Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.


# Supporting information

## A Practical *ortho*-Acylation of Aryl Iodides via Palladium/Norbornene Catalysis Enabled by Moisture-insensitive Activated Esters

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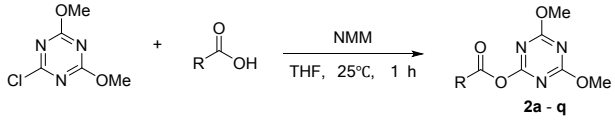
## 1. General Information

Organic solvents were used without further purification. Purifications of reactions products were carried out by flash chromatography using silica gel (200-300 m). $^{1}H$ NMR (400 MHz), $^{13}C$ NMR (100 MHz), $^{19}F$ NMR (376 MHz) were measured on a Brucker Avance 400 MHz spectrometer. Chemical shifts are reported in parts per million (ppm, δ) downfield from residual solvents peaks and coupling constants are reported as Hertz (Hz). Splitting patterns are designated as singlet (s), doublet (d), triplet (t), … Splitting patterns that could not be interpreted or easily visualized are designated as multiplet (m). Electrospray mass spectra were obtained using Bruker micrOTOF-Q II 10410 Mass Spectrometer. FT-IR spectra were obtained with a Nicolet 5700 spectrophotometer. Unless otherwise noted, all other commercially available reagents and solvents were used without further purification. All reactions were monitored by TLC.

## 2. Experimental Procedures

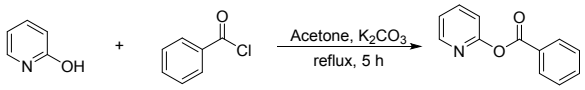
**General procedure for the synthesis of different triazine ester:** [1]




**2a - q**

To the THF (8 mL) solution of 2-chloro-4,6-dimethoxy-1,3,5-triazine (1.404g, 8 mmol, 1.0 equiv) and 4-Methylmorpholine (NMM) (1.012g, 10 mmol, 1.25 equiv), carboxylic acid (8 mmol, 1.0 equiv) in THF (8 mL) was added dropwise. The reaction mixture was stirred at room temperature for 1 h. The by-product, NMM-HCl was filtrated off, and the crude product was obtained by rotary evaporation to remove the solvent. The solid was dissolved in ethyl acetate, and then washed with 10% citric acid solution for three times, water, and 0.5 mol/L $NaHCO_3$, respectively. The organic layer was collected and dried by $Na_2SO_4$, and recrystallized to afford pure triazine esters **2a - q** (yield: 80-98%).

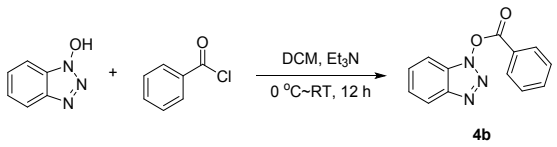
**General procedure for the synthesis of pyridine ester 4a:** [2]




**4a**

To the dry acetone (16 mL) solution of 2-hydroxypyridine (951.0 mg, 10 mmol, 1.0 equiv) and potassium (2.764 g, 20 mmol, 2.0 equiv) was added BzCl (2.811 g, 20 mmol, 2.0 equiv) dropwise under $N_2$ atmosphere. The reaction mixture was refluxed for 5 h. The reaction mixture was cooled to room temperature, washed with 1N hydrochloric acid, and dried over anhydrous $Na_2SO_4$. The residue was separated by column chromatography on a silica gel with eluent (petroleum ether/ethyl acetate = 15:1) to afford the corresponding pyridine ester **4a** as colorless oil (1.693 g, yield: 85%). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.35 (d, *J* = 4.4 Hz, 1H), 8.13 (d, *J* = 8.0 Hz, 2H), 7.73 (t, *J* = 7.6 Hz, 1H), 7.53 (t, *J* = 8.4 Hz, 1H), 7.40 (t, *J* = 7.6 Hz, 2H), 7.17-7.10 (m, 2H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 164.7, 158.1, 148.6, 139.5, 133.8, 130.3, 129.0, 128.5, 122.1, 116.6; HRMS (ESI) calcd for $C_{12}H_9NO_2$ $[M]^+$ 199.0633, found 199.0631.

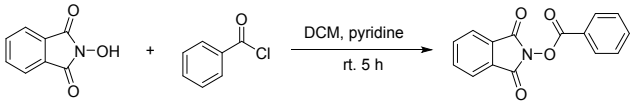
**General procedure for the synthesis of N-hydroxybenzotriazole ester 4b:**




**4b**

To a solution of 1*H*-benzo[*d*][1,2,3]triazol-1-ol (540.5 mg, 4 mmol, 1.0 equiv) and BzCl (618.6 mg, 4.4 mmol, 1.1 equiv) in DCM (10 mL) was added triethylamine (445.3 mg, 4.4 mmol, 1.1 equiv) dropwise for 15 minutes at 0 °C. The solvent was removed in vacuo after performing the reaction for 12 h under room temperature. The solid was dissolved in ethyl acetate, and then washed with 1N hydrochloric acid, water, and 0.5 mol/L $NaHCO_3$, respectively. The organic layer was collected and dried by $Na_2SO_4$, and recrystallized to afford pure activated ester product **4b** as white solid (0.775 g, yield: 81%). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.26 (d, *J* = 8.0 Hz, 2H), 8.10 (d, *J* = 8.8 Hz, 1H), 7.76 (t, *J* = 7.6 Hz, 1H), 7.60-7.52 (m, 3H), 7.48-7.41 (m, 2H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 162.7, 143.4, 135.5, 130.6, 129.1, 128.7, 128.7, 124.8, 124.6, 120.4, 108.3; HRMS (ESI) calcd for $C_{13}H_9N_3O_2$ $[M]^+$ 239.0695, found 239.0697.

**General procedure for the synthesis of N-hydroxysuccinimide ester 4c:** [3]

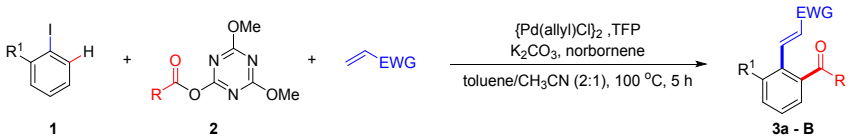



**4c**

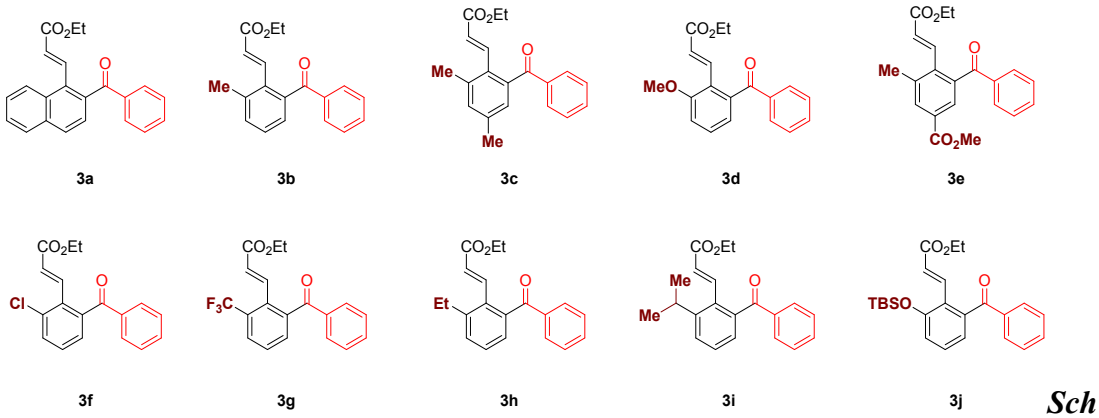
To a solution of 2-hydroxyisoindoline-1,3-dione (978.7 mg, 6 mmol, 1.0 equiv) and BzCl (927.8mg, 6.6 mmol, 1.1 equiv) in DCM (20 mL) was added pyridine (1.42 mL, 18 mmol, 3.0 equiv) drop wise for 15 minutes at room temperature. The solvent was removed in vacuo after performing the reaction for 5 h. Subsequently, distilled water (30 mL) with a drop of concentrated 1N hydrochloric acid was added to the crude mixture. The solid product was filtered and washed with water (twice) to afford pure N-hydroxysuccinimide ester **4c** as white solid (1.28g, yield: 80%). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.19 (d, *J* = 7.6 Hz, 2H), 7.93-7.91 (m, 2H), 7.82-7.80 (m, 2H),

7.70 (t, $J$ = 7.4 Hz, 1H), 7.54 (t, $J$ = 7.4 Hz, 2H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 162.8, 162.0, 134.9, 134.8, 130.6, 128.9, 128.8, 125.2, 124.0; HRMS (ESI) calcd for $C_{15}H_9NO_4$ $[M]^+$ 267.0532, found 267.0529.

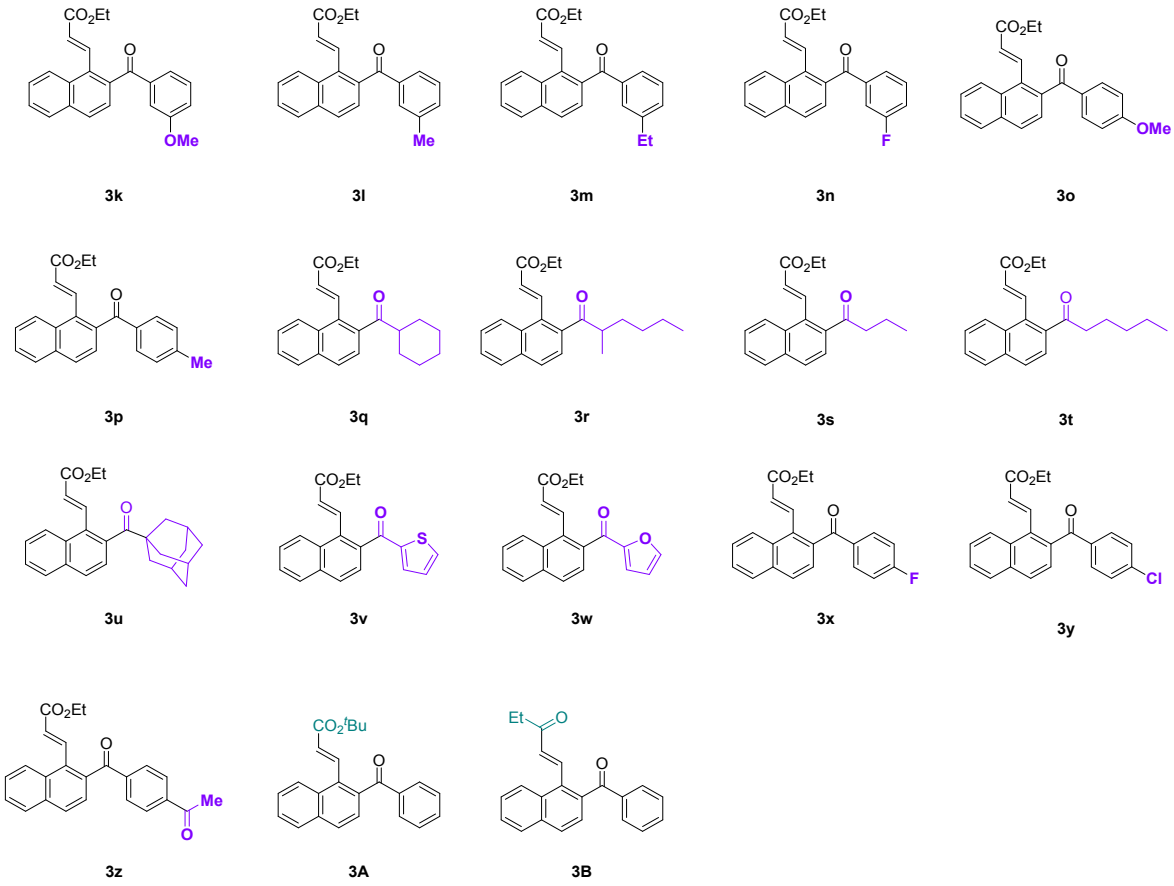
**Typical Experiment Procedure for isolated products:**




To a 25 mL of Schlenk tube were added triazine ester **2** (0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv). The mixture was evacuated and backfilled with $N_2$ for three times, aryl iodide **1** (0.20 mmol, 1.0 equiv), acrylate (0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv), toluene (1.35 mL) and acetonitrile (0.65 mL) were then added. The Schlenk tube was screw capped and put into a preheated oil bath (100 °C). After stirring for 5h, the reaction mixture was cooled to room temperature and purified with silica gel chromatography to give product **3a - B**.

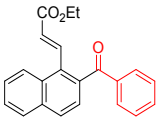



***Scheme S1.*** Reaction scope with respect to the aryl iodide.



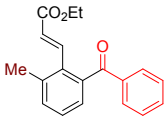

***Scheme S2.*** Scope of esters and alkenes.

## 3. Characterization Data




**Ethyl (E)-3-(2-benzoylnaphthalen-1-yl)acrylate (3a)**

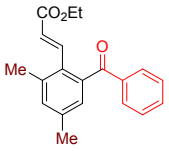
The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), ester **2a** (156.6 mg, 0.60 mmol, 3.0 equiv), {Pd(allyl)Cl}$_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3a** as colorless oil (60.6 mg, 92%) (petroleum ether/ethyl acetate = 25 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.18-8.10 (m, 2H), 7.92 (d, *J* = 8.8 Hz, 2H), 7.74 (d, *J* = 7.6 Hz, 2H), 7.63-7.58 (m, 2H), 7.57-7.50 (m, 2H), 7.41 (t, *J* = 7.8 Hz, 2H), 6.11 (d, *J* = 16.0 Hz, 1H), 4.17 (q, *J* = 7.2 Hz, 2H), 1.25 (t, *J* = 6.8 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 198.2, 165.5, 140.9, 137.5, 136.5, 133.8, 133.2, 132.0, 130.9, 129.8, 128.8, 128.5, 128.5, 127.4, 127.4, 126.7, 125.2, 124.9, 60.5, 14.1; IR (KBr) υ 3059, 2980, 1717, 1666, 1307, 1279, 1249, 1179, 1033, 969, 818, 751, 715 cm$^{-1}$; HRMS (ESI) calcd for $C_{22}H_{18}O_3$ $[M]^+$ 330.1256, found 330.1257.




**Ethyl (E)-3-(2-benzoyl-6-methylphenyl)acrylate (3b)**

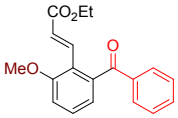
The reaction of 1-iodo-2-methylbenzene **1b** (43.6 mg, 0.20 mmol, 1.0 equiv), ester **2a** as colorless oil (156.6 mg, 0.60 mmol, 3.0 equiv), {Pd(allyl)Cl}$_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3b** (42.4 mg, 88%) (petroleum ether/ethyl acetate = 25 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.64 (t, *J* = 8.4 Hz, 3H), 7.45 (t, *J* = 7.8 Hz, 1H), 7.34-7.22 (m, 4H), 7.17 (d, *J* = 6.4 Hz, 1H), 5.79 (d, *J* = 16.0 Hz, 1H), 4.03 (q, *J* = 6.8 Hz, 2H), 2.33 (s, 3H), 1.13 (t, *J* = 6.8 Hz, 3H); **$^{13}$C NMR** (100 MHz,

$CDCl_3$): δ 198.3, 165.8, 141.7, 139.5, 137.5, 137.5, 133.3, 133.2, 132.1, 129.8, 128.4, 128.2, 126.4, 125.0, 60.4, 20.4, 14.1; IR (KBr) υ 3061, 2990, 1717, 1668, 1449, 1311, 1280, 1178, 1035, 973, 783, 714 $cm^{-1}$; HRMS (ESI) calcd for $C_{19}H_{18}O_3$ $[M]^+$ 294.1256, found 294.1250.




**Ethyl (E)-3-(2-benzoyl-4,6-dimethylphenyl)acrylate (3c)**

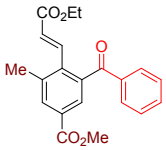
The reaction of 1-iodo-2,4-dimethylbenzene **1c** (50.8 mg, 0.20 mmol, 1.0 equiv), ester **2a** (156.6 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3c** as colorless oil (39.5 mg, 64%) (petroleum ether/ethyl acetate = 25 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.72-7.67 (m, 3H), 7.53 (t, $J$ = 7.6 Hz, 1H), 7.41-7.38 (m, 1H), 7.17 (s, 1H), 7.06 (s, 1H), 5.85 (d, $J$ = 16.4 Hz, 1H), 4.10 (q, $J$ = 6.8 Hz, 2H), 2.38 (s, 3H), 2.35 (s, 3H), 1.20 (t, $J$ = 7.2 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 198.6, 166.0, 141.5, 139.7, 138.5, 137.5, 137.5, 133.1, 133.0, 130.3, 129.8, 128.4, 126.9, 124.3, 60.3, 21.0, 20.4, 14.1; IR (KBr) υ 2980, 1716, 1667, 1448, 1305, 1176, 1037, 977, 721, 697 $cm^{-1}$; HRMS (ESI) calcd for $C_{20}H_{20}O_3$ $[M]^+$ 308.1412, found 308.1407.




**Ethyl (E)-3-(2-benzoyl-6-methoxyphenyl)acrylate (3d)**

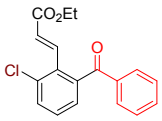
The reaction of 1-iodo-2-methoxybenzene **1d** (46.8 mg, 0.20 mmol, 1.0 equiv), ester **2a** (156.6 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3d** as white solid (55.8 mg, 89%) (petroleum ether/ethyl acetate =20: 1 as

eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.76 (d, *J* = 7.6 Hz, 2H), 7.65 (d, *J* = 16.0 Hz, 1H), 7.55 (t, *J* = 7.2 Hz, 1H), 7.43-7.37 (m, 3H), 7.06 (d, *J* = 8.4 Hz, 1H), 6.96 (d, *J* = 7.2 Hz, 1H), 6.44 (d, *J* = 16.4 Hz, 1H), 4.11 (q, *J* = 6.8 Hz, 2H), 3.93 (s, 3H), 1.20 (t, *J* = 7.2 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 197.7, 166.8, 158.7, 141.8, 137.8, 137.0, 133.5, 130.0, 128.5, 123.7, 121.8, 120.3, 112.3, 60.2, 55.7, 14.1; IR (KBr) υ 2982, 2925, 1711, 1670, 1630, 1584, 1449, 1285, 1173, 1068, 979, 889, 717 $cm^{-1}$; HRMS (ESI) calcd for $C_{19}H_{18}O_4$ $[M]^+$ 310.1205, found 310.1206.




**Methyl (E)-3-benzoyl-4-(3-ethoxy-3-oxoprop-1-en-1-yl)-5-methylbenzoate (3e)**

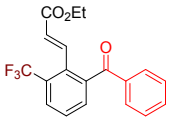
The reaction of methyl 4-iodo-3-methylbenzoate **1e** (55.2 mg, 0.20 mmol, 1.0 equiv), ester **2a** (156.6 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3e** as white solid (69.3 mg, 90%) (petroleum ether/ethyl acetate = 25 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.01 (s, 1H), 7.90 (s, 1H), 7.7.2-7.68 (m, 3H), 7.56 (t, *J* = 7.2 Hz, 1H), 7.41 (t, *J* = 7.6 Hz, 2H), 5.92 (d, *J* = 16.0 Hz, 1H), 4.12 (q, *J* = 7.2 Hz, 2H), 3.89 (s, 3H), 2.44 (s, 3H), 1.21 (t, *J* = 7.2 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 197.2, 165.9, 165.4, 140.7, 139.7, 138.0, 137.7, 137.0, 133.5, 132.7, 129.82, 129.6, 128.6, 127.2, 126.0, 60.6, 52.3, 20.4, 14.1; IR (KBr) υ 2954, 2926, 1721, 1670, 1442, 1307, 1249, 1178, 1033, 980, 769, 720 $cm^{-1}$; HRMS (ESI) calcd for $C_{21}H_{20}O_5$ $[M]^+$ 352.1311, found 352.1313.




**Ethyl (E)-3-(2-benzoyl-6-chlorophenyl)acrylate (3f)**

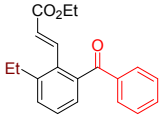
The reaction of 1-chloro-2-iodobenzene **1f** 47.7 mg, 0.20 mmol, 1.0 equiv), ester **2a** (156.6 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv),

norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3f** as colorless oil (30.6 mg, 37%) (petroleum ether/ethyl acetate = 25 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.75-7.69 (m, 3H), 7.59-7.55 (m, 2H), 7.45-7.35 (m, 4H), 7.33-7.31 (m, 1H), 6.00 (d, *J* = 16.4 Hz, 1H), 4,12 (q, *J* = 6.7 Hz, 2H), 1.22 (t, *J* = 7.2 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 196.7, 165.4, 141.1, 139.2, 136.8, 134.7, 133.7, 132.4, 131.4, 129.9, 129.5, 128.7, 127.0, 126.4, 60.6, 14.1; IR (KBr) υ 2955, 2924, 1715, 1667, 1954, 1448, 1265, 1176, 1029, 975, 711, 622 $cm^{-1}$; HRMS (ESI) calcd for $C_{18}H_{15}ClO_3$ $[M]^+$ 314.0710, found 314.0713.



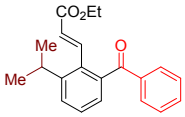

**Ethyl (E)-3-(2-benzoyl-6-(trifluoromethyl)phenyl)acrylate (3g)**

The reaction of 1-iodo-2-(trifluoromethyl)benzene **1g** (54.4 mg, 0.20 mmol, 1.0 equiv), ester **2a** (156.6 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3g** as colorless oil (30.6 mg, 42%) (petroleum ether/ethyl acetate = 25 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.86 (d, *J* = 7.6 Hz, 1H), 7.79 (d, *J* = 16.4 Hz, 1H), 7.67 (d, *J* = 8.0 Hz, 2H), 7.63 (d, *J* = 8.0 Hz, 1H), 7.59-7.55 (m, 2H), 7.44 (t, *J* = 7.2 Hz, 2H), 5.89 (d, *J* = 16.0 Hz, 1H), 4.11 (q, *J* = 7.2 Hz, 2H), 1.21 (t, *J* = 7.2 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 196.7, 164.9, 141.0, 138.9, 136.9, 133.8, 133.3, 131.8, 133.3 (q, *J* = 1.8 Hz), 131.8, 129.7, 129.6 (q, *J* = 30.2 Hz), 128.8, 128.4, 127.5 (q, *J* = 5.4 Hz), 127.4, 123.5 (q, *J* = 210.9 Hz), 60.7, 14.1; **$^{19}$F NMR** (376 MHz, $CDCl_3$): δ -59.4 (s, 3F); IR (KBr) υ 2959, 2930, 1720, 1672, 1597, 1329, 1312, 1278, 1171, 1128, 1033, 764, 714 $cm^{-1}$; HRMS (ESI) calcd for $C_{19}H_{15}F_3O_3$ $[M]^+$ 348.0973, found 348.0968.



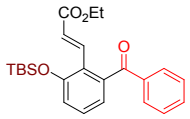

**Ethyl (E)-3-(2-benzoyl-6-ethylphenyl)acrylate (3h)**

The reaction of 1-ethyl-2-iodobenzene **1h** (46.4 mg, 0.20 mmol, 1.0 equiv), ester **2a** (156.6 mg, 0.60 mmol, 3.0 equiv), {Pd(allyl)Cl}$_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3h** as colorless oil (57.1 mg, 83%) (petroleum ether/ethyl acetate = 25 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.69 (d, *J* = 16.0 Hz, 1H), 7.61 (d, *J* = 8.0 Hz, 2H), 7.44 (t, *J* = 7.6 Hz, 1H), 7.33-7.28 (m, 4H), 7.20-7.15 (m, 1H), 5.77 (d, *J* = 16.0 Hz, 1H), 4.02 (q, *J* = 6.8 Hz, 2H), 2.65 (q, *J* = 7.6 Hz, 2H), 1.15-1.10 (m, 6H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 198.4, 165.7, 143.5, 141.6, 139.6, 137.5, 133.1, 132.8, 130.4, 129.7, 128.4, 126.3, 125.2, 60.4, 26.5, 15.1, 14.1; IR (KBr) υ 2970, 2934, 1718, 1448, 1310, 1276, 1179, 1034, 981, 763, 715 cm$^{-1}$; HRMS (ESI) calcd for $C_{20}H_{20}O_3$ $[M]^+$ 308.1412, found 308.1409.



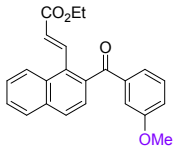

**Ethyl (E)-3-(2-benzoyl-6-isopropylphenyl)acrylate (3i)**

The reaction of 1-iodo-2-isopropylbenzene **1i** (49.22 mg, 0.20 mmol, 1.0 equiv), ester **2a** (156.6 mg, 0.60 mmol, 3.0 equiv), {Pd(allyl)Cl}$_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3i** as colorless oil (51.5 mg, 80%) (petroleum ether/ethyl acetate = 25 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.75 (d, *J* = 16.0 Hz, 1H), 7.63 (d, *J* = 7.6 Hz, 2H), 7.46 (d, *J* = 7.6 Hz, 1H), 7.40 (d, *J* = 8.0 Hz, 1H), 7.33 (t, *J* = 7.2 Hz, 3H), 7.19 (t, *J* = 4.0 Hz, 1H), 5.75 (d, *J* = 16.0 Hz, 1H), 4.04 (q, *J* = 7.2 Hz, 2H), 3.15-3.08 (m, 1H), 1.18 (d, *J* = 6.8 Hz, 6H), 1.14 (t, *J* = 7.2 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 198.4, 165.6, 147.9, 142.2, 139.5, 137.7, 133.1, 132.5, 129.7, 128.5, 128.4, 127.1, 126.2, 125.4, 60.4, 29.8, 23.5, 14.1; IR (KBr) υ 2956, 2924, 1719, 1669, 1460, 1376, 1310, 1286, 1176, 977, 761, 715, 670 cm$^{-1}$; HRMS (ESI) calcd for $C_{21}H_{22}O_3$ $[M]^+$ 322.1569, found 322.1570.




**Ethyl (E)-3-(2-benzoyl-6-((tert-butyldimethylsilyl)oxy)phenyl)acrylate (3j)**

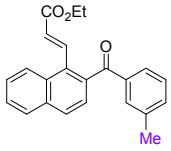
The reaction of tert-butyl((2-iodocyclohexa-2,4-dien-1-yl)oxy)dimethylsilane **1j** (69.66 mg, 0.20 mmol, 1.0 equiv), ester **2a** (156.6 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3j** as colorless oil (59.8 mg, 64%) (petroleum ether/ethyl acetate = 25 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 7.74-7.67 (m, 4H), 7.54 (t, *J* = 6.8 Hz, 1H), 7.46-7.38 (m, 3H), 7.35 (d, *J* = 7.6 Hz, 1H), 5.92 (d, *J* = 16.0 Hz, 1H), 4.75 (s, 3H), 4.12 (q, *J* = 7.2 Hz, 2H), 1.21 (t, *J* = 7.2 Hz, 3H), 0.95 (s, 9H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 198.0, 165.2, 140.6, 140.3, 139.3, 137.5, 133.2, 132.3, 129.8, 129.0, 128.4, 128.3, 127.5, 125.4, 62.8, 60.4, 25.8, 18.3, 14.10 -5.4; IR (KBr) υ 3462, 2928, 1776, 1717, 1665, 1995, 1449, 1285, 1179, 1027, 763, 714 $cm^{-1}$; HRMS (ESI) calcd for $C_{24}H_{30}O_4Si$ $[M]^+$ 410.1913, found 410.1916.




**Ethyl (E)-3-(2-(3-methoxybenzoyl)naphthalen-1-yl)acrylate (3k)**

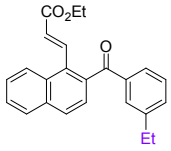
The reaction of 1-iodo-2,4-dimethylbenzene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), ester **2b** (174.8 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3k** as colorless oil (48.9 mg, 68%) (petroleum ether/ethyl acetate =20 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.08-8.01 (m, 2H), 7.82 (d, *J* = 8.0 Hz, 2H), 7.52-7.50 (m, 2H), 7.42 (d, *J* = 8.4 Hz, 2H), 7.28 (s, 1H), 7.19 (t, *J* = 7.6 Hz, 1H), 7.10 (d, *J* = 6.8 Hz, 1H), 7.00 (d, *J* = 8.0 Hz, 1H), 6.00 (d, *J* = 16.0 Hz, 1H), 4.09 (q, *J*

= 7.2 Hz, 2H), 3.72 (s, 3H), 1.17 (t, $J$ = 7.2 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 198.1, 165.6, 159.7, 140.9, 138.9, 136.6, 133.8, 132.0, 130.9, 129.5, 128.8, 128.5, 127.4, 126.7, 125.3, 124.9, 122.9, 119.9, 113.5, 60.5, 55.4, 14.1; IR (KBr) υ 2960, 1715, 1667, 1594, 1368, 1283, 1179, 1036, 978, 878, 799, 759, 685 $cm^{-1}$; HRMS (ESI) calcd for $C_{23}H_{20}O_4$ $[M]^+$ 360.1362, found 360.1360.




**Ethyl (E)-3-(2-(3-methylbenzoyl)naphthalen-1-yl)acrylate (3l)**

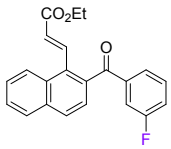
The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), ester **2c** (181.9 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3l** as colorless oil (59.0 mg, 86%) (petroleum ether/ethyl acetate = 25 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.11-8.03 (m, 2H), 7.87-7.84 (m, 2H), 7.55-7.52 (m, 3H), 7.44-7.42 (m, 2H), 7.29 (d, $J$ = 8.0 Hz, 1H), 7.23-7.19 (m, 1H), 6.04 (d, $J$ = 8.4 Hz, 1H), 4.11 (q, $J$ = 7.2 Hz, 2H), 2.28 (s, 3H), 1.19 (t, $J$ = 7.6 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 198.4, 165.6, 140.9, 138.3, 137.5, 136.7, 134.1, 133.8, 131.9, 130.9, 130.1, 128.8, 128.5, 128.3, 127.4, 127.3, 127.2, 126.6, 125.3, 124.9, 60.5, 21.2, 14.1; IR (KBr) υ 2979, 2926, 1717, 1666, 1601, 1464, 1369, 1281, 1179, 1035, 975, 800, 755, 685 $cm^{-1}$; HRMS (ESI) calcd for $C_{23}H_{20}O_3$ $[M]^+$ 344.1412, found 344.1415.




**Ethyl (E)-3-(2-(3-ethylbenzoyl)naphthalen-1-yl)acrylate (3m)**

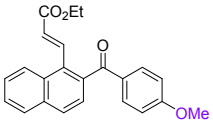
The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), ester **2d** (173.4 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv),

norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3m** as colorless oil (68.4 mg, 87%) (petroleum ether/ethyl acetate = 25 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.11-8.04 (m, 2H), 7.86-7.83 (m, 2H), 7.60 (d, *J* = 8.0 Hz, 2H), 7.55-7.51 (m, 2H), 7.42 (d, *J* = 8.4 Hz, 1H), 7.16 (d, *J* = 8.0 Hz, 2H), 6.05 (d, *J* = 16.0 Hz, 1H), 4.10 (q, *J* = 7.2 Hz, 2H), 2.62 (q, *J* = 7.6 Hz, 2H), 1.20-1.16 (m, 6H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 197.9, 165.7, 150.4, 140.9, 136.9, 135.1, 133.8, 131.7, 131.0, 130.2, 128.8, 128.5, 128.0, 127.4, 127.3, 126.6, 125.3, 124.9, 60.5, 28.9, 15.1, 14.1; IR (KBr) υ 2968, 2933, 1717, 1665, 1604, 1462, 1370, 1279, 1034, 970, 819, 762 $cm^{-1}$; HRMS (ESI) calcd for $C_{24}H_{22}O_3$ $[M]^+$ 358.1569, found 358.1574.



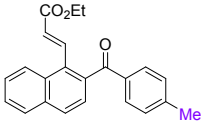

**Ethyl (E)-3-(2-(3-fluorobenzoyl)naphthalen-1-yl)acrylate (3n)**

The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), ester **2e** (167.4 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3n** as colorless oil (57.1 mg, 82%) (petroleum ether/ethyl acetate = 25 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.13-8.06 (m, 2H), 7.91 (d, *J* = 8.4 Hz, 2H), 7.61-7.56 (m, 2H), 7.48 (d, *J* = 8.4 Hz, 1H), 7.44-7.41 (m, 2H), 7.37-7.31 (m, 1H), 7.24-7.20 (m, 1H), 6.04 (d, *J* = 16.0 Hz, 1H), 4.15 (q, *J* = 7.2 Hz, 2H), 1.23 (t, *J* = 7.2 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 197.0, 165.5, 163.9, 161.4, 140.8, 139.8, 139.7, 135.9, 134.0, 132.3, 130.9, 130.3, 130.2, 129.1, 128.5, 127.6, 127.6, 127.1, 125.7, 125.6, 125.3, 124.8, 120.4, 120.2, 116.3, 116.0, 60.6, 14.1; **$^{19}$F NMR** (376 MHz, $CDCl_3$): δ -111.7 (s, 1F); IR (KBr) υ 3064, 2926, 1716, 1669, 1587, 1442, 1368, 1256, 1179, 1033, 978, 884, 805, 759 $cm^{-1}$; HRMS (ESI) calcd for $C_{22}H_{17}FO_3$ $[M]^+$ 348.1162, found 348.1165.




**Ethyl (E)-3-(2-(4-methoxybenzoyl)naphthalen-1-yl)acrylate (3o)**

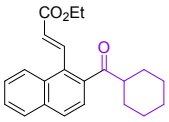
The reaction of 1-iodo-2,4-dimethylbenzene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), ester **2f** (174.8 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3o** as white solid (44.7 mg, 62%) (petroleum ether/ethyl acetate =20 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.18-8.11 (m, 2H), 7.94-7.91 (m, 2H), 7.72 (d, *J* = 8.4 Hz, 2H), 7.63-7.58 (m, 2H), 7.48 (d, *J* = 8.4 Hz, 1H), 6.89 (d, *J* = 8.4 Hz, 2H), 6.13 (d, *J* = 16.0 Hz, 1H), 4.18 (q, *J* = 7.2 Hz, 2H), 3.86 (s, 3H), 1.26 (t, *J* = 7.2 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 196.9, 165.8, 163.8, 140.9, 137.1, 133.7, 132.3, 131.3, 131.0, 130.3, 128.9, 128.5, 127.4, 127.2, 126.5, 125.2, 124.8, 113.8, 60.6, 55.5, 14.2; IR (KBr) υ 2958, 2926, 1716, 1658, 1597, 1509, 1463, 1257, 1179, 1029, 970, 821, 767 $cm^{-1}$; HRMS (ESI) calcd for $C_{23}H_{20}O_4$ $[M]^+$ 360.1362, found 360.1367.




**Ethyl (E)-3-(2-(4-methylbenzoyl)naphthalen-1-yl)acrylate (3p)**

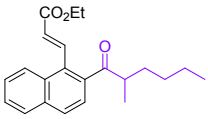
The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), ester **2g** (174.6 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3p** as colorless oil (57.8 mg, 84%) (petroleum ether/ethyl acetate = 25 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.18-8.11 (m, 2H),7.94-7.91 (m, 2H), 7.65-7.59 (m, 4H), 7.49 (d, *J* = 8.4 Hz, 1H), 7.21 (d, *J* = 8.0 Hz, 2H), 6.12 (d, *J* = 16.0 Hz, 1H), 4.18 (q, *J* = 7.2 Hz, 2H), 2.40 (s, 3H), 1.26 (t, *J* = 7.2 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 198.0, 165.7, 144.3, 141.0, 136.9, 135.0, 133.8, 131.7, 131.0, 130.1,

129.3, 128.8, 128.6, 127.4, 127.3, 126.6, 125.3, 124.9, 60.6, 21.7, 14.2; IR (KBr) υ 2923, 2857, 1714, 1659, 1603, 1461, 1377, 1263, cm$^{-1}$; HRMS (ESI) calcd for $C_{23}H_{20}O_3$ [M]$^+$ 344.1412, found 344.1411.




**Ethyl (E)-3-(2-(cyclohexanecarbonyl)naphthalen-1-yl)acrylate (3q)**

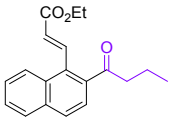
The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), ester **2h** (160.4 mg, 0.60 mmol, 3.0 equiv), {Pd(allyl)Cl}$_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3q** as colorless oil (57.9 mg, 86%) (petroleum ether/ethyl acetate = 25 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.38 (d, *J* = 16.0 Hz, 1H), 8.10 (t, *J* = 4.0 Hz, 1H), 7.86 (d, *J* = 8.4 Hz, 2H), 7.56 (t, *J* = 3.2 Hz, 2H), 7.47 (d, *J* = 8.4 Hz, 1H), 6.10 (d, *J* = 16.0 Hz, 1H), 4.30 (q, *J* = 7.2 Hz, 2H), 2.90 (t, *J* = 11.6 Hz, 1H), 1.80-1.66 (m, 5H), 1.45 (q, *J* = 11.6 Hz, 2H), 1.34 (t, *J* = 7.2 Hz, 3H), 1.28-1.18 (m, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 210.0, 165.8, 141.9, 137.6, 133.8, 131.3, 130.9, 129.0, 128.3, 127.3, 127.2, 126.6, 125.3, 124.1, 60.7, 50.6, 29.0, 25.7, 25.5, 14.2; IR (KBr) υ 2931, 2854, 1718, 1639, 1449, 1369, 1306, 1263, 1177, 1035, 980, 824, 767 cm$^{-1}$; HRMS (ESI) calcd for $C_{22}H_{24}O_3$ [M]$^+$ 336.1725, found 336.1725.




**Ethyl (E)-3-(2-(2-methylhexanoyl)naphthalen-1-yl)acrylate (3r)**

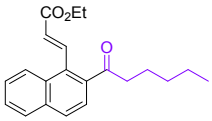
The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), ester **2i** (161.6 mg, 0.60 mmol, 3.0 equiv), {Pd(allyl)Cl}$_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3r** as colorless oil (40.2 mg, 60%) (petroleum ether/ethyl acetate = 25 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.38 (d, *J* = 16.4 Hz, 1H), 8.12 (t, *J* = 5.2 Hz,

1H), 7.90-7.89 (m, 2H), 7.59-7.57 (m, 2H), 7.52 (d, $J$ = 8.4 Hz, 1H), 6.10 (d, $J$ = 16.4 Hz, 1H), 4.34-4.26 (m, 2H), 3.15-3.07 (m, 1H), 1.71-1.65 (m, 1H), 1.42-1.33 (m, 4H), 1.25 (s, 4H), 1.13 (d, $J$ = 6.8 Hz, 3H), 0.84 (t, $J$ = 6.4 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 210.5, 165.8, 142.1, 137.7, 134.0, 131.7, 131.0, 129.1, 128.4, 127.5, 127.3, 126.5, 125.6, 124.4, 60.8, 45.9, 33.0, 29.3, 22.7, 16.6, 14.3, 13.9; IR (KBr) υ 2959, 2859, 1719, 1640, 1462, 1373, 1305, 1263, 1177, 1036, 980, 822, 761 cm$^{-1}$; HRMS (ESI) calcd for $C_{22}H_{26}O_3$ $[M]^+$ 338.1882, found 338.1879.




**Ethyl (E)-3-(2-butyrylnaphthalen-1-yl)acrylate (3s)**

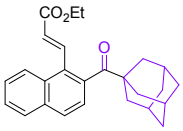
The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), ester **2k** (136.3 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3s** as colorless oil (49.8 mg, 84%) (petroleum ether/ethyl acetate = 30 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.37 (d, $J$ = 16.0 Hz, 1H), 8.13 (d, $J$ = 8.4 Hz, 1H), 7.88 (d, $J$ = 8.0 Hz, 2H), 7.58 (d, $J$ = 7.2 Hz, 3H), 6.09 (d, $J$ = 16.0 Hz, 1H), 4.31 (q, $J$ = 7.1 Hz, 2H), 2.83 (t, $J$ = 7.2 Hz, 2H), 1.72 (q, $J$ = 7.3 Hz, 2H), 1.36 (t, $J$ = 7.0 Hz, 3H), 0.96 (t, $J$ = 7.4 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 206.0, 165.9, 142.3, 137.1, 134.1, 132.1, 131.0, 129.1, 128.3, 127.5, 127.3, 126.1, 125.8, 124.0, 60.7, 44.9, 18.0, 14.3, 13.7; IR (KBr) υ 2964, 2875, 1717, 1639, 1463, 1368, 1265, 1177, 1037, 983, 817, 751 cm$^{-1}$; HRMS (ESI) calcd for $C_{19}H_{20}O_3$ $[M]^+$ 296.1412, found 296.1416.




**Ethyl (E)-3-(2-hexanoylnaphthalen-1-yl)acrylate (3t)**

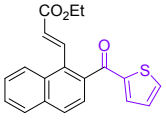
The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), ester **2j** (152.6 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0

equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3t** as colorless oil (56.3 mg, 87%) (petroleum ether/ethyl acetate = 30 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.37 (d, *J* = 16.0 Hz, 1H), 8.14-8.12 (m, 1H), 7.88 (d, *J* = 8.4 Hz, 2H), 7.59-7.57 (m, 3H), 6.10 (d, *J* = 16.0 Hz, 1H), 4.31 (q, *J* = 7.1 Hz, 2H), 2.84 (t, *J* = 7.4 Hz, 2H), 1.71-1.68 (m, 2H), 1.38-1.31 (m, 7H), 0.89 (t, *J* = 6.4 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 206.2, 165.9, 142.3, 137.1, 134.1, 132.1, 131.0, 129.1, 128.3, 127.5, 127.3, 126.1, 125.8, 124.0, 60.7, 43.1, 31.3, 24.3, 22.4, 14.2, 13.9; IR (KBr) υ 2957, 2871, 1717, 1638, 1464, 1368, 1263, 1177, 1038, 816, 749 cm$^{-1}$; HRMS (ESI) calcd for $C_{21}H_{24}O_3$ $[M]^+$ 324.1725, found 324.1722.



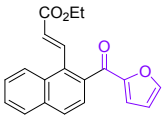

**Ethyl (E)-3-(2-((1s,3s)-adamantane-1-carbonyl)naphthalen-1-yl)acrylate (3u)**

The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), ester **2l** (191.6 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3u** as colorless oil (35.8 mg, 46%) (petroleum ether/ethyl acetate = 30 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.21 (d, *J* = 16.0 Hz, 1H), 8.07 (d, *J* = 8.0 Hz, 1H), 7.86 (t, *J* = 9.2 Hz, 2H), 7.56 (p, *J* = 6.8 Hz, 2H), 7.20 (d, *J* = 8.4 Hz, 1H), 6.21 (d, *J* = 16.0 Hz, 1H), 4.29 (q, *J* = 7.1 Hz, 2H), 1.99 (s, 3H), 1.86 (s, 6H), 1.65 (q, *J* = 12.5 Hz, 6H), 1.34 (t, *J* = 7.2 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 214.5, 166.1, 141.4, 138.1, 132.9, 130.8, 128.7, 128.5, 128.4, 127.3, 127.0, 126.7, 124.7, 122.9, 60.7, 47.5, 38.9, 36.3, 27.9, 14.3; IR (KBr) υ 2905, 2851, 1717. 1686, 1452, 1280, 1179, 1033, 988, 860, 828, 799, 748 cm$^{-1}$; HRMS (ESI) calcd for $C_{26}H_{28}O_3$ $[M]^+$ 388.2038, found 388.2042.



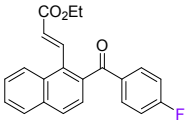

**Ethyl (E)-3-(2-(thiophene-2-carbonyl)naphthalen-1-yl)acrylate (3v)**

The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), ester **2m** (160.2 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3v** as yellow oil (41.7 mg, 62%) (petroleum ether/ethyl acetate =20 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.23 (d, *J* = 16.0 Hz, 1H), 8.15-8.11 (m, 1H), 7.95-7.92 (m, 2H), 7.72 (d, *J* = 5.2 Hz, 1H), 7.64-7.59 (m, 2H),7.57 (d, *J* = 8.4 Hz, 1H), 7.34 (d, *J* = 3.6 Hz, 1H), 7.08 (t, *J* = 4.0 Hz, 1H), 6.18 (d, *J* = 16.0 Hz, 1H), 4.21 (q, *J* = 7.2 Hz, 2H), 1.29 (t, *J* = 7.2 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 190.0, 165.8, 144.6, 140.8, 136.4, 135.4, 135.2, 133.9, 131.7, 131.0, 128.9, 128.6, 128.2, 127.5, 127.5, 126.6, 125.4, 124.6, 60.6, 14.2; IR (KBr) υ 2927, 1716, 1642, 1512 , 1463, 1410, 1369, 1281, 1179, 1047, 981, 861, 803, 727 $cm^{-1}$; HRMS (ESI) calcd for $C_{20}H_{16}O_3S$ $[M]^+$ 336.0820, found 336.0821.



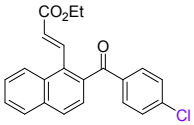

**Ethyl (E)-3-(2-(furan-2-carbonyl)naphthalen-1-yl)acrylate (3w)**

The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), ester **2n** (150.7 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3w** as colorless oil (34.6 mg, 54%) (petroleum ether/ethyl acetate = 20 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.26 (d, *J* = 16.0 Hz, 1H), 8.12 (t, *J* = 6.0 Hz, 1H), 7.94-7.92 (m, 2H), 7.63-7.59 (m, 4H), 7.02 (d, *J* = 4 Hz, 1H), 6.54-6.53 (m, 1H), 6.11 (d, *J* = 16.4 Hz, 1H), 4.21 (q, *J* = 6.8 Hz, 2H), 1.28 (t, *J* = 4.0 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 185.0, 165.8, 152.6, 147.5, 141.0, 135.3, 134.1, 132.7, 131.0, 128.9, 128.6, 127.6, 127.5, 126.6, 125.5, 124.8, 120.8, 112.6, 60.7, 14.2; IR (KBr) υ 2926, 2853, 1715, 1653, 1563, 1463, 1391, 1279, 1179, 1029, 762 $cm^{-1}$; HRMS (ESI) calcd for $C_{26}H_{10}O_4$ $[M]^+$ 320.1049, found 320.1049.




**Ethyl (E)-3-(2-(4-fluorobenzoyl)naphthalen-1-yl)acrylate (3x)**

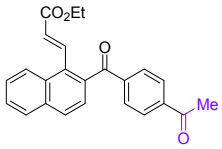
The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), ester **2o** (167.4 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3x** as colorless oil (32.8 mg, 47%) (petroleum ether/ethyl acetate = 25 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.15-8.09 (m, 2H), 7.94 (t, *J* = 3.6 Hz, 2H), 7.76 (m, 2H), 7.64-7.60 (m, 2H), 7.49 (d, *J* = 8.4 Hz, 1H), 7.09 (t, *J* = 8.4 Hz, 2H), 6.09 (d, *J* = 16.0 Hz, 1H), 4.18 (q, *J* = 6.8 Hz, 2H), 1.27 (t, *J* = 7.2 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 196.8, 165.8 (d, *J* = 254.4 Hz), 165.6, 140.8, 136.3, 133.96 (CC,F, d, *J* = 2.9 Hz), 133.91, 132.49 (CC,F, d, *J* = 9.4 Hz), 131.9, 131.0, 129.1, 128.6, 127.56 (CC,F, d, *J* = 3.7 Hz), 126.9, 125.3, 124.8, 115.8 (d, *J* = 21.9 Hz), 60.6, 14.2; **$^{19}$F NMR** (376 MHz, $CDCl_3$): δ -104.3 (s, 1F); IR (KBr) υ 2924, 2853, 1713, 1664, 1594, 1502, 1462, 1369, 1302, 1176, 1095, 1029, 966, 820, 763 $cm^{-1}$; HRMS (ESI) calcd for $C_{22}H_{17}FO_3$ $[M]^+$ 348.1162, found 348.1165.




**Ethyl (E)-3-(2-(4-chlorobenzoyl)naphthalen-1-yl)acrylate (3y)**

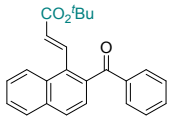
The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), ester **2p** (177.0 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3y** as colorless oil (40.2 mg, 55%) (petroleum ether/ethyl acetate = 25 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.15-8.10 (m, 2H), 7.95-7.93 (m, 2H), 7.67 (d, *J* = 8.4 Hz, 2H), 7.64-7.61 (m, 2H), 7.49 (d, *J* = 8.4 Hz, 1H), 7.39 (d, *J* = 8.4 Hz, 2H), 6.08 (d, *J* = 16.0 Hz, 1H), 4.19 (q, *J* = 6.8 Hz, 2H), 1.27 (t, *J* = 7.2 Hz, 3H); **$^{13}$C NMR**

(100 MHz, $CDCl_3$): δ 197.1, 165.5, 140.8, 139.8, 136.0, 135.9, 133.9, 132.1, 131.2, 131.0, 129.1, 128.9, 128.6, 127.6, 127.0, 125.3, 124.8, 60.7, 14.7; IR (KBr) υ 2925, 2854, 1715, 1666, 1586, 1462, 1370, 1262, 1176, 1091, 1030, 969, 816, 761 $cm^{-1}$; HRMS (ESI) calcd for $C_{22}H_{17}ClO_3$ $[M]^+$ 364.0866, found 364.0865.




**Ethyl (E)-3-(2-(4-acetylbenzoyl)naphthalen-1-yl)acrylate (3z)**

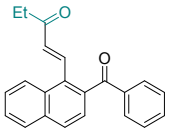
The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), ester **2q** (181.9 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3z** as colorless oil (60.6 mg, 92%) (petroleum ether/ethyl acetate = 25 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.14-8.10 (m, 2H), 7.98-7.95 (m, 4H), 7.79 (d, *J* = 7.2 Hz, 2H), 7.65-7.63 (m, 2H), 7.53 (d, *J* = 8.0 Hz, 1H), 6.04 (d, *J* = 16.0 Hz, 1H), 4.76 (q, *J* = 7.2 Hz, 2H), 2.63 (s, 3H), 1.25 (t, *J* = 6.8 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 197.7, 197.4, 165.4, 141.0, 140.9, 140.1, 135.9, 134.1, 132.5, 131.0, 129.9, 129.2, 128.6, 128.4, 127.8, 127.7, 127.2, 125.4, 124.9, 60.7, 26.9, 14.2; IR (KBr) υ 2926, 2854, 1716, 1689, 1464, 1365, 1265, 1179, 1032, 958, 820, 767 $cm^{-1}$; HRMS (ESI) calcd for $C_{24}H_{20}O_4$ $[M]^+$ 372.1362, found 372.1363.




**Butyl (E)-3-(2-benzoylnaphthalen-1-yl)acrylate (3A)**

The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), ester **2a** (156.6 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), butyl acrylate (51.3 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3A** as colorless oil (52.3 mg, 73%) (petroleum ether/ethyl acetate = 25 : 1 as

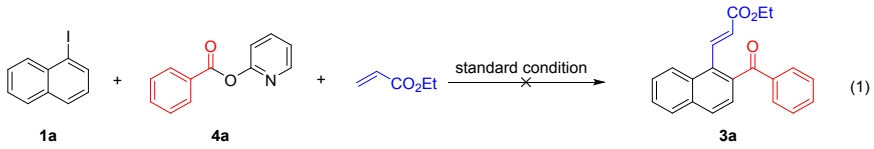
eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.15-8.10 (m, 2H), 7.93 (d, *J* = 8 Hz, 2H), 7.72 (d, *J* = 8 Hz, 2H), 7.63-7,60 (m, 2H), 7.59-7.51 (m, 2H), 7.41 (t, *J* = 8 Hz, 2H), 6.10 (d, *J* = 16 Hz, 1H), 4.11 (t, *J* = 8 Hz, 2H), 1.64-1.57 (m, 2H), 1.39-1.30 (m, 2H), 0.93 (t, *J* = 8 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 198.4, 165.6, 140.8, 137.5, 136.6 133.9 133.2 131.9, 130.9, 129.8 128.9, 128.5, 128.5 127.4, 126.8, 125.3, 125.0, 64.4, 30.5, 19.0, 13.7; IR (KBr) υ 2959, 2872, 1717, 1667, 1595, 1463, 1279, 1175, 967, 618, 751, 715 $cm^{-1}$; HRMS (ESI) calcd for $C_{24}H_{22}O_3$ $[M]^+$ 358.1569, found 358.1565.




**(E)-1-(2-benzoylnaphthalen-1-yl)pent-1-en-3-one (3B)**

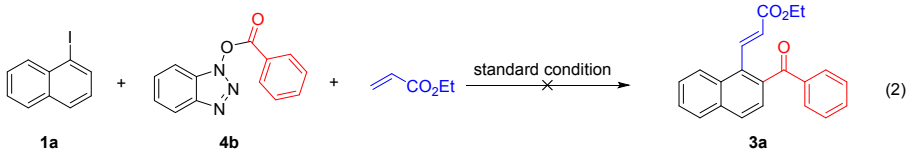
The reaction of 1-iodonaphthalene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), ester **2a** (156.6 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), pent-1-en-3-one (33.6 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h afforded **3B** as colorless oil (49.1 mg, 78%) (petroleum ether/ethyl acetate = 25 : 1 as eluent). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 8.09 (d, *J* = 9.2 Hz, 1H), 8.02 (d, *J* = 16.0 Hz, 1H), 7.94 (d, *J* = 8.0 Hz, 2H), 7.72 (d, *J* = 7.6 Hz, 2H), 7.61 (t, *J* = 4.0 Hz, 2H), 7.57-7.52 (m, 2H), 7.41 (t, *J* = 10 Hz, 2H), 6.37 (d, *J* = 16.0 Hz, 1H), 2.49 (q, *J* = 7.6 Hz, 2H), 1.03 (t, *J* = 7.2 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 199.8, 198.5, 138.4, 137.6, 136.4, 134.1, 133.9, 133.3, 132.3, 131.0, 129.7, 129.0, 128.6, 127.5, 125.3, 125.0, 34.1, 7.9; IR (KBr) υ 3058, 2935, 1668, 1618, 1462, 1330, 1280, 1189, 964, 819, 751, 714 $cm^{-1}$; HRMS (ESI) calcd for $C_{22}H_{18}O_2$ $[M]^+$ 314.1307, found 314.1304.

## 4. Mechanistic Investigations



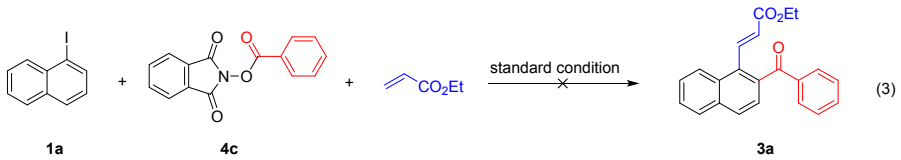

**Typical procedure using electrophile 4a:** The reaction of 1-iodo-2,4-dimethylbenzene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), pyridine ester **4a** (119.4 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h under $N_2$ atmosphere. The mixture was detected by GC-MS, and almost no corresponding product **3a** was detected.

**Conclusion: 3a** was not observed in the presence of pyridine ester possessing weak electron-withdrawing ability, which indicates strong electron-withdrawing ability of triazine ester is important in this reaction system.




**Typical procedure using electrophile 4b:** The reaction of 1-iodo-2,4-dimethylbenzene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), triazole ester **4b** (143.5 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h under $N_2$ atmosphere. The mixture was detected by GC-MS, and almost trace corresponding product **3a** was detected.

**Conclusion:** Low yield of **3a** was observed in the presence of triazole ester possessing weak electron-withdrawing ability, which also indicates strong electron-withdrawing ability of triazine ester is important in this reaction system.

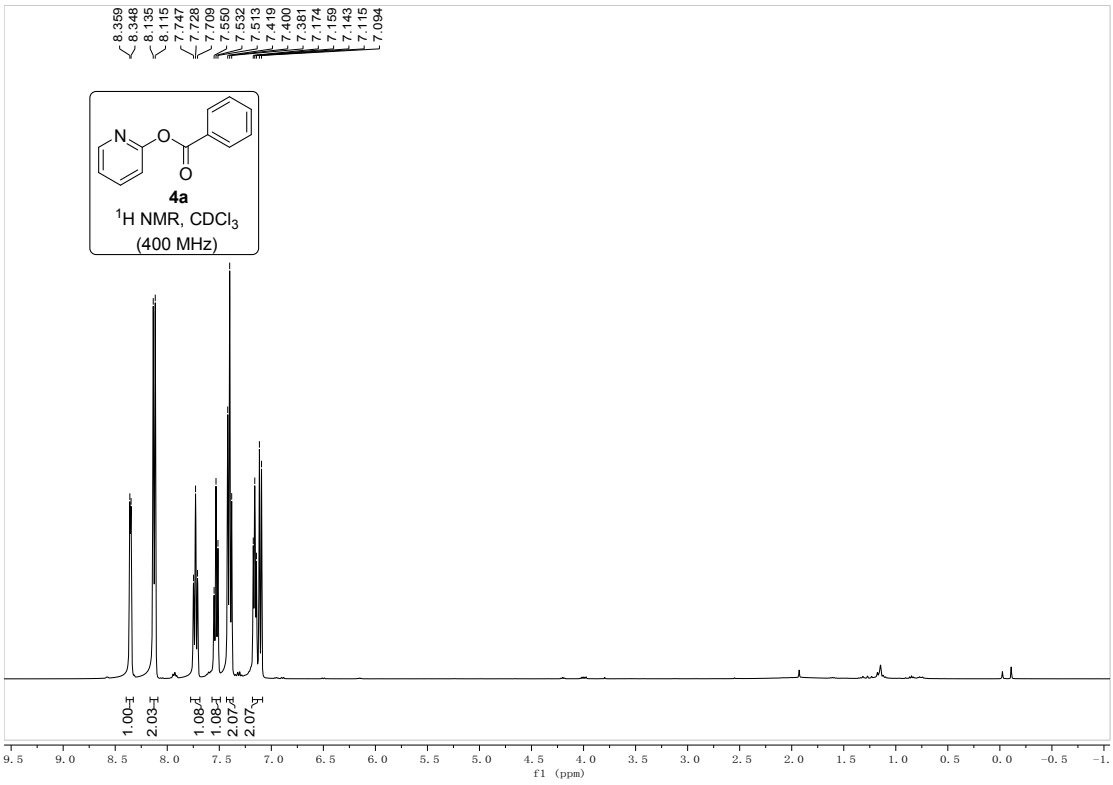


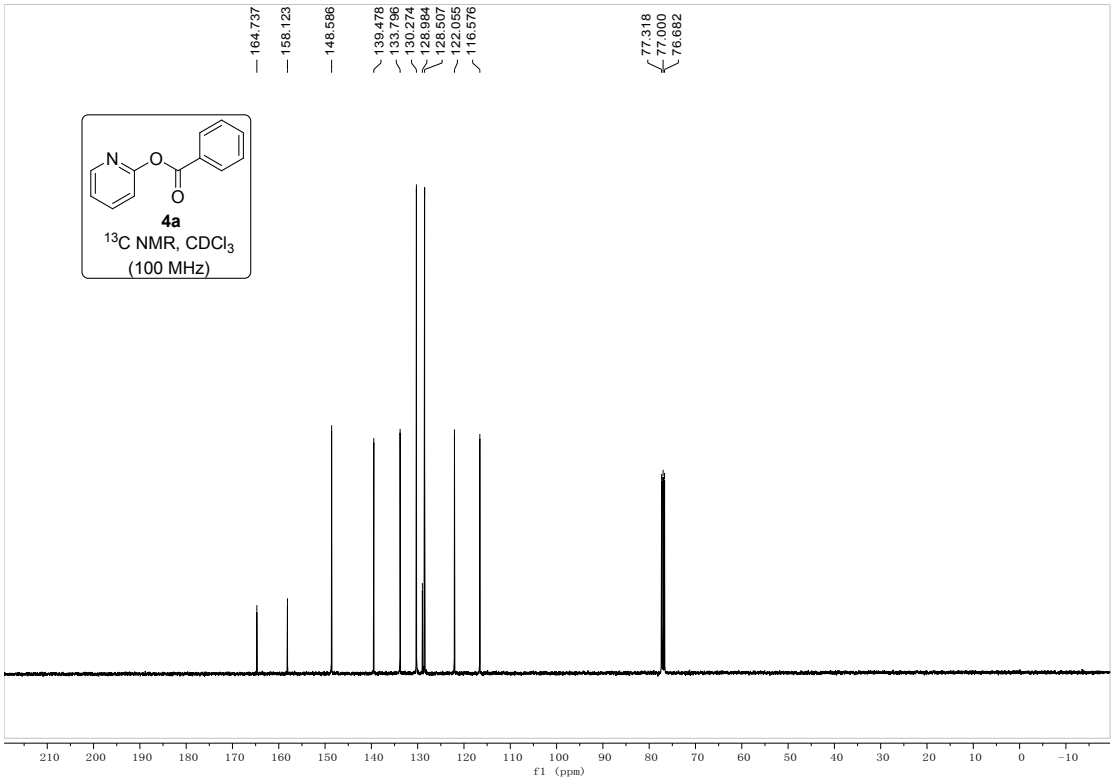

1a 4c 3a

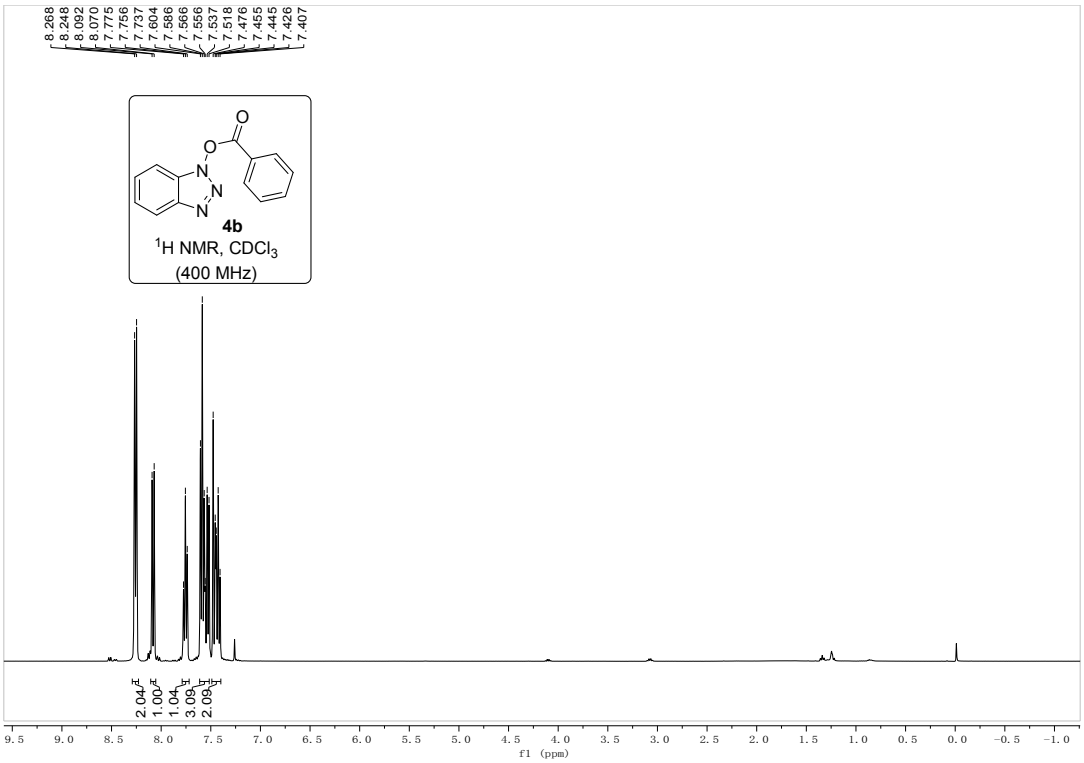
**Typical procedure using electrophile 4c investigation:** The reaction of 1-iodo-2,4-dimethylbenzene **1a** (50.8 mg, 0.20 mmol, 1.0 equiv), N-hydroxysuccinimide ester **4c** (160.2 mg, 0.60 mmol, 3.0 equiv), $\{Pd(allyl)Cl\}_2$ (1.8 mg, 0.005 mmol, 5 mol %), TFP (4.7 mg, 0.01 mmol, 10 mol %), ethyl acrylate (40.0 mg, 0.40 mmol, 2.0 equiv), norbornene (56.5 mg, 0.60 mmol, 3.0 equiv) and $K_2CO_3$ (55.3 mg, 0.40 mmol, 2.0 equiv) in a mixture of toluene (1.35 mL) and acetonitrile (0.65 mL), at 100 °C for 5 h under $N_2$ atmosphere. The mixture was detected by GC-MS, and no corresponding product **3a** was detected.

**Conclusion:** Corresponding product **3a** was not observed, sugesting strong electron-withdrawing property of activator alone could not facilitate C-O bond cleavage.

# 5. Data of the NMR Spectra.

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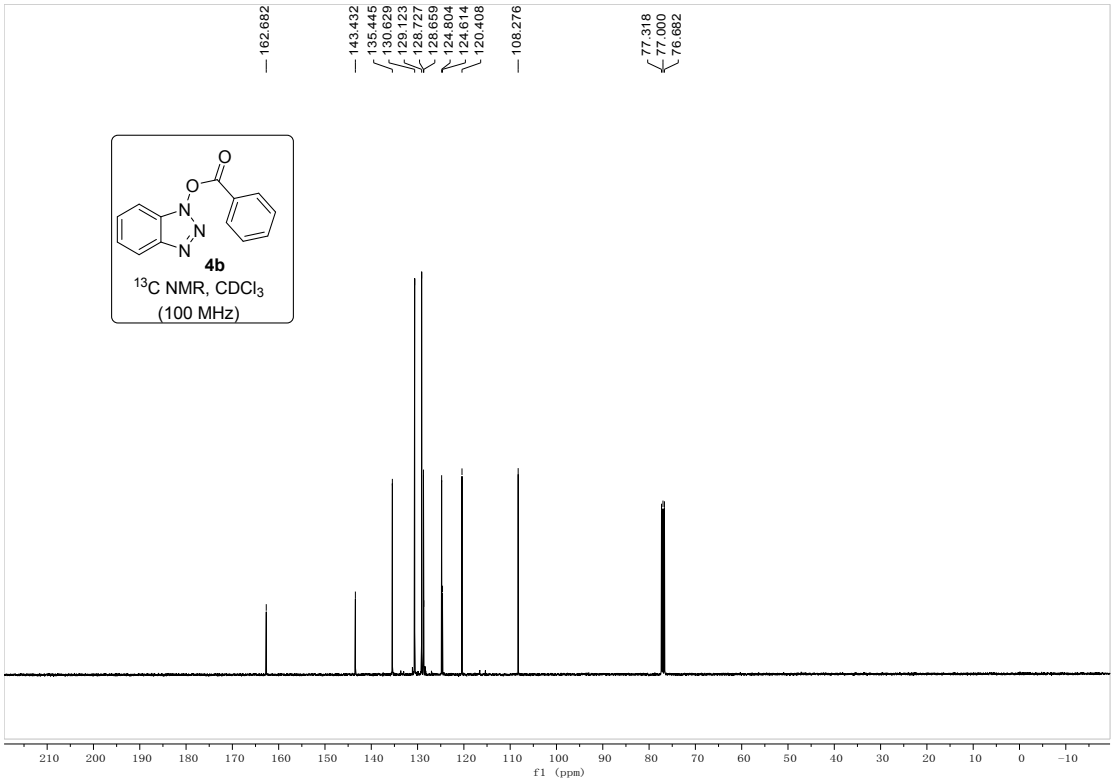
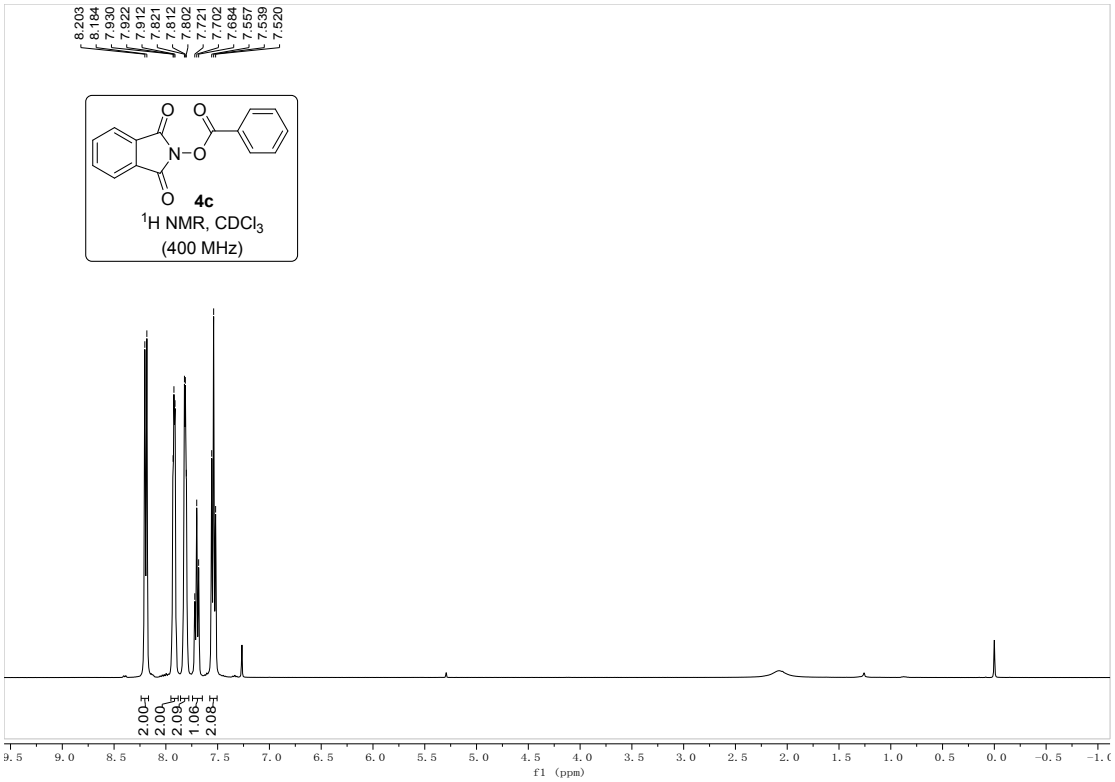
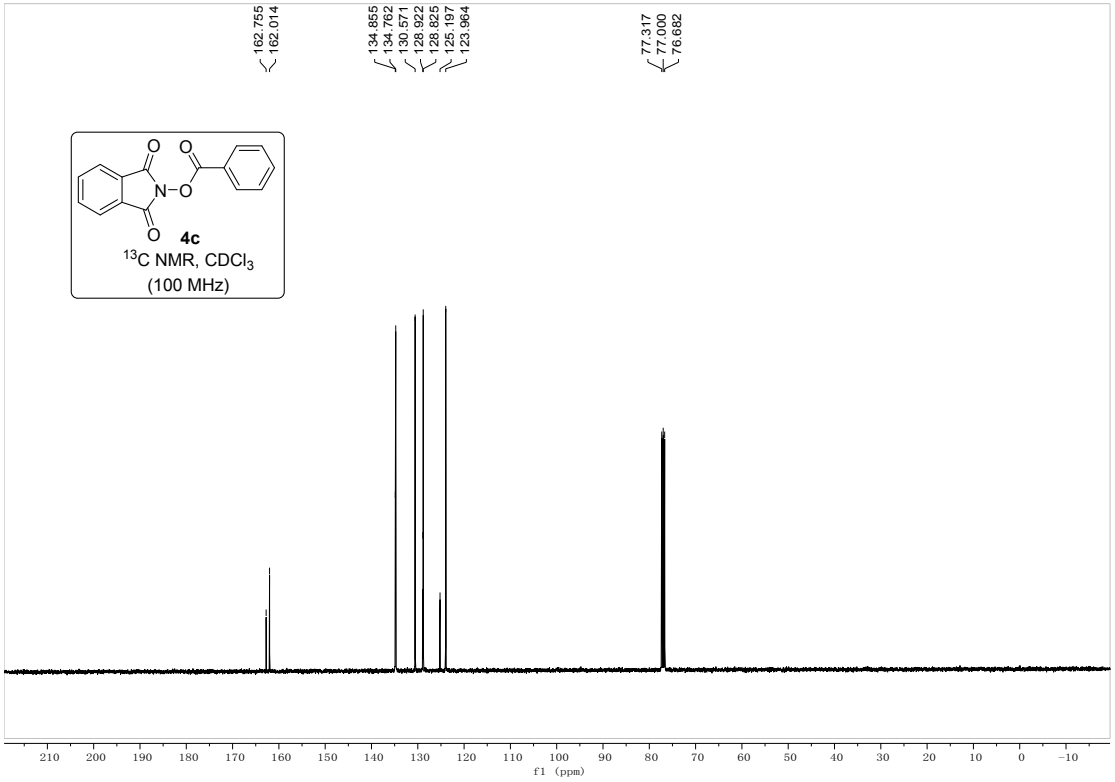
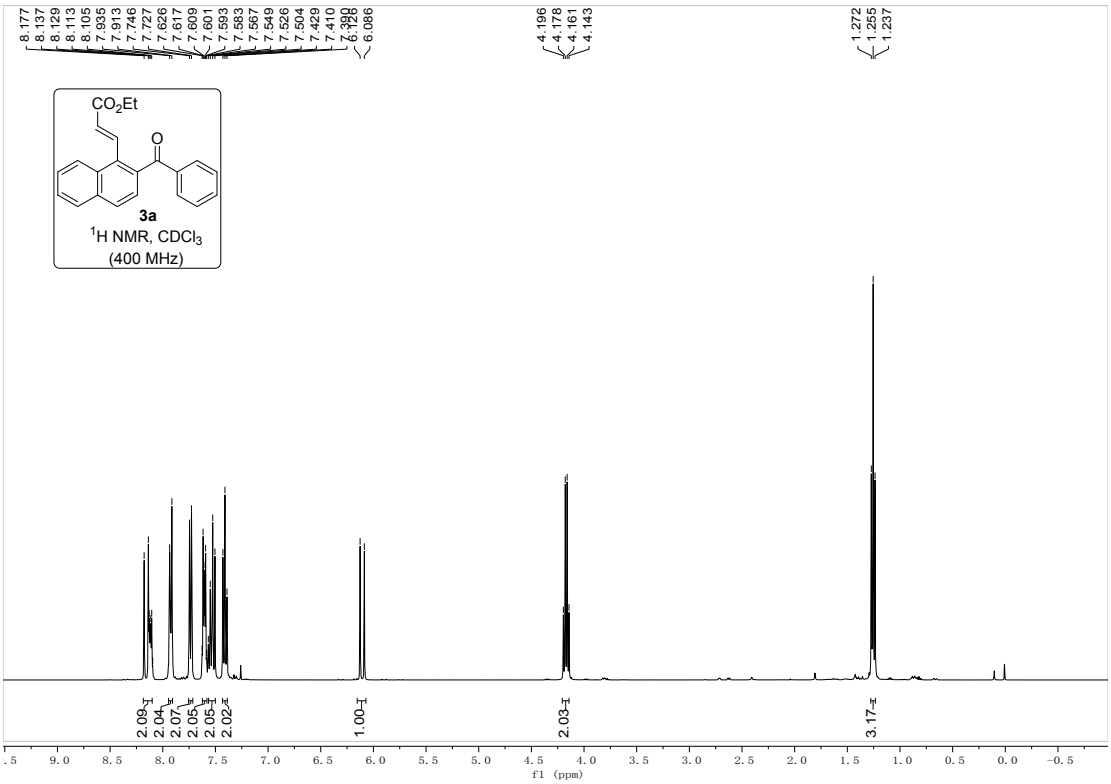
8.248
8.092
8.070
7.775
7.756
7.737
7.604
7.586
7.566
7.556
7.537
7.518
7.476
7.455
7.445
7.426
7.407
4b
$^{1}$H NMR, CDCl$_3$
(400 MHz)
2.04
1.00
1.04
3.09
2.09
f1 (ppm)


162.682
143.432
135.445
130.629
129.123
128.727
128.659
124.804
124.614
120.408
108.276
77.318
77.000
76.682
4b
$^{13}$C NMR, CDCl$_3$
(100 MHz)
f1 (ppm)


8.203
8.184
7.930
7.922
7.912
7.821
7.812
7.802
7.721
7.702
7.684
7.557
7.539
7.520
4c
$^{1}$H NMR, $CDCl_3$
(400 MHz)
2.00
2.00
2.09
1.06
2.08
f1 (ppm)


162.755
162.014
134.855
134.762
130.571
128.922
128.825
125.197
123.964
77.317
77.000
76.682
4c
$^{13}$C NMR, $CDCl_3$
(100 MHz)
f1 (ppm)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8.137
8.129
8.113
8.105
7.935
7.913
7.746
7.727
7.626
7.617
7.609
7.601
7.593
7.583
7.567
7.549
7.526
7.504
7.429
7.410
7.390
6.128
6.086
4.196
4.178
4.161
4.143
1.272
1.255
1.237
CO2Et
O
3a
1H NMR, CDCl3
(400 MHz)
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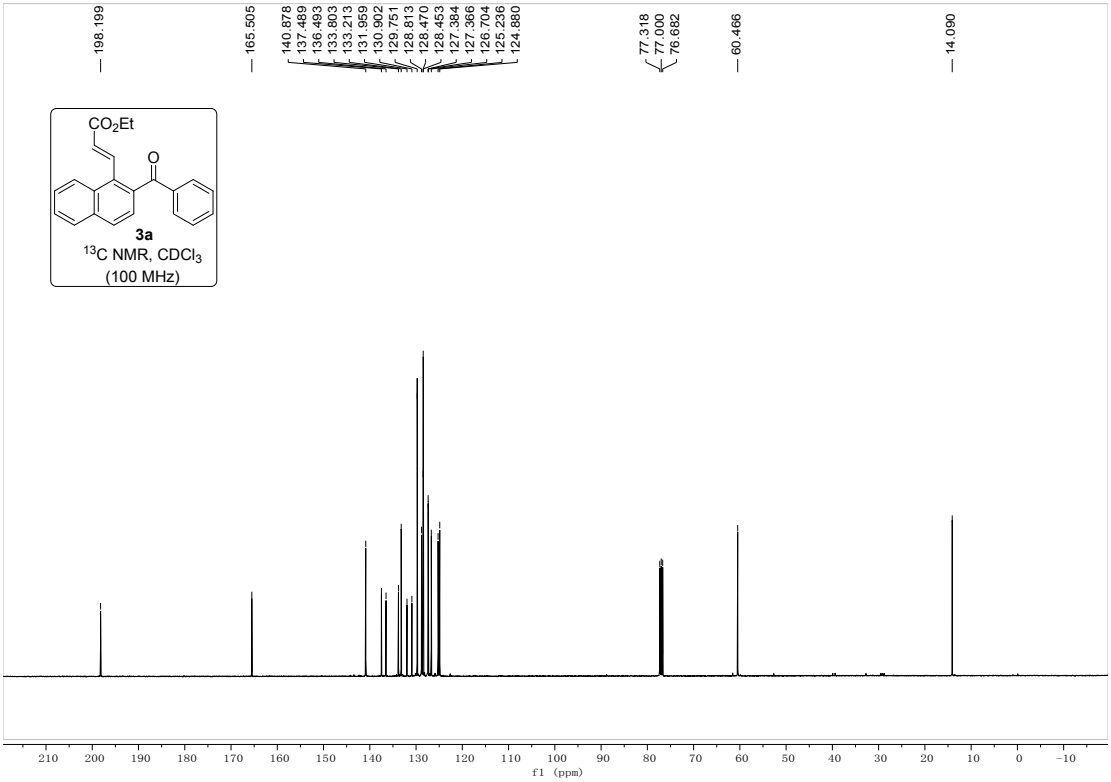
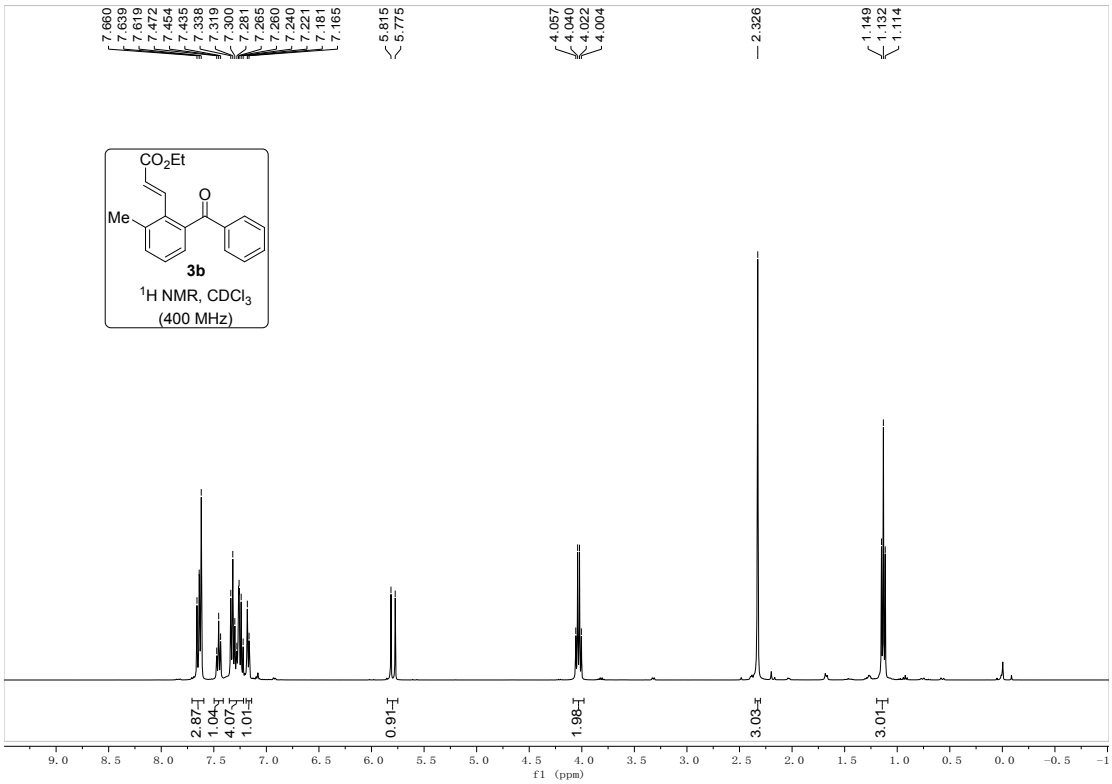
2.04
2.07
2.05
2.05
2.02
1.00
2.03
3.17
f1 (ppm)


198.199
165.505
140.878
137.489
136.493
133.803
133.213
131.959
130.902
129.751
128.813
128.470
128.453
127.384
127.366
126.704
125.236
124.880
77.318
77.000
76.682
60.466
14.090
CO2Et
O
3a
13C NMR, CDCl3
(100 MHz)
f1 (ppm)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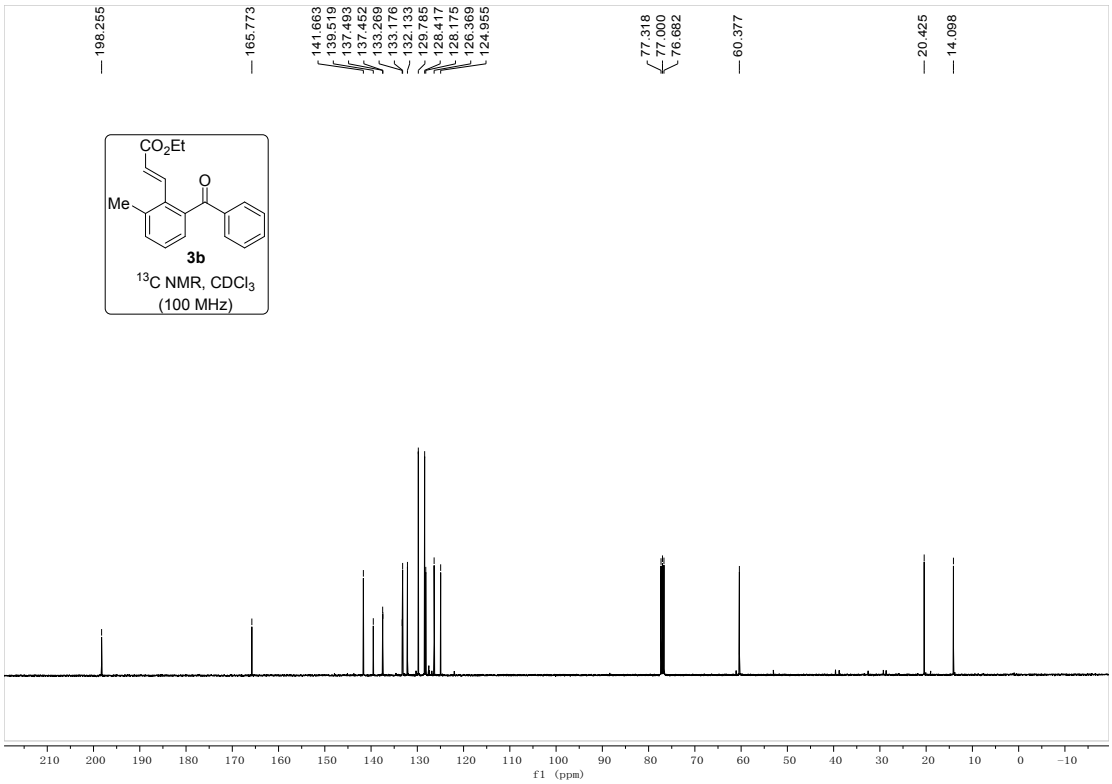
7.639
7.619
7.472
7.454
7.435
7.338
7.319
7.300
7.281
7.265
7.260
7.240
7.221
7.181
7.165
5.815
5.775
4.057
4.040
4.022
4.004
2.326
1.149
1.132
1.114
CO2Et
O
Me
3b
1H NMR, CDCl3
(400 MHz)
2.87
1.04
4.07
1.01
0.91
1.98
3.03
3.01
f1 (ppm)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139.519
137.493
137.452
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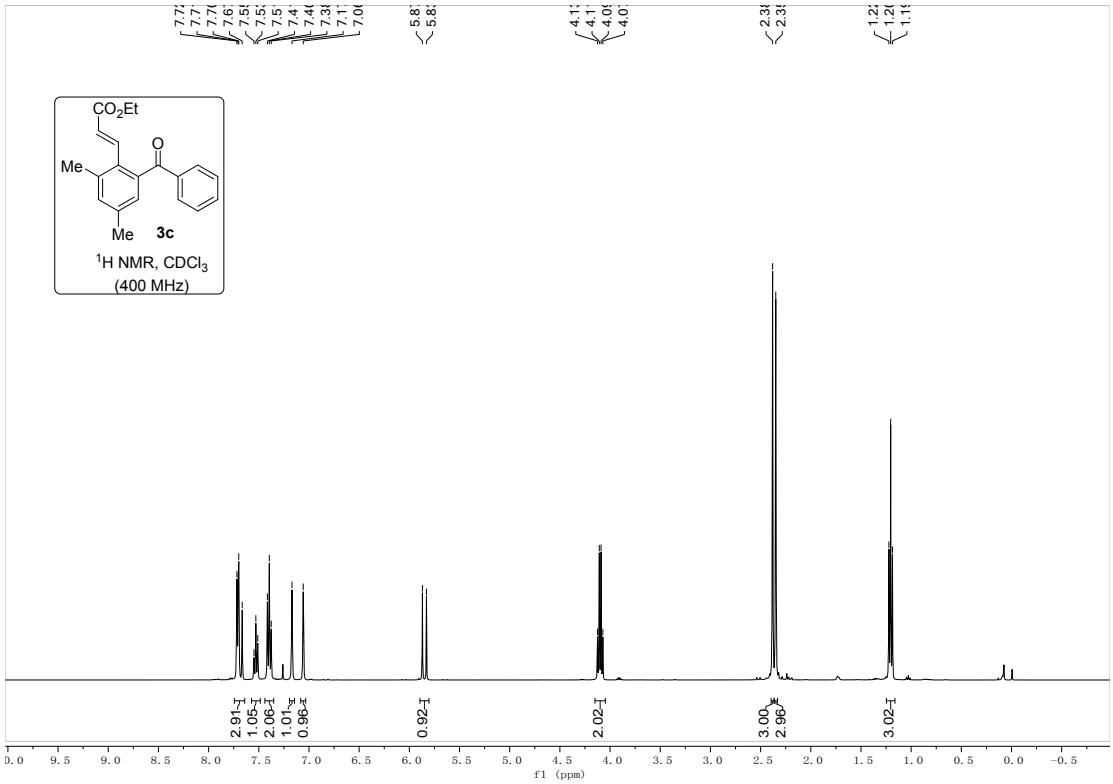
133.176
132.133
129.785
128.417
128.175
126.369
124.955
77.318
77.000
76.682
60.377
20.425
14.098
CO2Et
O
Me
3b
13C NMR, CDCl3
(100 MHz)
f1 (ppm)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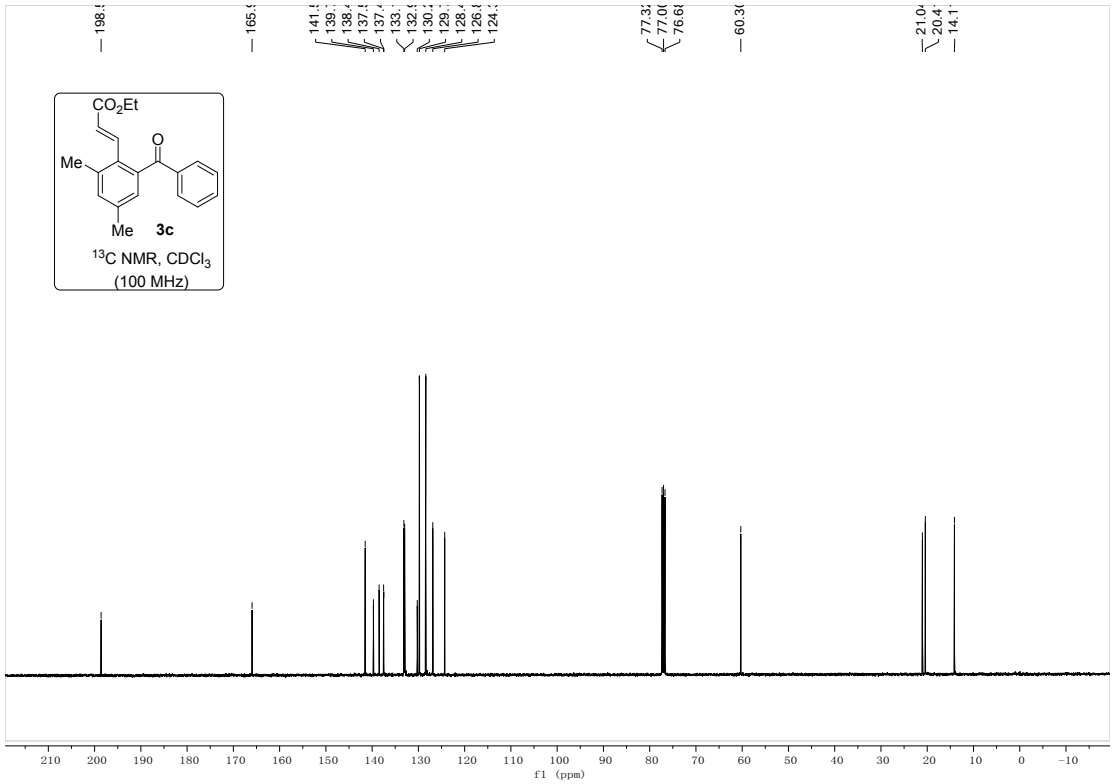
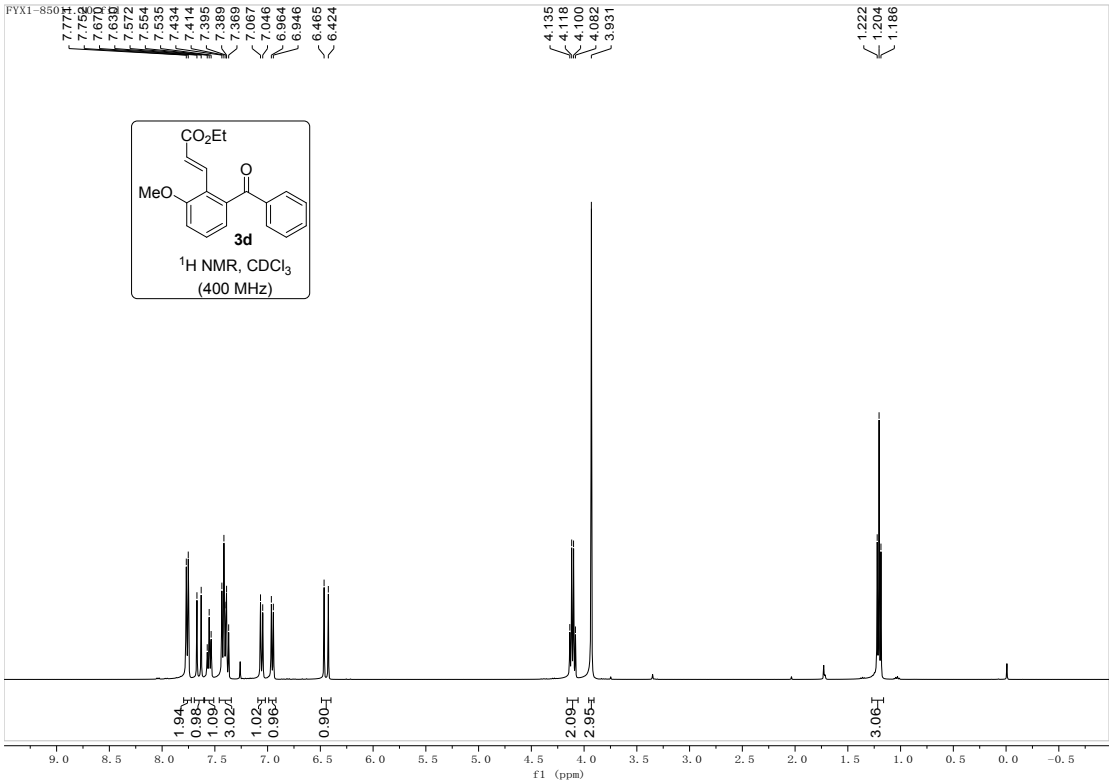
O
Me
Me
3c
1H NMR, CDCl3
(400 MHz)
f1 (ppm)


CO2Et
O
Me
Me
3c
13C NMR, CDCl3
(100 MHz)
f1 (ppm)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7.678
7.638
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7.554
7.535
7.434
7.414
7.395
7.389
7.369
7.067
7.046
6.964
6.946
6.465
6.424
4.135
4.118
4.100
4.082
3.931
1.222
1.204
1.186
CO2Et
O
MeO
3d
1H NMR, CDCl3
(400 MHz)
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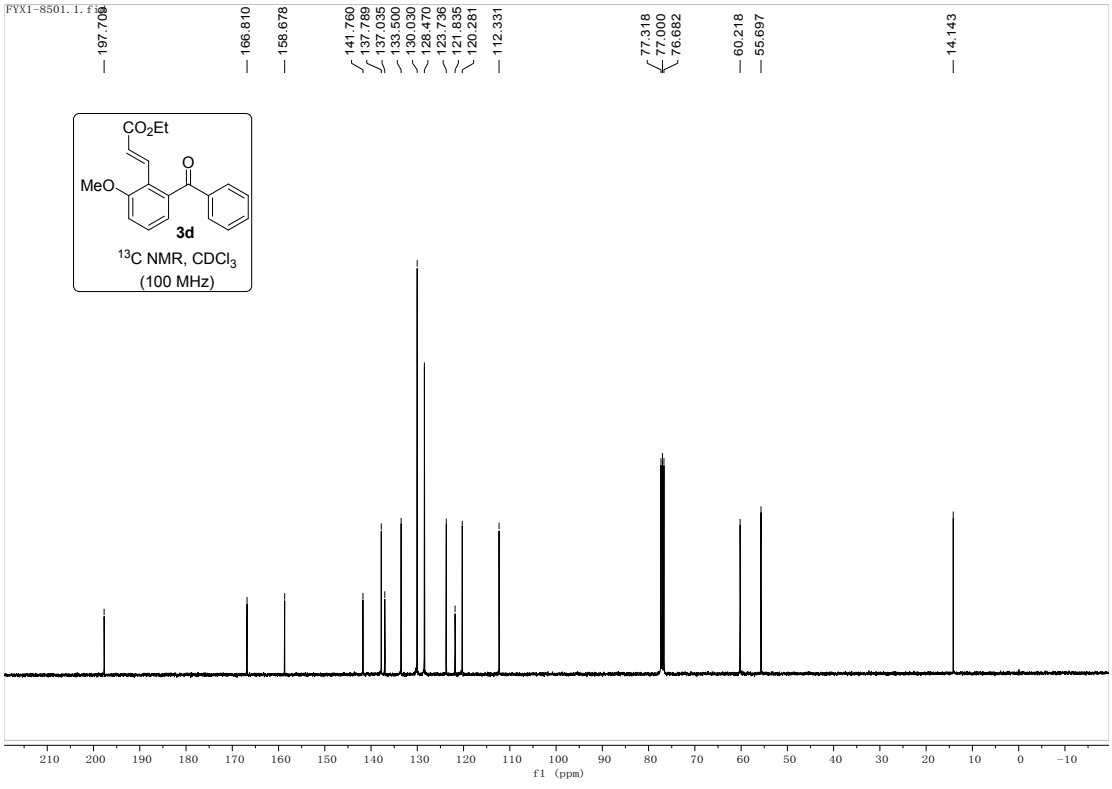
1.09
3.02
1.02
0.96
0.90
2.09
2.95
3.06
f1 (ppm)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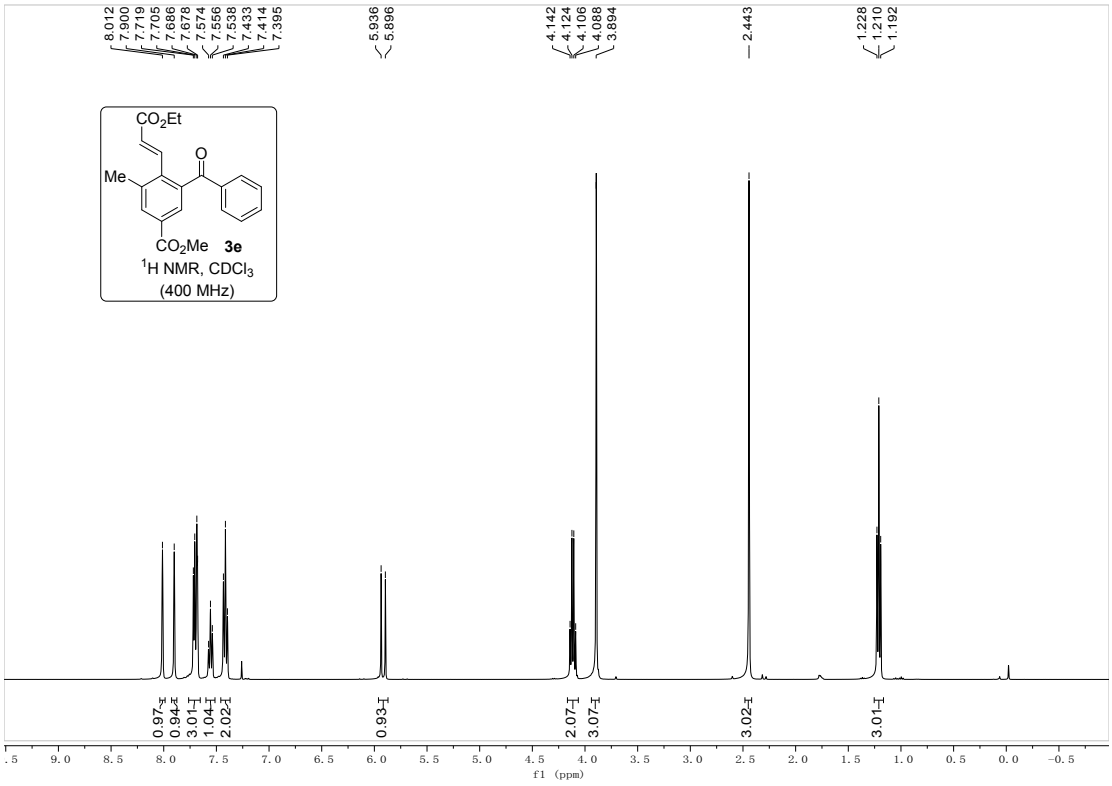
158.678
141.760
137.789
137.035
133.500
130.030
128.470
123.736
121.835
120.281
112.331
77.318
77.000
76.682
60.218
55.697
14.143
CO2Et
O
MeO
3d
13C NMR, CDCl3
(100 MHz)
f1 (ppm)


8.012
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7.705
7.686
7.678
7.574
7.556
7.538
7.433
7.414
7.395
5.936
5.896
4.142
4.124
4.106
4.088
3.894
2.443
1.228
1.210
1.192
CO2Et
O
Me
CO2Me 3e
1H NMR, CDCl3
(400 MHz)
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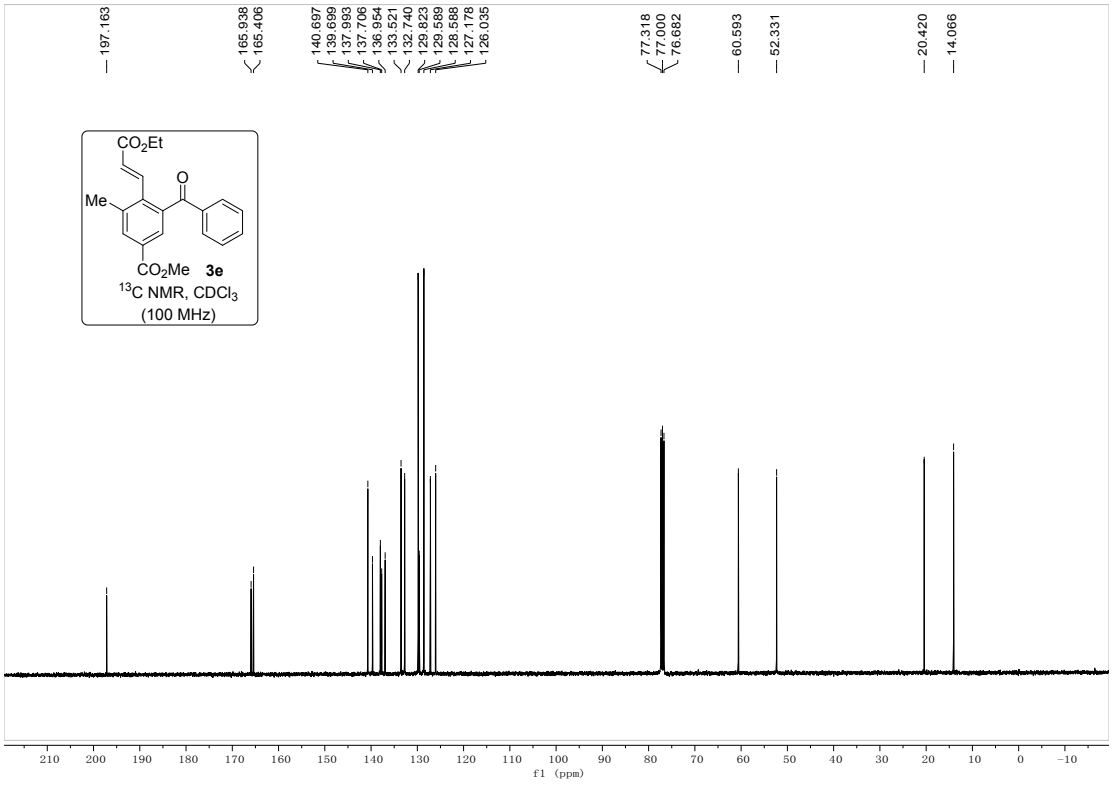
2.02
0.93
2.07
3.07
3.02
3.01
f1 (ppm)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165.406
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139.699
137.993
137.706
136.954
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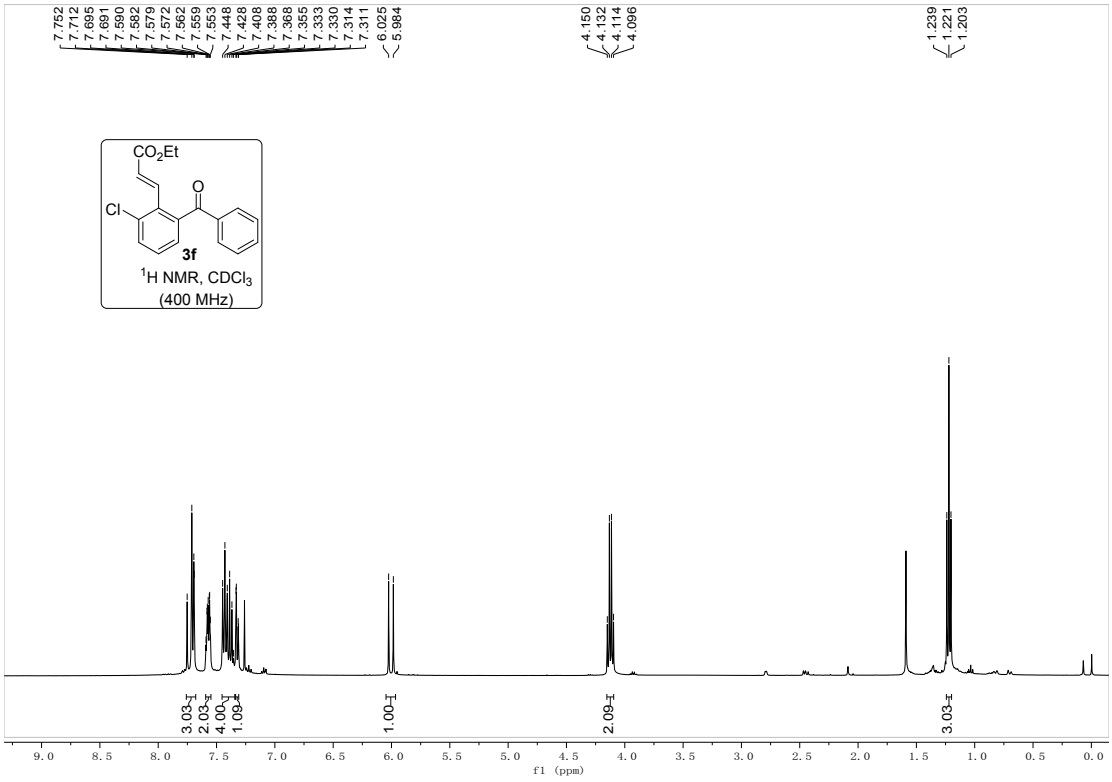
132.740
129.823
129.589
128.588
127.178
126.035
77.318
77.000
76.682
60.593
52.331
20.420
14.066
CO2Et
O
Me
CO2Me 3e
13C NMR, CDCl3
(100 MHz)
f1 (ppm)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7.590
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7.572
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7.559
7.553
7.448
7.428
7.408
7.388
7.368
7.355
7.333
7.330
7.314
7.311
6.025
5.984
4.150
4.132
4.114
4.096
1.239
1.221
1.203
CO2Et
O
Cl
3f
1H NMR, CDCl3
(400 MHz)
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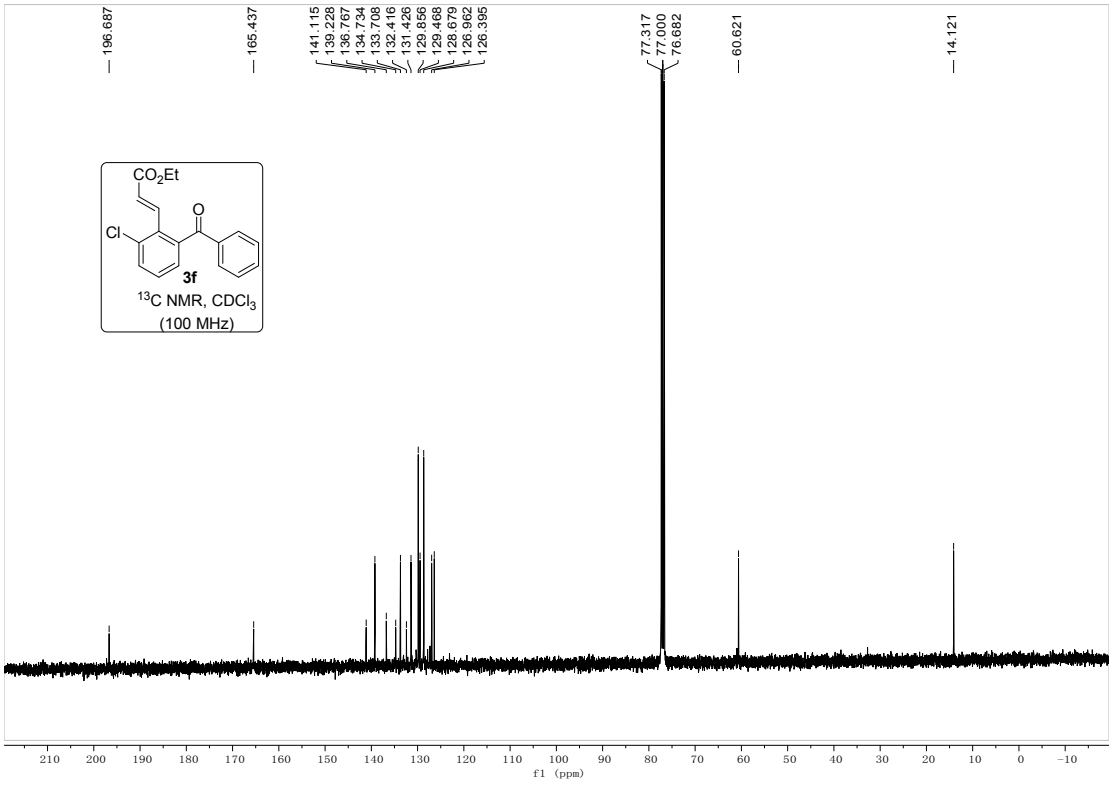
2.03
4.00
1.09
1.00
2.09
3.03
f1 (ppm)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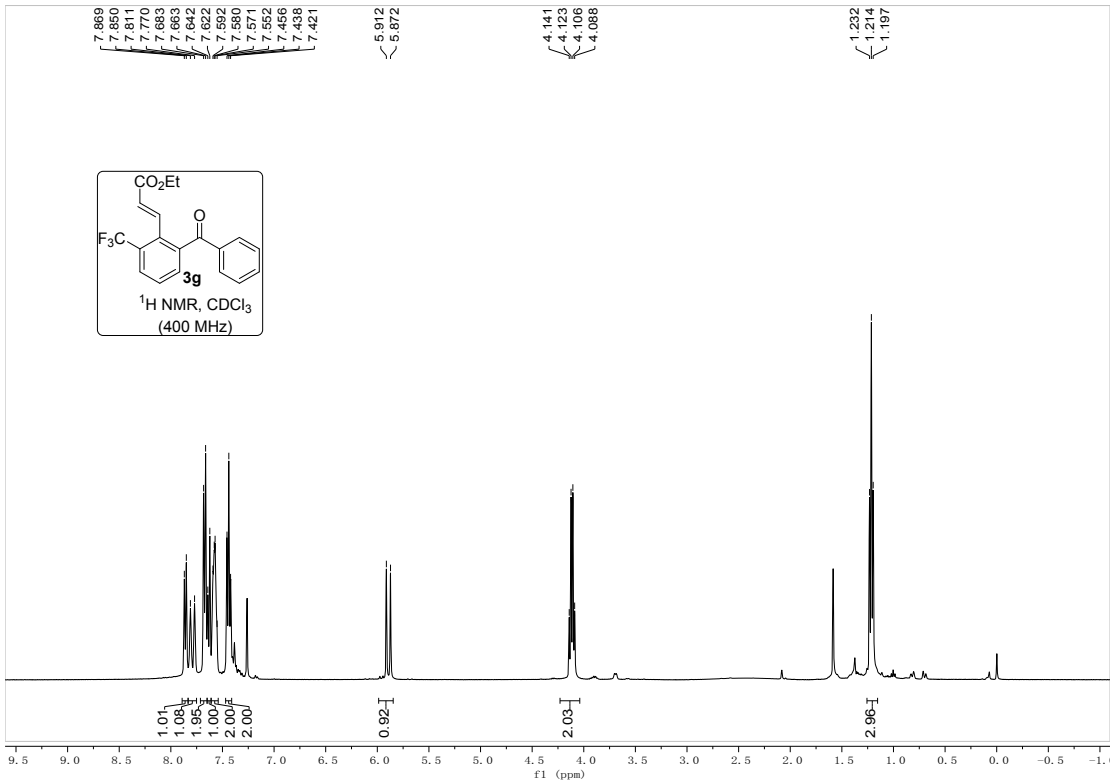
134.734
133.708
132.416
131.426
129.856
129.468
128.679
126.962
126.395
77.317
77.000
76.682
60.621
14.121
CO2Et
O
Cl
3f
13C NMR, CDCl3
(100 MHz)
f1 (ppm)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7.850
7.811
7.770
7.683
7.663
7.642
7.622
7.592
7.580
7.571
7.552
7.456
7.438
7.421
5.912
5.872
4.141
4.123
4.106
4.088
1.232
1.214
1.197
CO2Et
O
F3C
3g
1H NMR, CDCl3
(400 MHz)
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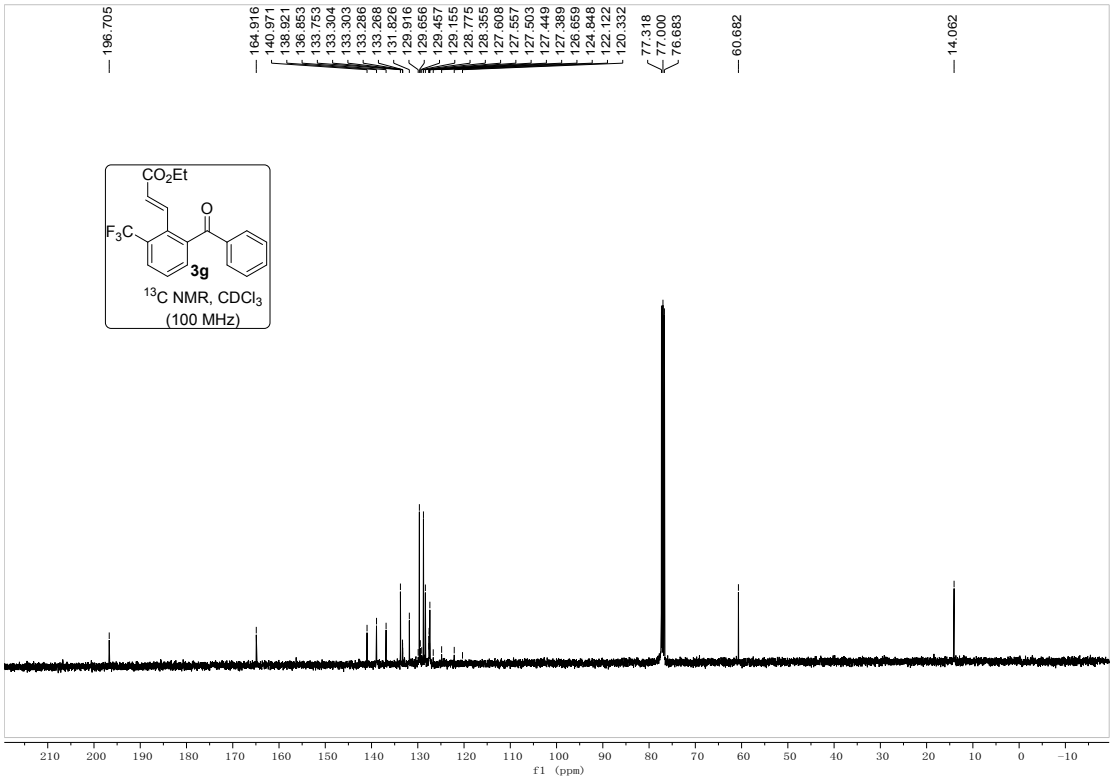
1.08
1.95
1.00
2.00
2.00
0.92
2.03
2.96
f1 (ppm)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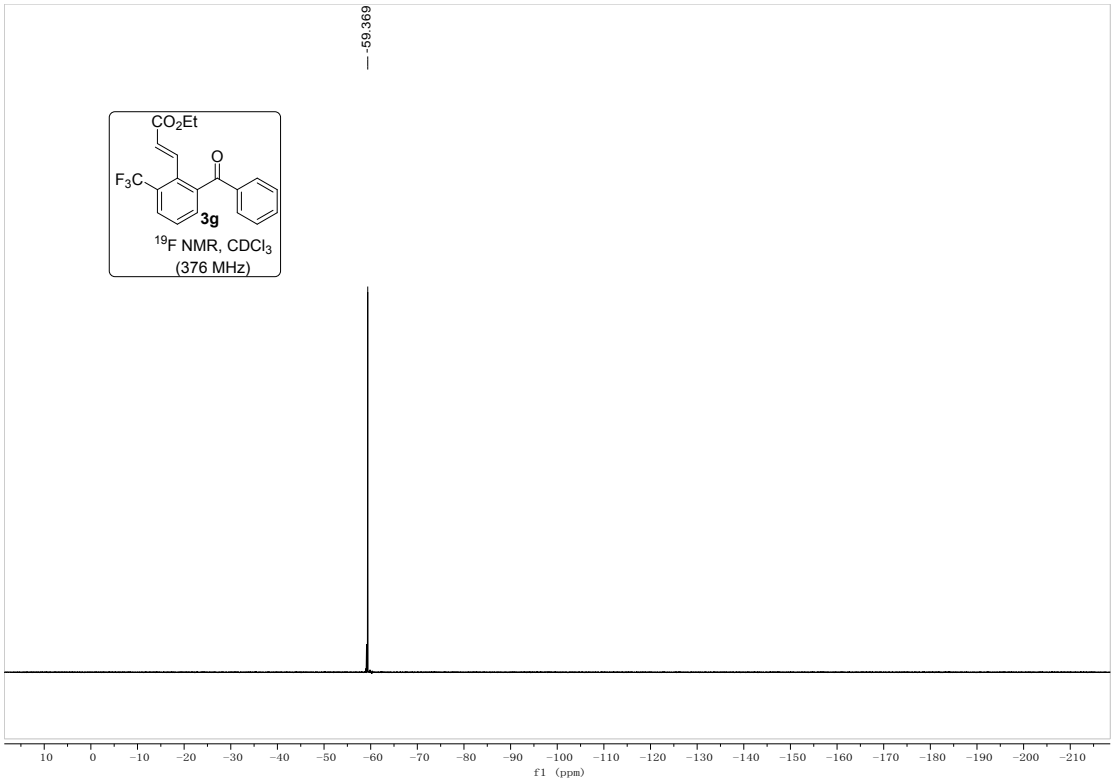
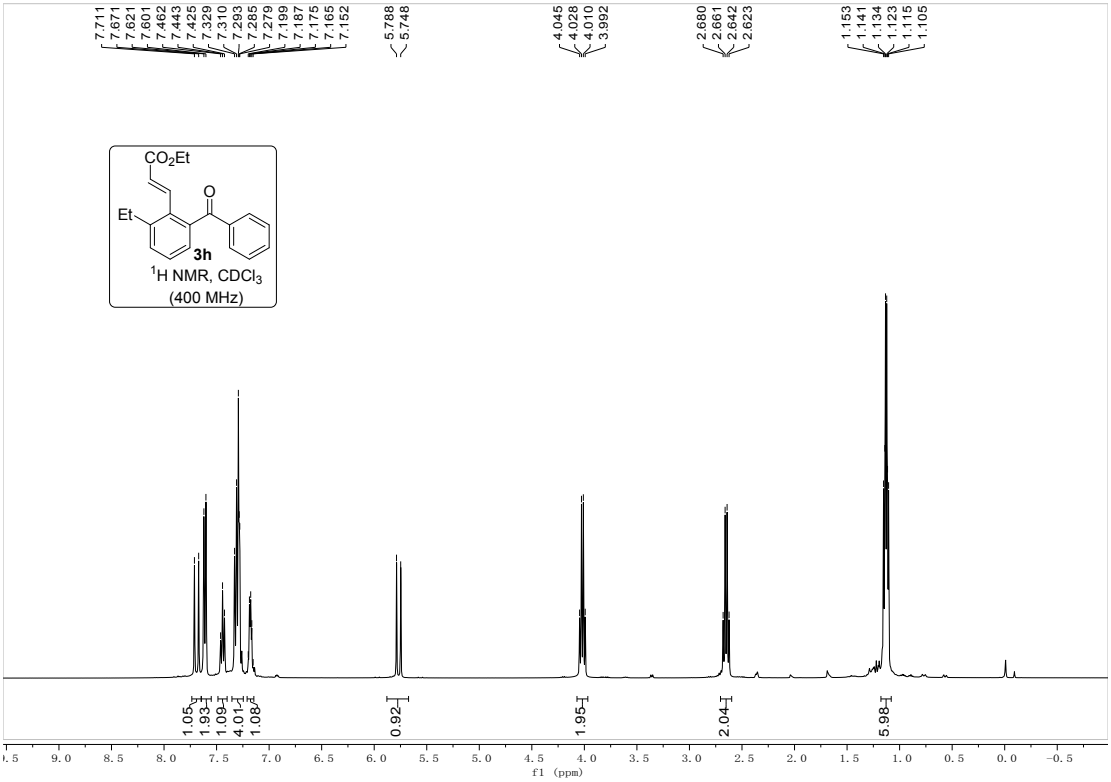
131.826
129.916
129.656
129.457
129.155
128.775
128.355
127.608
127.557
127.503
127.449
127.389
126.659
124.848
122.122
120.332
77.318
77.000
76.683
60.682
14.062
CO2Et
O
F3C
3g
13C NMR, CDCl3
(100 MHz)
f1 (ppm)


-59.369
CO2Et
O
F3C
3g
19F NMR, CDCl3
(376 MHz)
f1 (ppm)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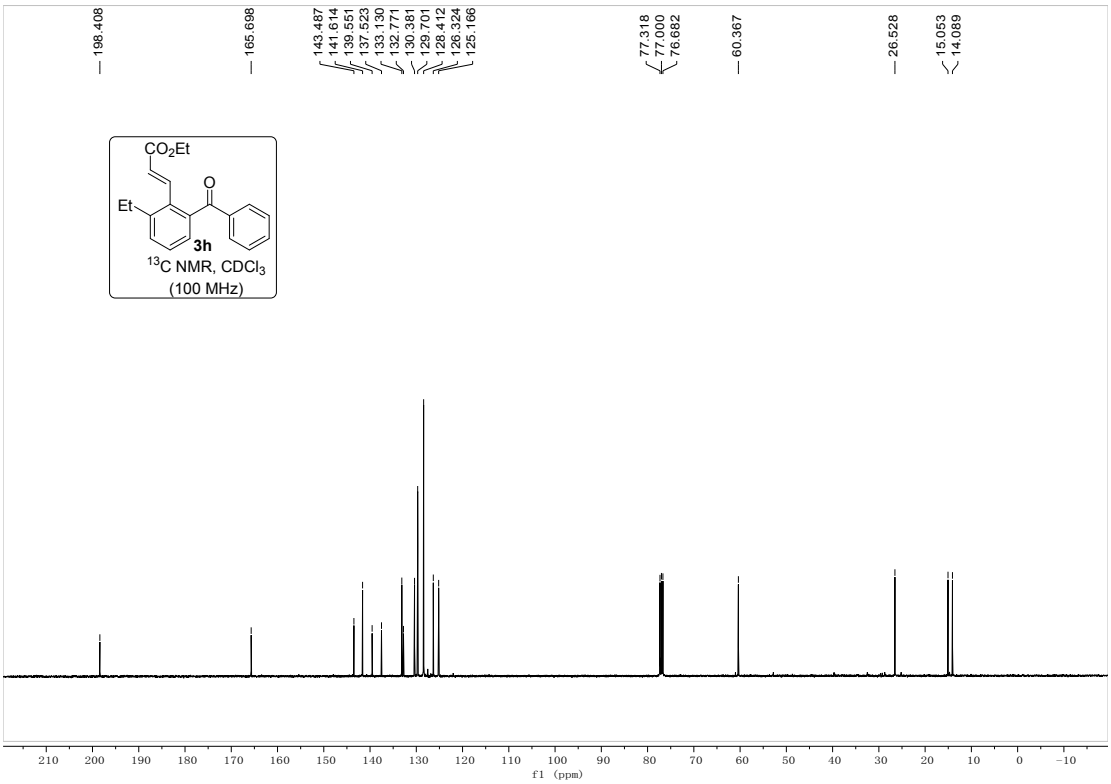
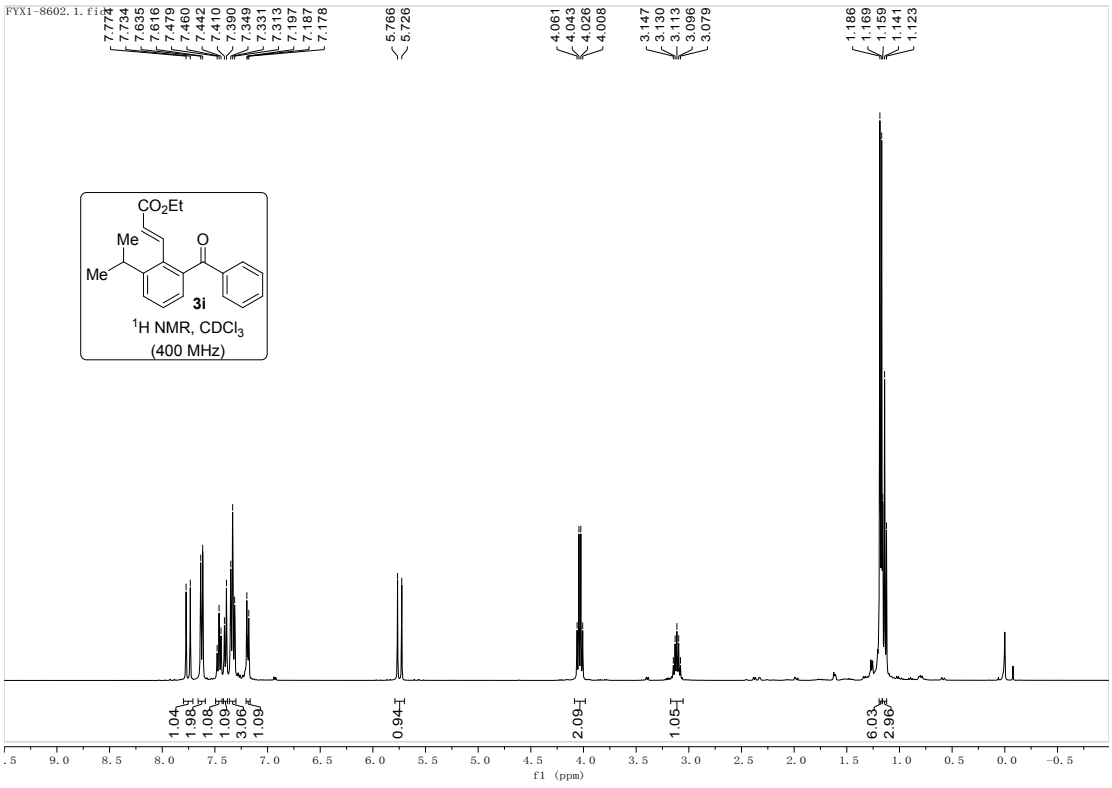
7.671
7.621
7.601
7.462
7.443
7.425
7.329
7.310
7.293
7.285
7.279
7.199
7.187
7.175
7.165
7.152
5.788
5.748
4.045
4.028
4.010
3.992
2.680
2.661
2.642
2.623
1.153
1.141
1.134
1.123
1.115
1.105
CO2Et
O
Et
3h
1H NMR, CDCl3
(400 MHz)
1.05
1.93
1.09
4.01
1.08
0.92
1.95
2.04
5.98
f1 (ppm)


198.408
165.698
143.487
141.614
139.551
137.523
133.130
132.771
130.381
129.701
128.412
126.324
125.166
77.318
77.000
76.682
60.367
26.528
15.053
14.089
CO2Et
O
Et
3h
13C NMR, CDCl3
(100 MHz)
f1 (ppm)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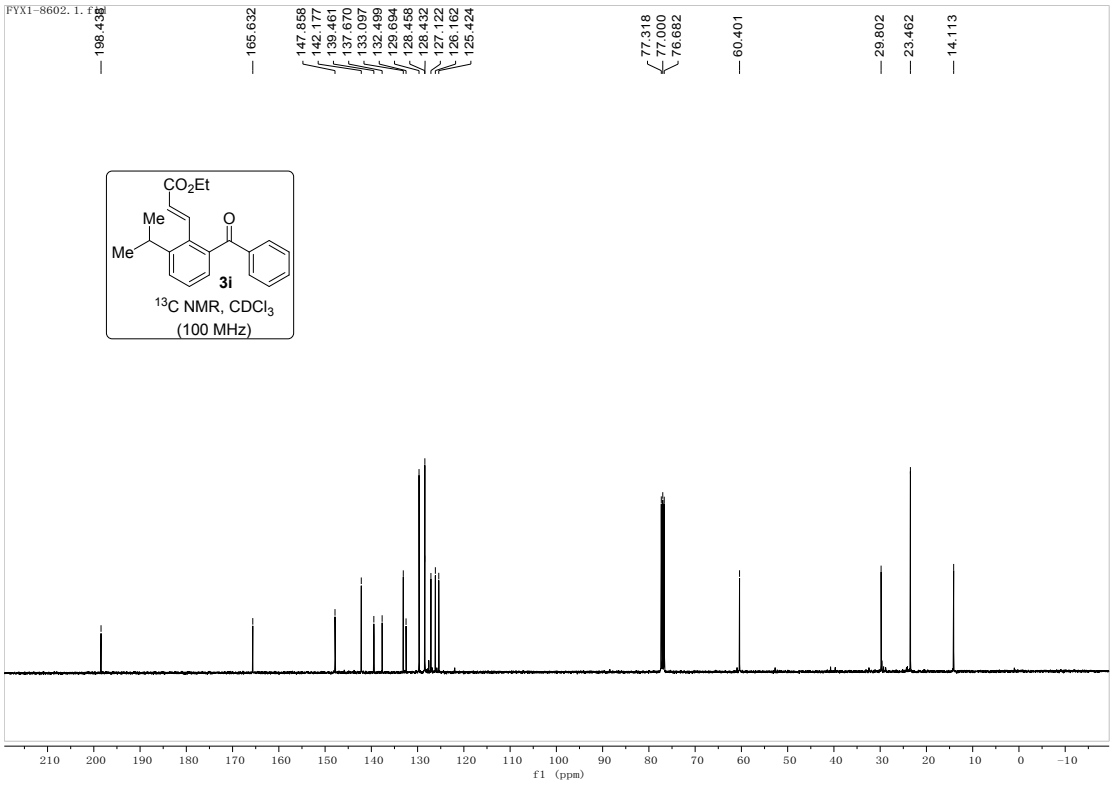
7.734
7.635
7.616
7.479
7.460
7.442
7.410
7.390
7.349
7.331
7.313
7.197
7.187
7.178
5.766
5.726
4.061
4.043
4.026
4.008
3.147
3.130
3.113
3.096
3.079
1.186
1.169
1.159
1.141
1.123
CO2Et
Me
O
Me
3i
1H NMR, CDCl3
(400 MHz)
1.04
1.98
1.08
1.09
3.06
1.09
0.94
2.09
1.05
6.03
2.96
f1 (ppm)


FYX1-8602.1.fid
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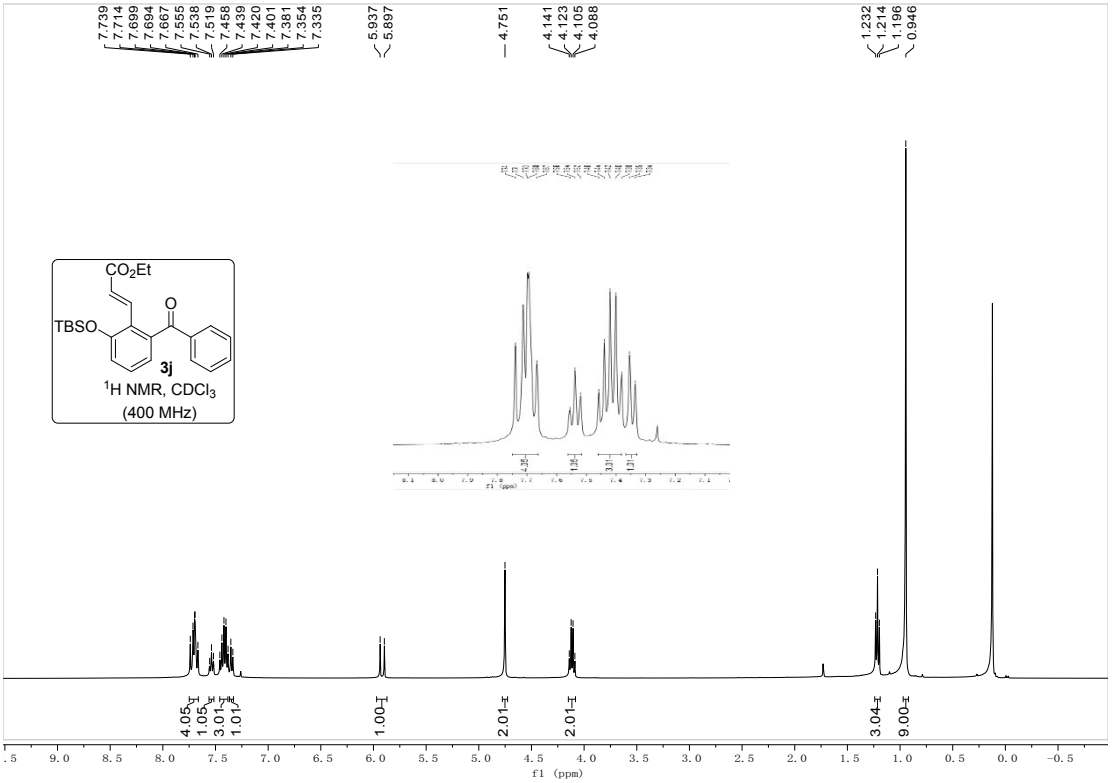
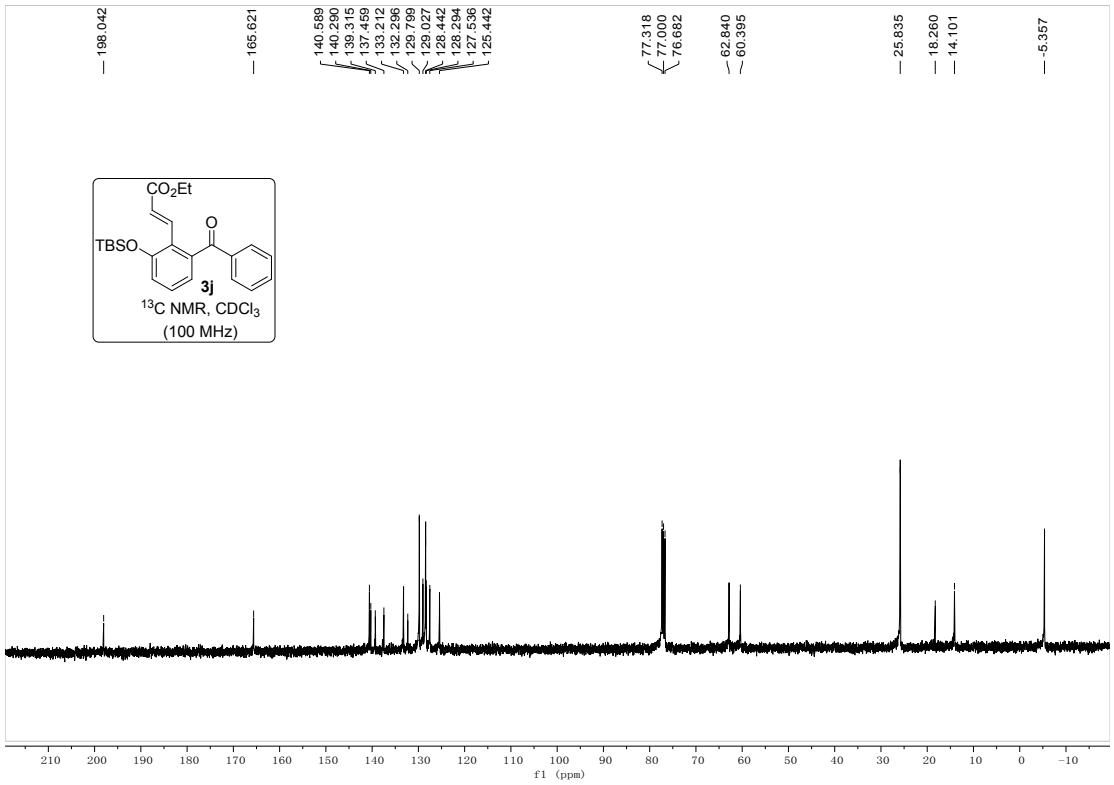
165.632
147.858
142.177
139.461
137.670
133.097
132.499
129.694
128.458
128.432
127.122
126.162
125.424
77.318
77.000
76.682
60.401
29.802
23.462
14.113
CO2Et
Me
Me
O
3i
13C NMR, CDCl3
(100 MHz)
f1 (ppm)


7.739
7.714
7.699
7.694
7.667
7.555
7.538
7.519
7.458
7.439
7.420
7.401
7.381
7.354
7.335
5.937
5.897
4.751
4.141
4.123
4.105
4.088
1.232
1.214
1.196
0.946
CO2Et
TBSO
O
3j
1H NMR, CDCl3
(400 MHz)
4.05
1.05
3.01
1.01
1.00
2.01
2.01
3.04
9.00
f1 (ppm)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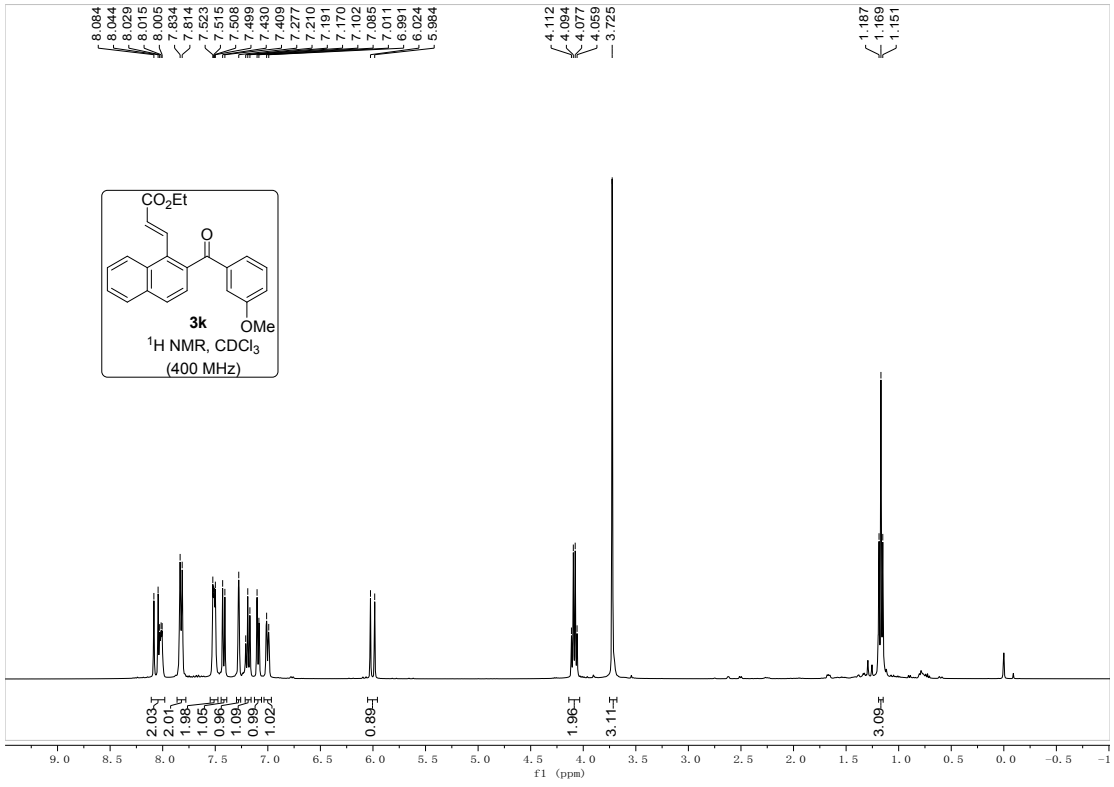
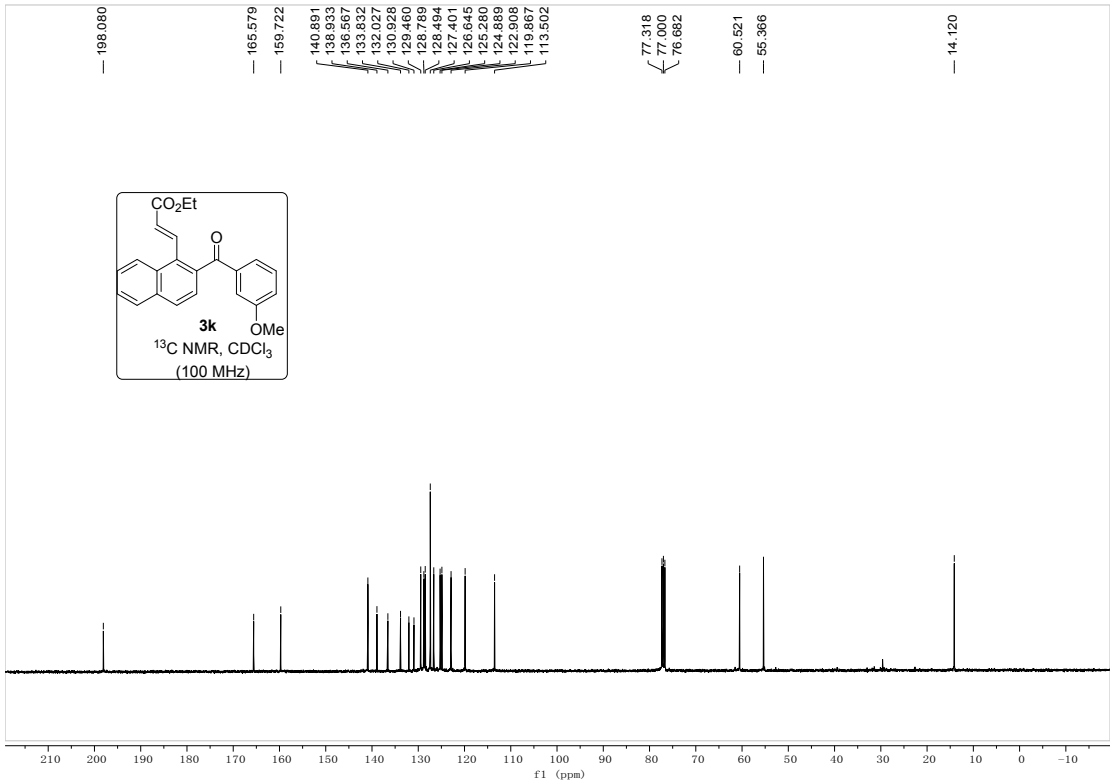
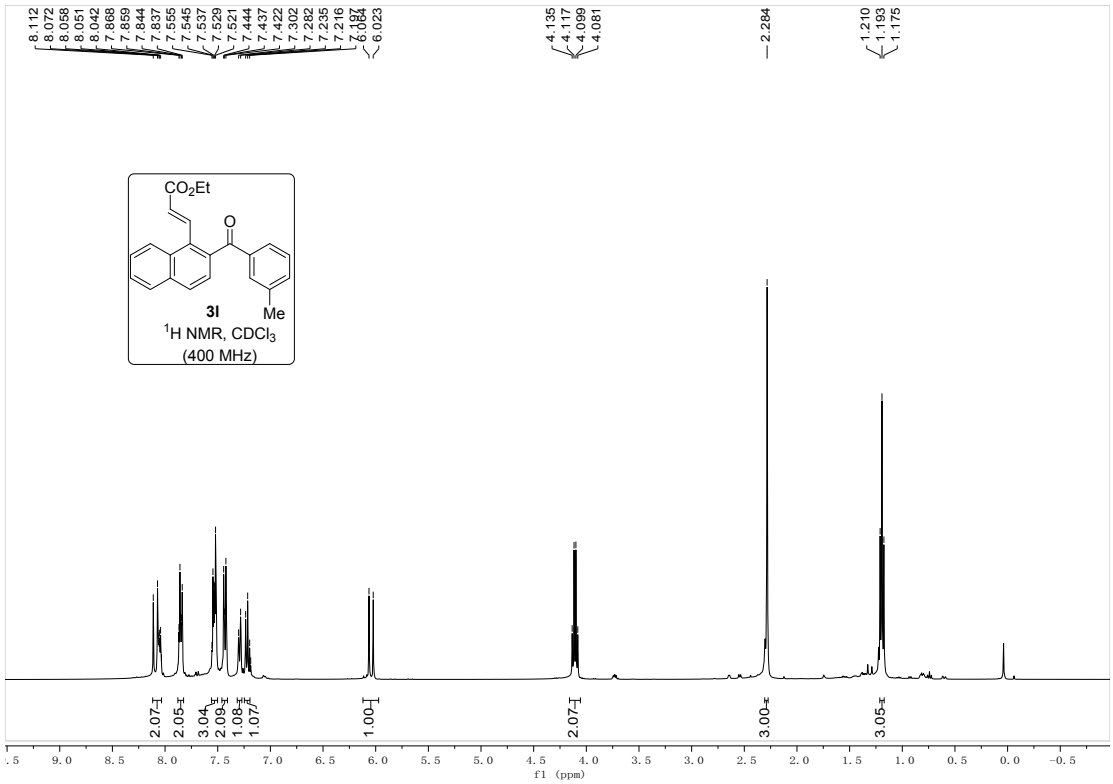
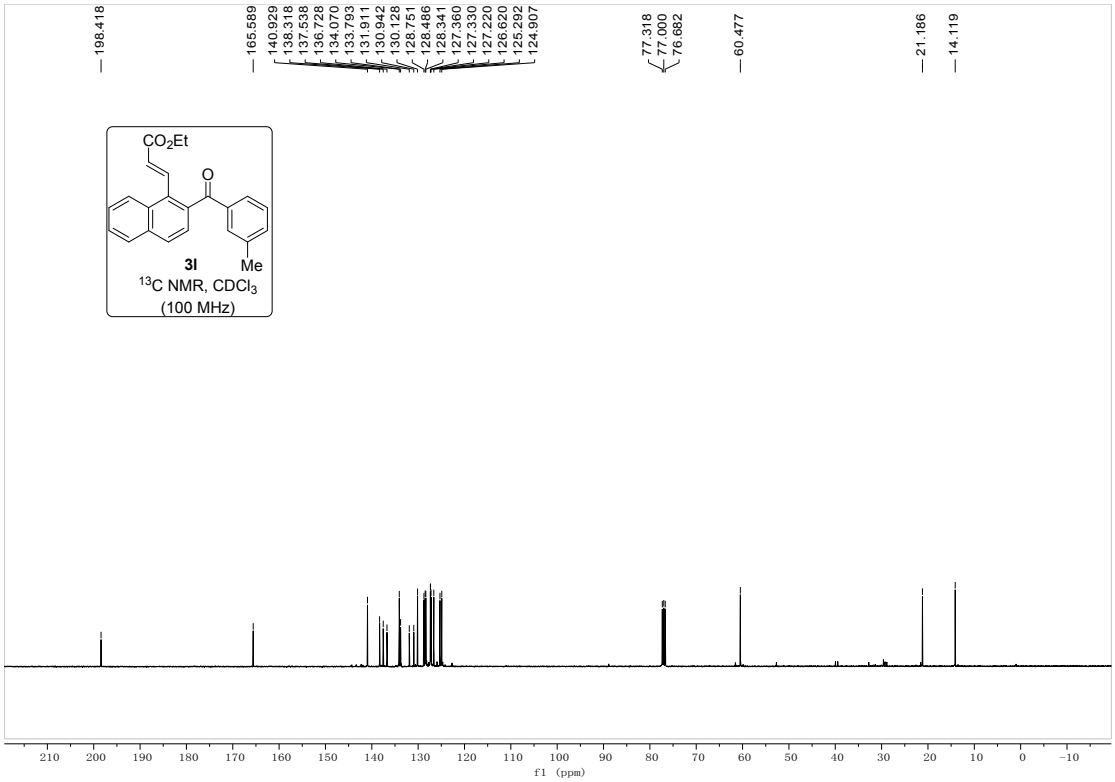
165.621
140.589
140.290
139.315
137.459
133.212
132.296
129.799
129.027
128.442
128.294
127.536
125.442
77.318
77.000
76.682
62.840
60.395
25.835
18.260
14.101
-5.357
CO2Et
O
TBSO
3j
13C NMR, CDCl3
(100 MHz)
f1 (ppm)


8.084
8.044
8.029
8.015
8.005
7.834
7.814
7.523
7.515
7.508
7.499
7.430
7.409
7.277
7.210
7.191
7.170
7.102
7.085
7.011
6.991
6.024
5.984
4.112
4.094
4.077
4.059
3.725
1.187
1.169
1.151
CO2Et
O
3k
OMe
1H NMR, CDCl3
(400 MHz)
2.03
2.01
1.98
1.05
0.96
1.09
0.99
1.02
0.89
1.96
3.11
3.09
f1 (ppm)


198.080
165.579
159.722
140.891
138.933
136.567
133.832
132.027
130.928
129.460
128.789
128.494
127.401
126.645
125.280
124.889
122.908
119.867
113.502
77.318
77.000
76.682
60.521
55.366
14.120
CO2Et
O
3k
OMe
13C NMR, CDCl3
(100 MHz)
f1 (ppm)


8.112
8.072
8.058
8.051
8.042
7.868
7.859
7.844
7.837
7.555
7.545
7.537
7.529
7.521
7.444
7.437
7.422
7.302
7.282
7.235
7.216
7.084
6.023
4.135
4.117
4.099
4.081
2.284
1.210
1.193
1.175
CO2Et
O
3l
Me
1H NMR, CDCl3
(400 MHz)
2.07
2.05
3.04
2.09
1.08
1.07
1.00
2.07
3.00
3.05
f1 (ppm)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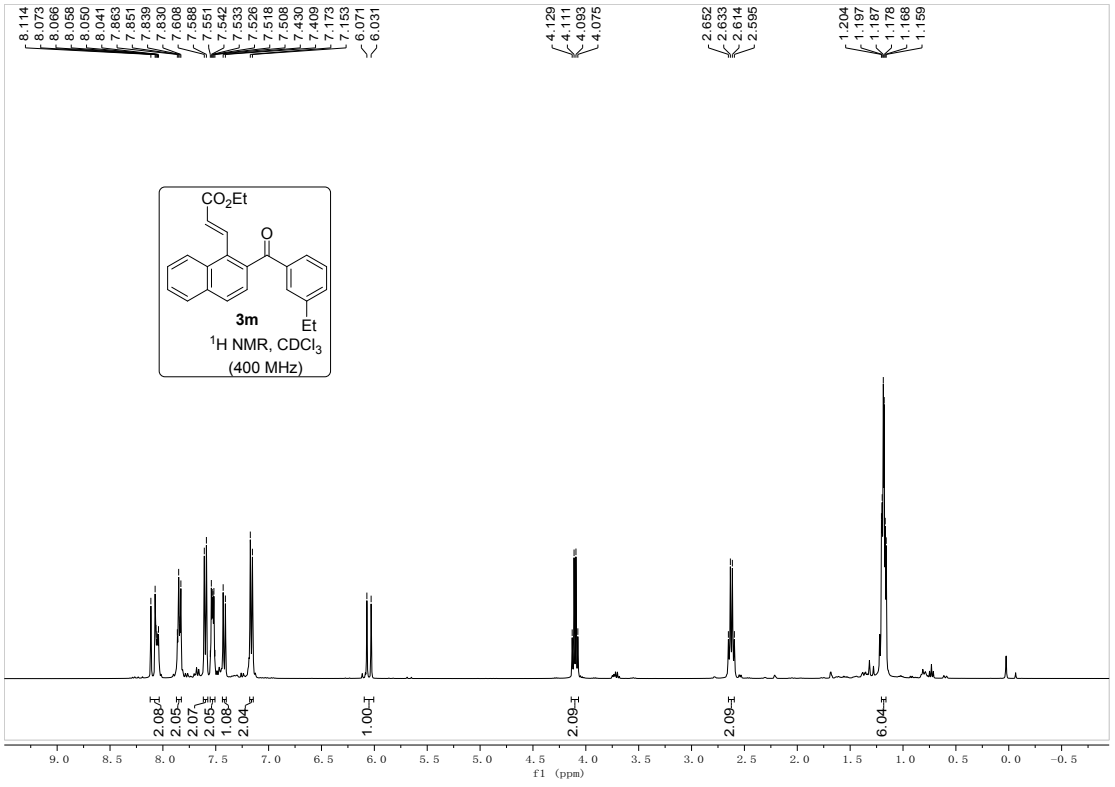
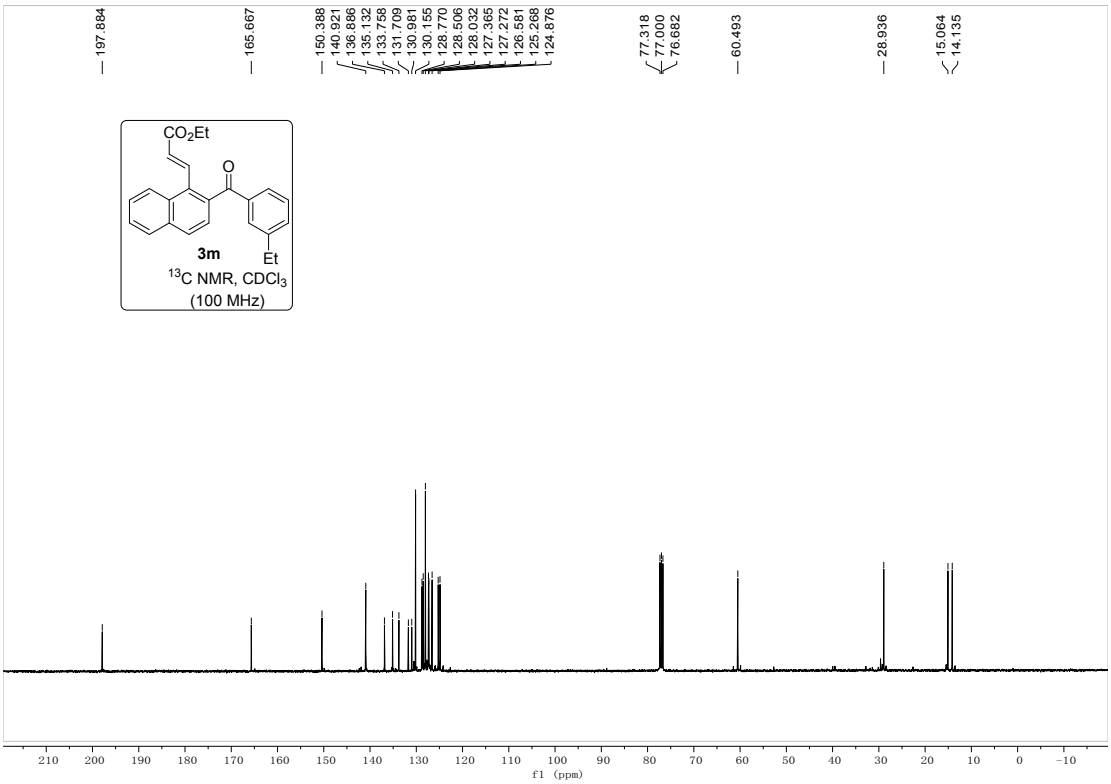
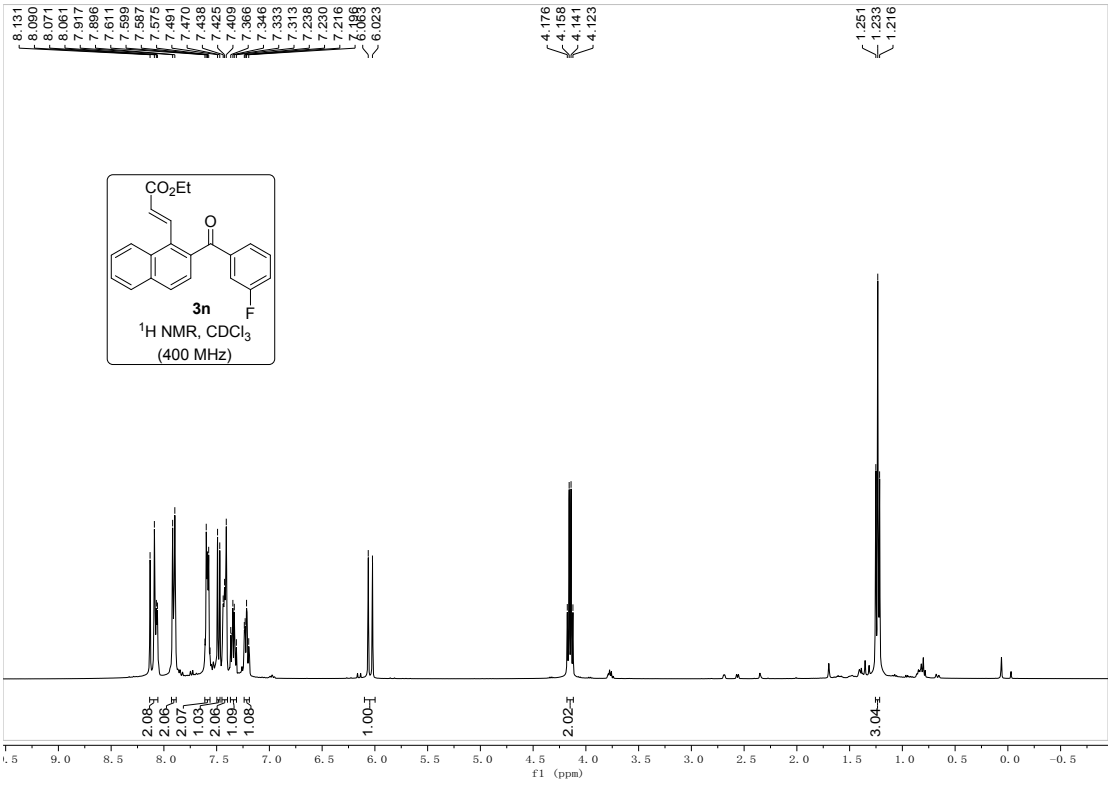
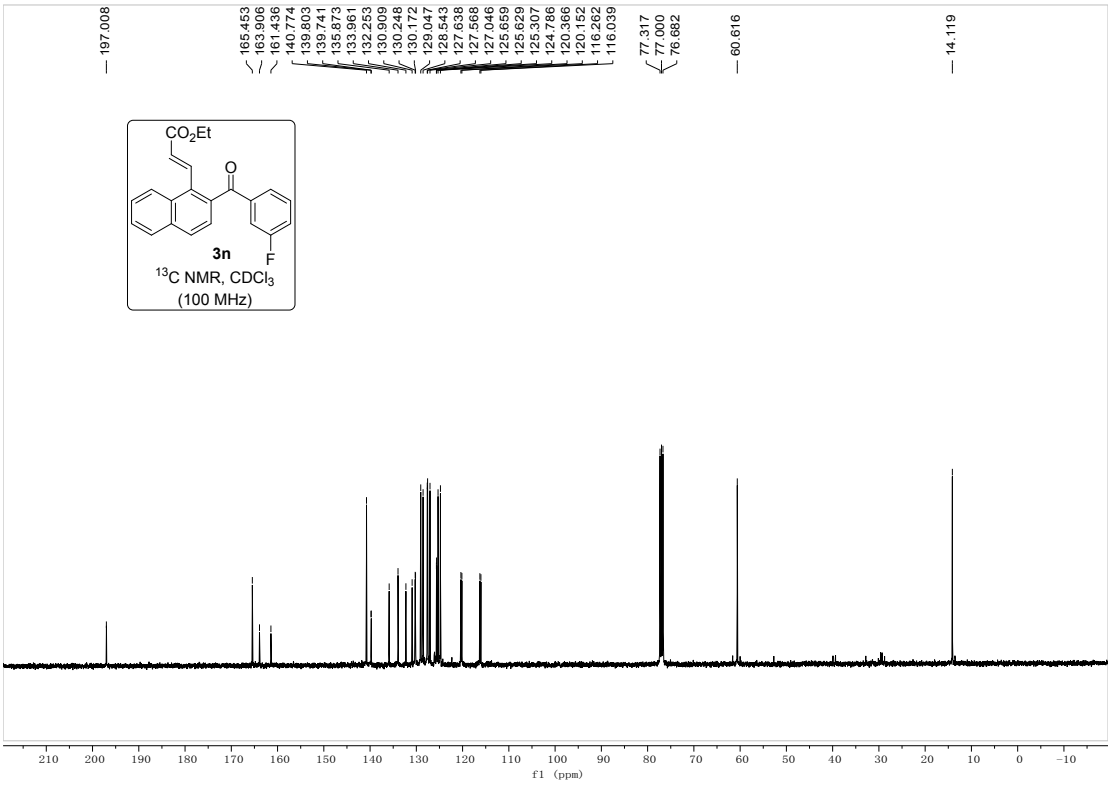
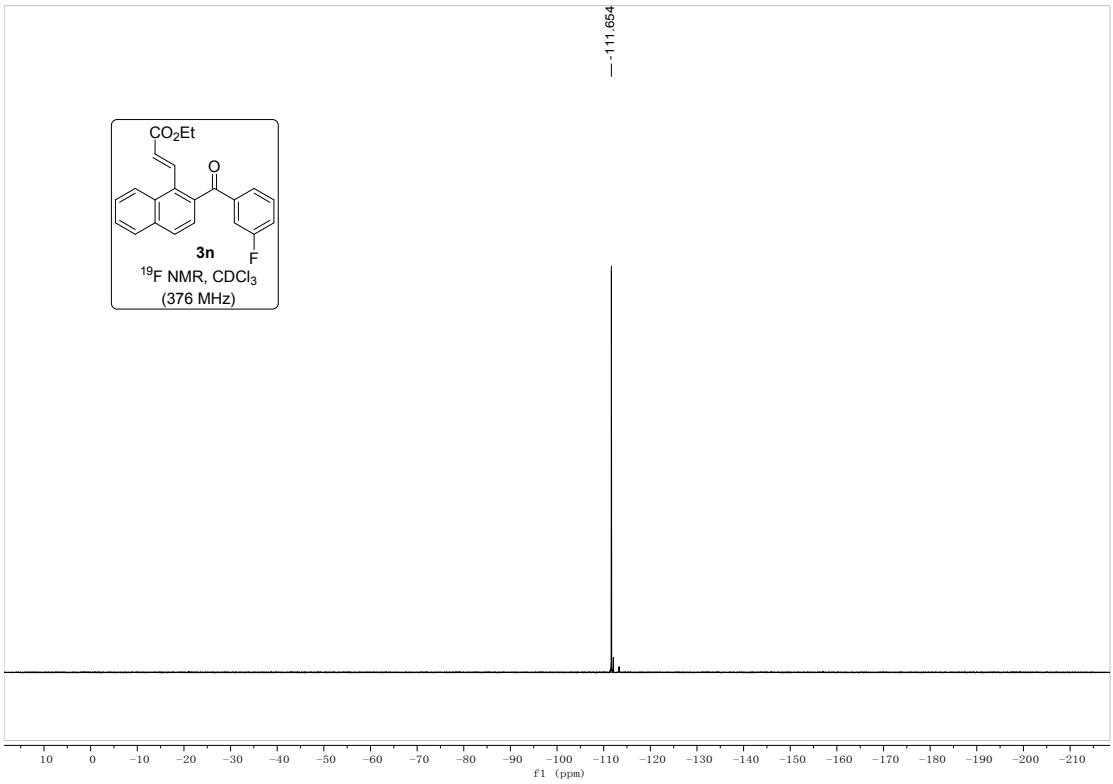
140.929
138.318
137.538
136.728
134.070
133.793
131.911
130.942
130.128
128.751
128.486
128.341
127.360
127.330
127.220
126.620
125.292
124.907
77.318
77.000
76.682
60.477
21.186
14.119
CO2Et
O
Me
3l
13C NMR, CDCl3
(100 MHz)
f1 (ppm)

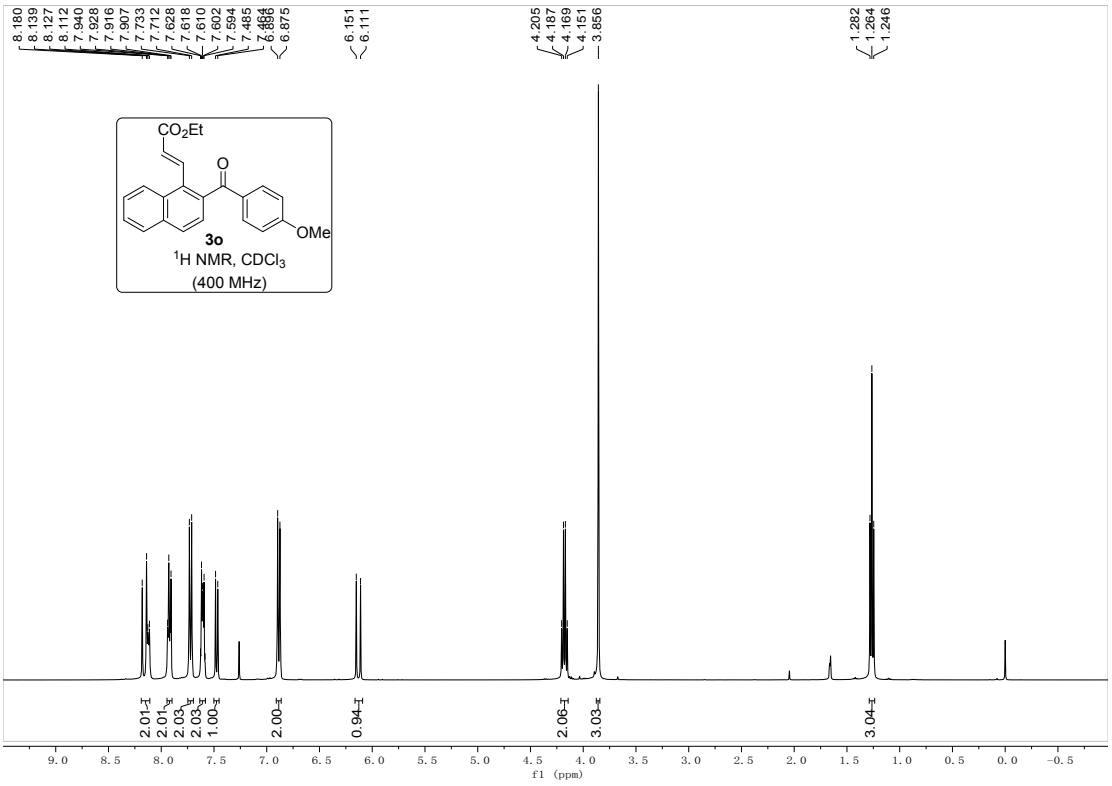

8.114
8.073
8.066
8.058
8.050
8.041
7.863
7.851
7.839
7.830
7.608
7.588
7.551
7.542
7.533
7.526
7.518
7.508
7.430
7.409
7.173
7.153
6.071
6.031
4.129
4.111
4.093
4.075
2.652
2.633
2.614
2.595
1.204
1.197
1.187
1.178
1.168
1.159
CO2Et
O
Et
3m
1H NMR, CDCl3
(400 MHz)
2.08
2.05
2.07
2.05
1.08
2.04
1.00
2.09
2.09
6.04
f1 (ppm)


197.884
165.667
150.388
140.921
136.886
135.132
133.758
131.709
130.981
130.155
128.770
128.506
128.032
127.365
127.272
126.581
125.268
124.876
77.318
77.000
76.682
60.493
28.936
15.064
14.135
CO2Et
O
3m
Et
13C NMR, CDCl3
(100 MHz)
f1 (ppm)


8.131
8.090
8.071
8.061
7.917
7.896
7.611
7.599
7.587
7.575
7.491
7.470
7.438
7.425
7.409
7.366
7.346
7.333
7.313
7.238
7.230
7.216
6.023
4.176
4.158
4.141
4.123
1.251
1.233
1.216
CO2Et
O
3n
F
1H NMR, CDCl3
(400 MHz)
2.08
2.06
2.07
1.03
2.06
1.09
1.08
1.00
2.02
3.04
f1 (ppm)


197.008
165.453
163.906
161.436
140.774
139.803
139.741
135.873
133.961
132.253
130.909
130.248
130.172
129.047
128.543
127.638
127.568
127.046
125.659
125.629
125.307
124.786
120.366
120.152
116.262
116.039
77.317
77.000
76.682
60.616
14.119
CO2Et
O
F
3n
13C NMR, CDCl3
(100 MHz)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)


-111.654
CO2Et
O
F
3n
19F NMR, CDCl3
(376 MHz)
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210
f1 (ppm)


8.180
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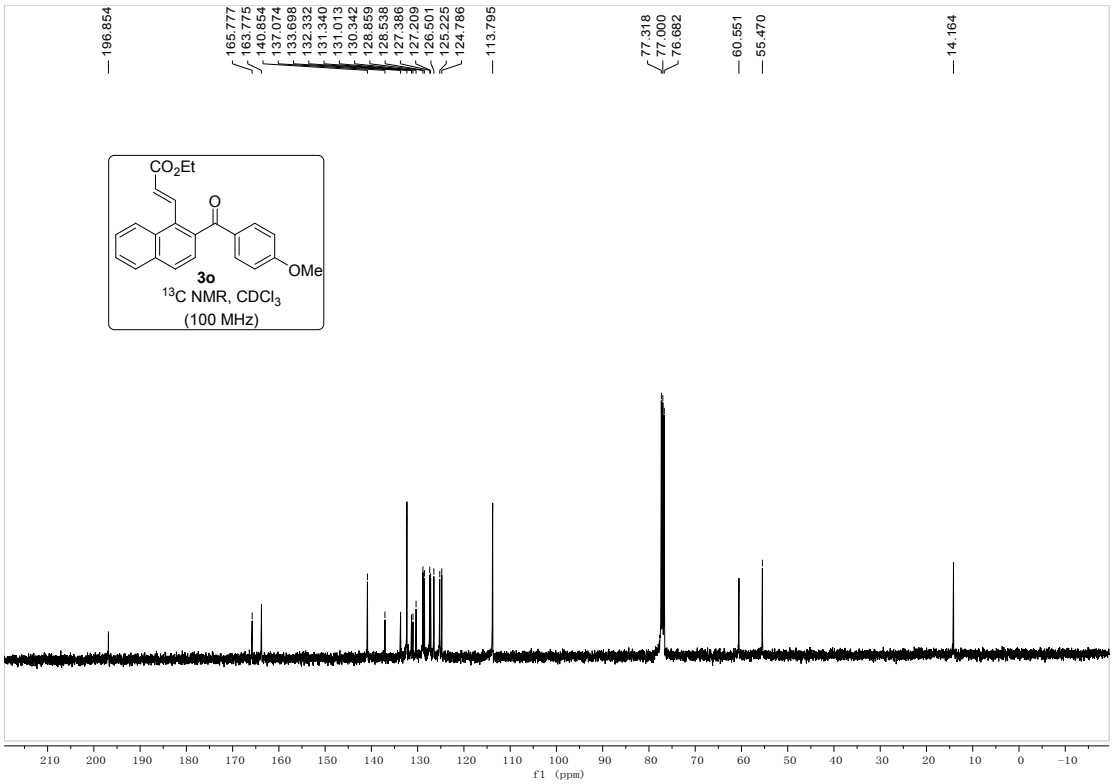
8.127
8.112
7.940
7.928
7.916
7.907
7.733
7.712
7.628
7.618
7.610
7.602
7.594
7.485
6.886
6.875
6.151
6.111
4.205
4.187
4.169
4.151
3.856
1.282
1.264
1.246
CO2Et
O
OMe
3o
1H NMR, CDCl3
(400 MHz)
2.01
2.01
2.03
2.03
1.00
2.00
0.94
2.06
3.03
3.04
f1 (ppm)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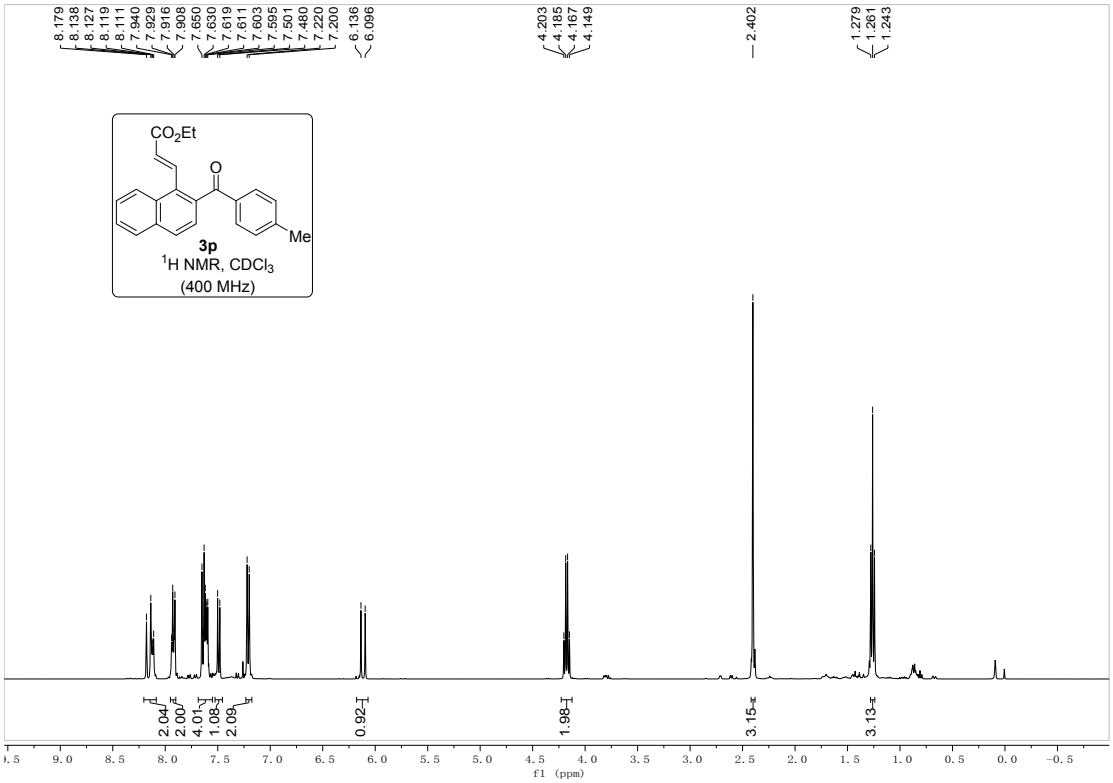
163.775
140.854
137.074
133.698
132.332
131.340
131.013
130.342
128.859
128.538
127.386
127.209
126.501
125.225
124.786
113.795
77.318
77.000
76.682
60.551
55.470
14.164
CO2Et
O
OMe
3o
13C NMR, CDCl3
(100 MHz)
f1 (ppm)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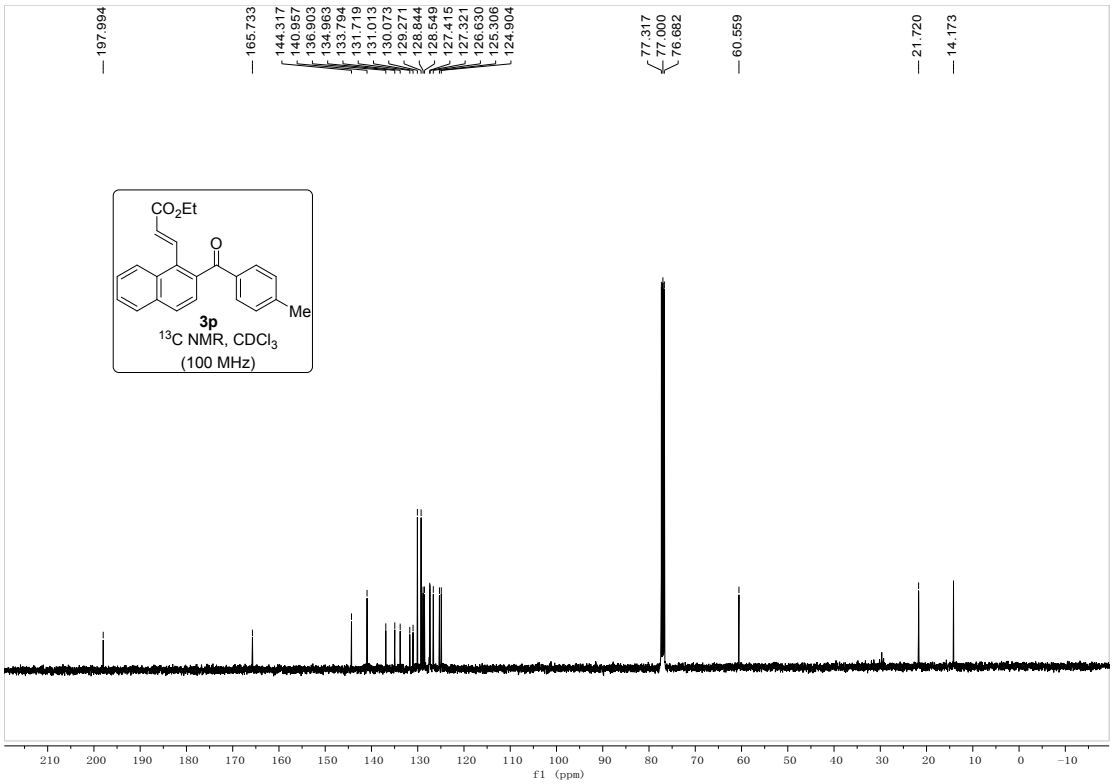
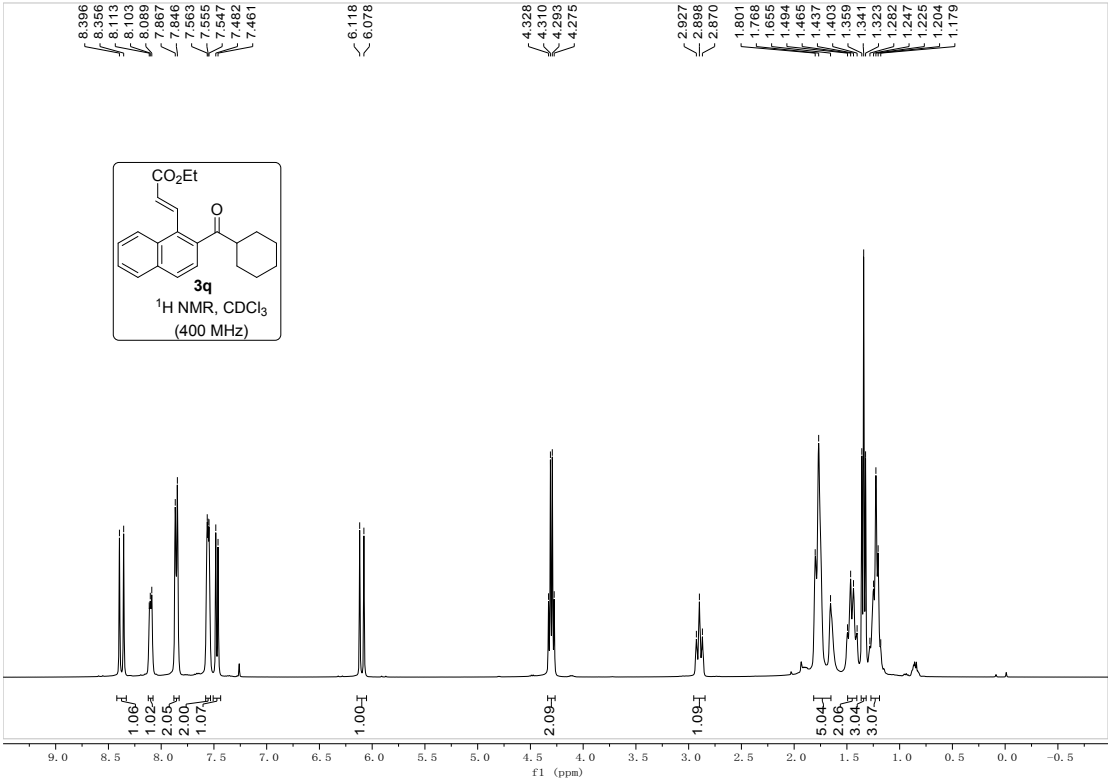
8.111
7.940
7.929
7.916
7.908
7.650
7.630
7.619
7.611
7.603
7.595
7.501
7.480
7.220
7.200
6.136
6.096
4.203
4.185
4.167
4.149
2.402
1.279
1.261
1.243
CO2Et
O
Me
3p
1H NMR, CDCl3
(400 MHz)
2.04
2.00
4.01
1.08
2.09
0.92
1.98
3.15
3.13
f1 (ppm)


197.994
165.733
144.317
140.957
138.903
134.963
133.794
131.719
131.013
130.073
129.271
128.844
128.549
127.415
127.321
126.630
125.306
124.904
77.317
77.000
76.682
60.559
21.720
14.173
CO2Et
O
Me
3p
13C NMR, CDCl3
(100 MHz)
f1 (ppm)


8.396
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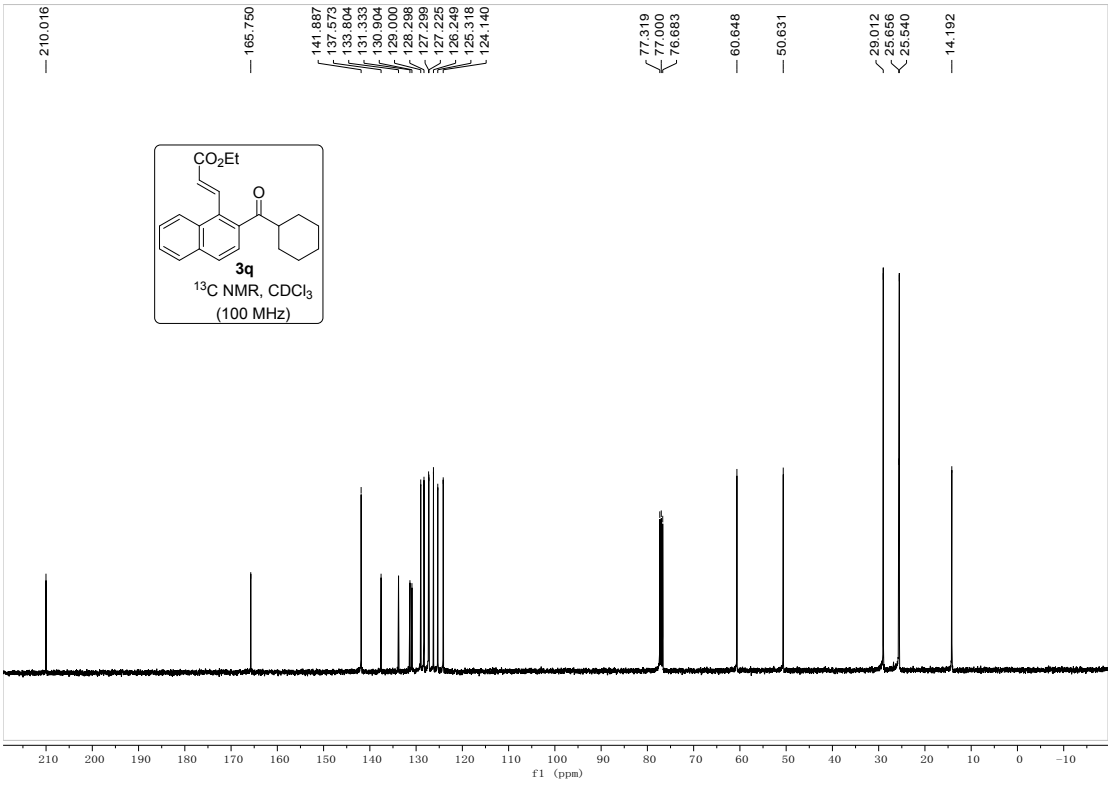
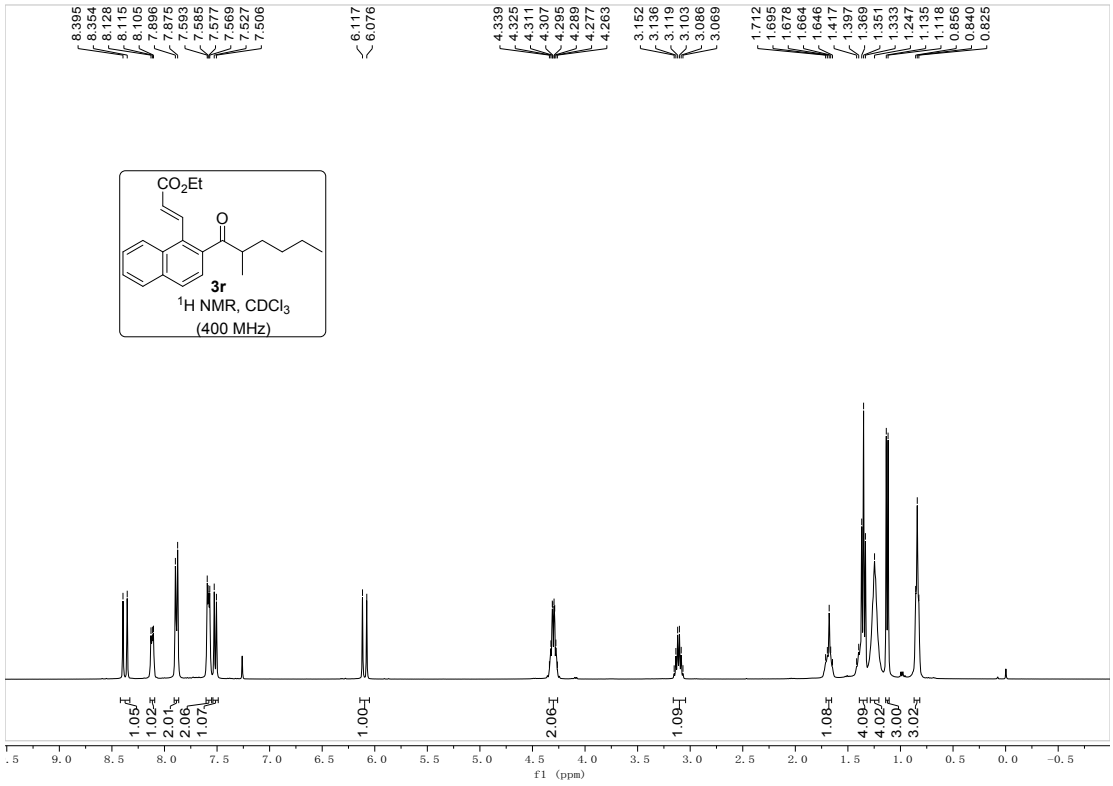
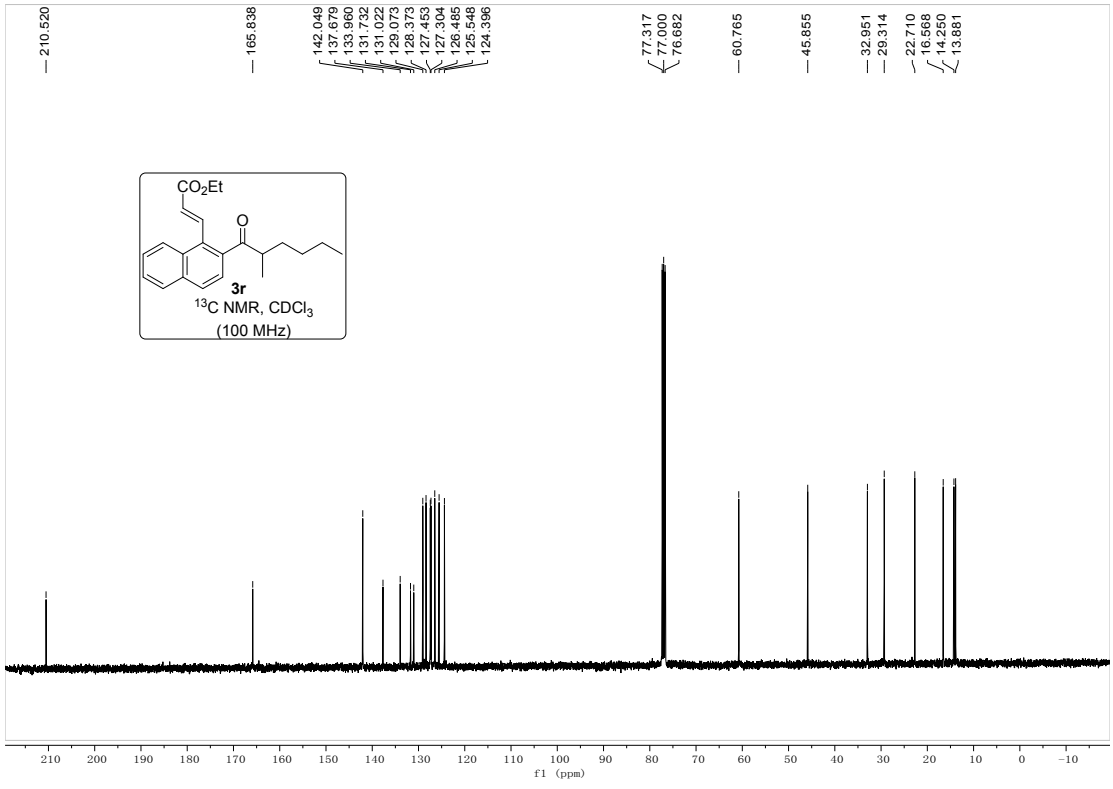
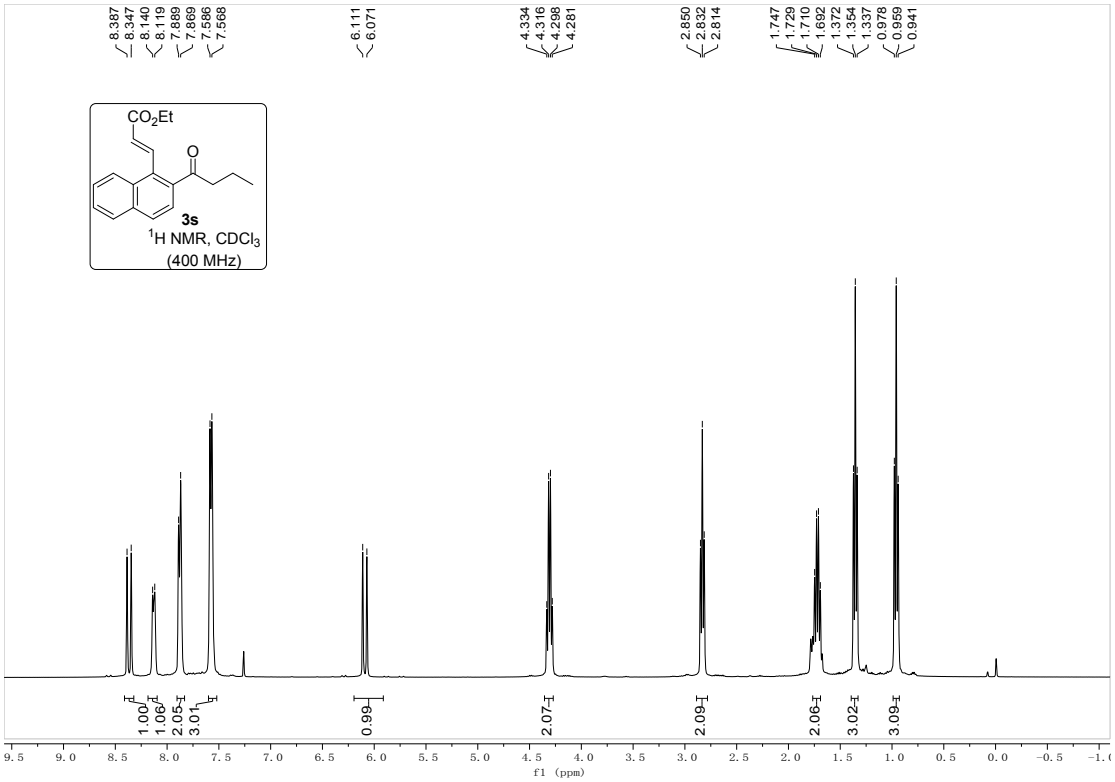
8.113
8.103
8.089
7.867
7.846
7.563
7.555
7.547
7.482
7.461
6.118
6.078
4.328
4.310
4.293
4.275
2.927
2.898
2.870
1.801
1.768
1.655
1.494
1.465
1.437
1.403
1.359
1.341
1.323
1.282
1.247
1.225
1.204
1.179
CO2Et
O
3q
1H NMR, CDCl3
(400 MHz)
1.06
1.02
2.05
2.00
1.07
1.00
2.09
1.09
5.04
2.06
3.04
3.07
f1 (ppm)


210.016
165.750
141.887
137.573
133.804
131.333
130.904
129.000
128.298
127.299
127.225
126.249
125.318
124.140
77.319
77.000
76.683
60.648
50.631
29.012
25.656
25.540
14.192
CO2Et
O
3q
13C NMR, CDCl3
(100 MHz)
f1 (ppm)


8.395
8.354
8.128
8.115
8.105
7.896
7.875
7.593
7.585
7.577
7.569
7.527
7.506
6.117
6.076
4.339
4.325
4.311
4.307
4.295
4.289
4.277
4.263
3.152
3.136
3.119
3.103
3.086
3.069
1.712
1.695
1.678
1.664
1.646
1.417
1.397
1.369
1.351
1.333
1.247
1.135
1.118
0.856
0.840
0.825
CO2Et
O
3r
1H NMR, CDCl3
(400 MHz)
1.05
1.02
2.01
2.06
1.07
1.00
2.06
1.09
1.08
4.09
4.02
3.00
3.02
f1 (ppm)


210.520
165.838
142.049
137.679
133.960
131.732
131.022
129.073
128.373
127.453
127.304
126.485
125.548
124.396
77.317
77.000
76.682
60.765
45.855
32.951
29.314
22.710
16.568
14.250
13.881
CO2Et
O
3r
13C NMR, CDCl3
(100 MHz)
f1 (ppm)


8.387
8.347
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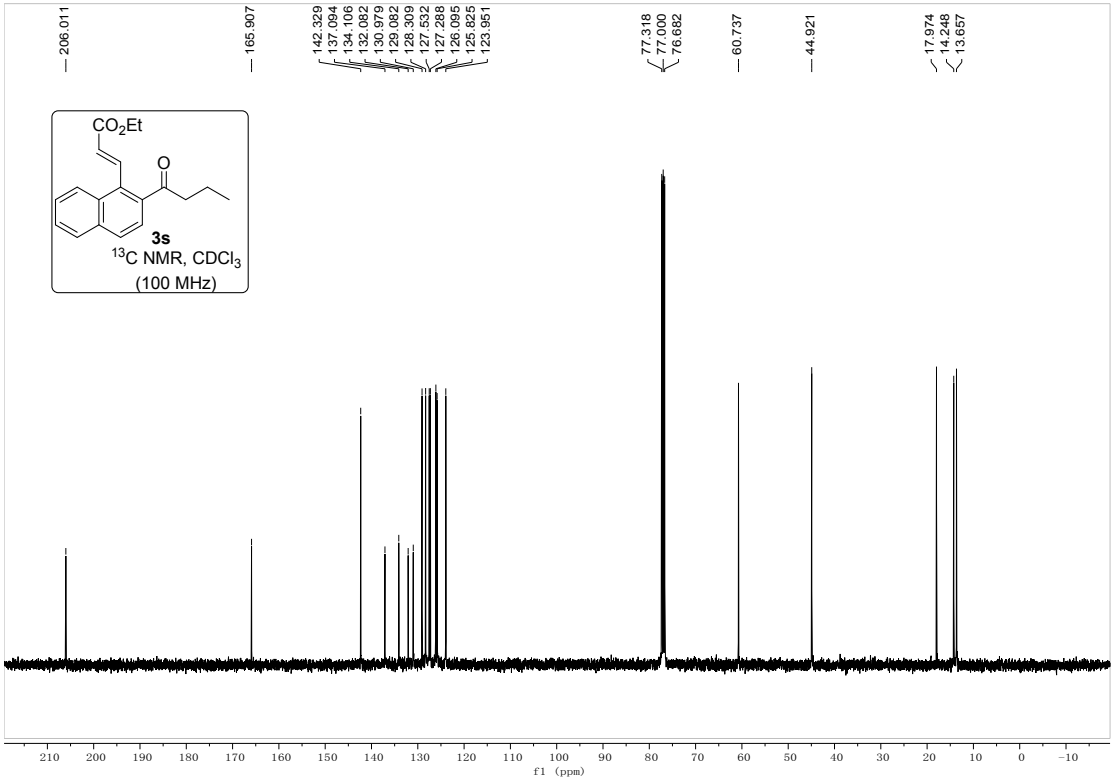
8.119
7.889
7.869
7.586
7.568
6.111
6.071
4.334
4.316
4.298
4.281
2.850
2.832
2.814
1.747
1.729
1.710
1.692
1.372
1.354
1.337
0.978
0.959
0.941
CO2Et
O
3s
1H NMR, CDCl3
(400 MHz)
1.00
1.06
2.05
3.01
0.99
2.07
2.09
2.06
3.02
3.09
f1 (ppm)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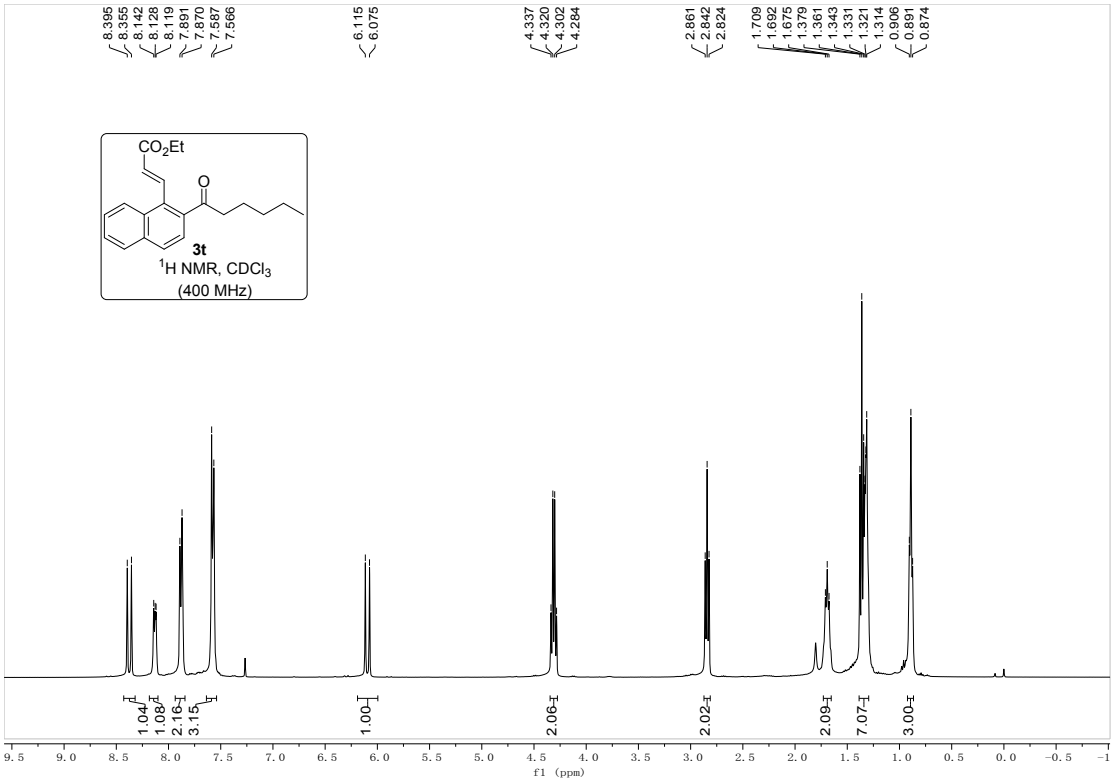
129.082
128.309
127.532
127.288
126.095
125.825
123.951
77.318
77.000
76.682
60.737
44.921
17.974
14.248
13.657
CO2Et
O
3s
13C NMR, CDCl3
(100 MHz)
f1 (ppm)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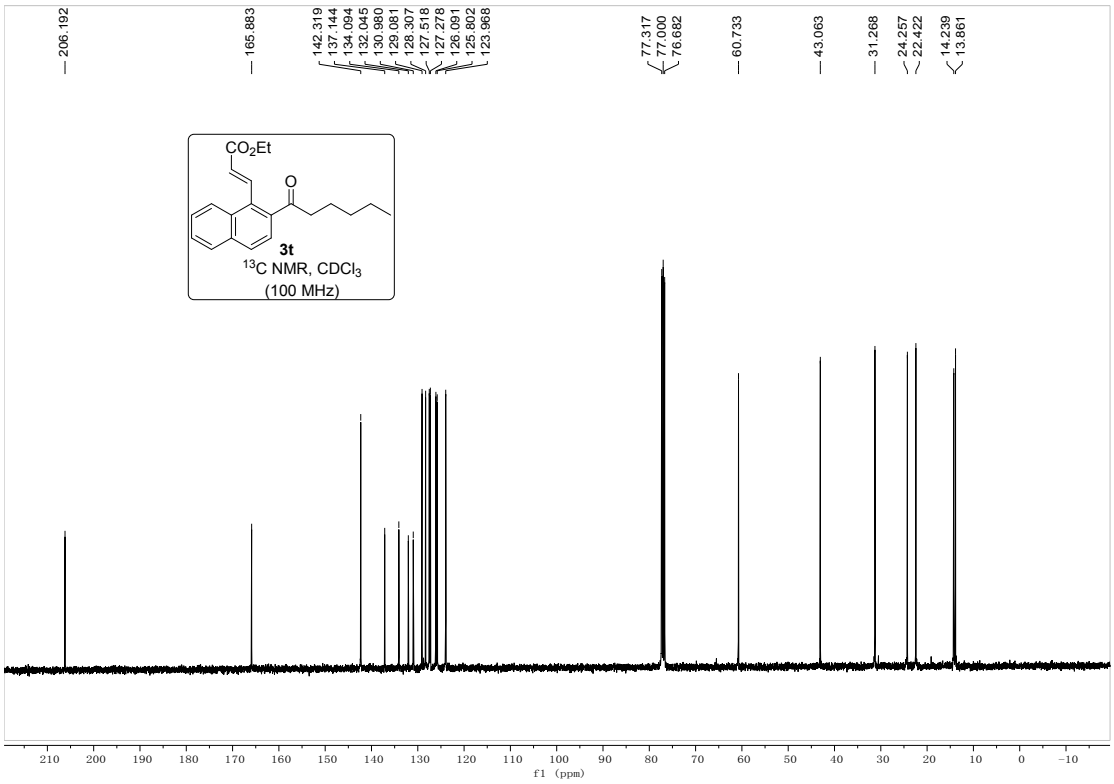
8.355
8.142
8.128
8.119
7.891
7.870
7.587
7.566
6.115
6.075
4.337
4.320
4.302
4.284
2.861
2.842
2.824
1.709
1.692
1.675
1.379
1.361
1.343
1.331
1.321
1.314
0.906
0.891
0.874
CO2Et
O
3t
1H NMR, CDCl3
(400 MHz)
1.04
1.08
2.16
3.15
1.00
2.06
2.02
2.09
7.07
3.00
f1 (ppm)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142.319
137.144
134.094
132.045
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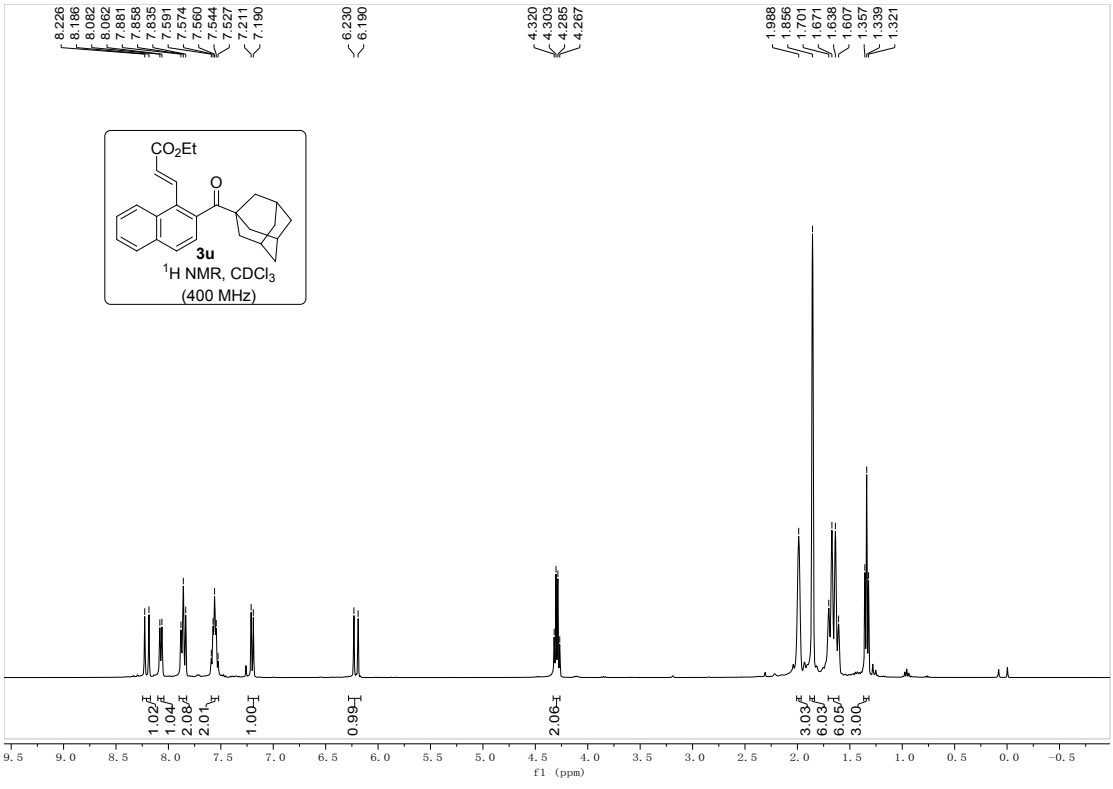
129.081
128.307
127.518
127.278
126.091
125.802
123.968
77.317
77.000
76.682
60.733
43.063
31.268
24.257
22.422
14.239
13.861
CO2Et
O
3t
13C NMR, CDCl3
(100 MHz)
f1 (ppm)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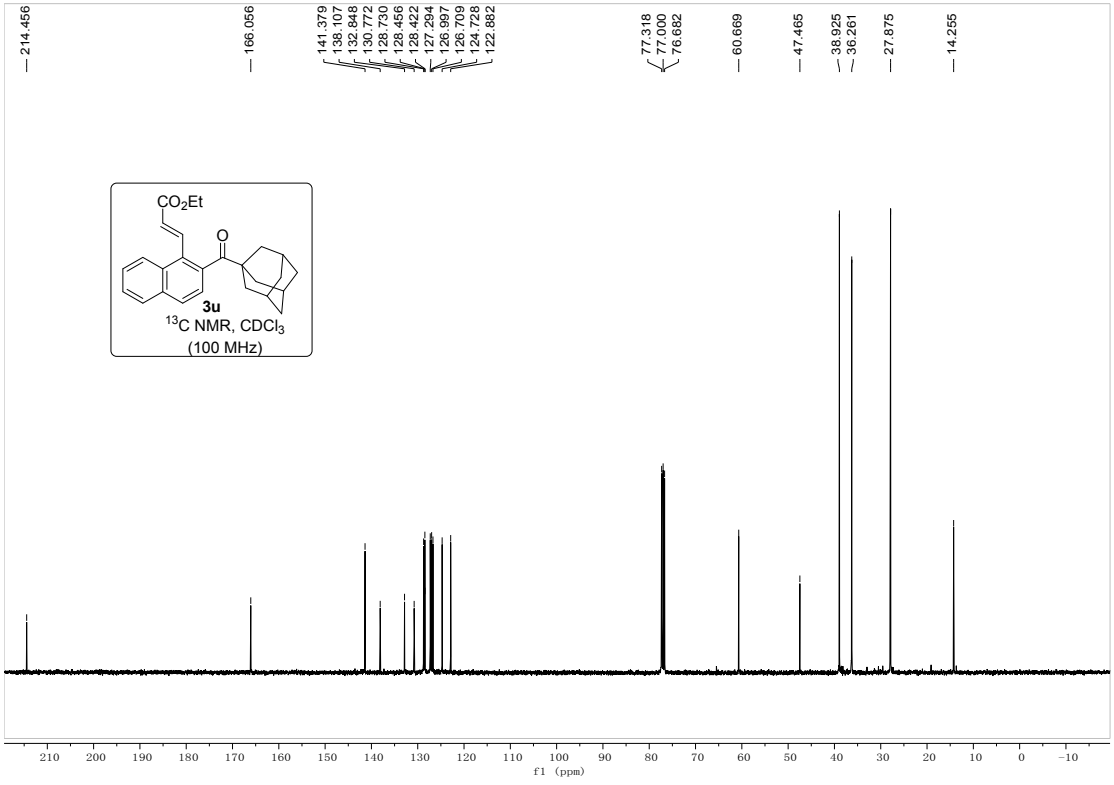
8.186
8.082
8.062
7.881
7.858
7.835
7.591
7.574
7.560
7.544
7.527
7.211
7.190
6.230
6.190
4.320
4.303
4.285
4.267
1.988
1.856
1.701
1.671
1.638
1.607
1.357
1.339
1.321
CO2Et
O
3u
1H NMR, CDCl3
(400 MHz)
1.02
1.04
2.08
2.01
1.00
0.99
2.06
3.03
6.03
6.05
3.00
f1 (ppm)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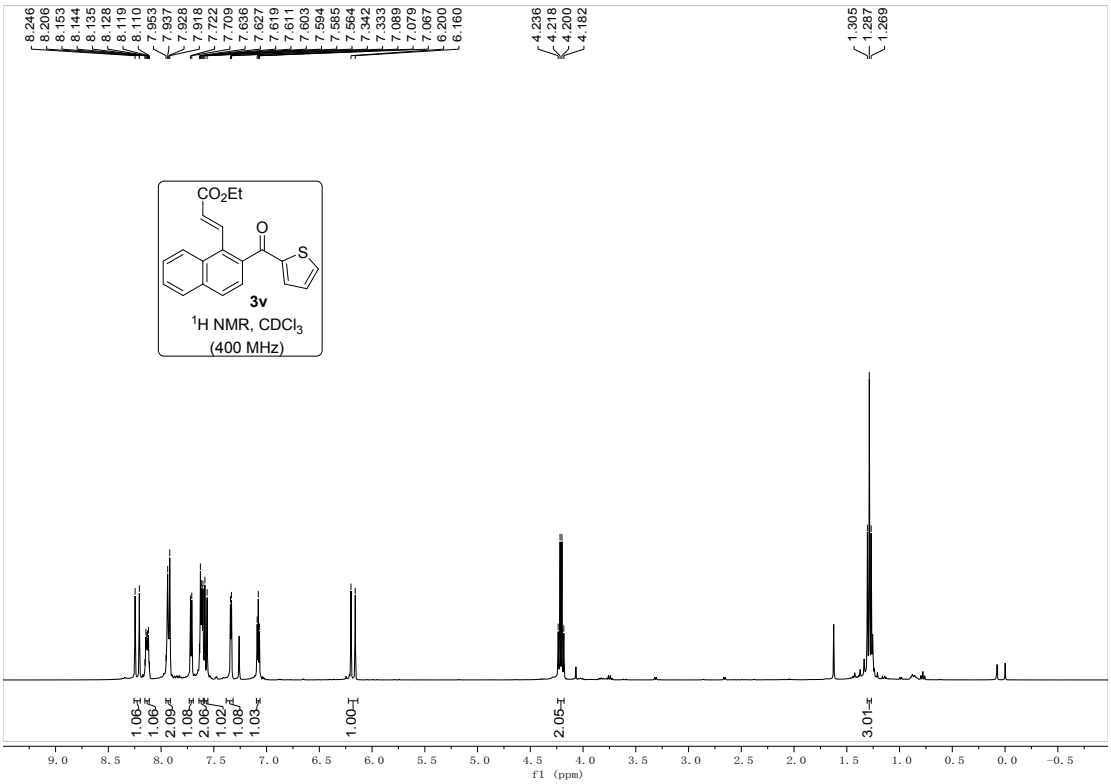
166.056
141.379
138.107
132.848
130.772
128.730
128.456
128.422
127.294
126.997
126.709
124.728
122.882
77.318
77.000
76.682
60.669
47.465
38.925
36.261
27.875
14.255
CO2Et
O
3u
13C NMR, CDCl3
(100 MHz)
f1 (ppm)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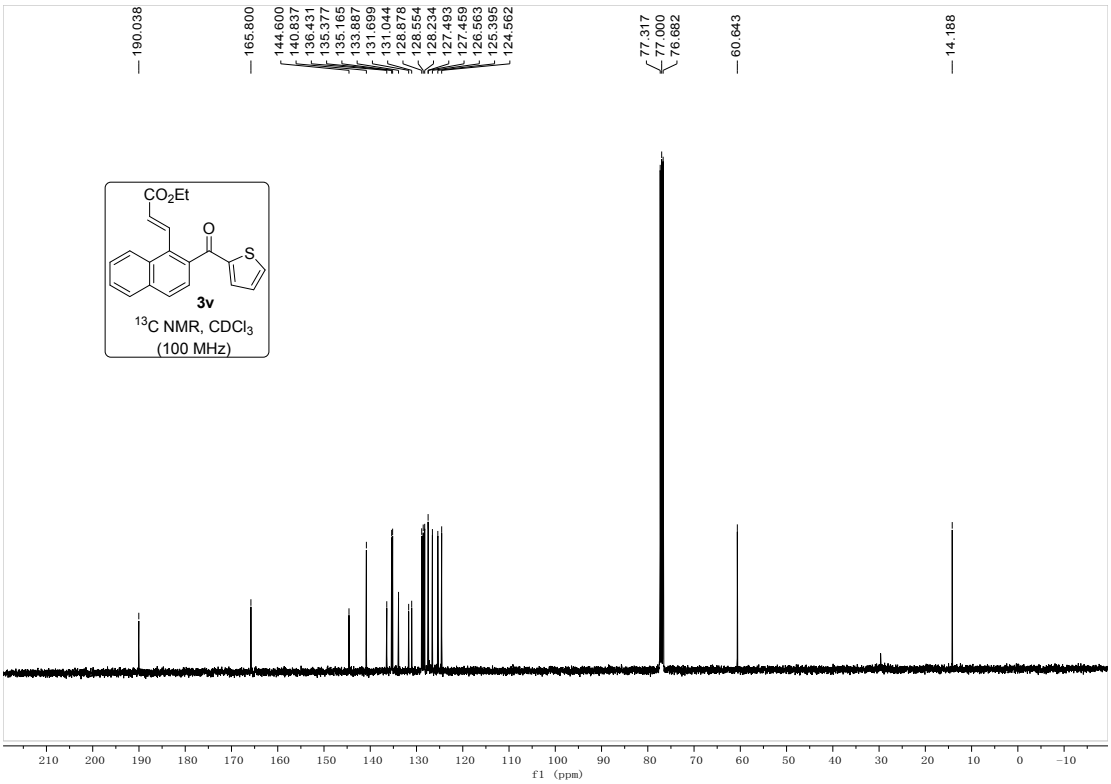
8.144
8.135
8.128
8.119
8.110
7.953
7.937
7.928
7.918
7.722
7.709
7.636
7.627
7.619
7.611
7.603
7.594
7.585
7.564
7.342
7.333
7.089
7.079
7.067
6.200
6.160
4.236
4.218
4.200
4.182
1.305
1.287
1.269
CO2Et
O
S
3v
1H NMR, CDCl3
(400 MHz)
1.06
1.06
2.09
1.08
2.06
1.02
1.08
1.03
1.00
2.05
3.01
f1 (ppm)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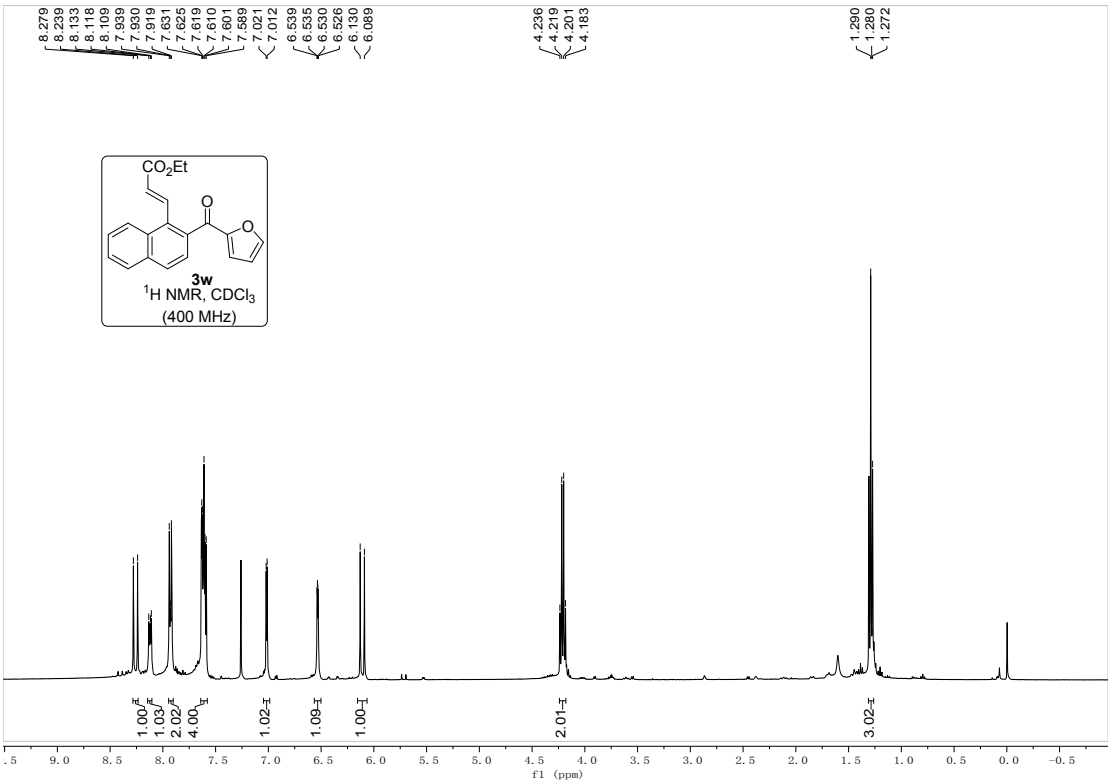
144.600
140.837
136.431
135.377
135.165
133.887
131.699
131.044
128.878
128.554
128.234
127.493
127.459
126.563
125.395
124.562
77.317
77.000
76.682
60.643
14.188
CO2Et
O
S
3v
13C NMR, CDCl3
(100 MHz)
f1 (ppm)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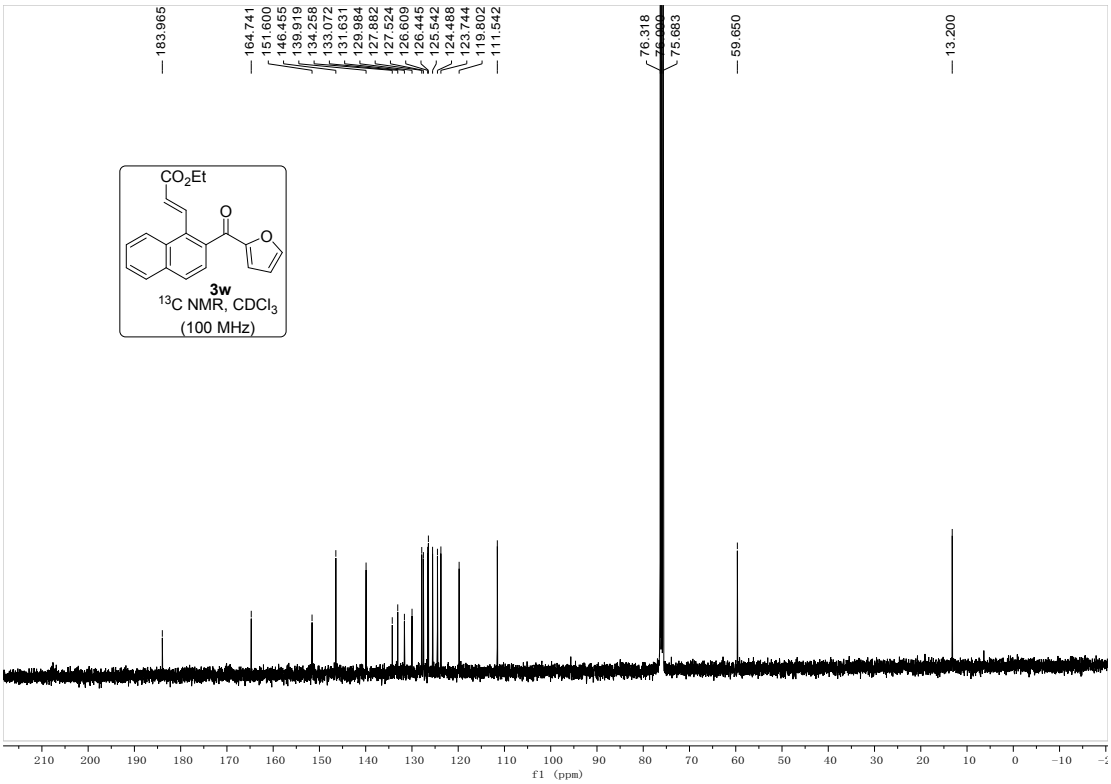
8.239
8.133
8.118
8.109
7.939
7.930
7.919
7.631
7.625
7.619
7.610
7.601
7.589
7.021
7.012
6.539
6.535
6.530
6.526
6.130
6.089
4.236
4.219
4.201
4.183
1.290
1.280
1.272
CO2Et
O
O
3w
1H NMR, CDCl3
(400 MHz)
1.00
1.03
2.02
4.00
1.02
1.09
1.00
2.01
3.02
f1 (ppm)


183.965
164.741
151.600
146.455
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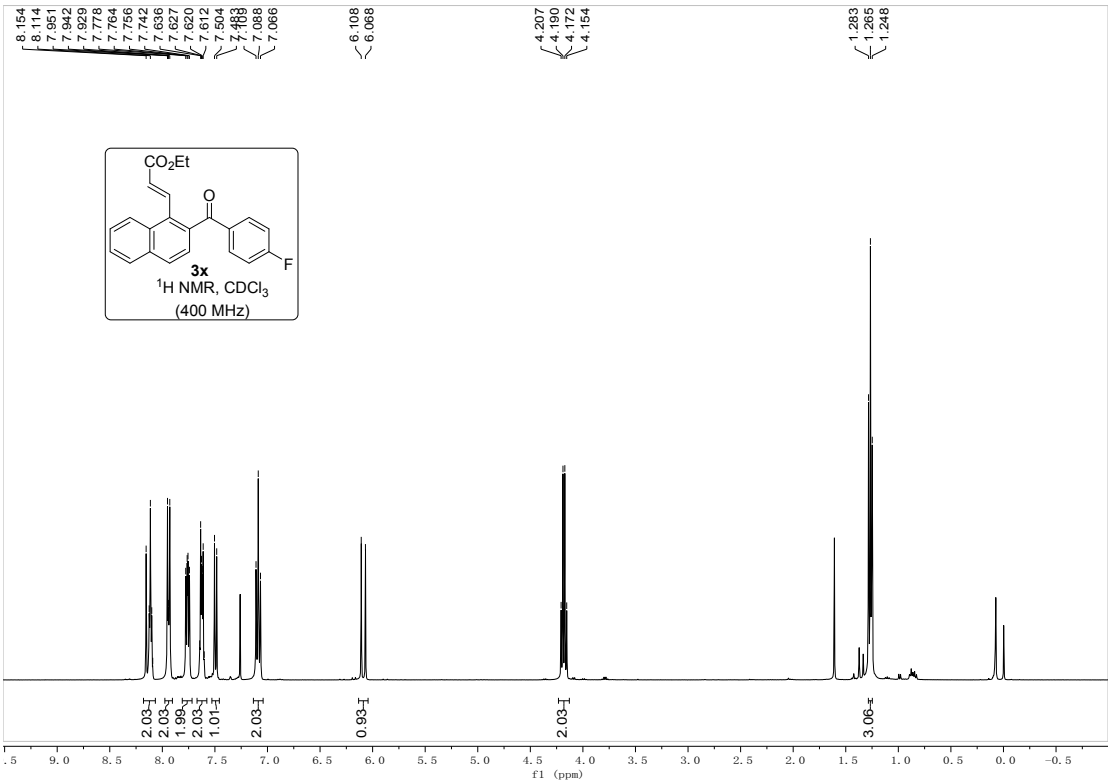
134.258
133.072
131.631
129.984
127.882
127.524
126.609
126.445
125.542
124.488
123.744
119.802
111.542
76.318
76.000
75.683
59.650
13.200
CO2Et
O
O
3w
13C NMR, CDCl3
(100 MHz)
f1 (ppm)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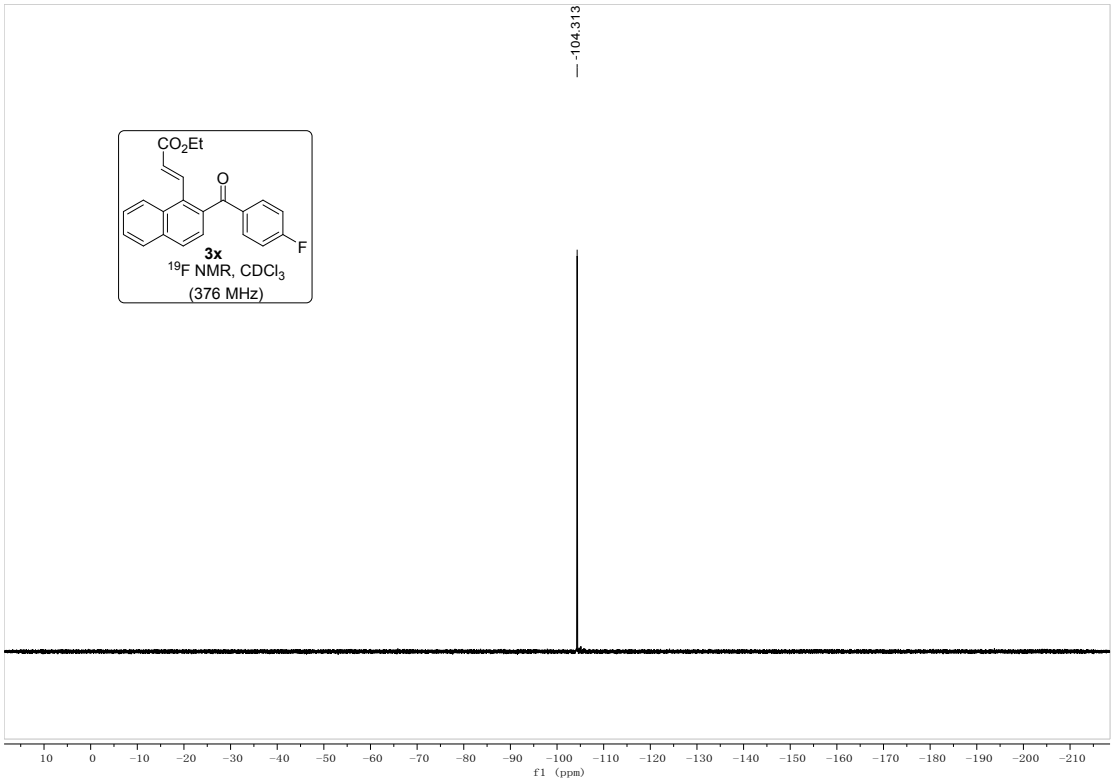
8.114
7.951
7.942
7.929
7.778
7.764
7.756
7.742
7.636
7.627
7.620
7.612
7.504
7.483
7.109
7.088
7.066
6.108
6.068
4.207
4.190
4.172
4.154
1.283
1.265
1.248
CO2Et
O
F
3x
1H NMR, CDCl3
(400 MHz)
2.03
2.03
1.99
2.03
1.01
2.03
0.93
2.03
3.06
f1 (ppm)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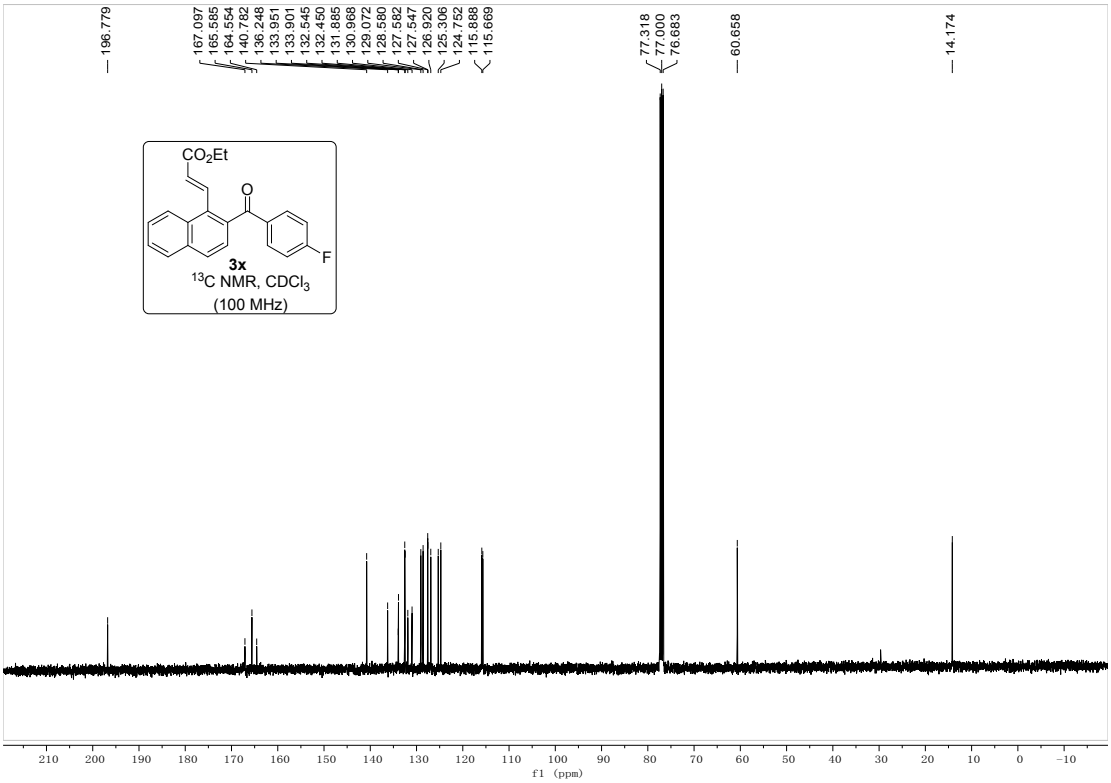
CO2Et
O
F
3x
19F NMR, CDCl3
(376 MHz)
f1 (ppm)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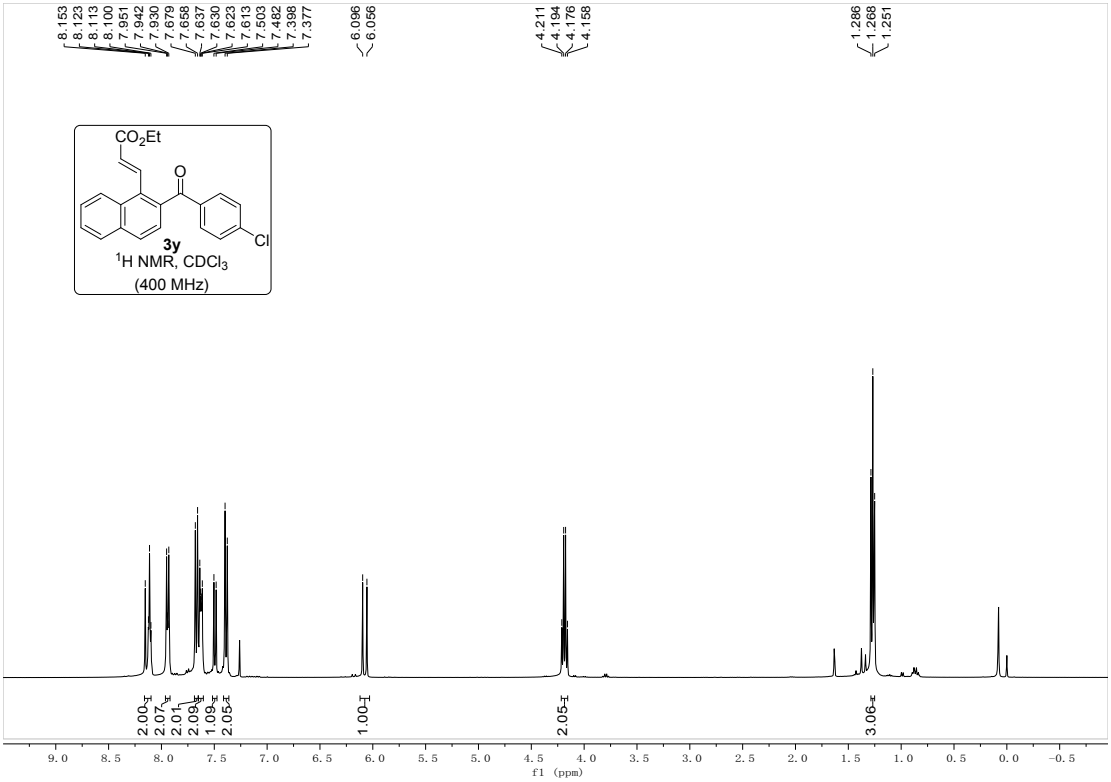
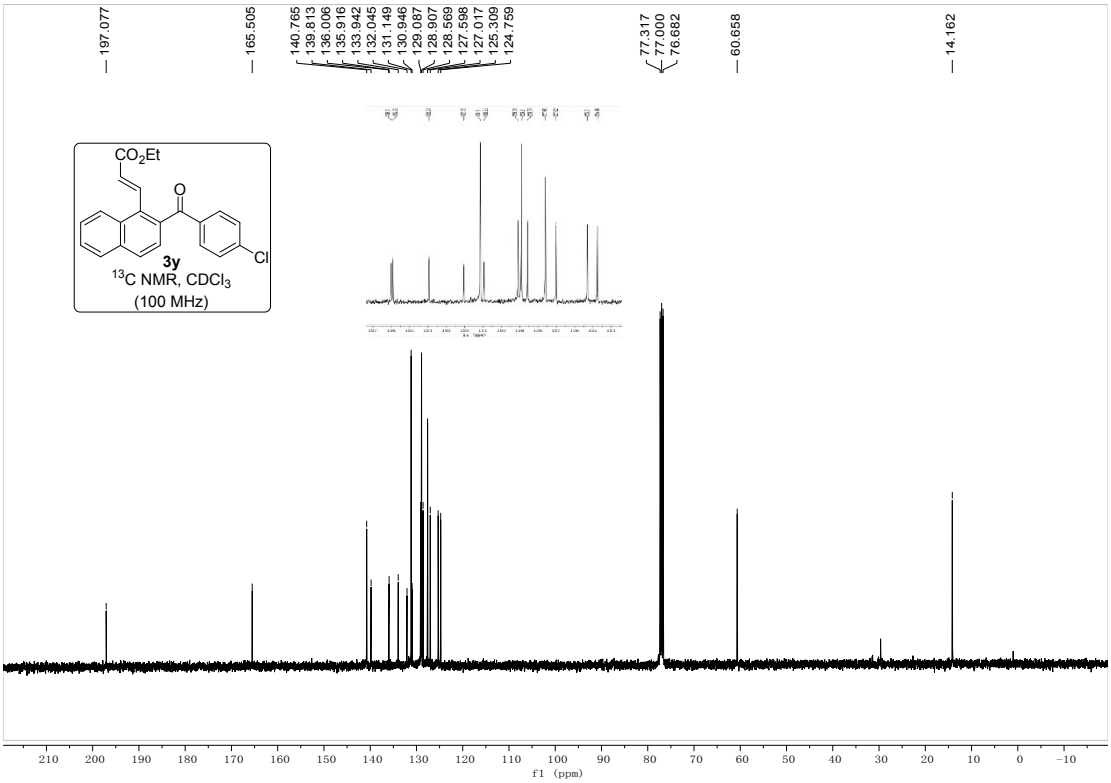
165.585
164.554
140.782
136.248
133.951
133.901
132.545
132.450
131.885
130.968
129.072
128.580
127.582
127.547
126.920
125.306
124.752
115.888
115.669
77.318
77.000
76.683
60.658
14.174
CO2Et
O
F
3x
13C NMR, CDCl3
(100 MHz)
f1 (ppm)


8.153
8.123
8.113
8.100
7.951
7.942
7.930
7.679
7.658
7.637
7.630
7.623
7.613
7.503
7.482
7.398
7.377
6.096
6.056
4.211
4.194
4.176
4.158
1.286
1.268
1.251
CO2Et
O
Cl
3y
1H NMR, CDCl3
(400 MHz)
2.00
2.07
2.01
2.09
1.09
2.05
1.00
2.05
3.06
f1 (ppm)


197.077
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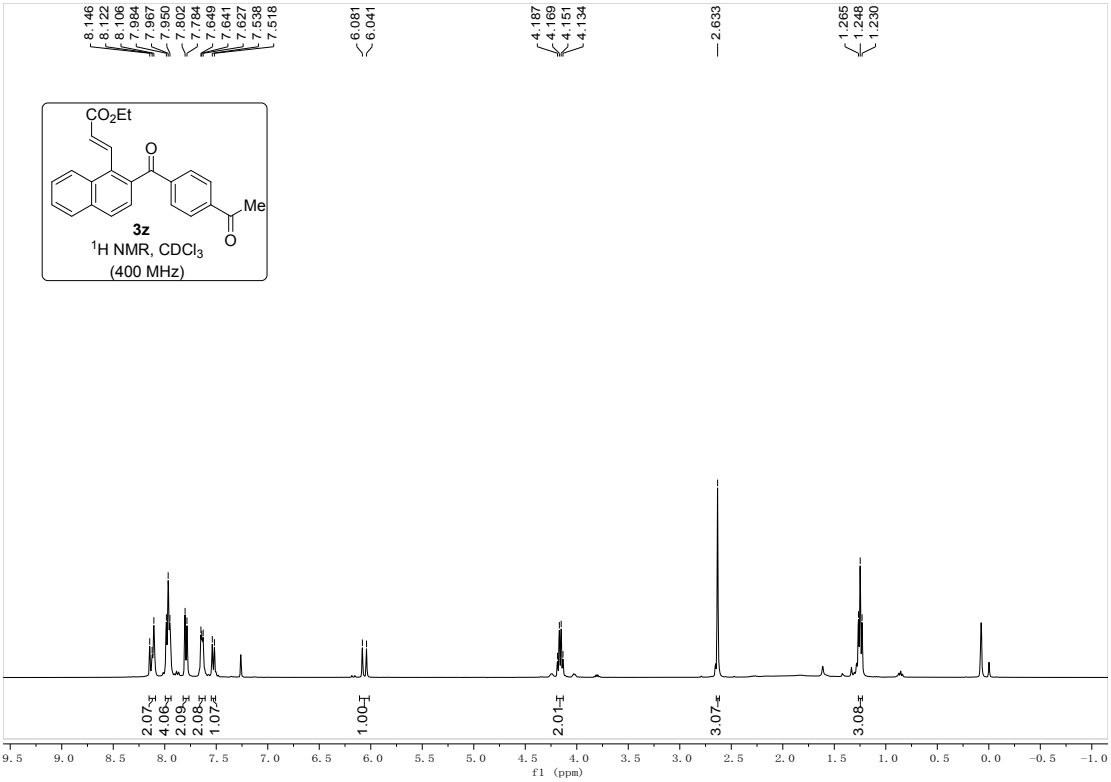
139.813
136.006
135.916
133.942
132.045
131.149
130.946
129.087
128.907
128.569
127.598
127.017
125.309
124.759
77.317
77.000
76.682
60.658
14.162
CO2Et
O
Cl
3y
13C NMR, CDCl3
(100 MHz)
f1 (ppm)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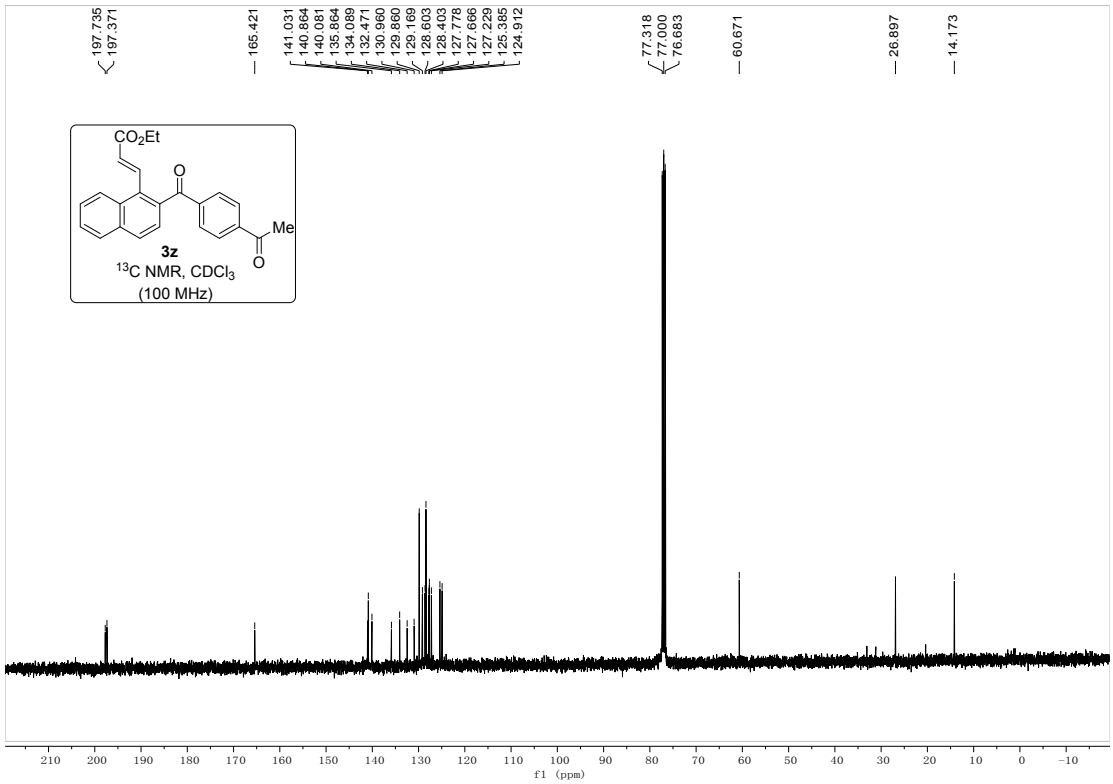
8.106
7.984
7.967
7.950
7.802
7.784
7.649
7.641
7.627
7.538
7.518
6.081
6.041
4.187
4.169
4.151
4.134
2.633
1.265
1.248
1.230
CO2Et
O
Me
O
3z
1H NMR, CDCl3
(400 MHz)
2.07
4.06
2.09
2.08
1.07
1.00
2.01
3.07
3.08
f1 (ppm)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140.864
140.081
135.864
134.089
132.471
130.960
129.860
129.169
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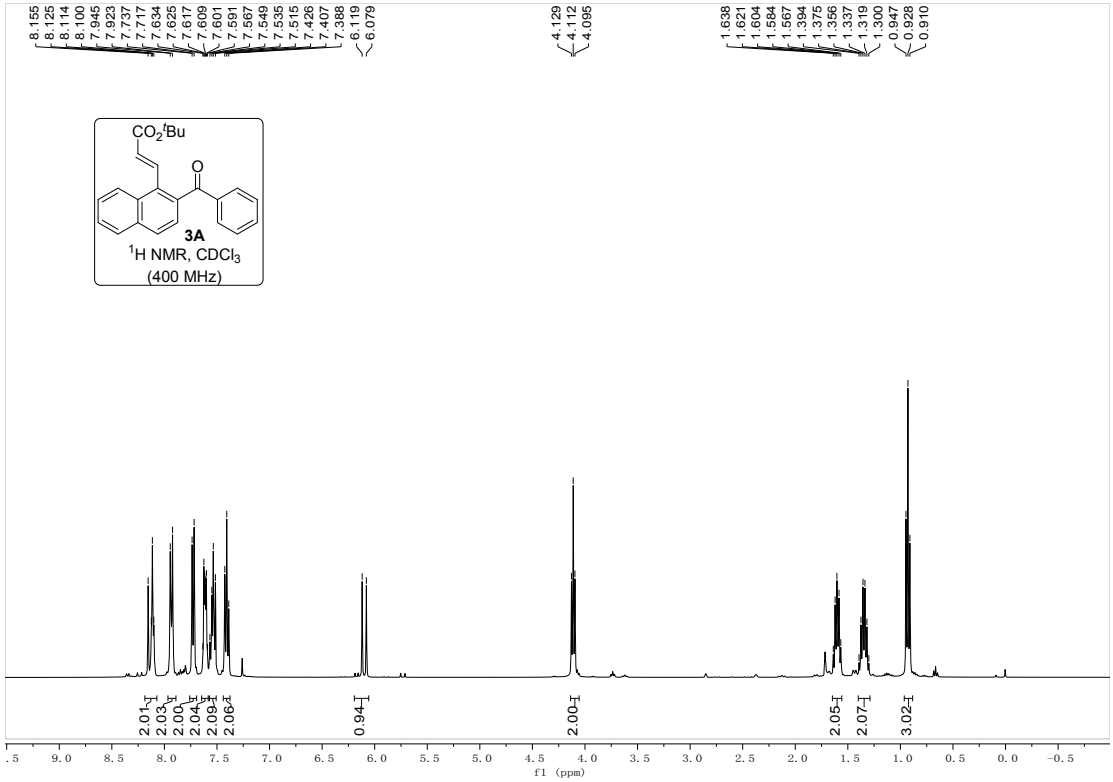
128.403
127.778
127.666
127.229
125.385
124.912
77.318
77.000
76.683
60.671
26.897
14.173
CO2Et
O
Me
O
3z
13C NMR, CDCl3
(100 MHz)
f1 (ppm)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7.717
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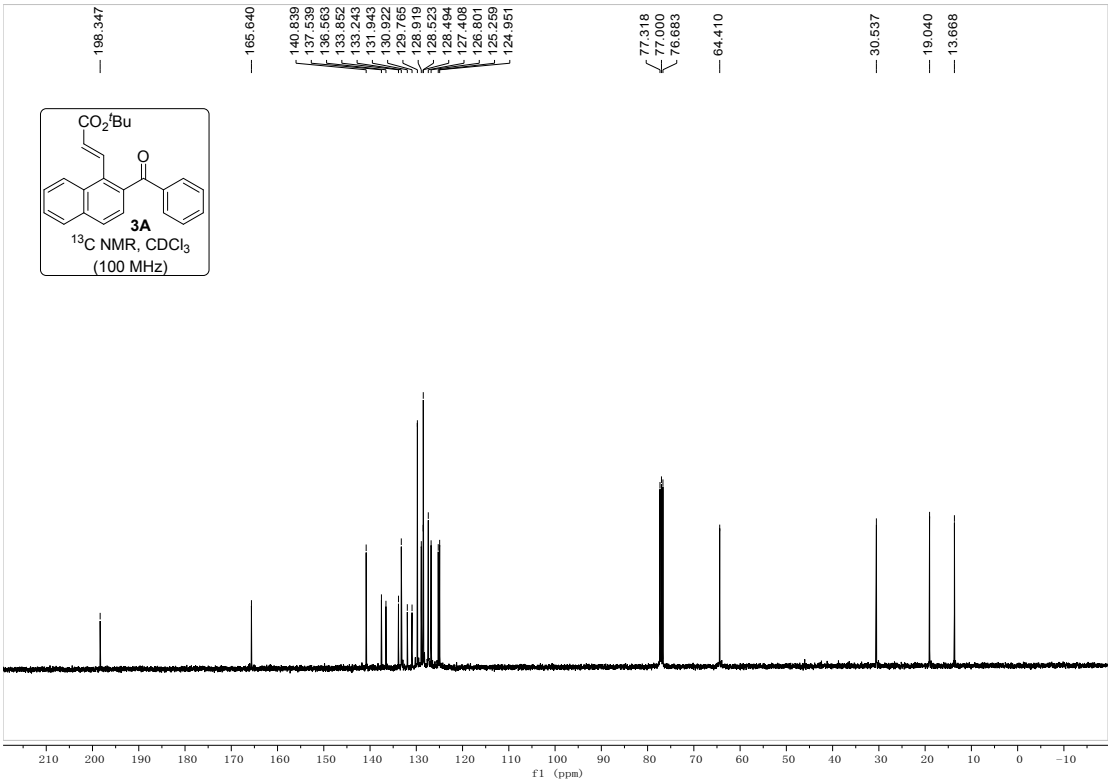
7.601
7.591
7.567
7.549
7.535
7.515
7.426
7.407
7.388
6.119
6.079
4.129
4.112
4.095
1.638
1.621
1.604
1.584
1.567
1.394
1.375
1.356
1.337
1.319
1.300
0.947
0.928
0.910
CO2tBu
O
3A
1H NMR, CDCl3
(400 MHz)
2.01
2.03
2.00
2.04
2.09
2.06
0.94
2.00
2.05
2.07
3.02
f1 (ppm)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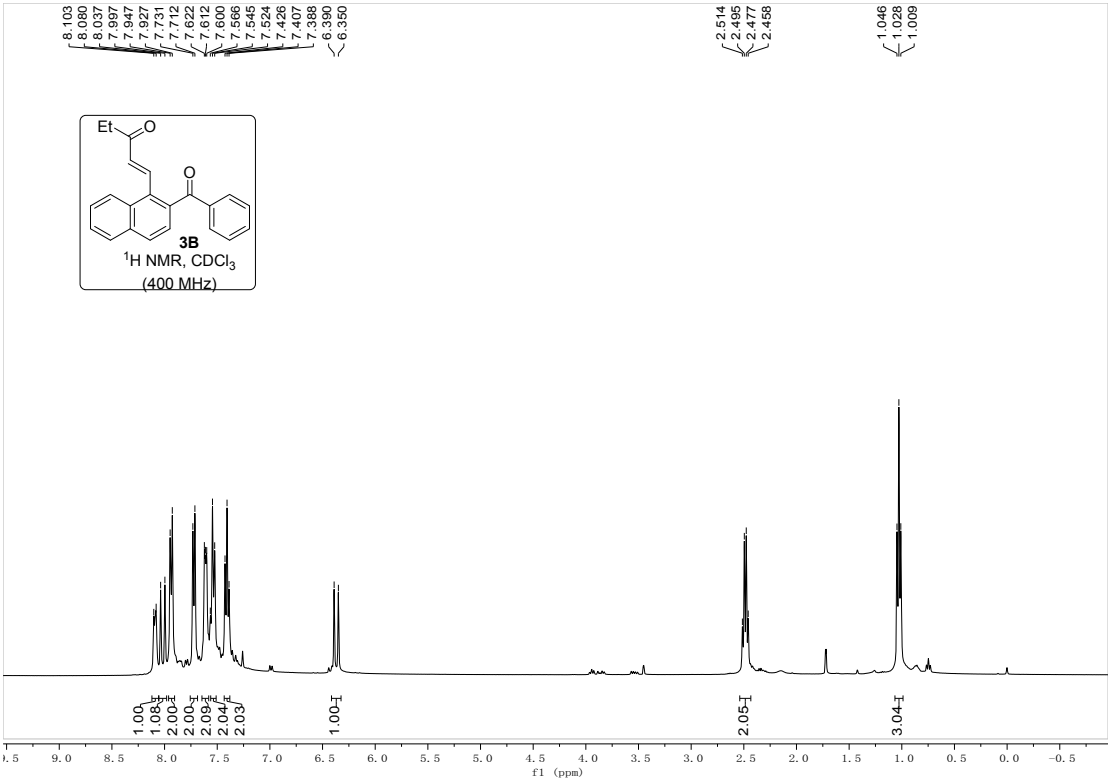
136.563
133.852
133.243
131.943
130.922
129.765
128.919
128.523
128.494
127.408
126.801
125.259
124.951
77.318
77.000
76.683
64.410
30.537
19.040
13.668
CO2tBu
O
3A
13C NMR, CDCl3
(100 MHz)
f1 (ppm)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8.080
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7.947
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7.731
7.712
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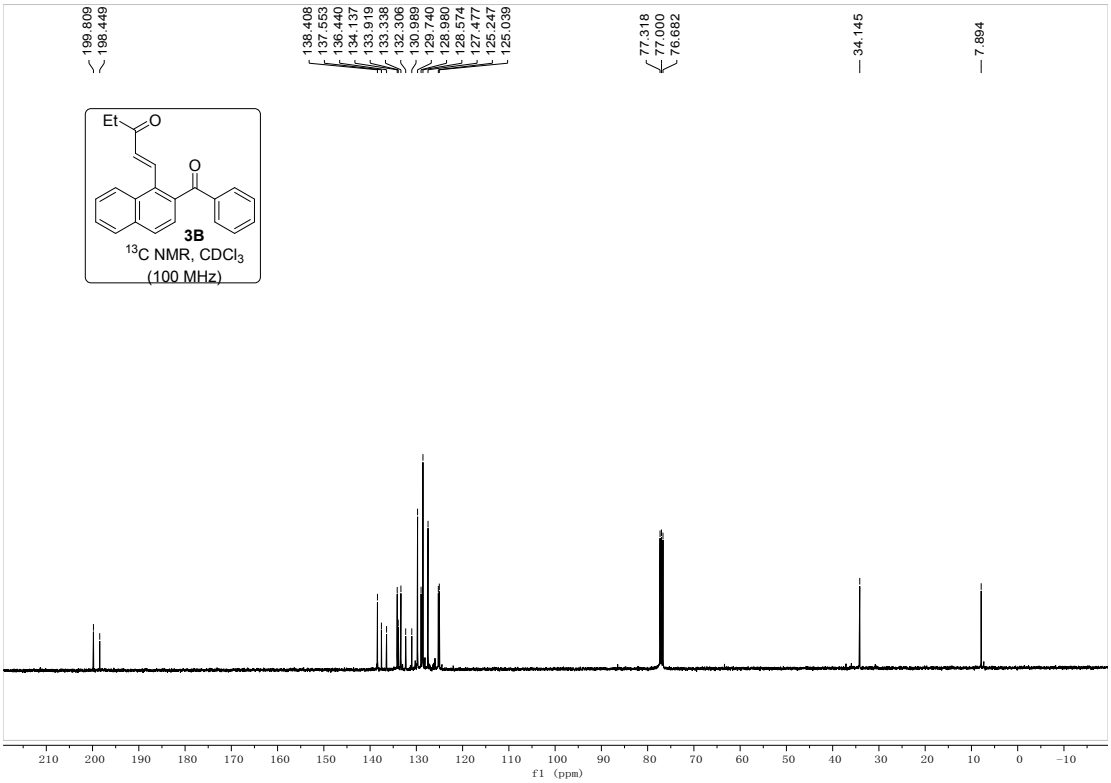
7.612
7.600
7.566
7.545
7.524
7.426
7.407
7.388
6.390
6.350
2.514
2.495
2.477
2.458
1.046
1.028
1.009
Et
O
O
3B
1H NMR, CDCl3
(400 MHz)
1.00
1.08
2.00
2.00
2.09
2.04
2.03
1.00
2.05
3.04
f1 (ppm)


199.809
198.449
138.408
137.553
136.440
134.137
133.919
133.338
132.306
130.989
129.740
128.980
128.574
127.477
125.247
125.039
77.318
77.000
76.682
34.145
7.894
Et
O
O
3B
13C NMR, CDCl3
(100 MHz)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

# 6. References


1. B. Yu, H. Sun, Z. Xie, G. Zhang, L. W. Xu, W. Zhang and Z. Gao, *Org. Lett.,* 2015, **17**, 3298-3301.
2. E. Kumarasamy, J. L. Jesuraj, J. N. Omlid, A. Ugrinov and J. Sivaguru, *J. Am. Chem. Soc.,* 2011, **133**, 17106-17109.
3. M. R. Yadav, M. Shankar, E. Ramesh, K. Ghosh and A. K. Sahoo, *Org. Lett.,* 2015*,* **17**, 1886-1889.