Supplementary Information

Synthesis of Phthalides utilizing one-pot intramolecular domino protocol

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A. General remarks

All reactions were carried out using oven-dried glassware. Commercial grade reagents were used without further purification. Solvents were dried and distilled following usual protocols prior to use. All yields refer to isolated yields after column purification. Column chromatography was carried out using Silica gel (60-120 mesh) purchased from Rankem, India. TLC was performed on aluminium-backed plates coated with Silica gel 60 with F254 indicator (Merck).

The ^{1}H NMR spectra were measured with Bruker-200 (200 MHz) or Bruker-400 (400 MHz) and ^{13}C NMR spectra were measured with Bruker-200 (50 MHz) or Bruker-400 (100 MHz) using $CDCl_3$. Coupling constants in ^{1}H NMR are in Hz. EIMS (70 eV) spectra were taken using a VG Autospec mass spectrometer. Melting points were measured in Toshniwal (India) melting point apparatus.

General procedure for the synthesis of *o*-alkenylbenzoic acid (2):

To a solution of *o*-alkenylbenzaldehyde (1 mmol) in acetonitrile at 0 °C, NaH_2PO_4 (1 mmol) dissolved in 1 mL water was added. To it, H_2O_2 (1 mol%) was added. Then $NaClO_2$ (1.4 mmol) dissolved in minimum amount of water, was added drop wise and stirred for 1-2 h. Upon completion of the reaction, excess saturated aq Na_2CO_3 solution was added, washed with ether, aq solution was acidified with conc. HCl and then extracted with ether (3 X 25 mL). The combined organic layer was washed with brine, dried over Na_2SO_4 and then evaporated to give pure *o*-alkenylbenzoic acid.

General Procedure for the synthesis of 3-alkylidine phthalides (3a-f):

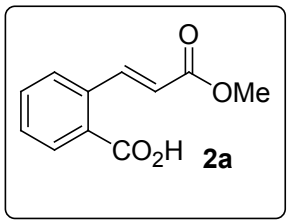
To a solution of *o*-alkenylbenzoic acid (1 mmol) in dry DCM (5 mL), *m*CPBA (1 mmol) and *p*-TsOH (1 mmol) was added and the mixture was stirred at rt for 5-6 h. Then the reaction mixture was quenched with saturated aq $NaHCO_3$ solution and extracted with DCM. The organic solvent was washed with aq $NaHCO_3$ and brine solution, and then dried over anhy Na_2SO_4. The solvent

was evaporated and then the product was purified by column chromatography using ethyl acetate/ petroleum ether as eluent.

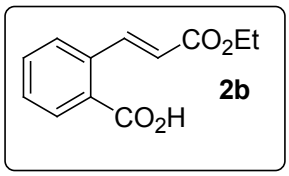
General procedure for the synthesis of lactones (5):

To a solution of 3-(2-formyl-cycloalkenyl)-acrylic esters **4.1** or 3-(2-formyl cycloalkenyl)-acrylonitriles **4.2** (1 mmol) in acetonitrile at 0 °C, NaH_2PO_4 (1 mmol) dissolved in 1 mL water was added. To it H_2O_2 (1 mol%) was added. Then $NaClO_2$ (1.4 mmol) dissolved in minimum amount of water, was added drop wise and stirred for 1-2 h. Upon completion of the reaction the mixture was diluted with EtOAc, washed with saturated aq $NaHCO_3$ and then brine, dried over Na_2SO_4 and then evaporated to give pure lactone.

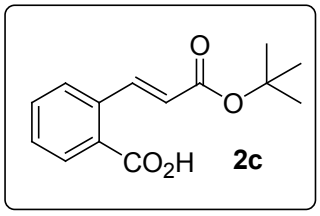
Spectral and analytical data of 2a-f, 3a-f and 5a-k:



2-(2-Methoxycarbonylvinyl)-benzoic acid (**2a**):[1] White solid. M. P. 102-104 °C. ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 3.80 (s, 3H), 6.30 (d, *J* = 16.0 Hz, 1H), 7.44–7.51 (m, 3H), 8.07 (d, *J* = 7.4 Hz, 1H), 8.52 (d, *J* = 16.0 Hz, 1H), 8.98 (bs, 1H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 51.92, 120.98, 128.22, 128.52, 129.53, 131.72, 133.31, 137.18, 144.05, 167.14, 171.84. *Elemental Anal*. Calcd for $C_{11}H_{10}O_4$: C, 64.07; H, 4.89. Found C, 63.85; H, 5.07.

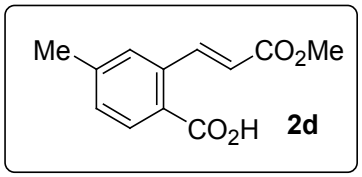


2-(2-Ethoxycarbonyl-vinyl)-benzoic acid (**2b**):[2] White solid. ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 1.41 (t, *J* = 7.2 Hz, 3H), 4.36 (q, *J* = 7.2 Hz, 2H), 6.39 (d, *J* = 15.8 Hz, 1H), 8.63 (d, *J* = 15.8 Hz, 1H), 7.61–7.70 (m, 3H), 8.19 (m, 1H).

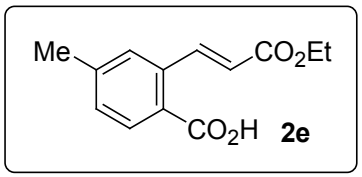


2-(2-tert-Butoxycarbonylvinyl)-benzoic acid (**2c**): Colourless liquid. ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 1.52 (s, 9H), 6.275 (d, *J* = 16.0 Hz, 1H), 7.33–7.69 (m, 3H), 8.06 (d, *J* = 7.8 Hz, 1H), 8.45 (d, *J* = 15.8 Hz, 1H), 9.41 (bs, 1H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 28.18 (3C),

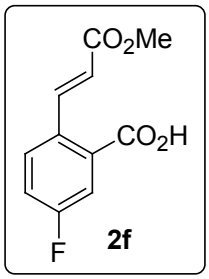
80.81, 123.19, 128.09, 128.59, 129.25, 131.64, 133.13, 137.31, 142.64, 166.08, 172.08. *Elemental Anal*. Calcd for $C_{14}H_{16}O_4$: C, 67.73; H, 6.50. Found C, 67.93; H, 6.59.



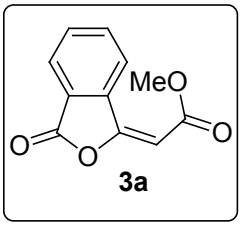
2-(2-Methoxycarbonylvinyl)-4-methylbenzoic acid (**2d**): Puffy white solid. M. P. 96 °C. ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 2.42 (s, 3H), 3.77 (s, 3H), 6.31 (d, J = 16.0 Hz, 1H), 7.27 (dd, J_1 = 2.0 Hz, J_2 = 7.4 Hz, 1H), 7.40 (s, 1H), 8.01 (d, J = 8.0 Hz, 1H), 8.55 (d, J = 16.0 Hz, 1H), 8.73 (bs, 1H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 14.05, 51.85, 120.71, 125.78, 128.91, 130.28, 131.92, 137.31, 144.09, 144.40, 167.21, 171.75. *Elemental Anal*. Calcd for $C_{12}H_{12}O_4$: C, 65.45; H, 5.49. Found C, 65.32; H, 5.61.



2-(2-Ethoxycarbonylvinyl)-4-methylbenzoic acid (**2e**): White solid. M. P. 74-76 °C. ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 1.34 (t, J = 7.2 Hz, 3H), 2.43 (s, 3H), 4.28 (q, J = 7.2 Hz, 2H), 6.31 (d, J = 16.0 Hz, 1H), 7.28 (dd, J_1 = 2.4 Hz, J_2 = 7.6 Hz, 1H), 7.41 (s, 1H), 8.01 (d, J = 7.8 Hz, 1H), 8.50 (bs, 1H), 8.55 (d, J = 16.0 Hz, 1H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 14.41, 21.69, 60.76, 121.28, 125.86, 129.01, 130.32, 132.00, 137.52, 144.22 (2C), 166.88, 171.88. *Elemental Anal*. Calcd for $C_{13}H_{14}O_4$: C, 66.66; H, 6.02. Found C, 66.78; H, 6.20.

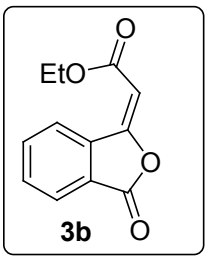


5-Fluoro-2-(2-methoxycarbonyl-vinyl)-benzoic acid (**2f**): White solid. M. P. 158-160 °C. ^{1}H NMR ($CDCl_3$$^+$$d_6$-DMSO, 200 MHz): δ = 3.65 (s, 3H), 4.62(bs), 6.03 (d, J = 15.8 Hz, 1H), 7.04–7.14 (m, 1H), 7.43–7.58 (m, 2H), 8.35 (d, J = 15.8 Hz, 1H), 8.91.^{13}C NMR ($CDCl_3$$^+$$d_6$-DMSO, 50 MHz): δ = 51.60, 117.76 (d, J = 23 Hz), 118.11 (d, J = 21.5 Hz), 119.95, 129.57, 129.72, 132.22(d, J = 3.5 Hz), 132.71, 143.09, 166.92, 167.36. *Elemental Anal*. Calcd for $C_{11}H_9FO_4$: C, 58.93; H, 4.05. Found C, 58.69; H, 4.21.



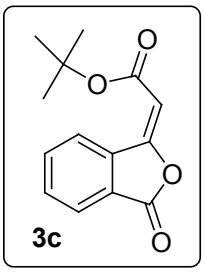
3a

(3-Oxo-3*H*-isobenzofuran-1-ylidene)-acetic acid methyl ester (**3a**):[3] White solid. M. P. 97–98 °C. *FTIR* (KBr, cm^{-1}): 2966, 2363, 1806, 1718, 1653, 1479, 1435, 1208, 1147, 1035, 973, 849, 776, 690. ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 3.80 (s, 3H), 6.11 (s, 1H), 7.67 (t, *J* = 7.4 Hz, 1H), 7.79 (t, *J* = 7.6 Hz, 1H), 7.93 (d, *J* = 7.6 Hz, 1H), 9.01 (d, *J* = 7.8 Hz, 1H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 51.95, 101.93, 125.38, 126.52, 128.17, 132.56, 135.30, 136.05, 158.00, 165.65, 165.97. HRMS calcd for $C_{11}H_8O_4Na$ (MNa^+) *m/z* = 227.0320, found *m/z* = 227.0320. *Elemental Anal*. Calcd for $C_{11}H_8O_4$: C, 64.71; H, 3.95. Found C, 64.57; H, 4.03.



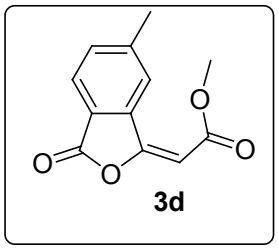
3b

(3-Oxo-3*H*-isobenzofuran-1-ylidene)-acetic acid ethyl ester (**3b**): White solid. M. P. 74 °C. ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 1.36 (t, *J* = 7.2 Hz, 3H), 4.30 (q, *J* = 7.2 Hz, 2H), 6.14 (s, 1H), 7.70 (t, *J* = 7.4 Hz, 1H), 7.82 (t, *J* = 7.6 Hz, 1H), 7.96 (d, *J* = 8.0 Hz, 1H), 9.05 (d, *J* = 7.8 Hz, 1H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 14.22, 60.92, 102.45, 125.32, 126.52, 128.19, 132.46, 135.24, 136.11, 157.80, 165.52, 165.69. *Elemental Anal*. Calcd for $C_{12}H_{10}O_4$: C, 66.05; H, 4.62. Found C, 65.90; H, 4.79.



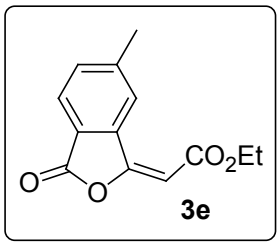
3c

(3-Oxo-3*H*-isobenzofuran-1-ylidene)-acetic acid tert-butyl ester (**3c**): White solid. M. P. 70 °C. ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 1.56 (s, 9H), 6.09 (s, 1H), 7.68 (t, *J* = 7.2 Hz, 1H), 7.82 t, *J* = 7.4 Hz, 1H), 7.96 (d, *J* = 8.0 Hz, 1H), 9.03 (d, *J* = 7.8 Hz, 1H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 28.33 (3C), 81.69, 104.72, 125.40, 126.66, 128.35, 132.38, 135.32, 136.43, 157.19, 165.02, 166.12. *Elemental Anal*. Calcd for $C_{14}H_{14}O_4$: C, 68.28; H, 5.73. Found C, 68.10; H, 5.91.

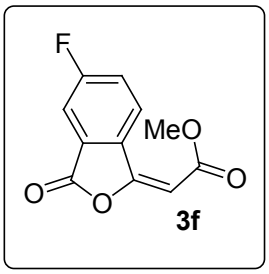


3d

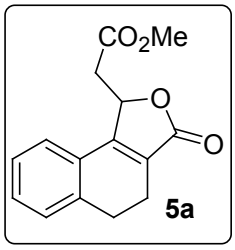
(6-Methyl-3-oxo-3*H*-isobenzofuran-1-ylidene)-acetic acid methyl ester (**3d**): White solid. M. P. 122-124 °C. ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 2.54 (s, 3H), 3.80 (s, 3H), 6.08 (s, 1H), 7.47 (d, *J* = 7.8 Hz, 1H), 7.80 (d, *J* = 7.8 Hz, 1H), 8.81 (s, 1H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 22.41, 51.90, 101.56, 124.05, 125.17, 128.35, 133.63, 136.44, 146.81, 158.23, 165.63, 166.11. *Elemental Anal*. Calcd for $C_{12}H_{10}O_4$: C, 66.05; H, 4.62. Found C, 66.13; H, 4.87.



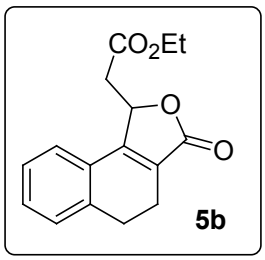
(6-Methyl-3-oxo-3*H*-isobenzofuran-1-ylidene)-acetic acid ethyl ester (**3e**): White solid. M. P. 74-76 °C. ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 1.30 (t, *J* = 7.2 Hz, 3H), 2.53 (s, 3H), 2.26 (q, *J* = 7.2 Hz, 3H), 2.53 (s, 2H), 6.08 (s, 1H), 7.23 (d, *J* = 0.6 Hz, 1H), 4.47 (d, *J* = 7.8 Hz, 1H), 7.81 (d, *J* = 8.0 Hz, 1H), 8.82 (s, 1H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 14.24, 22.68, 60.87, 102.12, 124.07, 125.14, 128.37, 133.55, 146.73, 158.02, 165.66, 166.10. *Elemental Anal*. Calcd for $C_{13}H_{12}O_4$: C, 67.23; H, 5.21. Found C, 67.10; H, 5.37.



(5-Fluoro-3-oxo-3*H*-isobenzofuran-1-ylidene)-acetic acid methyl ester (**3f**): White solid. M. P. 80-82 °C. ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 3.81 (s, 3H), 6.11 (s, 1H), 7.52–7.61 (m, 2H), 9.08 (dd, J_1 = 4.8 Hz, J_2 = 8.6 Hz, 1H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 51.99, 101.82, 111.79, 112.43, 119.11, 122.80, 124.44, 130.81, 156.79, 165.40, 165.88. *Elemental Anal*. Calcd for $C_{11}H_7FO_4$: C, 59.47; H, 3.18. Found C, 59.58; H, 3.33.

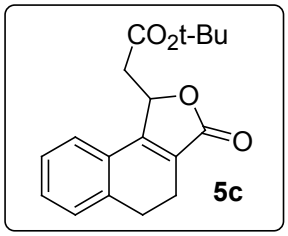


(3-Oxo-1,3,4,5-tetrahydronaphtho[1,2-*c*]furan-1-yl)-acetic acid methyl ester (**5a**): Colourless crystalline solid. M. P. 130-132°C. *FTIR* (KBr, cm^{-1}): 2942, 1749, 1733, 1650, 1439, 1368, 1318, 1167, 1063, 1011, 766, 613.^{1}H NMR ($CDCl_3$, 200 MHz): δ = 2.25–2.66 (m, 3H), 2.86–3.05 (m, 3H), 3.65 (s, 3H), 5.67–5.73 (m, 1H), 7.07–7.31 (m, 4H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 18.16, 27.85, 38.71, 52.23, 76.37, 123.90, 125.96, 127.15, 127.32, 128.92, 131.05, 137.81, 157.73, 169.79, 171.89. HRMS calcd for $C_{15}H_{15}O_4$ (MH^+) *m/z* = 259.0970, found *m/z* = 259.0967. *Elemental Anal*. Calcd for $C_{15}H_{14}O_4$: C, 69.76; H, 5.46. Found C, 69.62; H, 5.33.

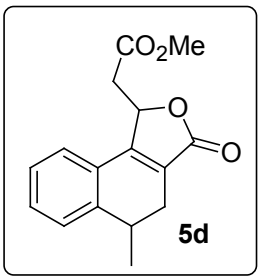


(3-Oxo-1,3,4,5-tetrahydronaphtho[1,2-*c*]furan-1-yl)-acetic acid ethyl ester (**5b**): White solid. M. P. 83–85 °C. ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 1.22 (t, *J* = 7.2 Hz, 3H), 2.37–2.70 (m, 3H), 2.89–3.04 (m, 3H), 4.15 (q, *J* = 7.2 Hz, 2H), 5.70–5.77 (m, 1H), 7.09–7.35 (m, 4H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 14.22, 18.29, 27.99, 39.07, 61.43, 76.60, 124.02, 126.12,

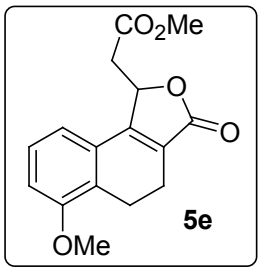
127.25, 127.49, 129.03, 131.14, 137.93, 157.90, 169.47, 172.11. *Elemental Anal*. Calcd for $C_{16}H_{16}O_4$: C, 70.57; H, 5.92. Found C, 70.79; H, 6.05.



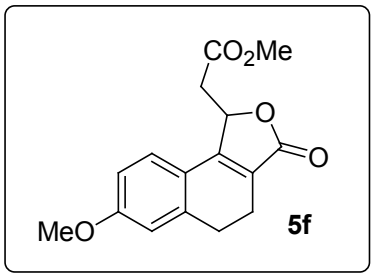
(3-Oxo-1,3,4,5-tetrahydronaphtho[1,2-*c*]furan-1-yl)-acetic acid tert-butyl ester (**5c**): Colourless viscous liquid. ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 1.47 (s, 9H), 2.42–2.75 (m, 3H), 2.92–3.02 (m, 3H), 5.70–5.77 (m, 1H), 7.14–7.35 (m, 4H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 18.23, 28.05 (3C), 29.70, 40.09, 76.82, 81.95, 124.03, 126.01, 127.15, 127.52, 128.91, 130.97, 137.83, 157.93, 168.57, 172.19. *Elemental Anal*. Calcd for $C_{18}H_{20}O_4$: C, 71.98; H, 6.71. Found C, 71.83; H, 6.82.



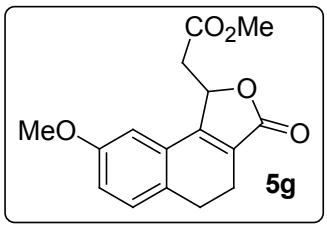
(5-Methyl-3-oxo-1,3,4,5-tetrahydronaphtho[1,2-*c*]furan-1-yl)-acetic acid methyl ester (**5d**): Yellow solid. M. P. 78 °C. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 1.32-1.39 (m, 3H), 2.45-2.73 (m, 3H), 2.95-3.19 (m, 2H), 3.77 (s, 3H), 5.73-5.78 (m, 1H), 7.11-7.40 (m, 4H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 22.67, 25.59, 33.18, 38.86, 52.52, 76.47, 124.12, 124.32, 126.11, 127.34, 128.86, 131.61, 143.93, 156.92, 169.94, 172.70. *Elemental Anal*. Calcd for $C_{16}H_{16}O_4$: C, 70.57; H, 5.92. Found C, 70.43; H, 6.02.



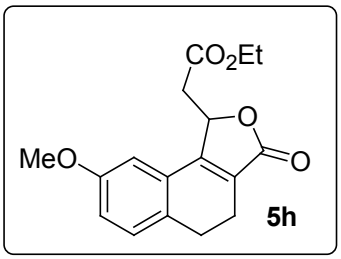
(6-Methoxy-3-oxo-1,3,4,5-tetrahydronaphtho[1,2-*c*]furan-1-yl)-acetic acid methyl ester (**5e**): White crystalline solid. M. P. 138 °C. ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 2.21 2.67 (m, 4H), 2.96 (dd, J_1 = 2.6 Hz, J_2 = 16.4 Hz, 1H), 3.17–3.28 (m, 1H), 3.66 (s, 1H), 3.77 (s, 1H), 5.65–5.71 (m, 1H), 6.69 (d, J = 7.4 Hz, 1H), 6.88 (d, J = 8.2 Hz, 1H), 7.09–7.20 (m, 1H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 17.64, 20.21, 39.02, 52.38, 55.72, 76.73, 113.46, 116.35, 125.82, 126.18, 127.71, 128.28, 157.15, 157.75, 170.01, 172.19. *Elemental Anal*. Calcd for $C_{16}H_{16}O_5$: C, 66.66; H, 5.59. Found C, 66.71; H, 5.73.



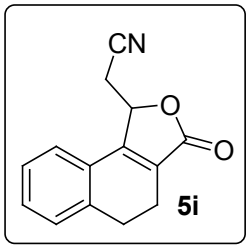
(7-Methoxy-3-oxo-1,3,4,5-tetrahydronaphtho[1,2-*c*]furan-1-yl)-acetic acid methyl ester (**5f**): Off-White solid. M. P. 130-132 °C. ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 2.47–2.70 (m, 3H), 2.90–3.06 (m, 3H), 3.77 (s, 3H), 3.81 (s, 3H), 5.71–5.76 (m, 1H), 6.74–6.83 (m, 2H), 7.09 (d, J = 8.4 Hz, 1H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 18.08, 28.42, 39.01, 52.34, 55.45, 76.30, 111.90, 115.15, 120.29, 123.13, 125.45, 140.25, 158.00, 161.78, 170.05, 172.34. *Elemental Anal*. Calcd for $C_{16}H_{16}O_5$: C, 66.66; H, 5.59. Found C, 66.79; H, 5.80.



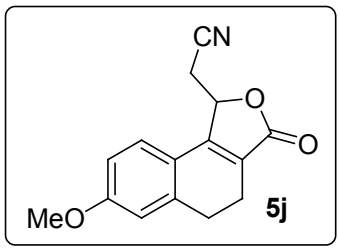
(8-Methoxy-3-oxo-1,3,4,5-tetrahydronaphtho[1,2-*c*]furan-1-yl)-acetic acid methyl ester (**5g**): Off-white solid. M. P. 131-133 °C. ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 2.35 – 2.75 (m, 3H), 2.83–3.05 (m, 3H), 3.72 (s, 3H), 3.78 (s, 1H), 5.71–5.76 (m, 1H), 6.65 (d, J = 1.8 Hz, 1H), 6.86 (dd, J_1 = 2.0 Hz, J_2 = 8.2 Hz, 1H), 7.19 (d, J = 8.4 Hz, 1H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 18.53, 17.04, 38.79, 52.32, 55.56, 76.39, 110.37, 115.30, 126.73, 128.22, 129.70, 157.66, 158.59, 169.83, 171.89. *Elemental Anal*. Calcd for $C_{16}H_{16}O_5$: C, 66.66; H, 5.59. Found C, 66.50; H, 5.73.



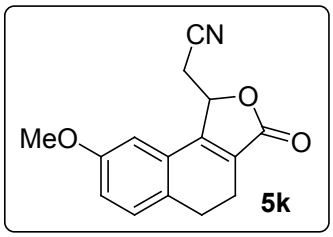
(8-Methoxy-3-oxo-1,3,4,5-tetrahydronaphtho[1,2-c]furan-1-yl)-acetic acid ethyl ester (**5h**): Yellow solid. M. P. 72-74 °C. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 1.27 (t, J = 7.2 Hz, 3H), 2.38–2.48 (m, 1H), 2.53–2.59 (m, 1H), 2.63–2.70 (m, 1H), 2.87-2.92 (m, 2H), 2.99 (dd, J_1 = 1.4 Hz, J_2 = 8.2 Hz, 1H), 3.81 (s, 3H), 4.20 (q, J = 7.2 Hz, 2H), 5.75 (dd, J_1 = 2.8 Hz, J_2 = 6.4 Hz, 1H), 6.69 (s, 1H), 6.88 (dd, , J_1 = 2.4 Hz, J_2 = 8.0 Hz, 1H), 7.21 (d, J = 8.0 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 14.06, 18.46, 26.99, 38.91, 55.49, 61.29, 76.36, 110.39, 115.19, 126.64, 128.21, 129.60, 129.63, 157.65, 158.53, 169.29, 171.88. *Elemental Anal*. Calcd for $C_{17}H_{18}O_5$: C, 67.54; H, 6.00. Found C, 67.43; H, 6.19.



(3-Oxo-1,3,4,5-tetrahydronaphtho[1,2-c]furan-1-yl)-acetonitrile (**5i**): Yellow solid. M. P. 150 °C. *FTIR* (KBr, cm^{-1}): 2925, 2254, 1745, 1657, 1395, 1308, 1064, 1049, 1010, 780. ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 2.43–2.58 (m, 1H), 2.67–3.07 (m, 4H), 3.25 (dd, J_1 = 3.2 Hz, J_2 = 17.2 Hz, 1H), 5.56 (m, 1H), 7.13 (d, J = 7.4 Hz, 1H), 7.26–7.43 (m, 3H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 18.27, 23.27, 27.80, 74.13, 114.65, 123.70, 126.73, 127.37 (2C), 129.21, 131.59, 138.07, 156.19, 171.01. *Elemental Anal*. Calcd for $C_{14}H_{11}NO_2$: C, 74.65; H, 4.92; N, 6.22. Found C, 74.77; H, 5.03; N, 6.10.



(7-Methoxy-3-oxo-1,3,4,5-tetrahydronaphtho[1,2-c]furan-1-yl)-acetonitrile (**5j**): Yellow solid. M. P. 106-108 °C. ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 2.64–2.74 (m, 1H), 2.75–3.03 (m, 4H), 3.16 (dd, J_1 = 4.2 Hz, J_2 = 17.2 Hz, 1H), 3.82 (s, 3H), 5.44 (m, 1H), 6.77 (dd, J_1 = 2.2 Hz, J_2 = 8.4 Hz, 1H), 6.84 (d, J = 2.2 Hz, 1H), 2.02 (d, J = 8.4 Hz, 1H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 18.20, 23.45, 28.38, 55.33, 73.86, 112.11, 114.48, 115.45, 119.69, 124.54, 125.12, 140.53, 156.10, 162.20, 171.04. *Elemental Anal*. Calcd for $C_{15}H_{13}NO_3$: C, 70.58; H, 5.13; N, 5.49. Found C, 70.39; H, 5.31; N, 5.32.



(8-Methoxy-3-oxo-1,3,4,5-tetrahydronaphtho[1,2-c]furan-1-yl)-acetonitrile (**5k**): White solid. M. P. 174-176 °C. ^{1}H NMR ($CDCl_3$, 200 MHz): δ = 2.41–2.56 (m, 1H), 2.63-2.96 (m, 4H), 3.21 (dd, J_1 = 4.0 Hz, J_2 = 17.0 Hz, 1H), 3.83 (s, 3H), 5.53 (m, 1H), 6.65 (d, J = 1.8 Hz, 1H), 6.92 (dd, J_1 = 1.8 Hz, J_2 = 7.8 Hz, 1H), 7.25 (d, J = 7.8 Hz, 1H). ^{13}C NMR ($CDCl_3$, 50 MHz): δ = 18.60, 23.26, 26.93, 55.64, 74.05, 110.33, 114.48, 115.66, 127.55, 128.12, 129.89, 129.99, 155.99, 158.68, 170.83. *Elemental Anal*. Calcd for $C_{15}H_{13}NO_3$: C, 70.58; H, 5.13; N, 5.49. Found C, 70.71; H, 5.25; N, 5.39.

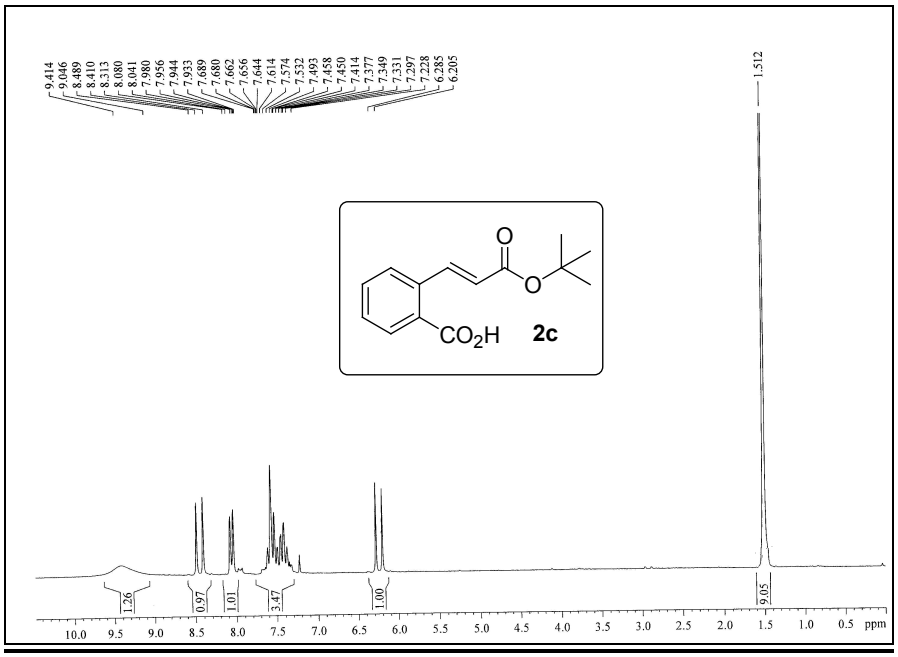
References:

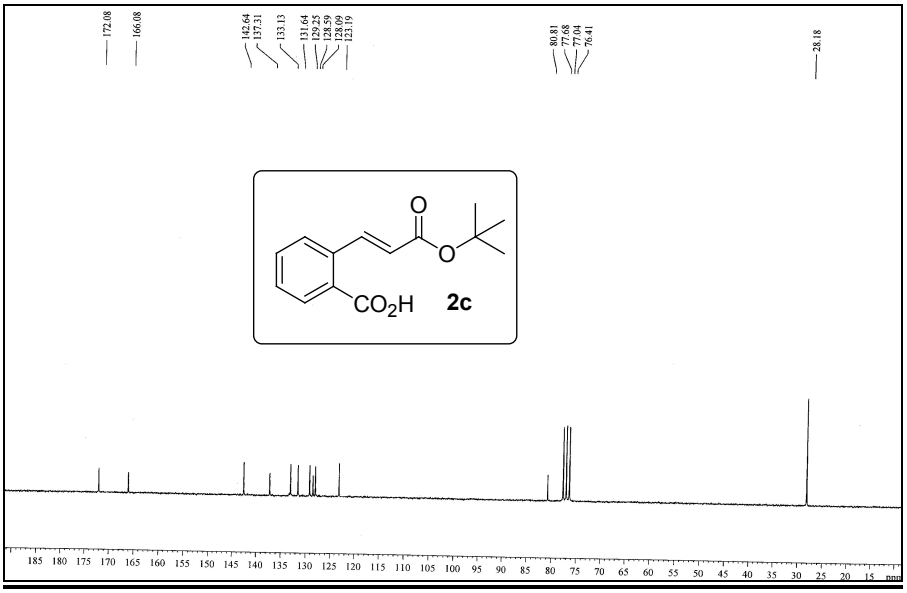
1. H. Fernholz, *Chem. Ber.*, 1951, **84**, 110.
2. E. Artuso, M. Barbero, I. Degani, S. Dughera, R. Fochi, *Tetrahedron*, 2006, **62**, 3146.
3. M. M. Kayser, K. L. Hatt, D. L. Hooper, *Can. J. Chem.*, 1992, **70,** 1985.

Electronic Supplementary Material (ESI) for RSC Advances

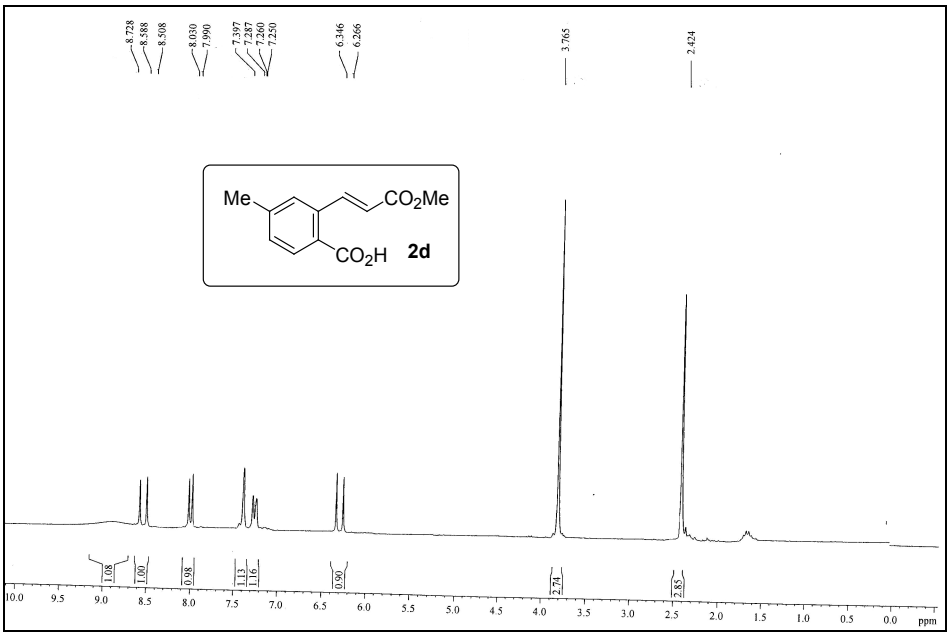
Spectra

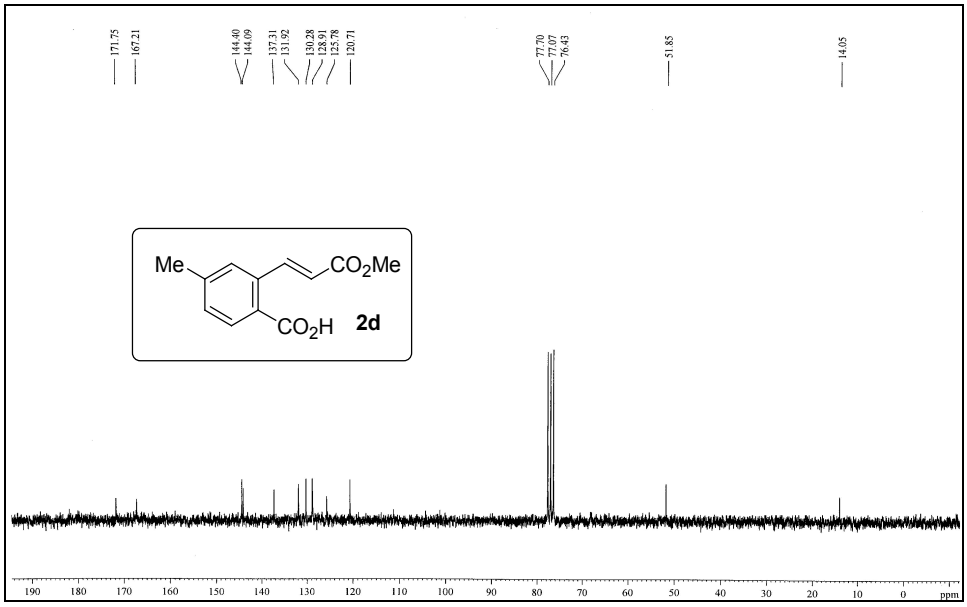
^{1}H NMR ($CDCl_3$, 200 MHz) and ^{13}C NMR ($CDCl_3$, 50 MHz) of **2c**



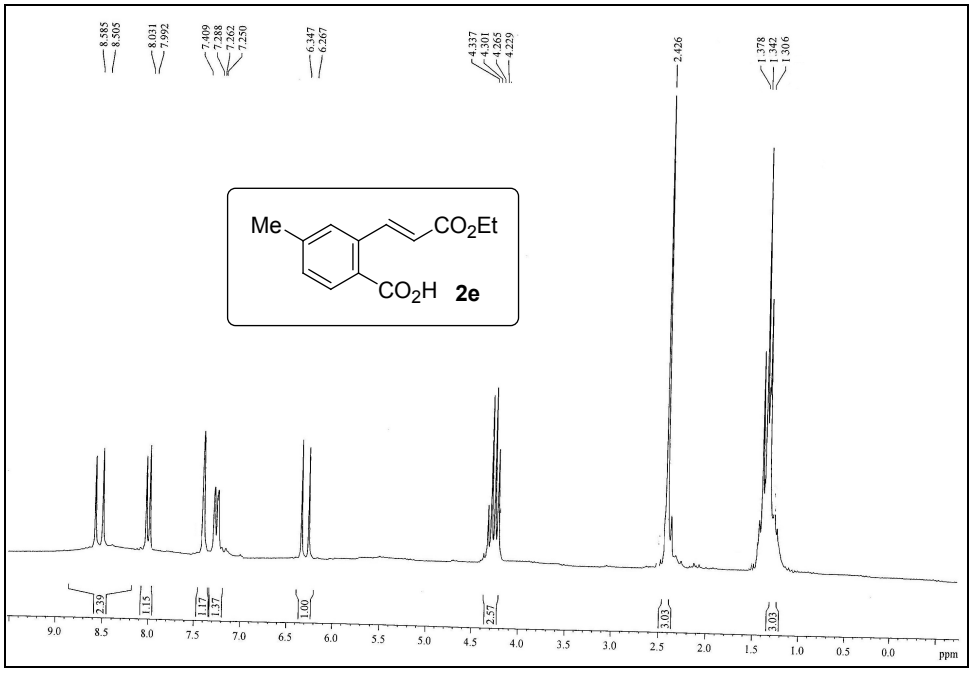


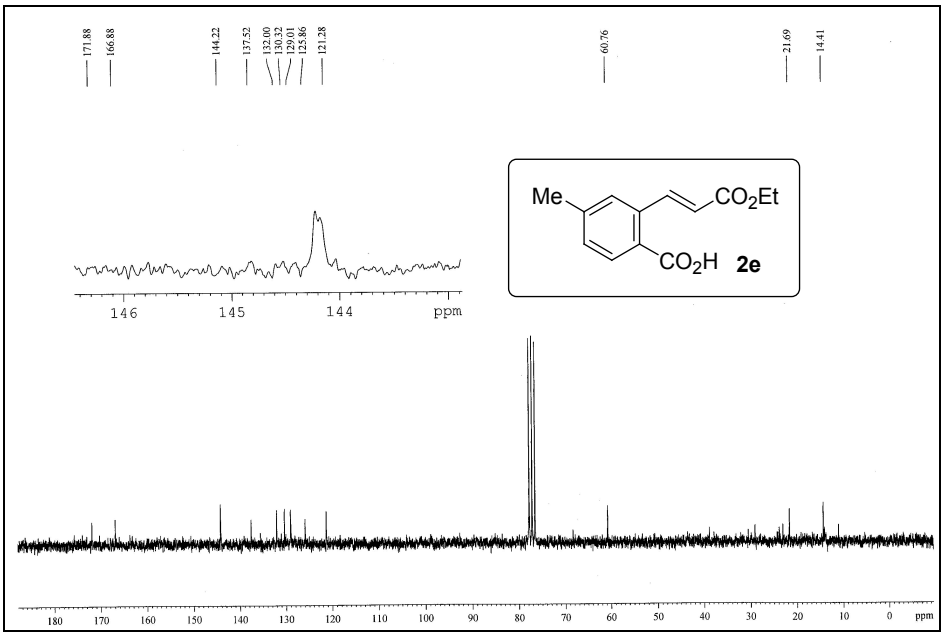
^{1}H NMR ($CDCl_3$, 200 MHz) and ^{13}C NMR ($CDCl_3$, 50 MHz) of **2d**



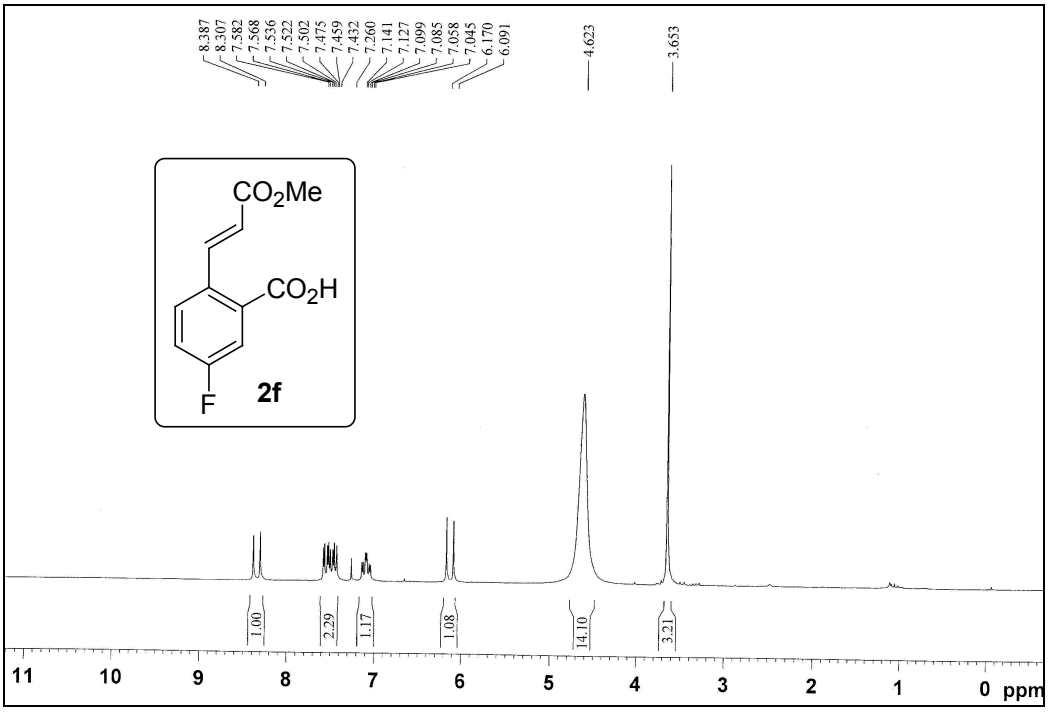


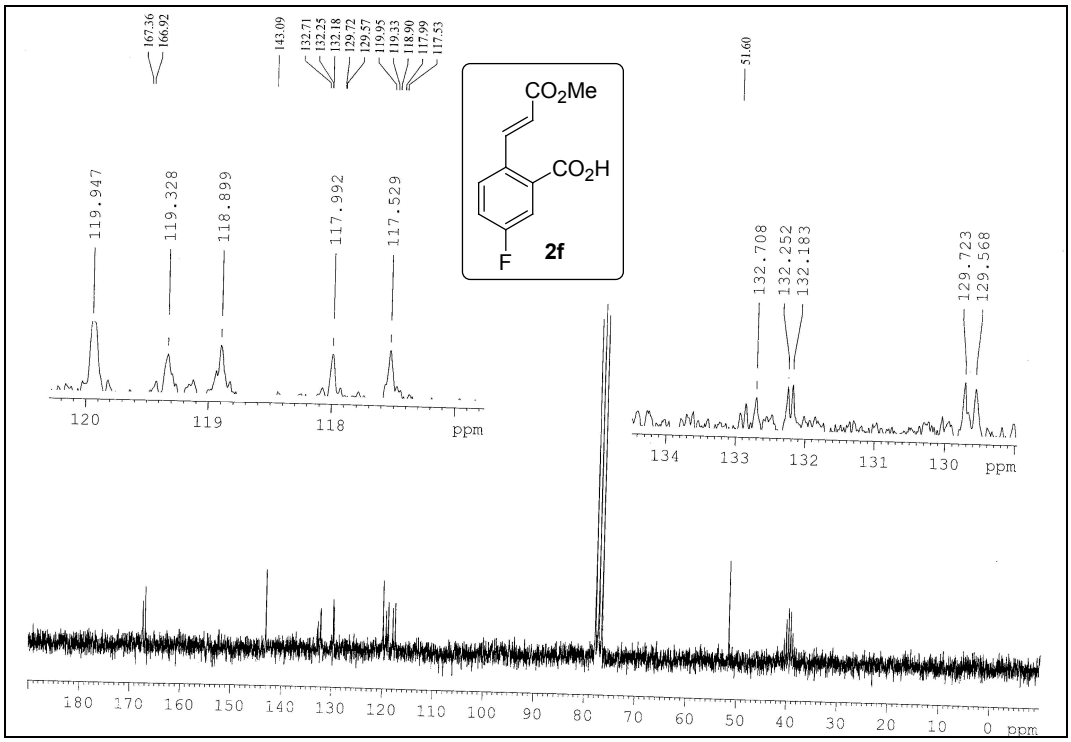
^{1}H NMR ($CDCl_3$, 200 MHz) and ^{13}C NMR ($CDCl_3$, 50 MHz) of **2e**



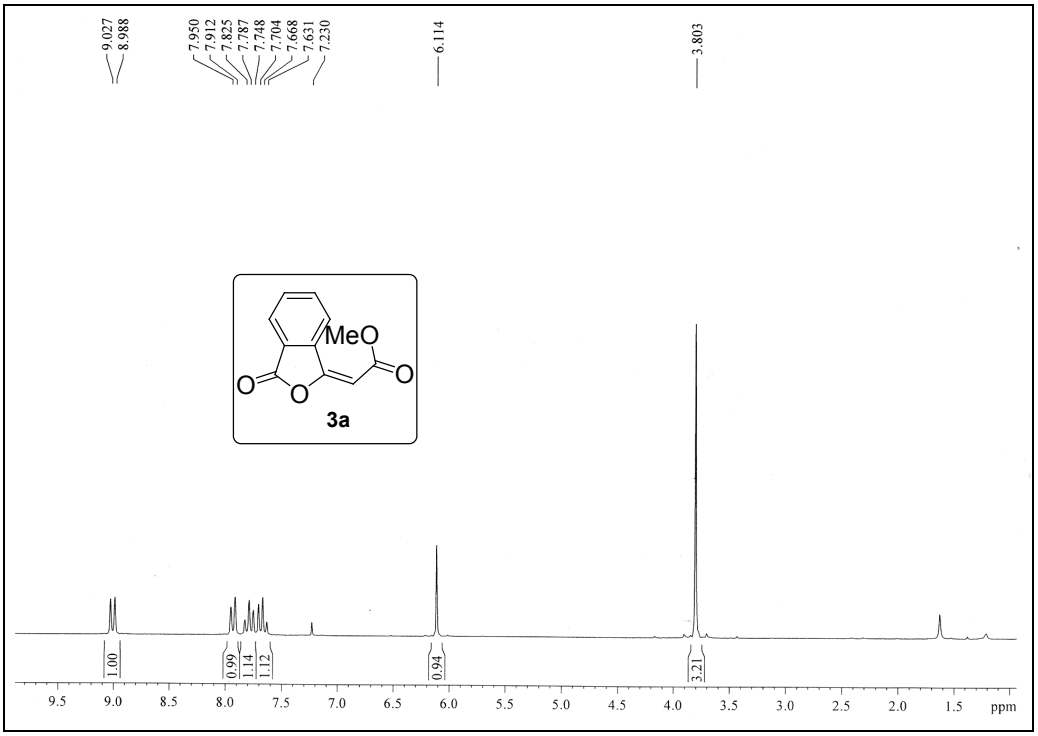


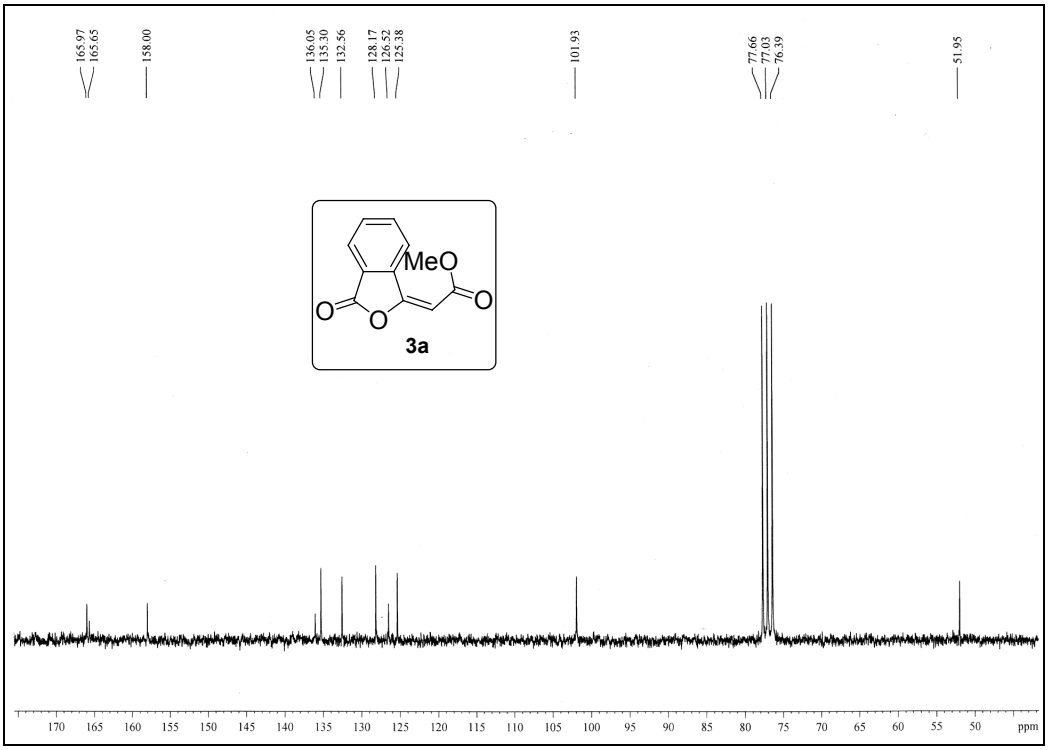
^{1}H NMR ($CDCl_3$ +d_6-DMSO, 200 MHz) and ^{13}C NMR ($CDCl_3$ +d_6-DMSO, 50 MHz) of **2f**



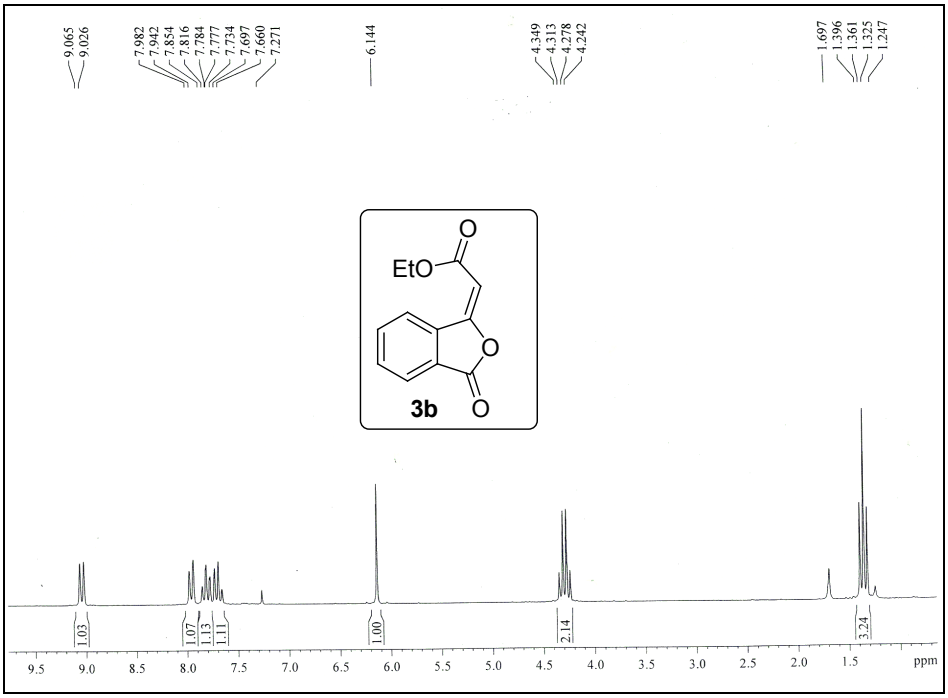


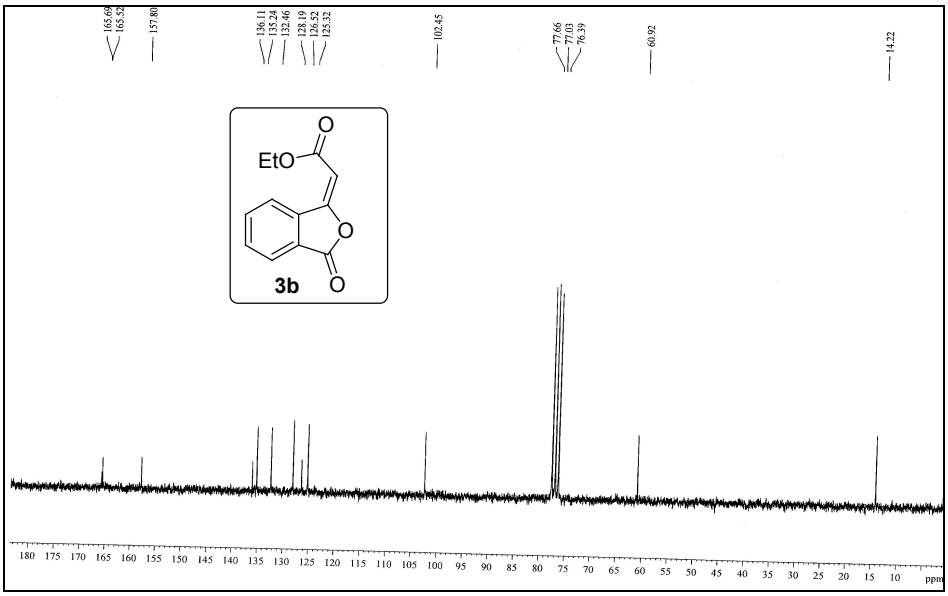
^{1}H NMR ($CDCl_3$, 200 MHz) and ^{13}C NMR ($CDCl_3$, 50 MHz) of **3a**



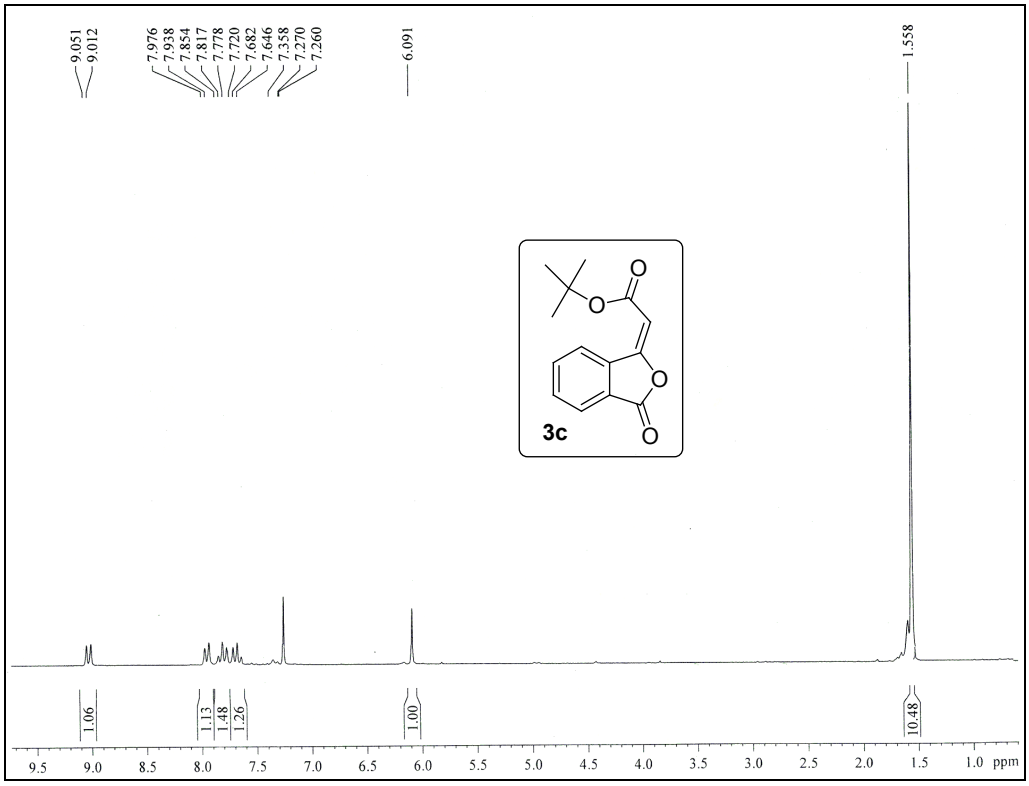


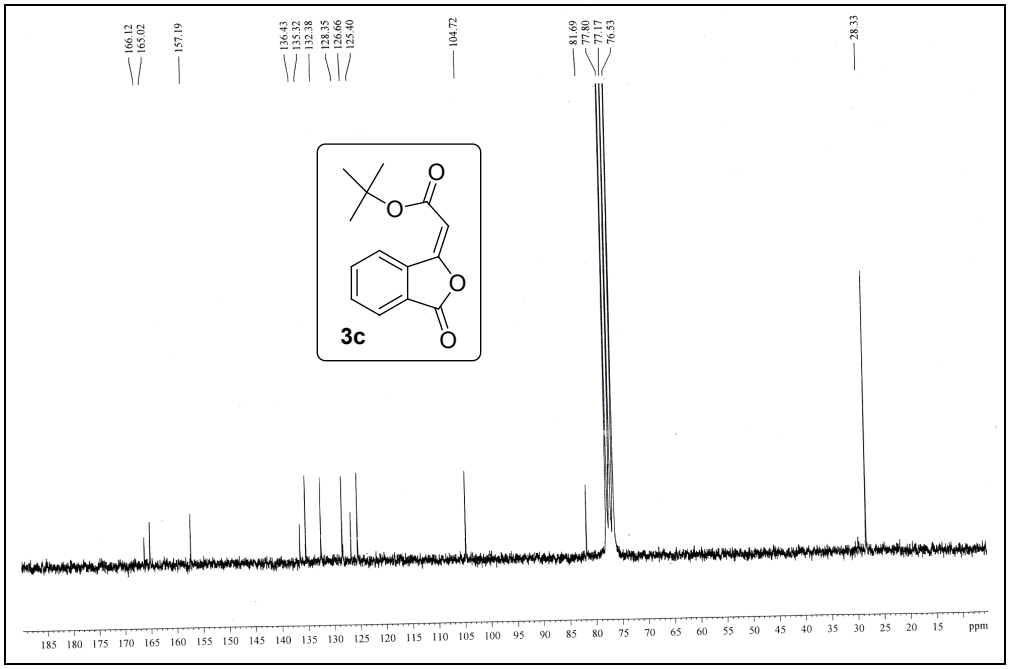
^{1}H NMR ($CDCl_3$, 200 MHz) and ^{13}C NMR ($CDCl_3$, 50 MHz) of **3b**



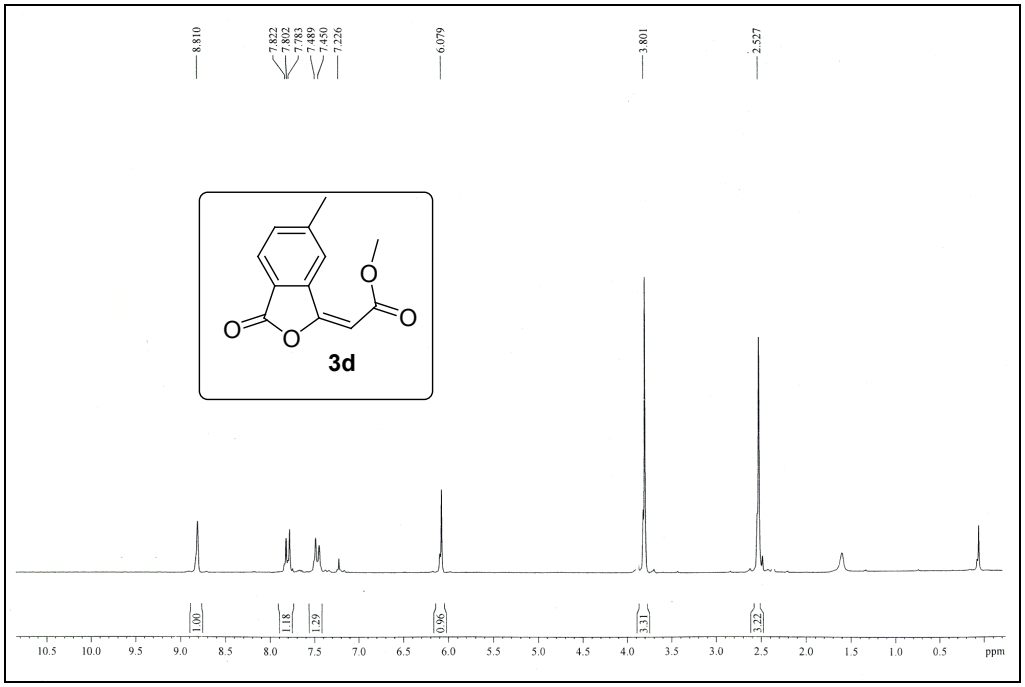


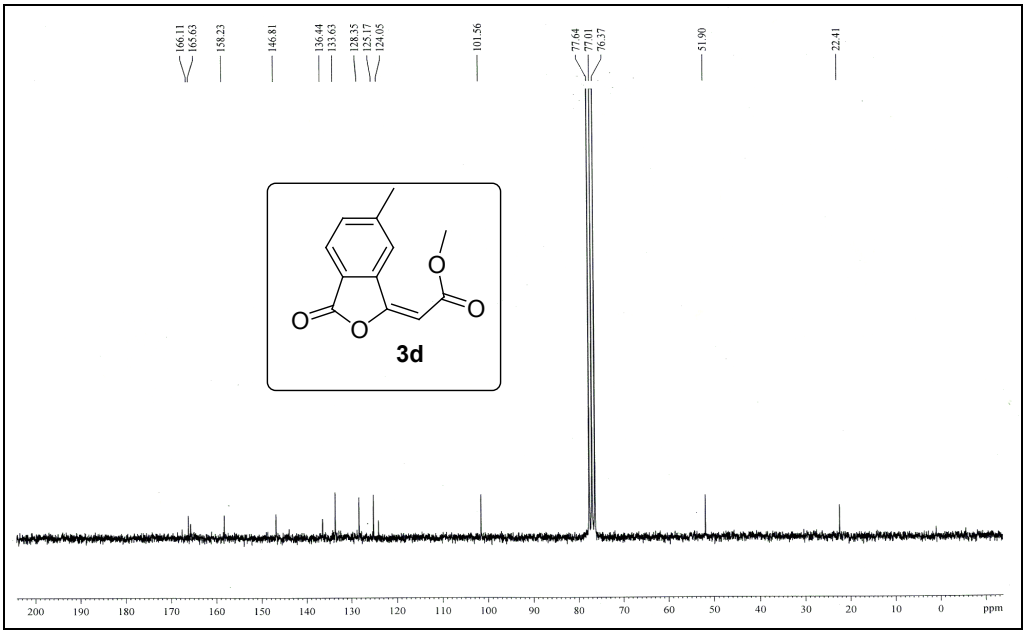
^{1}H NMR ($CDCl_3$, 200 MHz) and ^{13}C NMR ($CDCl_3$, 50 MHz) of **3c**



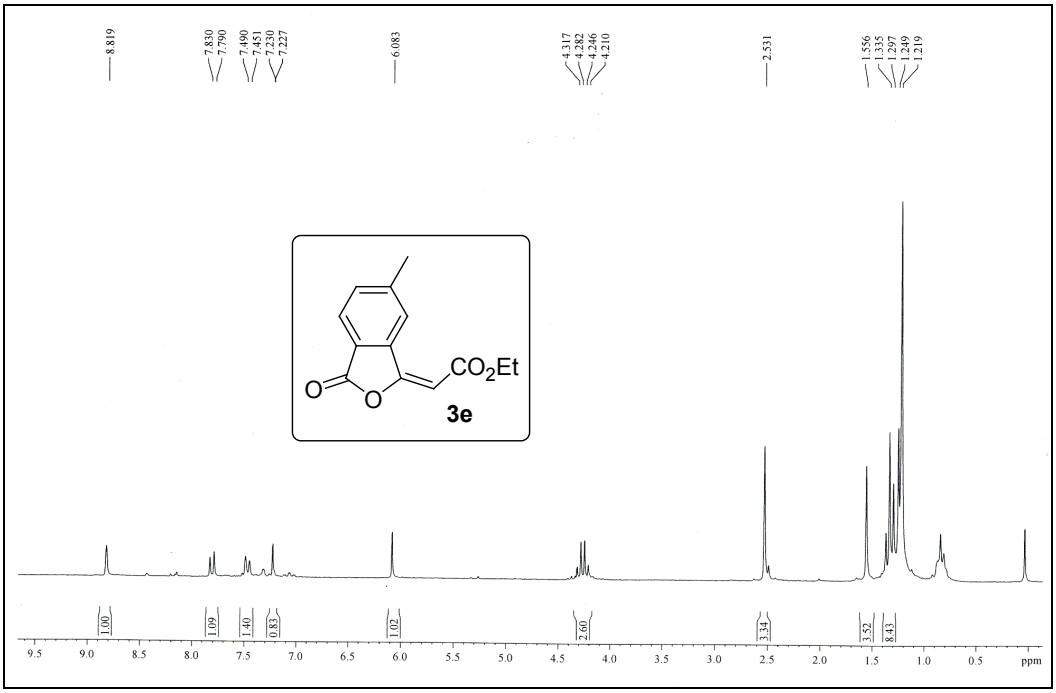


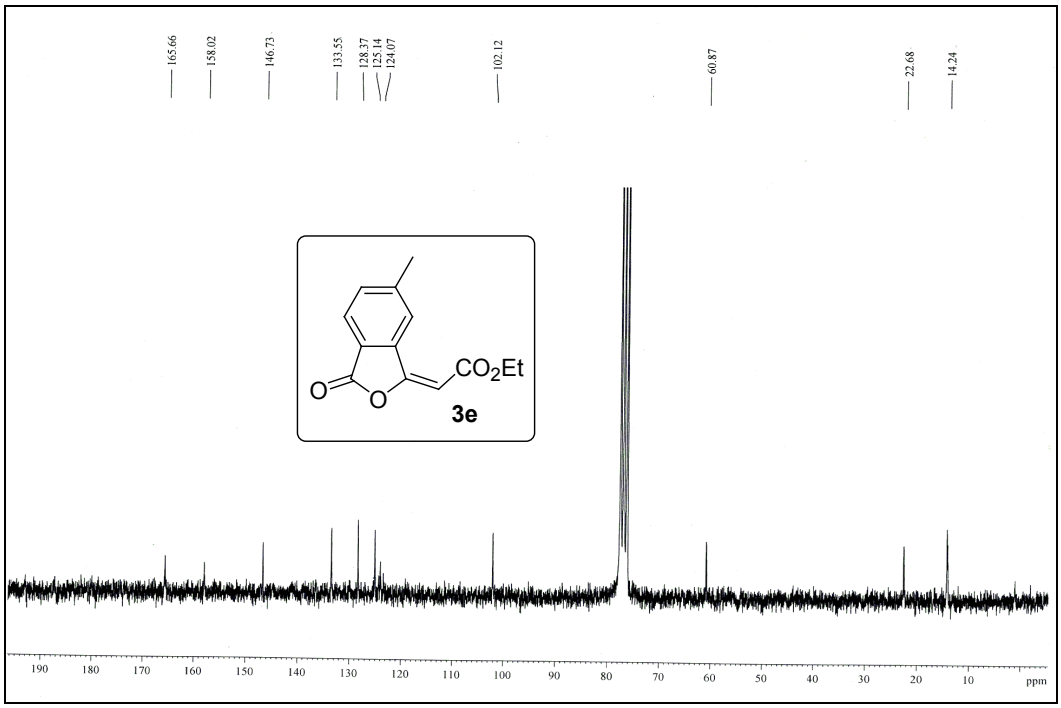
^{1}H NMR ($CDCl_3$, 200 MHz) and ^{13}C NMR ($CDCl_3$, 50 MHz) of **3d**



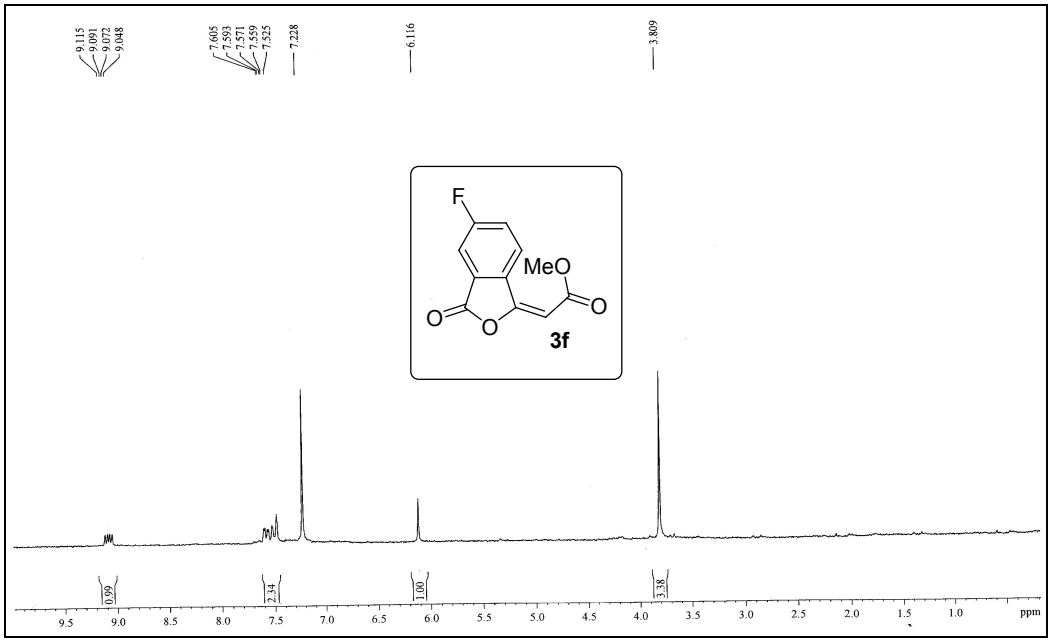


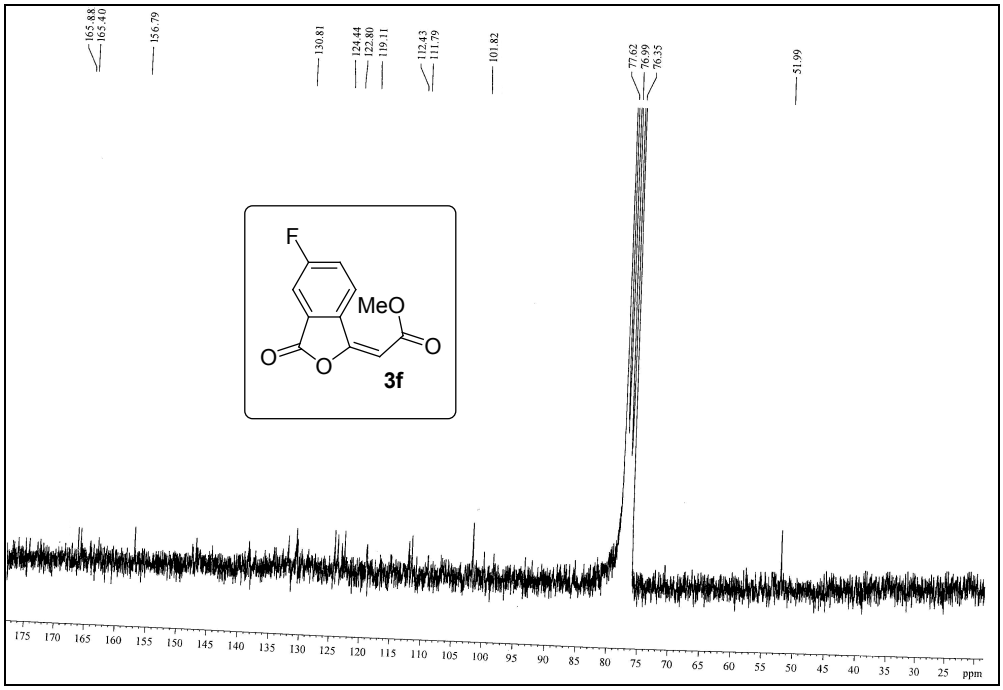
^{1}H NMR ($CDCl_3$, 200 MHz) and ^{13}C NMR ($CDCl_3$, 50 MHz) of **3e**



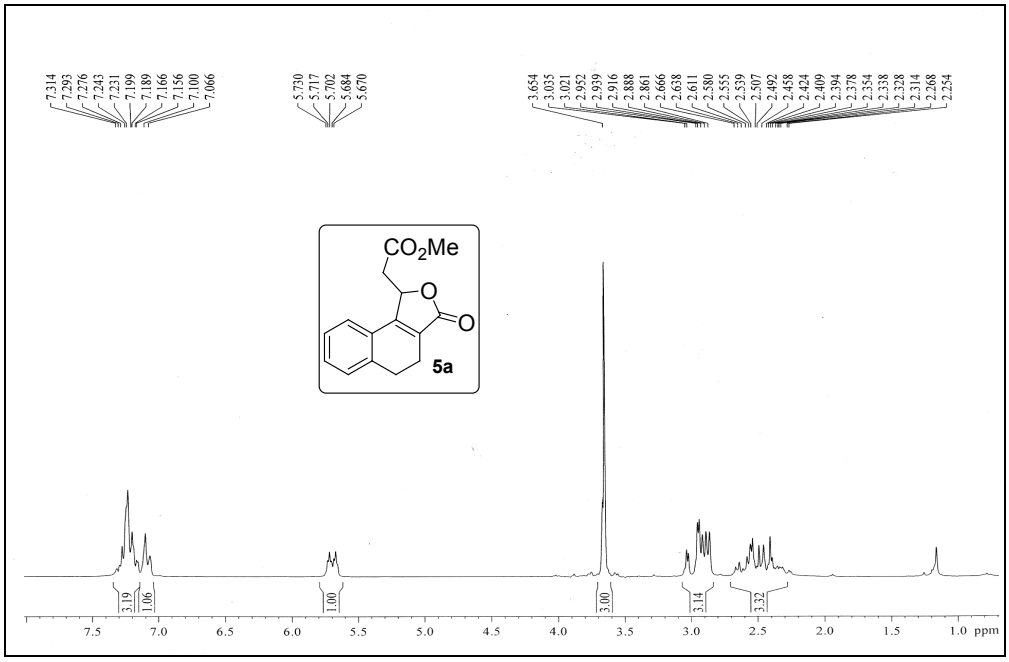


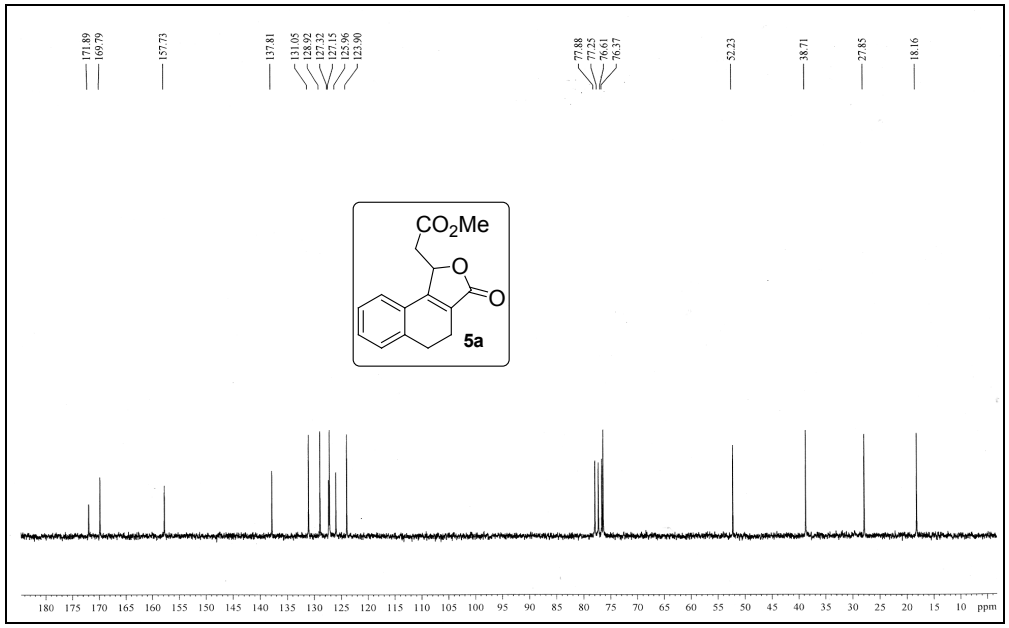
^{1}H NMR ($CDCl_3$, 200 MHz) and ^{13}C NMR ($CDCl_3$, 50 MHz) of **3f**



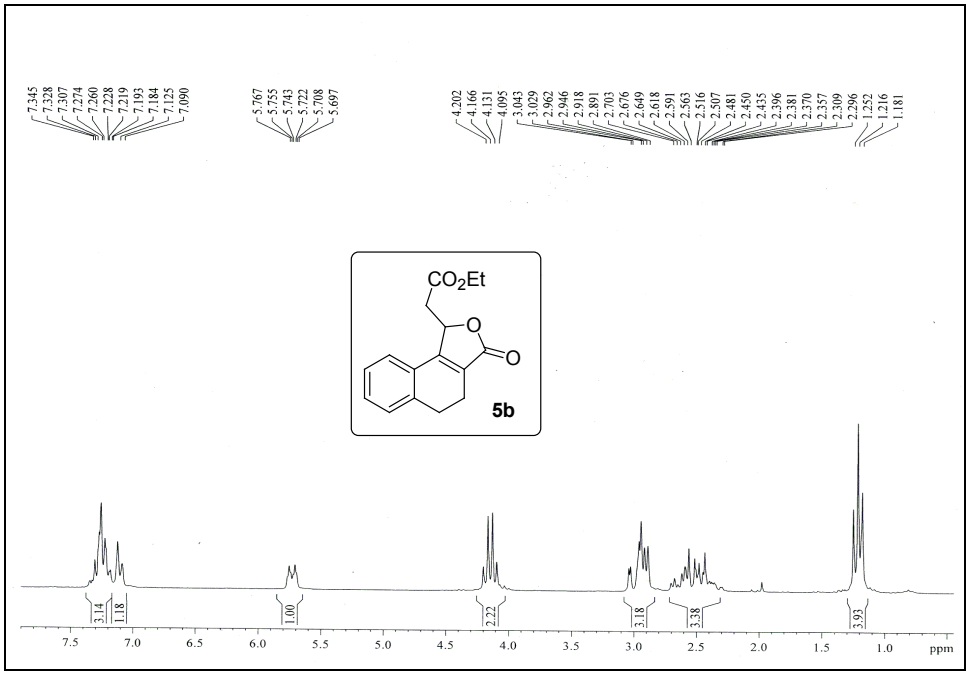


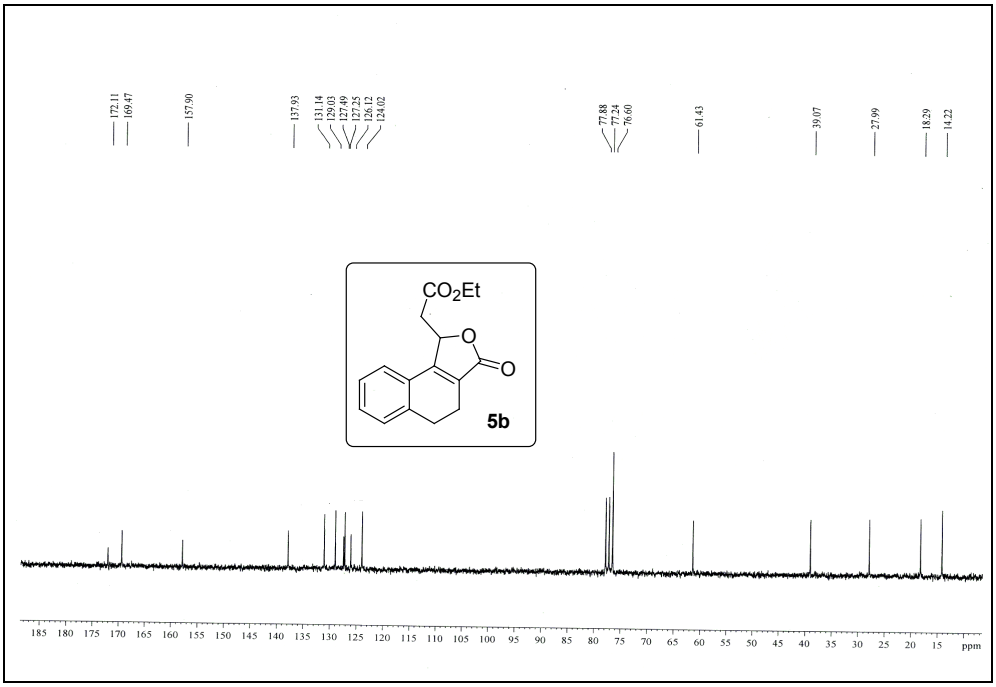
^{1}H NMR ($CDCl_3$, 200 MHz) and ^{13}C NMR ($CDCl_3$, 50 MHz) of **5a**



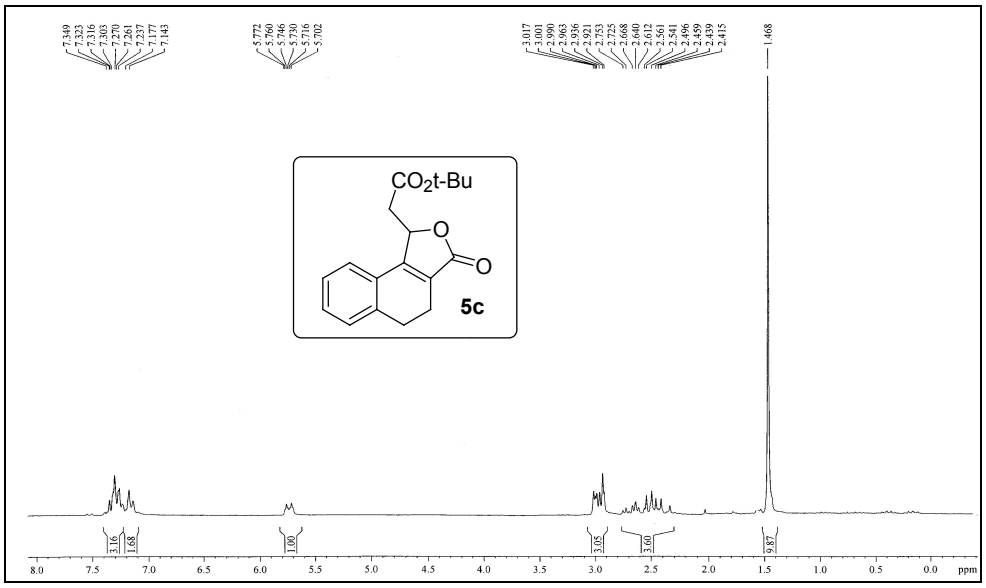


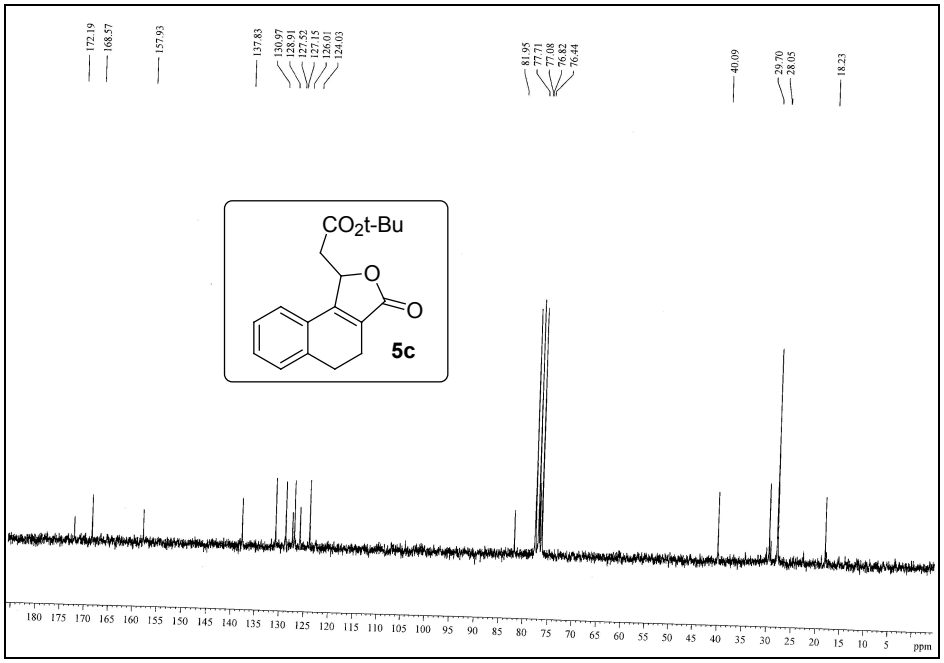
^{1}H NMR ($CDCl_3$, 200 MHz) and ^{13}C NMR ($CDCl_3$, 50 MHz) of **5b**



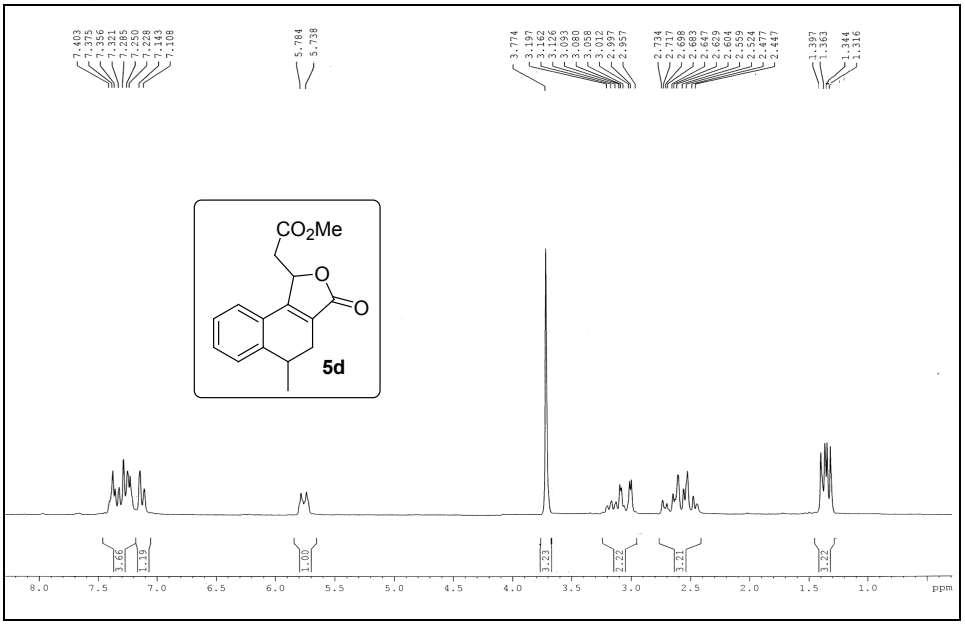


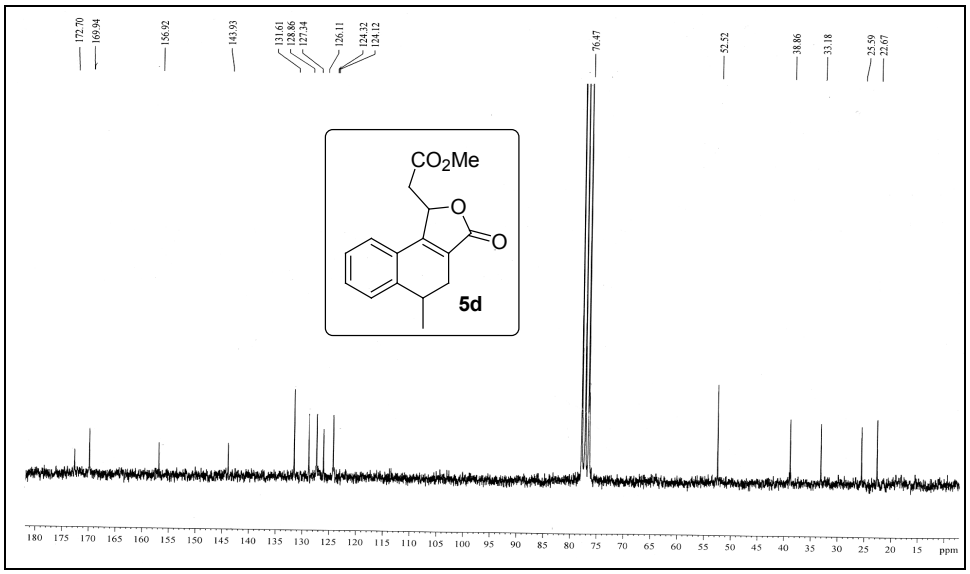
^{1}H NMR ($CDCl_3$, 200 MHz) and ^{13}C NMR ($CDCl_3$, 50 MHz) of **5c**



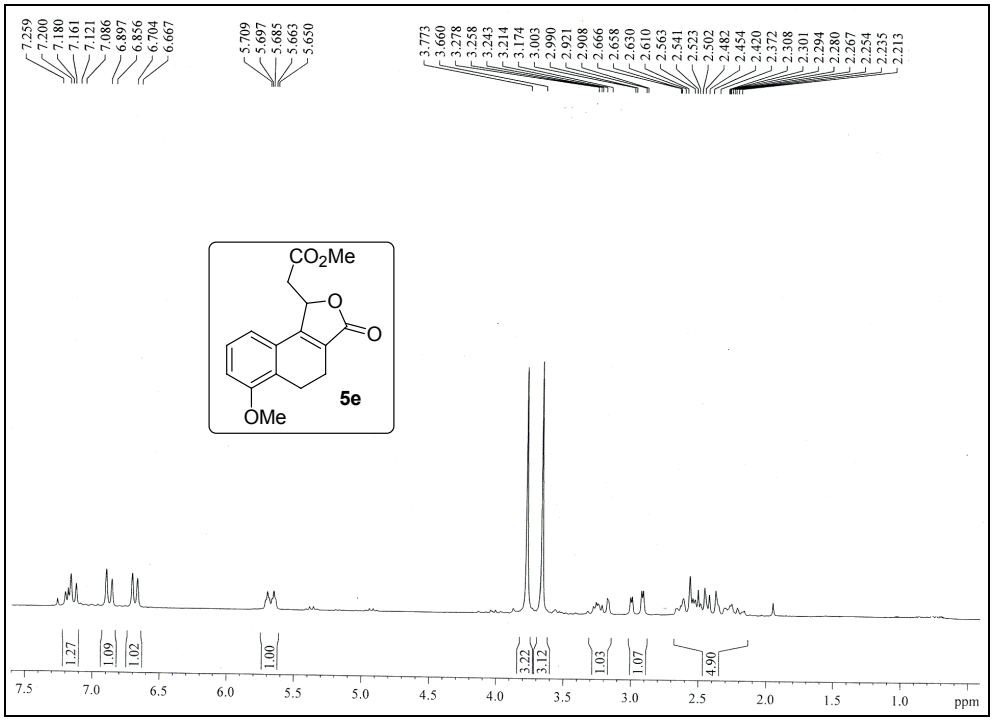


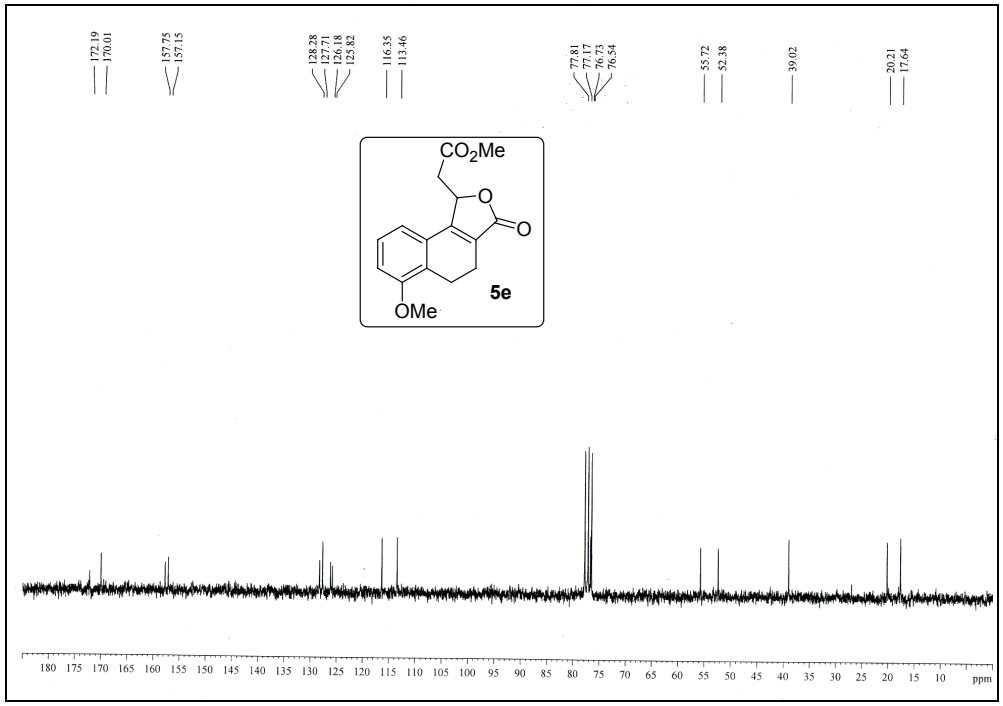
^{1}H NMR ($CDCl_3$, 200 MHz) and ^{13}C NMR ($CDCl_3$, 50 MHz) of **5d**



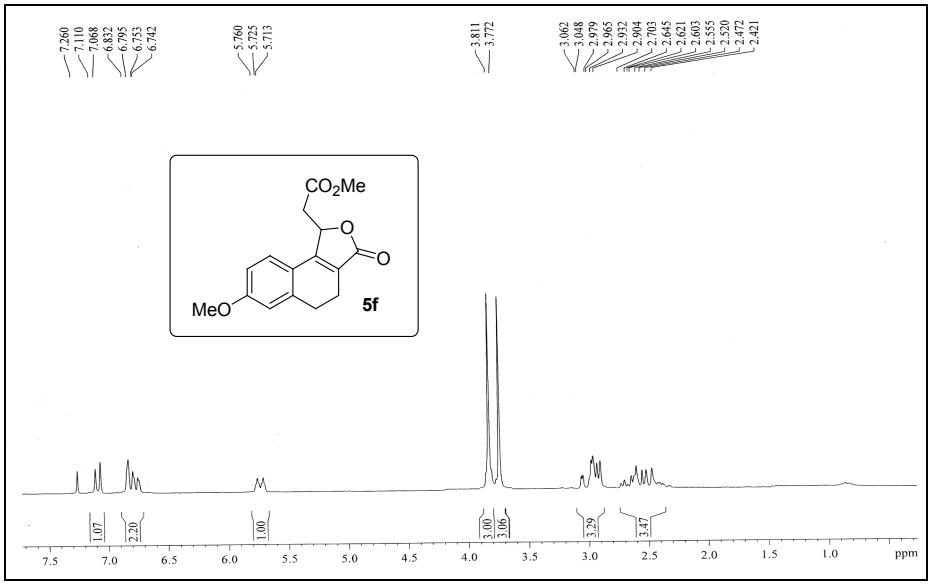


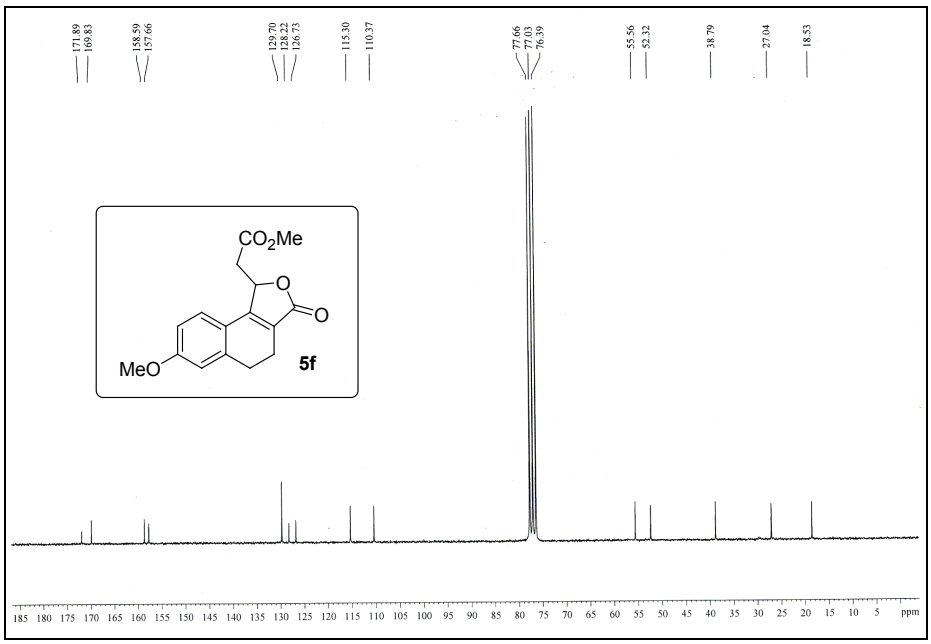
^{1}H NMR ($CDCl_3$, 200 MHz) and ^{13}C NMR ($CDCl_3$, 50 MHz) of **5e**



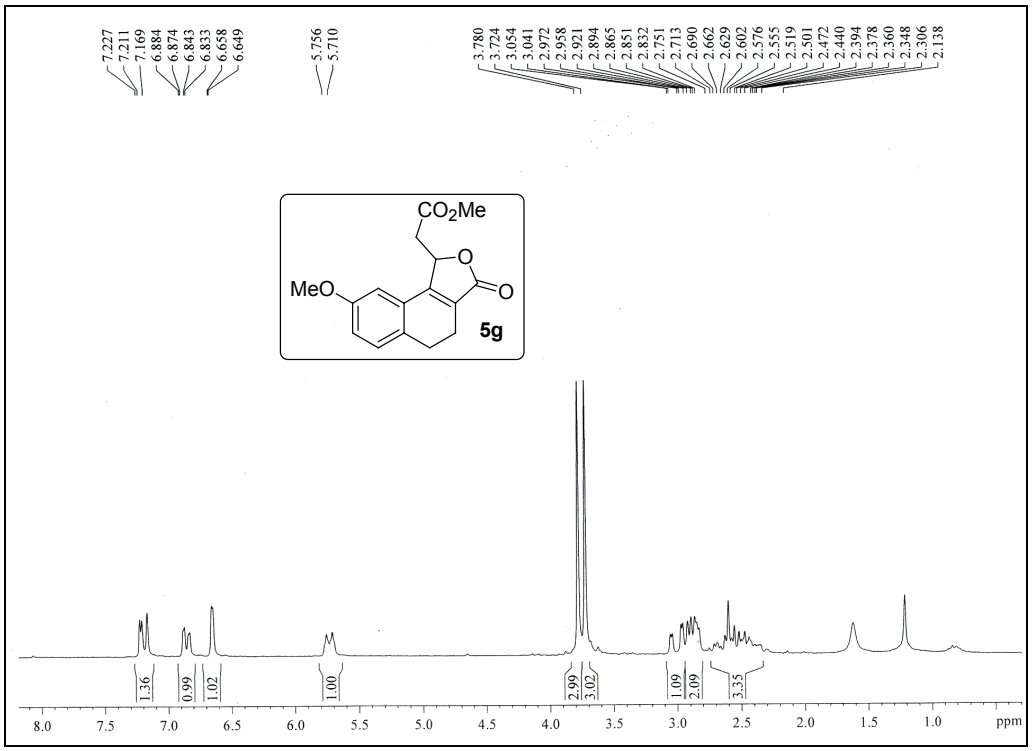


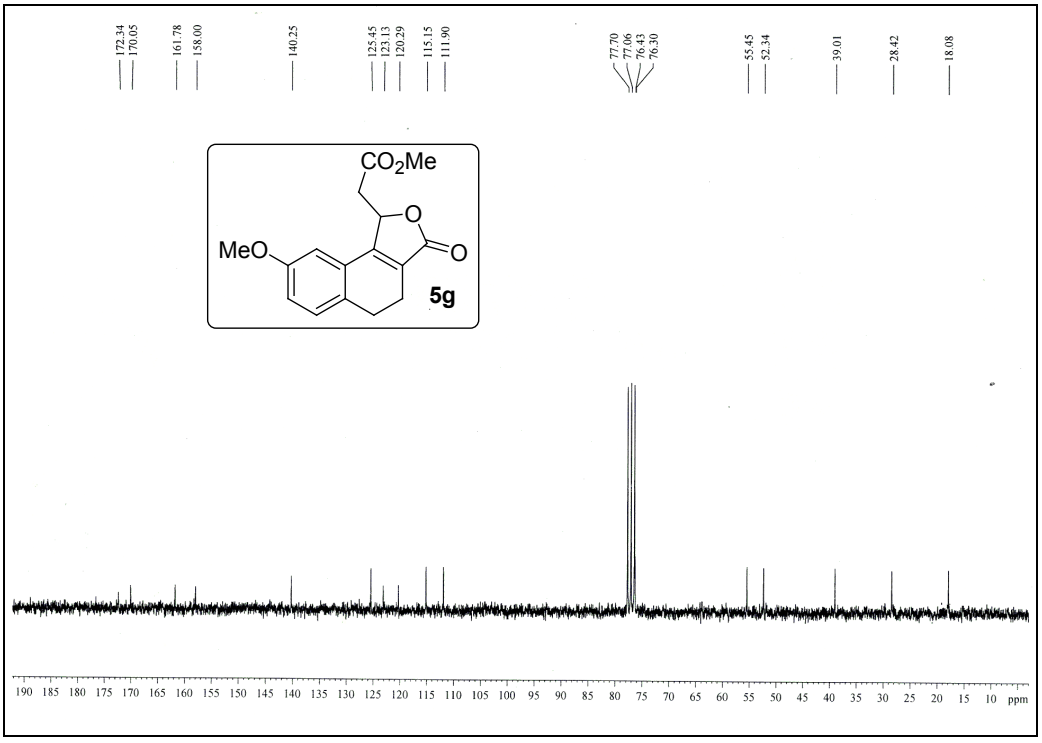
^{1}H NMR ($CDCl_3$, 200 MHz) and ^{13}C NMR ($CDCl_3$, 50 MHz) of **5f**



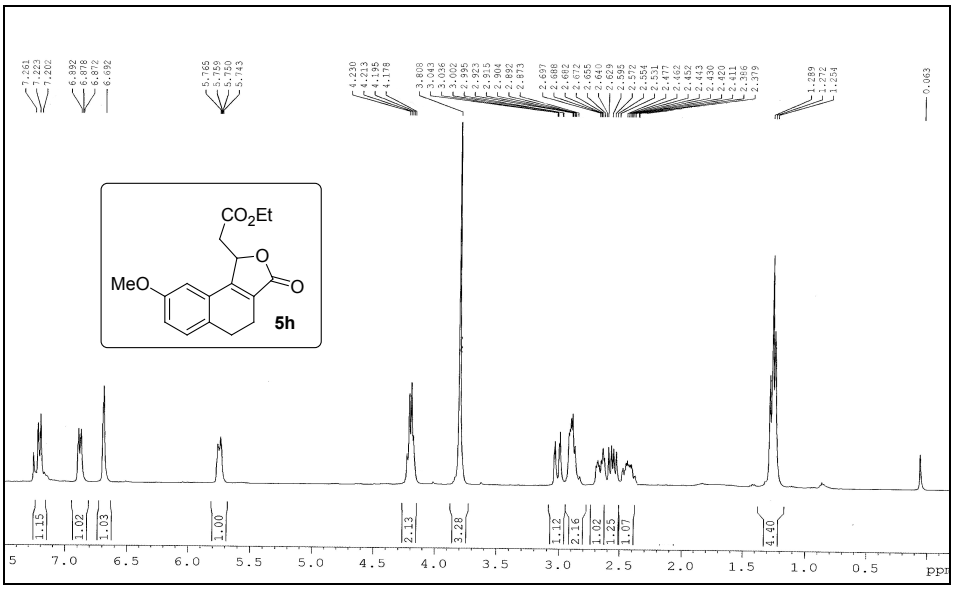


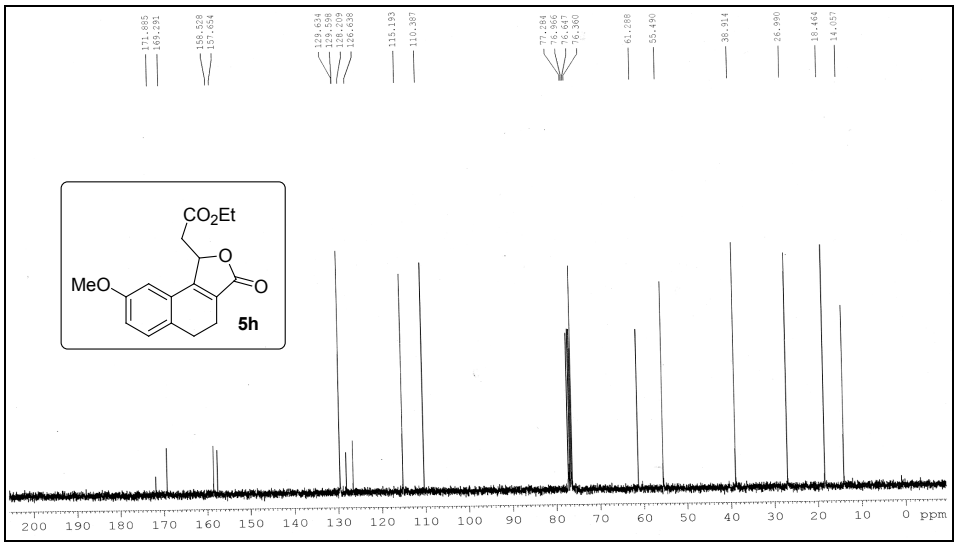
^{1}H NMR ($CDCl_3$, 200 MHz) and ^{13}C NMR ($CDCl_3$, 50 MHz) of **5g**



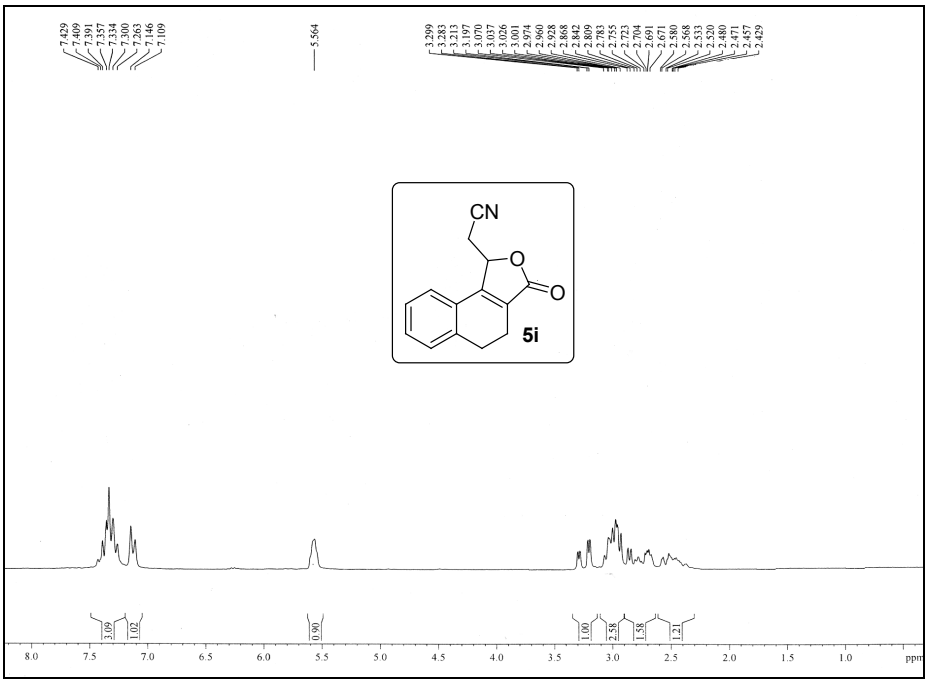


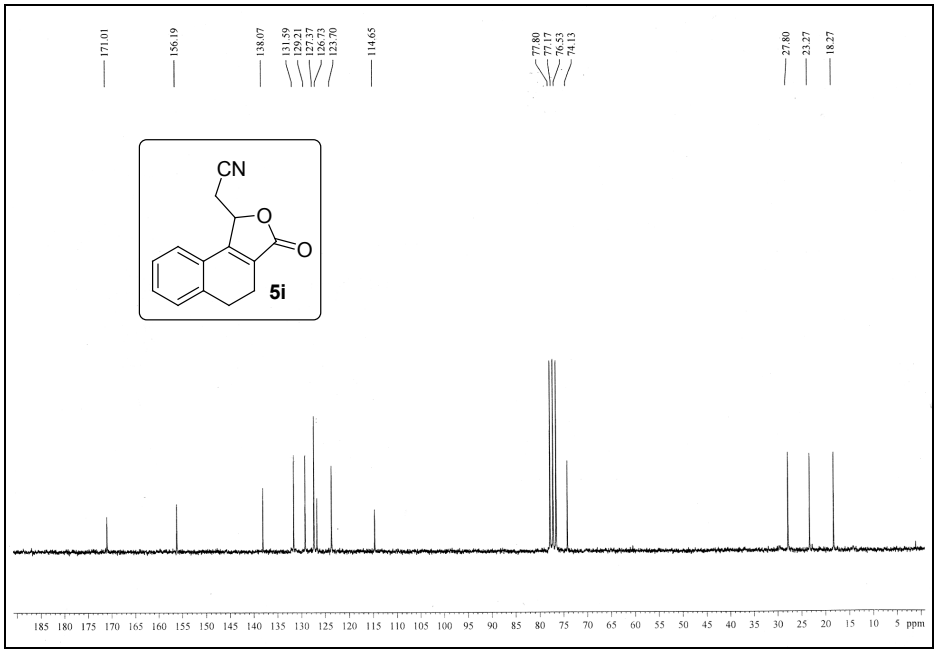
^{1}H NMR ($CDCl_3$, 400 MHz) and ^{13}C NMR ($CDCl_3$, 100 MHz) of **5h**



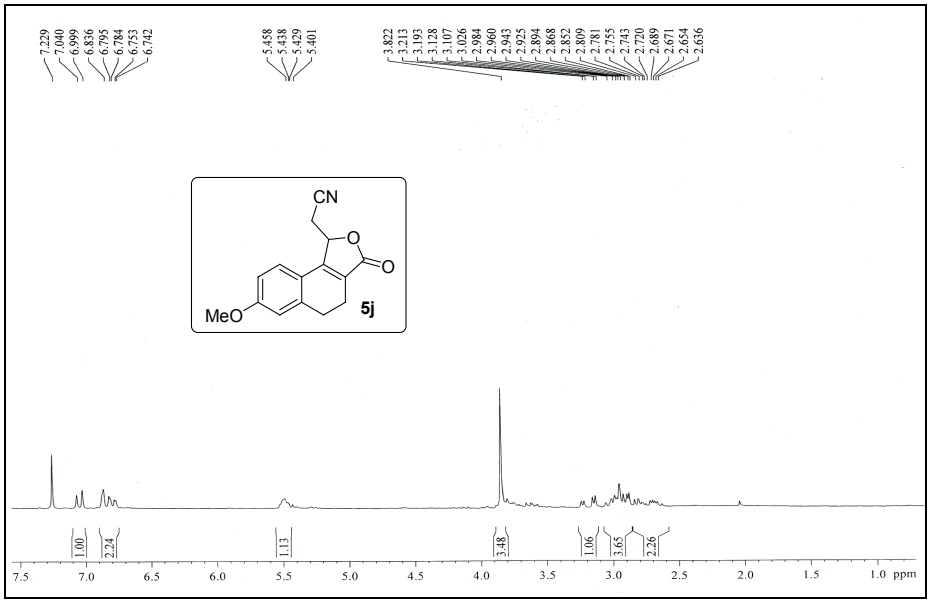


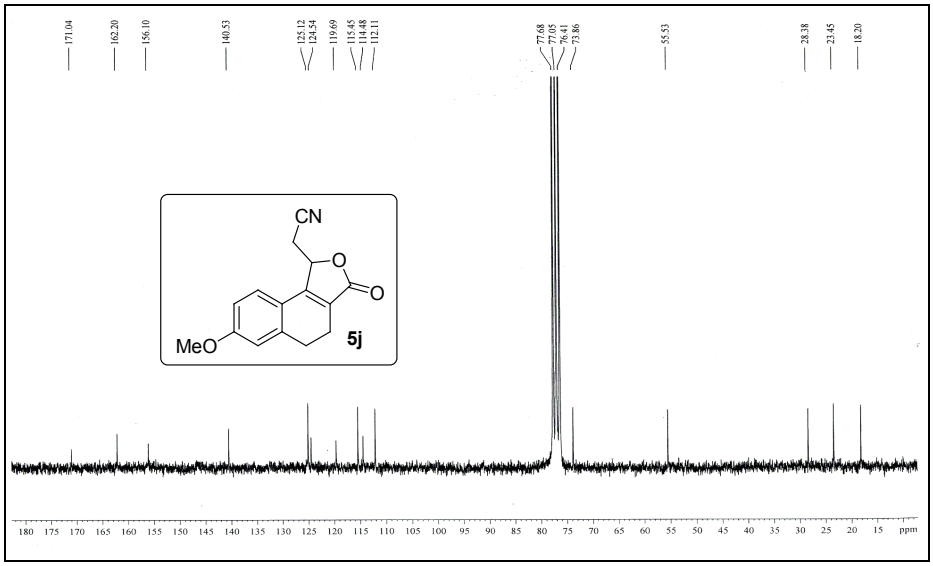
^{1}H NMR ($CDCl_3$, 200 MHz) and ^{13}C NMR ($CDCl_3$, 50 MHz) of **5i**





^{1}H NMR ($CDCl_3$, 200 MHz) and ^{13}C NMR ($CDCl_3$, 50 MHz) of **5j**





^{1}H NMR ($CDCl_3$, 200 MHz) and ^{13}C NMR ($CDCl_3$, 50 MHz) of **5k**