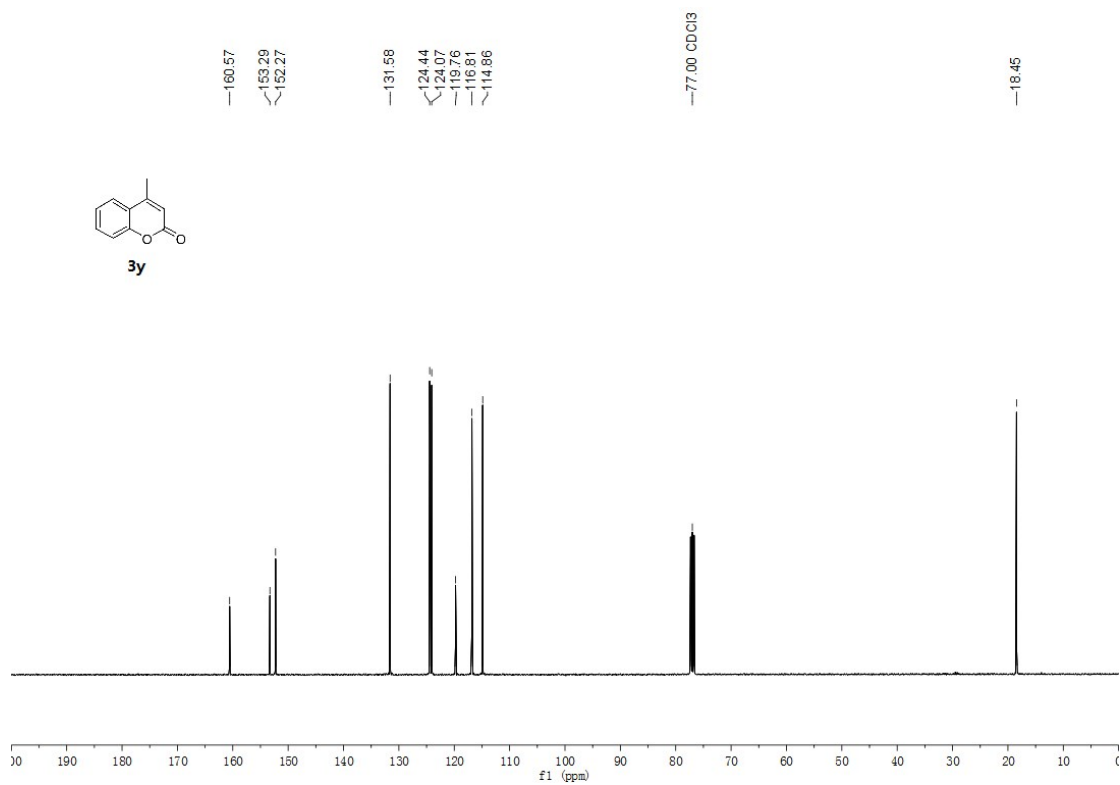
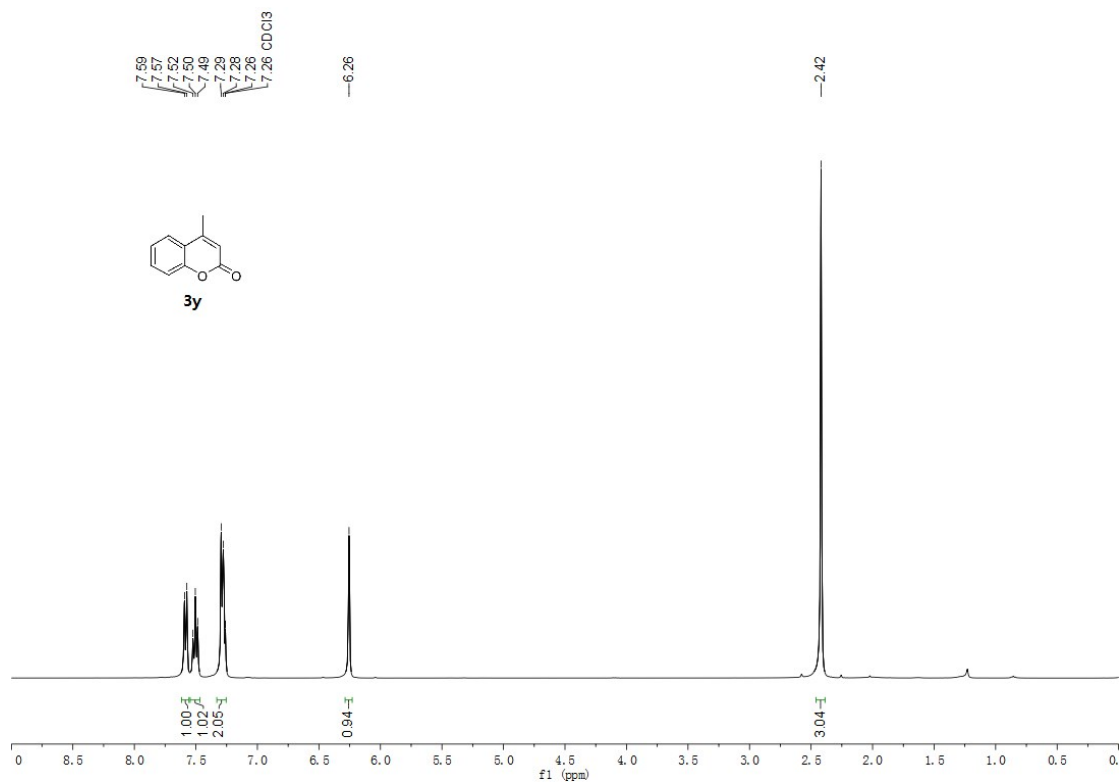


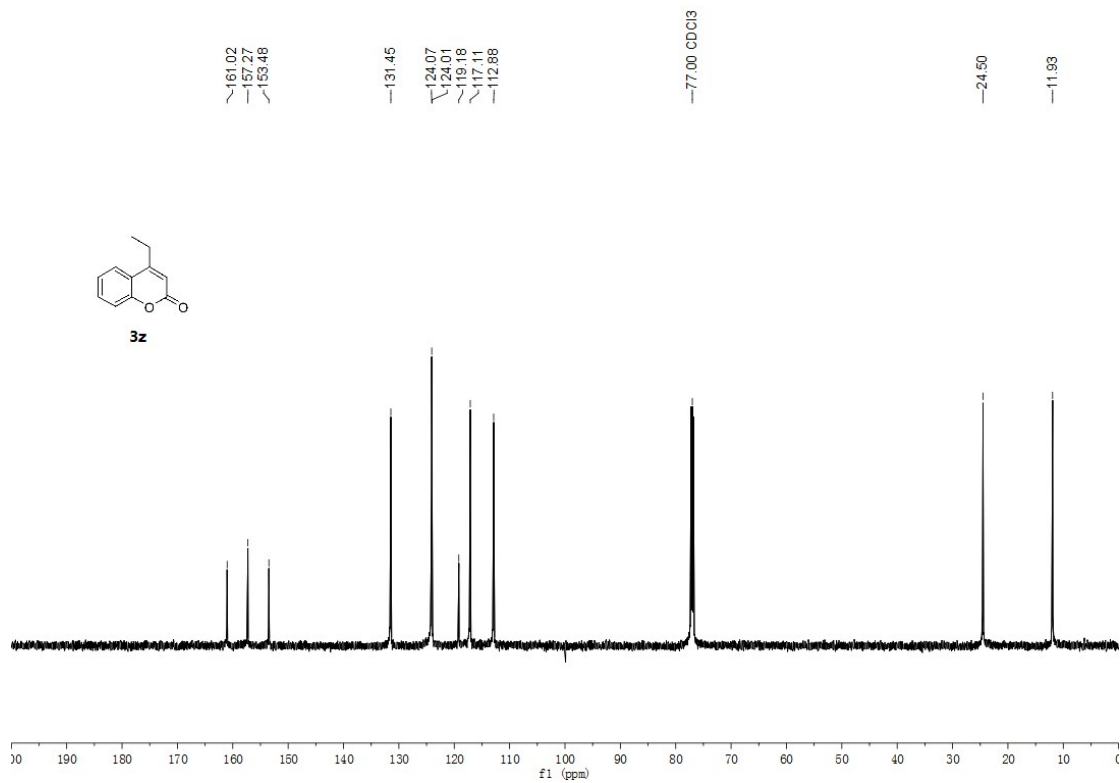
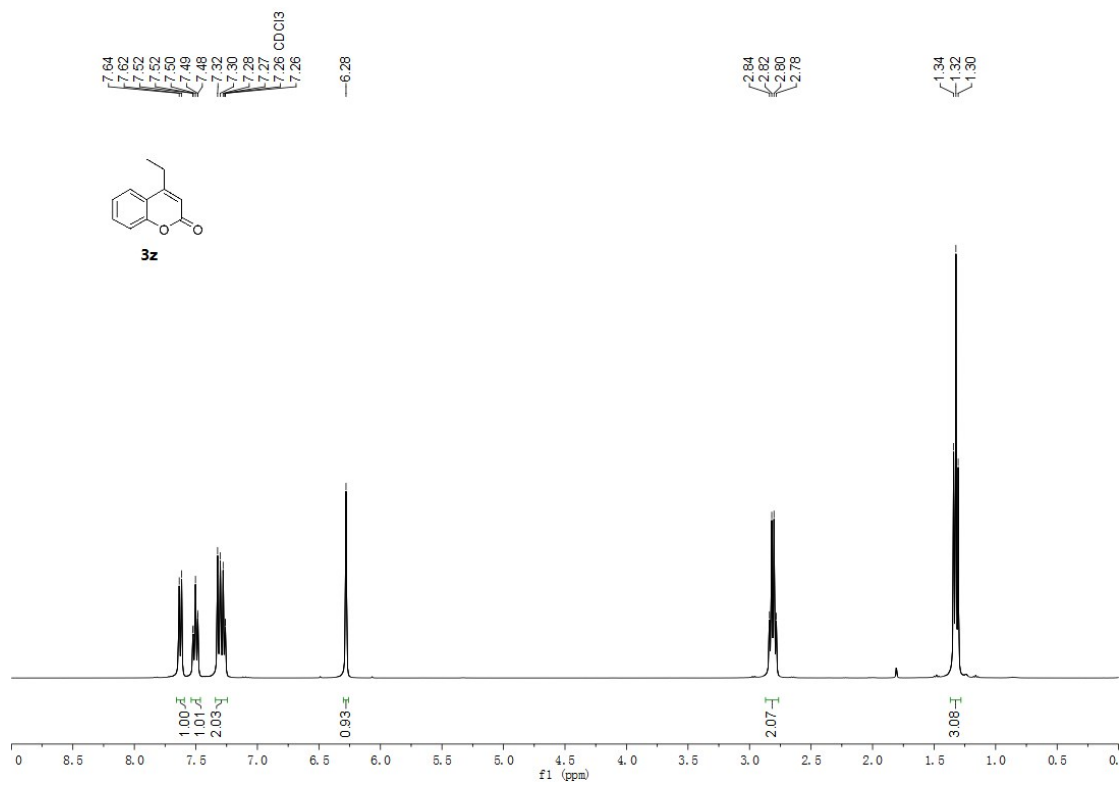


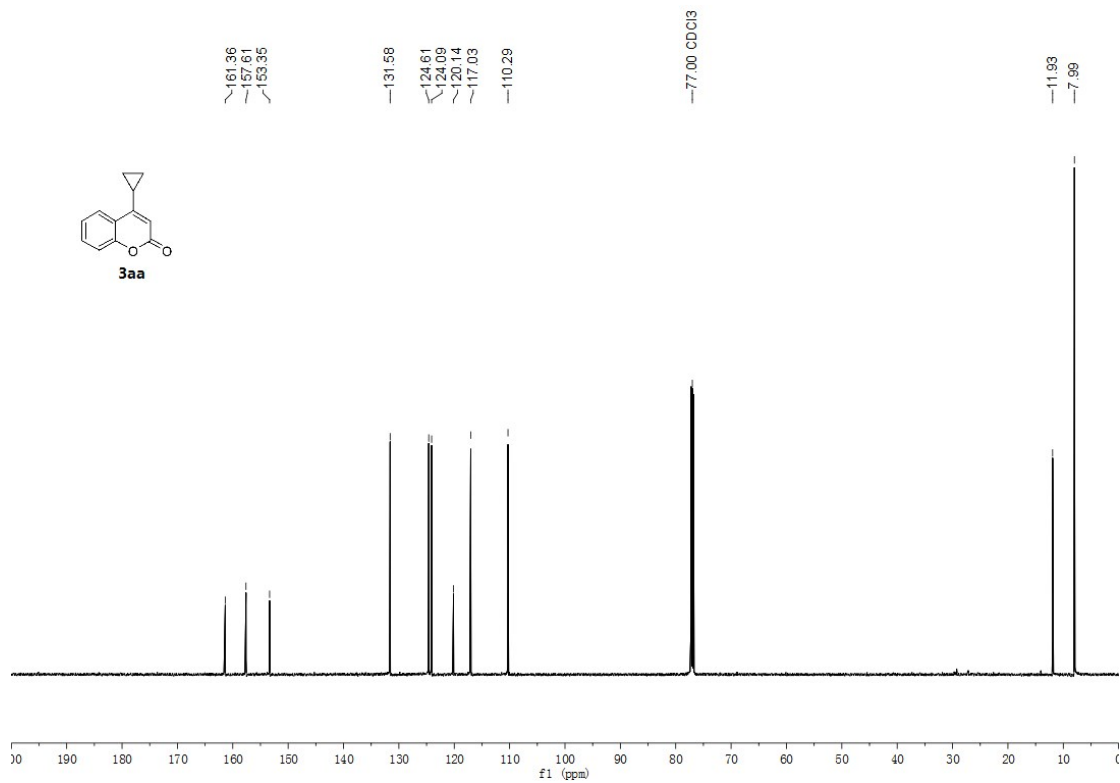
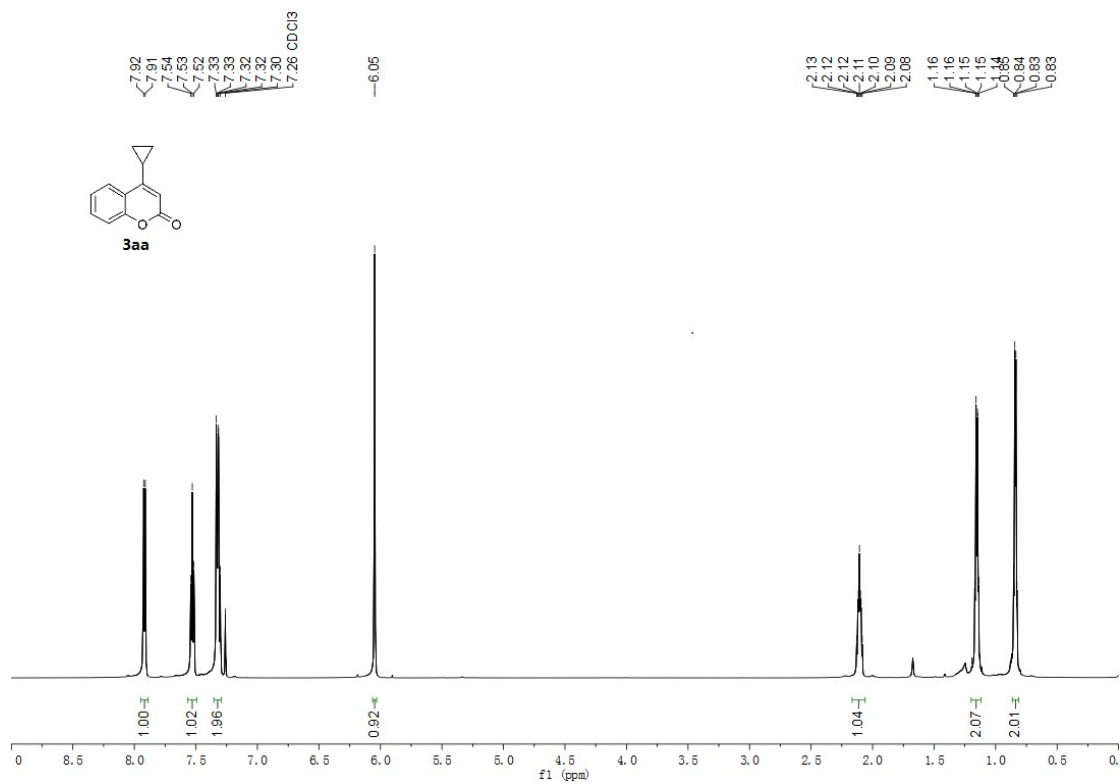


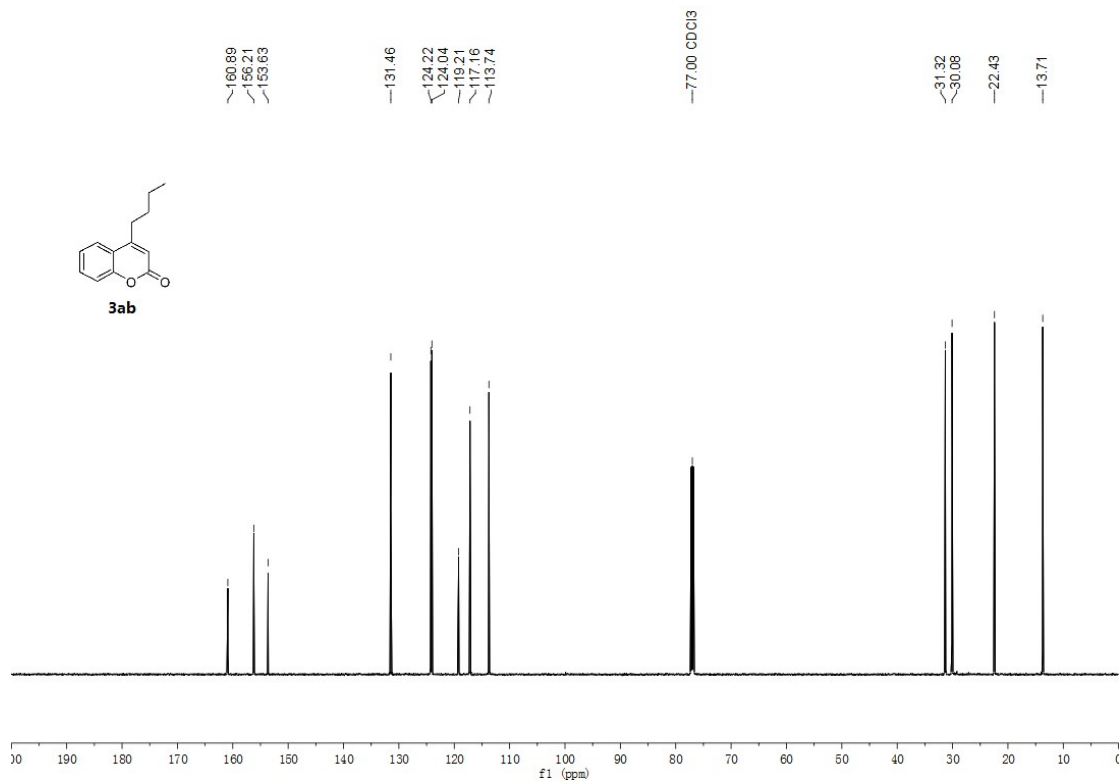
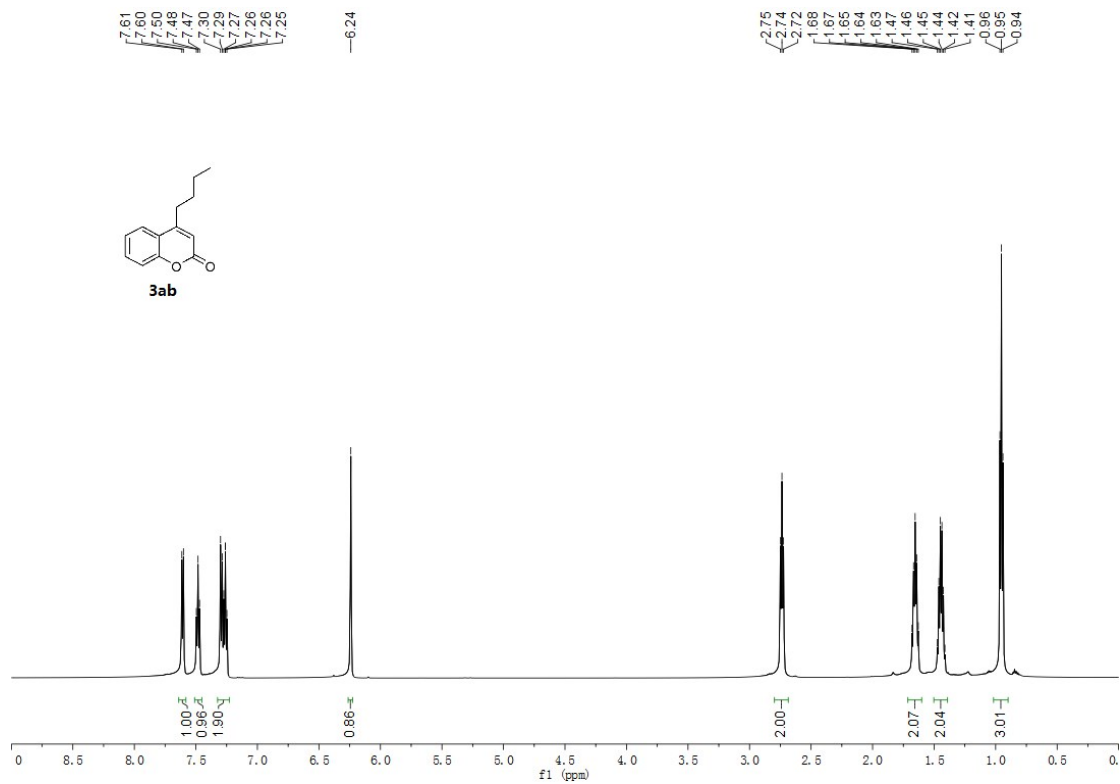


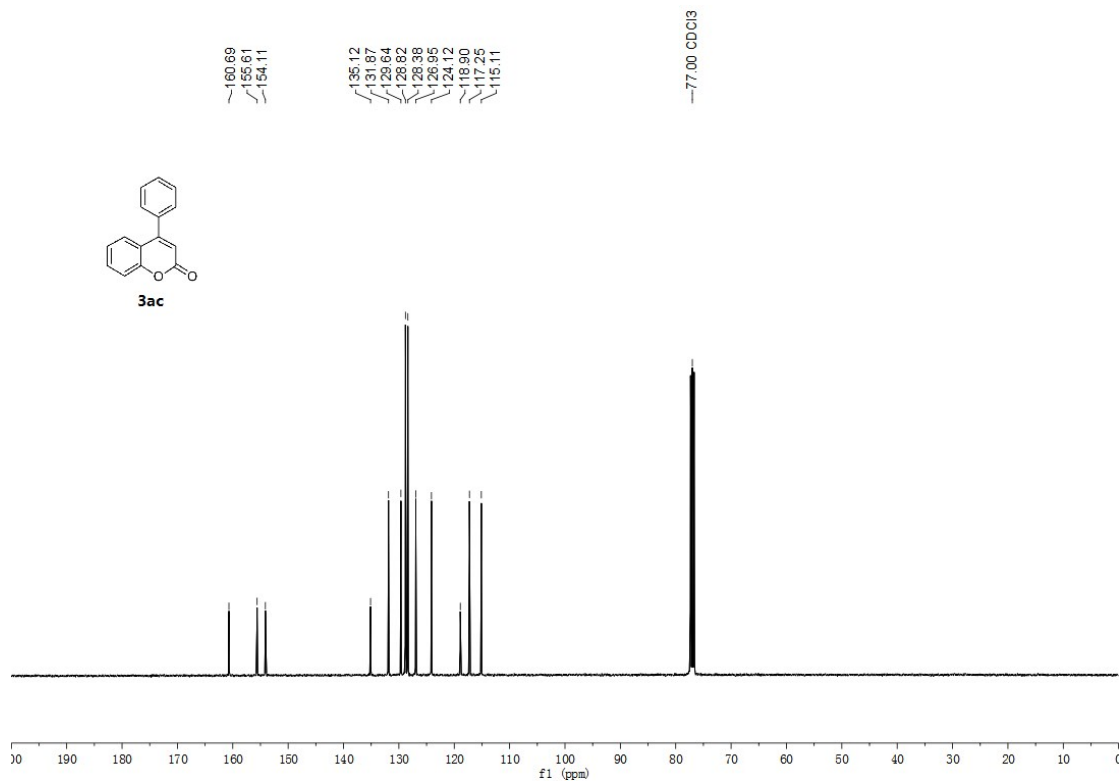
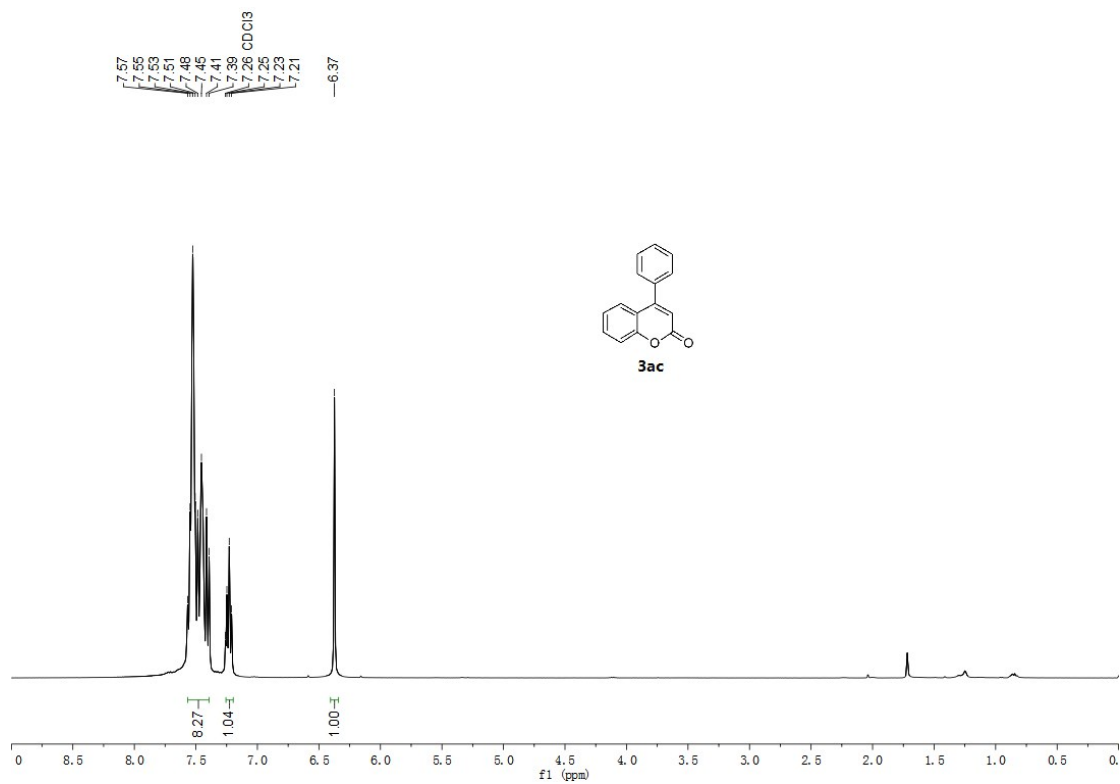


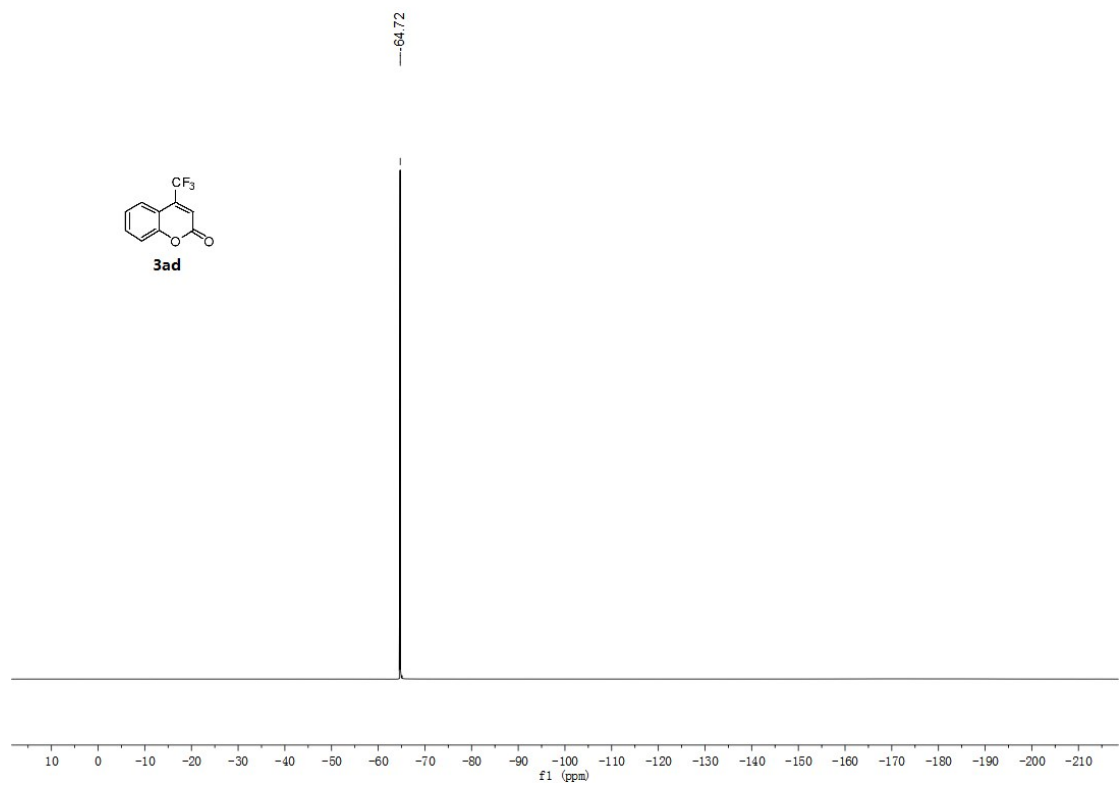
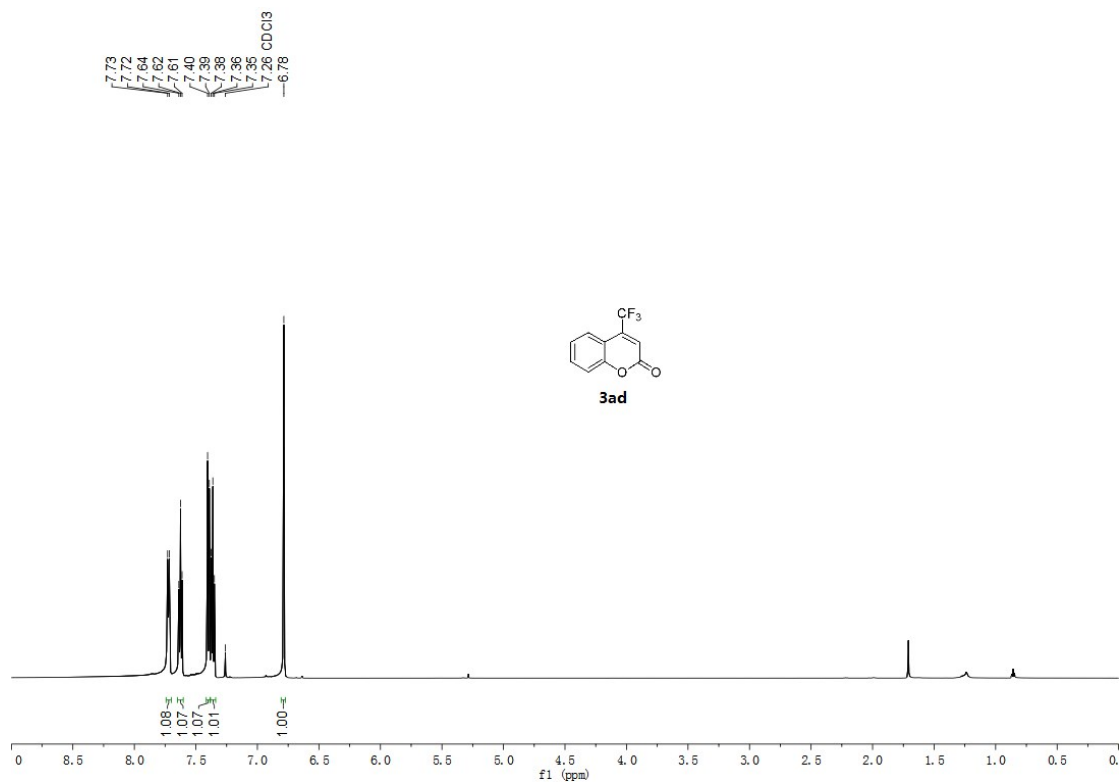


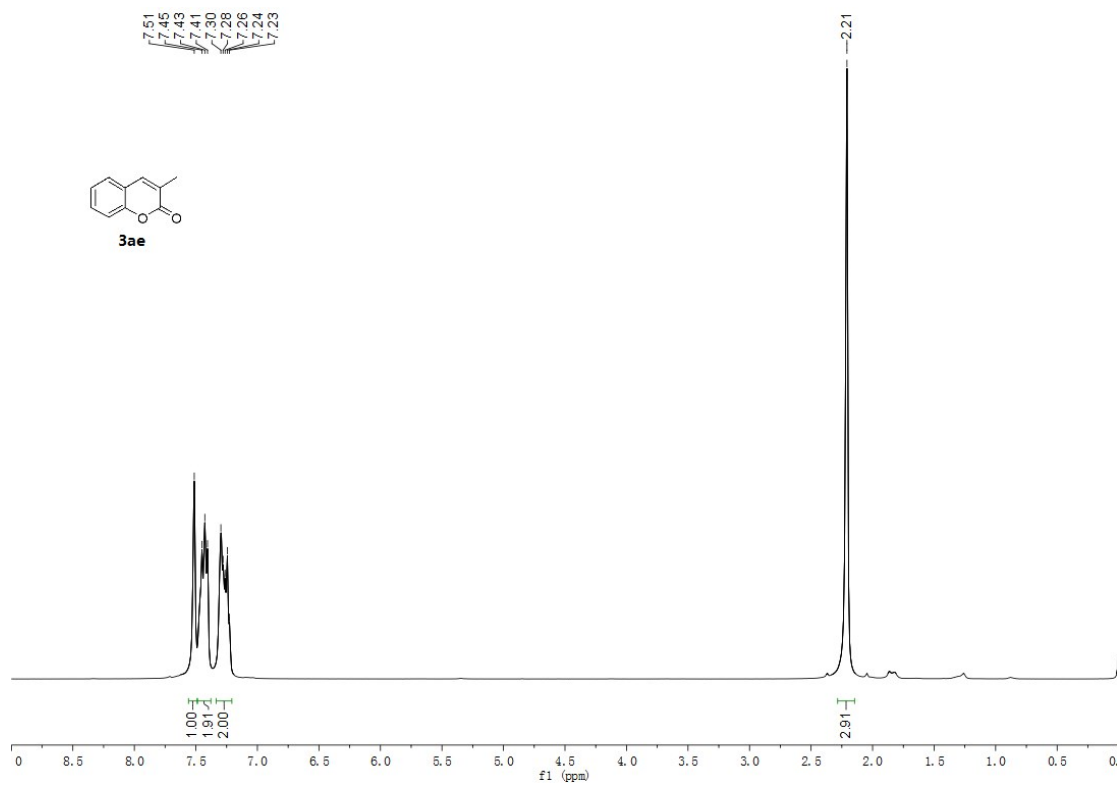
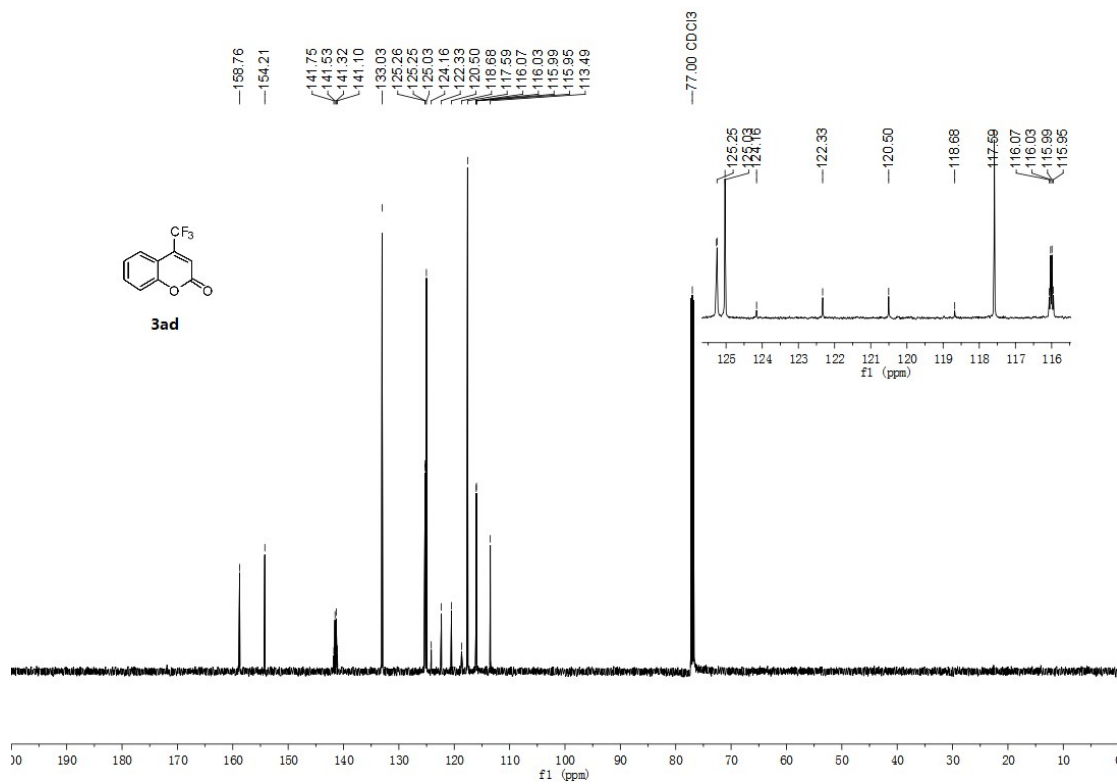


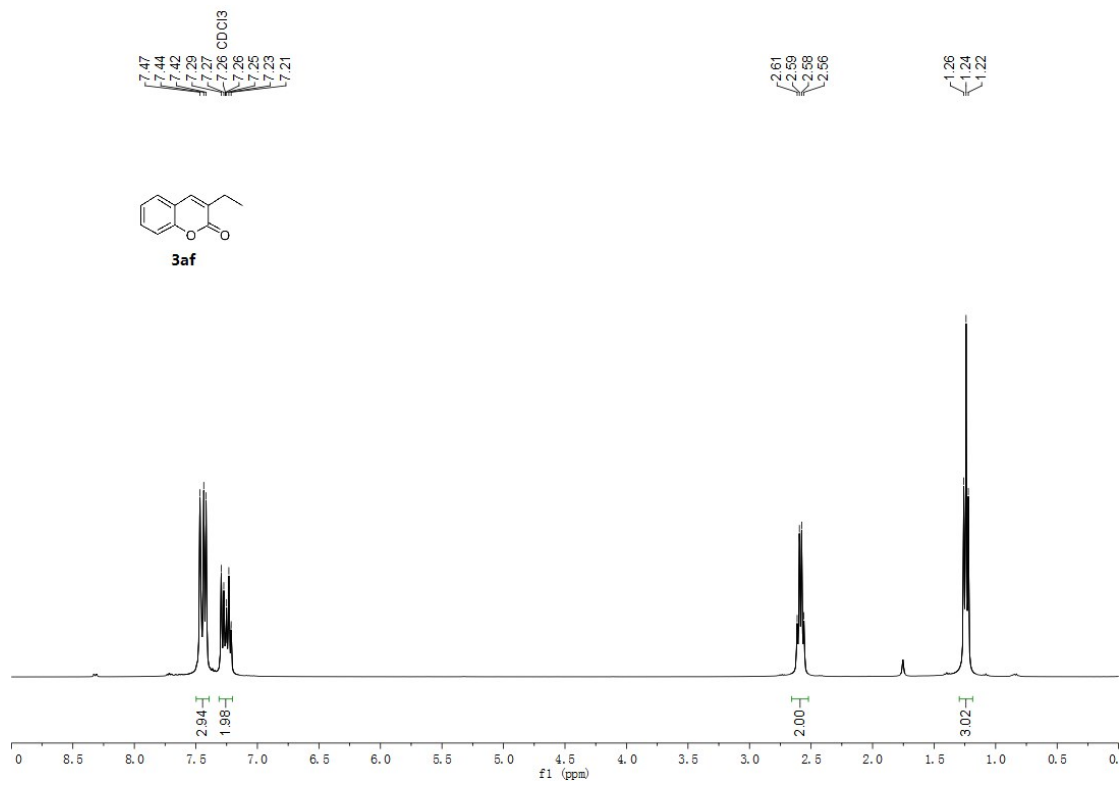
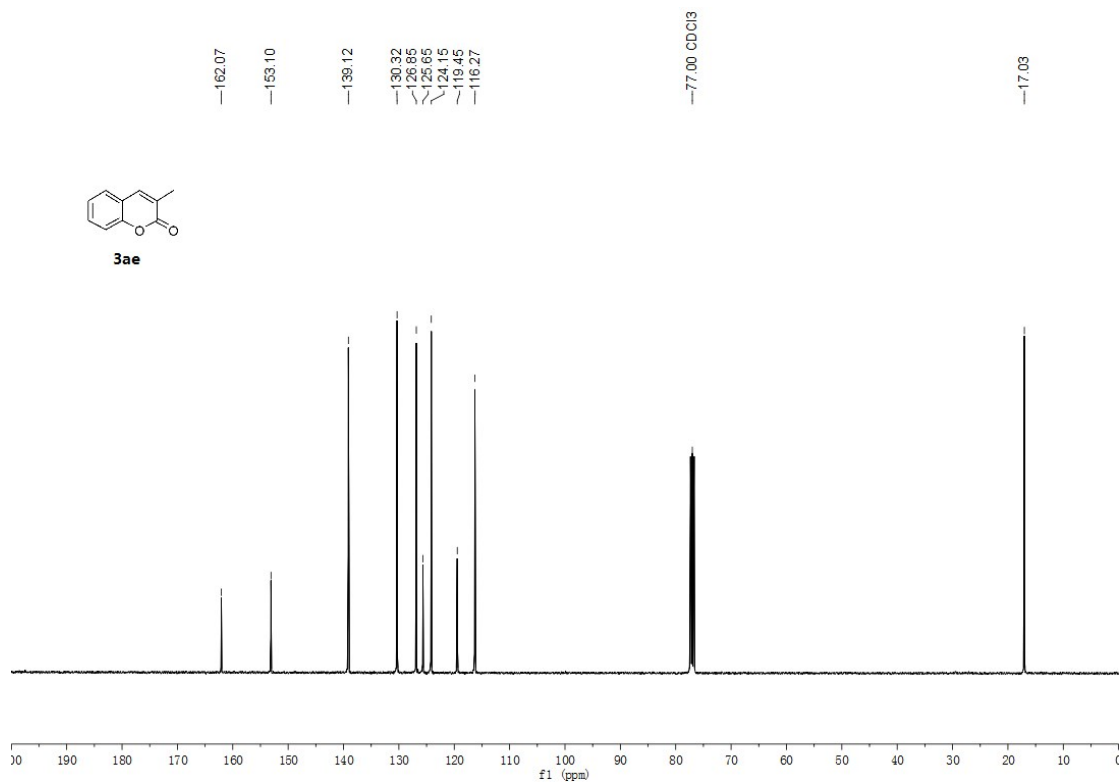


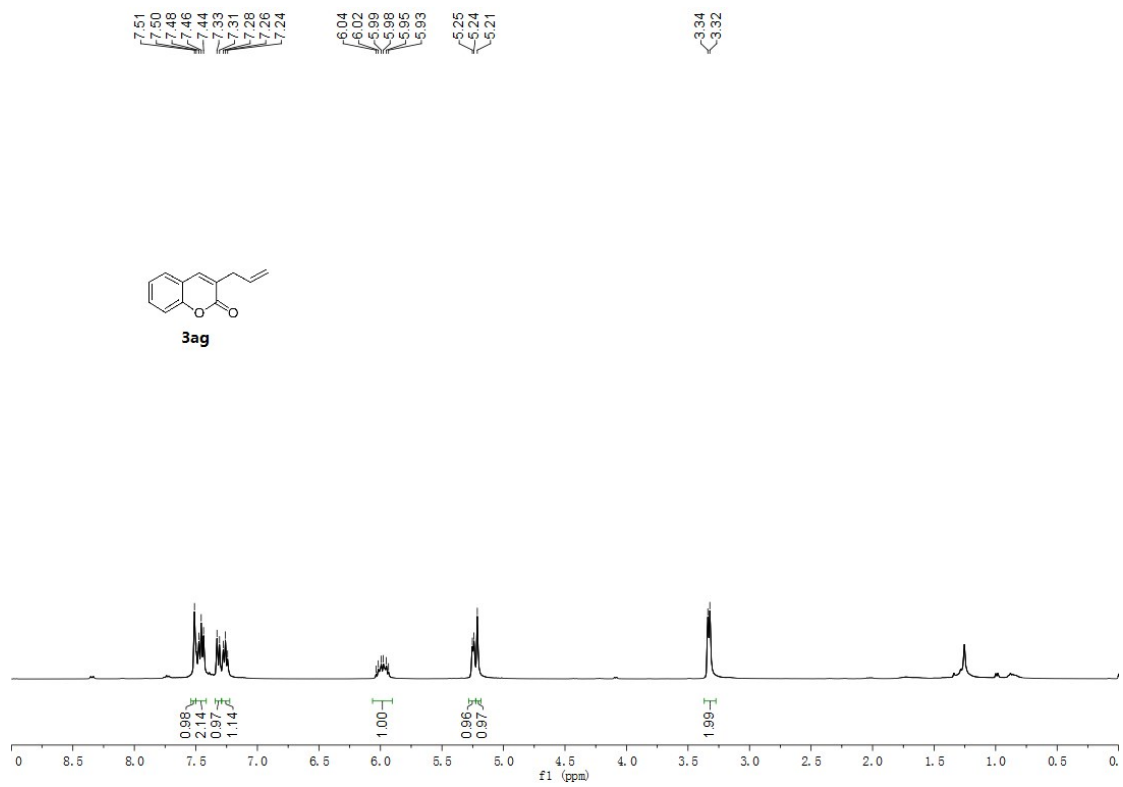
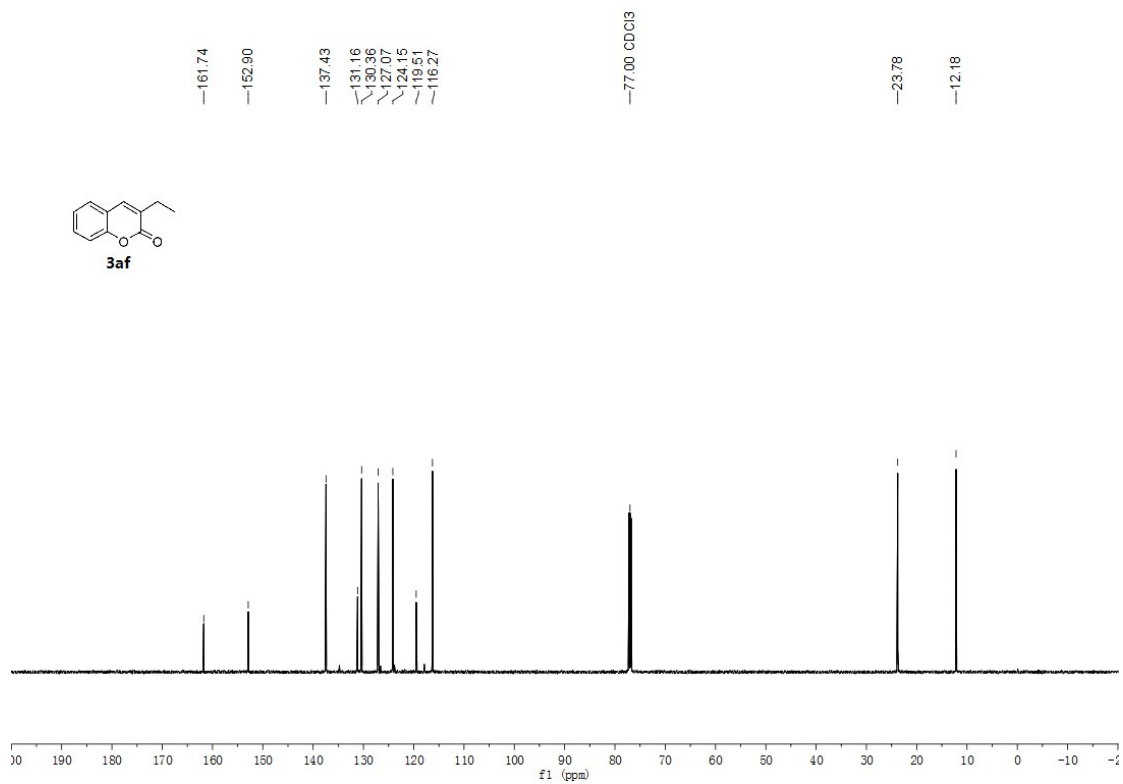


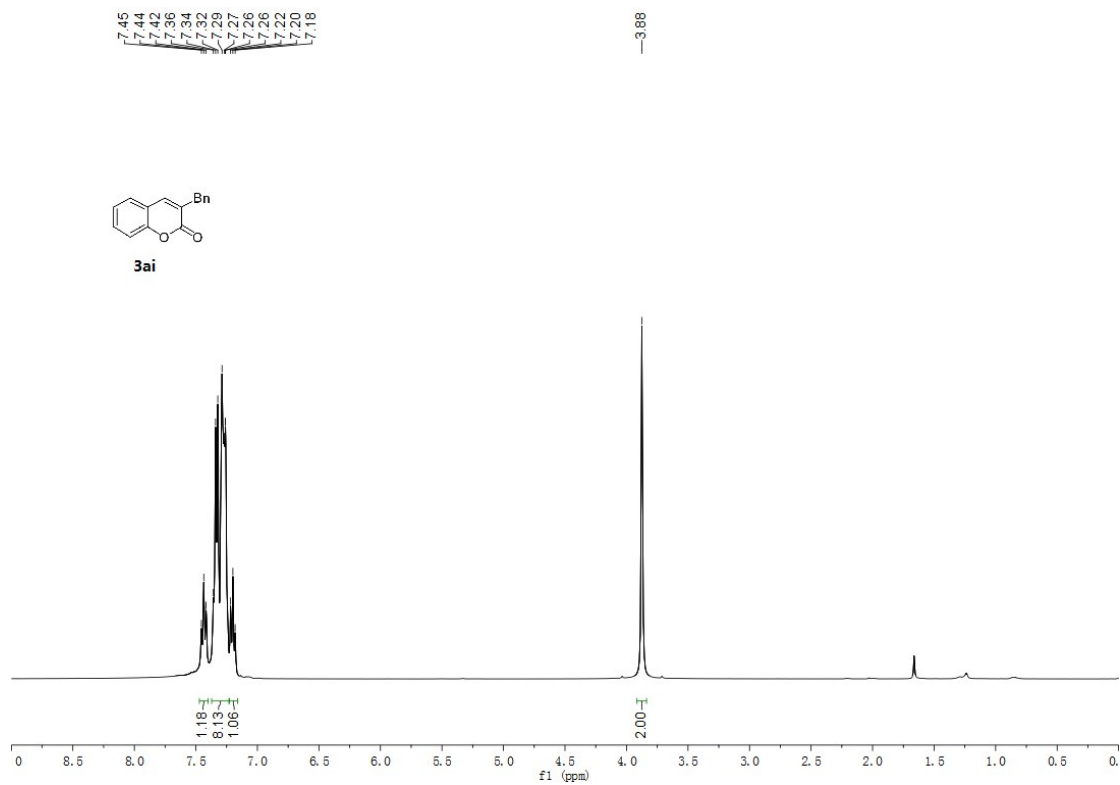
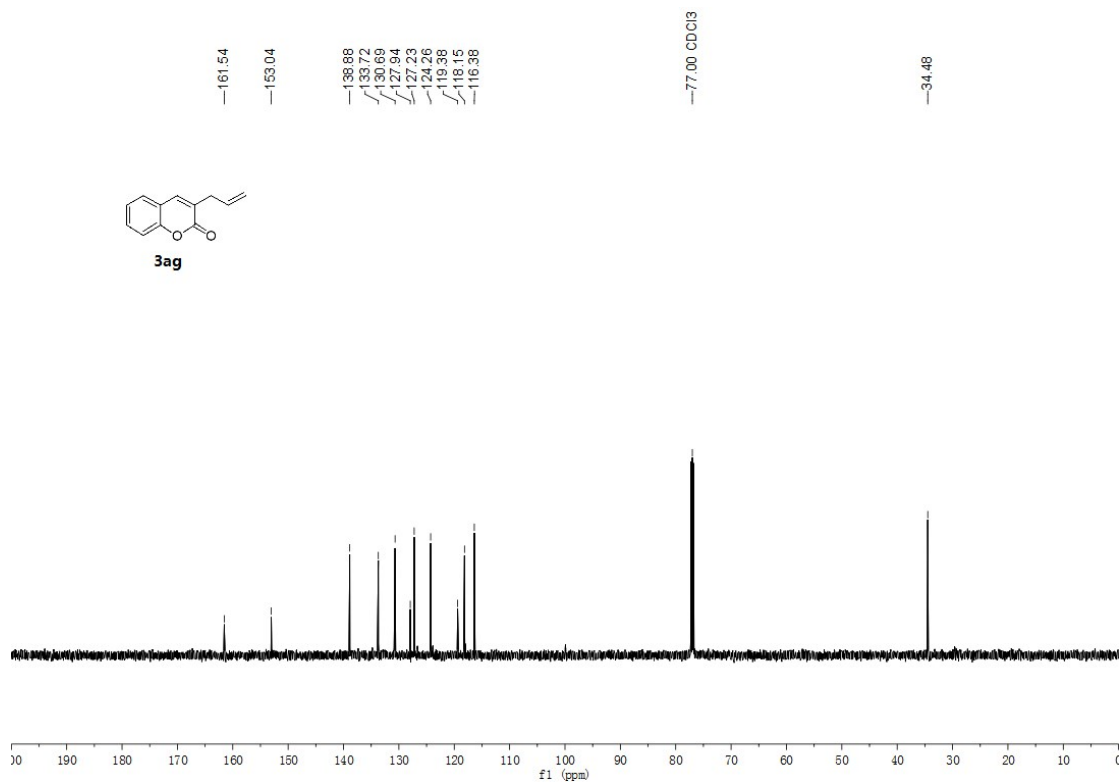


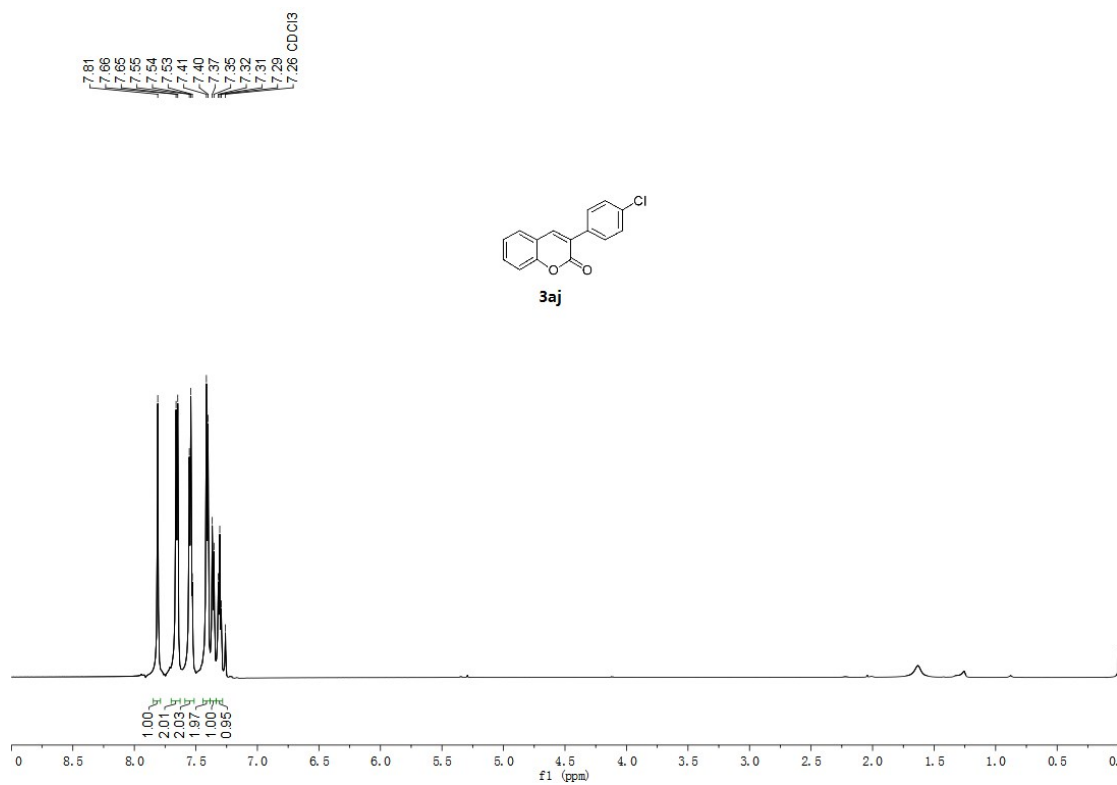
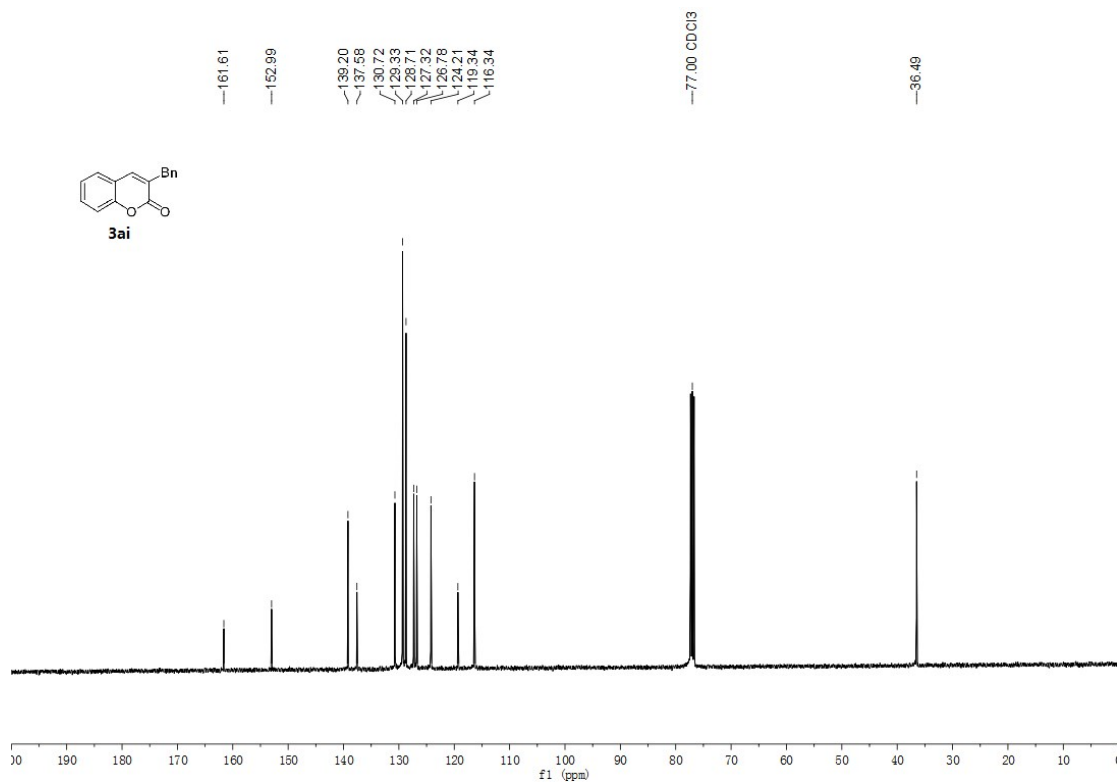


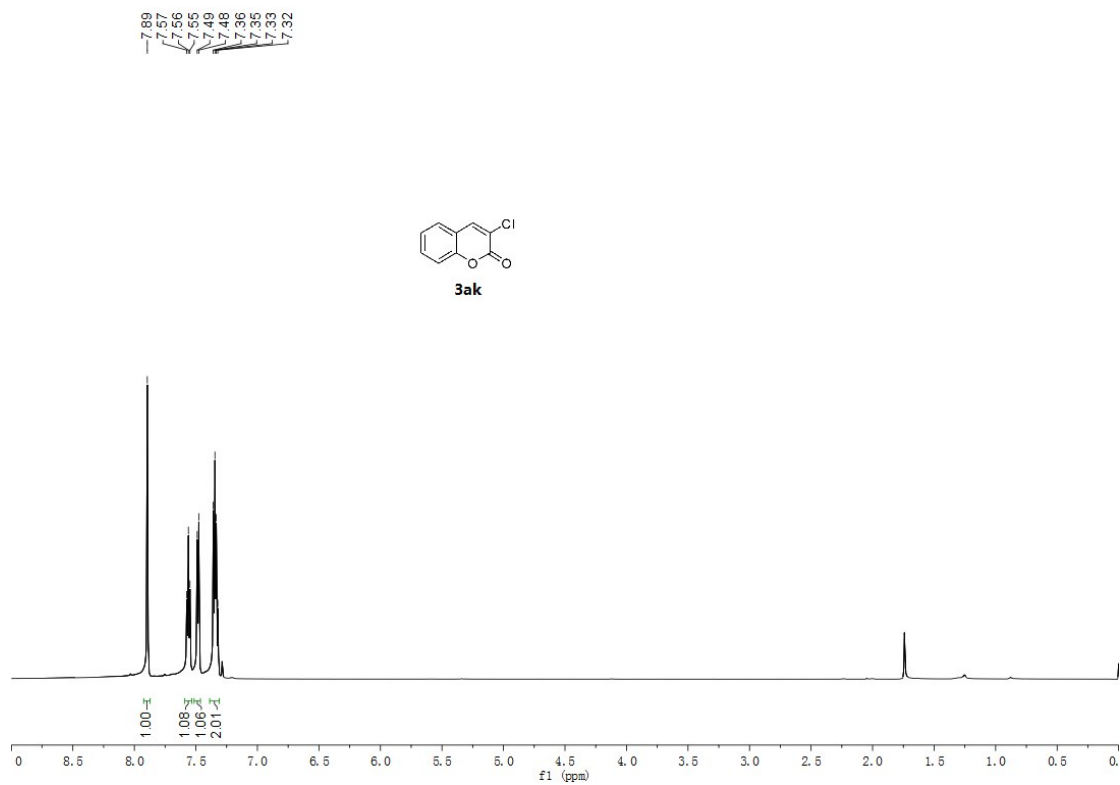
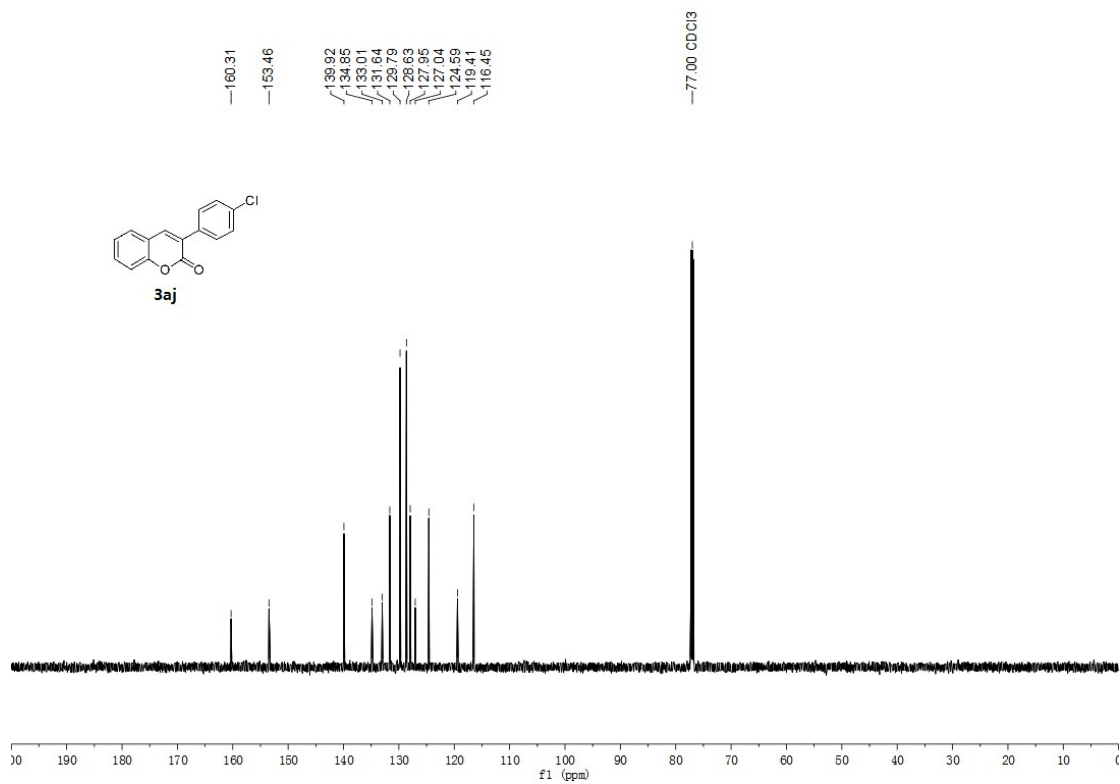


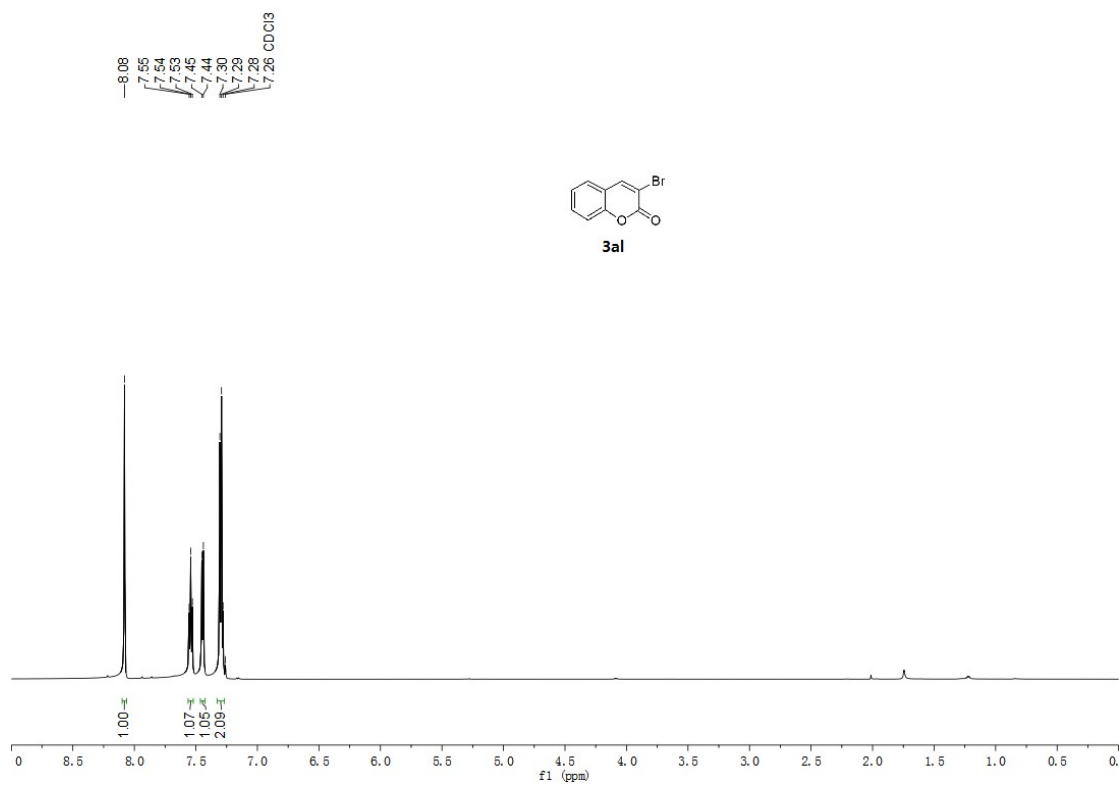
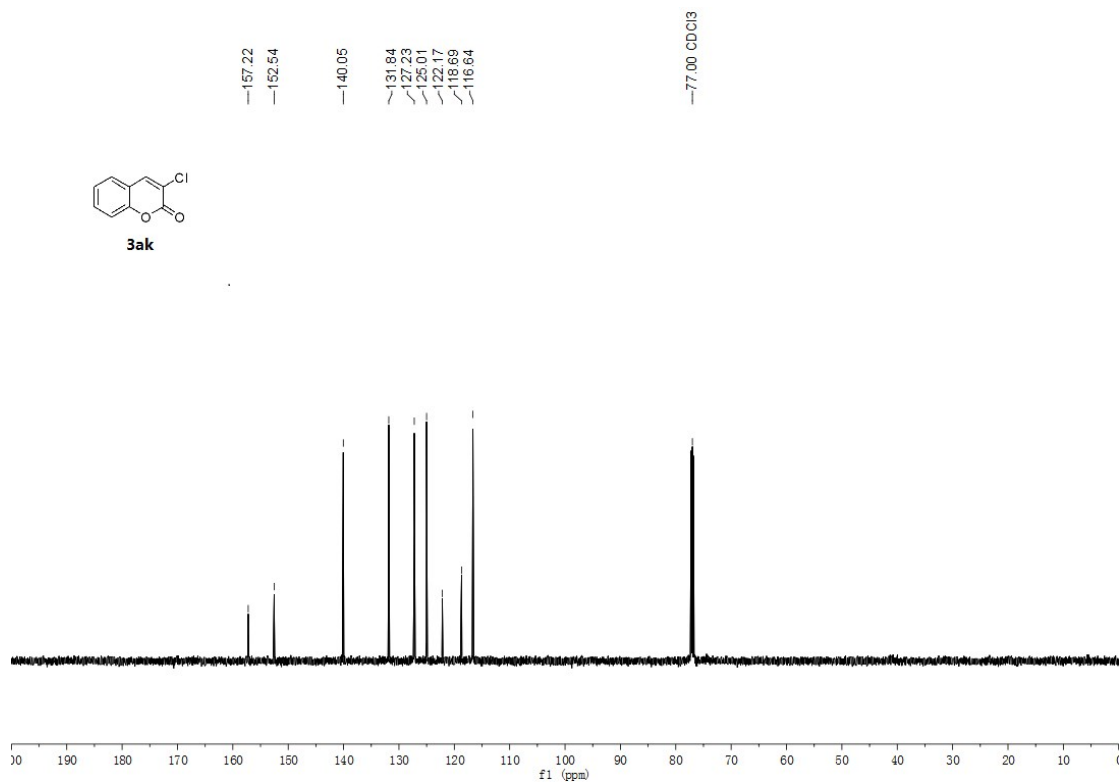


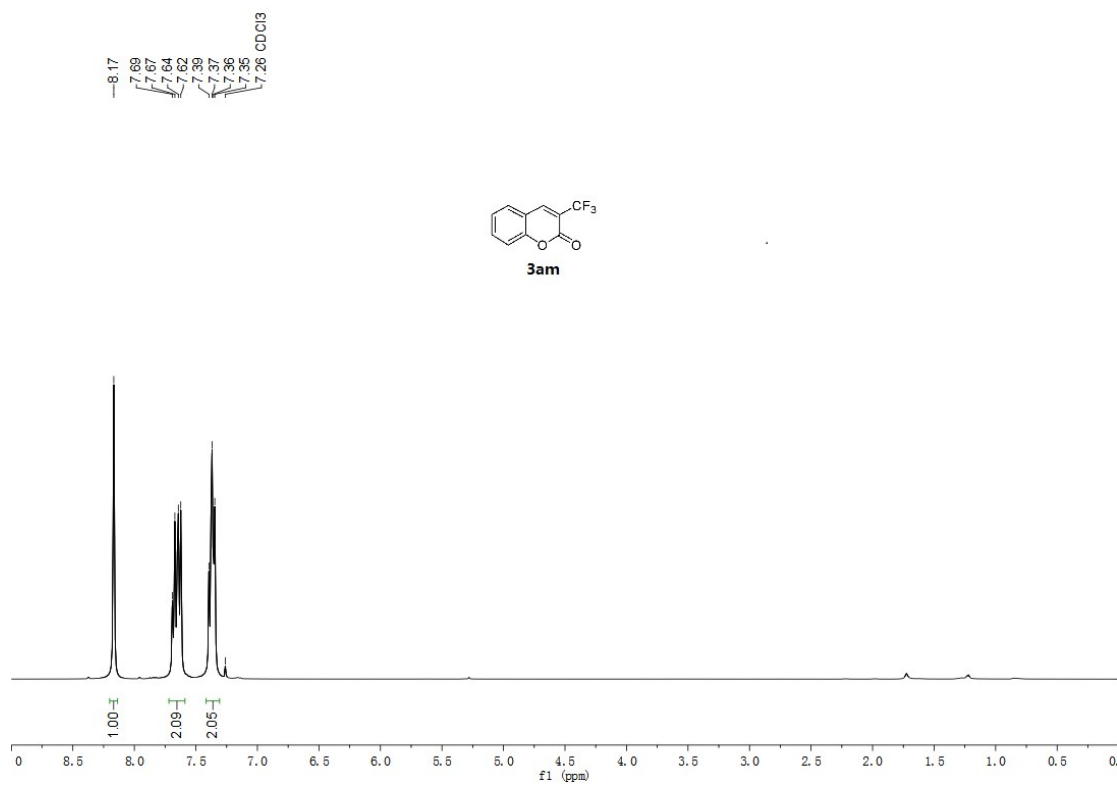
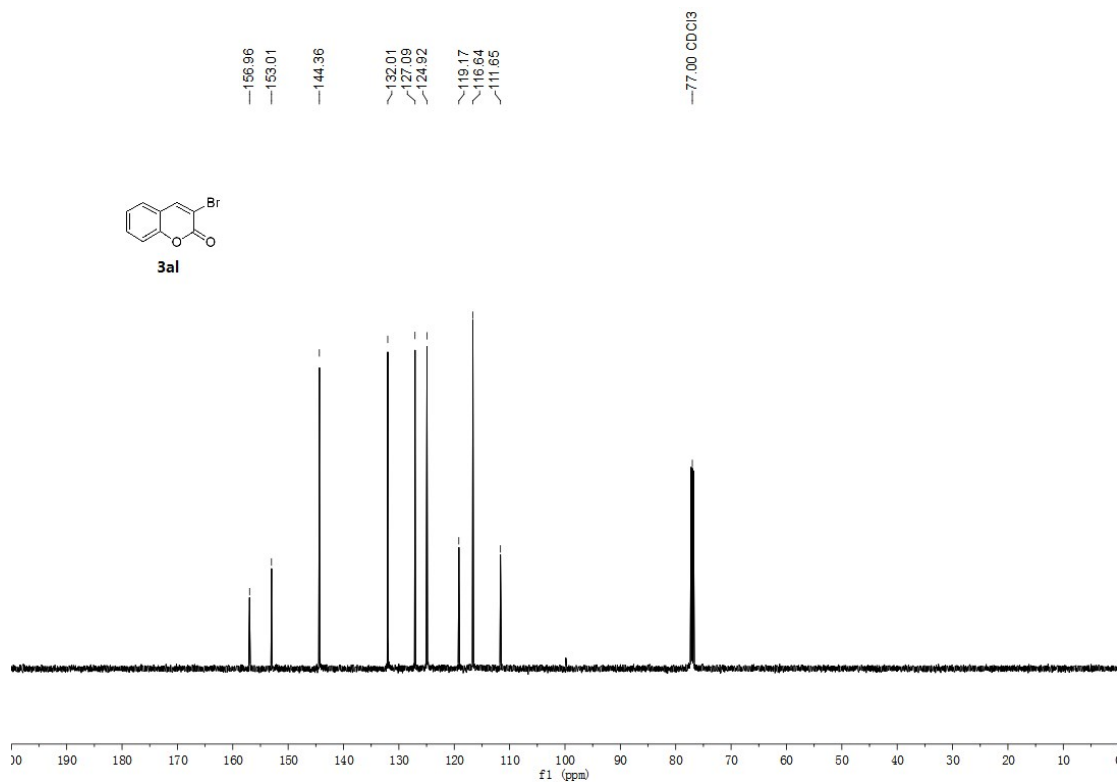


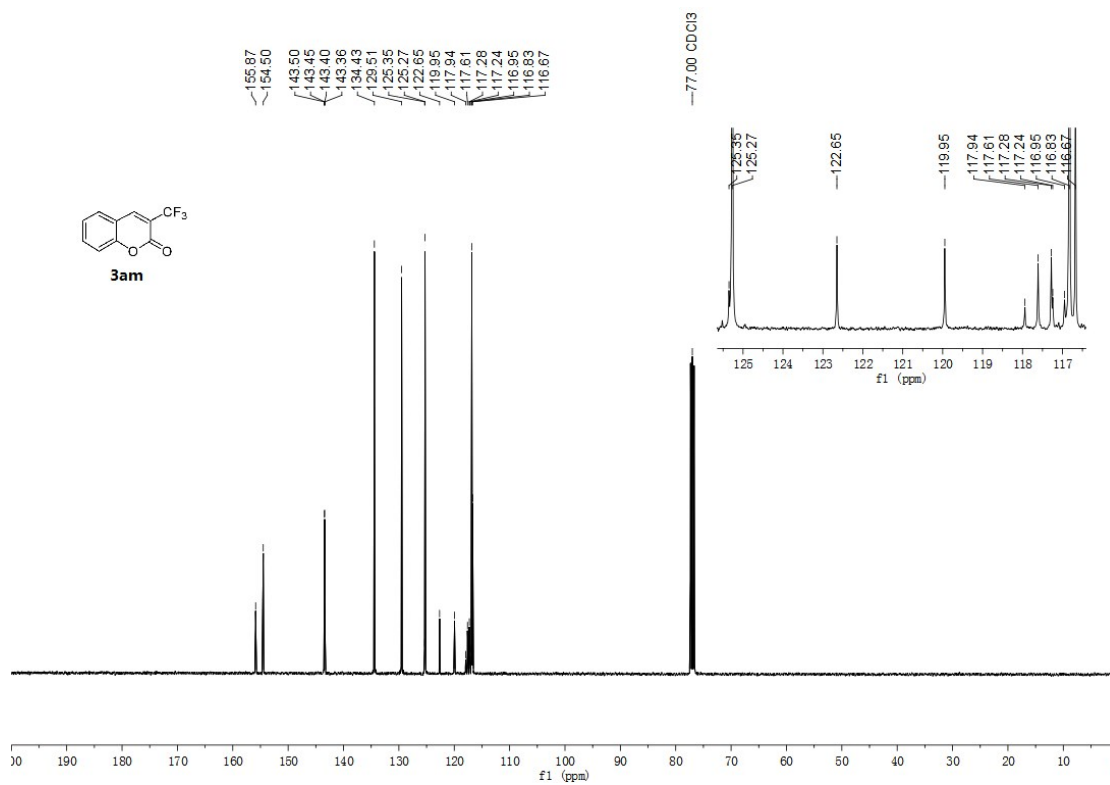
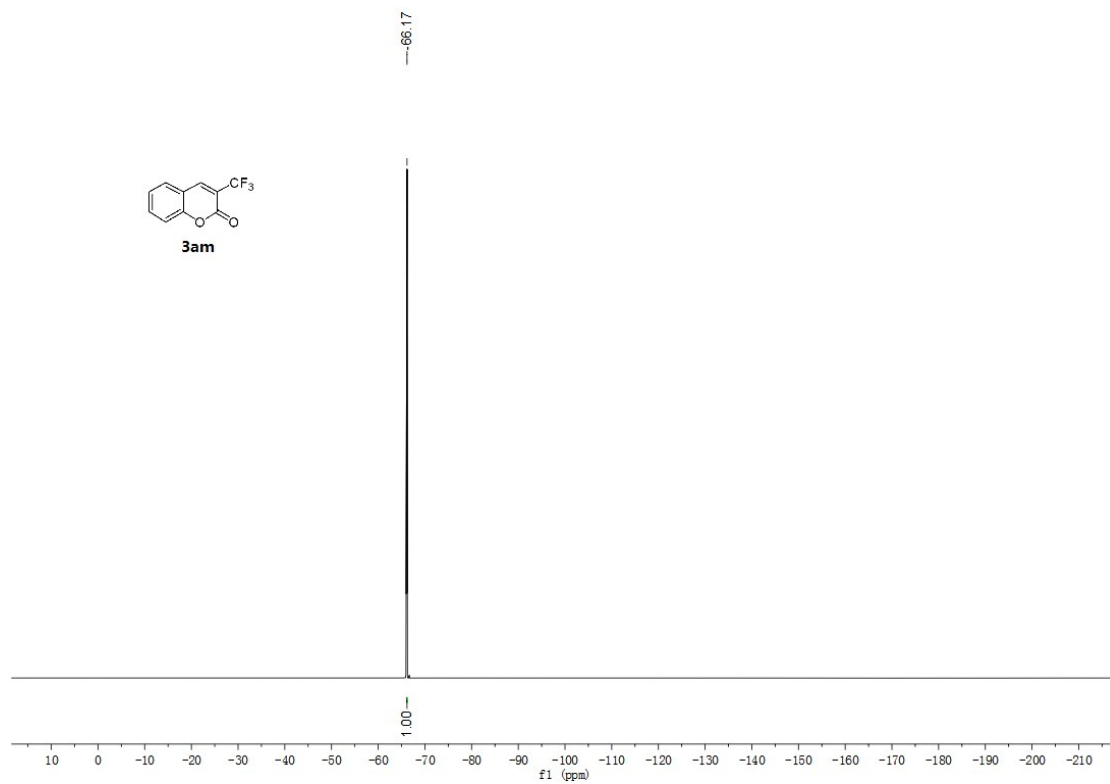


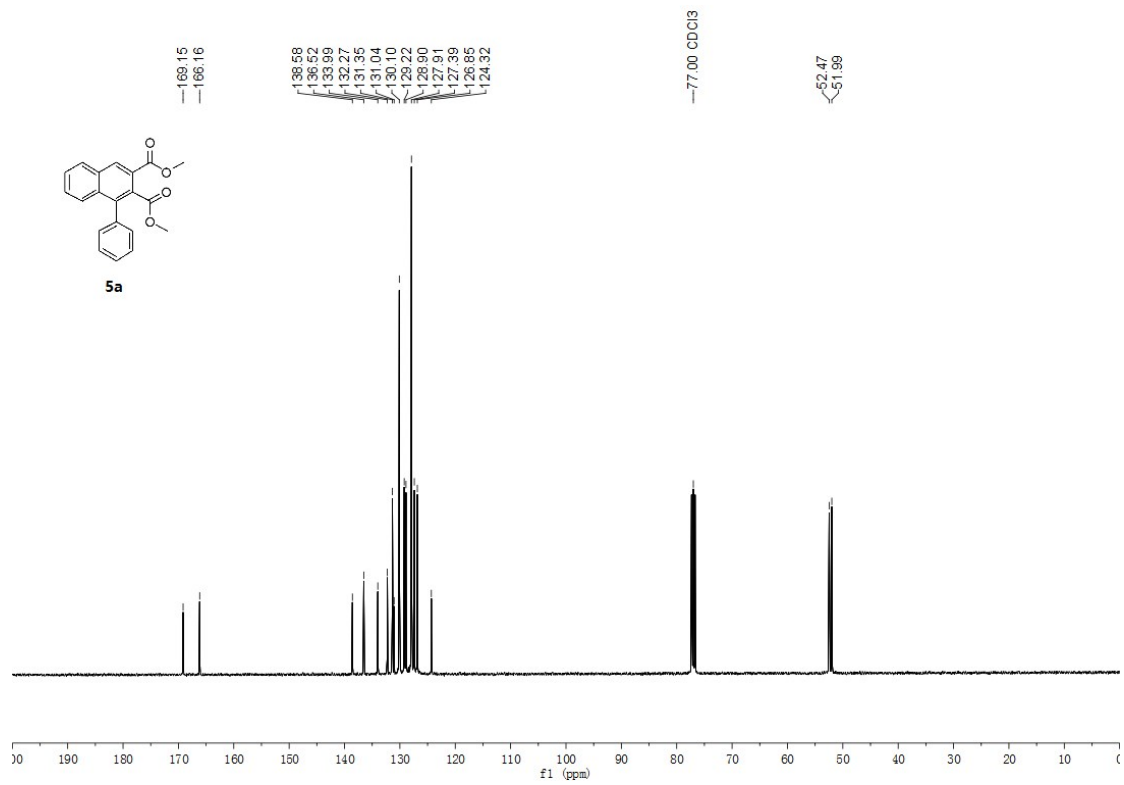
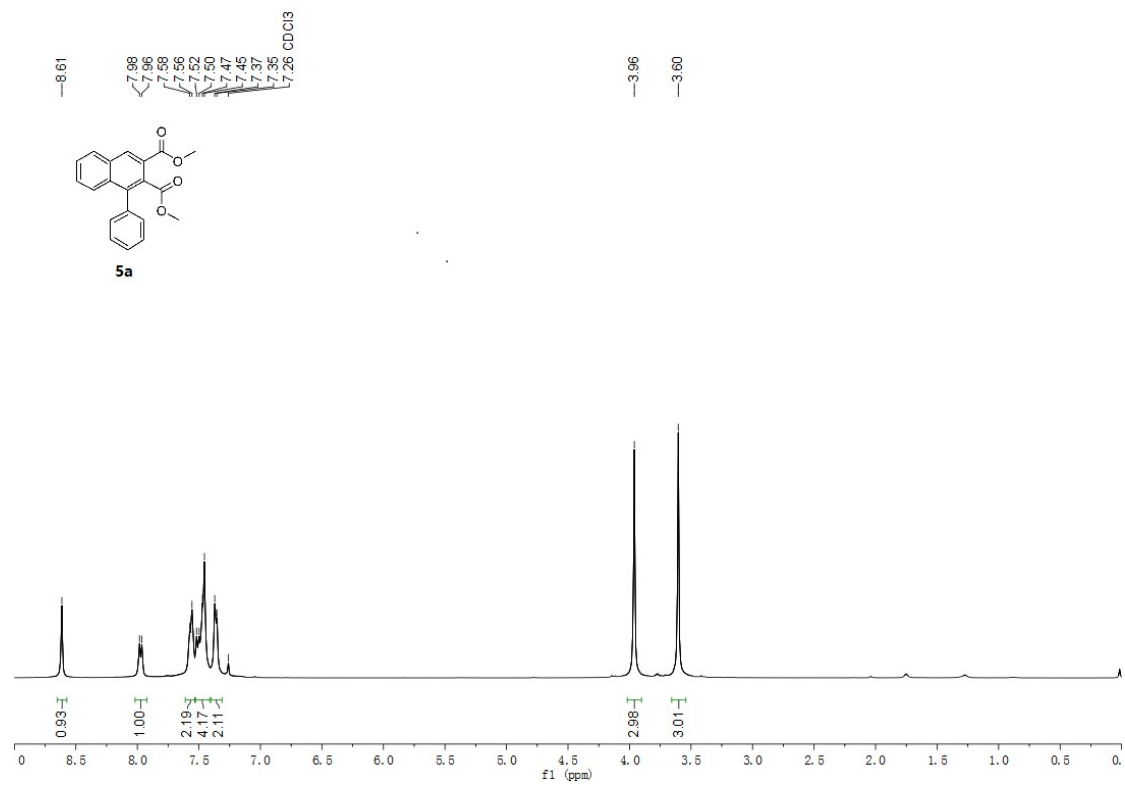


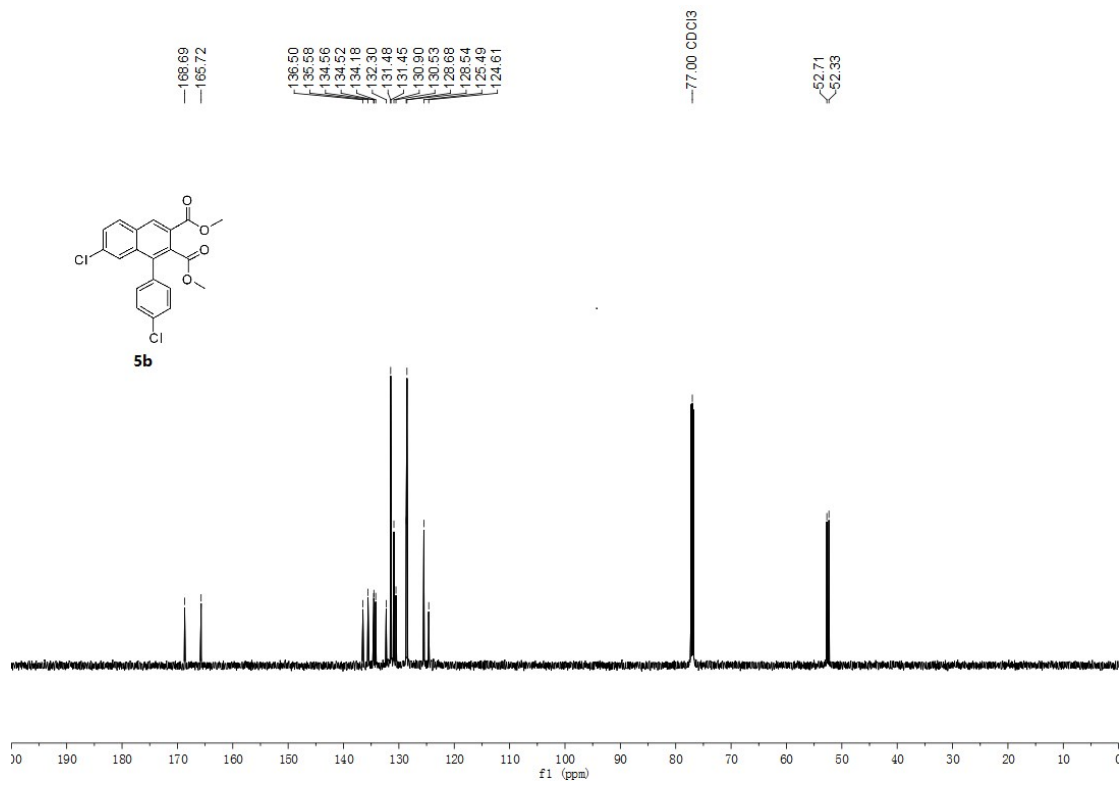
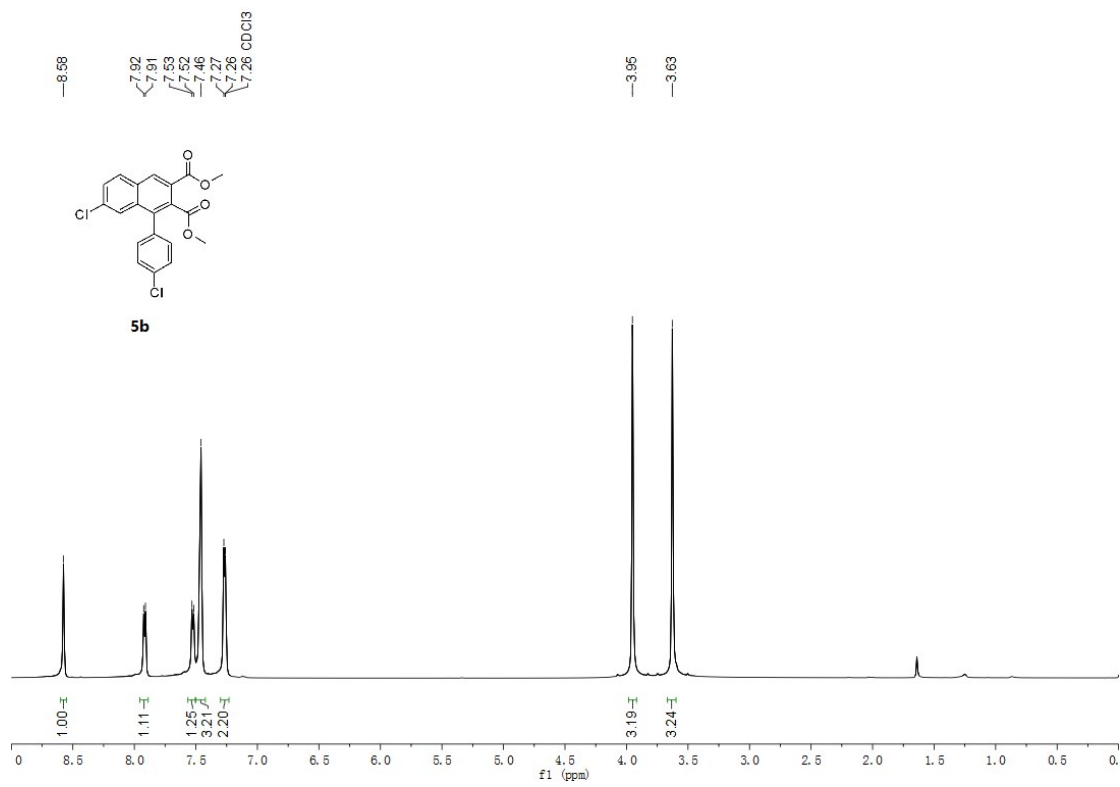


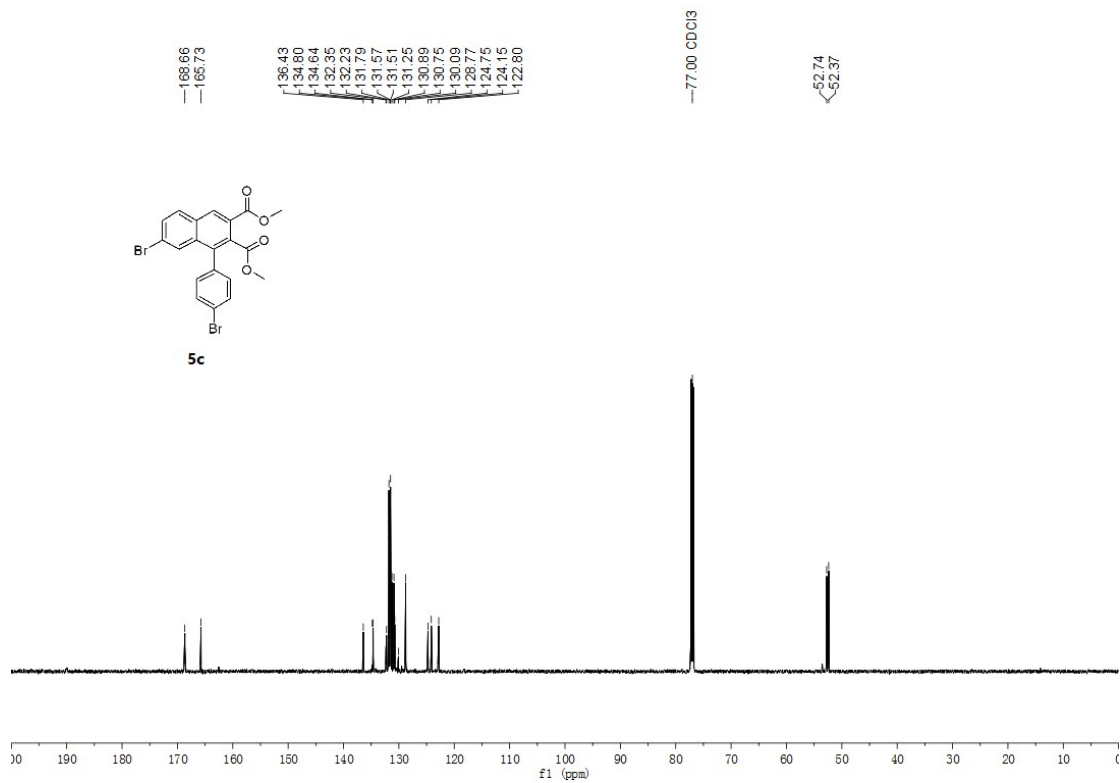
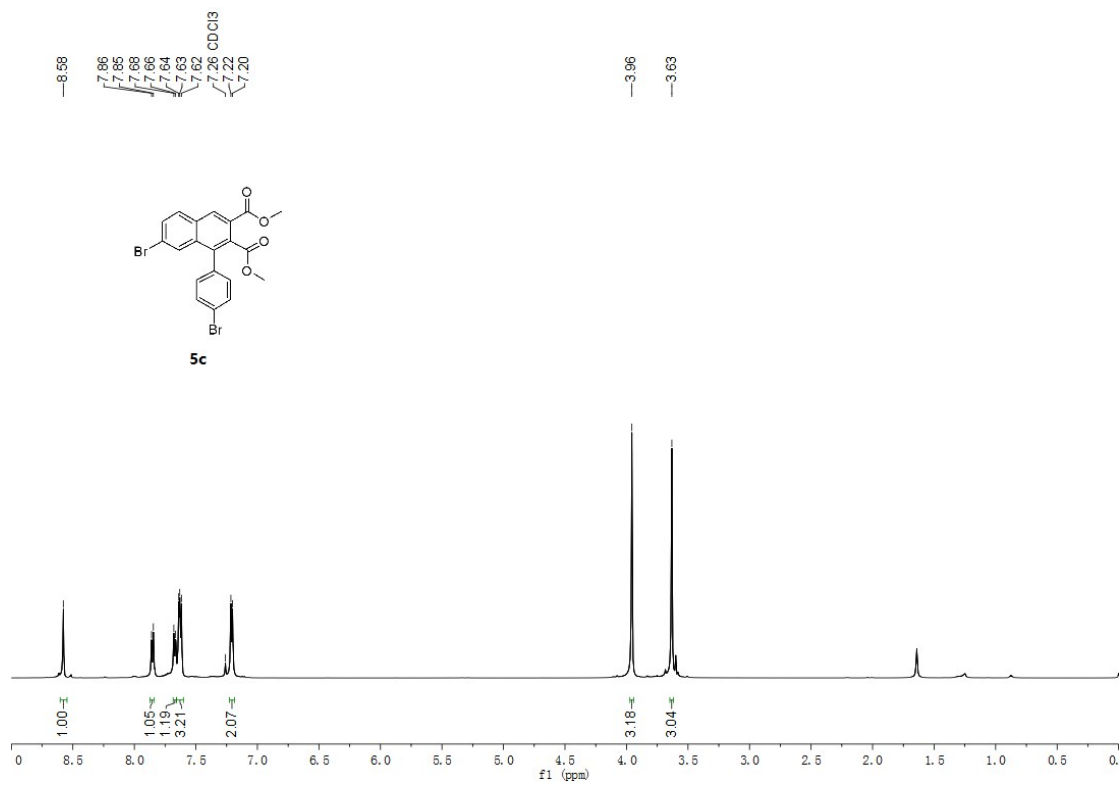


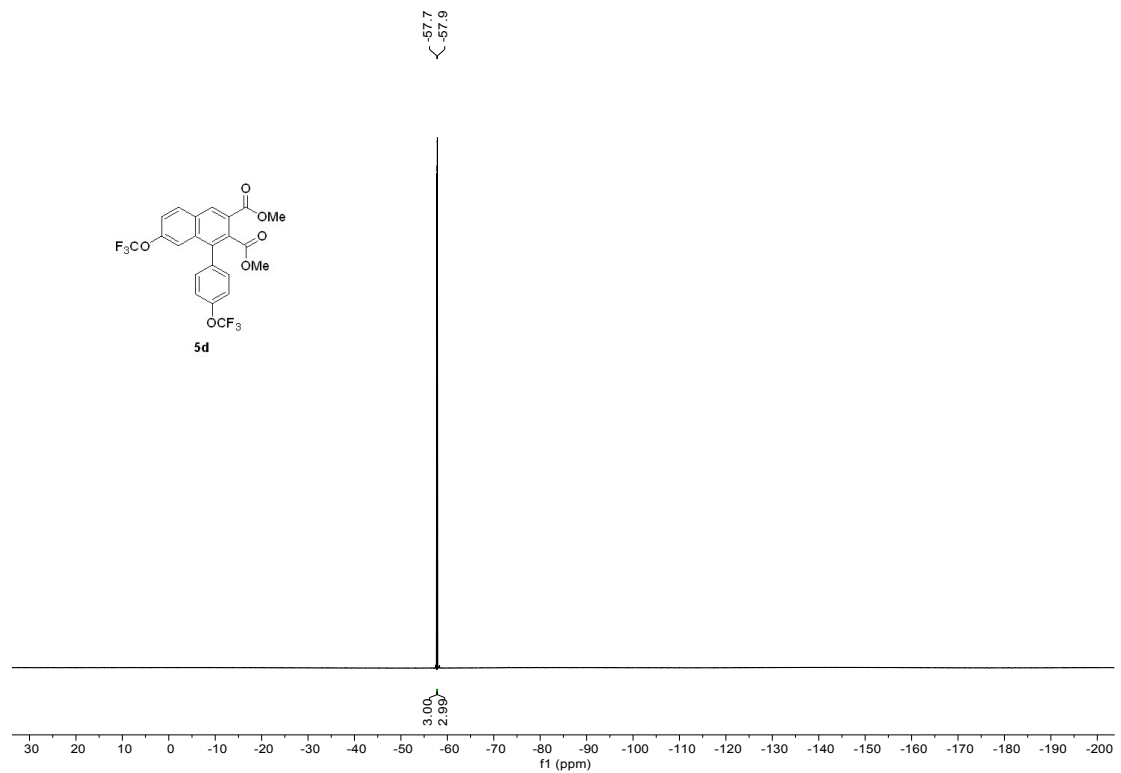
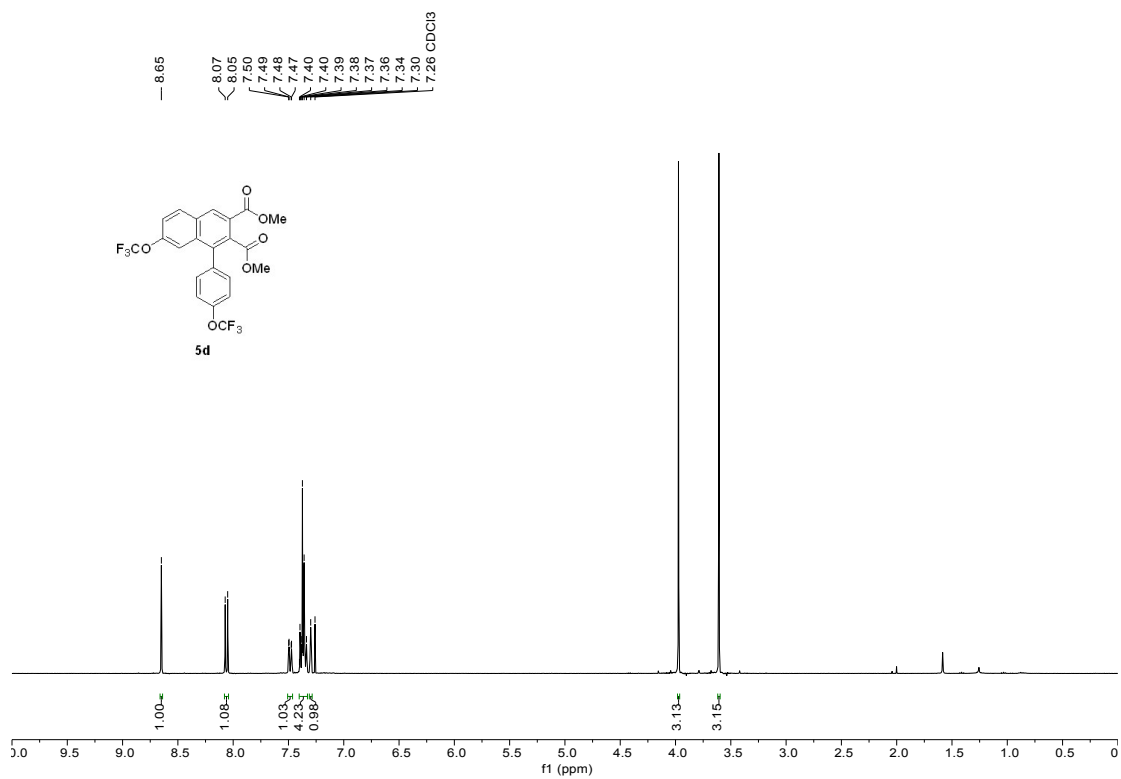


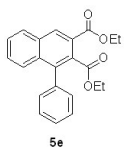
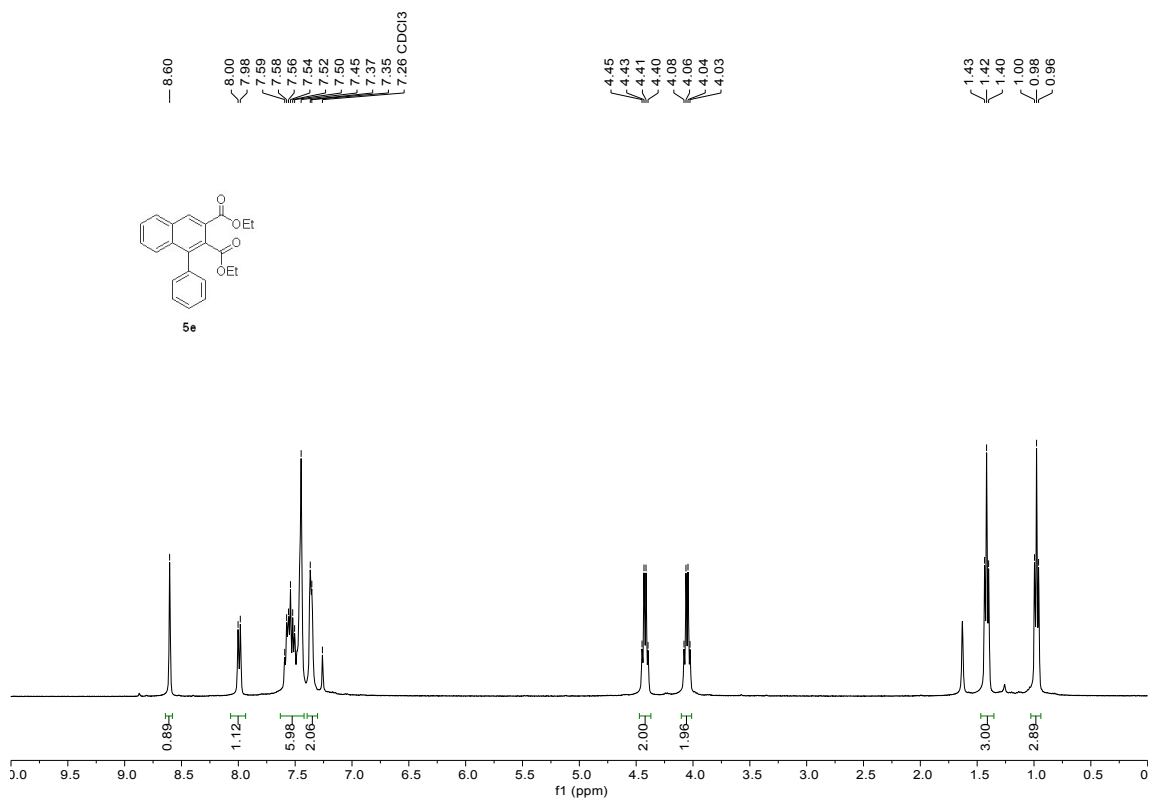
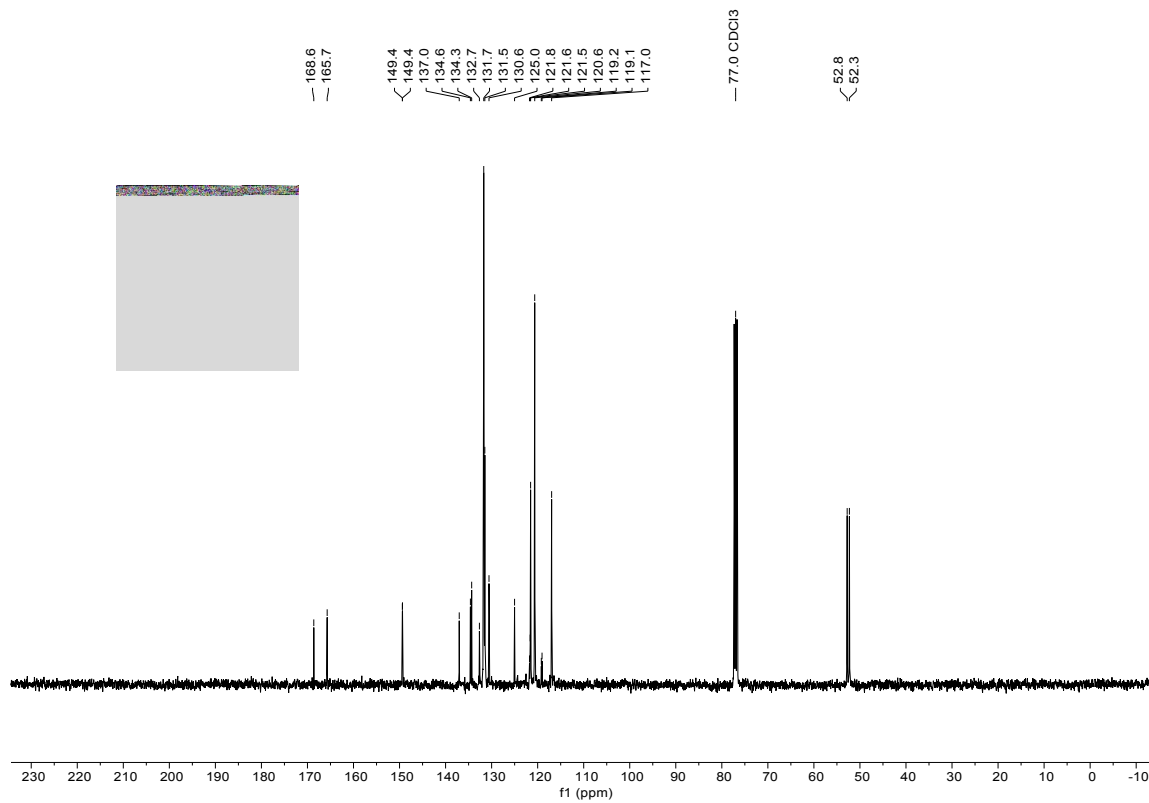


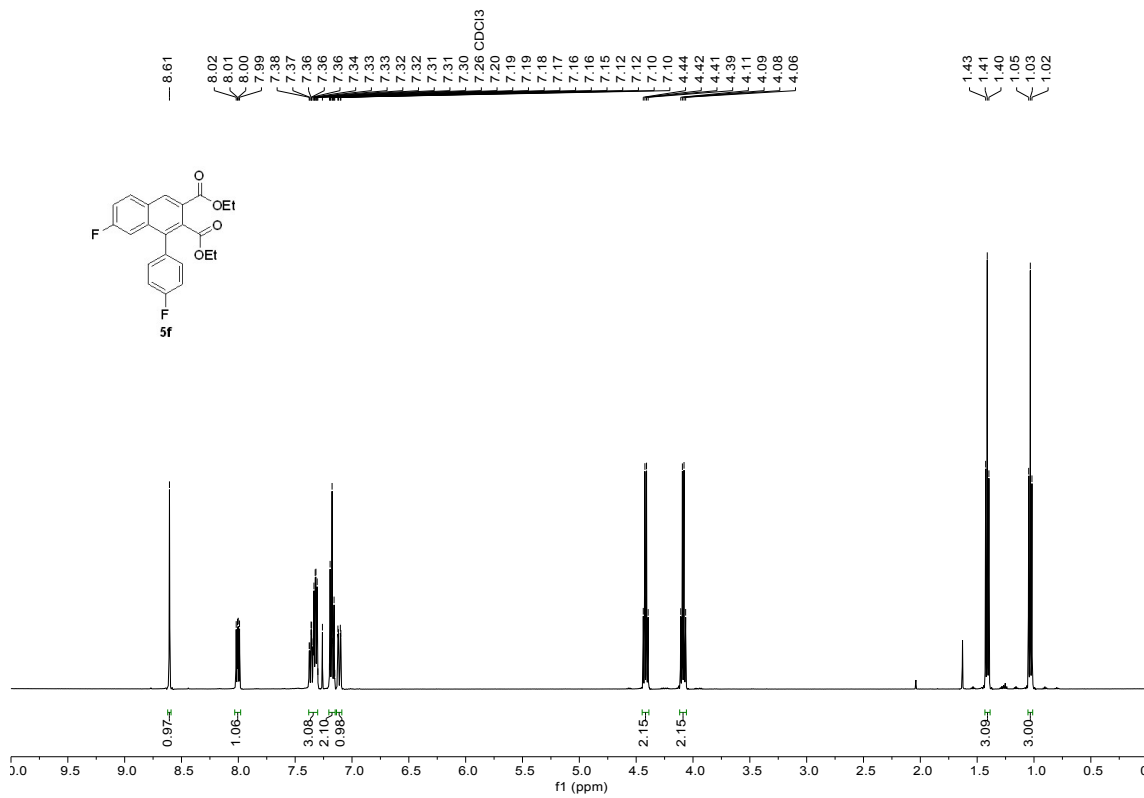
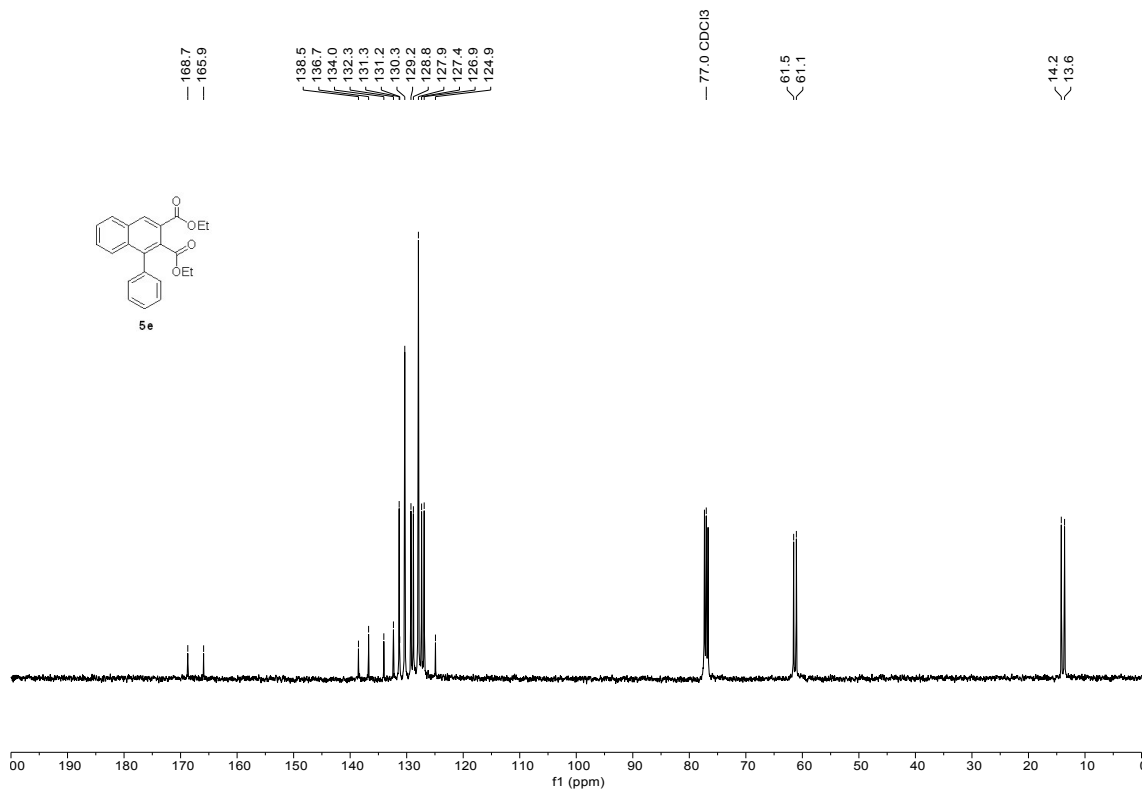


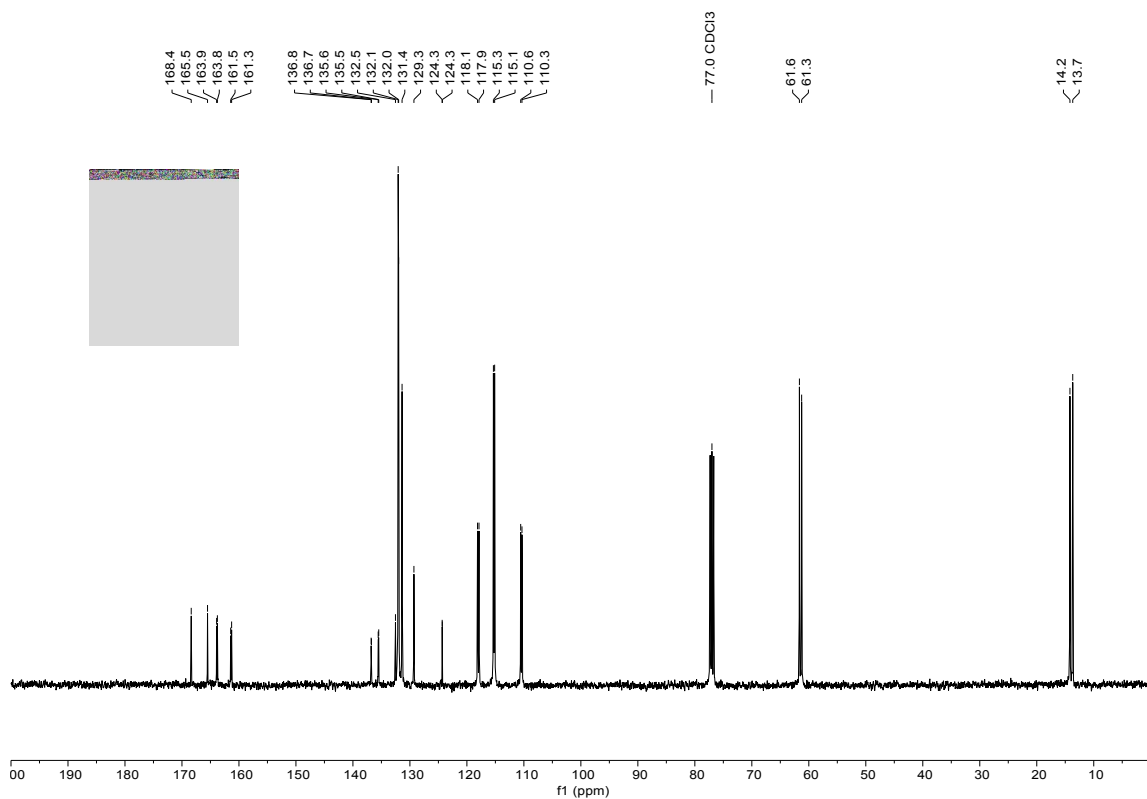
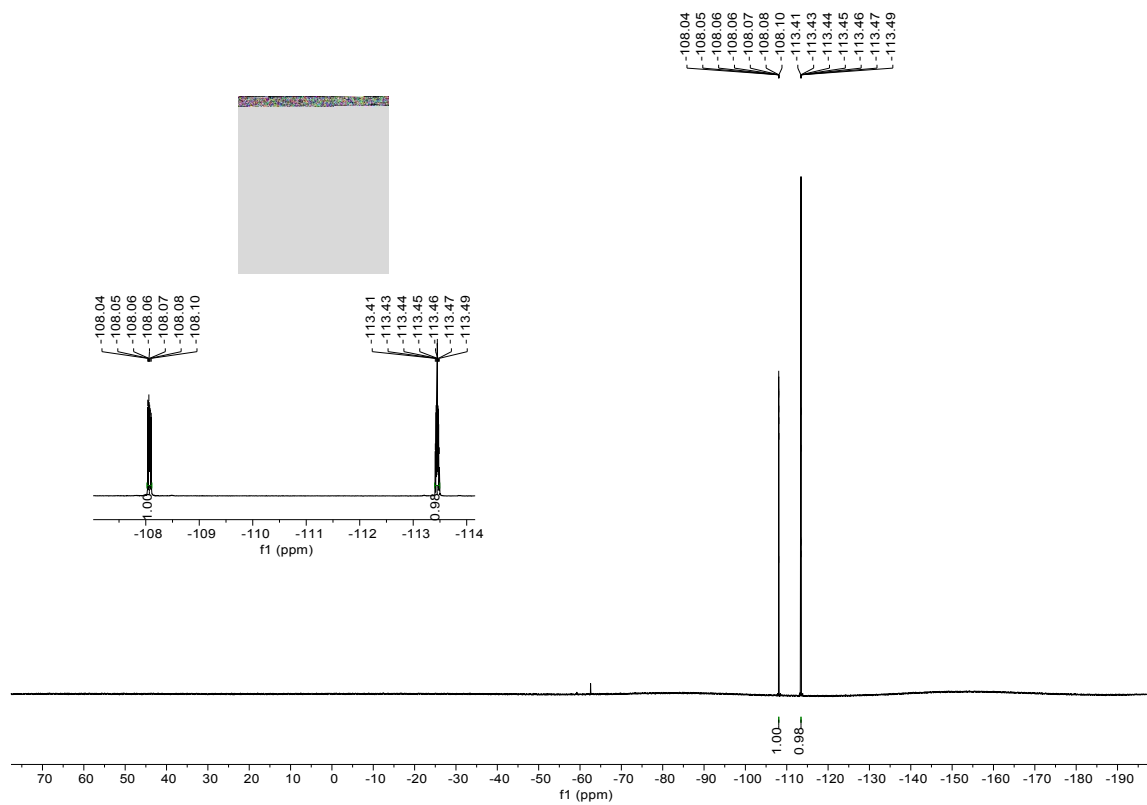


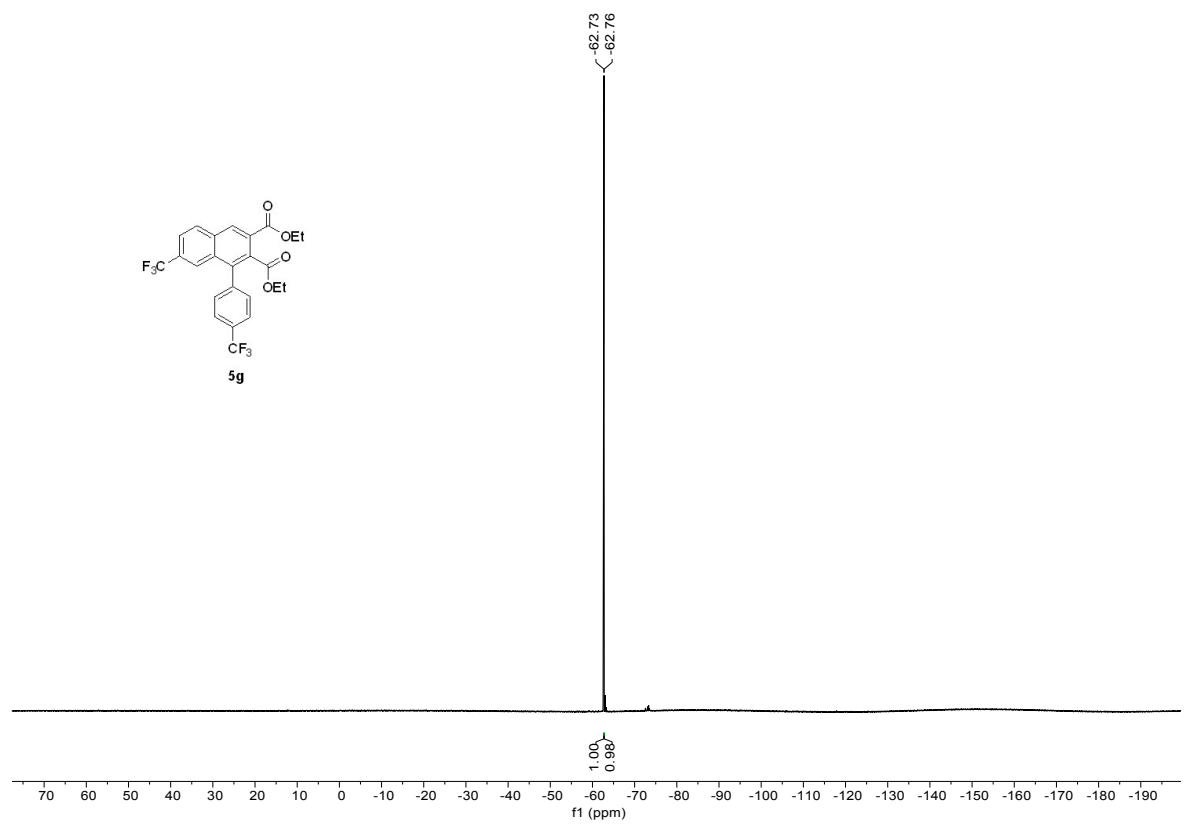
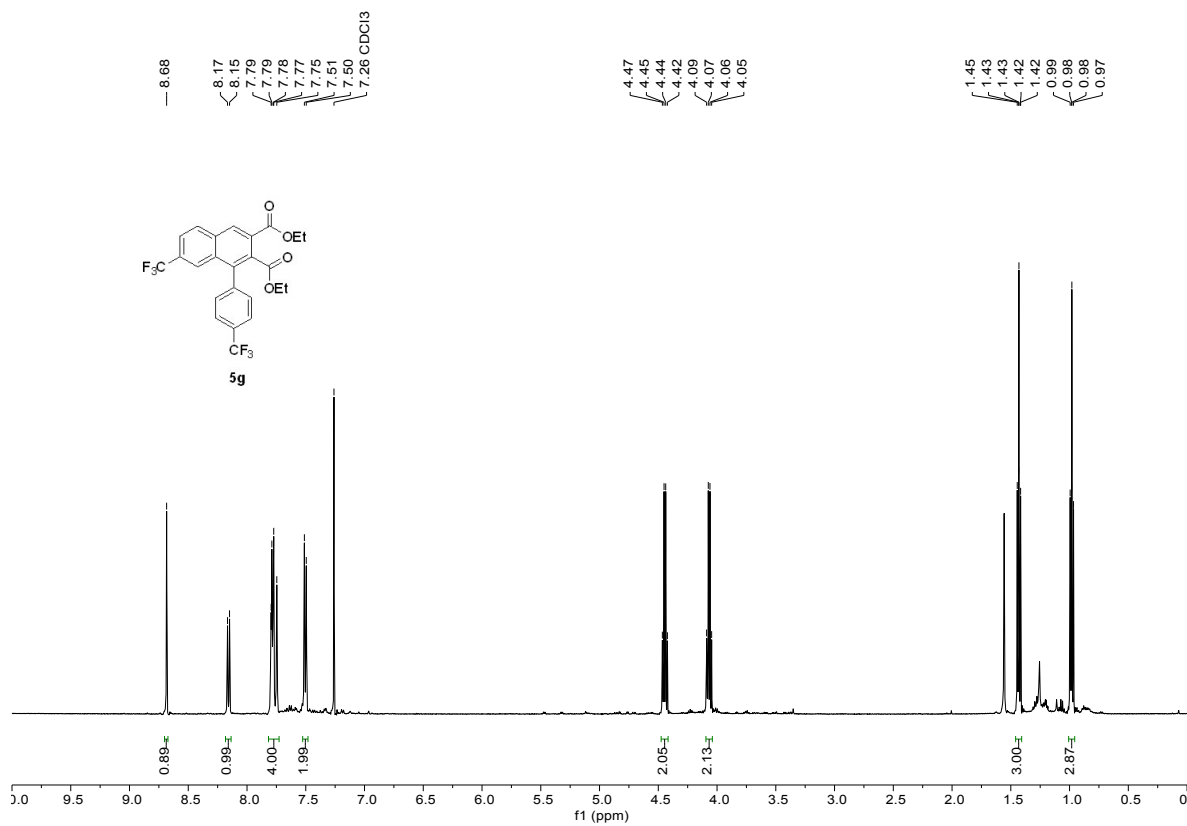


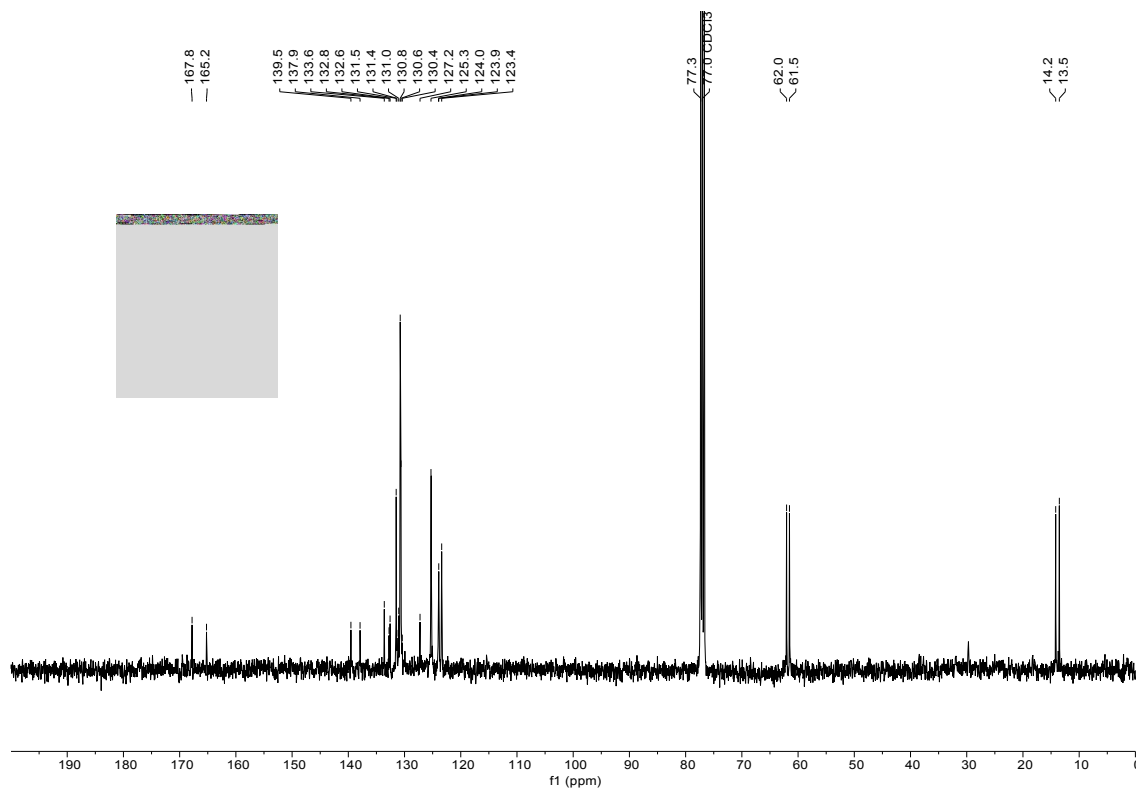












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