Supporting Information for

Dispersion-Induced Cooperative Hydrogen Atom Transfer for Radical Iodoalkylation

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1. General information

Unless otherwise noted, all commercially available compounds were used as provided without further purification. Solvents used in reactions were p.A. grade and dried only if indicated. Solvents for chromatography were technical grade and distilled prior to use. Analytical thin-layer chromatography (TLC) was performed on Merck silica gel aluminium plates with F-254 indicator, visualized by irradiation with UV light. Column chromatography was performed using silica gel Merck 60 (particle size 0.063–0.2 mm). Melting points were measured on a Yanaco Micro Melting Point Apparatus. ^{1}H NMR, ^{13}C NMR and ^{19}F NMR were recorded on a Variance VNMR 400 or Bruker AV-600 spectrometer in $CDCl_3$. For ^{1}H NMR spectra, data are quoted in the following order: chemical shift (δ) in parts per million (ppm) downfield of tetramethylsilane, using residual protonated solvent as internal standard ($CDCl_3$ at 7.26 ppm). Multiplicities are indicated s (singlet), d (doublet), t (triplet), m (multiplet), dd (doublet of doublets), coupling constants (*J*) are in Hertz (Hz). For proton-decoupled ^{13}C NMR spectra, chemical shifts (*J*) are also quoted in parts per million (ppm) downfield of tetramethylsilane, using deuterated solvent as internal standard ($CDCl_3$ at 77.0 ppm). High resolution mass spectra (HRMS) were obtained on AB 5800 MALDI-TOF/TOF and are recorded using electrospray ionization (ESI). X-ray crystallographic data were collected using D8 quest X-ray diffractometer. UV-Visible absorption spectroscopies were recorded on a fluorescence photometer (UV-1750, 190-1100nm).

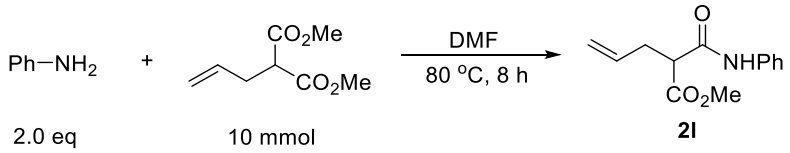
2. Preparation of starting materials

2.1) General procedure for the synthesis of indole-3-carboxaldehyde derivatives

2f-2r, **1x-1ad** were prepared according to the previous reported procedures. Citations to the references containing characterization data for these compounds: **2f**[1], **2g**[2], **2h**[3], **2i**[3], **2j**[4], **2k**[5], **2m**, **2n**[3],

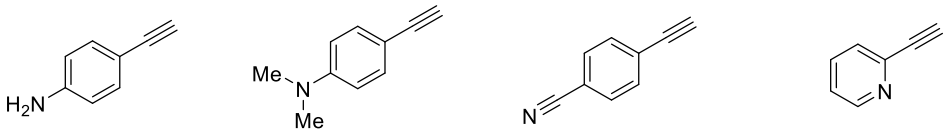
2q[4]**, 1x**[6]**, 1y**[6]**, 1z**[6]**, 1aa**[6]**, 1ab**[6]**, 1ac**[6]**, 1ad**[6] are consistent with the previous reports.**1a-1w** were purchased from energy-chemical, bidepharm and leyan chemical.

2.2) Preparation of 2l



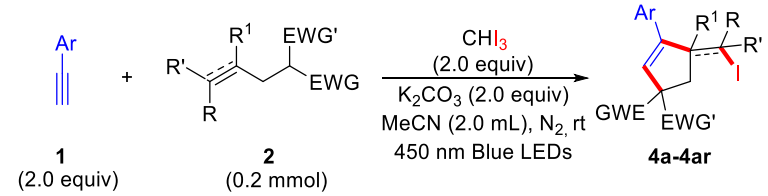
To a solution of aniline (2.0 equiv, 20 mmol, 1.86 g, 1.8 mL) in DMF (20.0 mL), dimethyl allylmalonate (10 mmol, 1.63 mL) was added dropwise at room temperature. The mixture was stirred at 80 ℃ for 8 h and then diluted with H_2O and extracted with ethyl acetate. Combined organic layers were dried over Na_2SO_4, concentrated under reduced pressure and purified by flash column chromatography on silica gel (ethyl acetate /petroleum ether = 1:15) to give the desired product **2l** as reddish brown soild (1.0 g, 43% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.75 (s, 1H), 7.53 (d, *J* = 8.0 Hz, 2H), 7.29 (t, *J* = 8.0 Hz, 2H), 7.10 (t, *J* = 8.0 Hz, 1H), 5.82-5.72 (m, 1H), 5.09 (dd, *J* = 28.0, 12.0 Hz, 2H), 3.74 (s, 3H), 3.48 (t, *J* = 8.0 Hz, 1H). 2.73 (t, *J* = 8.0 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.5, 166.0, 137.4, 133.6, 128.8, 124.5, 120.1, 117.9, 53.0, 52.5, 34.6. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{13}H_{16}NO_3^+$ 234.1125; found 234.1128.

3. Unsuccessful examples



The above terminal alkynes were tested under both of the radical iodoalkylative cyclization and iodoalkylation conditions, and very low reaction conversions were observed for those cases.

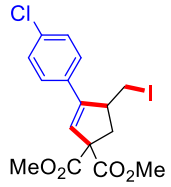
4. General procedure for the three-component radical iodoalkylative cyclization



Terminal alkynes **1** (2.0 equiv, 0.4 mmol), K_2CO_3 (2.0 equiv, 0.4 mmol, 55.3 mg), CHI_3 (2.0 equiv, 0.4 mmol, 156.0 mg) and a stir bar were added to a sealed tube under a nitrogen atmosphere, MeCN (2.0 mL) as solvent and **2** (0.2 mmol, 1.0 equiv) were added, respectively. The reaction mixture was then stirred under a 450 nm blue LEDs irradiation for 12 h at room temperature. The crude mixture was directly purified by flash column chromatography on silica gel (ethyl acetate/petroleum ether = 1:30 to 1:3) to give the desired products **4a-4ar**.

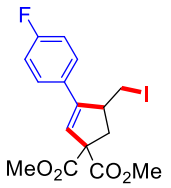
5. Characterization data of iodoalkylative cyclization products

Dimethyl 3-(4-chlorophenyl)-4-(iodomethyl) cyclopent-2-ene-1,1-dicarboxylate (4a)



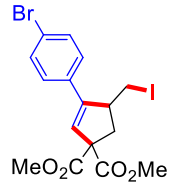
Yellow solid (69.0 mg, 79% yield); m.p. 106-108 °C; R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35-7.30 (m, 4H), 6.21 (d, *J* = 1.6 Hz, 1H), 3.79 (s, 3H), 3.74 (s, 3H), 3.64-3.58 (m, 1H), 3.36 (dd, *J* = 10.0, 2.8 Hz, 1H), 3.09 (t, *J* = 12.0 Hz, 1H), 2.92 (q, *J* = 8.0 Hz, 1H), 2.50 (dd, *J* = 12.0, 4.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 170.9, 147.7, 134.3, 132.3, 128.9, 127.9, 126.1 (d, *J* = 5.1 Hz, 1C), 64.8, 53.0 (d, *J* = 4.0 Hz, 1C), 47.3, 38.4, 10.9. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{16}H_{16}ClINaO_4^+$ 456.9674; found 456.9685.

Dimethyl 3-(4-fluorophenyl)-4-(iodomethyl) cyclopent-2-ene-1,1-dicarboxylate (4b)



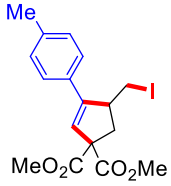
Yellow oil (70.6 mg, 84% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38-7.34 (m, 2H), 7.07-7.03 (m, 2H), 6.16 (d, *J* = 4.0 Hz, 1H), 3.79 (s, 3H), 3.74 (s, 3H), 3.65-3.58 (m, 1H), 3.37 (dd, *J* = 8.0, 4.0 Hz, 1H), 3.09 (t, *J* = 8.0 Hz, 1H), 2.92 (dd, *J* = 12.0, 8.0 Hz, 1H), 2.50 (dd, *J* = 16.0, 4.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.0, 162.7 (d, J_{C-F} = 249.5 Hz, 1C), 147.8, 129.9, 128.4 (d, J_{C-F} = 8.1 Hz, 1C), 125.3, 115.7 (d, *J* = 21.2 Hz, 1C), 64.8 53.0, 47.5, 38.5, 11.1. ^{19}F NMR (376 MHz, $CDCl_3$): -112.61. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{16}H_{16}FINaO_4^+$ 440.9970; found 440.9977.

Dimethyl 3-(4-bromophenyl)-4-(iodomethyl) cyclopent-2-ene-1,1-dicarboxylate (4c)



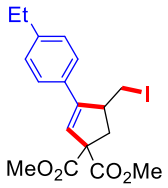
Yellow solid (83.9 mg, 88% yield); m.p. 78-80 °C; R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50-7.47 (m, 2H), 7.25 (dd, *J* = 1.2, 4.0 Hz, 2H), 6.22 (d, *J* = 1.6 Hz, 1H), 3.79 (s, 3H), 3.74 (s, 3H), 3.64-3.57 (m, 1H), 3.35 (q, *J* = 4.0 Hz, 1H), 3.09 (t, *J* = 8.0 Hz, 1H), 2.92 (q, *J* = 8.0 Hz, 1H), 2.49 (dd, *J* = 8.0, 16.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 170.9, 147.8, 132.7, 131.9, 128.2, 126.1 (d, *J* = 5.1 Hz, 1C), 122.5, 64.8, 53.0 (d, *J* = 4.0 Hz, 1C), 47.2, 38.4, 10.9. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{16}H_{16}BrINaO_4^+$ 500.9169; found 500.9168.

Dimethyl 4-(iodomethyl)-3-(p-tolyl) cyclopent-2-ene-1,1-dicarboxylate (4d)



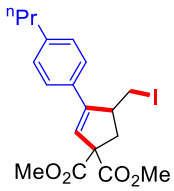
Yellow solid (75.3 mg, 91% yield); m.p. 74-76 °C; R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.28 (d, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 6.17 (d, *J* = 4.0 Hz, 1H), 3.79 (s, 3H), 3.73 (s, 3H), 3.68-3.62 (m, 1H), 3.41 (dd, *J* = 12.0, 4.0 Hz, 1H), 3.09 (t, *J* = 8.0 Hz, 1H), 2.91 (q, *J* = 8.0 Hz, 1H), 2.51 (dd, *J* = 16.0, 4.0 Hz, 1H), 2.35 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.2, 171.2, 148.7, 138.5, 130.9, 129.4, 126.51, 124.4 (d, *J* = 4.0 Hz, 1C), 64.7, 52.9, 47.5, 38.5, 21.2 (d, *J* = 2.0 Hz, 1C), 11.5. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{17}H_{19}INaO_4^+$ 437.0220; found 437.0215.

Dimethyl 3-(4-ethylphenyl)-4-(iodomethyl) cyclopent-2-ene-1,1-dicarboxylate (4e)



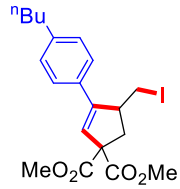
Yellow oil (71.2 mg, 83% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:20); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31 (d, *J* = 8.0 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 2H), 6.18 (d, *J* = 1.6 Hz, 1H), 3.79 (s, 3H), 3.73 (s, 3H), 3.70-3.63 (m, 1H), 3.43 (dd, *J* = 8.0, 2.8 Hz, 1H), 3.09 (t, *J* = 12.0 Hz, 1H), 2.92 (q, *J* = 8.0 Hz, 1H), 2.65 (q, *J* = 8.0 Hz, 2H), 2.50 (dd, *J* = 16.0, 4.0 Hz, 1H), 1.23 (t, *J* = 8.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): 171.2, 171.2, 148.7, 144.8, 131.1 128.2, 126.6, 124.4, 64.7, 52.9 (d, *J* = 4.0 Hz, 1C), 47.5, 38.5, 28.6, 15.4, 11.5. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{18}H_{21}INaO_4^+$ 451.0377; found 451.0369.

Dimethyl 4-(iodomethyl)-3-(4-propylphenyl) cyclopent-2-ene-1,1-dicarboxylate (4f)



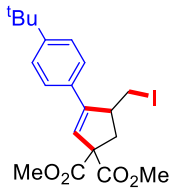
Yellow oil (63.0 mg, 71% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30 (d, *J* = 8.0 Hz, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 6.18 (d, *J* = 4.0 Hz, 1H), 3.79 (s, 3H), 3.73 (s, 3H), 3.70-3.63 (m, 1H), 3.43 (dd, *J* = 12.0, 4.0 Hz, 1H), 3.09 (t, *J* = 12.0 Hz, 1H), 2.91 (dd, *J* = 12.0, 8.0 Hz, 1H), 2.58 (t, *J* = 8.0 Hz, 2H), 2.51 (dd, *J* = 12.0, 4.0 Hz, 1H), 1.64 (dd, *J* = 12.0, 8.0 Hz, 2H), 0.94 (t, *J* = 8.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.2, 171.2 148.7, 143.3, 131.1, 128.8, 126.5, 124.4, 64.7, 52.9 (d, *J* = 4.0 Hz, 1C), 47.5, 38.5, 37.7, 24.4, 13.8 11.5. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{19}H_{23}INaO_4^+$ 465.0533; found 465.0524.

Dimethyl 3-(4-butylphenyl)-4-(iodomethyl) cyclopent-2-ene-1,1-dicarboxylate (4g)



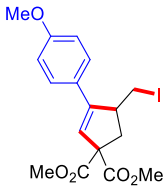
Yellow oil (76.5 mg, 84% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30 (d, *J* = 8.0 Hz, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 6.18 (d, *J* = 4.0 Hz, 1H), 3.79 (s, 3H), 3.73 (s, 3H), 3.70-3.62 (m, 1H), 3.43 (dd, *J* = 12.0, 4.0 Hz, 1H), 3.09 (t, *J* = 12.0 Hz, 1H), 2.91 (dd, *J* = 12.0, 4.0 Hz, 1H), 2.60 (t, *J* = 8.0 Hz, 2H), 2.51 (dd, *J* = 12.0, 4.0 Hz, 1H), 1.61-1.57 (m, 2H), 1.40-1.32 (m, 2H), 0.93 (t, *J* = 8.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.2 (d, *J* = 6.1 Hz, 1C), 148.7, 143.5, 131.0, 128.8, 126.5, 124.4, 64.7, 52.9 (d, *J* = 1.0 Hz, 1C), 47.5, 38.5, 35.4, 33.5, 22.3, 13.9, 11.5. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{20}H_{25}INaO_4^+$ 479.0690; found 479.0701.

Dimethyl 3-(4-(tert-butyl) phenyl)-4-(iodomethyl) cyclopent-2-ene-1,1-dicarboxylate (4h)



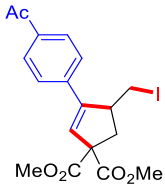
Yellow oil (83.5 mg, 92% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 (d, *J* = 4.0 Hz, 1H), 7.32 (d, *J* = 4.0 Hz, 1H), 6.19 (d, *J* = 4.0 Hz, 1H), 3.79 (s, 3H), 3.73 (s, 3H), 3.71-3.64 (m, 1H), 3.45 (dd, *J* = 8.0, 4.0 Hz, 1H), 3.08 (t, *J* = 8.0 Hz, 1H), 2.92 (dd, *J* = 12.0, 8.0 Hz, 1H), 2.50 (dd, *J* = 16.0, 4.0 Hz, 1H), 1.32 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.2, 171.2 151.7, 148.5, 130.8, 126.3, 125.6, 124.5, 64.7, 52.9 (d, *J* = 5.0 Hz, 1C), 47.6, 38.6, 34.6, 31.2, 11.5. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{20}H_{25}INaO_4^+$ 479.0690; found 479.0689.

Dimethyl 4-(iodomethyl)-3-(4-methoxyphenyl) cyclopent-2-ene-1,1-dicarboxylate (4i)



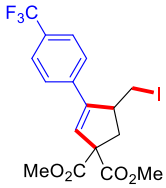
Yellow oil (58.6 mg, 68% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32 (dd, *J* = 4.0, 2.0 Hz, 2H), 6.88 (dd, *J* = 4.0, 2.0 Hz, 2H), 6.11 (d, *J* = 4.0 Hz, 1H), 3.81 (s, 3H), 3.79 (s, 3H), 3.73 (s, 3H), 3.66-3.60 (m, 1H), 3.41 (dd, *J* = 12.0, 4.0 Hz, 1H), 3.09 (t, *J* = 8.0 Hz, 1H), 2.89 (q, *J* = 8.0 Hz, 1H), 2.51 (dd, *J* = 16.0, 4.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.3, 171.3, 159.8, 148.2, 127.9, 126.2, 123.3, 114.1, 64.7, 55.3, 52.9 (d, *J* = 3.0 Hz, 1C), 47.6, 38.5, 11.5. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{17}H_{19}INaO_5^+$ 453.0169; found 453.0176.

Dimethyl 3-(4-acetylphenyl)-4-(iodomethyl) cyclopent-2-ene-1,1-dicarboxylate (4j)



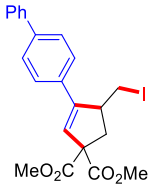
Yellow solid (61.5 mg, 70% yield); m.p. 130-132 °C; R_f = 0.25 (ethyl acetate/petroleum ether, 1:9); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94 (d, *J* = 8.0 Hz, 2H), 7.47 (d, *J* = 8.0 Hz, 2H), 6.33 (d, *J* = 1.2 Hz, 1H), 3.80 (s, 3H), 3.74 (s, 3H), 3.70-3.65 (m, 1H), 3.36 (dd, *J* = 12.0, 4.0 Hz, 1H), 3.11 (t, *J* = 12.0 Hz, 1H), 2.94 (q, *J* = 8.0 Hz, 1H), 2.60 (s, 3H), 2.50 (dd, *J* = 12.0, 4.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 197.3, 170.7, 147.9, 138.4, 136.7, 128.7, 127.8, 126.8, 64.9, 53.0, 47.1, 38.4, 26.6, 10.8. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{18}H_{19}INaO_5^+$ 465.0170; found 465.0162.

Dimethyl 4-(iodomethyl)-3-(4-(trifluoromethyl) phenyl) cyclopent-2-ene-1,1-dicarboxylate (4k)



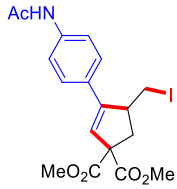
Yellow solid (79.6 mg, 85% yield); m.p. 78-80 °C; R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62 (d, *J* = 8.0 Hz, 2H), 7.49 (d, *J* = 8.0 Hz, 2H), 6.31 (d, *J* = 1.6 Hz, 1H), 3.81 (s, 3H), 3.75 (s, 3H), 3.69-3.62 (m, 1H), 3.36 (dd, *J* = 12.0, 4.0 Hz, 1H), 3.10 (t, *J* = 12.0 Hz, 1H), 2.96 (q, *J* = 8.0 Hz, 1H), 2.50 (dd, *J* = 16.0, 4.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 170.7, 147.7, 137.4 (d, J_{C-F} = 1.0 Hz, 1C), 130.3 (q, J_{C-F} = 33.3 Hz, 1C), 127.8 (d, J_{C-F} = 8.1 Hz, 1C), 127.0, 125.7 (d, J_{C-F} = 3.0 Hz, 1C), 122.6, 64.9, 53.1 (d, *J* =7.1 Hz, 1C), 47.2, 38.4, 10.7. ^{19}C NMR (376 MHz, $CDCl_3$): -62.7. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{17}H_{16}F_3INaO_4^+$ 490.9938; found 490.9943.

Dimethyl 3-([1,1'-biphenyl]-4-yl)-4-(iodomethyl) cyclopent-2-ene-1,1-dicarboxylate (4l)



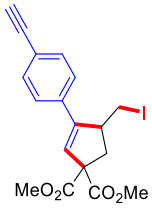
Yellow solid (87.3 mg, 92% yield); m.p. 128-130 °C; R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 (d, *J* = 8.0 Hz, 4H), 7.48-7.43 (m, 4H), 7.36 (t, *J* = 8.0 Hz, 1H), 6.28 (s, 1H), 3.81 (s, 3H), 3.75 (s, 3H), 3.71 (s, 1H), 3.46 (d, *J* = 12.0 Hz, 1H), 3.14 (t, *J* = 12.0 Hz, 1H), 2.95 (dd, *J* = 12.0, 8.0 Hz, 1H), 2.54 (dd, *J* = 12.0, 4.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.1, 171.1, 148.4, 141.3, 140.3, 132.7, 128.8, 127.6, 127.4, 127.1, 127.0, 125.3, 64.8, 53.0, 47.5, 38.5, 11.3. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{22}H_{21}INaO_4^+$ 499.0377; found 499.0367.

Dimethyl 3-(4-acetamidophenyl)-4-(iodomethyl) cyclopent-2-ene-1,1-dicarboxylate (4m)



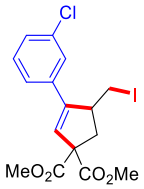
Yellow oil (41.1 mg, 45% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:2); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51 (d, *J* = 8.0 Hz, 1H), 7.43 (s, 1H), 7.33 (d, *J* = 8.0 Hz, 2H), 6.16 (s, 1H), 3.79 (s, 3H), 3.74 (s, 3H), 3.65-3.59 (m, 1H), 3.39 (dd, *J* = 8.0, 4.0 Hz, 1H), 3.09 (t, *J* = 8.0 Hz, 1H), 2.89 (dd, *J* = 12.0, 4.0 Hz, 1H), 2.50 (d, *J* = 16.0, 4.0 Hz, 1H), 2.18 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.3, 171.0, 149.0, 136.0, 134.1, 130.7, 128.4, 128.2, 127.9, 125.7, 64.9, 52.9, 49.1, 38.7, 20.4, 11.5. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{18}H_{20}INNaO_5^+$ 480.0278; found 480.027.

Dimethyl 3-(4-ethynylphenyl)-4-(iodomethyl) cyclopent-2-ene-1,1-dicarboxylate (4n)



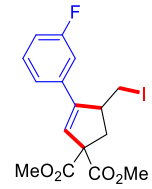
Yellow oil (43.1 mg, 51% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.48 (d, *J* = 8.0 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 6.25 (d, *J* = 4.0 Hz, 1H), 3.80 (s, 3H), 3.74 (s, 3H), 3.67-3.61 (m, 1H), 3.37 (dd, *J* = 8.0, 4.0 Hz, 1H), 3.14 (s, 1H), 3.09 (t, *J* = 12.0 Hz, 1H), 2.92 (dd, *J* = 12.0, 8.0 Hz, 1H), 2.51 (dd, *J* = 16.0, 4.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.9, 148.1, 134.2, 132.4, 126.5, 126.4, 122.1, 83.2, 78.4, 64.8, 53.0, 47.2, 38.4, 11.0. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{18}H_{17}INaO_4^+$ 447.0064; found 447.0075.

Dimethyl 3-(3-chlorophenyl)-4-(iodomethyl) cyclopent-2-ene-1,1-dicarboxylate (4o)



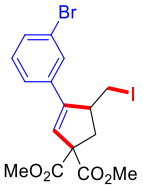
Yellow oil (45.5 mg, 84% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38 (s, 1H), 7.30-7.27 (m, 2H), 7.25-7.24 (m, 1H), 6.24 (d, *J* = 1.6 Hz, 1H), 3.80 (s, 3H), 3.74 (s, 3H), 3.64-3.58 (m, 1H), 3.37 (dd, *J* = 12.0, 4.0 Hz, 1H), 3.10 (t, *J* = 8.0 Hz, 1H), 2.92 (dd, *J* = 16.0, 8.0 Hz, 1H), 2.50 (dd, *J* = 12.0, 4.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 170.8, 147.6, 135.7, 134.7, 130.0, 128.5 (d, *J* = 3.0 Hz, 1C), 126.9, 126.8, 124.8, 64.8, 53.1 (d, *J* = 1.6 Hz, 1C), 47.2, 38.4, 10.9. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{16}H_{16}ClINaO_4^+$ 456.9674; found 456.9676.

Dimethyl 3-(3-fluorophenyl)-4-(iodomethyl) cyclopent-2-ene-1,1-dicarboxylate (4p)



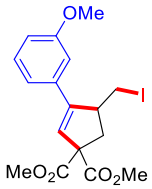
Yellow oil (57.0 mg, 68% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35-7.30 (m, 1H), 7.17-7.07 (m, 2H), 7.03-6.99 (m, 1H),6.24 (d, *J* = 4.0 Hz, 1H), 3.80 (s, 3H), 3.74 (s, 3H), 3.65-3.58 (m, 1H), 3.38 (dd, *J* = 12.0, 4.0 Hz, 1H), 3.11 (t, *J* = 12.0 Hz, 1H), 2.93 (dd, *J* = 12.0, 8.0 Hz, 1H), 2.51 (dd, *J* = 12.0, 4.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 170.8, 163.0 (d, $J_{C\text{-}F}$ = 248.5 Hz, 1C), 147.7 (d, $J_{C\text{-}F}$ = 3.0 Hz, 1C), 136.0 (d, $J_{C\text{-}F}$ = 7.1 Hz, 1C), 130.3 (d, $J_{C\text{-}F}$ = 8.1 Hz, 1C), 126.7, 122.3, 115.4 (d, $J_{C\text{-}F}$ = 22.2 Hz, 1C), 113.5 (d, $J_{C\text{-}F}$ = 12.2 Hz, 1C), 64.8, 53.0, 47.3, 38.4, 10.9. ^{19}F NMR (376 MHz, $CDCl_3$): -112.5. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{16}H_{16}FINaO_4^+$ 440.9970; found 440.9978.

Dimethyl 3-(3-bromophenyl)-4-(iodomethyl) cyclopent-2-ene-1,1-dicarboxylate (4q)



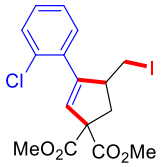
Yellow oil (63.4 mg, 66% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54 (t, *J* = 4.0 Hz, 1H), 7.45-7.43 (m, 1H), 7.31-7.28 (m, 1H), 7.23 (t, *J* = 8.0 Hz, 1H), 6.23 (d, *J* = 4.0 Hz, 1H), 3.80 (s, 3H), 3.74 (s, 3H), 3.64-3.57 (m, 1H), 3.36 (dd, *J* = 12.0, 4.0 Hz, 1H), 3.10 (t, *J* = 8.0 Hz, 1H), 2.92 (dd, *J* = 12.0, 8.0 Hz, 1H), 2.49 (dd, *J* = 16.0, 8.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 170.8, 170.8, 147.5, 136.0, 131.4, 130.3, 129.6, 126.9, 125.2, 122.9, 64.8, 53.0, 47.2 38.4, 10.9. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{16}H_{16}BrINaO_4^+$ 500.9169; found 500.9177.

Dimethyl 4-(iodomethyl)-3-(3-methoxyphenyl) cyclopent-2-ene-1,1-dicarboxylate (4r)



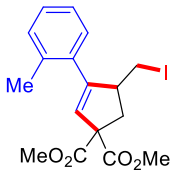
Yellow oil (58.1 mg, 68% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:15); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.28 (t, *J* = 8.0 Hz, 1H), 6.97 (d, *J* = 8.0 Hz, 1H), 6.92 (d, *J* = 4.0 Hz, 1H), 6.88-6.85 (m, 1H), 6.22 (d, *J* = 1.6 Hz, 1H), 3.83 (s, 3H), 3.80 (s, 3H), 3.74 (s, 3H), 3.68-3.61 (m, 1H), 3.42 (dd, *J* = 12.0, 4.0 Hz, 1H), 3.11 (t, *J* = 8.0 Hz, 1H), 2.93 (dd, *J* = 16.0, 8.0 Hz, 1H), 2.50 (dd, *J* = 16.0, 4.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.1, 171.0, 159.8, 148.7, 135.2, 129.7, 125.6, 119.0, 113.9, 112.4, 64.8, 55.3, 53.0, 47.5, 38.5, 11.3. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{17}H_{19}INaO_5^+$ 453.0169; found 453.0161.

Dimethyl 3-(2-chlorophenyl)-4-(iodomethyl) cyclopent-2-ene-1,1-dicarboxylate (4s)



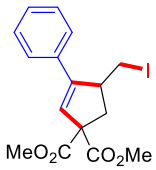
Yellow oil (73.7 mg, 85% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38-7.36 (m, 1H), 7.32-7.30 (m, 1H), 7.27-7.23 (m, 2H), 6.11 (d, *J* = 1.6 Hz, 1H), 3.81 (s, 4H), 3.75 (s, 3H), 3.23-3.20 (m, 1H), 3.04-2.95 (m, 2H), 2.35-2.30 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.0, 170.6, 147.9, 133.7, 132.3, 131.2, 130.1 (d, *J* = 5.1 Hz, 1H), 130.0, 129.4, 126.8, 64.7, 53.0 (t, *J* = 4.0 Hz, 1C), 47.9, 38.9, 11.4. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{16}H_{16}ClINaO_4^+$ 456.9674; found 456.9674.

Dimethyl 4-(iodomethyl)-3-(o-tolyl) cyclopent-2-ene-1,1-dicarboxylate (4t)



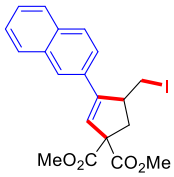
Yellow oil (79.8 mg, 96% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.21-7.13 (m, 4H), 5.95 (d, *J* = 4.0 Hz, 1H), 3.81 (s, 3H), 3.74 (s, 3H), 3.62-3.54 (m, 1H),3.20 (dd, *J* = 8.0, 4.0 Hz, 1H), 3.03 (d, *J* = 8.0 Hz, 1H), 2.98 (t, *J* = 8.0 Hz, 1H), 2.35 (s, 3H), 2.31 (t, *J* = 4.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.3, 170.9, 149.0, 135.9, 134.0, 130.7, 128.3, 128.2, 127.8, 125.6, 64.8, 52.9 (d, *J* = 2.0 Hz, 1C), 49.0, 38.7, 20.4, 11.5. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{17}H_{19}INaO_4^+$ 437.0220; found 437.0216.

Dimethyl 4-(iodomethyl)-3-phenylcyclopent-2-ene-1,1-dicarboxylate (4u)



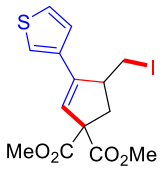
Yellow oil (64.7 mg, 81% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.40-7.29 (m, 5H), 6.22 (d, *J* = 4.0 Hz, 1H), 3.80 (s, 3H), 3.74 (s, 3H), 3.71-3.64 (m, 1H), 3.41 (dd, *J* = 12.0, 4.0 Hz, 1H), 3.10 (t, *J* = 8.0 Hz, 1H), 2.93 (q, *J* = 8.0 Hz, 1H), 2.50 (dd, *J* = 16.0, 4.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.1, 171.1, 148.8, 133.8, 128.7, 128.5, 126.6, 125.3 (d, *J* = 4.04 Hz, 1C), 64.8, 53.0, 47.4, 38.5, 11.3. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{16}H_{17}INaO_4^+$ 423.0064; found 423.0069.

Dimethyl 4-(iodomethyl)-3-(naphthalen-2-yl) cyclopent-2-ene-1,1-dicarboxylate (4v)



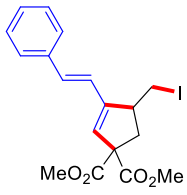
Yellow solid (52.9 mg, 59% yield); m.p. 103-105 °C; R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85-7.81 (m, 3H), 7.79 (s, 1H), 7.56 (dd, *J* = 8.0, 4.0 Hz, 1H), 7.52-7.47 (m, 2H), 6.36 (d, *J* = 4.0 Hz, 1H), 3.83 (s, 3H), 3.81-3.78 (m, 1H), 3.76 (s, 3H), 3.49 (dd, *J* = 12.0, 4.0 Hz, 1H), 3.17 (t, *J* = 12.0 Hz, 1H), 2.98 (dd, *J* = 12.0, 8.0 Hz, 1H), 2.58 (dd, *J* = 12.0, 4.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.1, 171.1, 148.8, 133.2, 133.2, 131.2, 128.5, 128.2, 127.7, 126.5, 126.5, 125.8, 125.6, 124.6, 64.9, 53.0 (q, *J* = 1.0 Hz, 1C), 47.5, 38.6, 11.4. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{20}H_{19}INaO_4^+$ 473.0220; found 473.0220.

Dimethyl 4-(iodomethyl)-3-(thiophen-2-yl) cyclopent-2-ene-1,1-dicarboxylate (4w)



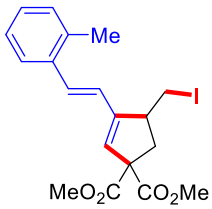
Yellow oil (49.5 mg, 61% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32 (dd, *J* = 4.8, 2.8 Hz, 1H), 7.24 (dd, *J* = 2.8, 1.2 Hz, 1H), 7.20 (dd, *J* = 5.2, 1.2 Hz, 1H), 6.16 (d, *J* = 1.2 Hz, 1H), 3.78 (s, 3H), 3.74 (s, 3H), 3.60-3.53 (m, 1H), 3.50 (dd, *J* = 12.0, 4.0 Hz, 1H), 3.14 (t, *J* = 8.0 Hz, 1H), 2.85 (q, *J* = 8.0 Hz, 1H), 2.56 (dd, *J* = 16.0, 4.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.1, 171.1, 143.6, 135.4, 126.4, 126.2, 124.3 (d, *J* = 4.04 Hz, 1C), 122.4, 64.8, 53.0, 48.7, 38.5, 11.1. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{14}H_{15}INaO_4S^+$ 428.9628; found 428.9619.

Dimethyl (E)-4-(iodomethyl)-3-styrylcyclopent-2-ene-1,1-dicarboxylate (4x)



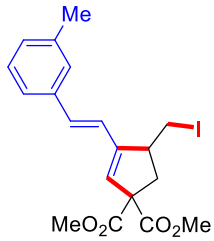
Yellow oil (34.4mg, 40% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42 (d, *J* = 8.0 Hz, 2H), 7.34 (t, *J* = 8.0 Hz, 2H), 7.29 (t, *J* = 8.0 Hz, 1H), 6.80 (d, *J* = 16.0 Hz, 1H), 6.61 (d, *J* = 16.0 Hz, 1H), 6.04 (s, 1H), 3.78 (s, 3H), 3.75 (s, 3H), 3.57-3.54 (m, 1H), 3.49 (t, *J* = 8.0 Hz, 1H), 3.18 (t, *J* = 8.0 Hz, 1H), 2.79 (q, *J* =8.0 Hz, 1H), 2.59 (dd, *J* = 16.0, 8.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.1, 171.1, 147.1, 136.3, 132.1, 128.7, 128.2, 128.1, 126.6, 122.3, 64.6, 53.0 (d, *J* = 6.1 Hz, 1C), 46.9, 38.6, 11.0. HRMS (ESI): $[M+Na]^+$ calcd for $C_{18}H_{19}INaO_4^+$ 449.0220; found 449.0224.

Dimethyl (E)-4-(iodomethyl)-3-(2-methylstyryl)cyclopent-2-ene-1,1-dicarboxylate (4y)



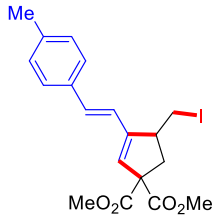
Yellow oil (63.4mg, 72% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46 (t, *J* = 4.0 Hz, 1H), 7.18 (q, *J* = 4.0 Hz, 3H), 6.85 (d, *J* = 16.0 Hz, 1H), 6.67 (d, *J* = 16.0 Hz, 1H), 6.04 (s, 1H), 3.78 (s, 3H), 3.76 (s, 3H), 3.56 (dd, *J* = 12.0, 4.0 Hz, 1H), 3.50 (t, *J* = 8.0 Hz, 1H), 3.21 (t, *J* = 8.0 Hz, 1H), 2.81 (dd, *J* = 12.0, 8.0 Hz, 1H), 2.58 (dd, *J* = 4.0, 12.0 Hz, 1H), 2.38 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.1, 147.5, 135.9, 135.5, 130.5, 130.1, 128.1, 127.9, 126.3, 125.3, 123.5, 77.4, 77.0, 76.7, 64.7, 53.0, 47.2, 38.6, 19.9, 10.8. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{19}H_{21}INaO_4^+$ 463.0377; found 463.0381.

Dimethyl (E)-4-(iodomethyl)-3-(2-methylstyryl)cyclopent-2-ene-1,1-dicarboxylate (4z)



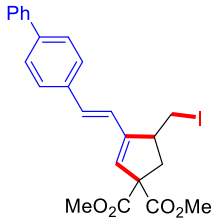
Yellow oil (45.8 mg, 52% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.23 (d, *J* = 8.0 Hz, 3H), 7.10-7.08 (m, 1H), 6.78 (d, *J* = 8.0 Hz, 1H), 6.58 (d, *J* = 20.0 Hz, 1H), 6.03 (s, 1H), 3.78 (s, 3H), 3.75 (s, 3H),3.56 (dd, *J* = 12.0, 4.0 Hz, 1H), 3.48 (t, *J* = 12.0 Hz, 1H), 3.18 (t, *J* = 8.0 Hz, 1H), 2.79 (dd, *J* = 12.0, 8.0 Hz, 1H), 2.59 (dd, *J* = 12.0, 4.0 Hz, 1H), 2.36 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.1, 147.2, 138.3, 136.3, 132.3, 129.1, 128.6, 127.9, 127.4, 123.7, 122.1, 64.6, 53.0, 47.0, 38.7, 21.4, 11.0. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{19}H_{21}INaO_4^+$ 463.0377; found 463.0365.

Dimethyl (E)-4-(iodomethyl)-3-(4-methylstyryl)cyclopent-2-ene-1,1-dicarboxylate (4aa)



Yellow oil (34.9mg, 43% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32 (d, *J* = 8.0 Hz, 2H), 7.15 (d, *J* = 8.0 Hz, 2H), 6.75 (d, *J* = 16.0 Hz, 1H), 6.58 (d, *J* = 16.0 Hz, 1H), 6.01 (s, 1H), 3.77 (s, 3H), 3.75(s, 3H), 3.55 (dd, *J* = 12.0, 4.0 Hz, 1H), 3.48 (t, *J* = 12.0 Hz, 1H), 3.17 (t, *J* = 12.0 Hz, 1H), 2.78 (dd, *J* = 12.0, 8.0 Hz, 1H), 2.59 (dd, *J* = 12.0, 4.0 Hz,, 1H), 2.35 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.1, 147.3, 138.3, 133.6, 132.1, 129.4, 127.5, 126.5, 121.3, 64.6, 53.0, 47.1, 38.7, 21.2, 11.0, 1.0. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{19}H_{21}INaO_4^+$ 463.0377; found 463.0385.

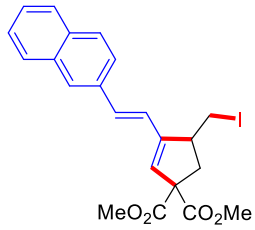
Dimethyl (E)-3-(2-([1,1'-biphenyl]-4-yl)vinyl)-4-(iodomethyl)cyclopent-2-ene-1,1-dicarboxylate (4ab)



Yellow solid (49.3mg, 49% yield); m.p. 95-97 °C; R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60 (t, *J* = 8.0 Hz, 4H), 7.50 (d, *J* = 8.0 Hz, 2H), 7.45 (t, *J* = 8.0 Hz, 2H), 7.38-7.33 (m, 1H), 6.85 (d, *J* = 20.0 Hz, 1H), 6.65 (d, *J* = 16.0 Hz, 1H), 6.07 (s, 1H), 3.79 (s, 3H), 3.76 (s, 3H), 3.58 (dd, *J* = 12.0, 4.0 Hz, 1H), 3.51 (t, *J* = 8.0 Hz, 1H), 3.20 (t, *J* = 8.0 Hz, 1H), 2.81 (q, *J* = 8.0 Hz, 1H), 2.61 (dd, *J* = 12.0, 4.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.1, 147.2, 141.0, 140.4, 135.4, 131.7, 128.8, 128.2, 127.5, 127.4, 127.1, 126.9, 122.3,

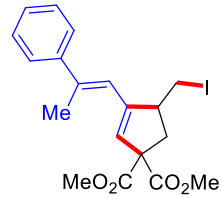
64.7, 53.0, 47.0, 38.7, 11.0. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{24}H_{23}INaO_4^+$ 525.0533; found 525.0535.

Dimethyl (E)-4-(iodomethyl)-3-(2-(naphthalen-2-yl)vinyl)cyclopent-2-ene-1,1-dicarboxylate (4ac)



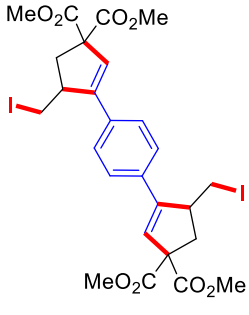
Yellow solid (53.6mg, 56% yield); m.p. 88-90 °C; R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83-7.79 (m, 4H), 7.62 (dd, *J* = 8.0 Hz, 1.6 Hz, 1H), 7.48-7.45 (m, 2H), 6.93 (d, *J* = 16.0 Hz, 1H), 6.78 (d, *J* = 16.0 Hz, 1H), 6.09 (s, 1H), 3.79 (s, 3H), 3.76 (s, 3H), 3.61 (dd, *J* = 12.0, 4.0 Hz, 1H), 3.58-3.51 (m, 1H), 3.22 (t, *J* = 8.0 Hz, 1H), 2.82 (q, *J* = 8.0 Hz, 1H), 2.62 (dd, *J* = 16.0, 4.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.1, 147.2, 133.9, 133.5, 133.3, 132.3, 128.4, 128.3, 128.1, 127.7, 127.2, 126.5, 126.3, 123.1, 122.6, 64.7, 53.0, 47.1, 38.7, 11.0. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{22}H_{21}INaO_4^+$ 499.0377; found 499.0367.

Dimethyl (E)-4-(iodomethyl)-3-(2-phenylprop-1-en-1-yl)cyclopent-2-ene-1,1-dicarboxylate (4ad)



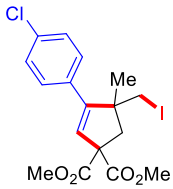
Yellow oil (44.3mg, 50% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44-7.41 (m, 2H), 7.37-7.33 (m, 2H), 7.30-7.28 (m, 1H), 6.08 (d, *J* = 4.0 Hz, 1H), 5.96 (s, 1H), 3.79 (s, 3H), 3.75 (s, 3H), 3.40 (dd, *J* = 12.0, 4.0 Hz, 1H), 3.24-3.21 (m, 1H), 3.17 (t, *J* = 8.0 Hz, 1H),2.84 (dd, *J* = 16.0, 8.0 Hz, 1H), 2.30 (t, *J* = 8.0 Hz, 1H), 2.26 (d, *J* = 4.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.4, 171.2, 146.1, 143.2, 142.0, 128.4, 127.6, 127.3, 126.0, 120.0 (d, *J* = 6.1 Hz, 1C), 65.0, 53.0, 49.6, 38.2, 18.4, 11.9. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{19}H_{22}IO_4^+$ 441.0557; found 441.0549.

Tetramethyl 3,3'-(1,4-phenylene)bis(4-(iodomethyl)cyclopent-2-ene-1,1-dicarboxylate) (4ae)



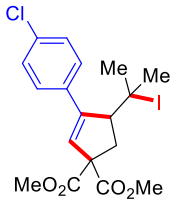
Yellow solid (78.3 mg, 54% yield); m.p. 64-66 °C; R_f = 0.25 (ethyl acetate/petroleum ether, 1:6); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39 (s, 4H), 6.25 (d, *J* = 1.2 Hz, 2H), 3.80 (s, 6H), 3.74 (s, 6H), 3.68-3.61 (m, 2H), 3.42-3.37 (m, 2H), 3.13-3.07 (m, 2H), 2.93 (dd, *J* = 12.0, 8.0 Hz, 2H).2.51 (dd, *J* = 12.0, 4.0 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.0 (d, *J* = 2.0 Hz, 1C), 148.1 (d, *J* = 4.0 Hz, 1C), 133.9 (d, *J* = 2.0 Hz, 1C), 127.0 (d, *J* = 1.0 Hz, 1C), 125.9, 64.8, 53.0 (d, *J* = 1.0 Hz, 1C), 47.3, 38.4, 11.1 (d, *J* = 2.0 Hz, 1C). HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{26}H_{28}I2NaO_8^+$ 744.9766; found 744.9764.

Dimethyl 3-(4-chlorophenyl)-4-(iodomethyl)-4-methylcyclopent-2-ene-1,1-dicarboxylate (4af)



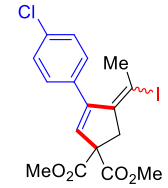
Yellow solid (60.8 mg, 68% yield); m.p. 83-85 °C; R_f = 0.25 (ethyl acetate/petroleum ether, 1:15); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32-7.29 (m, 2H), 7.27 (s, 1H), 7.25 (dd, *J* = 2.0, 1.6 Hz, 1H), 5.91 (s, 1H), 3.79 (s, 3H), 3.77 (s, 3H), 3.28 (dd, *J* = 16.0, 8.0 Hz, 2H), 2.77 (d, *J* = 12.0 Hz, 1H), 2.52 (d, *J* = 16.0 Hz, 1H), 1.42 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.1, 171.0, 151.0, 134.1, 133.4, 129.4, 128.5, 127.5, 63.3, 53.1, 53.0, 51.4, 46.2, 26.8, 20.0. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{17}H_{19}ClIO_4^+$ 449.0011; found 449.0011.

Dimethyl 3-(4-chlorophenyl)-4-(2-iodopropan-2-yl)cyclopent-2-ene-1,1-dicarboxylate (4ag)



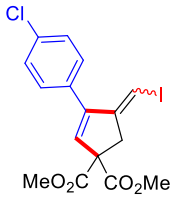
Yellow oil (48.2 mg, 52% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.29 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 12.0 Hz, 2H), 5.91 (s, 1H), 3.76 (s, 6H), 3.73 (d, *J* = 4.0 Hz, 1H), 3.26 (s, 2H), 1.76 (s, 3H), 1.31 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 171.2, 148.3, 133.6, 131.7, 129.5, 128.1, 127.1, 62.8, 52.9, 39.2, 23.5, 21.7. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{18}H_{20}ClINaO_4^+$ 484.9987; found 484.9977.

Dimethyl 3-(4-chlorophenyl)-4-(1-iodoethylidene)cyclopent-2-ene-1,1-dicarboxylate (4ah)



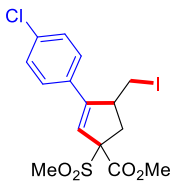
Yellow oil (61.0 mg, 66% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30 (d, *J* = 8.0 Hz, 2H), 7.15 (d, *J* = 8.0 Hz, 2H), 6.22 (s, 1H), 3.77 (s, 6H), 3.41 (s, 2H), 2.62 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 170.2, 148.8, 142.1, 137.0, 134.0, 133.4, 131.2, 128.0, 85.1, 62.4, 53.2 (d, *J* = 3.0 Hz, 1C), 39.9, 34.0. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{27}H_{28}INNaO_4S_2^+$ 468.9674; found 468.9665.

Diethyl 3-(4-chlorophenyl)-4-(iodomethylene)cyclopent-2-ene-1,1-dicarboxylate (4ai)



Yellow oil (52.8 mg, 61% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36 (d, *J* = 8.0 Hz, 2H), 7.27 (d, *J* = 8.0 Hz, 1H), 6.39 (s, 1H), 6.24 (t, *J* = 4.0 Hz, 1H), 3.80 (s, 6H), 3.32 (d, *J* = 4.0 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 170.1, 152.6, 146.1, 134.3, 131.7, 129.6, 129.1, 128.8, 128.4, 73.4, 62.6, 53.3, 43.40. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{16}H_{14}ClINaO_4^+$ 454.9518; found 454.9525.

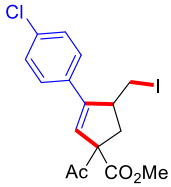
Methyl 3-(4-chlorophenyl)-4-(iodomethyl)-1-(methylsulfonyl)cyclopent-2-ene-1-carboxylate (4aj)



Yellow solid (65.4 mg, 72% yield); m.p. 108-110 °C; R_f = 0.25 (ethyl acetate/petroleum ether, 1:3); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36 (d, *J* = 8.0 Hz, 2H), 7.31 (dd, *J* = 8.0, 4.0 Hz, 2H), 6.16 (d, *J* = 4.0 Hz, 1H), 3.90 (s, 3H), 3.69-3.63 (m, 1H), 3.39-3.36 (m, 1H), 3.15 (dd, *J* = 8.0, 4.0 Hz, 1H), 3.05 (q, *J* = 8.0 Hz, 4H), 2.62 (q, *J* = 8.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 167.9, 154.1, 135.1, 131.8, 129.1,

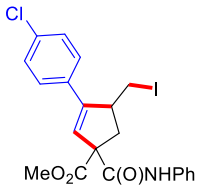
128.1, 121.2, 82.9, 53.8, 46.2, 37.5, 35.2, 11.9. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{15}H_{16}ClINaO_4S^+$ 476.9395; found 476.9392.

Methyl 1-acetyl-3-(4-chlorophenyl)-4-(iodomethyl)cyclopent-2-ene-1-carboxylate (4ak)



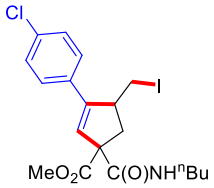
Yellow oil (45.1 mg, 54% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:6); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35-7.29 (m, 4H), 6.24 (dd, *J* = 8.0, 1.6 Hz, 1H), 3.77 (d, *J* = 24.0 Hz, 3H), 3.61-3.52 (m, 1H), 3.37-3.32 (m, 1H), 3.11-3.03 (m, 1H), 2.94-2.78 (m, 1H), 2.48-2.33 (m, 1H), 2.26 (d, *J* = 40.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 202.6 (d, *J* = 9.1 Hz, 1C), 171.4 (d, *J* = 8.1 Hz,), 148.1 (d, *J* = 7.1 Hz, 1C), 134.3, 132.3 (d, *J* = 8.0 Hz, 1C), 129.0, 127.9, 126.1 (d, *J* = 24.2 Hz, 1C), 71.7 (d, *J* = 8.1 Hz, 1C), 53.0, 47.1 (d, *J* = 27.3 Hz, 1C), 36.9 (d, *J* = 5.0 Hz, 1C), 26.8 (d, *J* = 68.7 Hz, 1C), 11.3 (d, *J* = 15.2 Hz, 1C). HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{16}H_{16}ClINaO_3^+$ 440.9725; found 440.9719.

Methyl 3-(4-chlorophenyl)-4-(iodomethyl)-1-(phenylcarbamoyl)cyclopent-2-ene-1-carboxylate (4al)



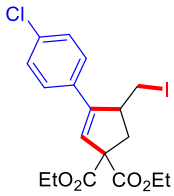
Yellow oil (68.7 mg, 69% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:6); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.07 (d, *J* = 16.0 Hz, 1H), 7.57-7.49 (m, 2H), 7.36-7.29 (m, 6H), 7.16-7.09 (m, 1H), 6.30 (dd, *J* = 16.0, 1.6 Hz, 1H), 3.82 (d, *J* = 28.0 Hz, 3H), 3.66-3.55 (m, 1H), 3.41-3.36 (m, 1H), 3.33-3.17 (m, 1H), 3.14-2.95 (m, 1H), 2.75-2.47 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 172.3 (d, *J* = 47.0 Hz, 1C), 167.5 (d, *J* = 8.0 Hz, 1C), 149.5, 148.5, 137.2 (d, *J* = 6.1 Hz, 1C), 134.5 (d, *J* = 9.1 Hz, 1C), 132.1, 128.9 (t, *J* = 2.0 Hz, 1C), 127.9 (d, *J* = 4.0 Hz, 1C), 126.2 (d, *J* = 4.0 Hz, 1C), 124.7 (d, *J* = 2.0 Hz, 1C), 119.9 (d, *J* = 11.1 Hz, 1C), 66.7 (d, *J* = 6.3 Hz, 1C), 53.3 (d, *J* = 11.1 Hz, 1C), 46.7 (d, *J* = 26.3 Hz, 1C), 38.3 (d, *J* = 11.1 Hz, 1C), 11.4. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{21}H_{20}ClINO_3^+$ 496.0171; found 496.0165.

Methyl 1-(tert-butylcarbamoyl)-3-(4-chlorophenyl)-4-(iodomethyl)cyclopent-2-ene-1-carboxylate (4am)



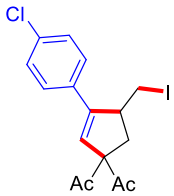
Yellow oil (39.2 mg, 41% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:6); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35-7.26 (m, 4H), 6.19 (dd, *J* = 20.0, 4.0 Hz, 1H),6.14-6.04 (m, 1H) 3.77 (d, *J* = 24.0 Hz, 3H), 3.59-3.49 (m, 1H), 3.38-3.33 (m, 1H), 3.31-3.22 (m, 2H), 3.21-2.80 (m, 2H), 2.68-2.32 (m, 1H), 1.56-1.42 (m, 2H), 1.39-1.29 (m, 2H), 0.95-0.88 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 172.3 (d, *J* = 33.3Hz, 1C), 169.7 (d, *J* = 49.5 Hz, 1C), 148.2 (d, *J* = 80.8 Hz, 1C), 134.3, 132.3, 129.0, 127.9 (d, *J* = 8.1 Hz, 1C), 126.6, 65.8, 53.1, 47.0, 39.8, 38.4, 31.4, 19.9, 13.7, 11.4. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{19}H_{23}ClINNaO_3^+$ 498.0303; found 498.0308.

Diethyl 3-(4-chlorophenyl)-4-(iodomethyl)cyclopent-2-ene-1,1-dicarboxylate (4an)



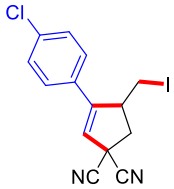
Yellow solid (49.9 mg, 54% yield); m.p. 95-97 °C; R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32 (s, 4H), 6.22 (d, *J* = 1.6 Hz, 1H), 4.29-4.15 (m, 4H), 3.64-3.58 (m, 1H), 3.36 (dd, *J* = 12.0, 8.0 Hz, 1H), 3.09 (t, *J* = 8.0 Hz, 1H), 2.89 (q, *J* = 8.0 Hz, 1H), 2.51 (dd, *J* = 16.0, 4.0 Hz, 1H), 1.30 (t, *J* = 8.0 Hz, 3H), 1.25 (t, *J* = 8.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 170.5, 147.5, 134.3, 132.4, 128.9, 127.9, 126.3, 65.0, 61.9, 47.4, 38.2, 14.1, (d, *J* = 6.1 Hz, 1C), 11.0. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{18}H_{20}ClINNaO_4^+$ 484.9987 found 484.9988.

1,1'-(3-(4-Chlorophenyl)-4-(iodomethyl)cyclopent-2-ene-1,1-diyl)bis(ethan-1-one) (4ao)



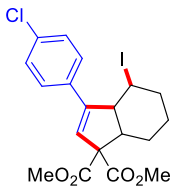
Yellow oil (27.5 mg, 35% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:9); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35 (d, *J* = 8.0 Hz, 2H), 7.30 (d, *J* =8.0 Hz, 2H), 6.36 (d, *J* = 1.6 Hz, 1H), 3.51-3.45 (m, 1H), 3.34 (dd, *J* = 12.0, 4.0 Hz 1H), 3.06 (dd, *J* = 10.1, 8.0 Hz, 1H), 2.86 (dd, *J* = 12.0, 8.0 Hz, 1H), 2.37 (dd, *J* = 12.0, 8.0 Hz), 2.27 (s, 3H), 2.16 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$): δ 204.7, 203.8, 148.5, 134.4, 132.4, 129.0, 127.8, 126.2, 79.6, 46.5, 35.8, 27.5, 26.8, 11.7. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{16}H_{16}ClINNaO_2^+$ 424.9776; not found (Confirmed by GC-MS).

3-(4-Chlorophenyl)-4-(iodomethyl)cyclopent-2-ene-1,1-dicarbonitrile (4ap)



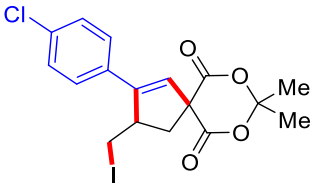
Yellow oil (21.3 mg, 45% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:9); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 6.02 (d, *J* = 2.4 Hz, 1H), 3.79-3.73 (m, 1H), 3.34 (q, *J* = 4.0 Hz, 1H), 3.21-3.08 (m, 2H), 2.81 (dd, *J* = 16.0, 4.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 152.8, 136.2, 130.0, 129.5, 128.2, 119.5, 114.8(d, *J* = 15.2, 1C), 47.0, 43.1, 37.4, 8.0. HRMS (ESI) m/z: calcd 367.9577; not found (Confirmed by GC-MS).

Dimethyl 3-(4-chlorophenyl)-4-iodo-3a,4,5,6,7,7a-hexahydro-1H-indene-1,1-dicarboxylate (4aq)



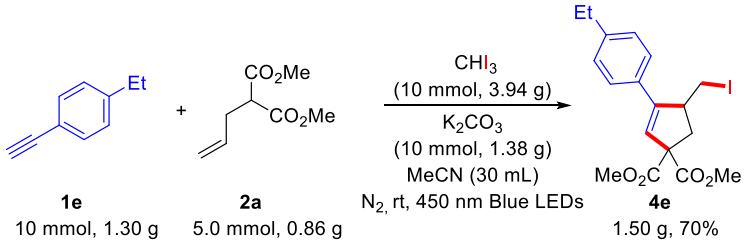
Yellow oil (39.4mg, 42% yield); R_f = 0.25 (ethyl acetate/petroleum ether, 1:30); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36-7.34 (m, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 5.97 (d, *J* = 4.0 Hz, 1H), 4.93 (d, *J* = 4.0 Hz, 1H), 3.99 (t, *J* = 4.0 Hz, 1H), 3.77 (s, 3H), 3.68 (s, 3H), 3.57-3.52 (m, 1H), 1.88-1.78 (m, 2H), 1.57-1.42 (m, 3H), 1.18-1.10 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 170.0, 168.9, 148.2, 134.1, 133.5, 128.9, 128.1, 127.0, 67.5, 54.0, 52.7, 52.5, 42.2, 32.9, 31.4, 23.7, 21.2. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{19}H_{20}ClINaO_4^+$ 496.9987; found 496.9984.

2-(4-Chlorophenyl)-3-(iodomethyl)-8,8-dimethyl-7,9-dioxaspiro[4.5]dec-1-ene-6,10-dione (4ar)



Yellow solid (40.2mg, 45% yield); m.p. 141-143 °C; R_f = 0.25 (ethyl acetate/petroleum ether, 1:6); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37-7.31 (m, 4H), 5.93 (d, *J* = 4.0 Hz, 1H), 3.96-3.89 (m, 1H), 3.41 (dd, *J* = 12.0, 4.0 Hz, 1H), 3.24 (t, *J* = 12.0 Hz, 1H), 2.99 (q, *J* = 8.0 Hz ,1H), 2.61 (dd, *J* = 16.0, 4.0 Hz, 1H), 1.81 (d, *J* = 4.0 Hz, 6H). ^{13}C NMR (101 MHz, CDCl3): ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.1, 168.8, 151.3, 134.9, 131.4, 128.5 (d, *J* = 4.0 Hz, 1C), 129.1, 128.1, 124.4, 105.4, 58.9, 49.0, 43.0, 29.3, 28.8, 9.5. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{17}H_{16}ClINaO_4^+$ 468.9674; not found.

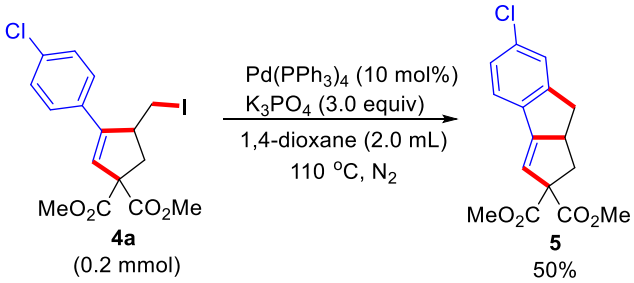
8. Procedure for the scale-up reaction



1-Ethyl-4-ethynylbenzene **1e** (2.0 equiv, 10.0 mmol, 1.30 g), dimethyl allylmalonate **2a** (8.0 mol, 0.86 g), CHI_3 (2.0 equiv, 10 mmol, 3.94 g), K_2CO_3 (2.0 equiv, 10.0 mmol, 1.38 g) and a stir bar were added to a sealed tube under nitrogen atmosphere, MeCN (30.0 mL) as solvent was then added, and the reaction mixture was stirred under a 450 nm blue LEDs irradiation for 12 h at room temperature. After **2a** was completely consumed (monitored by TLC), the crude mixture was directly purified by flash column chromatography on silica gel (ethyl acetate/petroleum ether = 1:30) to give the desired product **4e** as yellow oil (1.50 g, 70% yield).

9. Product derivation.

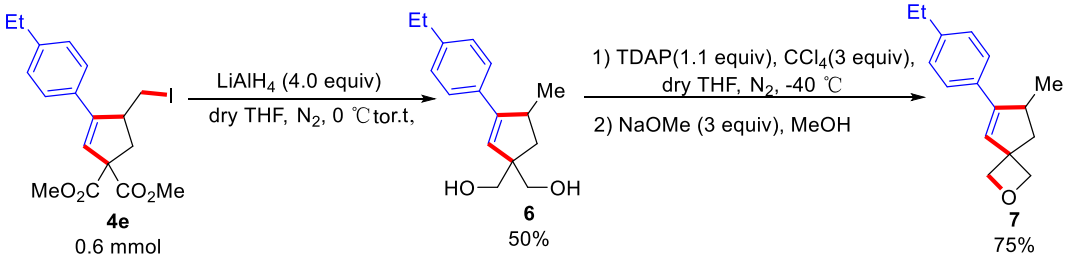
9.1) Intramolecular annulation[7]



Compound **4a** (0.2 mmol, 86.9 mg, 1.0 equiv), $Pd(PPh_3)_4$ (10 mol%, 23.1 mg), K_3PO_4 (0.6 mmol, 127.4 mg, 3.0 equiv) and a stir bar were added to a sealed tube in nitrogen atmosphere, then 1,4-dioxane (2.0 mL) were added. The reaction heated to 110 °C and stirred for 24 hours. After **4a** was completely consumed (monitored by TLC), the reaction mixture was allowed to cool down to room temperature, was then quenched with 1.0 mL 1M HCl and extracted with 2.0 mL CH_2Cl_2. The organic layer was dried over Na_2SO_4, concentrated and purified by flash column chromatography

on silica gel (ethyl acetate/petroleum ether = 1:40) to give the desired product **5** as colorless oil (38.3 mg, 50% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34 (d, *J* =4.0 Hz, 3H), 6.21 (s, 1H), 5.09 (d, *J* = 4.0 Hz, 1H), 5.03 (t, *J* = 4.0 Hz, 1H), 3.77 (s, 6H), 3.74 (t, *J* = 4.0 Hz, 1H), 3.35 (t, *J* = 0.2 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 170.7, 148.6, 147.5, 134.1, 132.4, 129.6, 128.6, 128.1, 106.7, 63.5, 53.0 (d, *J* = 5.0 Hz, 1C), 39.6 (d, *J* = 71.7 Hz, 1C), 38.8, 20.1. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{16}H_{15}ClNaO_4^+$ 329.0551; found 329.0547.

9.2) Reduction and spiro-cyclization[8, 9]

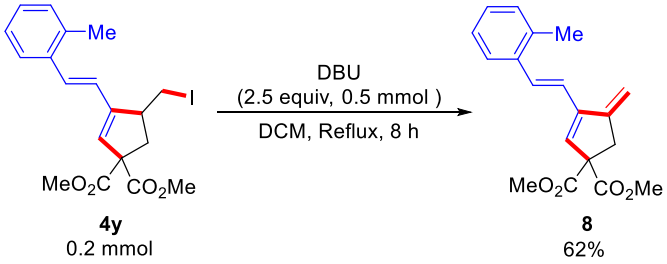


Compound **4e** (1.0 equiv, 0.6 mmol, 257.0 mg) was added to $LiAlH_4$ (4.0 equiv, 2.4 mmol, 91.1 mg) in THF (5.0 mL) at 0 ℃ within 30 min, and the reaction mixture was stirred at room temperature for 90 min, then EtOAc (2 mL) was added in 0 ℃ and the mixture was poured into 1.0 mL 1M HCl. The mixture was extracted with EAOAc, dried over Na_2SO_4, concentrated and purified by flash column chromatography on silica gel to obtain **6** (74.0 mg, 50%, white solid). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.32 (d, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 5.84 (d, *J* = 1.2 Hz, 1H), 3.74 (s, 2H), 3.66 (s, 2H), 3.38-3.30 (m, 1H), 2.65 (t, *J* = 8.0 Hz, 2H), 2.61-2.55 (m, 2H), 2.19 (dd, *J* = 12.0, 8.0 Hz, 1H), 1.49 (dd, *J* = 12.0, 4.0 Hz, 1H), 1.23 (t, *J* = 8.0 Hz, 3H),1.14 (d, *J* = 8.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 150.5, 143.6, 133.1, 127.9, 126.4, 126.1, 69.9, 69.3, 55.2, 38.9, 38.2, 28.6, 21.6, 15.5. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{16}H_{22}NaO_2^+$ 269.1512; found 269.1497.

A mixture of **6** (1.0 equiv, 0.3 mmol, 74.0 mg) and CCl_4 (3.0 equiv, 0.9 mmol, 138.4 mg) in THF (3.0 mL) cooled to -40 ℃, the solution of N, N, N', N', N'', N''-hexamethylphosphinetriamine (TDAP, 1.1 equiv, 0.33 mmol, 53.9 mg) in THF (2.0 mL) are added over three hours under a nitrogen atmosphere. Then the reaction mixture was stirred at room temperature for one hour, and then concentrated under vacuum. The solution of above mixture in the methanol is added to the sodium (3.0 equiv, 0.9 mmol, 20.7 mg) solution within 5 minutes, and after refluxing for one hour, the sodium chloride is filtered. Then the solution was concentrated in vacuum and purified by flash column chromatography on silica gel (ethyl acetate/petroleum ether = 30:1) to obtain **7** (Clear colorless liquid, 75%, 51.4 mg). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.34 (d, *J* = 8.0 Hz, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 6.24 (s, 1H), 4.77 (q, *J* = 4.0 Hz, 2H), 4.71 (d, *J* = 8.0 Hz, 1H), 4.66 (d, *J* = 4.0 Hz, 1H), 3.26-3.18 (m, 1H), 2.64 (dd, *J* = 12.0, 8.0 Hz, 2H), 2.54 (dd, *J* = 12.0, 8.0 Hz, 1H), 2.05 (dd, *J* = 12.0, 4.0 Hz, 1H), 1.24 (t, *J* = 8.0 Hz, 3H), 1.06 (d, *J* = 8.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$):

δ 149.1, 143.7, 132.6, 128.2, 127.9, 126.3, 83.9, 83.3, 51.6, 45.4, 39.2, 28.6, 21.2, 15.5. HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{16}H_{21}O^+$ 229.1587; found 229.1578.

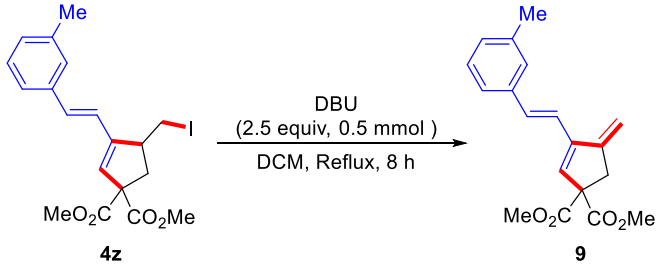
9.3) Elimination of hydroiodic acid[4]



4y
0.2 mmol

8
62%

Compound **4y** (0.2 mmol, 88.0 mg, 1.0 equiv), DBU (0.5 mmol, 76.6 mg, 2.5 equiv), DCM (2.0 mL) and a stir bar were added to a 10 mL sealed tube in nitrogen atmosphere. The solution was refluxed for 8 h. Then the solution was concentrated in vacuum and purified by flash column chromatography on silica gel (ethyl acetate/petroleum ether =30:1) to obtain **8** (38.7 mg, 62%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54-7.50 (m, 1H), 7.26 (t, *J* = 8.0 Hz, 1H), 7.21-7.16 (m, 3H), 6.65 (d, *J* = 16.0 Hz, 1H), 6.32 (s, 1H), 5.24 (t, *J* = 4.0 Hz, 1H), 5.07 (d, *J* = 1.2 Hz,1H), 3.78 (s, 6H), 3.30 (t, *J* = 4.0 Hz, 2H), 2.39 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 170.9, 148.7, 144.5, 136.0 (d, *J* = 4.0 Hz, 1C), 131.2, 130.4, 130.1 (d, *J* = 8.1 Hz, 1C), 128.0, 126.2, 125.6, 120.8, 105.0, 63.3, 53.0 (d, *J* = 6.1 Hz, 1C), 38.9, 19.9. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{19}H_{20}NaO_4^+$ 335.1254; found 335.1260.

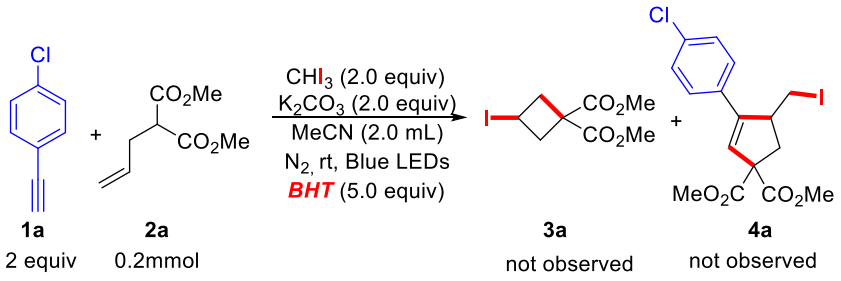


4z
0.2 mmol

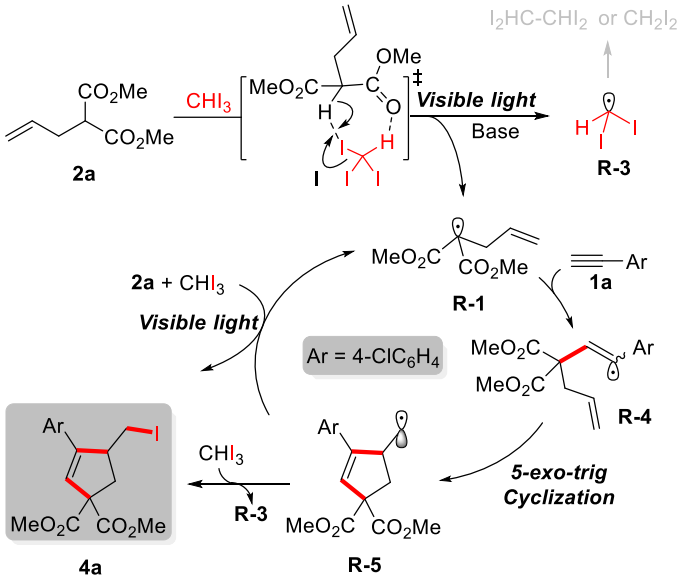
9
54%

Compound **4z** (0.2 mmol, 88.0 mg, 1.0 equiv), DBU (0.5 mmol, 76.6 mg, 2.5 equiv), DCM (2.0 mL) and a stir bar were added to a 10 mL sealed tube in nitrogen atmosphere. The solution was refluxed for 8 h. Then the solution was concentrated in vacuum and purified by flash column chromatography on silica gel (ethyl acetate/petroleum ether =30:1) to obtain **9** (33.7 mg, 54%). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.27 (s, 1H), 7.23 (t, *J* = 8.0 Hz, 2H), 7.09 (d, *J* = 8.0 Hz 1H), 7.02 (d, *J* = 16.0 Hz, 1H), 6.74 (d, *J* = 16.0 Hz, 1H), 6.32 (s, 1H), 5.23 (t, *J* = 4.0 Hz, 1H), 5.06 (dd, *J* = 4.0, 0.4 Hz, 1H), 3.77 (s, 6H), 3.28 (t, *J* = 4.0 Hz, 2H), 2.36 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 170.9, 148.7, 144.3, 138.2, 136.8, 133.5, 129.9 (d, *J* = 7.1 Hz, 1C), 129.0, 128.6, 127.3, 123.8, 119.1, 104.9, 63.3, 53.0 (d, *J* = 5.0 Hz, 1C), 38.8, 21.4. HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{19}H_{20}NaO_4^+$ 335.1254; found 335.1259.

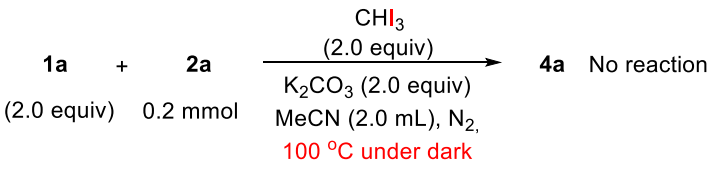
10. Control experiments and Reaction mechanism proposed



Mechanism proposed for the radical iodoalkylative cyclization



11. Reaction thermally under dark condition



1a (54.6 mg, 2.0 equiv), **2a** (34.8 mg, 0.2 mmol), CHI_3 (157.5 mg, 2.0 equiv), K_2CO_3 (55.3 mg, 2.0 equiv) and a stir bar were added to a sealed tube under a nitrogen atmosphere in 100 ℃ oil bath under dark. MeCN (2.0 mL) as solvent was then added. The mixture was stirred for 12 h at room temperature, and no reaction was observed.

12. Light on-off experiment

1a (273.0 mg, 2.0 equiv), **2a** (86.1 mg, 0.5 mmol), CHI_3 (393.8 mg, 2.0 equiv), K_2CO_3 (138.3 mg, 2.0 equiv), MeCN (5.0 mL) and a stir bar was added to a 10 mL sealed tube. Firstly, the mixture was irradiated by a 450 nm blue LEDs for 3 h under N_2 atmosphere. 0.5 mL reaction mixture was taken out from the reaction system under N_2 atmosphere for ^{1}H NMR analysis. The remaining mixture was stirred in the absence of light for an additional 1 h. Then another 0.5 mL reaction mixture was taken out from the reaction system under N_2 atmosphere for ^{1}H NMR analysis. Secondly, the blue LEDs were turned back on to irradiate the remaining mixture. After an additional 4 h of irradiation, the lamps were turned off and 0.5 mL reaction mixture was taken out from the reaction system for ^{1}H NMR analysis. The remaining mixture was stirred in the absence of light for an additional 1 h. Then another 0.5 mL reaction mixture was taken out from the reaction system ^{1}H NMR analysis. Finally, the LEDs were turned back on to irradiate the remaining mixture, and after an additional 5 h of irradiation, the lamps were turned off and 0.5 mL reaction mixture was taken out from the reaction system for ^{1}H NMR analysis. The remaining mixture was stirred in the absence of light for an additional 1 h. Then another 0.5 mL reaction mixture was taken out from the reaction system ^{1}H NMR analysis.

Time/hours	3	4	8	9	14	15
Conversion of 2a (%)	33	33	69	69	100	100

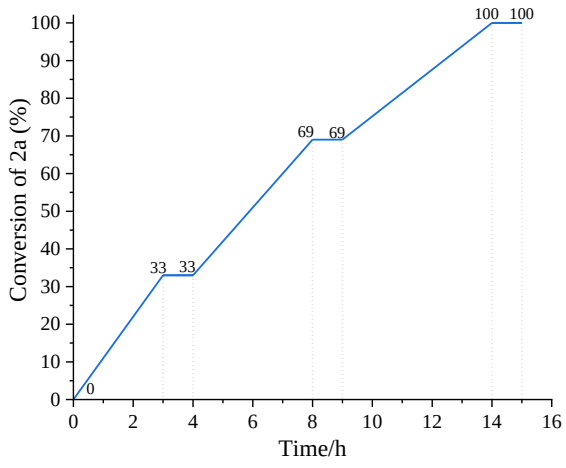


Figure S1: Light on-off experiment

13. The UV-Visible absorption spectroscopy experiment

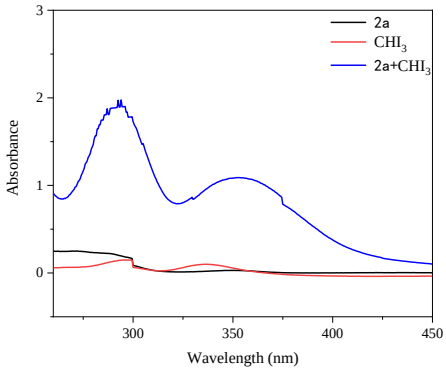


Figure S2: UV-Vis absorption spectroscopy of **2a**, CHI_3 and their mixture

The UV-Vis spectroscopy experiment was carried out using the 0.1 M dimethyl allyl malonate **2a** solution of MeCN solvent; 0.001 M CHI_3 solution of MeCN solvent as the sample, respectively. As the results shown in Fig S1, the maximum absorption wavelength of the malonate ester and CHI_3 were both recorded in the UV spectral region, while the malonate ester and CHI_3 were mixed together (1:1 v/v) and under the visible-light irradiation for five minutes, a significant red shift for the absorption spectroscopy of reaction components were observed.

14. X-ray Structure of product

Single crystal of **4a** was obtained by recrystallization from dichloromethane/n-hexane solution. The crystal structure was determined by standard crystallographic methods. A colorless block-shaped crystal (mm^3) was used for single-crystal X-ray diffraction. The data were collected at 273.15 K using a Bruker D8 QUEST X-ray diffractometer with graphite-monochromated Mo-Kα radiation (λ = 0.71073 Å). The structure was solved by Direct Methods and refined by full-matrix least-squares techniques on F2 with anisotropic displacement parameters for all atoms using SHELX-2014. All the processes were performed within Olex2. The final refinements included anisotropic displacements parameters for all atoms and a secondary extinction correction. The crystallographic parameter data is listed in **Table S1.**

Figure S1. Crystal Structure of **4a** with thermal ellipsoids drawn at the 30% probability level. Hydrogens are omitted for clearity and Crystallographic data for **4a** have been deposited with the Cambridge Crystallographic Data Center as CCDC: **2208470**. And the crystal data and details of the data collection are given in **Table S1**

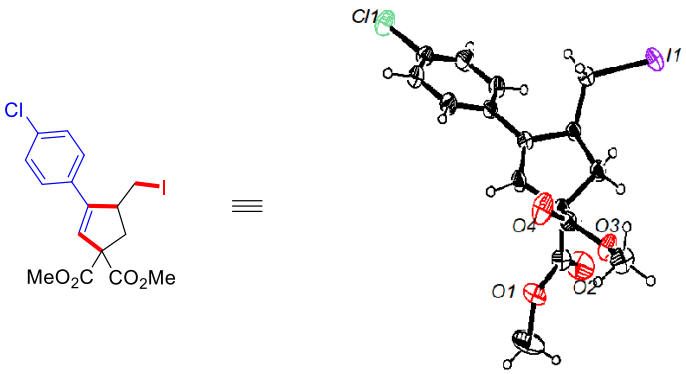


Figure S1: Crystal structure of **4a**

Table S1. Crystal data of **4a**

Empirical formula	$C_{16}H_{16}ClIO_4$	
CCDC	2208470	
Formula weight	434.64	
Temperature(K)	273.15	
Wavelength(Å)	0.71073	
Crystal system	triclinic	
space group	P-1	
Unit cell dimensions	a = 8.2223(3) Å b = 10.3432(5) Å c = 11.0230(6) Å	α =93.878(2)° β = 97.956(2) ° γ = 110.091(4)°
Volume($Å^3$)	865.22(7)	
Z	2	
Calculated density($g \cdot cm^{-3}$)	1.392	
Absorption coefficient(mm^{-1})	1.251	
F(000)	2512.0	
Crystal size(mm)	0.12 × 0.11 × 0.1	
θ range for data collection	3.658 to 55.046	
h, *k*, *l* ranges	-31<=h<=32, -13<=k<=16, -25<=l<=27	
Reflections collected	29006	
Independent reflections	6792 [R(int) = 0.0453]	
Completeness	99%	
Absorption correction	Multi-scan	
Data / restraints / parameters	6792/42/319	
Goodness-of-fit on F^2	1.066	
Final *R* indices [I>2σ (I)]	R_1 = 0.0608, wR_2 = 0.1731	
R indices (all data)	R_1 = 0.1020, wR_2 = 0.1960	
Largest diff. peak and hole	1.16 and -0.76 $e \cdot Å^{-3}$	

15. References

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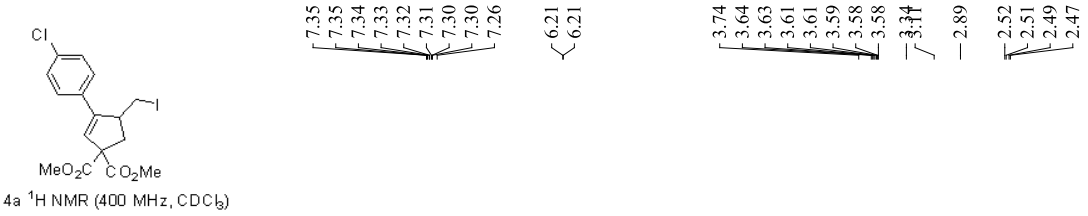
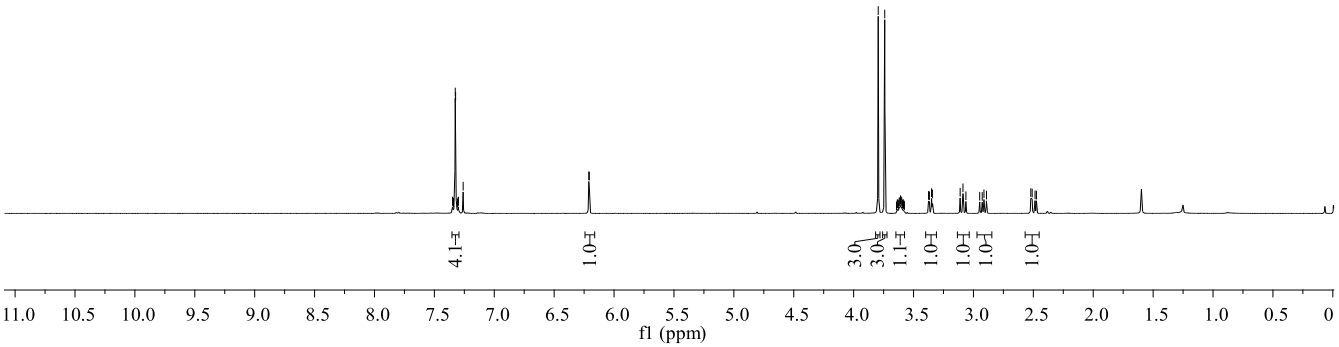
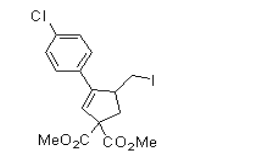
[9] B. Castro, C. Selve, *Tetrahedron Lett*. **1973**, 4459.

16. ^{1}H, ^{13}C and ^{19}F NMR spectra

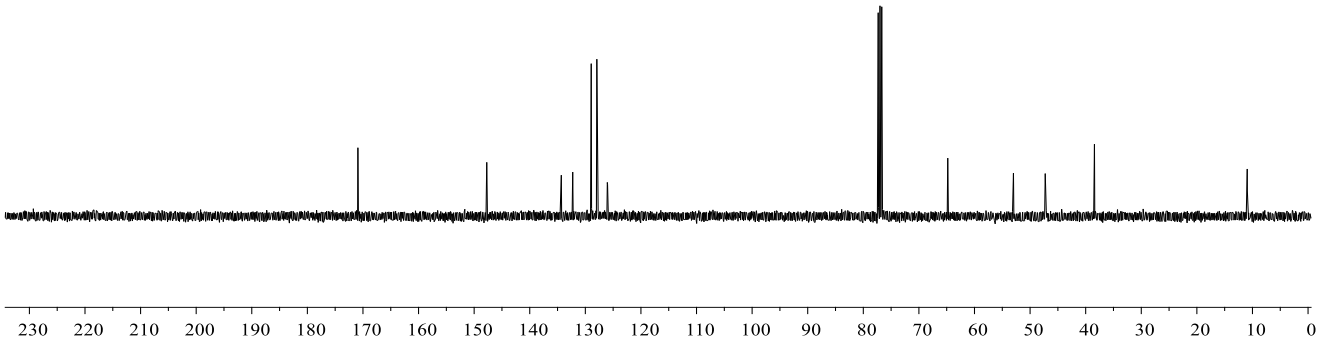


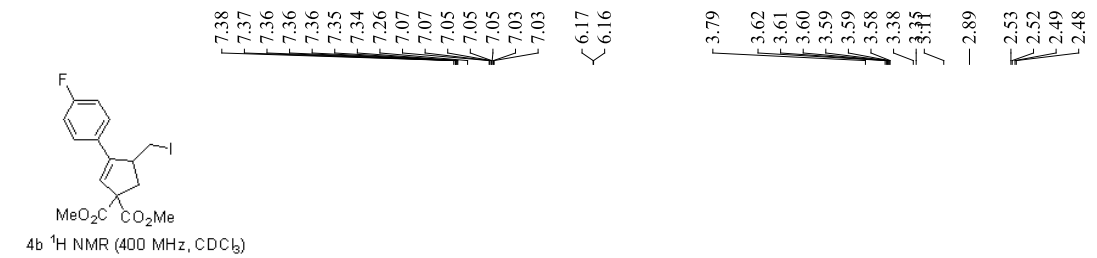
4a ^{1}H NMR (400 MHz, $CDCl_3$)



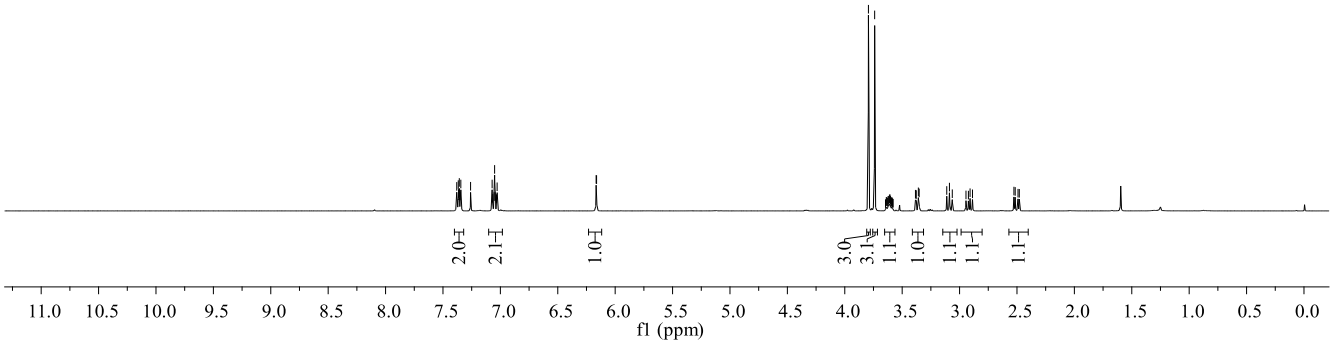


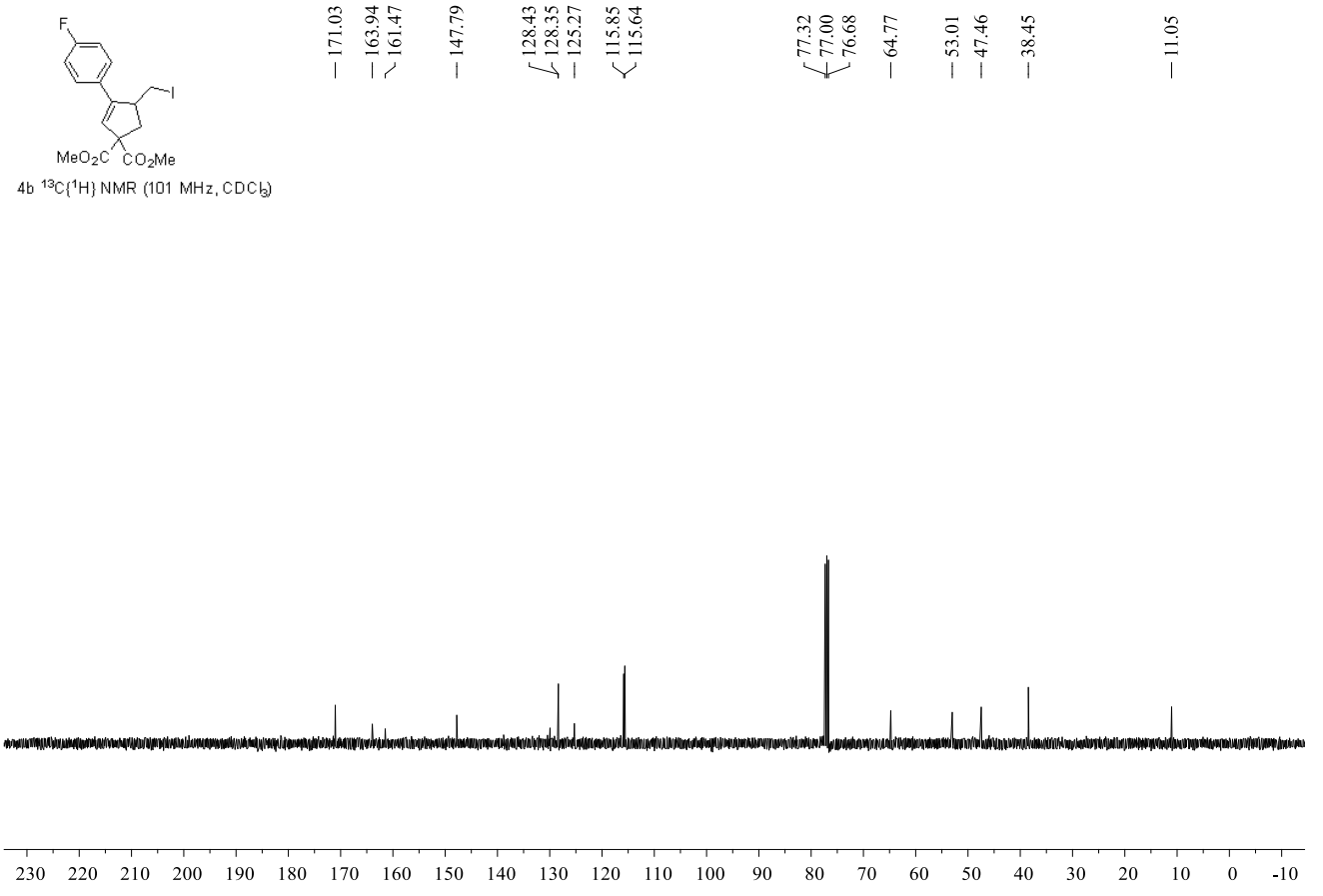
4a $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



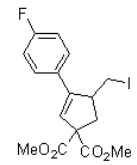


4b ^{1}H NMR (400 MHz, $CDCl_3$)

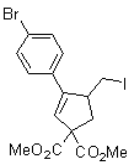




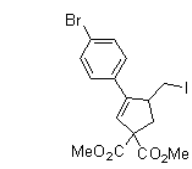
4b ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$)



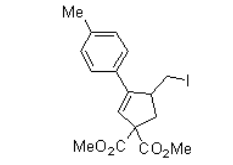
4b $^{19}F\{^{1}H\}$ NMR (376 MHz, $CDCl_3$)



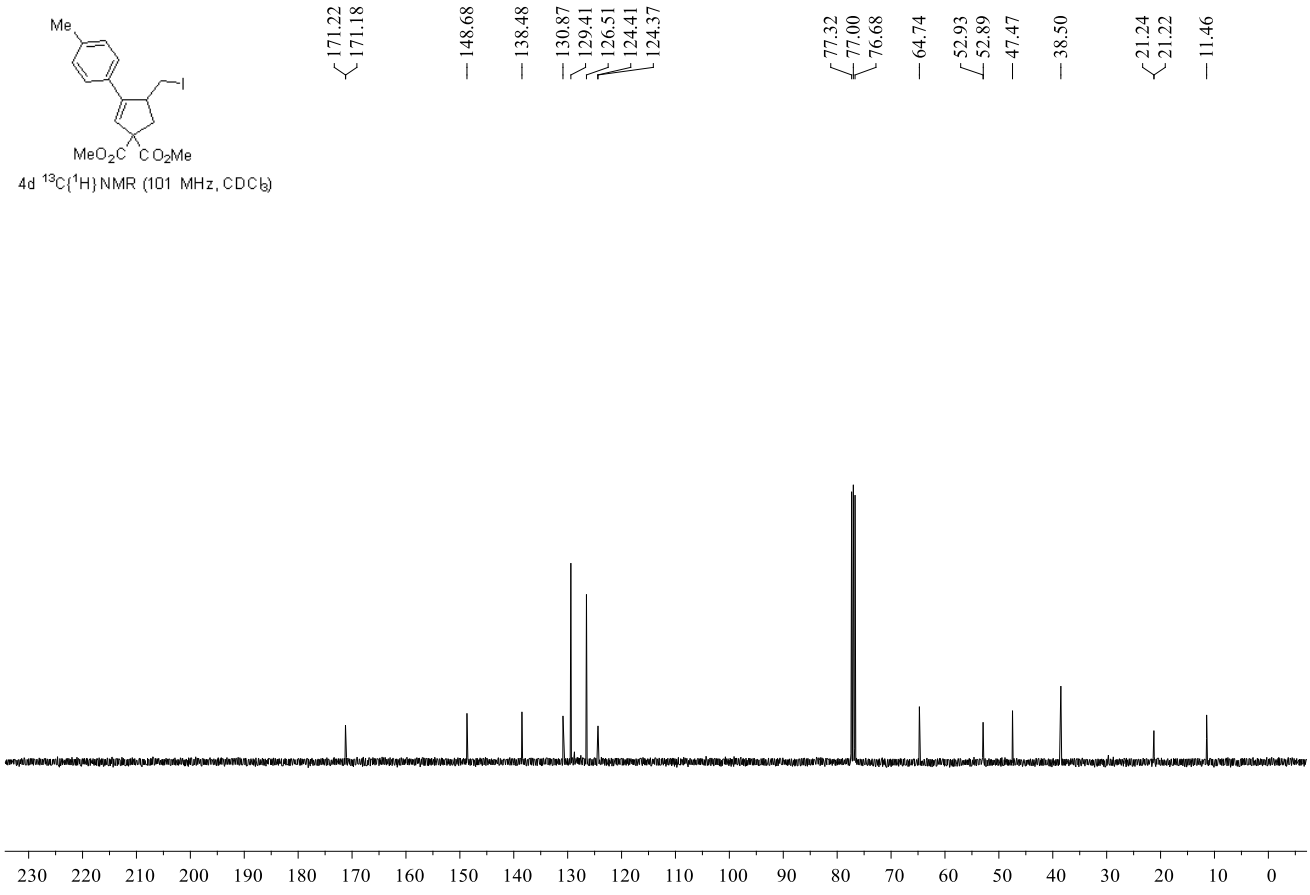
4c ^{1}H NMR (400 MHz, $CDCl_3$)



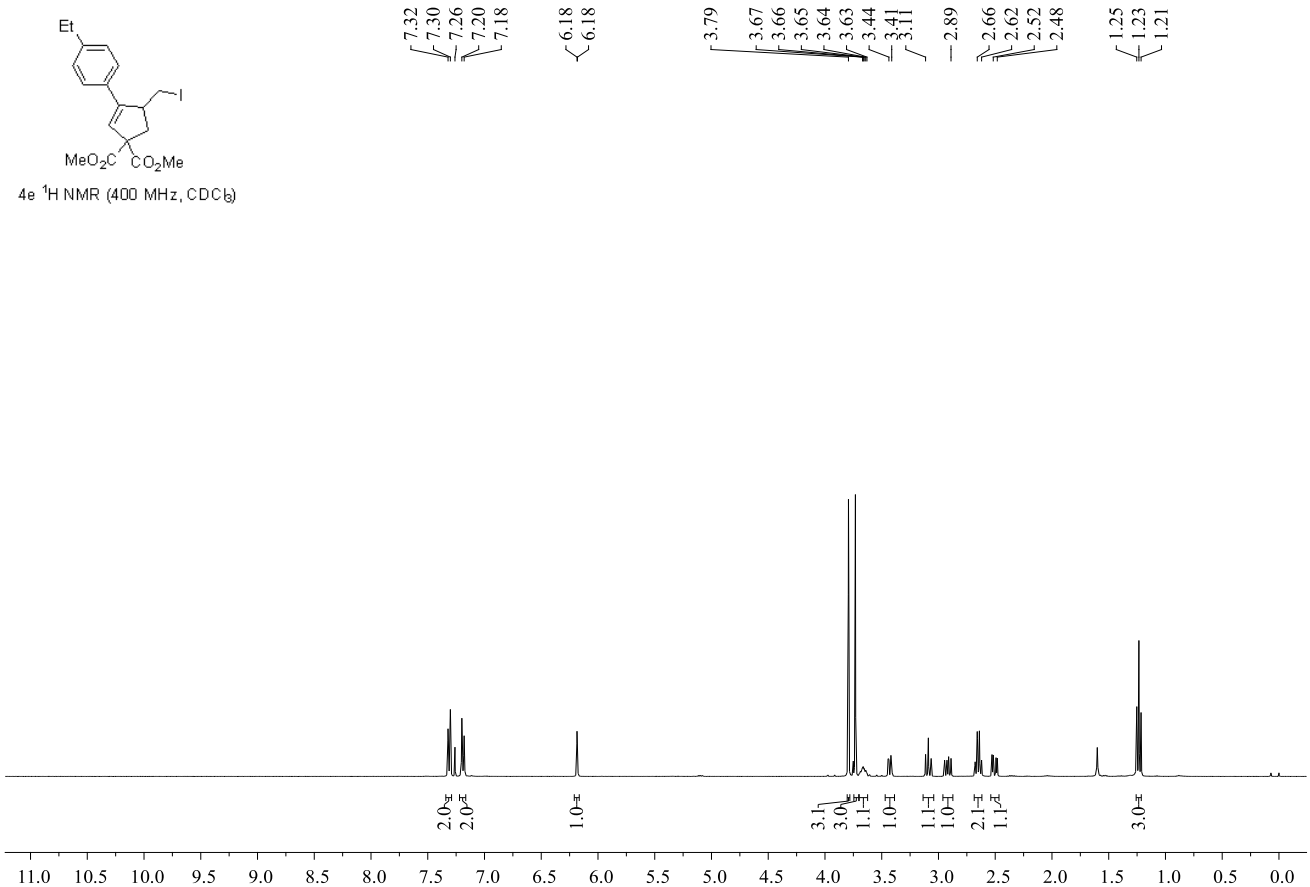
4c $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



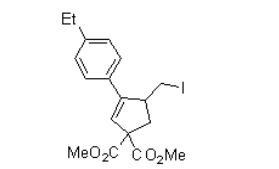
4d ^{1}H NMR (101 MHz, $CDCl_3$)



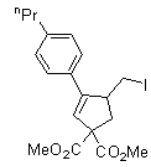
4d $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



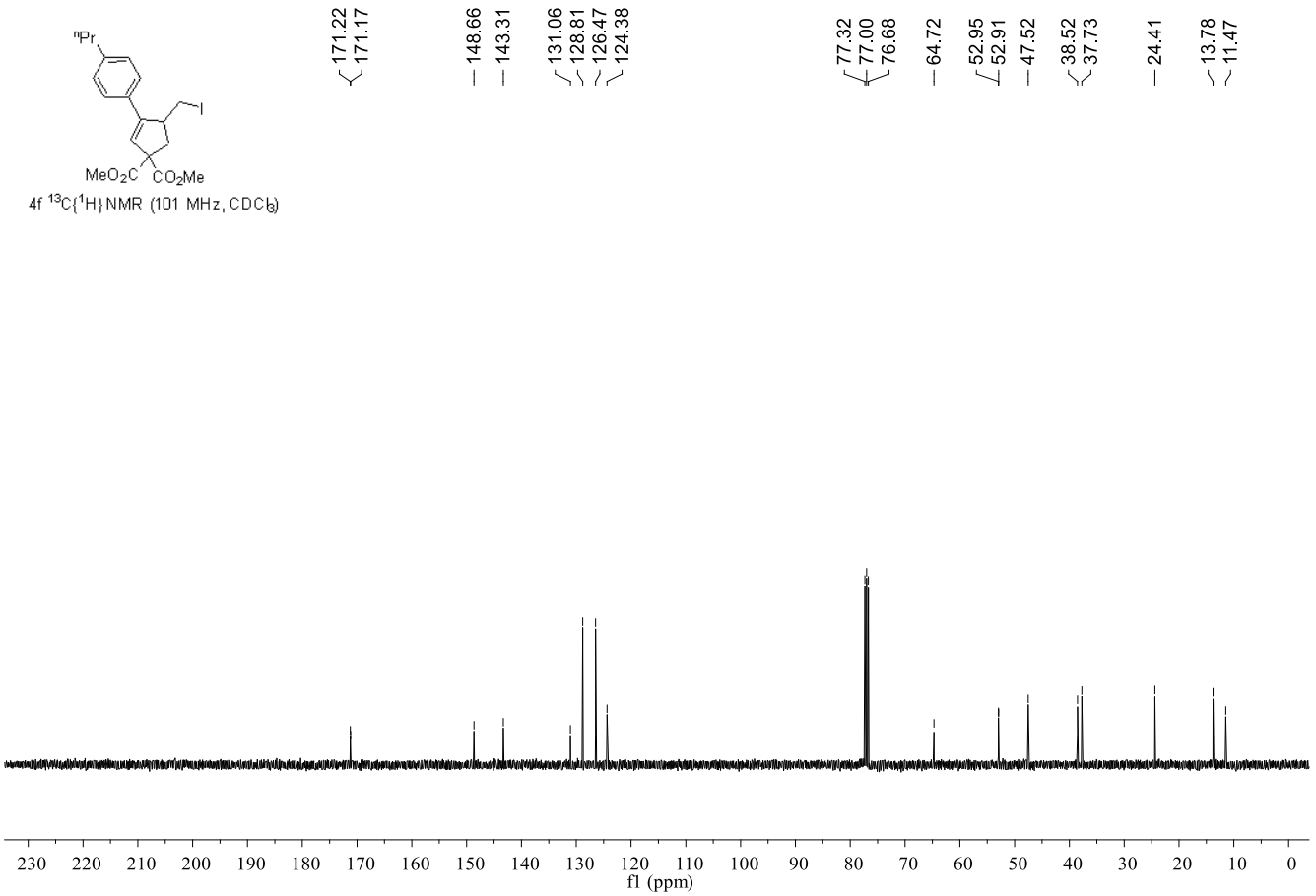
4e ^{1}H NMR (400 MHz, $CDCl_3$)



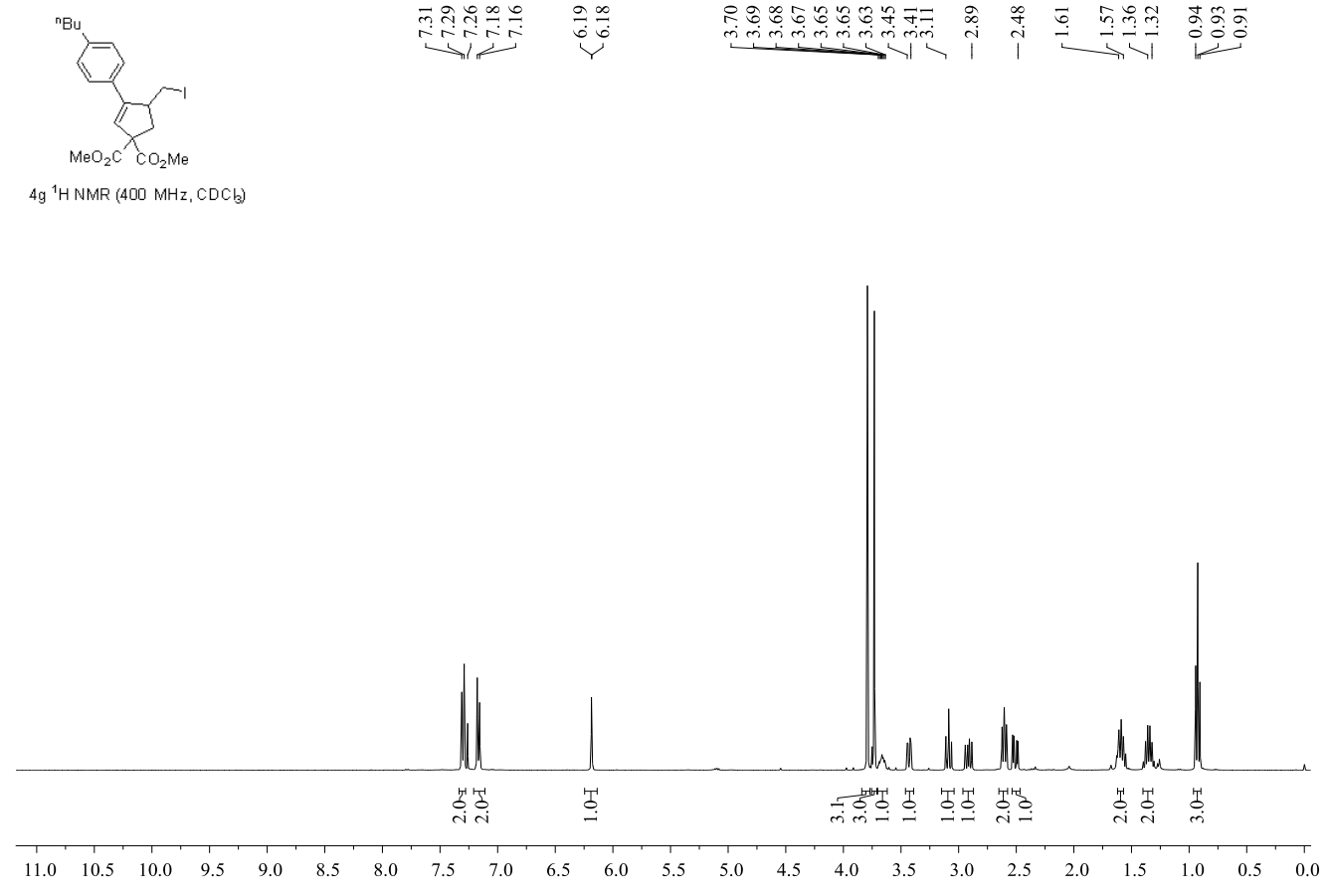
4e $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



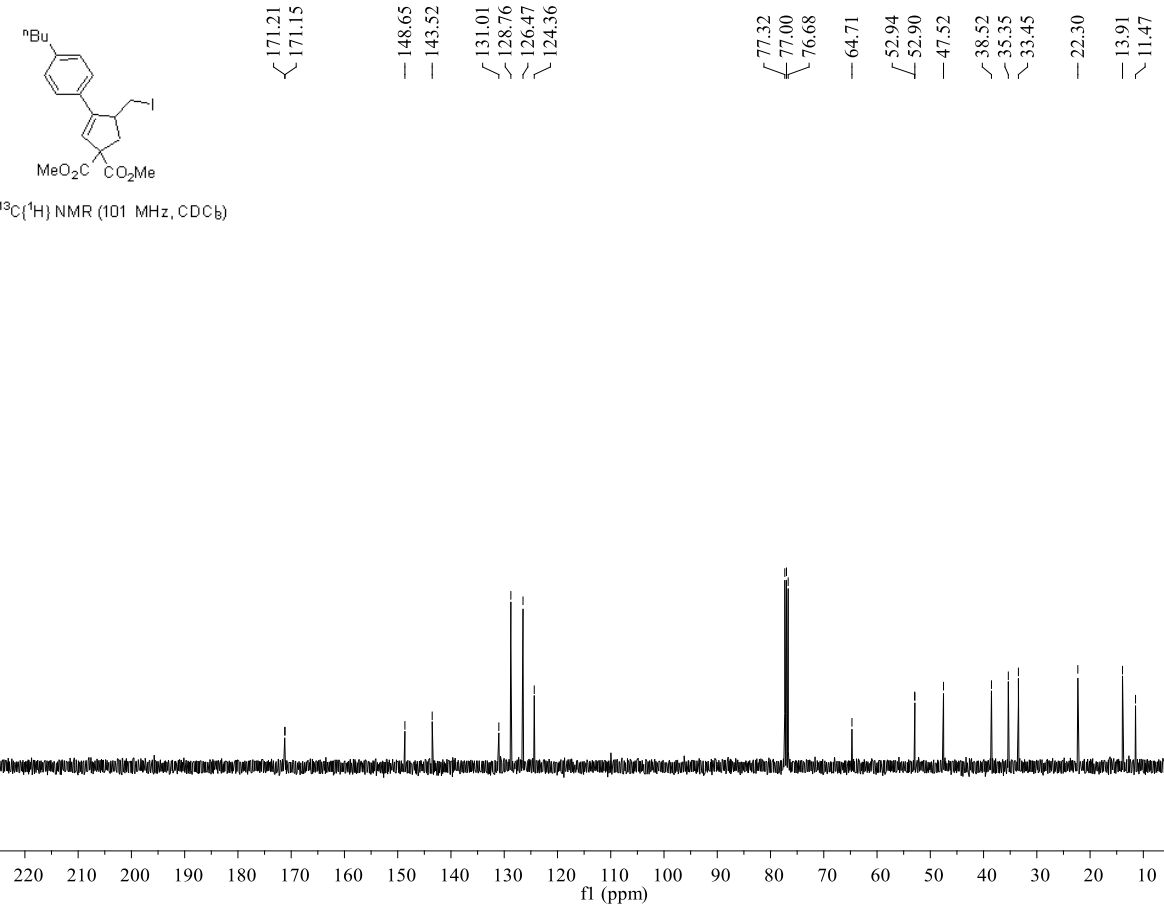
4f ^{1}H NMR (400 MHz, $CDCl_3$)



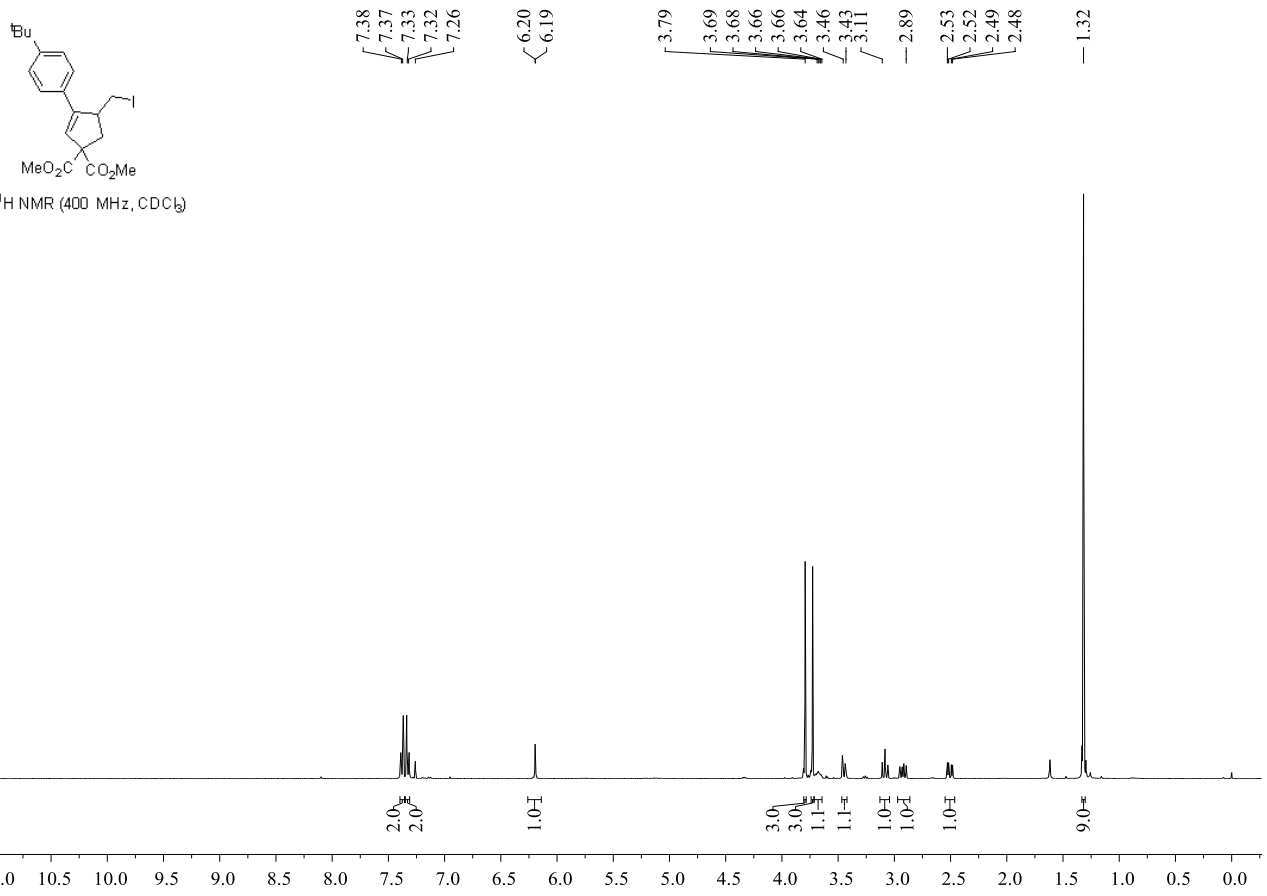
4f $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



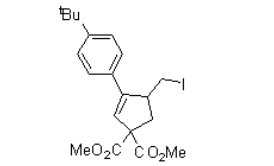
4g ^{1}H NMR (400 MHz, $CDCl_3$)



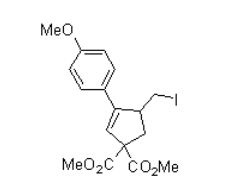
4g $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



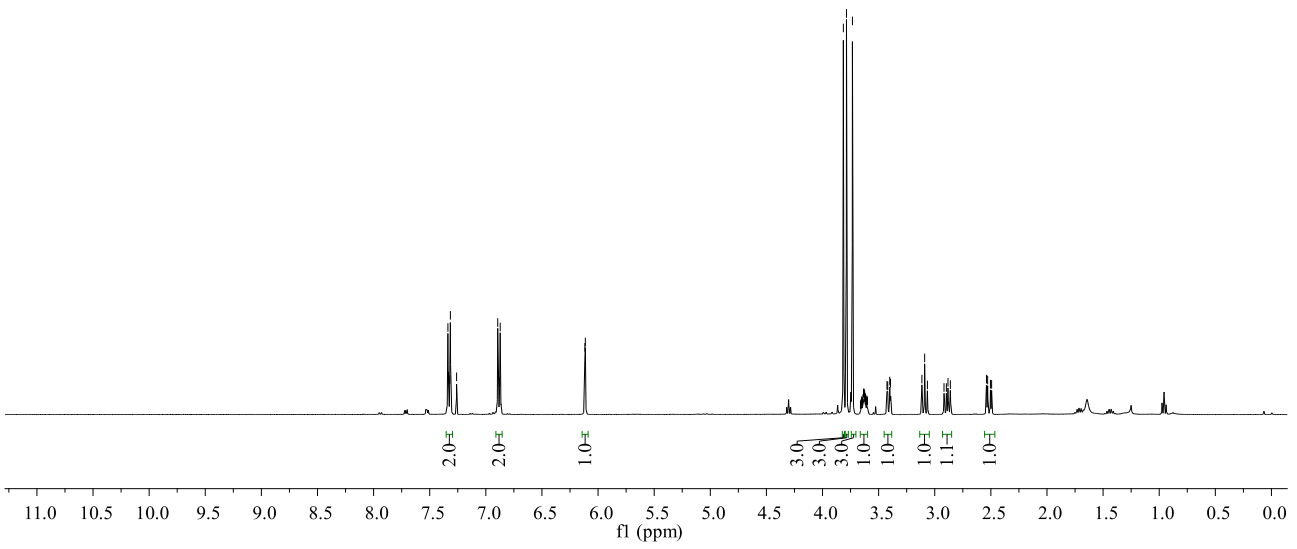
4h ^{1}H NMR (400 MHz, $CDCl_3$)

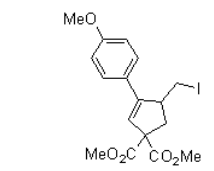


4h $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



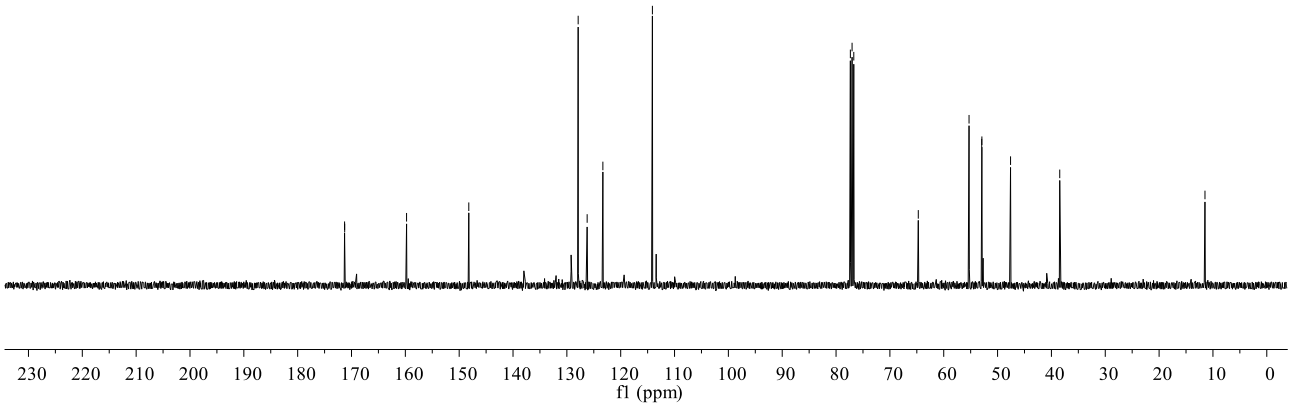
4i ^{1}H NMR (400 MHz, $CDCl_3$)



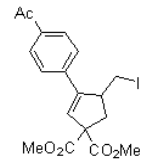


4i $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)

171.29
171.27
159.77
148.22
127.90
126.23
123.31
114.11
77.34
77.03
76.71
64.73
55.30
52.92
47.59
38.45
11.48

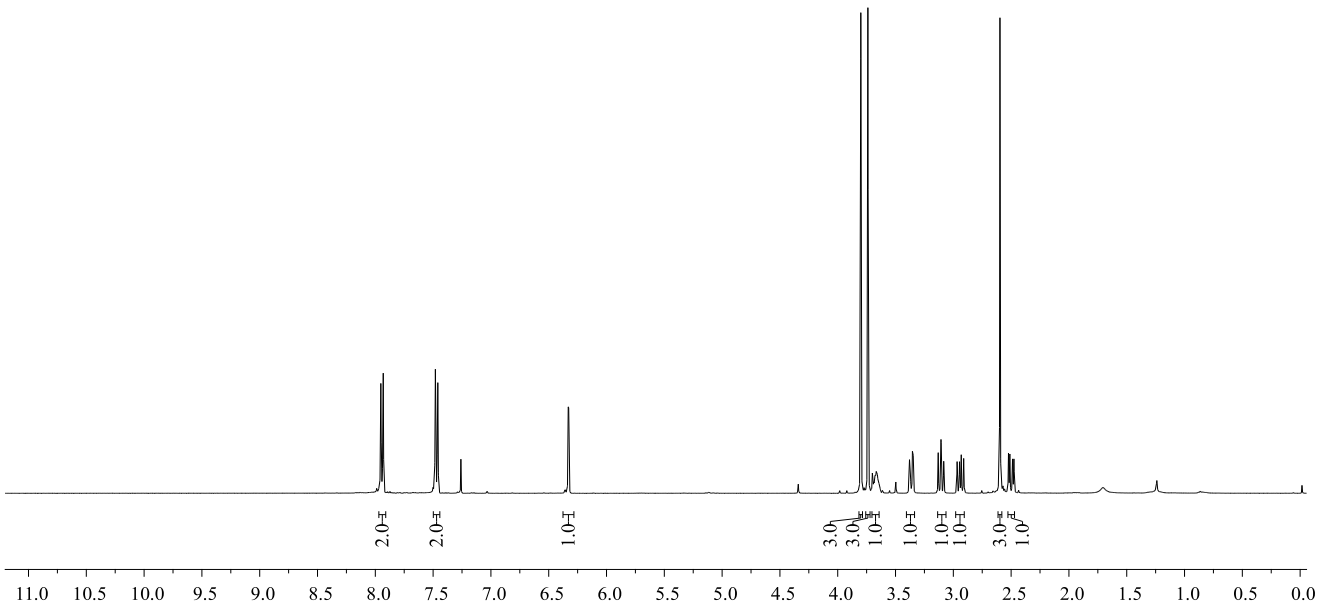


230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



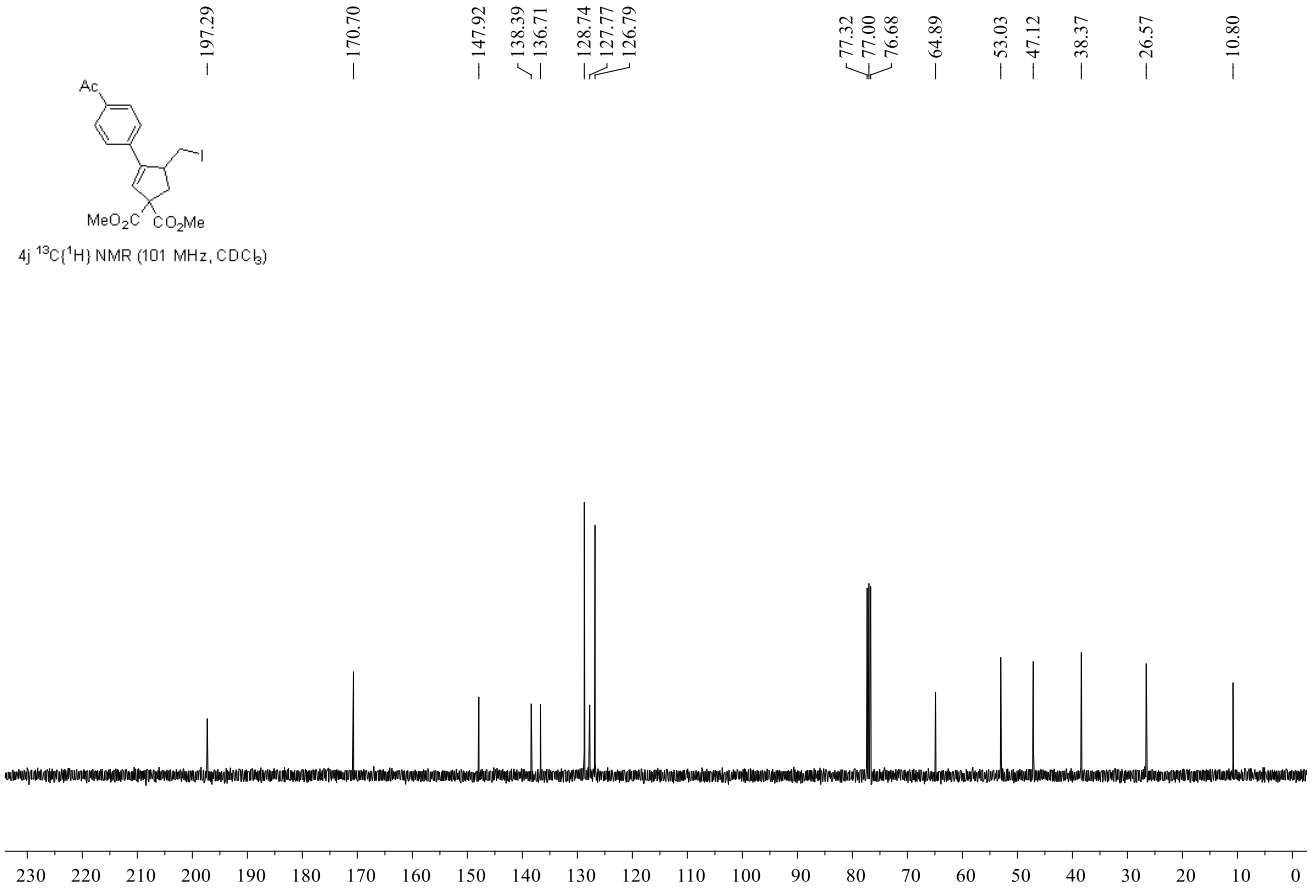
4j ^{1}H NMR (400 MHz, $CDCl_3$)

7.95
7.93
7.48
7.46
7.26
6.33
6.33
3.74
3.69
3.68
3.67
3.67
3.66
3.66
3.65
2.91
2.60
2.52
2.51
2.49
2.48

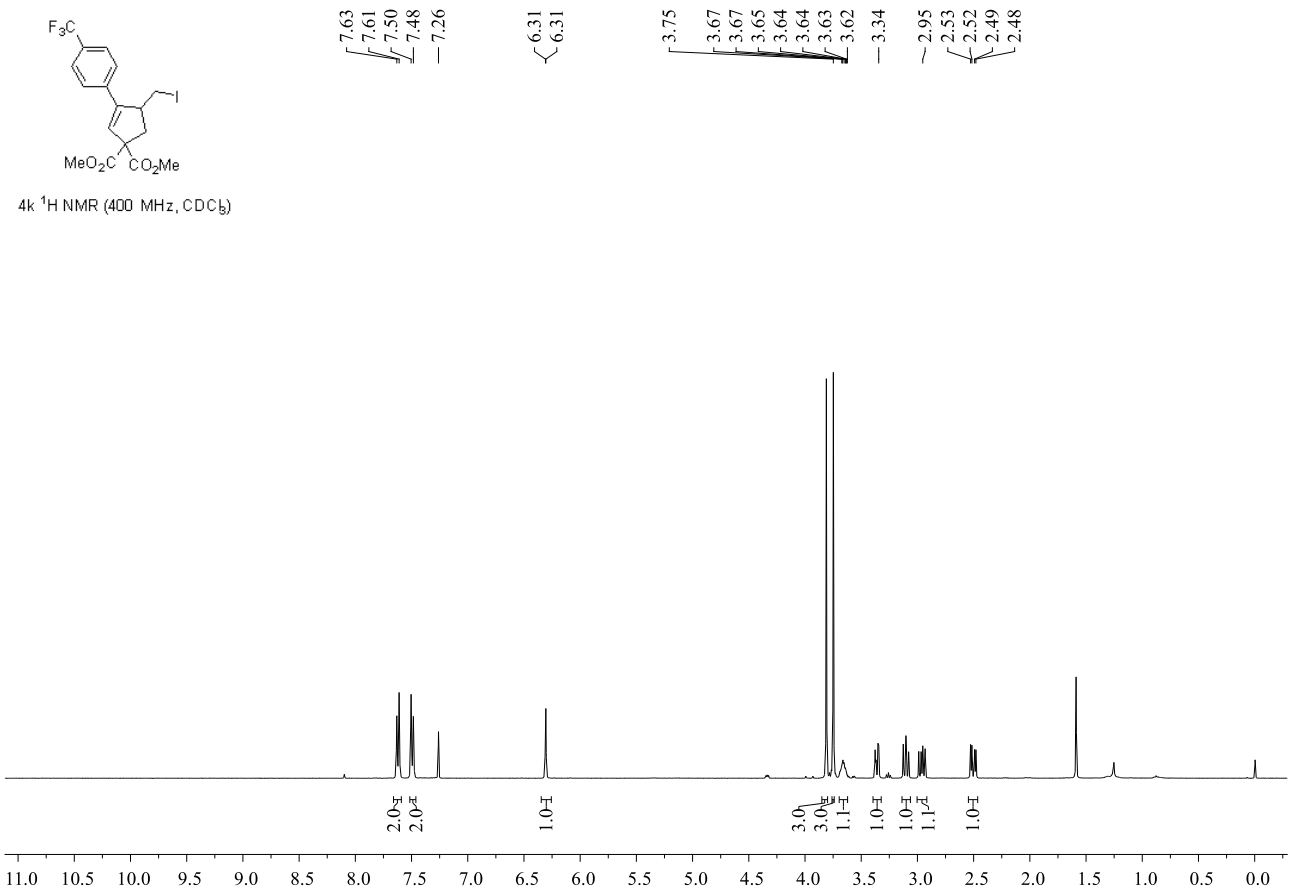


2.0
2.0
1.0
3.0
3.0
1.0
1.0
1.0
1.0
3.0
1.0

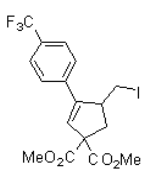
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0



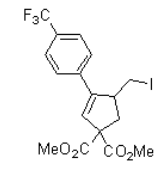
4j $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



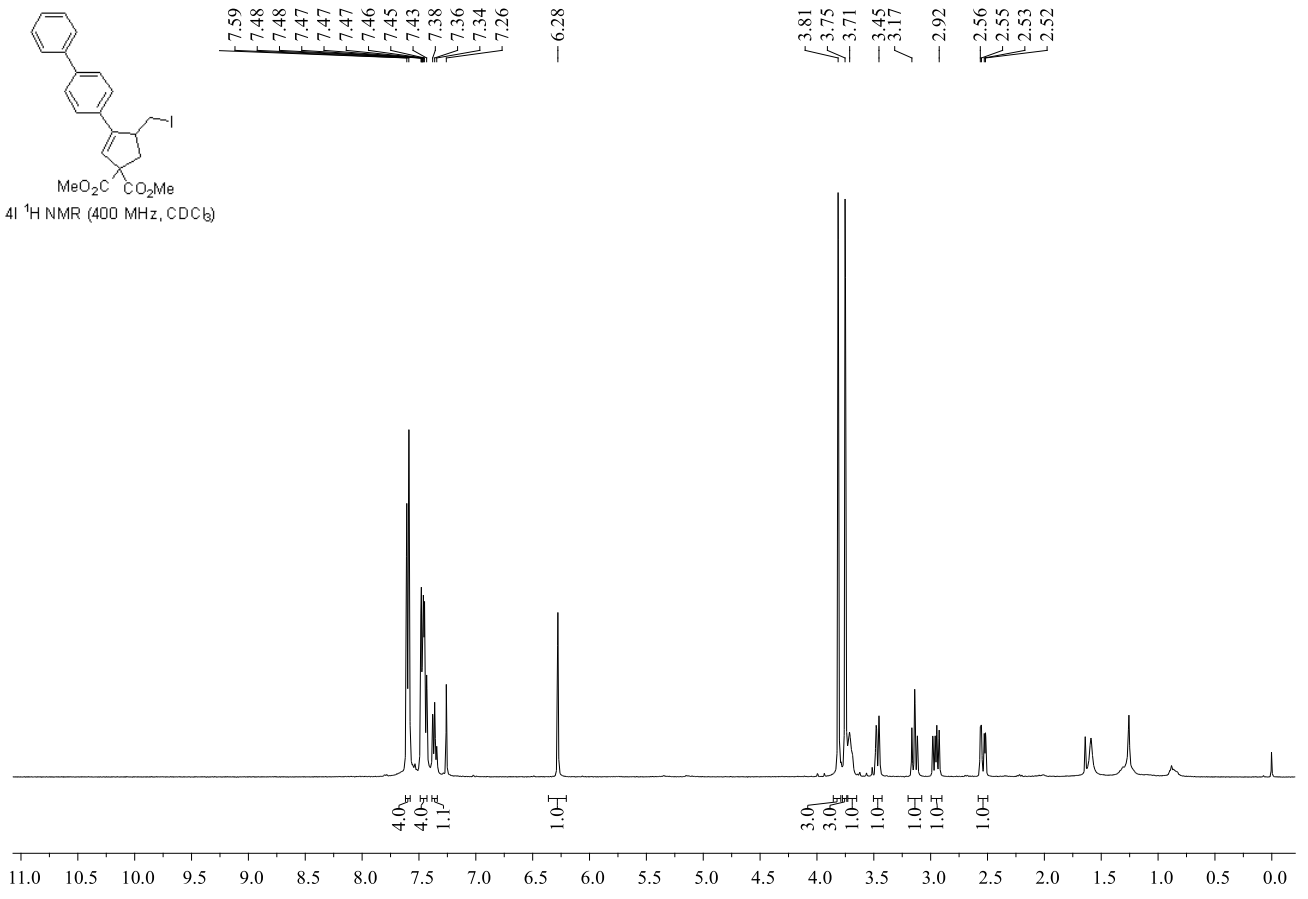
4k ^{1}H NMR (400 MHz, $CDCl_3$)

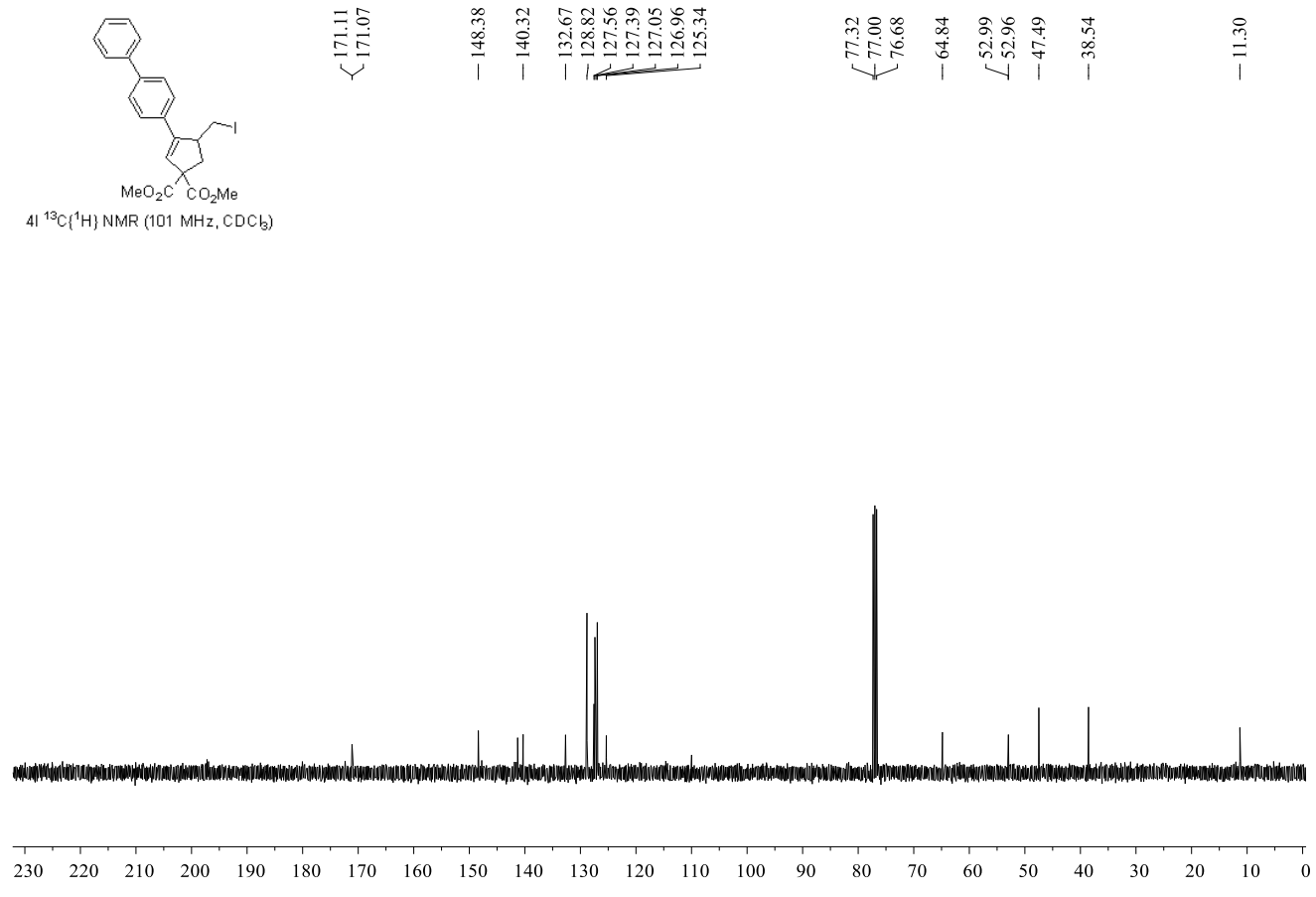


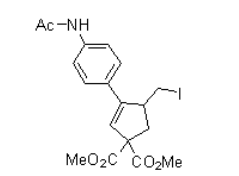
4k $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



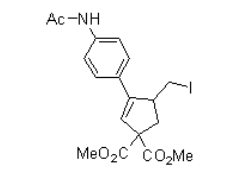
4k $^{19}F\{^{1}H\}$ NMR (376 MHz, $CDCl_3$)


MeO2C CO2Me
4l 1H NMR (400 MHz, CDCl3)
7.59
7.48
7.48
7.47
7.47
7.47
7.46
7.45
7.43
7.38
7.36
7.34
7.26
6.28
3.81
3.75
3.71
3.45
3.17
2.92
2.56
2.55
2.53
2.52
4.0
4.0
1.1
1.0
3.0
3.0
1.0
1.0
1.0
1.0
1.0
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

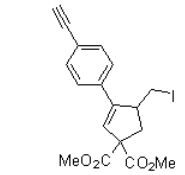

MeO2C CO2Me
4l 13C{1H} NMR (101 MHz, CDCl3)
171.11
171.07
148.38
140.32
132.67
128.82
127.56
127.39
127.05
126.96
125.34
77.32
77.00
76.68
64.84
52.99
52.96
47.49
38.54
11.30
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0



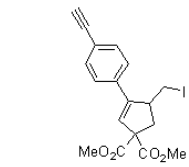
4m ^{1}H NMR (400 MHz, $CDCl_3$)



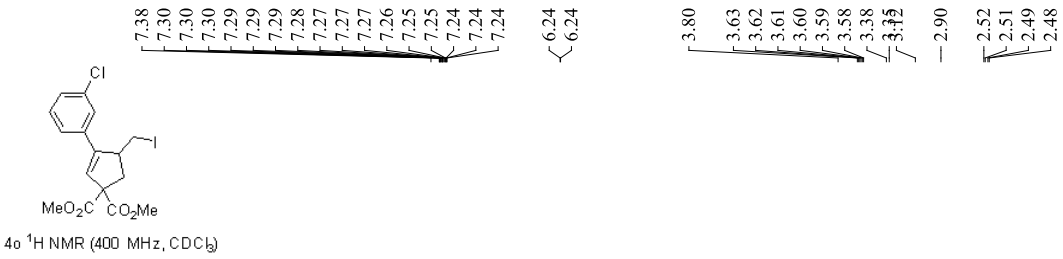
4m ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$)



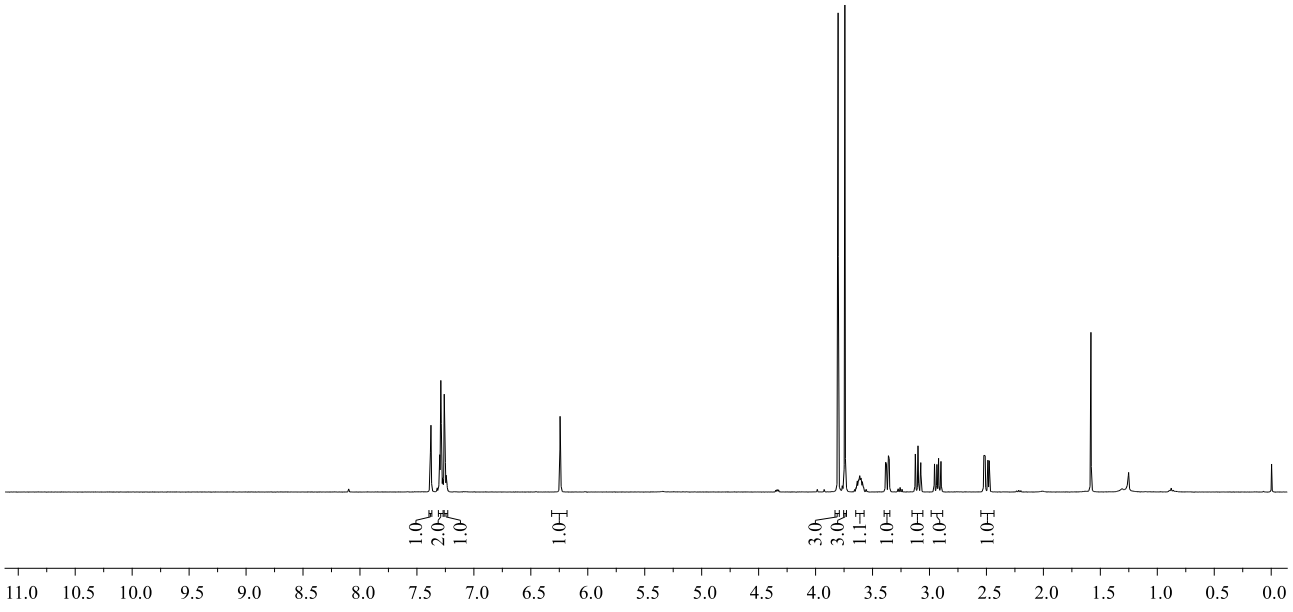
4n ^{1}H NMR (400 MHz, $CDCl_3$)

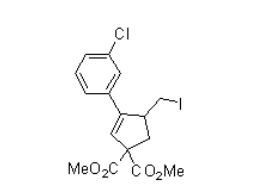


4n ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$)



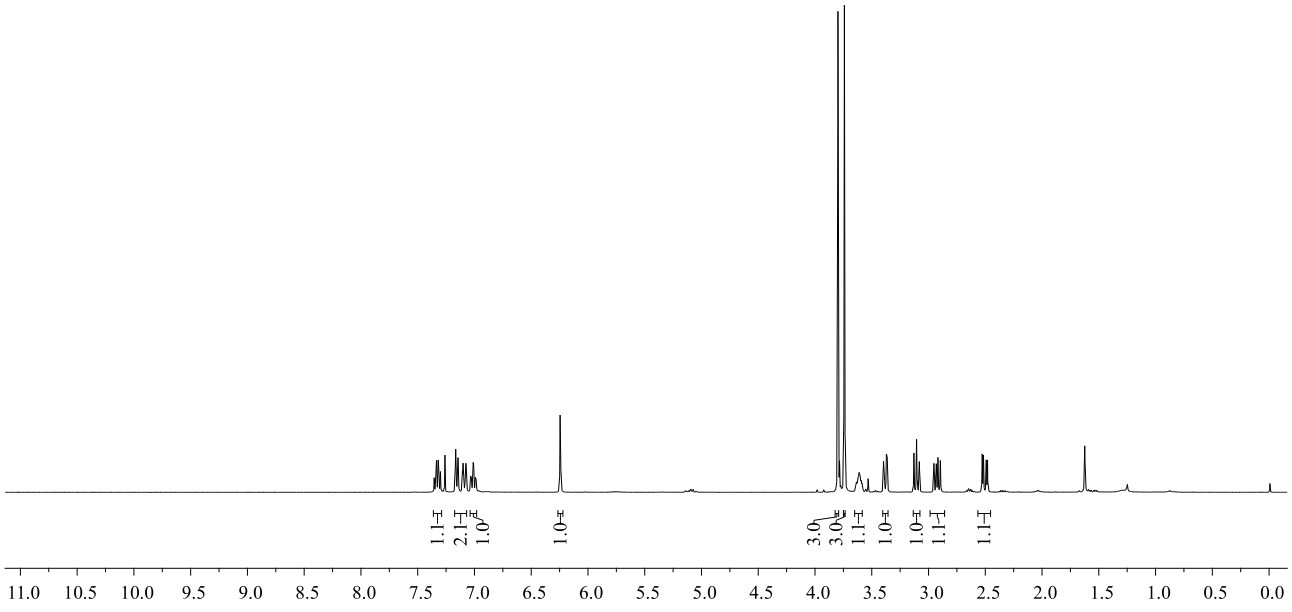
4o ^{1}H NMR (400 MHz, $CDCl_3$)

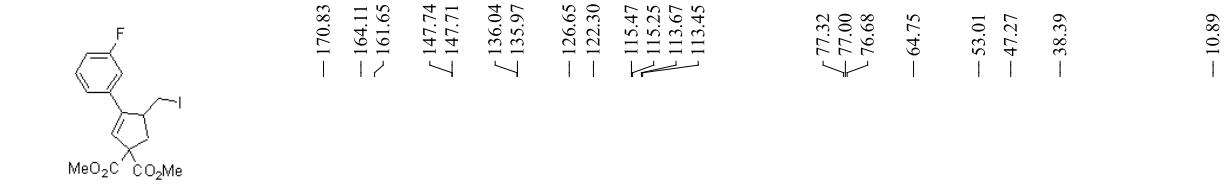




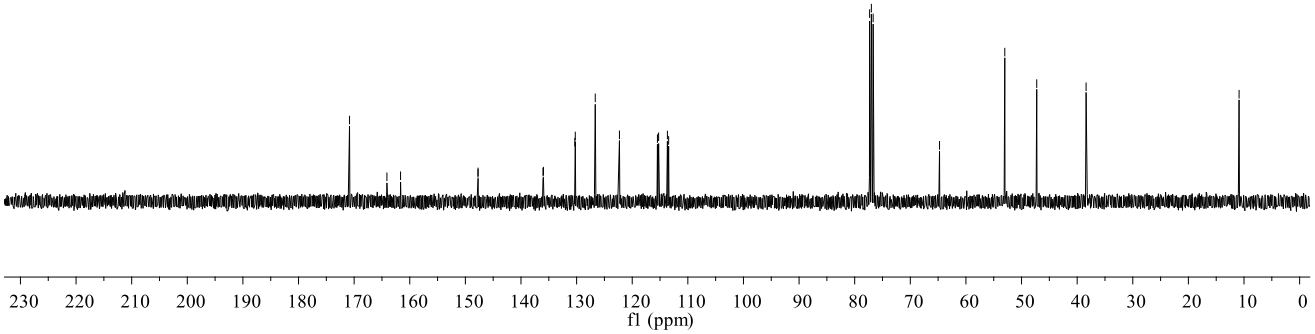
4o ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$)

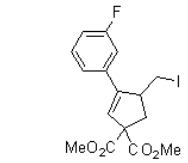
4p ^{1}H NMR (400 MHz, $CDCl_3$)



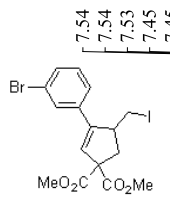


4p ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$)

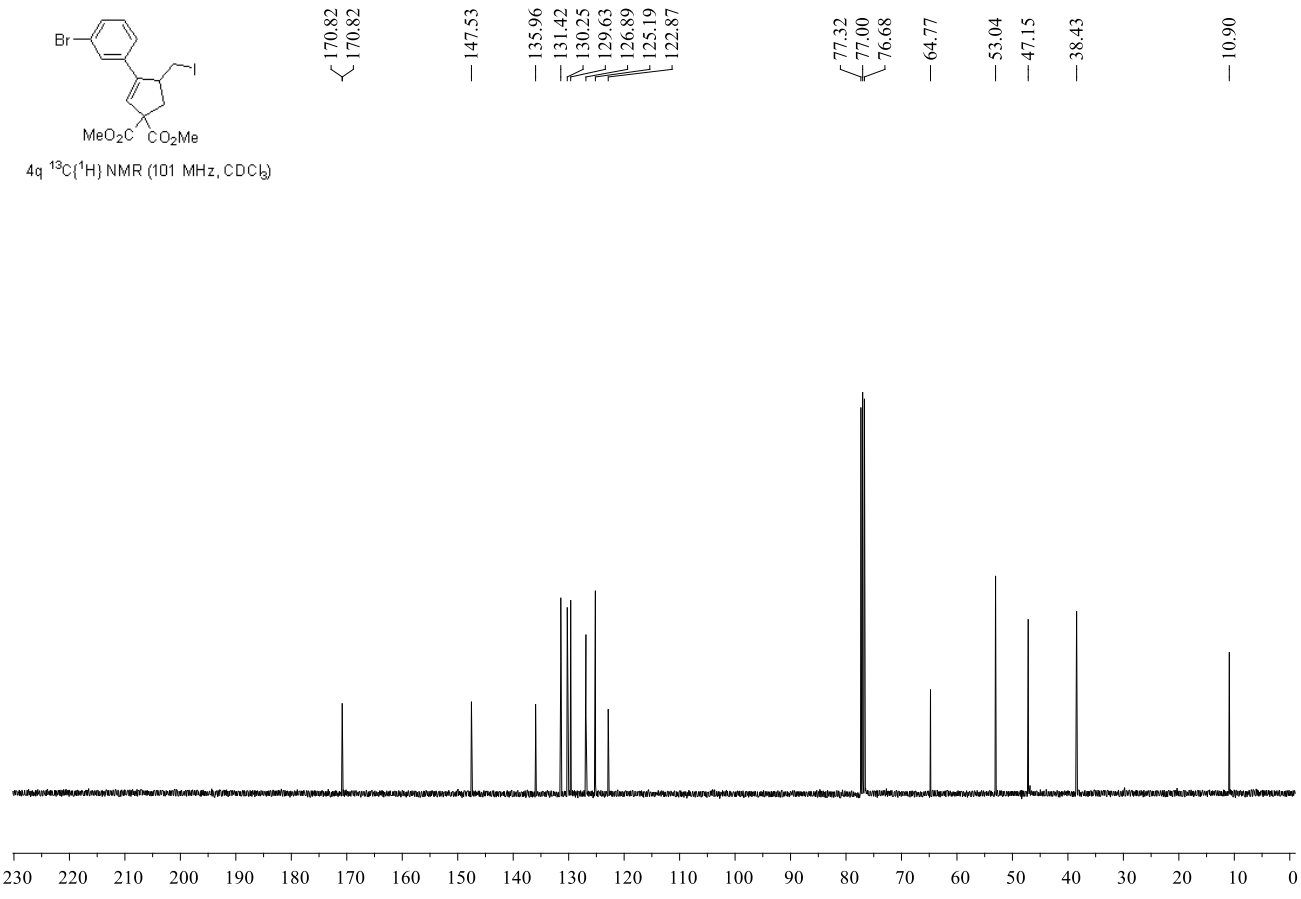




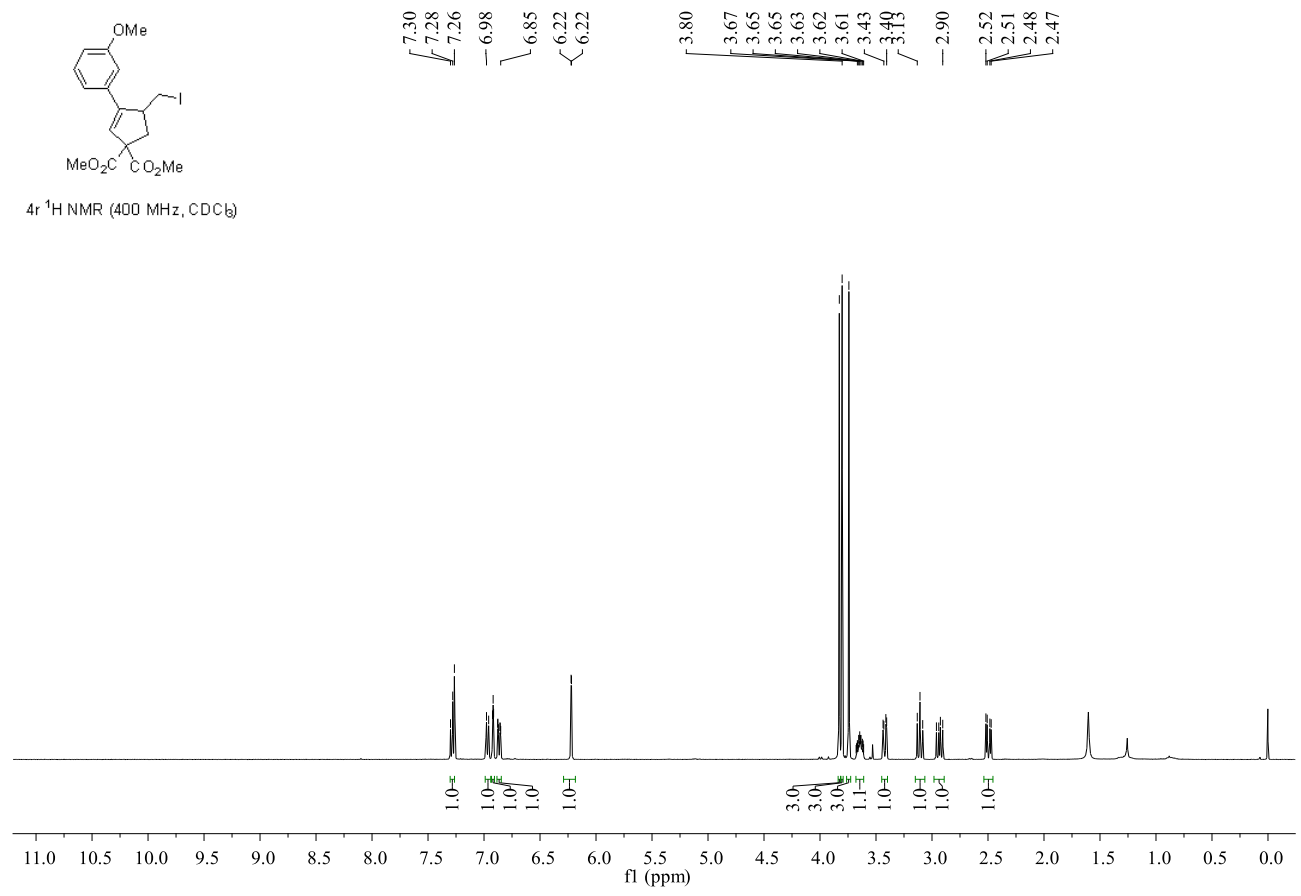
4p $^{19}F\{^{1}H\}$ NMR (376 MHz, $CDCl_3$)



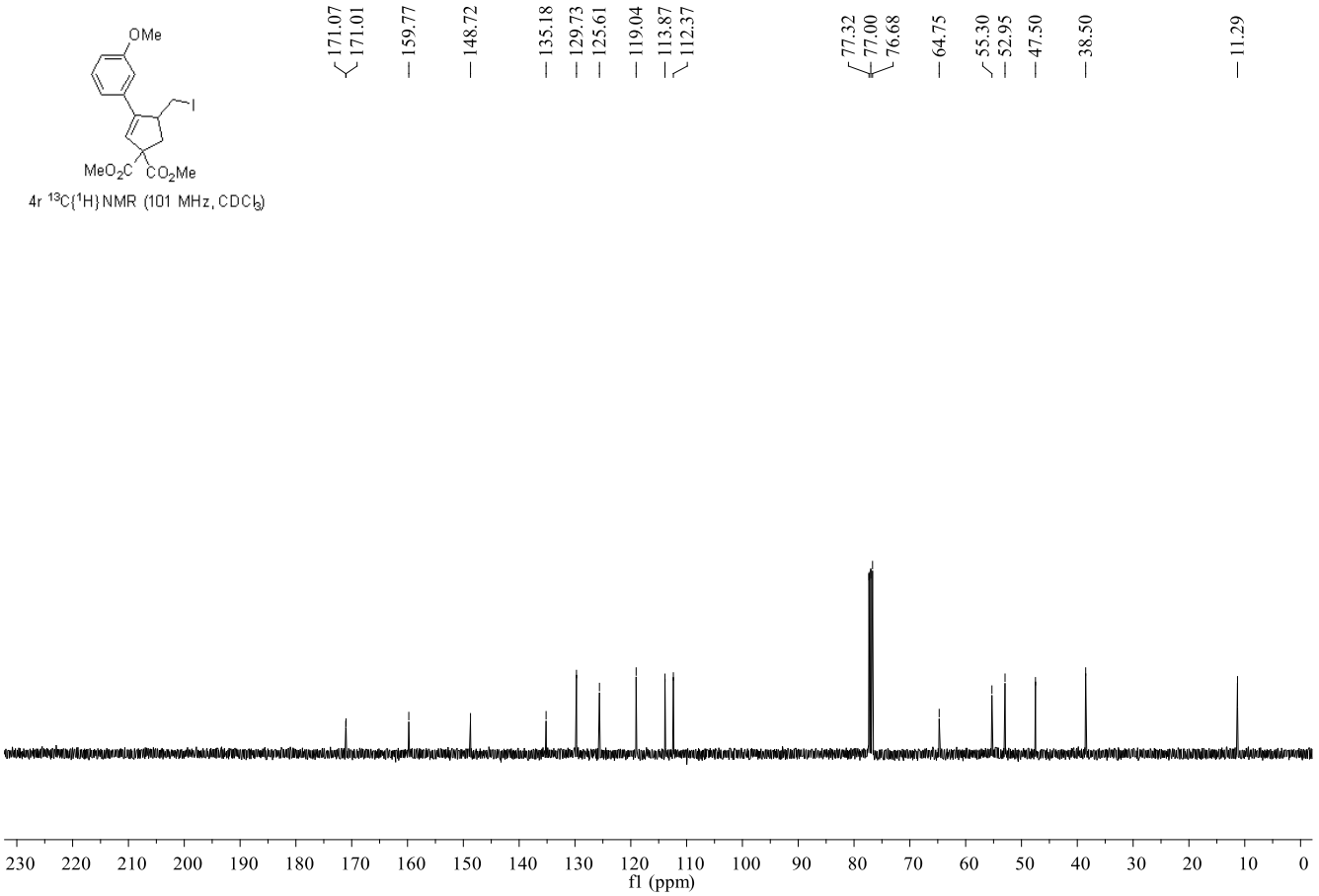
4q ^{1}H NMR (400 MHz, $CDCl_3$)



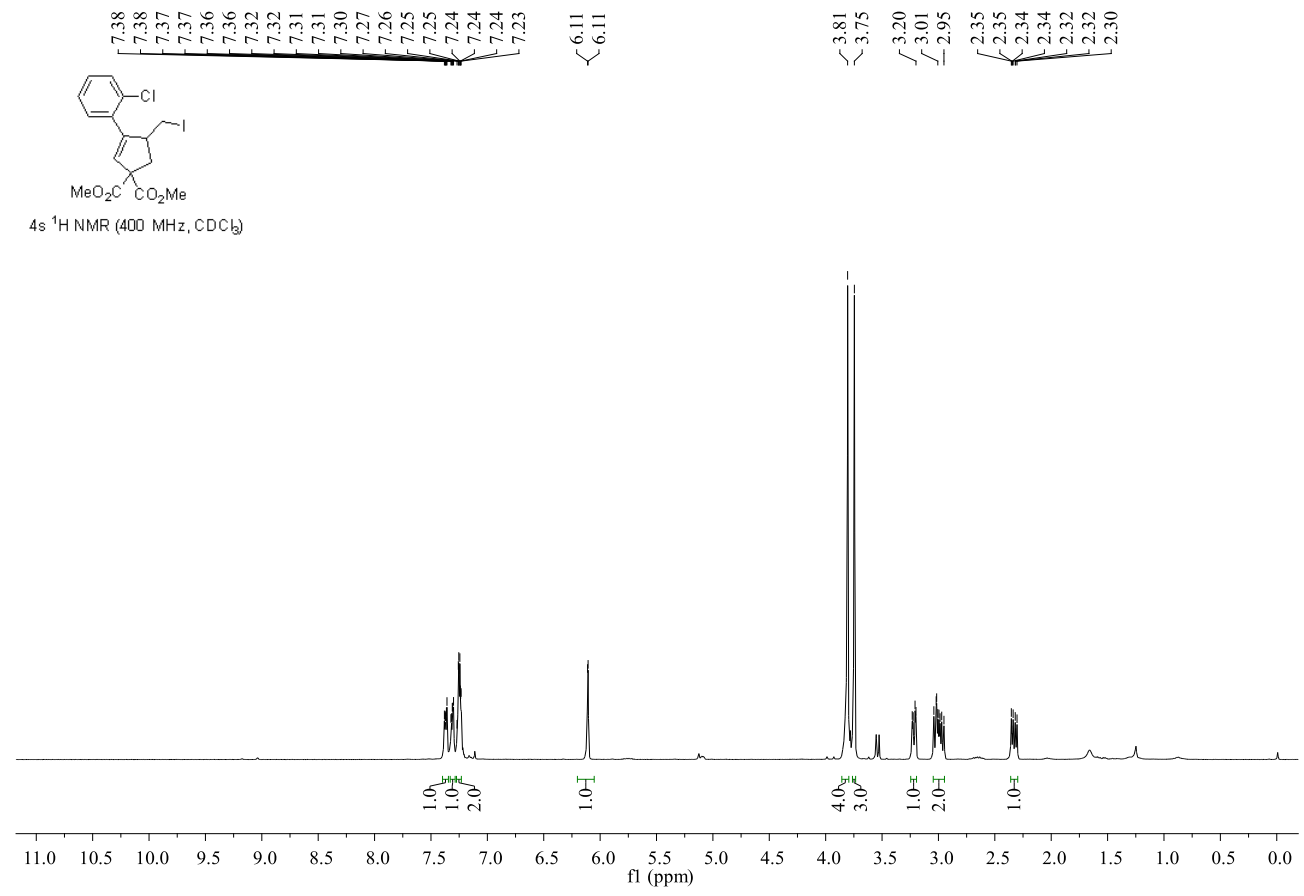
4q $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



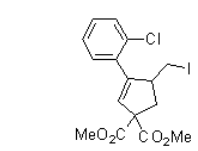
4r ^{1}H NMR (400 MHz, $CDCl_3$)



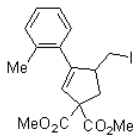
4r $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



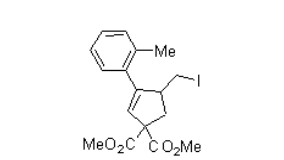
4s ^{1}H NMR (400 MHz, $CDCl_3$)



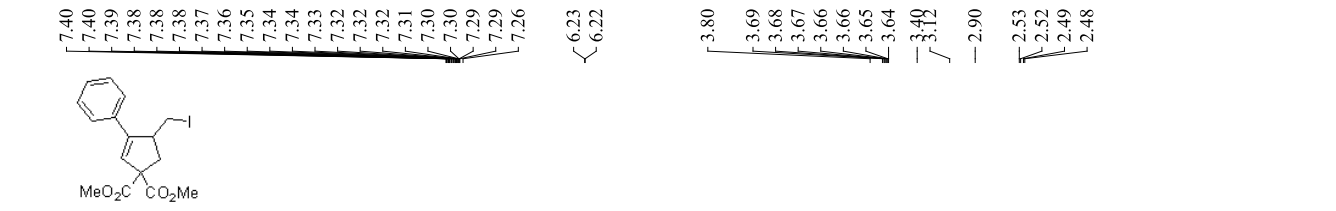
4s $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



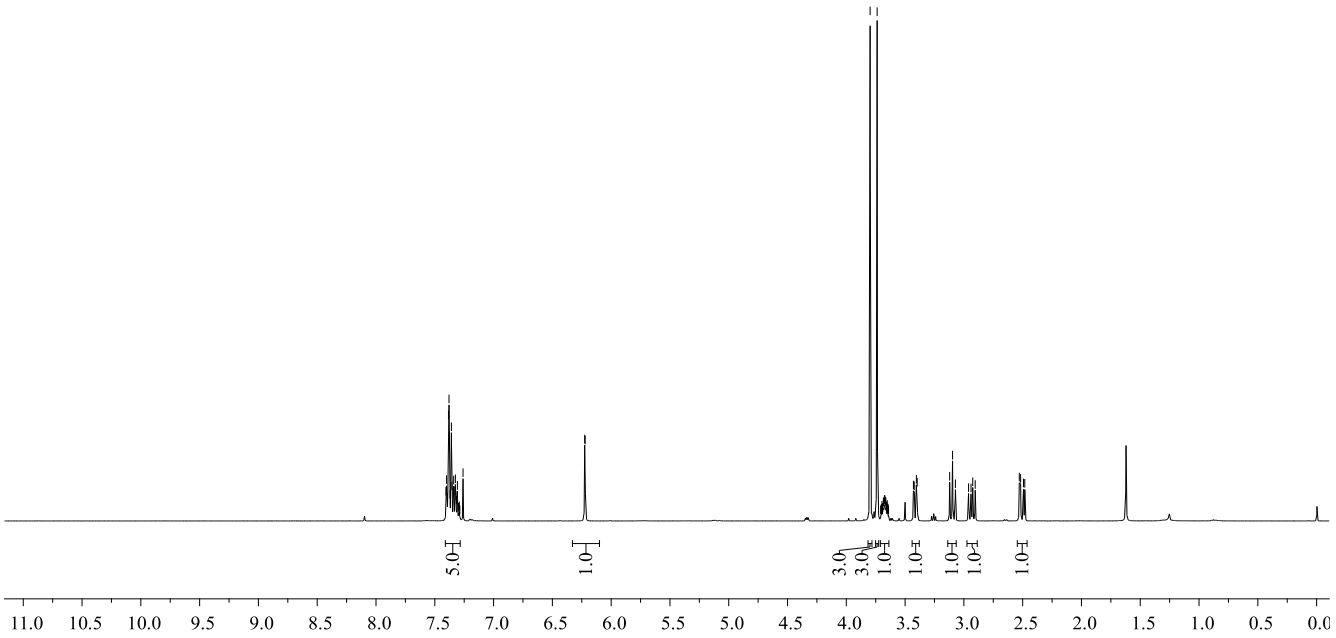
4t ^{1}H NMR (400 MHz, $CDCl_3$)

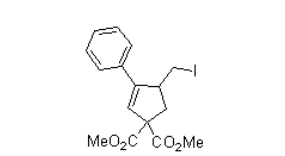


4t $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)

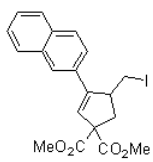


4u ^{1}H NMR (400 MHz, $CDCl_3$)

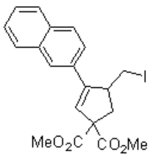




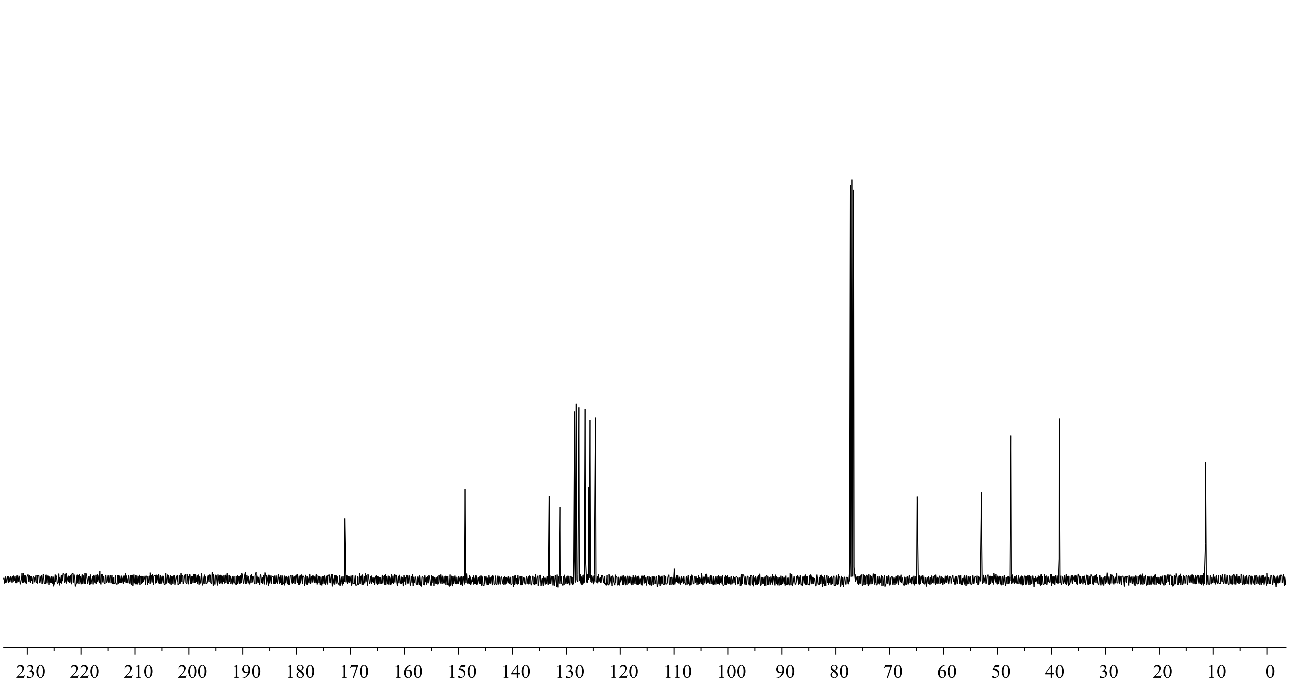
4u $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)

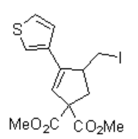


4v ^{1}H NMR (400 MHz, $CDCl_3$)

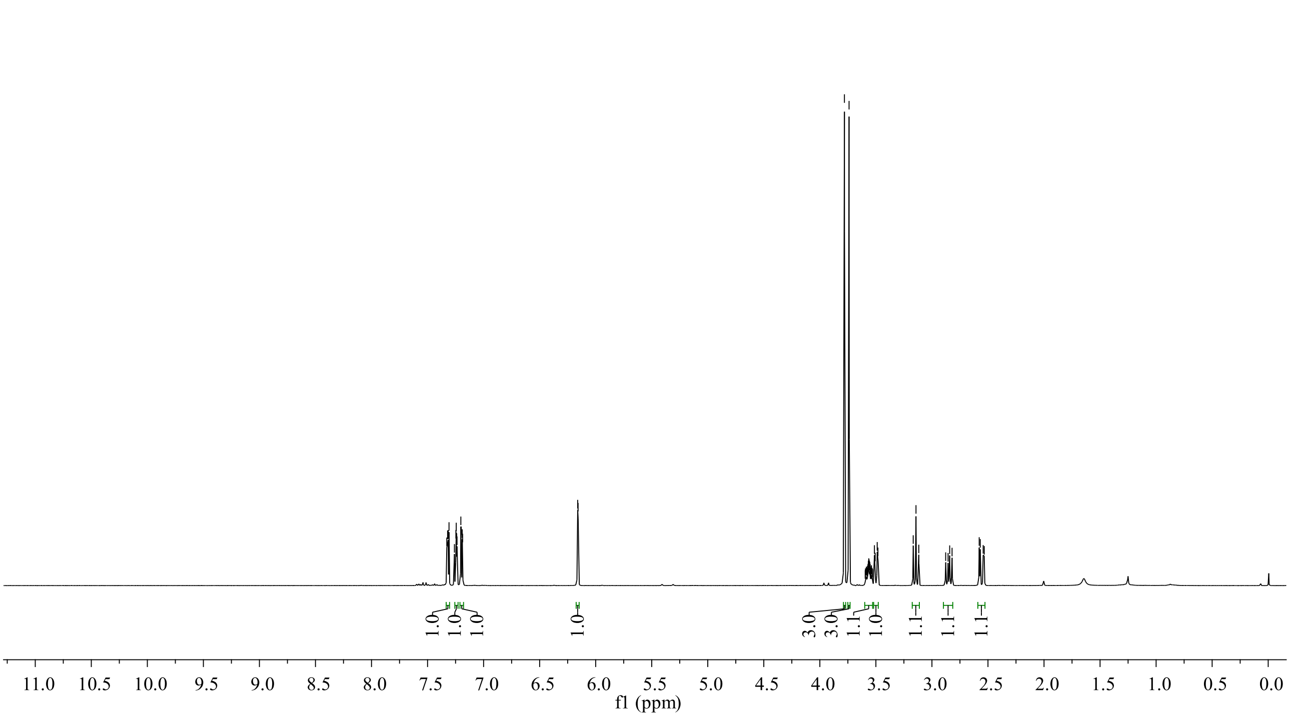


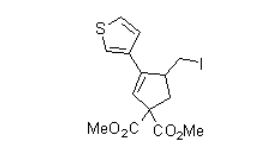
4v $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



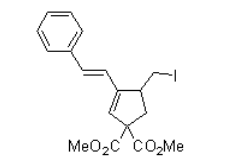


4w ^{1}H NMR (400 MHz, $CDCl_3$)

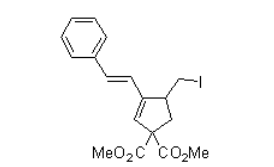




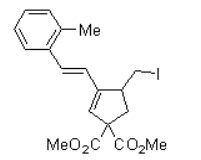
4w $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



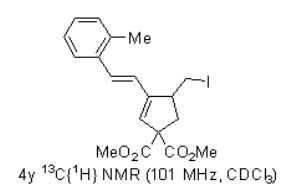
4x ^{1}H NMR (400 MHz, $CDCl_3$)



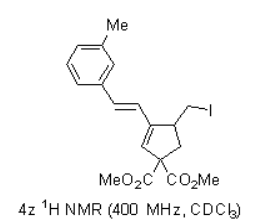
4x $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



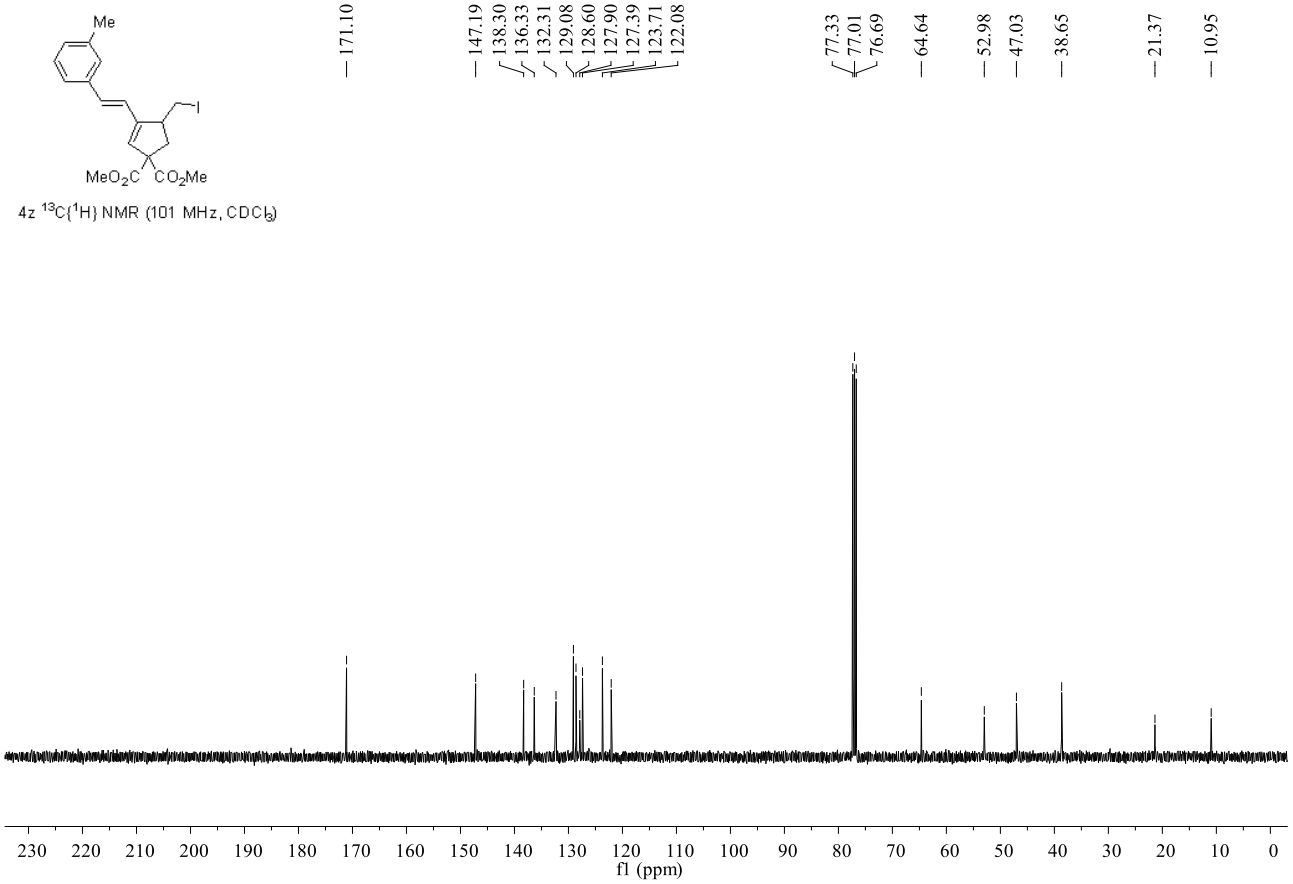
4y ^{1}H NMR (400 MHz, $CDCl_3$)



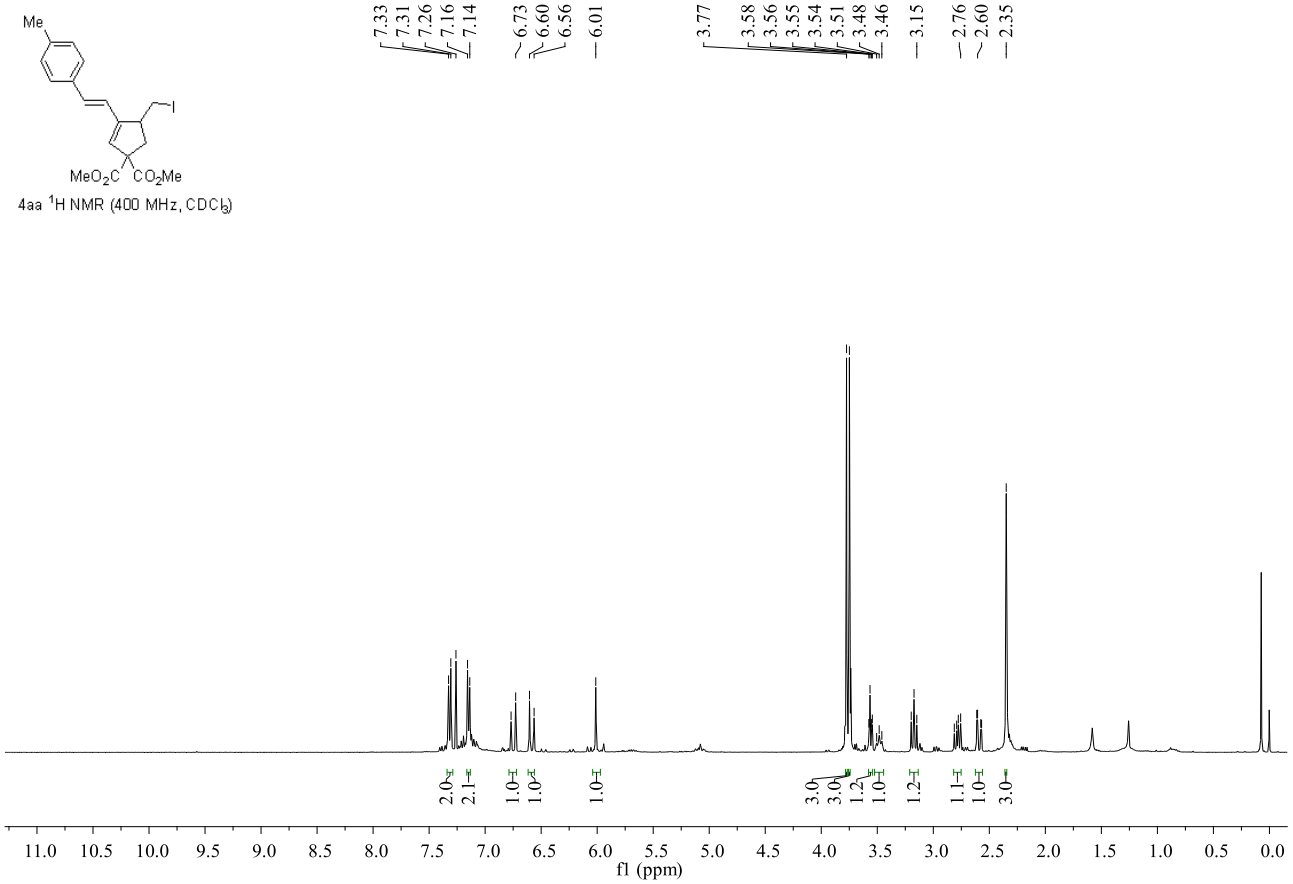
4y $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



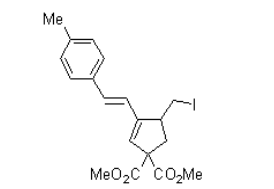
4z ^{1}H NMR (400 MHz, $CDCl_3$)



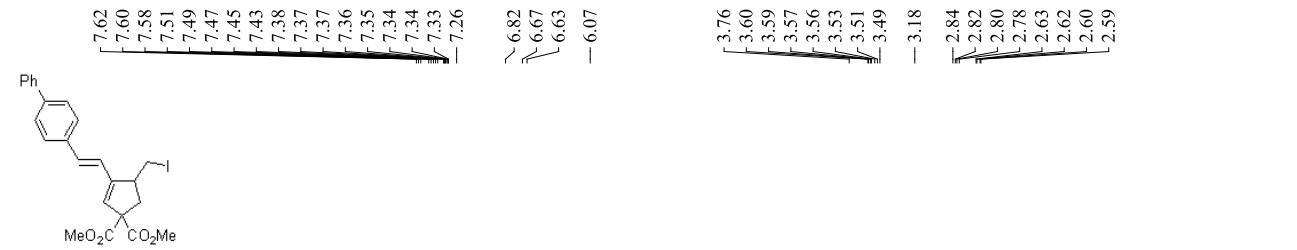
4z $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



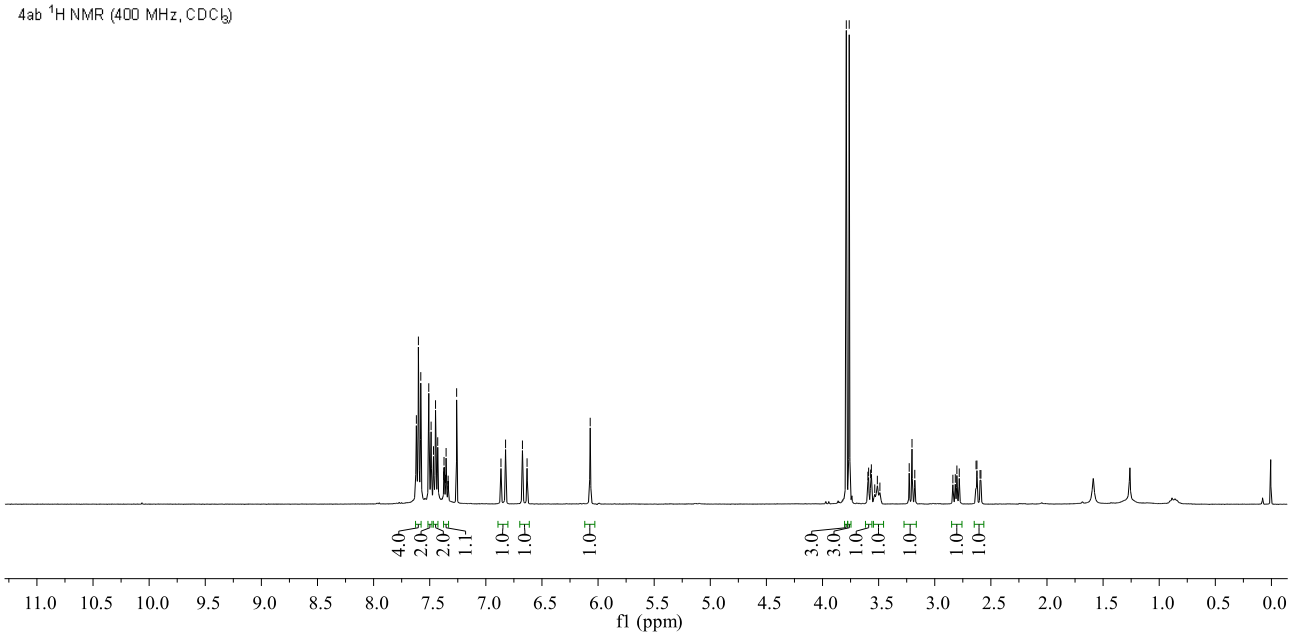
4aa ^{1}H NMR (400 MHz, $CDCl_3$)

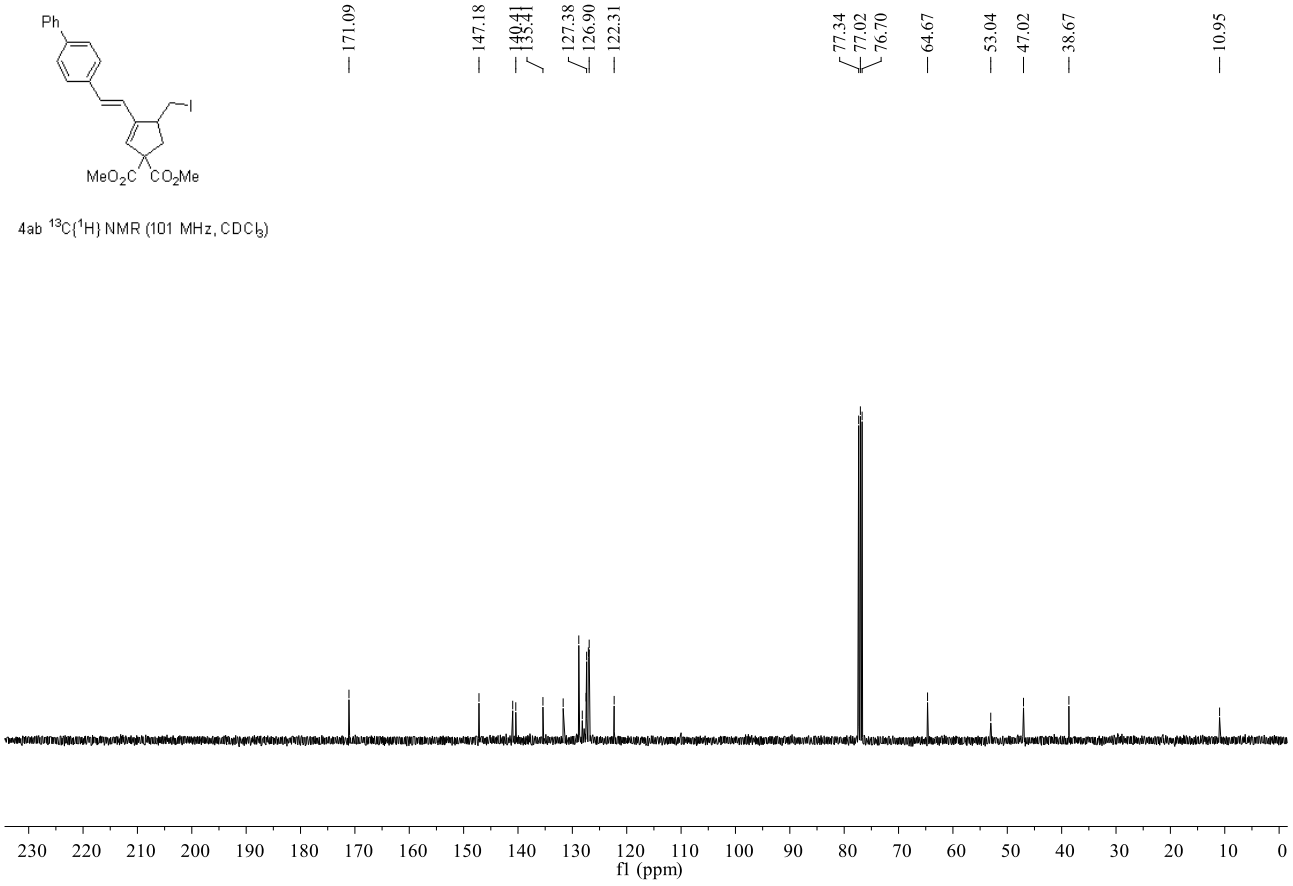


4aa $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)

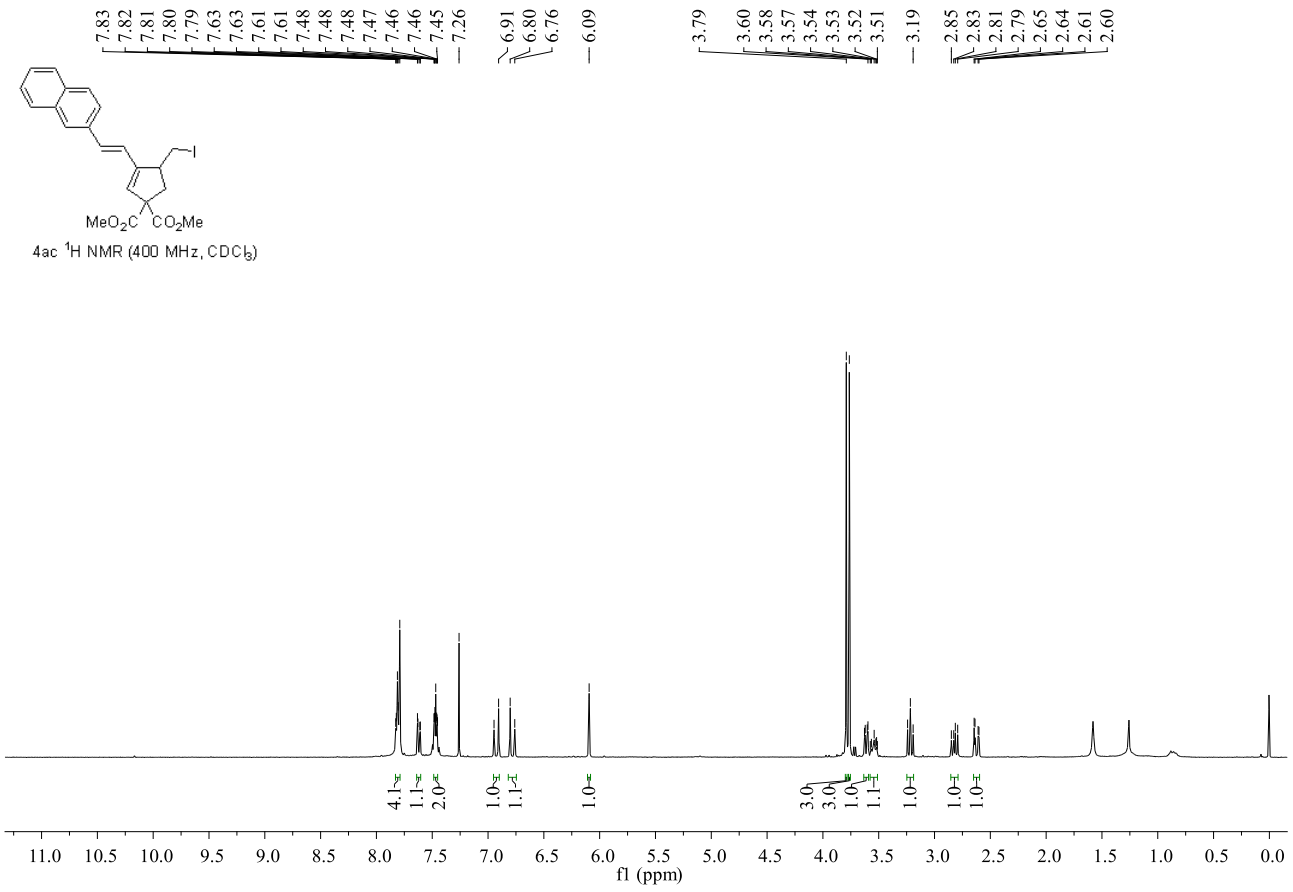


4ab ^{1}H NMR (400 MHz, $CDCl_3$)

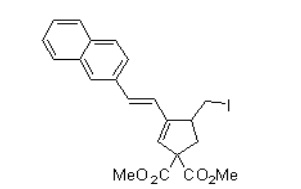




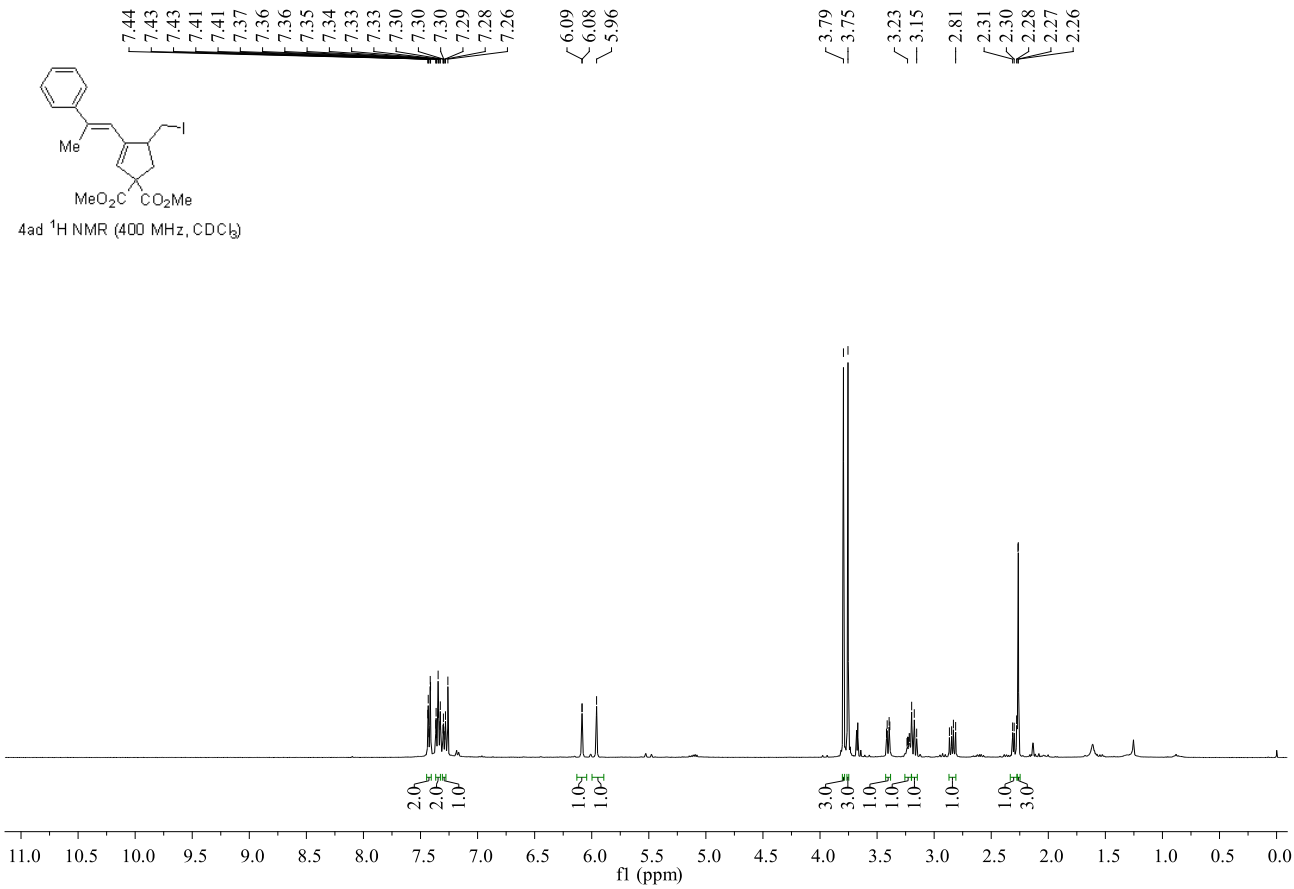
4ab $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



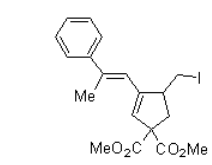
4ac ^{1}H NMR (400 MHz, $CDCl_3$)



4ac $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)

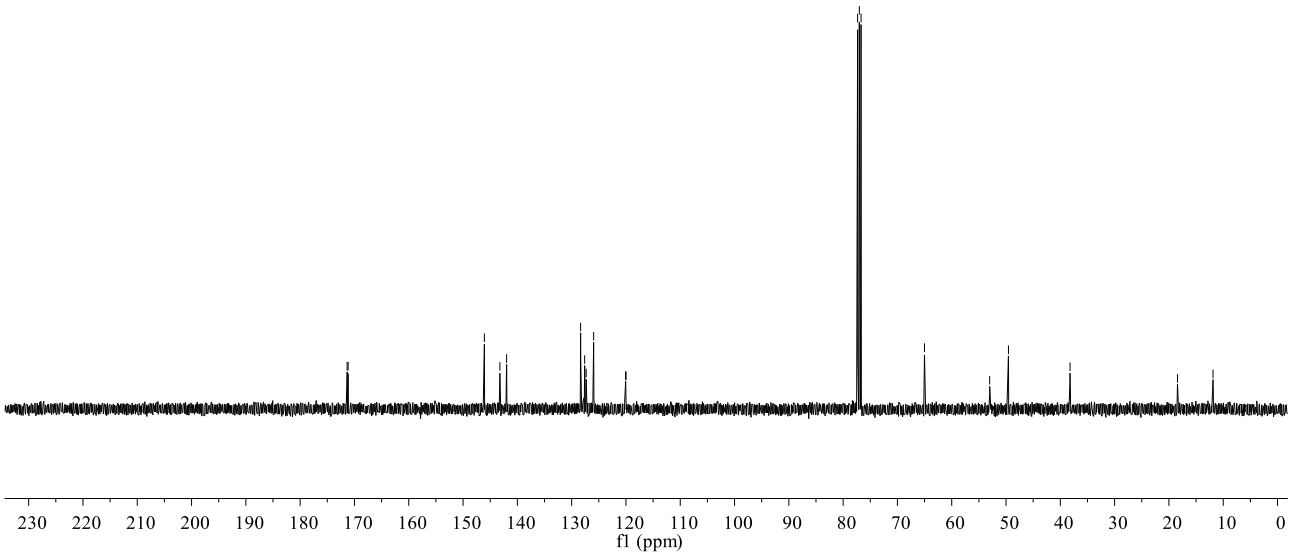


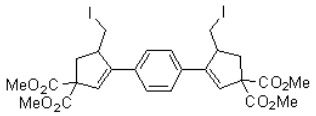
4ad ^{1}H NMR (400 MHz, $CDCl_3$)



4ad $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)

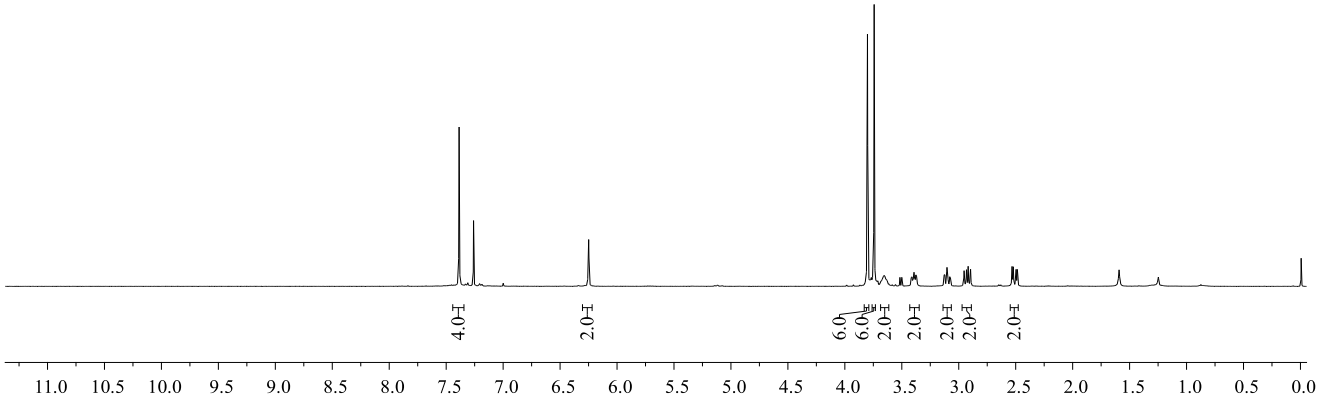
171.35
171.17
146.07
143.21
141.98
128.35
127.32
125.95
120.05
119.99
77.34
77.02
76.70
65.01
53.01
49.55
38.21
18.42
11.88

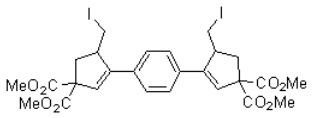




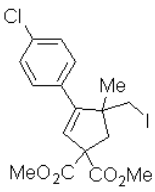
4ae ^{1}H NMR (400 MHz, $CDCl_3$)

7.39
7.26
6.25
6.25
3.74
3.66
3.65
3.64
3.63
3.42
3.40
3.39
3.37
3.08
2.90
2.53
2.52
2.50
2.49

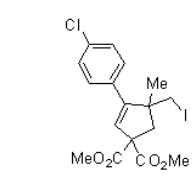




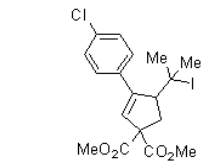
4ae $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



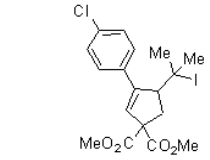
4af ^{1}H NMR (400 MHz, $CDCl_3$)



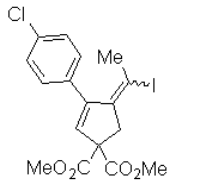
4af $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



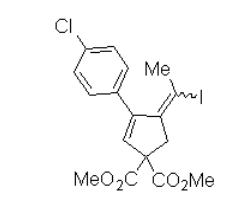
4ag ^{1}H NMR (400 MHz, $CDCl_3$)



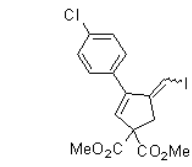
4ag $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



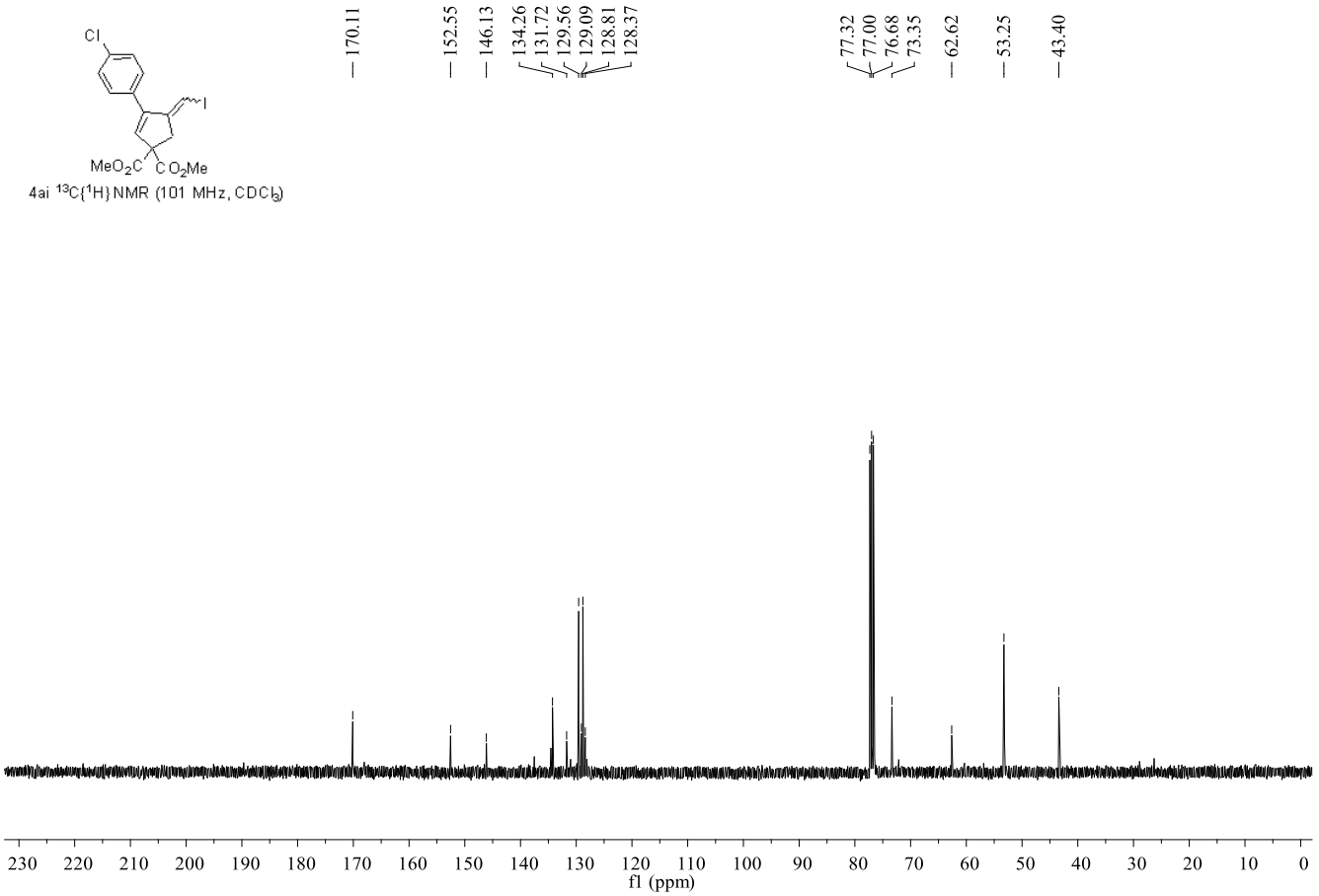
4ah ^{1}H NMR (400 MHz, $CDCl_3$)



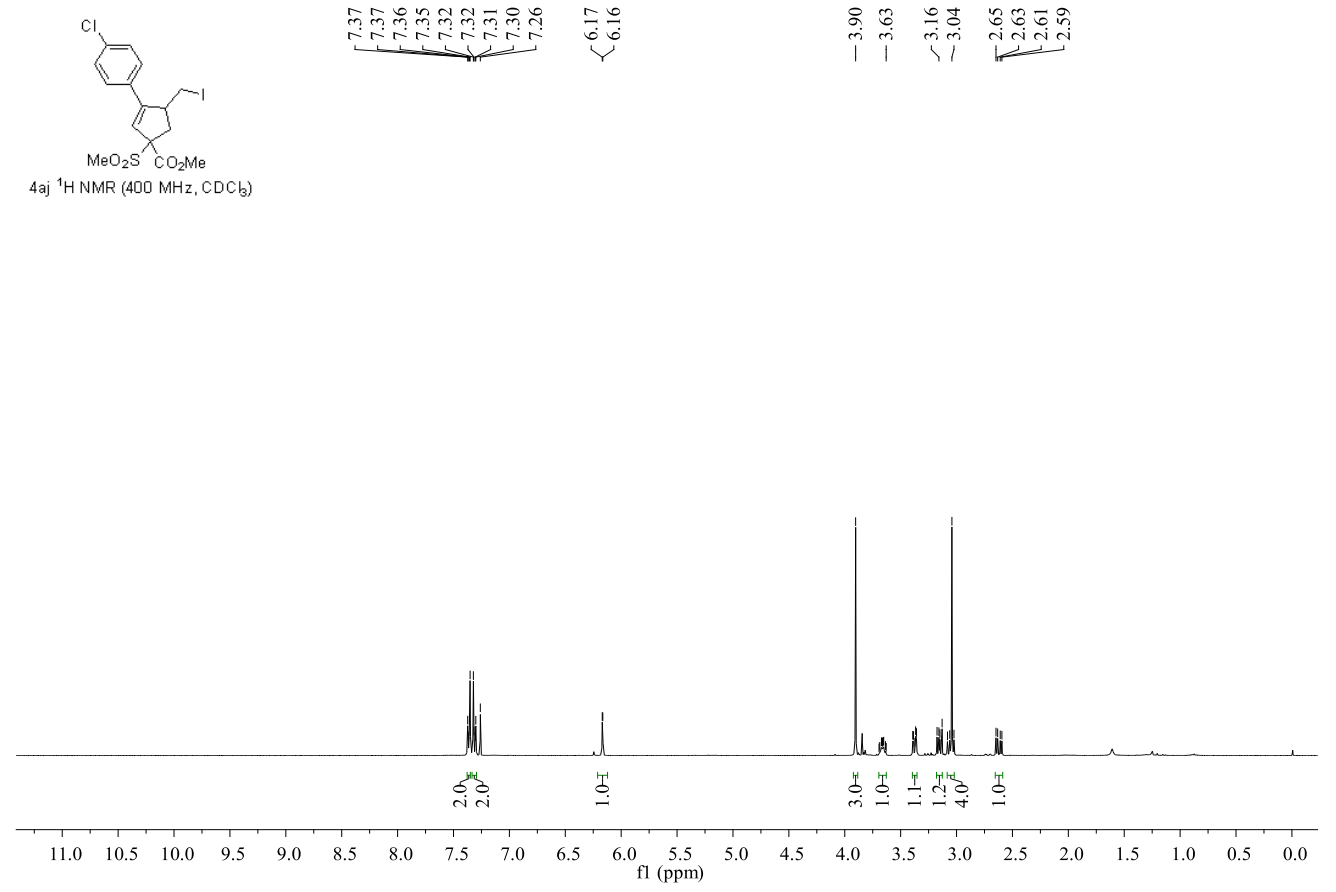
4ah $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



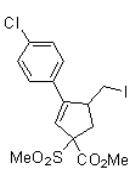
4ai ^{1}H NMR (400 MHz, $CDCl_3$)



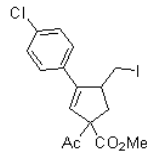
4ai $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



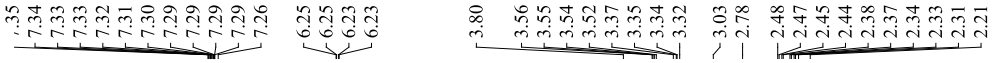
4aj ^{1}H NMR (400 MHz, $CDCl_3$)

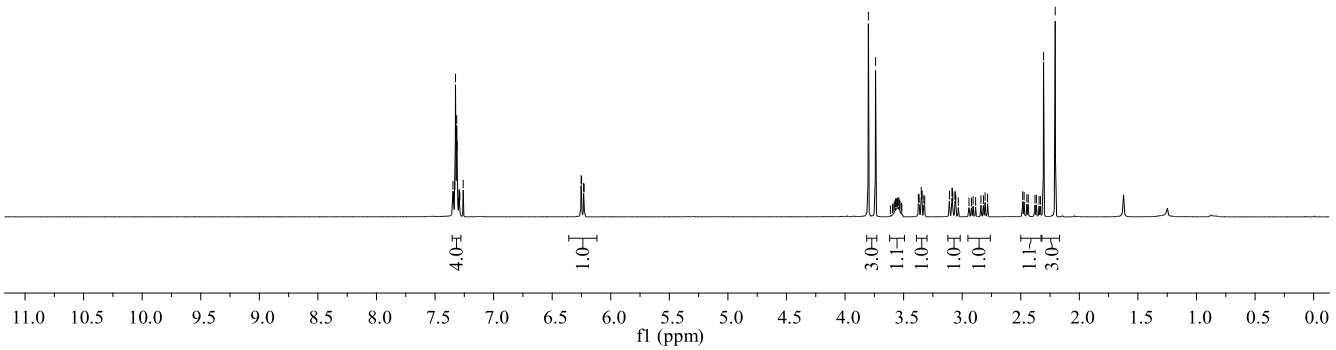


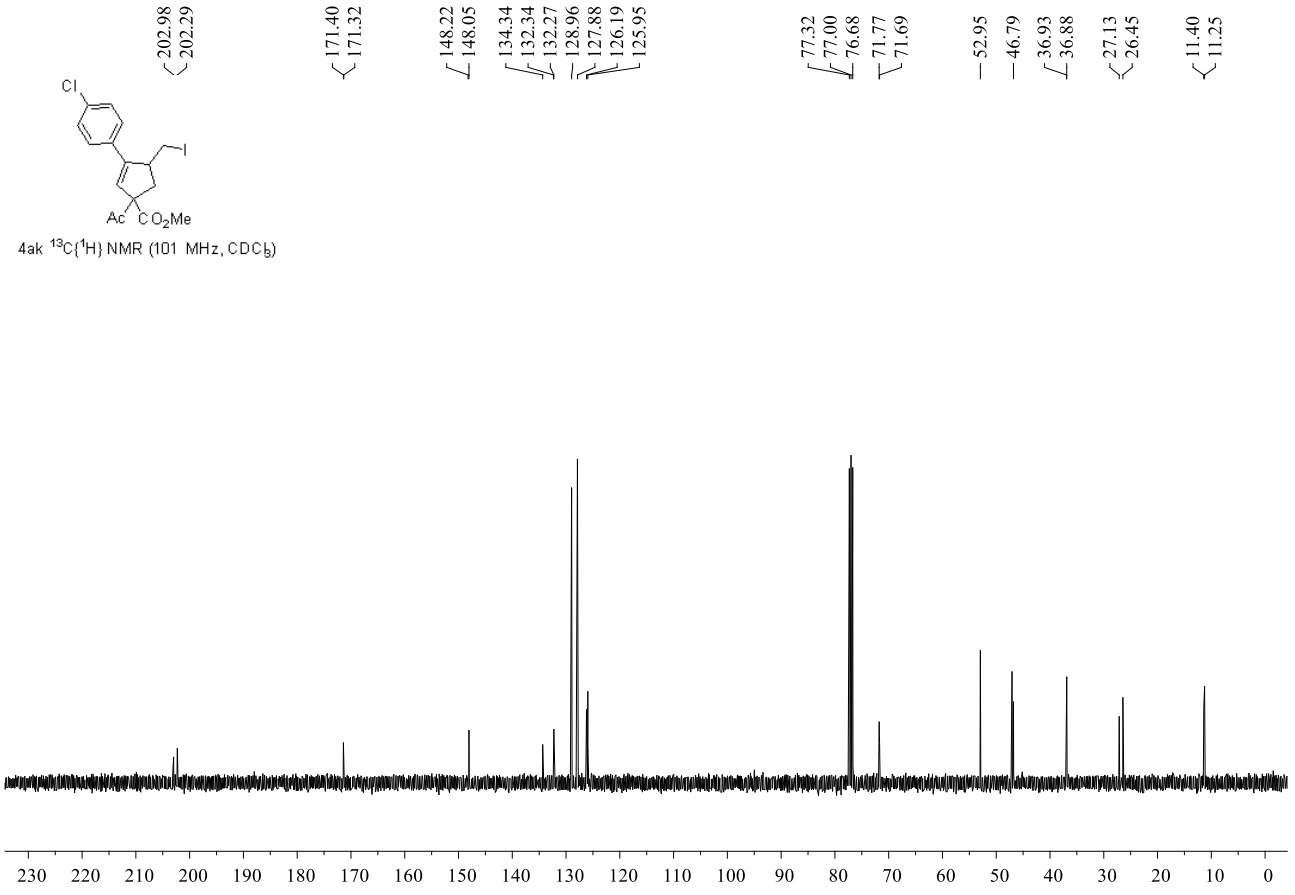
4aj $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



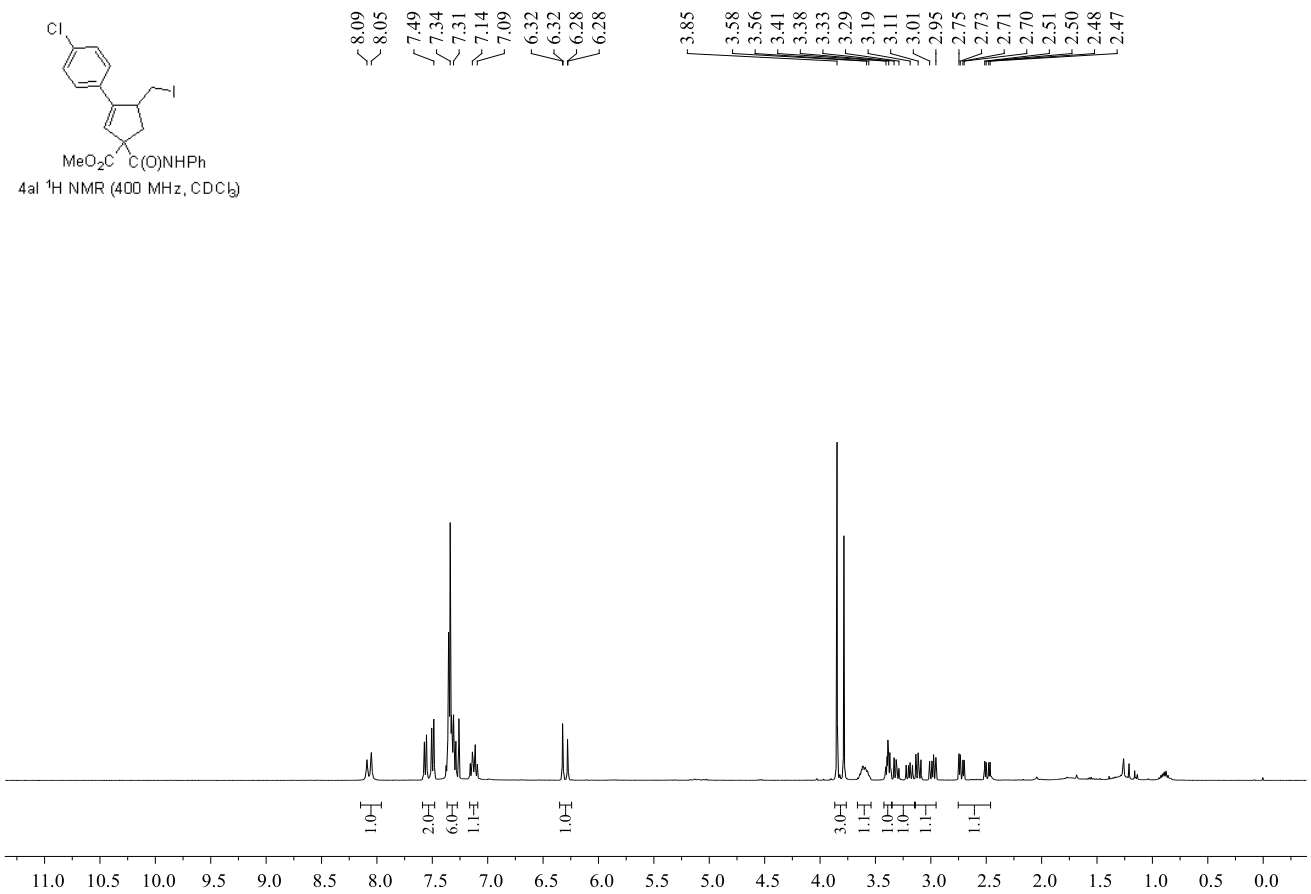
4ak ^{1}H NMR (400 MHz, $CDCl_3$)



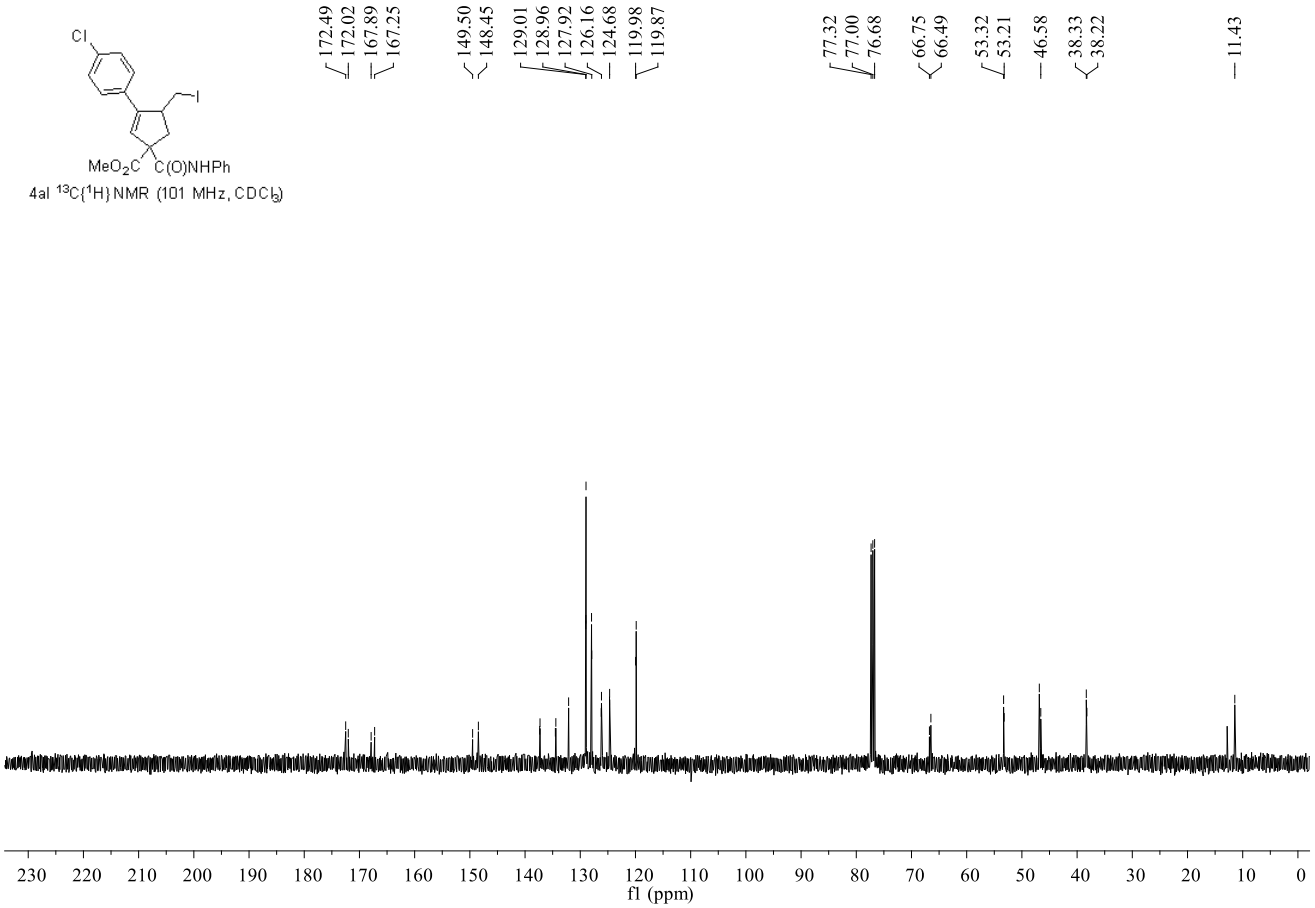




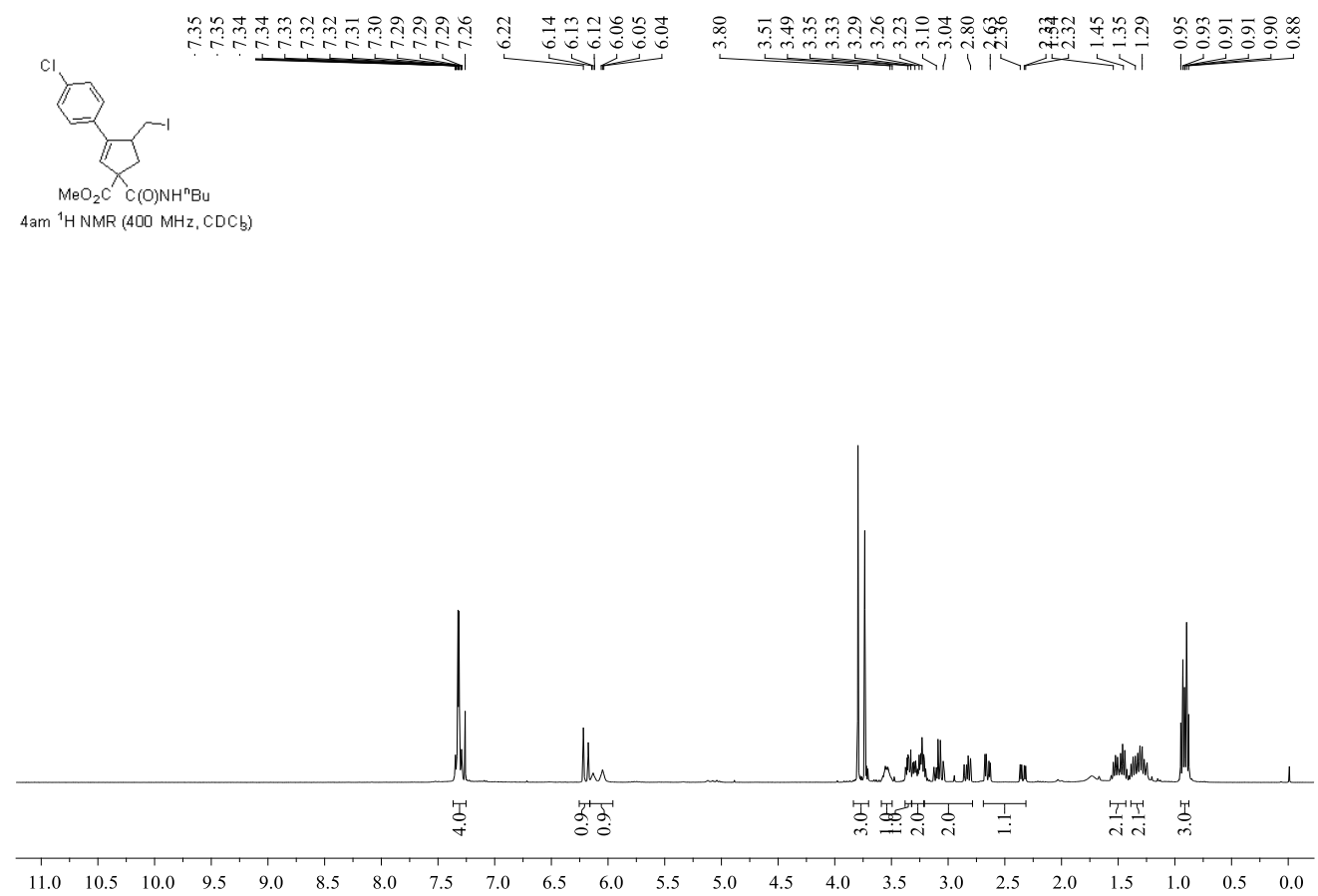
4ak $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



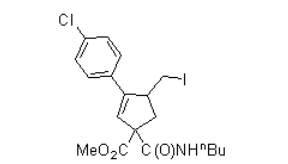
4al ^{1}H NMR (400 MHz, $CDCl_3$)



4al $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)

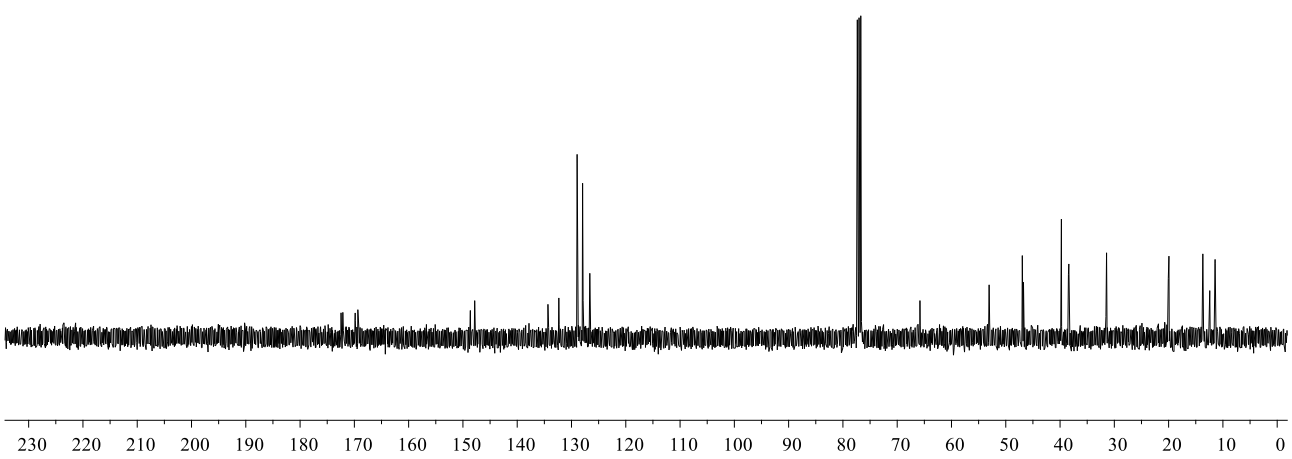


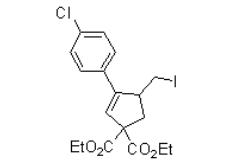
4am ^{1}H NMR (400 MHz, $CDCl_3$)



4am $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$)

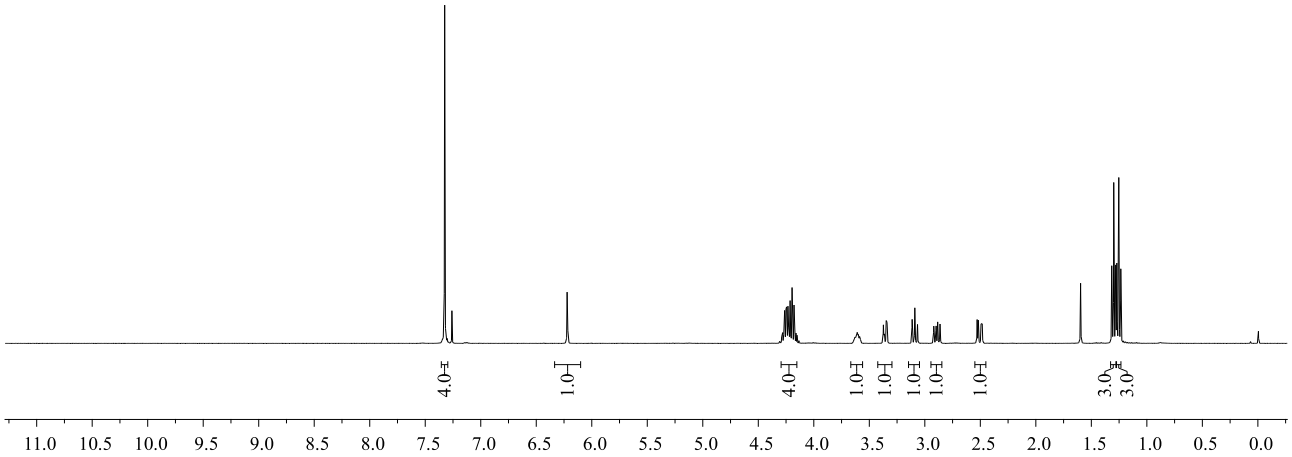
172.45
172.12
169.85
169.36
148.63
147.83
134.34
132.33
128.97
127.96
127.88
126.63
77.34
77.03
76.71
65.81
53.07
46.95
38.38
31.43
19.94
13.70
11.44

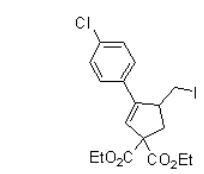




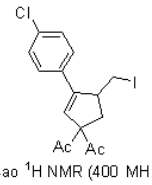
4an ^1H NMR (400 MHz, $CDCl_3$)

7.32
7.26
6.22
6.22
4.26
4.24
4.23
4.22
4.20
4.17
4.15
3.61
3.58
3.34
3.31
2.86
2.52
2.48
1.32
1.30
1.28
1.27
1.25
1.23

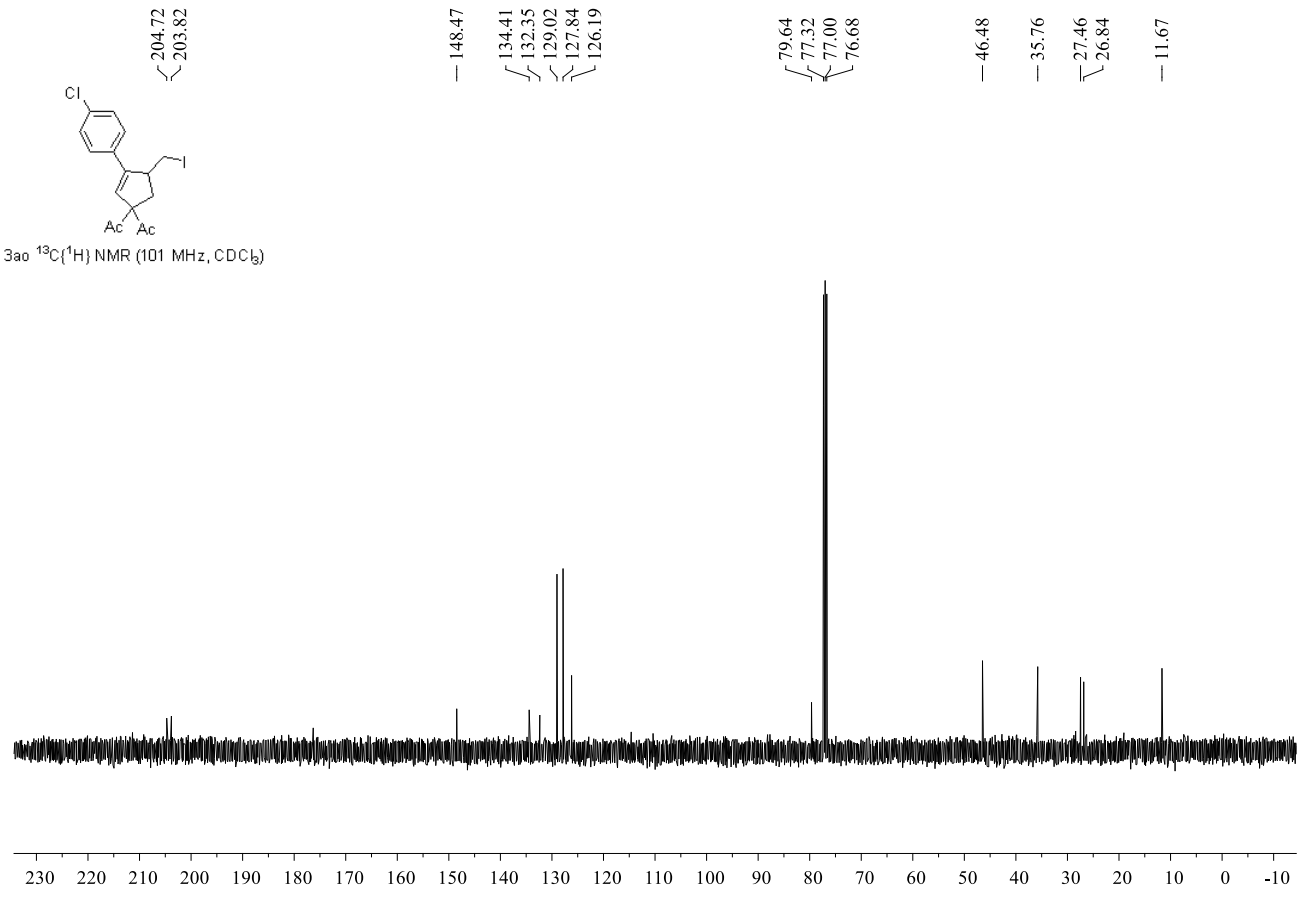




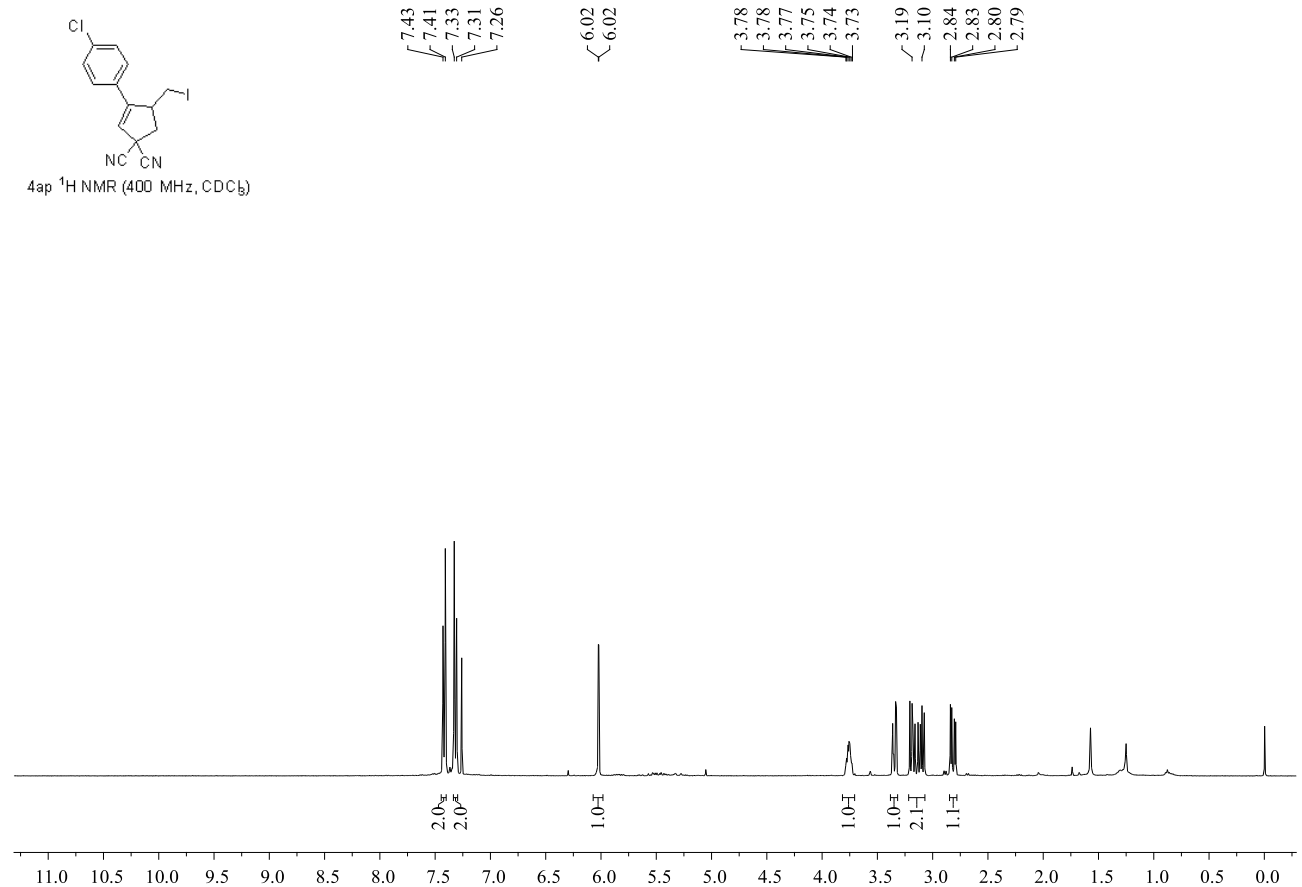
4an $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



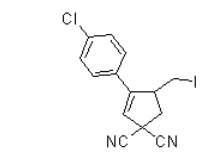
4ao ^{1}H NMR (400 MHz, $CDCl_3$)



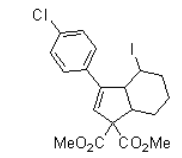
3ao $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



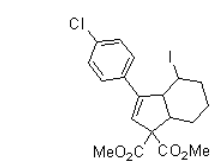
4ap ^{1}H NMR (400 MHz, $CDCl_3$)



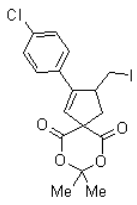
4ap $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



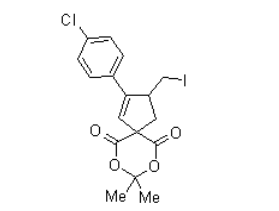
4aq ^{1}H NMR (400 MHz, $CDCl_3$)



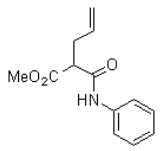
4aq $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



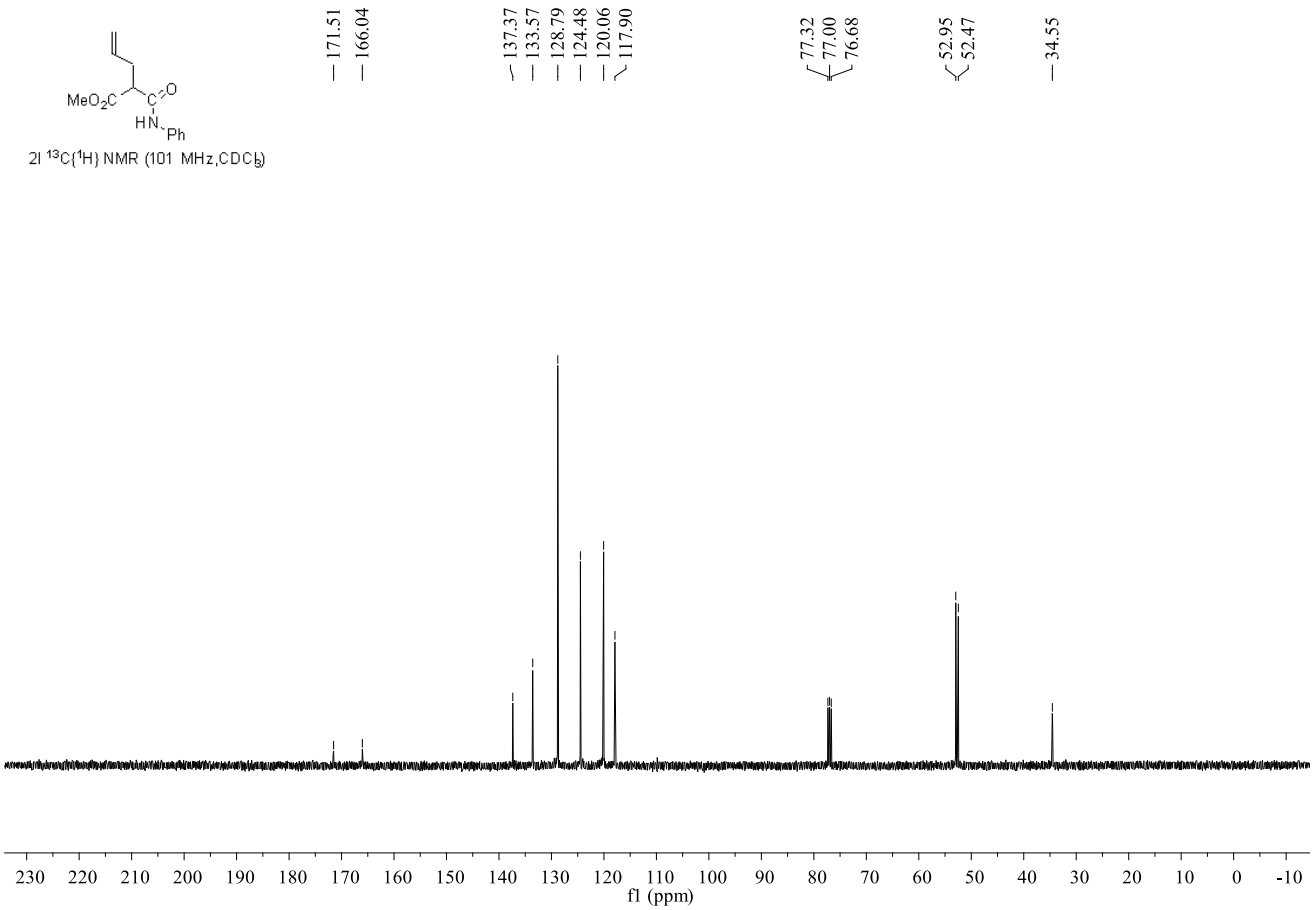
4ar ^{1}H NMR (400 MHz, $CDCl_3$)

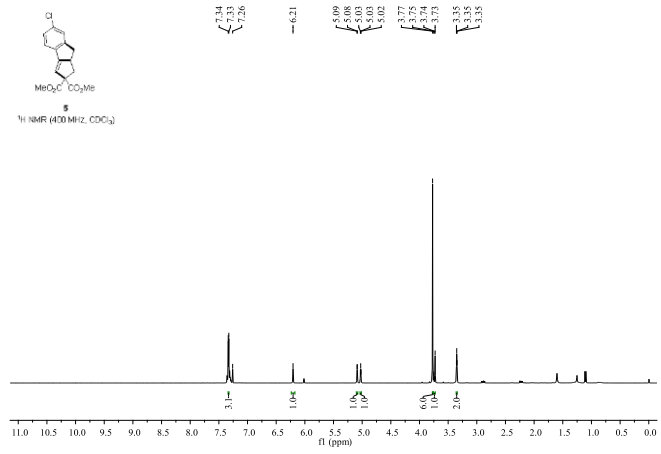


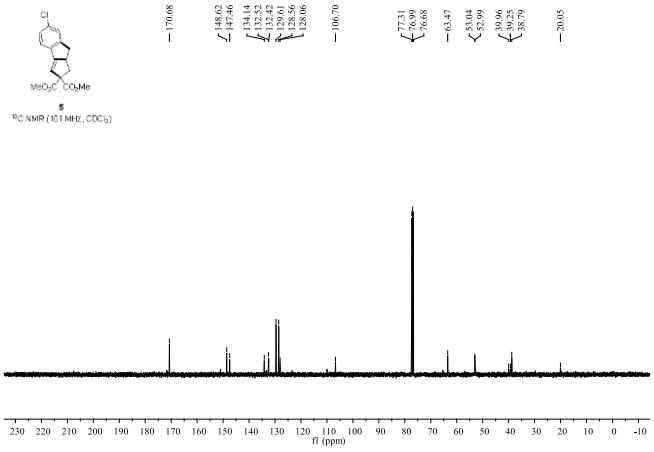
4ar $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)

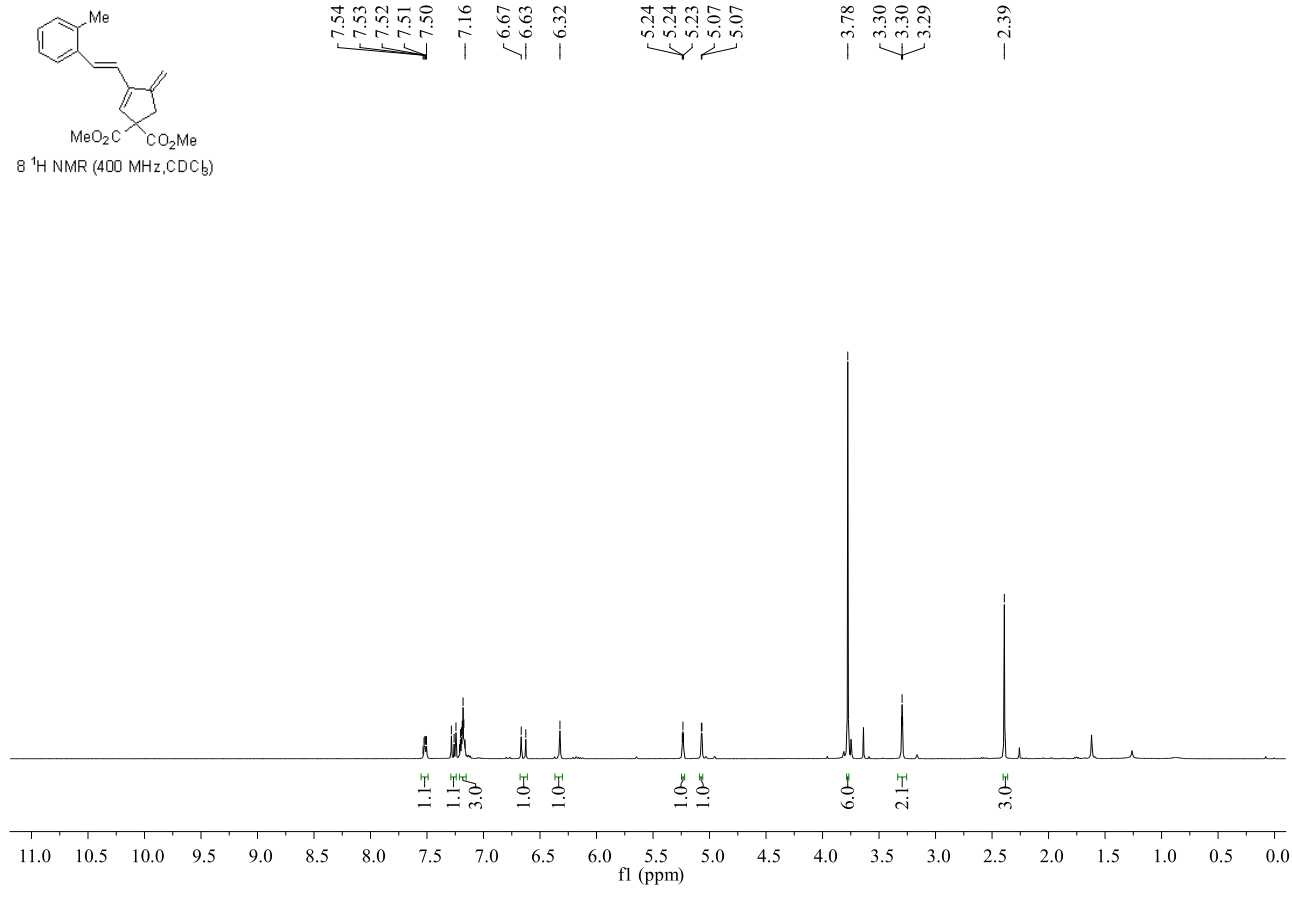


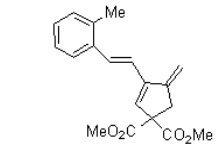
2l ^{1}H NMR (400 MHz, $CDCl_3$)


171.51
166.04
137.37
133.57
128.79
124.48
120.06
117.90
77.32
77.00
76.68
52.95
52.47
34.55
MeO2C
O
HN
Ph
2l 13C{1H} NMR (101 MHz,CDCl3)
fl (ppm)

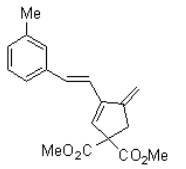

7.34
7.33
7.26
6.21
5.09
5.08
5.03
5.03
5.02
3.77
3.75
3.74
3.73
3.35
3.35
3.35
MeO2C
CO2Me
5
1H NMR (400 MHz, CDCl3)
fl (ppm)


Cl
MeO2C CO2Me
5
13C NMR (101 MHz, CDCl3)
170.68
148.62
147.46
134.14
132.52
132.42
129.61
128.56
126.06
106.70
77.31
76.99
76.68
63.47
53.04
52.99
39.96
39.25
38.79
20.05
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

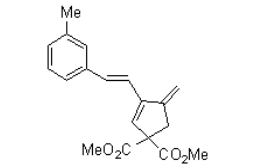

Me
MeO2C CO2Me
8 1H NMR (400 MHz,CDCl3)
7.54
7.53
7.52
7.51
7.50
7.16
6.67
6.63
6.32
5.24
5.24
5.23
5.07
5.07
3.78
3.30
3.30
3.29
2.39
1.1
1.1
3.0
1.0
1.0
1.0
1.0
6.0
2.1
3.0
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)



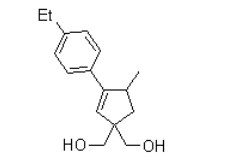
8 $^{13}C\{^{1}H\}$ NMR (101 MHz,$CDCl_3$)



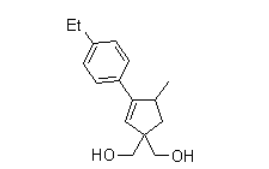
9 ^{1}H NMR (400 MHz,$CDCl_3$)



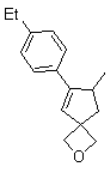
9 $^{13}C\{^{1}H\}$ NMR (101 MHz,$CDCl_3$)



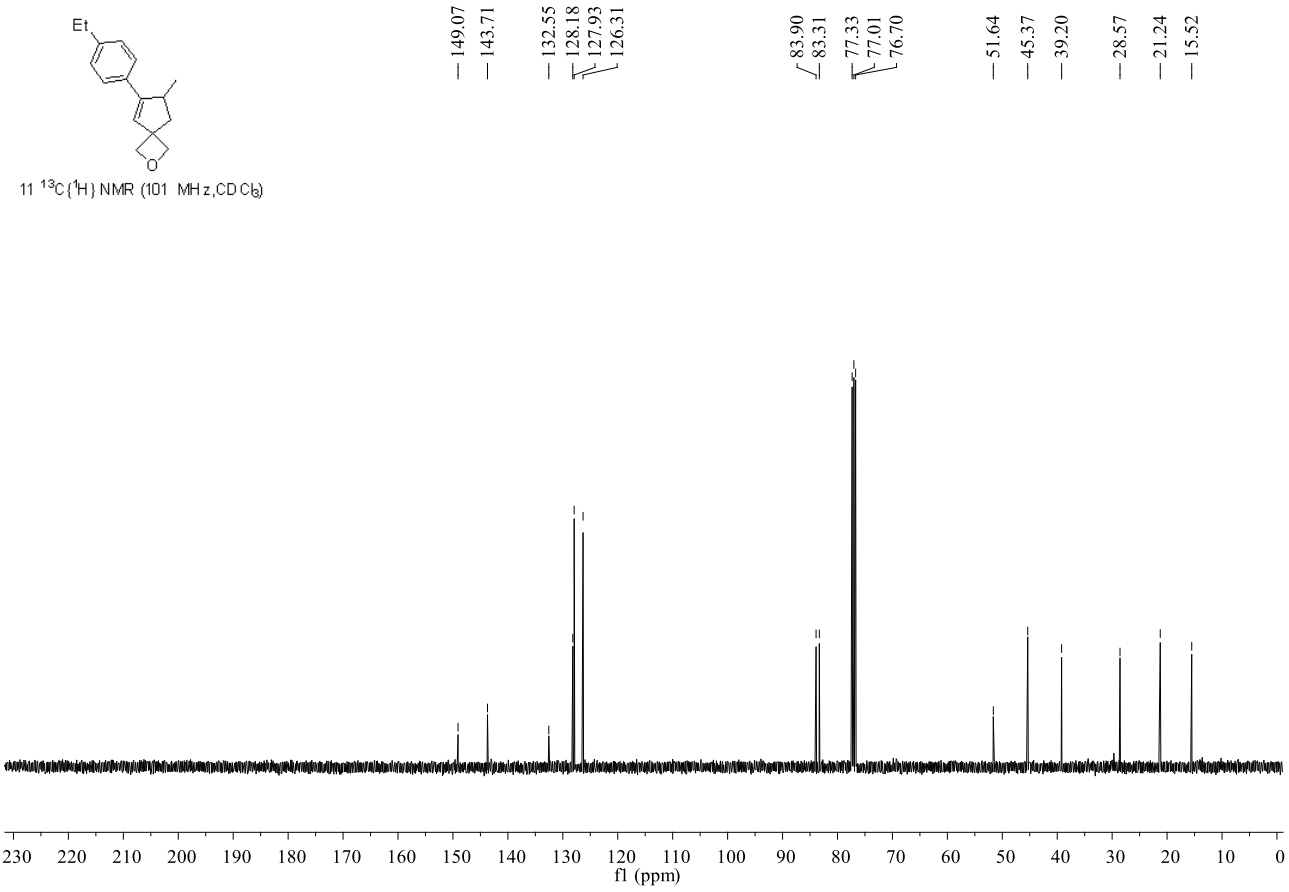
10 ^{1}H NMR (400 MHz,$CDCl_3$)



10 $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)



11 ^{1}H NMR (400 MHz, $CDCl_3$)



11 $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$)