Supporting Information for:

Nickel-Catalyzed [2+2+1] Cycloaddition of Alkynes, Acrylates and Isocyanates

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Contents

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Instrumentation and Chemicals

All manipulations of oxygen- and moisture-sensitive materials were conducted in a dry box or with a standard Schlenk technique under a purified argon atmosphere. Nuclear magnetic resonance spectra were taken on Varian UNITY INOVA 500 (^{1}H, 500 MHz; ^{13}C, 125.7 MHz) spectrometer using tetramethylsilane (^{1}H) as an internal standard. ^{1}H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sext = sextet, sept = septet, br = broad, m = multiplet), coupling constants (Hz), integration, and identification. High-resolution mass spectra were obtained with a Thermo Fisher SCIENTIFIC EXACTIVE spectrometer. Preparative recycling gel permeation chromatography (GPC) was performed with JAI LC-908 equipped with JAIGEL-1H and -2H columns (toluene as an eluent). Infrared spectra (IR) spectra were determined on a SHIMADZU IR Affinity-1 spectrometer. Melting points were determined using a YANAKO MP-500D. TLC analyses were performed by means of Merck Kieselgel 60 F_{254} (0.25 mm) Plates. Visualization was accomplished with UV light (254 nm) and/or an aqueous alkaline $KMnO_4$ solution followed by heating. Flash column chromatography was carried out using Kanto Chemical silica gel (spherical, 40–50 μm). Unless otherwise noted, commercially available reagents were used without purification. 1,4-Dioxane was purchased from Wako Pure Chemical Co. stored over slices of sodium. Bis(1,5-cyclooctadiene)nickel and 1,3-bis(2,6-diisopropylphenyl)imidazol-2-ylidene were purchased from Strem Chemicals, Inc.

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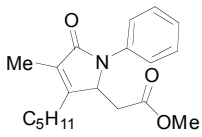
Experimental Procedure and Characterization Data for Products.

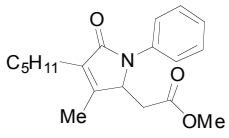
General procedure. The reaction was performed in a 15 mL sealed tube equipped with a Teflon-coated magnetic stirrer bar. A isocyanate (0.25 mmol), alkyne (1.0 mmol) and acrylate (0.25 mmol) were added to a solution of bis(1,5-dicyclooctadiene)nickel (7 mg, 0.025 mmol) and 1,3-bis(2,6-diisopropylphenyl)imidazol-2-ylidene (10 mg, 0.025 mmol) in 1,4-dioxane (1 mL) in a dry box. The flask was taken outside the dry box and heated at 100 °C for the indicated time under argon atmosphere. The resulting reaction mixture was cooled to ambient temperature and filtered through a silica gel pad, concentrated in vacuo. The residue was purified by flash silica gel column chromatography (20 g, 2x15 cm, hexane/ethyl acetate = 3:1) to give the corresponding product.

Methyl 2-(4-methyl-5-oxo-3-pentyl-1-phenyl-2,5-dihydro-1H-pyrrol-2-yl)acetate (4aaa).



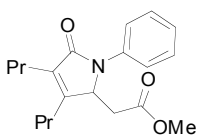
Yield: 63%, colorless oil. TLC: R_f 0.35(hexane/ethyl acetate = 3:1) ^{1}H NMR ($CDCl_3$) δ 7.49-7.46 (m, 2H; Ph–H), 7.41-7.37 (m, 2H; Ph–H), 7.18-7.15 (m, 1H; Ph–H), 4.98-4.95 (m, 1H; CH), 3.56 (s, 3H; CH_3), 2.63 (dd, *J* = 4.5, 15.5 Hz, 1H; CH-H), 2.55-2.47 (m, 1H; CH-H), 2.50 (dd, *J* = 7.5, 16 Hz, 1H; CH-H), 2.24-2.18 (m, 1H; CH-H), 1.87 (s, 3H; CH_3), 1.62-1.43 (m, 2H; CH_2), 1.38-1,27 (m, 4H; $2CH_2$), 0.92 (t, *J* = 7.0 Hz, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 170.7, 170.5, 153.3, 136.7, 129.4, 129.0, 124.9, 122.6, 58.6, 51.9, 35.7, 31.7, 28.1, 26.5, 22.3, 13.9, 8.8. IR (neat): 2954, 2871, 1737, 1694, 1599, 1501, 1381, 760 cm^{-1}. HRMS (ESI^+) found 316.1909, calcd for $[M+H]^+$ 316.1913.

Methyl 2-(3-methyl-5-oxo-4-pentyl-1-phenyl-2,5-dihydro-1H-pyrrol-2-yl)acetate (4aaa').



Yield: 13%, yellow oil. TLC: R_f 0.40 (hexane/ethyl acetate = 3:1). ^{1}H NMR ($CDCl_3$) δ 7.48-7.46 (m, 2H; Ph-H), 7.40-7.37 (m, 2H; PH-H), 7.17-7.14 (m, 1H; PH-H), 4.89-4.86 (m, 1H; CH), 2.71 (dd, *J* = 4.0, 15.5 Hz, 1H; CH-H), 2.50 (dd, *J* = 7.5, 16 Hz, 1H; CH-H), 2.31 (t, *J* = 7.0 Hz, 2H; CH_2), 2.00 (s, 3H; CH_3), 1.50 (quint, *J* = 8.0 Hz, 2H; CH2), 1.36-1.28 (m, 4H; $2CH_2$), 0.89 (t, *J* = 7.0 Hz, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 170.6, 170.0, 148.9, 136.7, 133.9, 129.0, 124.8, 122.4, 60.0, 51.8, 35.6, 31.6, 28.0, 23.5, 22.4, 14.0, 12.1. IR (neat): 2954, 2858, 1738, 1687, 1598, 1394, 1121,757 cm^{-1}. HRMS (ESI^+) found 316.1910, calcd for $[M+H]^+$ 316.1913.

Methyl 2-(5-oxo-1-phenyl-3,4-dipropyl-2,5-dihydro-1H-pyrrol-2-yl)acetate (4baa).



Yield: 66%, yellow oil. TLC: R_f 0.38 (hexane/ethyl acetate = 2:1). ^{1}H NMR ($CDCl_3$) δ 7.50-7.47 (m, 2H; Ph-H), 7.39-7.36 (m, 2H; Ph-H), 7.17-7.13 (m, 1H; Ph-H), 4.95 (dd, *J* = 2.0, 4.5 Hz, 1H; CH), 3.55 (s, 3H; CH_3), 2.64 (dd, *J*

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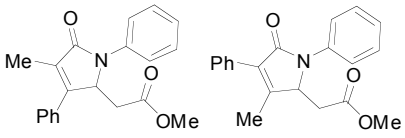
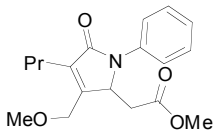
= 8.0, 15 Hz, 1H; CH-H), 2.55-2.49 (m, 1H; CH-H), 2.53 (dd, *J* = 7.0, 15.5 Hz, 1H; CH-H), 2.33-2.25 (m, 2H; CH_2), 2.25-2.18 (m, 1H; CH-H), 1.68-1.60 (m, 1H; CH-H), 1.56-1.52 (m, 2H; CH_2), 1.50-1.44 (m, 1H; CH-H), 0.98 (t, *J* = 7.5 Hz, 3H; CH_3), 0.94 (t, *J* = 7.5Hz, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 170.5, 170.0, 153.1, 136.7, 133.7, 128.9, 124.7, 122.4, 58.2, 51.7, 35.7, 28.4, 25.6, 22.0, 21.8, 14.1, 14.0. IR (neat): 2959, 2872, 1738, 1694, 1599, 1501, 1381, 757 cm^{-1}. HRMS (ESI^+) found 316.1908, calcd for $[M+H]^+$ 316.1913.

Methyl 2-(4-methyl-5-oxo-1,3-diphenyl-2,5-dihydro-1H-pyrrol-2-yl)acetate (4caa) and methyl 2-(3-methyl-5-oxo-1,4-diphenyl-2,5-dihydro-1H-pyrrol-2-yl)acetate (4caa') (2:1 mixture).



Yield: 56%, colorless oil. TLC: R_f 0.20 (hexane/ethyl acetate = 2:1). ^{1}H NMR ($CDCl_3$) δ 7.56-7.39 (m, 12.5H; Ph-H), 7.37-7.33 (m, 1H; Ph-H), 7.22-7.18 (m, 1.5H; Ph-H), 5.52-5.49 (m, 1H; CH), 5.05-5.03 (m, 0.5H; CH), 3.61 (s, 1.5H; CH_3), 3.29 (s, 3H; CH_3), 2.80 (dd, *J* = 4.5, 16 Hz, 0.5H; CH-H), 2.65 (dd, *J* = 7.0, 15.5 Hz, 0.5H; CH-H), 2.51 (dd, *J* = 5.0, 15 Hz, 1H; CH-H), 2.46 (dd, *J* = 6.0, 15.5 Hz, 1H; CH-H), 2.19 (s, 1.5H; CH_3), 2.07 (d, *J* = 1.8 Hz, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 174.9, 170.4, 170.4, 170.1, 168.6, 150.9, 150.6, 136.6, 132.9, 132.2, 131.0, 130.3, 129.3, 129.1,129.1, 129.0, 128.7, 128.4, 128.2, 128.0, 125.3, 125.2, 123.1, 122.8, 60.3, 59.2, 52.0, 51.6, 36.7, 35.6, 13.3, 10.0. IR (neat): 3060, 2952, 1738, 1729, 1694, 1674, 1597, 1494, 1385, 1176, 759, 696 cm^{-1}. HRMS (ESI^+) found 322.1438, calcd for $[M+H]^+$ 322.1443.

Methyl 2-(3-(methoxymethyl)-5-oxo-1-phenyl-4-propyl-2,5-dihydro-1H-pyrrol-2-yl) acetate (4daa).



Yield: 23%, yellow oil. TLC: R_f 0.41 (hexane/ethyl acetate = 2:1). ^{1}H NMR ($CDCl_3$) δ 7.49- 7.47 (m, 2H; Ph-H), 7.41-7.38 (m, 2H; Ph-H), 7.19-7.16 (m, 1H; Ph-H), 5.07 (dd, *J* = 3.5, 3.5 Hz, 1H; CH), 4.33 (dd, *J* = 13, 14.5 Hz, 2H; CH_2), 3.56 (s, 3H; CH_3), 3.37 (s, 3H; CH_3), 2.72 (dd, *J* = 4.0, 15 Hz, 1H; CH-H), 2.62 (dd, *J* = 7.0, 15.5 Hz, 1H; CH-H), 2.40-2.26 (m, 2H; CH_2), 1.59-1.52 (m, 2H; CH_2), 0.93 (t, *J* = 7.5 Hz, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 170.2, 169.4, 148.6, 136.5, 135.7, 129.0, 125.1, 122.7, 66.5, 58.7, 58.3, 51.6, 35.4, 25.7, 21.9, 13.8. IR (neat): 2958, 2873, 1728, 1678, 1598, 1500, 1172, 759, 694 cm^{-1}. HRMS (ESI^+) found 318.1700, calcd for $[M+H]^+$ 318.1705.

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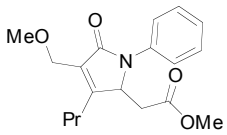
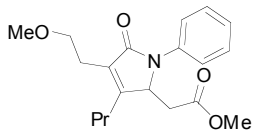
Methyl 2-(4-(methoxymethyl)-5-oxo-1-phenyl-3-propyl-2,5-dihydro-1H-pyrrol-2-yl)acetate (4daa').



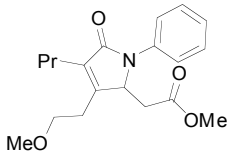
Yield: 23%, yellow oil. TLC: R_f 0.15 (hexane/ethyl acetate = 2:1). ^{1}H NMR ($CDCl_3$) δ 7.47-7.46 (m, 2H; Ph-H), 7.40-7.36 (m, 2H; Ph-H), 7.19-7.16 (m, 1H; Ph-H), 5.03 (dd, *J* = 4.5, 7.0 Hz, 1H; CH), 4.23 (d, *J* = 12, 1H; CH-H), 4.20 (d, *J* = 12 Hz, 1H; CH-H), 3.57 (s, 3H; CH_3), 3.39 (s, 3H; CH_3), 2.74-2.68 (m, 1H; CH-H), 2.66 (dd, *J* = 4.5, 15.5 Hz, 1H; CH-H), 2.56 (dd, *J* = 7.0, 16 Hz, 1H; CH-H), 2.29-2.24 (m, 1H; CH-H), 1.69-1.64 (m, 1H; CH-H), 1.57-1.50 (m, 1H; CH-H), 0.99 (t, *J* = 7.5 Hz, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 170.4, 169.1, 159.0, 136.4, 130.0, 129.0, 125.1, 122.7, 63.5, 58.8, 58.5, 35.5, 28.6, 22.1, 14.1. IR (neat): 2960, 2874, 1738, 1687, 1598, 1386, 1096, 695 cm^{-1}. HRMS (ESI^+) found 318.1701, calcd for $[M+H]^+$ 318.1705

Methyl 2-(4-(2-methoxyethyl)-5-oxo-1-phenyl-3-propyl-2,5-dihydro-1H-pyrrol-2-yl)acetate (4eaa).



Yield: 46%, yellow oil. TLC: R_f 0.32 (hexane/ethyl acetate = 2:1). ^{1}H NMR ($CDCl_3$) δ 7.49-7.46 (m, 2H; Ph-H), 7.40-7.36 (m, 2H; Ph-H), 7.18-7.15 (m, 1H; Ph-H), 4.99 (dd, *J* = 4.0, 6.5 Hz, 1H; CH), 3.56 (s, 3H; CH_3), 3.52 (dt, *J* = 0.5, 6.5 Hz, 2H; CH_2), 3.33 (s, 3H; CH_3), 2.67-2.50 (m, 5H; 3CH-H, CH_2), 2.24-2.19 (m, 1H; CH-H), 1.69-1.59 (m, 1H;CH-H), 1.54-1.44 (m, 1H; CH-H), 0.99 (t, *J* = 7.5 Hz, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 170.5, 169.8, 155.3, 136.6, 130.3, 129.0, 124.9, 122.5, 70.5, 58.5, 58.5, 51.8, 35.6, 28.5, 24.5, 22.1, 14.2. IR (neat): 2959, 2875, 1737, 1661, 1599, 1494, 1367, 758, 694 cm^{-1}. HRMS (ESI^+) found 332.1857, calcd for $[M+H]^+$ 332.1862.

Methyl 2-(3-(2-methoxyethyl)-5-oxo-1-phenyl-4-propyl-2,5-dihydro-1H-pyrrol-2-yl)acetate (4eaa').



Yield: 23%, yellow oil. TLC: R_f 0.35 (hexane/ethyl acetate = 2:1). ^{1}H NMR ($CDCl_3$) δ 7.49-7.47 (m, 2H; Ph-H), 7.40-7.37 (m, 2H; Ph-H), 7.18-7.15 (m, 1H; Ph-H), 4.99 (dd, *J* = 5.0, 5.0 Hz, 1H; CH), 3.59-3.55 (m, 1H; CH-H), 3.53 (s, 3H; CH_3), 3.52-3.47 (m, 1H; CH-H), 3.36 (s, 3H; CH_3), 2.85 (dt, *J* = 6.0, 14.5 Hz, 1H; CH-H), 2.68 (dd, *J* = 4.0, 15 Hz, 1H; CH-H), 2.64 (dd, *J* = 6.0, 15 Hz, 1H; CH-H), 2.54 (m, 1H; CH-H), 2.29 (m, 1H; CH-H), 1.54 (m, 2H; CH_2), 0.95 (t, *J* = 7.0 Hz, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 170.4, 169.8, 150.4, 136. 7, 135.0, 128.9, 124.9, 122.7, 71.0, 59.2, 58.7, 51.7, 35.3, 27.0, 25.8, 21.9, 14.0. IR (neat): 2957, 2873, 1737, 1694, 1598, 1500, 1112, 759, 694 cm^{-1}. HRMS (ESI^+) found 332.1857, calcd for $[M+H]^+$ 332.1862.

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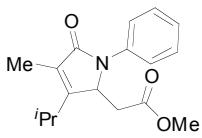
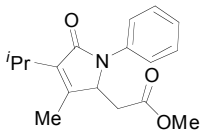
Methyl 2-(3-isopropyl-4-methyl-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrol-2-yl) acetate (4faa).



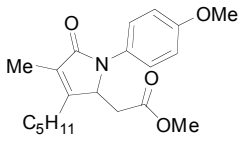
Yield: 63%, colorless crystal. Mp. 68-70 °C (dichloromethane). TLC: R_f 0.22 (hexane/ethyl acetate = 3:1). ^{1}H NMR ($CDCl_3$) δ 7.46-7.44 (m, 2H; Ph-H), 7.39-7.36 (m, 2H; Ph-H), 7.18-7.14 (m, 1H; Ph-H), 4.96-4.94 (m, 1H; CH), 3.50 (s, 3H; CH_3), 2.76 (sept, J = 7.0 Hz, 1H; CH), 2.65 (dd, J = 5.5, 16 Hz, 1H; CH-H), 2.58 (dd, J = 5.0, 16 Hz, 1H; CH-H), 1.94 (d, J = 1.0, 3H; CH_3), 1.30 (d, J = 7.0 Hz, 3H; CH_3), 1.26 (d, J = 7.0, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 170.6, 170.6, 157.3, 136.5, 128.9, 128.5, 125.0, 123.0, 59.0, 51.8, 35.7, 27.7, 21.3, 20.5, 9.4. IR (KBr): 2964, 2919, 2871, 1733, 1662, 1502, 1434, 1265, 760, 699 cm^{-1}. HRMS (ESI^+) found 288.1593, calcd for $[M+H]^+$ 288.1600.

Methyl 2-(4-isopropyl-3-methyl-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrol-2-yl) acetate (4faa').



Yield: 9%, colorless oil. TLC: R_f 0.33 (hexane/ethyl acetate = 3:1). ^{1}H NMR ($CDCl_3$) δ 7.47-7.45 (m, 2H; Ph-H), 7.39-7.36 (m, 2H; Ph-H), 7.17-7.14 (m, 1H; Ph-H), 4.81 (dd, J = 4.0, 7.0 Hz, 1H; CH), 3.57 (s, 3H; CH_3), 2.89 (sept, J = 7.0 Hz, 1H; CH), 2.66 (dd, J = 4.0, 15 Hz, 1H; CH-H), 2.51 (dd, J = 7.0, 15 Hz, 1H; CH-H), 2.02 (d, J = 0.5 Hz, 3H; CH_3), 1.28 (d, J = 7.7 Hz, 3H; CH_3), 1.24 (d, J = 7.0 Hz, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 170.5, 169.4, 147.5, 137.9, 136.6, 128.9, 124.8, 122.5, 59.9, 51.8, 35.6, 25.1, 20.5, 20.3, 12.1. IR (neat): 2962, 2932, 1736, 1688, 1501, 1392, 757, 694 cm^{-1}. HRMS (ESI^+) found 288.1596, calcd for $[M+H]^+$ 288.1600.

Methyl 2-(1-(4-methoxyphenyl)-4-methyl-5-oxo-3-pentyl-2,5-dihydro-1H-pyrrol-2-yl)acetate (4aab).



Yield: 47%, colorless oil. TLC: R_f 0.27 (hexane/ethyl acetate = 2:1). ^{1}H NMR ($CDCl_3$) δ 7.33-7.30 (m, 2H; Ph-H), 6.92-6.89 (m, 2H; Ph-H), 4.87-4.84 (m, 1H; CH), 3.79 (s, 3H; CH_3), 3.53 (s, 3H; CH_3), 2.60-2.47 (m, 3H; 3CH-H), 2.22-2.18 (m, 1H; CH-H), 1.85 (s, 3H; CH_3), 1.58-1.54 (m, 1H; CH-H), 1.49-1.41 (m, 1H; CH-H), 1.37- 1.29 (m, 4H; $2CH_2$), 0.91 (t, J = 6.5 Hz, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 170.6, 170,5, 157.2, 152.9, 129.3, 128.9, 125.0, 114.2, 59.3, 55.4, 51.7, 36.7, 31.7, 28.1, 26.4, 22.3,13.8, 8.7. IR (neat): 2955, 2870, 1737, 1682, 1514, 1248, 1170, 830 cm^{-1}. HRMS (ESI^+) found 346.2013, calcd for $[M+H]^+$ 346.2018.

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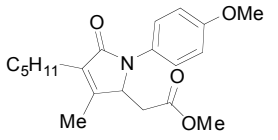
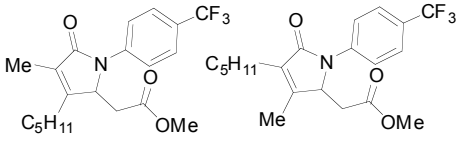
4-Methyl-3-pentyl-1*H*-isochromen-1-one (3ac').



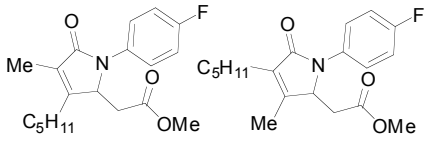
Yield: 9%, colorless oil. TLC: R_f 0.33 (hexane/ethyl acetate = 2:1). ^{1}H NMR ($CDCl_3$) δ 7.34-7.32 (m, 2H; Ph-H), 6.92-6.90 (m, 2H; Ph-H), 4.77 (dd, *J* = 5.0, 6.5 Hz, 1H; CH), 3.80 (s, 3H; CH_3), 3.56 (s, 3H; CH_3), 2.62 (dd, *J* = 4.5, 15.5 Hz, 1H; CH-H), 2.49 (dd, *J* = 7.0, 15.5 Hz, 1H; CH-H), 2.29 (t, *J* = 7.0 Hz, 2H; CH_2), 1.99 (s, 3H; CH_3), 1.53-1.47 (m, 2H; CH_2), 1.35-1.24 (m, 4H; $2CH_2$), 0.89 (t, *J* = 7.0, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 170.63, 170.1, 157.1, 148.5, 133.9, 129.7, 124.8, 114.3, 60.7, 55.4, 51.8, 35.7, 31.6, 28.0, 23.6, 22.4, 14.0, 12.1. IR (neat): 2956, 2871, 1737, 1667, 1514, 1248, 829, 805 cm^{-1}. HRMS (ESI^+) found 346.2013, calcd for $[M+H]^+$ 346.2018.

Methyl 2-(4-methyl-5-oxo-3-pentyl-1-(4-(trifluoromethyl)phenyl)-2,5-dihydro-1H-pyrrol-2-yl)acetate (4aac) and methyl 2-(3-methyl-5-oxo-4-pentyl-1-(4-(trifluorome thyl)phenyl)-2,5-dihydro-1H-pyrrol-2-yl)acetate (4aac') (1:1 mixture).



Yield: 61%, colorless oil. TLC: R_f 0.36 (hexane/ethyl acetate = 3:1). ^{1}H NMR ($CDCl_3$) δ 7.68-7.63 (m, 8H; Ph-H), 5.01-5.00 (m, 1H; CH), 4.92-4.90 (m, 1H; CH), 2.74 (dd, *J* = 3.5, 15.5 Hz, 1H; CH-H), 2.67 (dd, *J* = 4.0, 16Hz, 1H; CH-H), 2.55-2.50 (m, 3H; 3CH-H), 2.32-2.29 (m, 2H; CH_2), 2.25-2.19 (m, 1H; CH-H), 2.02 (s, 3H; CH_3), 1.88 (s, 3H; CH_3), 1.60-1.45 (m, 4H; $2CH_2$), 1.39-1.28 (m, 8H; $4CH_2$), 0.93-0.88 (m, 6H; $2CH_3$). ^{13}C NMR ($CDCl_3$) δ 170.5, 170.4, 170.3, 170.1, 154.1, 149.7, 139.9, 133.9, 129.3, 126.2, 126.2, 125.1 (q, *J* = 188 Hz), 123.0, 121.3, 121.2, 59.6, 58.2, 52.0, 52.0, 50.9, 35.4, 35.3, 33.5, 31.7, 31.6, 31.0, 29.7, 28.0, 26.7, 26.5, 23.5, 22.4, 22.4, 22.3, 13.9, 13.9, 12.1, 8.7. IR (neat): 2956, 2929, 1731, 1701, 1692, 1681, 1614, 1378, 1325, 1164, 1120, 1067 cm^{-1}. HRMS (ESI^+) found 384.1777, calcd for $[M+H]^+$ 384.1787.

Methyl 2-(1-(4-fluorophenyl)-4-methyl-5-oxo-3-pentyl-2,5-dihydro-1H-pyrrol-2-yl)acetate (4aad) and Methyl 2-(1-(4-fluorophenyl)-3-methyl-5-oxo-4-pentyl-2,5-dihydro-1H-pyrrol-2-yl)acetate (4aad') (5:1 mixture).



Yield: 66%, yellow oil. TLC: R_f 0.30 (hexane/ethyl acetate = 3:1). ^{1}H NMR ($CDCl_3$) δ 7.43-7.39 (m, 2.4H; Ph-H), 7.09-7.05 (m, 2.4H; Ph-H), 4.91-4.89 (m, 1H; CH), 4.80 (dd, *J* = 5.5, 7.0 Hz, 0.2H; CH), 3.57 (s, 0.6H; CH_3), 3.55 (s, 3H; CH_3), 2.64-2.49 (m, 3.4H; CH-H), 2.29 (dd, *J* = 7.5, 8.0 Hz, 0.4H; CH_2), 2.24-2.18 (m, 1H; CH-H), 2.00 (s, 0.6H; CH_3), 1.86 (s, 3H; CH_3), 1.60-1.55 (m, 1H; CH-H), 1.50-1.42 (m, 1.4H; CH-H,CH_2), 1.4o-1.29 (m, 4.8H; CH_2), 0.90-0.89 (m, 3.6H; CH_3). ^{13}C NMR ($CDCl_3$) δ 170.5, 170.5, 161.0,

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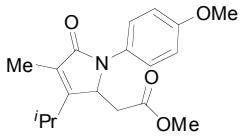
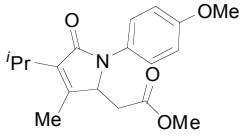
159.0, 153.3, 148.8, 133.8, 132.7, 132.7, 129.3, 124.8, 124.7, 124.6, 124.5, 115.8, 115.7, 60.5, 59.1, 51.9, 35.6, 35.6, 31.7, 31.6, 28.1, 28.0, 26.5, 23.5, 22.4, 223, 13.9, 13.9, 12.1, 8.7. IR (neat): 2955, 2932, 2872, 1738, 1733, 1694, 1674, 1511, 1383, 1222, 1157, 835 cm^{-1}. HRMS (ESI^+) found 334.1813, calcd for $[M+H]^+$ 334.1818.

Methyl 2-(3-isopropyl-1-(4-methoxyphenyl)-4-methyl-5-oxo-2,5-dihydro-1H-pyrrol-2-yl)acetate (4fab).



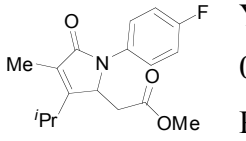
Yield: 47%, colorless crystal. Mp. 70-72 °C (dichloromethane). TLC: R_f 0.25 (hexane/ethyl acetate = 2:1). ^1H NMR ($CDCl_3$) δ 7.32-7.28 (m, 2H; Ph-H), 6.92-6.88 (m, 2H; Ph-H), 4.87-4.84 (m, 1H; CH), 3.79 (s, 3H; CH_3), 3.48 (s, 3H; CH_3), 2.76 (sept, J = 7.5 Hz, 1H; CH), 2.64 (dd, J = 5.5, 16 Hz, 1H; CH-H), 2.51 (dd, J = 5.5, 16Hz, 1H; CH-H), 1.93 (d, J = 2.0 Hz, 3H; CH_3), 1.28 (d, J = 7.5 Hz, 3H; CH_3), 1.25 (d, J = 7.5, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 170.7, 170.6, 157.3, 157.0, 129.5, 128.5, 125.4, 114.2, 59.7, 55.4, 51.7, 35.9, 27.7, 21.4, 20.5, 9.4. IR (KBr): 2966, 2930, 1732, 1682, 1516, 1437, 1246, 1036, 840 cm^{-1}. HRMS (ESI^+) found 318.1699, calcd for $[M+H]^+$ 318.1705

Methyl 2-(4-isopropyl-1-(4-methoxyphenyl)-3-methyl-5-oxo-2,5-dihydro-1H-pyrrol-2-yl)acetate (4fab').



Yield: 7%, yellow oil. TLC: R_f 0.52 (hexane/ethyl acetate = 2:1). ^1H NMR ($CDCl_3$) δ 7.33-7.30 (m, 2H; Ph-H), 6.92-6.89 (m, 2H; Ph-H), 4.71 (dd, J = 5.0, 6.0 Hz, 1H; CH), 3.79 (s, 3H; CH_3), 3.55 (s, 3H; CH_3), 2.89 (sept, J = 7.0 Hz, 1H; CH), 2.60 (dd, J = 4.5, 15 Hz, CH-H), 2.52 (dd, J = 6.5, 15Hz, 1H; CH-H), 2.01 (d, J = 0.5 Hz, 3H; CH_3),1.26 (d, J = 7.0 Hz, 3H; CH_3), 1.25 (d, J = 7.0 Hz, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 170.5, 169.5, 157.1, 147.1, 138.0, 129.6, 124.9, 114.2, 60.6, 55.4, 51.7, 35.7, 25.2, 20.5, 20.4, 12.1. IR (neat): 2961, 2936, 1737, 1682, 1514, 1248, 1034, 831 cm^{-1}. HRMS (ESI^+) found 318.1699, calcd for $[M+H]^+$ 318.1705.

Methyl 2-(1-(4-fluorophenyl)-3-isopropyl-4-methyl-5-oxo-2,5-dihydro-1H-pyrrol-2-yl)acetate (4fac).



Yield: 53%, colorless crystal. Mp. 108-109 °C (dichloromethane). TLC: R_f 0.27 (hexane/ethyl acetate = 3:1). ^1H NMR ($CDCl_3$) δ 7.41-7.37 (m, 2H; Ph-H), 7.09-7.04 (m, 2H; Ph-H), 4.90-4.88 (m, 1H; CH), 3.50 (s, 3H; CH_3), 2.77 (sept, J = 2.0 Hz, 1H; CH), 2.66 (dd, J = 5.5, 16 Hz, 1H; CH-H), 2.53 (dd, J = 5.0, 16 Hz, 1H; CH-H), 1.94 (d, J = 1.5 Hz, 3H; CH_3), 1.29 (d, J = 7.0 Hz, 3H; CH_3), 1.25 (d, J = 7.5 Hz, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 170.7, 170.4, 161.1 (d, J = 243 Hz), 157.3, 132.6, 128.5,

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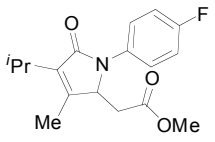
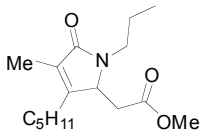
125.2 (d, J = 8.1 Hz), 115.8 (d, J = 22.3 Hz), 59.4, 51.8, 35.7, 27.7, 21.3, 20.4, 9.4. IR (KBr): 2966, 2929, 1736, 1673, 1509, 1217, 1158, 842, 757 cm^{-1}. HRMS (ESI^+) found 306.1500, calcd for $[M+H]^+$ 306.1505.

Methyl 2-(1-(4-fluorophenyl)-4-isopropyl-3-methyl-5-oxo-2,5-dihydro-1H-pyrrol-2-yl)acetate (4fac').



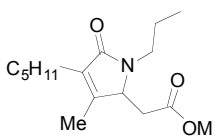
Yield: 8%, colorless oil. TLC: R_f 0.32 (hexane/ethyl acetate = 3:1). ^1H NMR ($CDCl_3$) δ 7.41-7.38 (m, 2H; Ph-H), 7.09-7.05 (m, 2H; Ph-H), 4.74 (dd, J = 5.0, 6.5 Hz, 1H; CH), 3.57 (s, 3H; CH_3), 2.89 (sept, J = 7.0 Hz, 1H; CH), 2.60 (dd, J = 4.5, 15.5 Hz, 1H; CH-H), 2.54 (dd, J = 6.5, 15.5 Hz, 1H; CH-H), 2.02 (d, J = 0.5 Hz, 3H; CH_3), 1.27 (d, J = 7.0 Hz, 3H; CH_3), 1.25 (d, J = 7.0 Hz, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 170.3, 169.5, 160.9 (d, J = 243 Hz), 147.5, 137.9, 132.7, 124.8 (d, J = 8.1 Hz), 115.8 (d, J = 22.3 Hz), 60.3, 51.8, 35.5, 25.2, 20.4, 20.3, 12.1. IR (neat): 2961, 2931, 1737, 1687, 1511, 1392, 1222, 1065, 835 cm^{-1}. HRMS (ESI^+) found 306.1499, calcd for $[M+H]^+$ 306.1505.

Methyl 2-(4-methyl-5-oxo-3-pentyl-1-propyl-2,5-dihydro-1H-pyrrol-2-yl)acetate (4aae).



Yield: 24%, yellow oil. TLC: R_f 0.13 (hexane/ethyl acetate = 2:1). ^1H NMR ($CDCl_3$) δ 4.32-4.29 (m, 1H; CH), 3.76-3.70 (m, 1H; CH-H), 3.69 (s, 3H; CH_3), 2.97-2.92 (m, 1H; CH-H), 2.61 (dd, J = 5.5, 16 Hz, 1H; CH-H), 2.48 (dd, J = 6.5, 16 Hz, 1H; CH), 2.45-2.41 (m, 1H; CH-H), 2.17-2.11 (m, 1H; CH-H), 1.79 (s, 3H; CH_3), 1.63-1.55 (m, 1H; CH-H), 1.53-1.45 (m, 2H; CH_2), 1.43-1.37 (m, 1H; CH-H), 1.36-1.24 (m, 4H; $2CH_2$), 0.89 (t, J = 7.5 Hz, 3H; CH_3), 0.86 (t, J = 7.5 Hz, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 172.0, 171.0, 152.3, 129.2, 57.7, 52.0, 41.8, 35.5, 31.6, 28.1, 26.3, 22.3, 21.7, 13.9, 11.2, 8.8. IR (neat): 2958, 2932, 2873, 1737, 1686, 1455, 1415, 1159, 1095 cm^{-1}. HRMS (ESI^+) found 282.2064, calcd for $[M+H]^+$ 282.2069.

Methyl 2-(3-methyl-5-oxo-4-pentyl-1-propyl-2,5-dihydro-1H-pyrrol-2-yl)acetate.



Yield: 5%, yellow oil. TLC: R_f 0.19 (hexane/ethyl acetate = 2:1). ^1H NMR ($CDCl_3$) δ 4.20 (dd, J = 5.5, 5.5 Hz, 1H; CH), 3.78-3.72 (m, 1H; CH-H), 3.67 (s, 3H; CH_3), 2.96-2.91 (m, 1H; CH-H), 2.61 (dd, J = 6.0, 15.5 Hz, 1H; CH-H), 2.55 (dd, J = 6.0, 15.5 Hz, 1H; CH-H), 2.24-2.19 (m, 2H; CH_2), 1.91 (s, 3H; CH_3), 1.61-1.55 (m, 1H; CH-H), 1.52-1.41 (m, 3H; CH-H,CH_2), 1.33-1.23 (m, 4H; $2CH_2$), 0.88-0.85 (m, 6H; $2CH_3$). ^{13}C NMR ($CDCl_3$) δ 171.6, 170.8, 147.9, 133.7, 59.2, 51.9, 41.5, 35.4, 31.6, 28.0, 23.6, 22.4, 21.7, 13.9, 11.9, 11.2. IR (neat): 2958, 2931, 1737, 1678, 1435, 1416, 1250, 1156 cm^{-1}. HRMS (ESI^+) found 282.2064, calcd for $[M+H]^+$ 282.2069.

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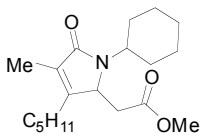
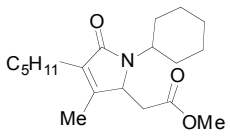
Methyl 2-(1-cyclohexyl-4-methyl-5-oxo-3-pentyl-2,5-dihydro-1H-pyrrol-2-yl) acetate (4aaf).



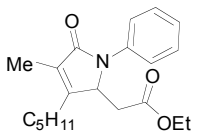
Yield: 20%, colorless oil. TLC: R_f 0.46 (hexane/ethyl acetate = 3:1). ^{1}H NMR ($CDCl_3$) δ 4.32-4.29 (m, 1H; CH), 3.68 (s, 3H; CH_3), 3.55 (tt, *J* = 4.0, 11.5 Hz, 1H; CH), 2.66 (dd, *J* = 4.5, 15.5 Hz, 1H; CH-H), 2.58 (dd, 6.5, 16 Hz, 1H; CH-H), 2.45-2.39 (m, 1H; CH-H), 2.11-2.05 (m, 1H; CH-H), 1.93-1.86 (m, 1H; CH-H), 1.83-1.70 (m, 8H; $4CH_2$), 1.50-1.46 (m, 1H; CH-H), 1.40-1.22 (m, 8H; $4CH_2$), 1.19-1.12 (m, 1H; CH-H), 0.88 (t, *J* = 7.0 Hz, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 172.4, 170.9, 152.7, 129.4, 58.1, 53.7, 51.8, 36.5, 31.7, 31.1, 30.8, 28.0, 26.3, 26.2, 26.1, 25.4, 22.3, 13.9, 8.6. IR (neat): 2930, 2855, 1737, 1667, 1452, 1372, 1256, 1156, 1024 cm^{-1}. HRMS (ESI^+) found 322.2376, calcd for $[M+H]^+$ 322.2382.

Methyl 2-(1-cyclohexyl-3-methyl-5-oxo-4-pentyl-2,5-dihydro-1H-pyrrol-2-yl) acetate (4aaf').



Yield: 4%, colorless oil. TLC: R_f 0.56 (hexane/ethyl acetate = 3:1). ^{1}H NMR ($CDCl_3$) δ 4.20 (dd, *J* = 4.0, 6.0 Hz, 1H; CH), 3.67 (s, 3H; CH_3), 3.59 (tt, *J* = 4.0, 12.5 Hz, 1H; CH), 2.73 (dd, *J* = 4.0, 15.5 Hz, 1H; CH-H), 2.58 (dd, *J* = 7.0, 16 Hz, 1H; CH-H), 2.24-2.13 (m, 2H; CH_2), 1.87 (s, 3H; CH_3), 1.82-1.78 (m, 2H; CH_2), 1.74-1.68 (m, 2H; CH_2), 1.47-1.37 (m, 2H; CH_2), 1.35-1.21 (m, 8H; $4CH_2$), 1.19-1.10 (m, 2H; CH_2), 0.87 (t, *J* = 7.5 Hz, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 172.0, 170.7, 148.4, 133.9, 59.5, 53.4, 51.8, 36.6, 31.7, 31.3, 30.9, 28.0, 26.2, 26.1, 25.5, 23.5, 22.4, 14.0, 11.9. IR (neat): 2931, 2856, 1737, 1682, 1450, 1257, 1116, 1031 cm^{-1}. HRMS (ESI^+) found 322.2376, calcd for $[M+H]^+$ 322.2382.

Ethyl 2-(4-methyl-5-oxo-3-pentyl-1-phenyl-2,5-dihydro-1H-pyrrol-2-yl)acetate (4aba).



Yield: 54%, colorless oil. TLC: R_f 0.43 (hexane/ethyl acetate = 3:1). ^{1}H NMR ($CDCl_3$) δ 7.49-7.47 (m, 2H; Ph-H), 7.40-7.37 (m, 2H; Ph-H), 7.16 (tt, *J* = 1.5, 7.0 Hz, 1H; Ph-H), 4.97-4.95 (m, 1H; CH), 4.08-3.94 (m, 2H; CH_2), 2.62 (dd, *J* = 4.5, 16 Hz, 1H; CH-H), 2.55-2.49 (m, 1H; CH-H), 2.50 (dd, *J* = 7.0, 15.5 Hz, 1H; CH-H), 2.26-2.20 (m, 1H; CH-H), 1.87 (dd, *J* = 1.0, 1.5 Hz, 3H; CH_3), 1.49-1.45 (m, 1H; CH-H), 1.38-1.23 (m, 4H; $2CH_2$), 1.16 (t, *J* = 7.0 Hz, 3H; CH_3), 0.91 (t, *J* = 7.0 Hz, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 170.5, 170.2, 153.3, 136.7, 129.4, 129.0, 124.8, 122.5, 60.9, 58.6, 35.8, 31.7, 28.1, 26.5, 22.3, 14.0, 13.9, 8.7. IR (neat): 2984, 2938, 1666, 1643, 1499, 1371, 1293, 1155, 759, 694 cm^{-1}. HRMS (ESI^+) found 330.2063, calcd for $[M+H]^+$ 330.2069.

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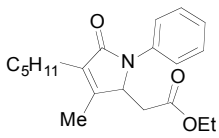
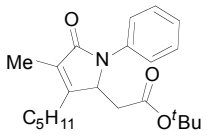
Ethyl 2-(3-methyl-5-oxo-4-pentyl-1-phenyl-2,5-dihydro-1H-pyrrol-2-yl)acetate (4aba').



Yield: 9%, colorless oil. TLC: R_f 0.48 (hexane/ethyl acetate = 3:1). ^{1}H NMR ($CDCl_3$) δ 7.49-7.47 (m, 2H; Ph-H), 7.40-7.36 (m, 2H; Ph-H), 7.15 (tt, *J* = 1.5, 7.0 Hz, 1H; Ph-H), 4.87 (dd, *J* = 4.0, 6.5 Hz, 1H; CH), 4.09-3.97 (m, 2H; CH_2), 2.67 (dd, *J* = 4.5, 15.5 Hz, CH-H), 2.50 (dd, *J* = 7.5, 15.5 Hz, 1H; CH-), 2.29 (t, *J* = 7.5 Hz, 2H; CH_2), 2.01 (s, 3H; CH_3), 1.57 (s, 3H; CH_3), 1.50 (quint, *J* = 7.5 Hz, 2H; CH_2), 1.35-1.26 (m, 4H; $2CH_2$), 1.18 (t, *J* = 7.5 Hz, 3H; CH_3), 0.88 (t, *J* = 7.0 Hz, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 170.1, 170.0, 148.9, 136.7, 133.8, 129.0, 124.7, 122.3, 60.9, 60.1, 35.8, 31.6, 28.0, 23.5, 22.4, 14.0, 14.0, 12.1. IR (neat): 2957, 2930, 1736, 1667, 1501, 1372, 1188, 759, 694 cm^{-1}. HRMS (ESI^+) found 330.2064, calcd for $[M+H]^+$ 330.2069

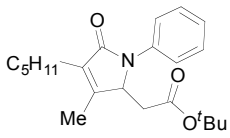
.

Tert-butyl 2-(4-methyl-5-oxo-3-pentyl-1-phenyl-2,5-dihydro-1H-pyrrol-2-yl)acetate (4aca).



Yield: 25%, colorless oil. TLC: R_f 0.45 (hexane/ethyl acetate = 3:1). ^{1}H NMR ($CDCl_3$) δ 7.51-7.49 (m, 2H; Ph-H), 7.40-7.36 (m, 2H; Ph-H), 7.15 (tt, *J* = 1.5, 7.0 Hz, 1H; Ph-H), 4.91-4.89 (m, 1H; CH), 2.58 (dd, *J* = 4.0, 16 Hz, 1H; CH-H), 2.54-2.50 (m, 1H; CH-H), 2.46 (dd, *J* = 7.0, 15.5 Hz, 1H; CH-H), 2.30-2.26 (m, 1H; CH-H), 1.86 (s, 3H; CH_3), 1.63-1.59 (m, 1H; CH-H), 1.52-1.48 (m, 1H; CH-H), 1.35 (s, 9H; $3CH_3$), 0.91 (t, *J* = 7.0 Hz, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 170.5, 169.1, 153.4, 136.9, 129.2, 129.0, 124.6, 122.2, 81.3, 58.6, 36.5, 31.7, 28.1, 27.8, 26.5, 22.4, 13.9, 8.7. IR (neat): 2957, 2931, 1725, 1693, 1501, 1381, 1143, 759, 693 cm^{-1}. HRMS (ESI^+) found 358.2376, calcd for $[M+H]^+$ 358.2382

Tert-butyl 2-(3-methyl-5-oxo-4-pentyl-1-phenyl-2,5-dihydro-1H-pyrrol-2-yl)acetate (4aca').



Yield: 3%, colorless oil. TLC: R_f 0.50 (hexane/ethyl acetate = 3:1). ^{1}H NMR ($CDCl_3$) δ 7.51-7.45 (m, 2H; Ph-H), 7.41-7.34 (m, 2H; Ph-H), 7.15 (m, 1H; Ph-H), 4.82 (dd, *J* = 3.0, 6.5 Hz, 1H; CH), 2.63 (dd, *J* = 3.5, 15 Hz, 1H; CH-H), 2.44 (dd, *J* = 7.5, 15.5 Hz, 1H; CH-H), 2.32-2.26 (m, 2H; CH_2), 2.03 (s, 3H; CH_3), 1.54-1.48 (m, 2H; CH_2), 1.37 (s, 9H; $3CH_3$), 1.34-1.26 (m, 4H; $2CH_2$), 0.89 (t, *J* = 6.5 Hz, 3H; CH_3). ^{13}C NMR ($CDCl_3$) δ 170.1, 169.2, 149.2, 133.7, 129.0, 128.9, 124.5, 122.1, 81.3, 60.1, 36.6, 31.7, 28.0, 27.9, 23.6, 22.4, 14.0, 12.2. IR (neat): 2957, 2932, 1721, 1667, 1501, 1393, 1367, 1147, 759, 694 cm^{-1}. HRMS (ESI^+) found 358.2376, calcd for $[M+H]^+$ 358.2382.

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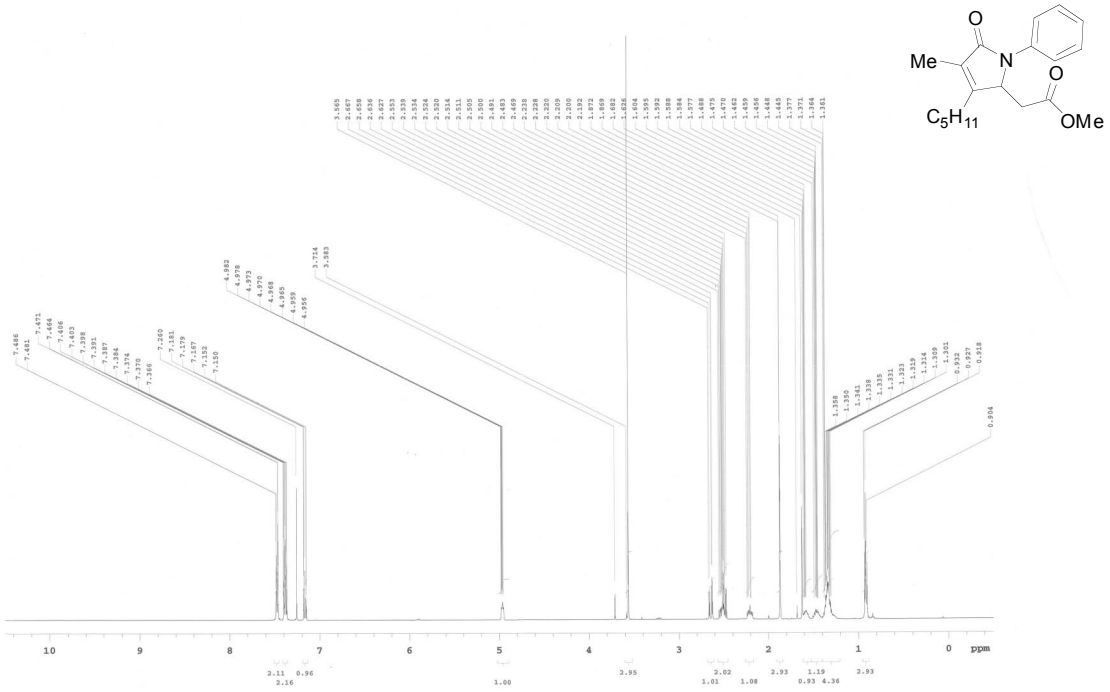
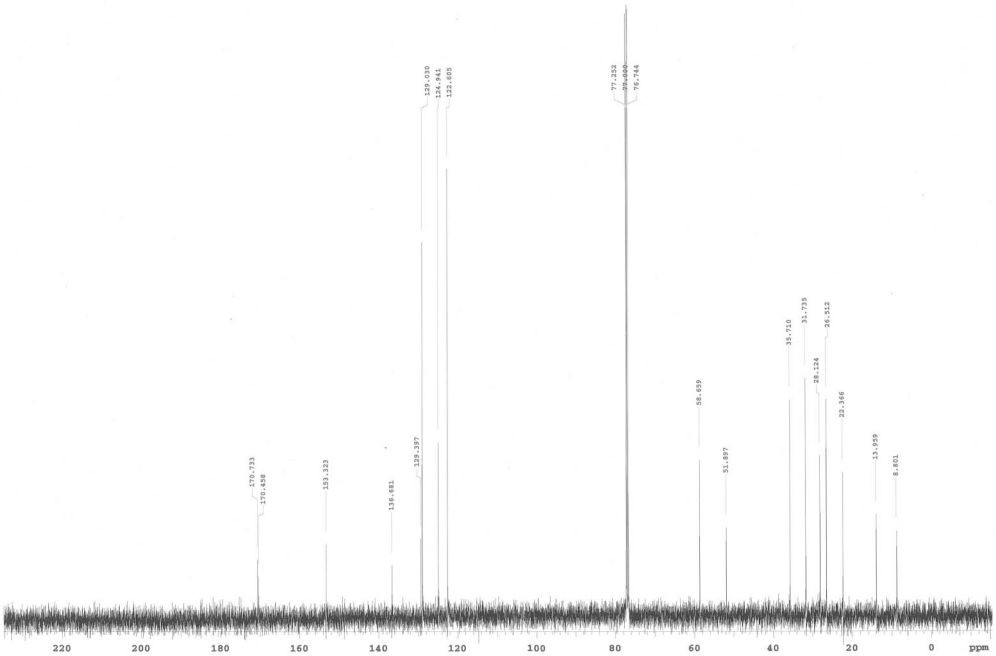
^{1}H NMR and ^{13}C NMR Spectra of Products





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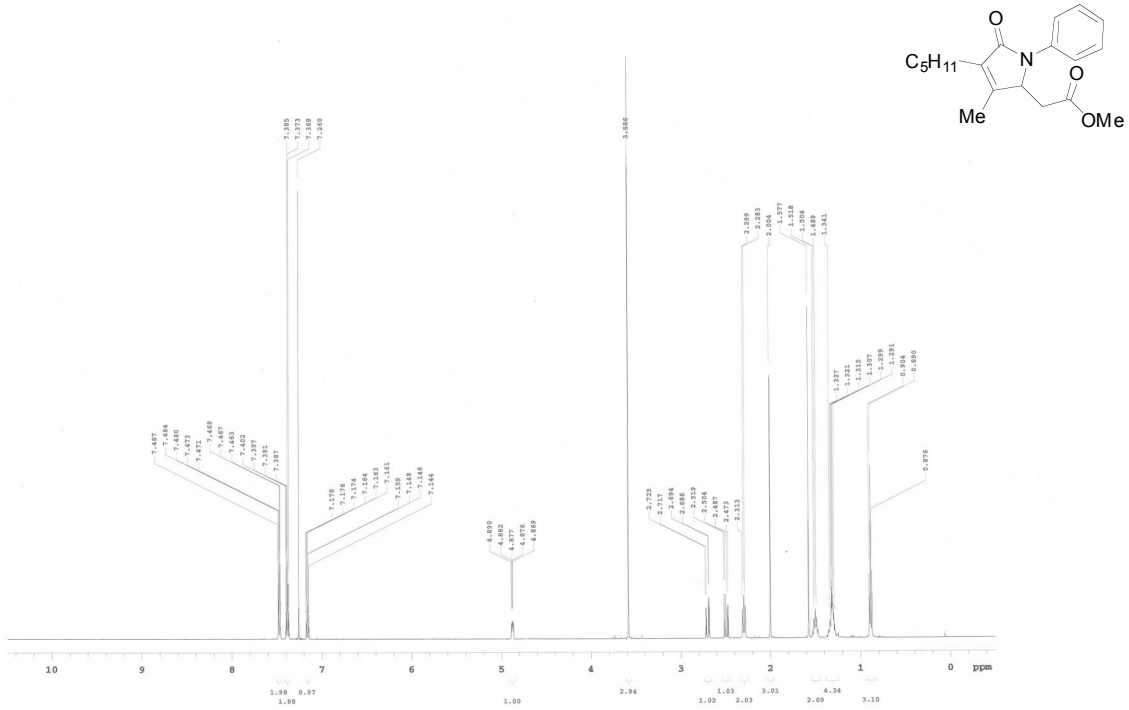

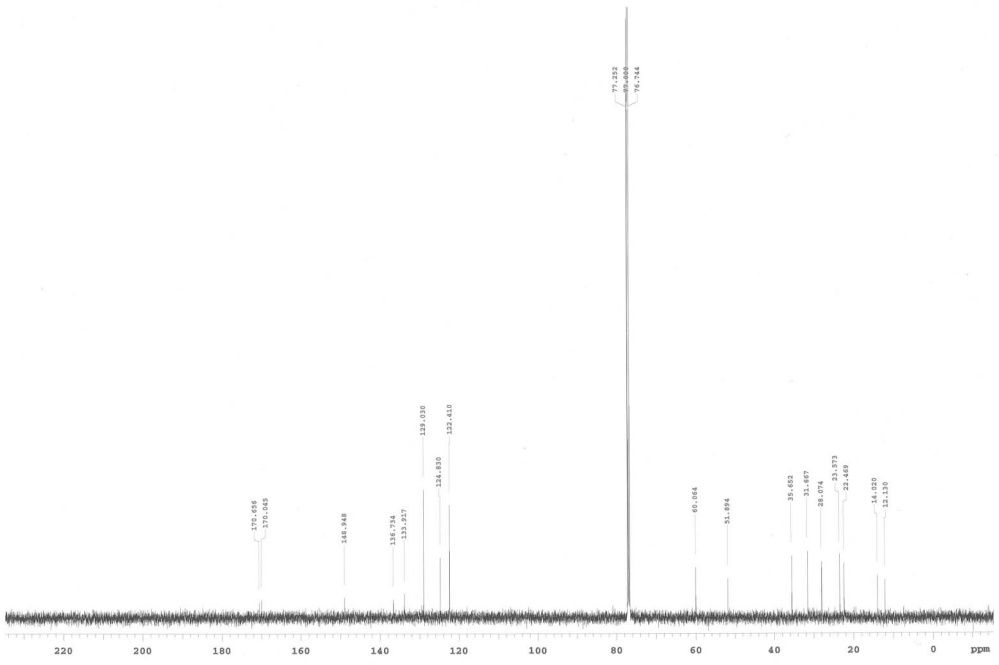

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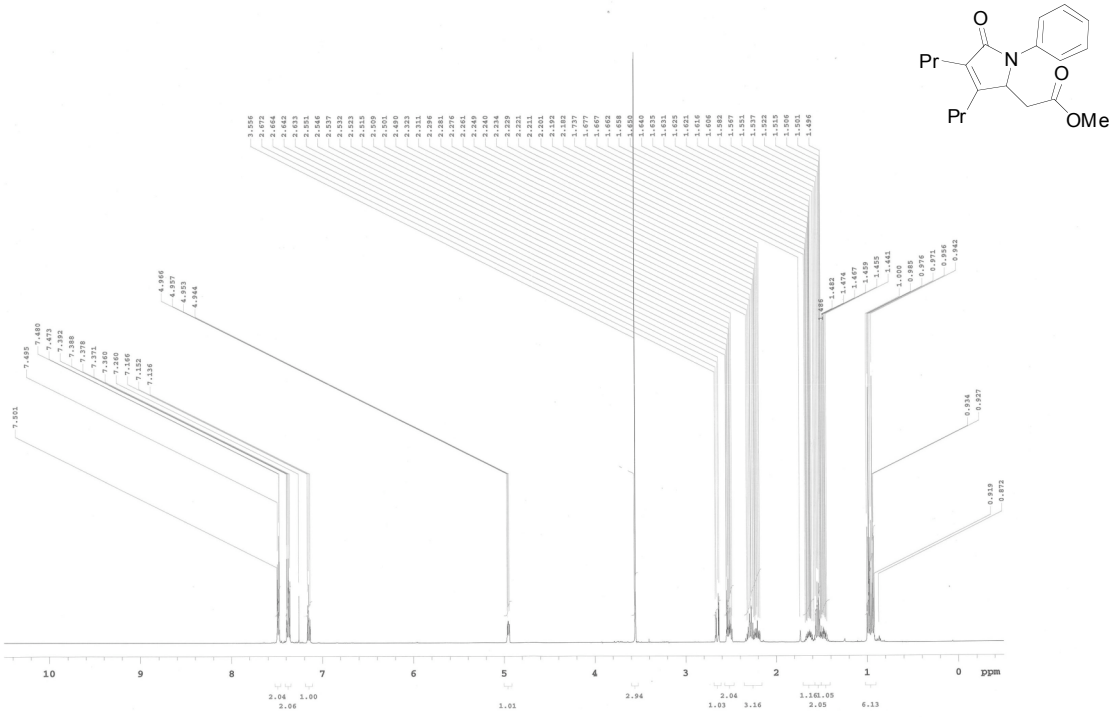

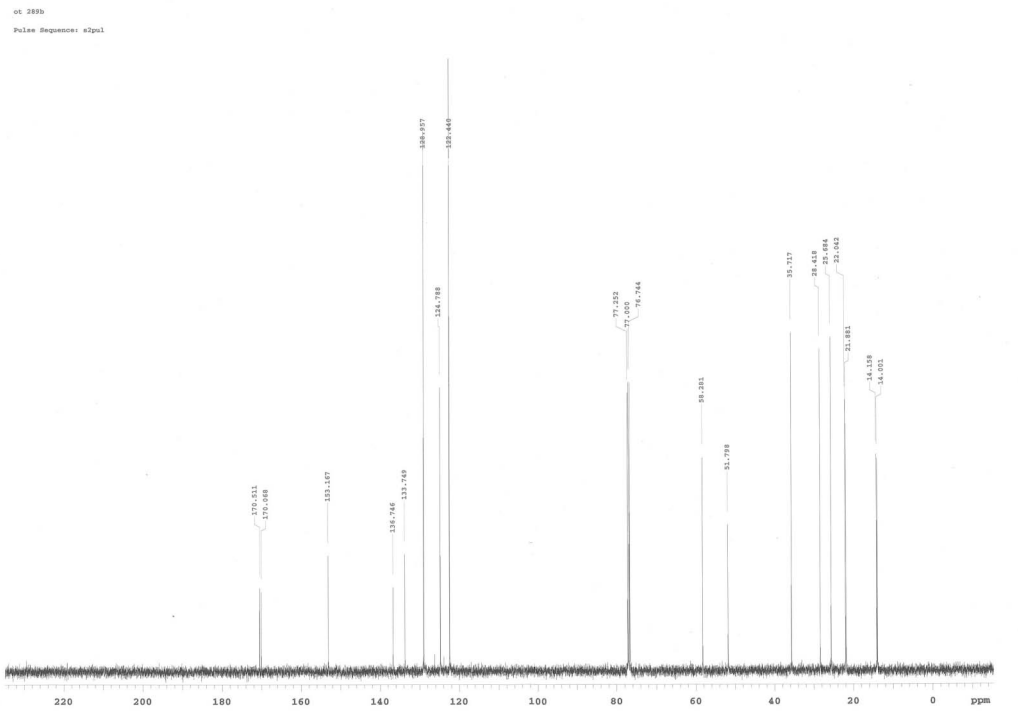

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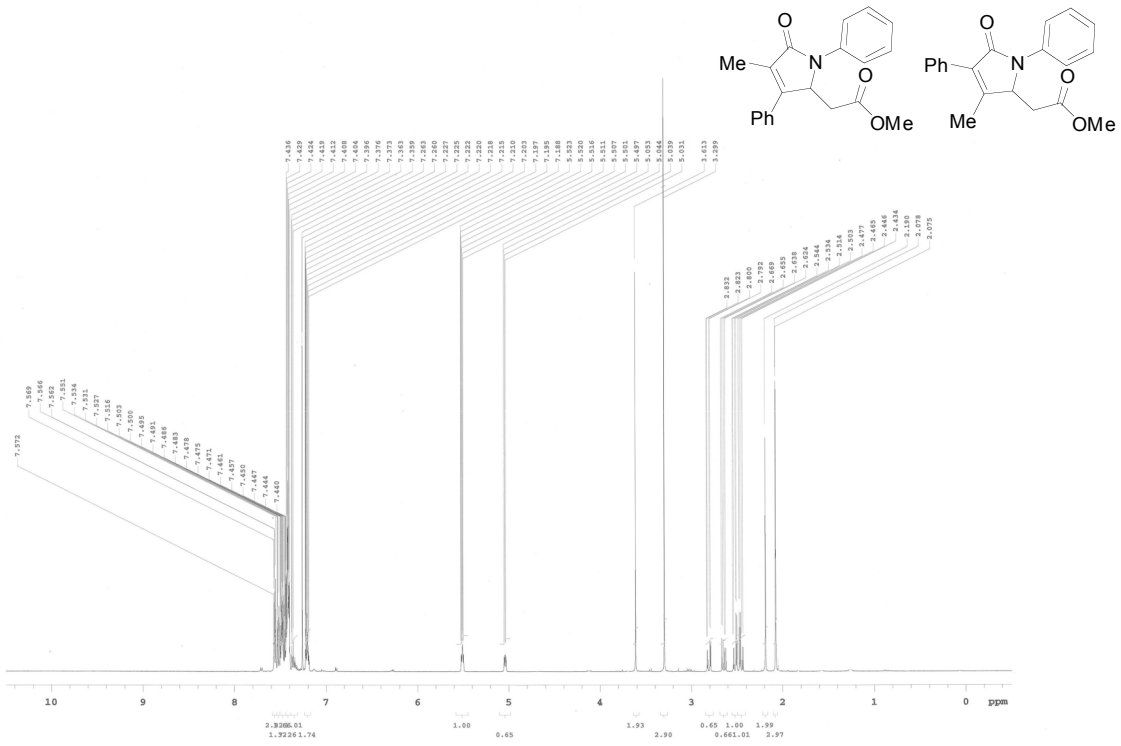

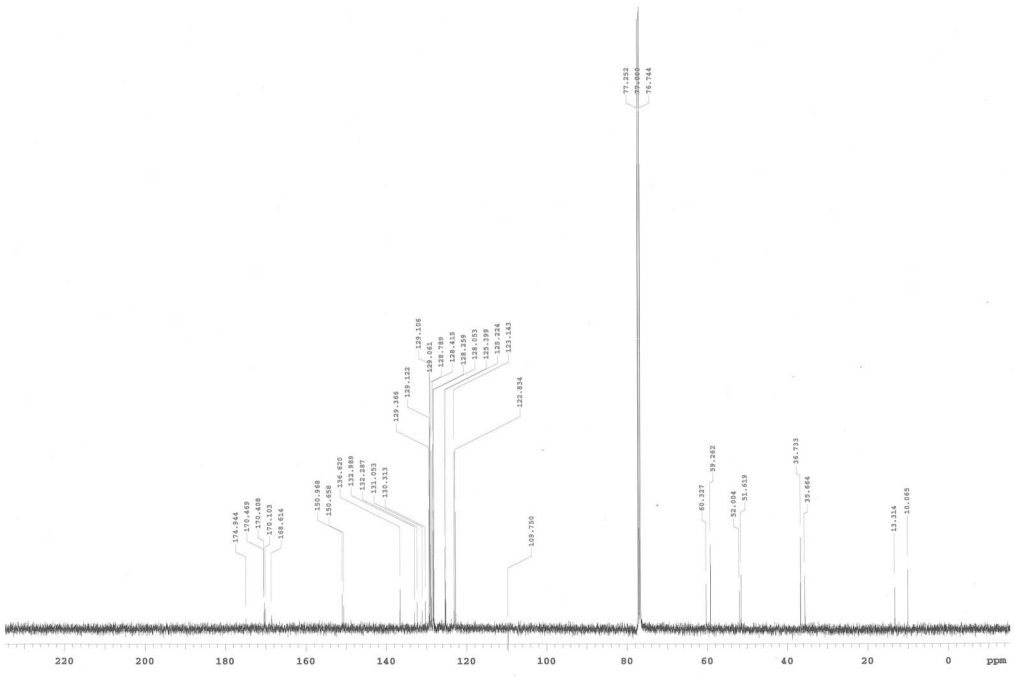

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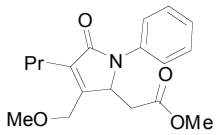

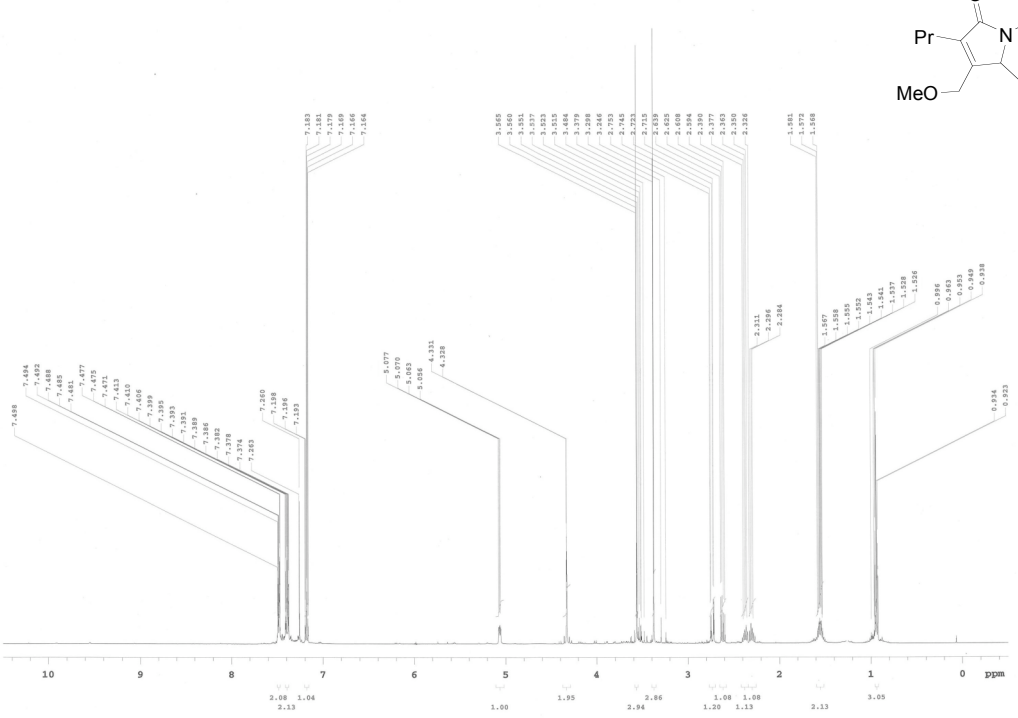

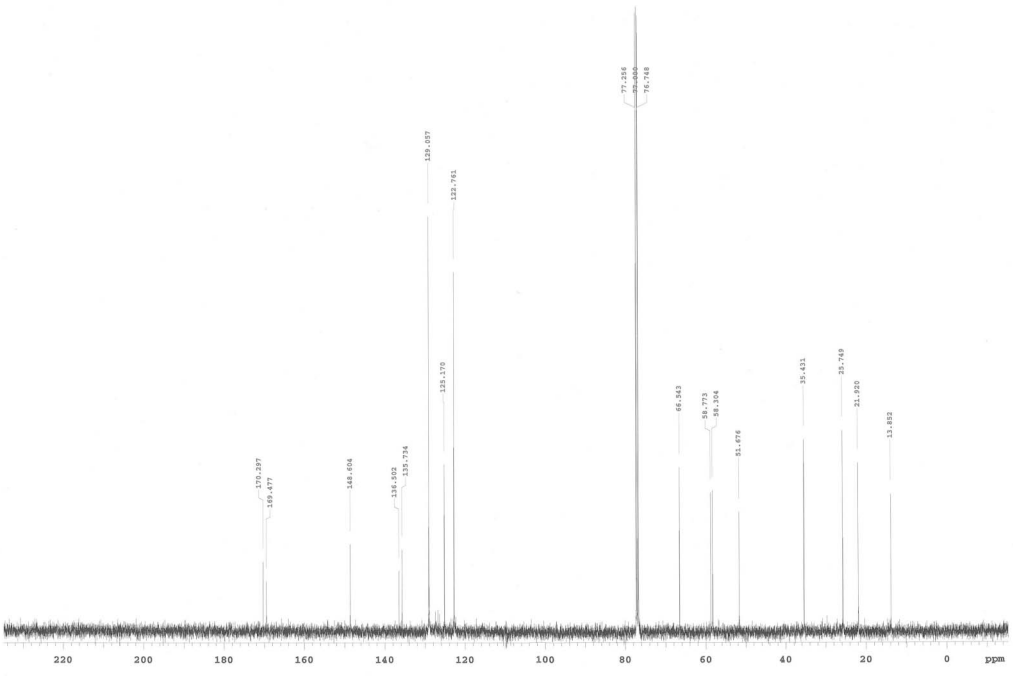

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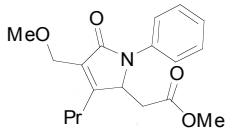

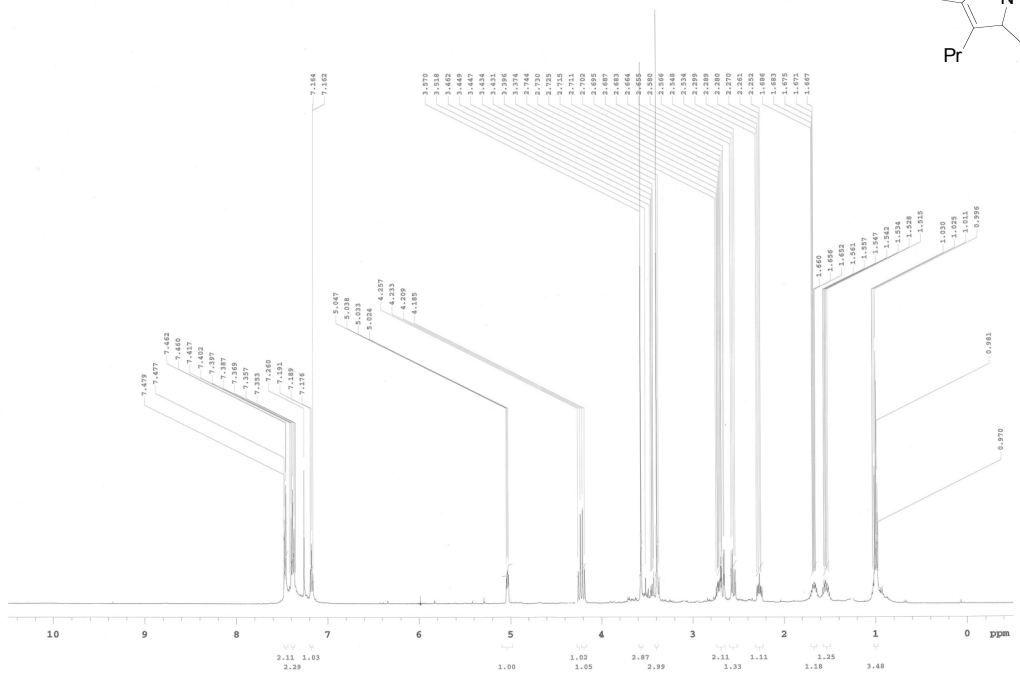

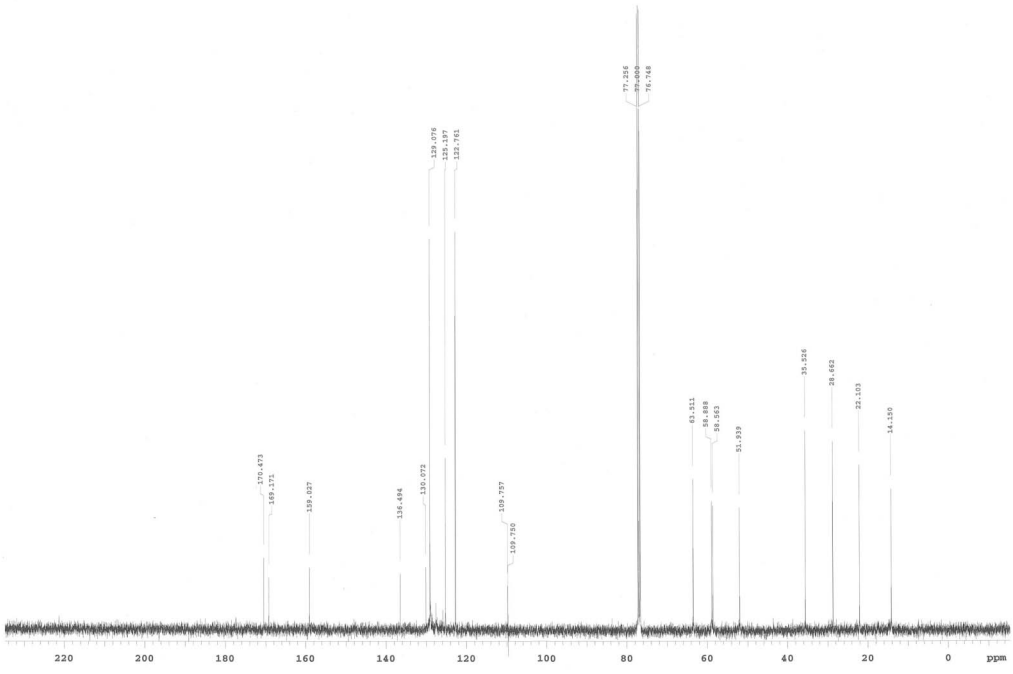

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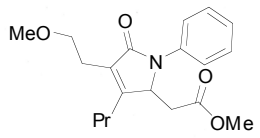

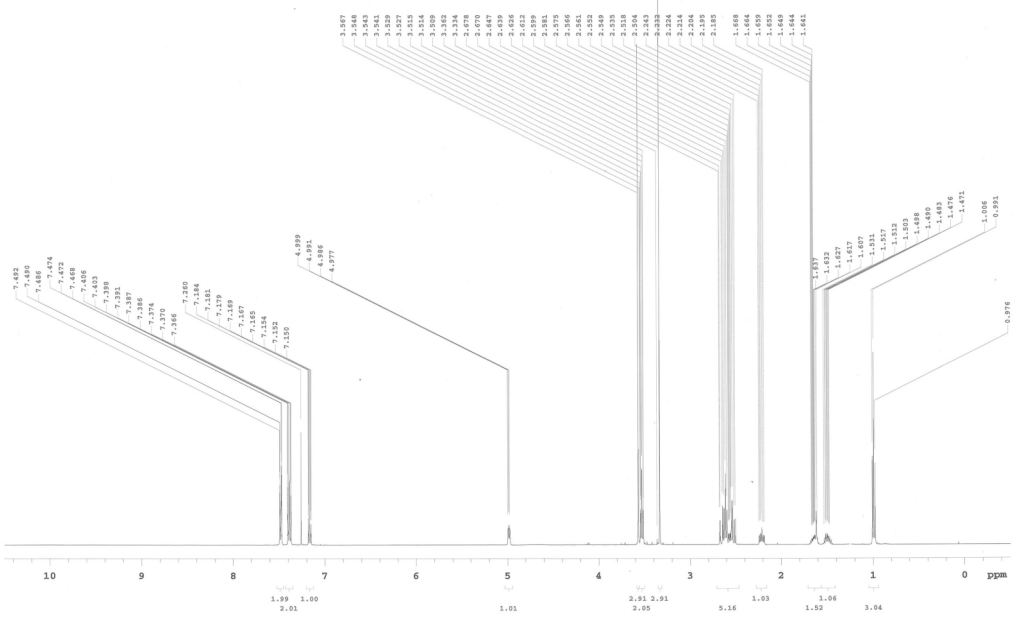

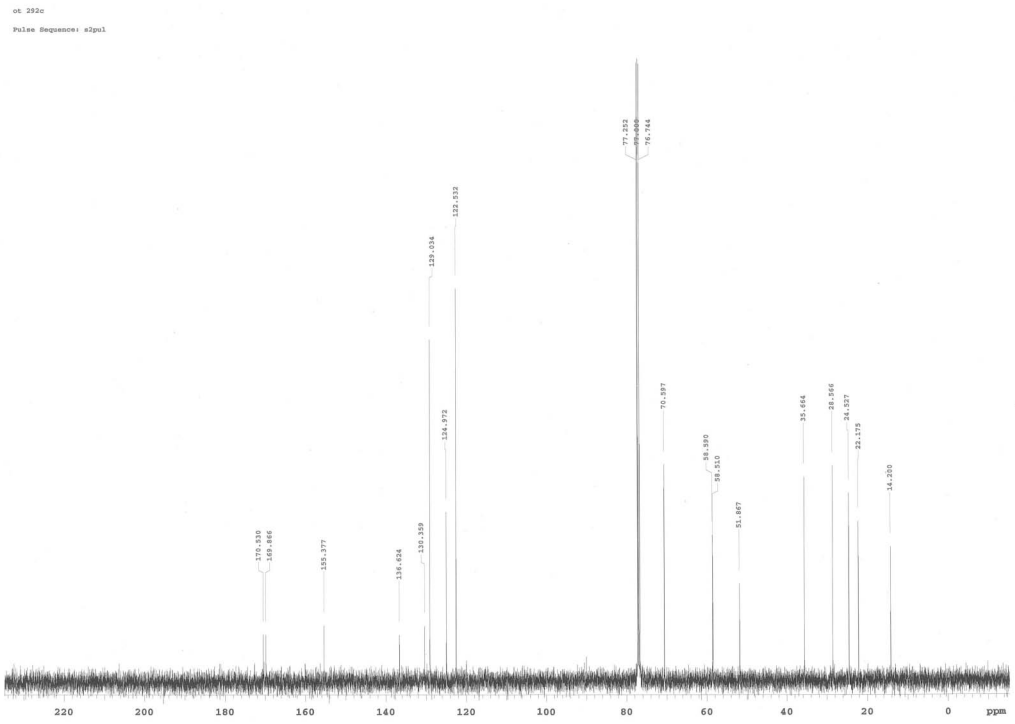

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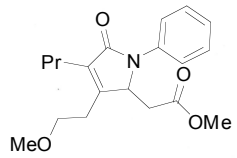

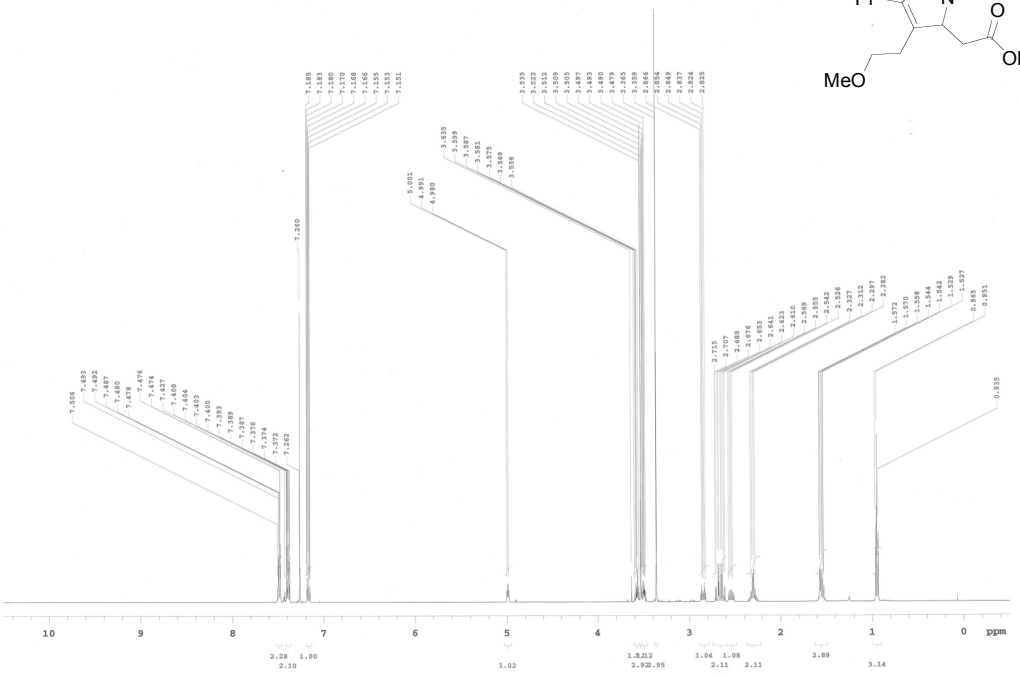

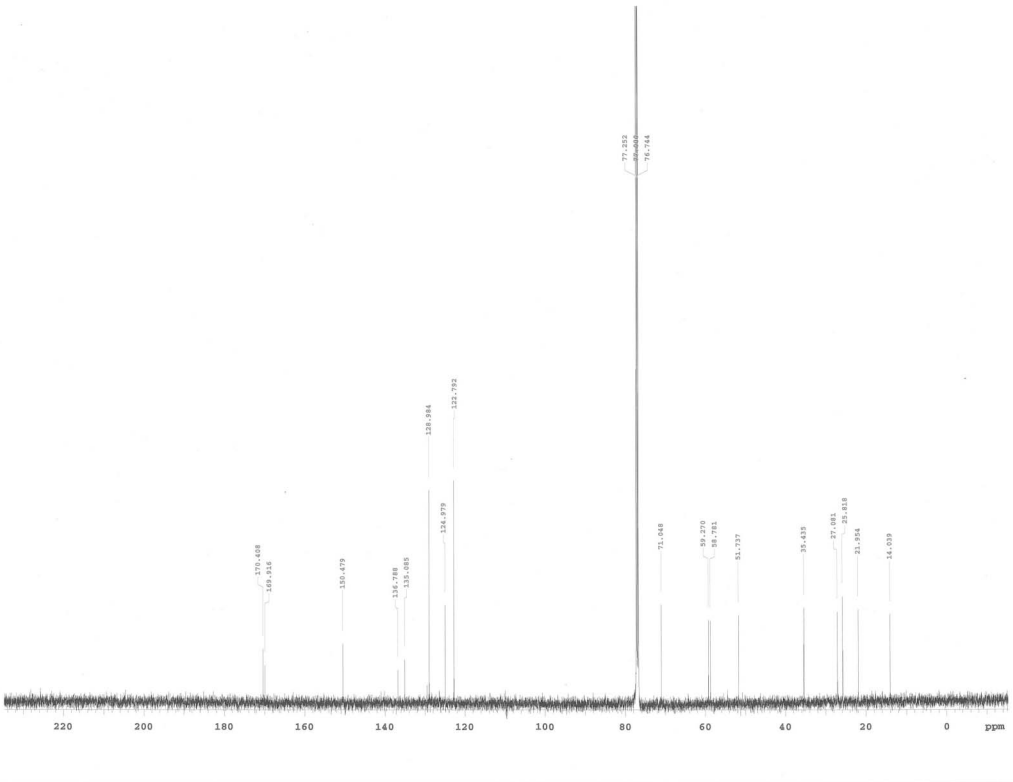

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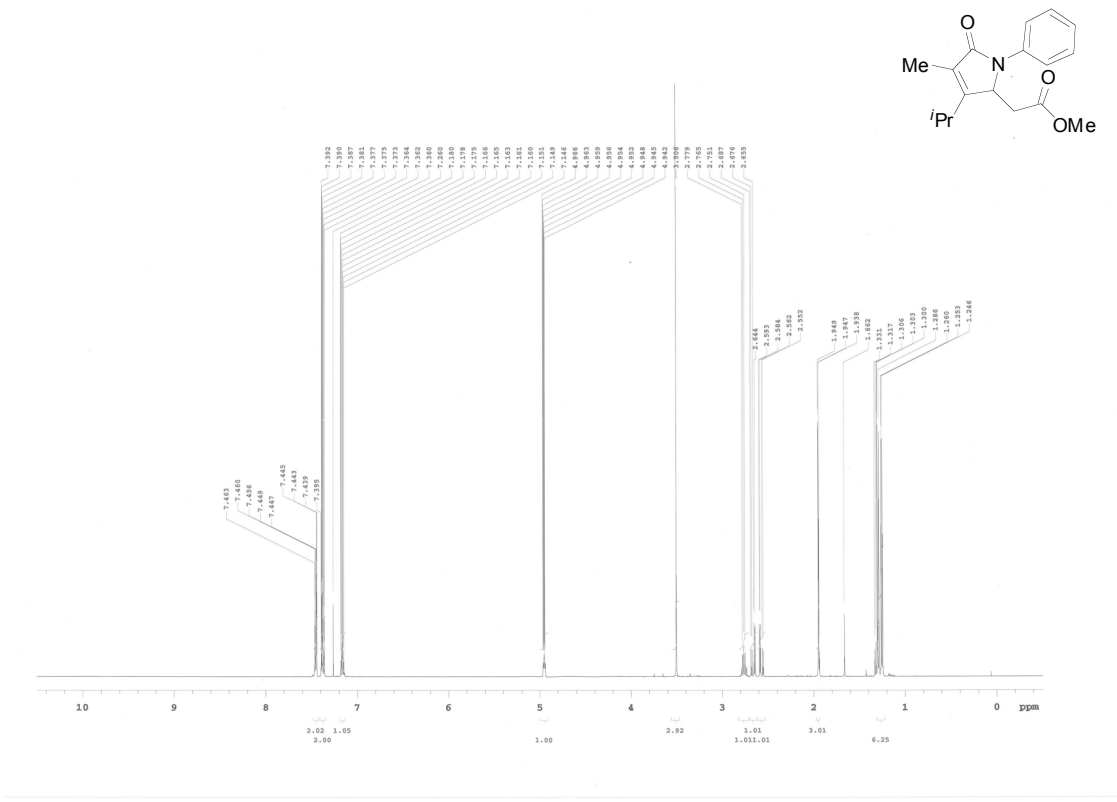

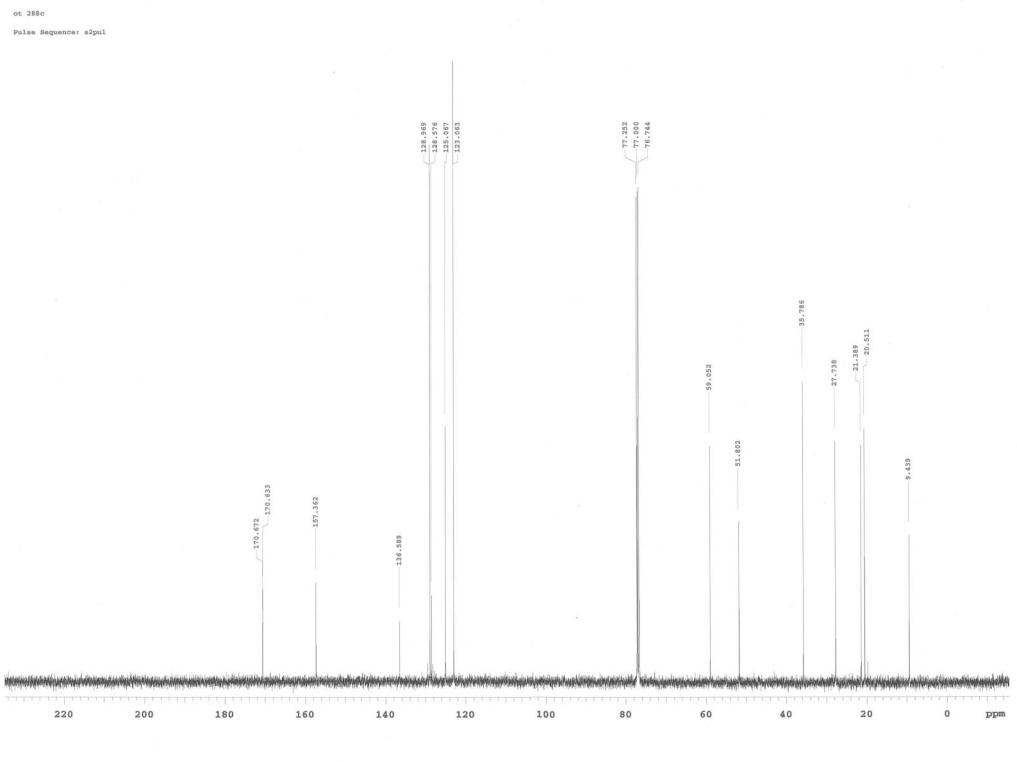

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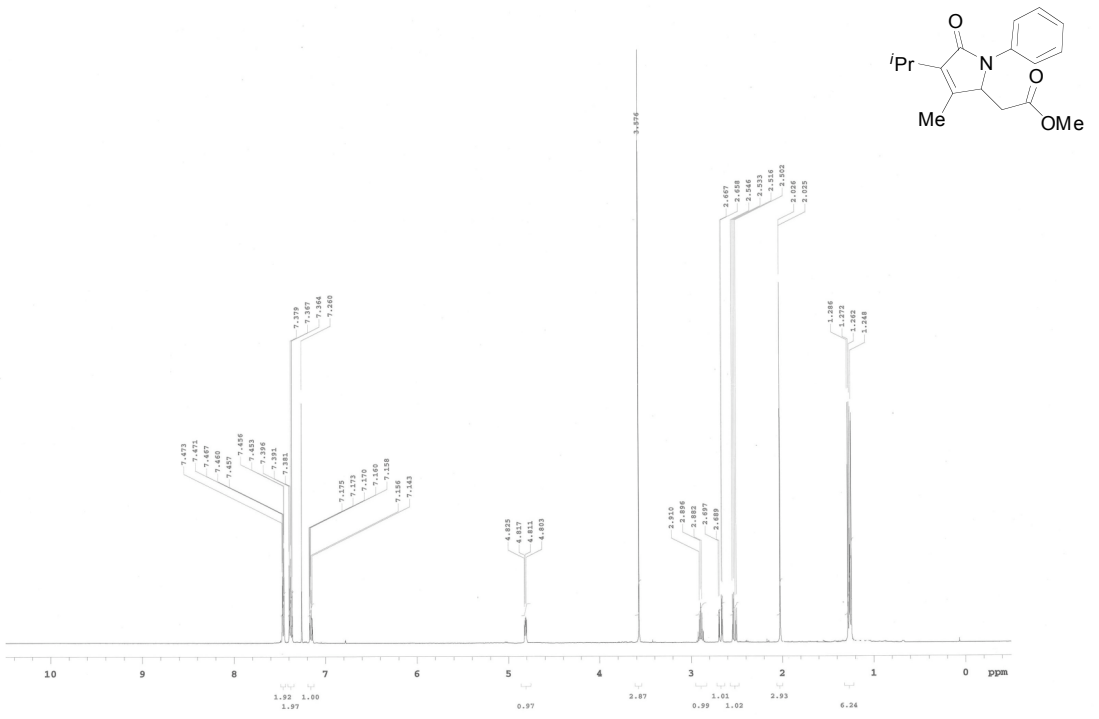

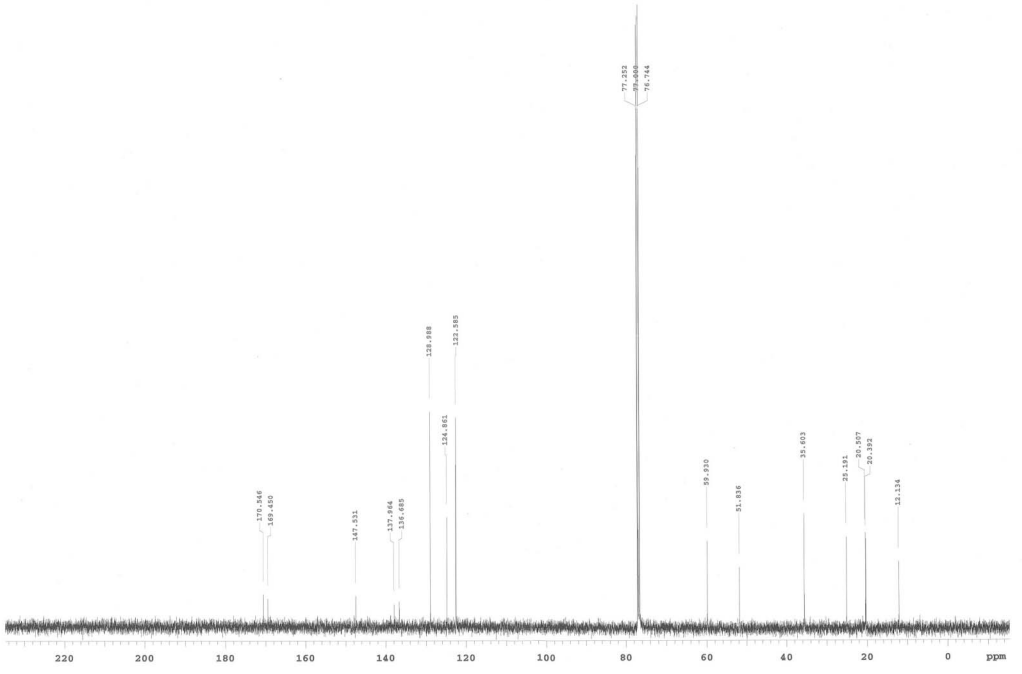

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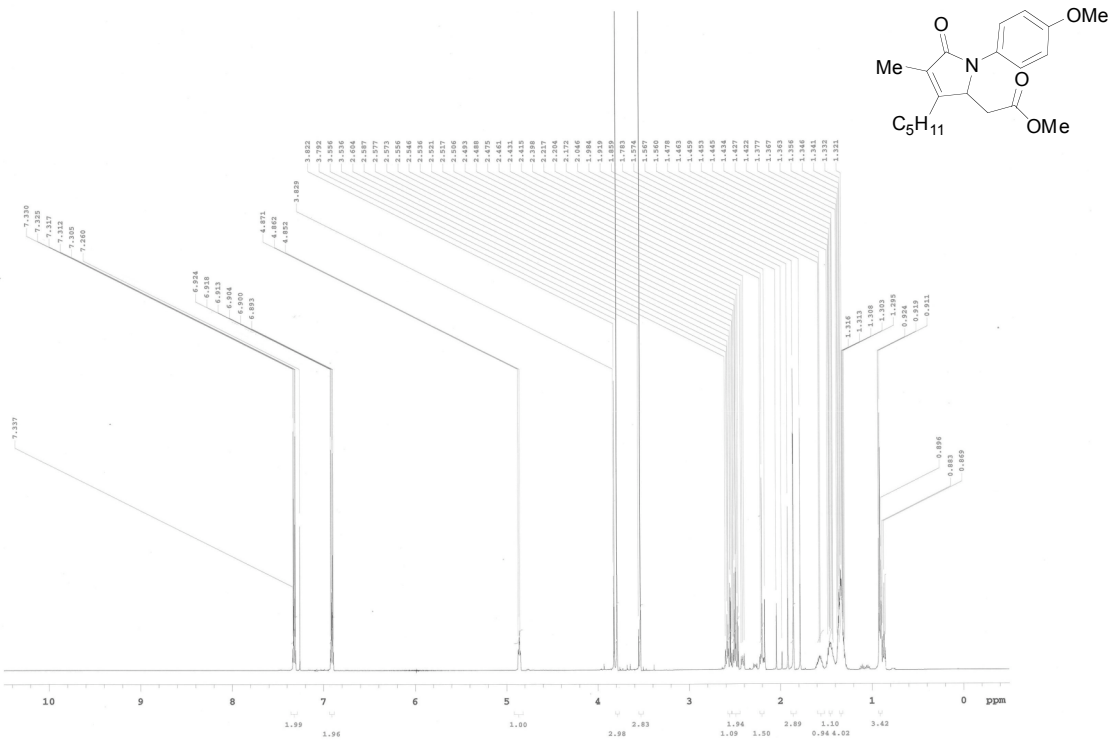

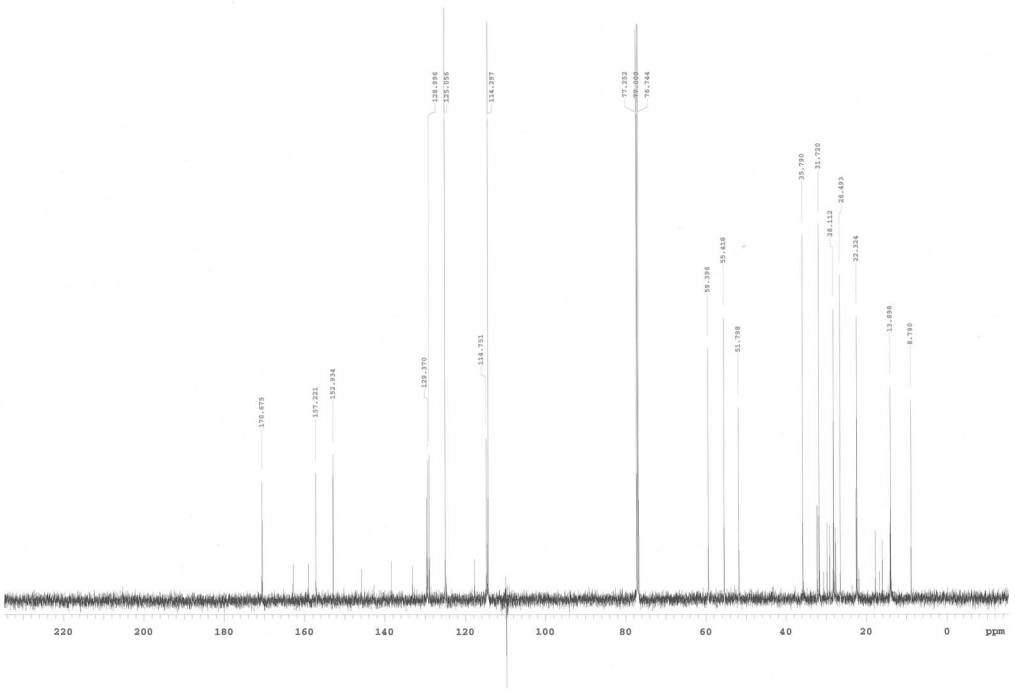

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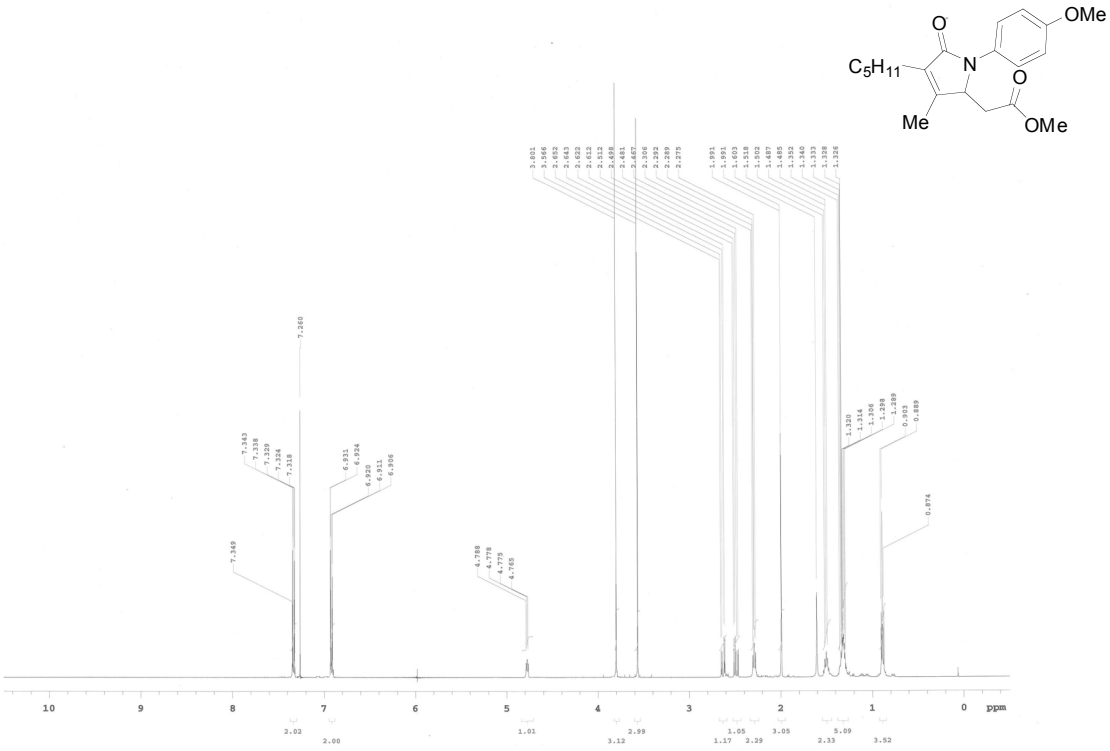

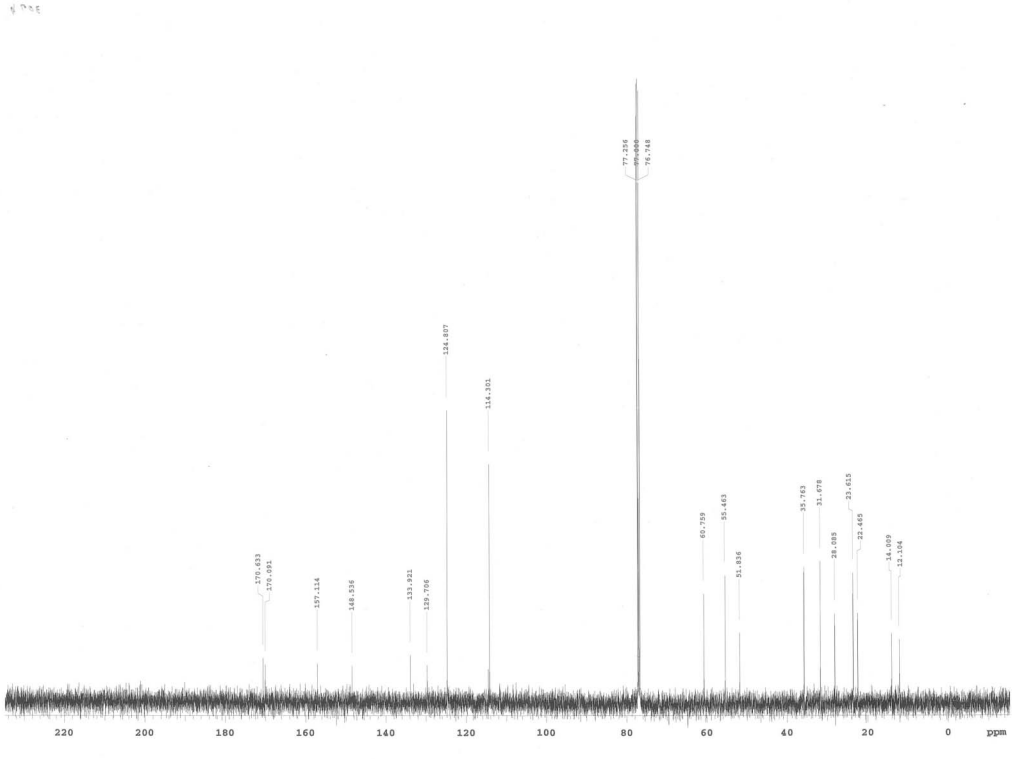

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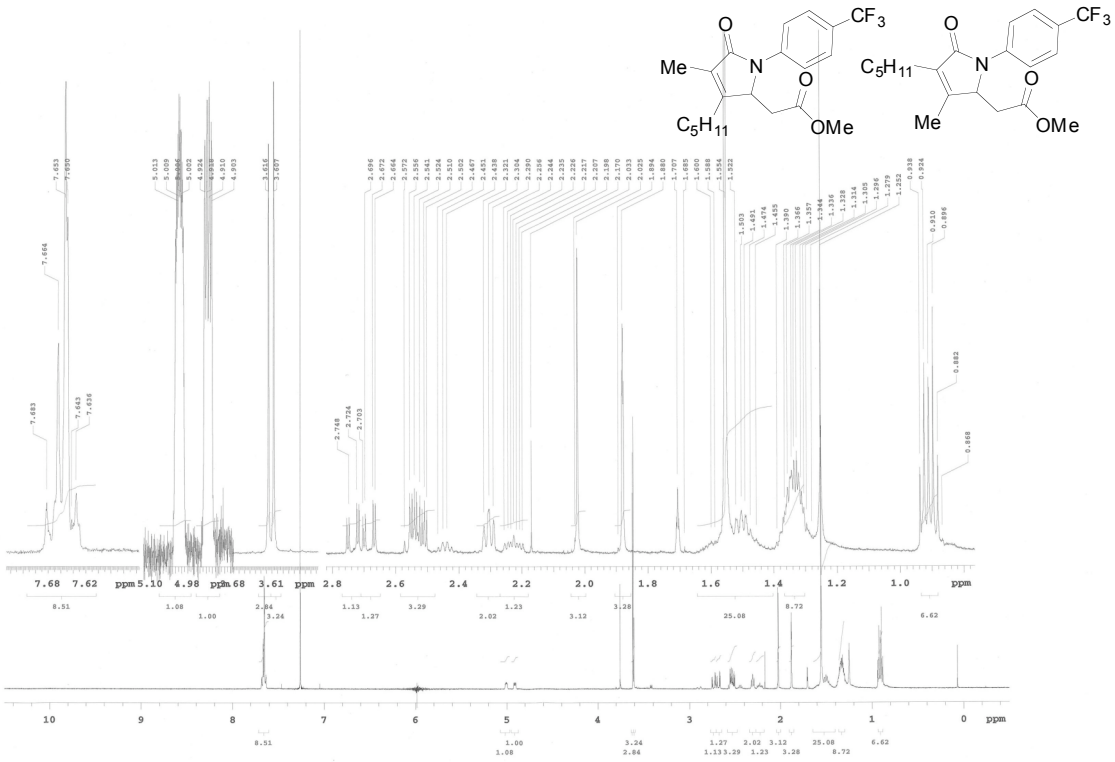

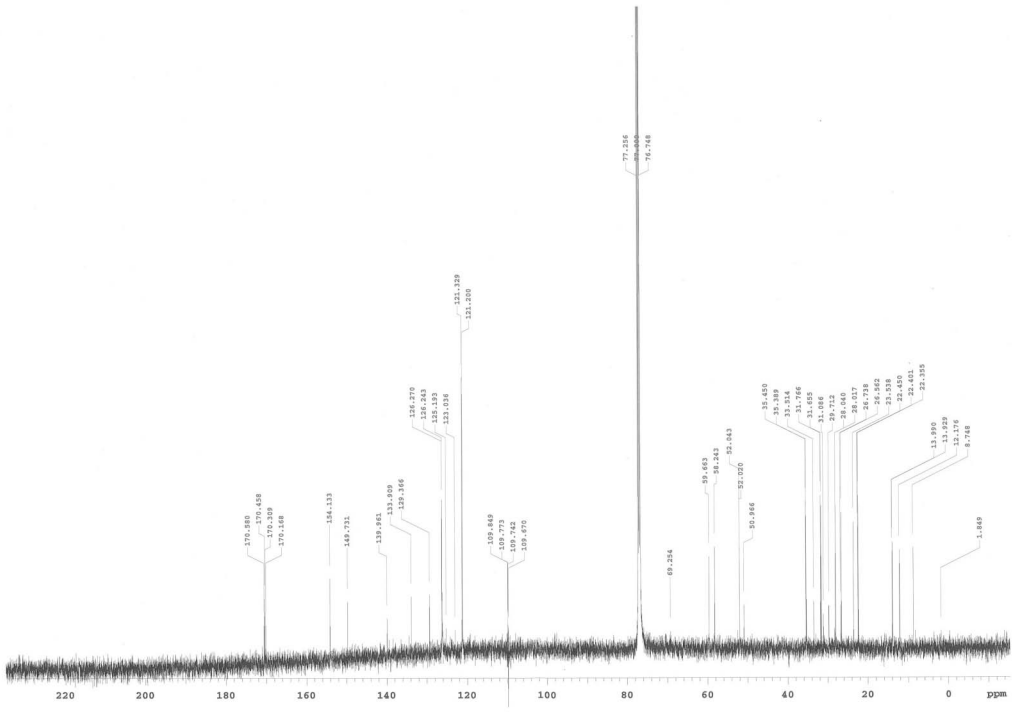

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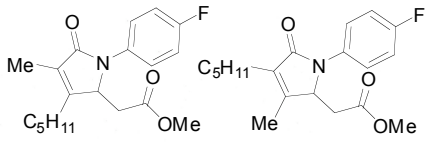

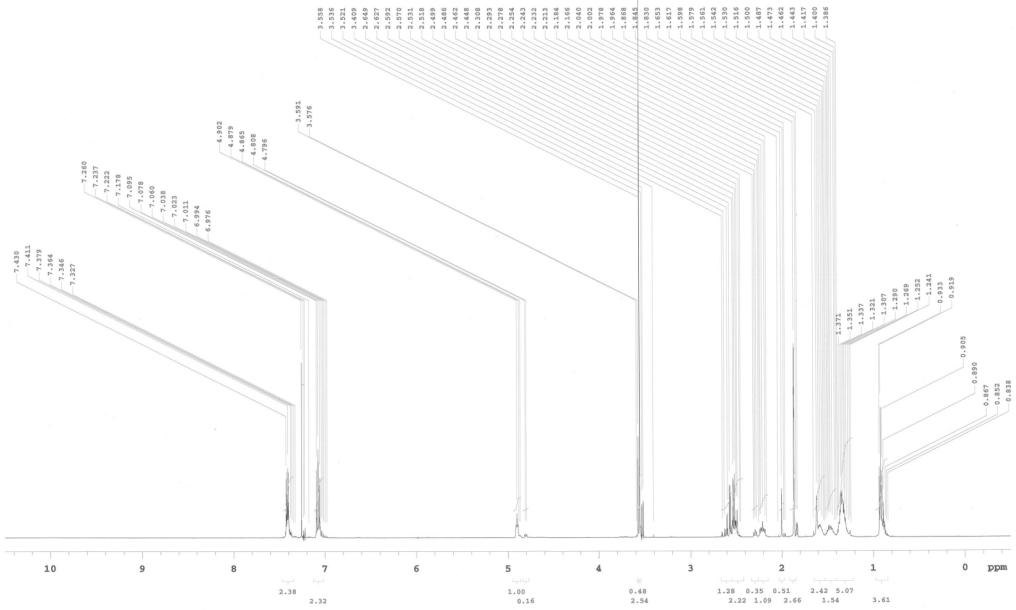

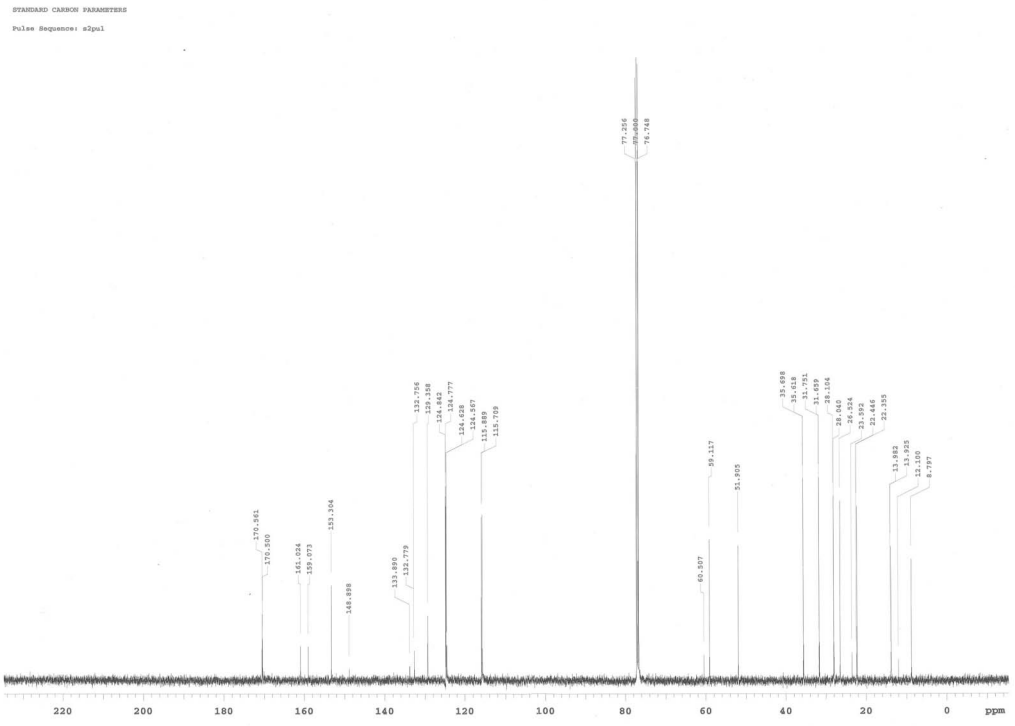

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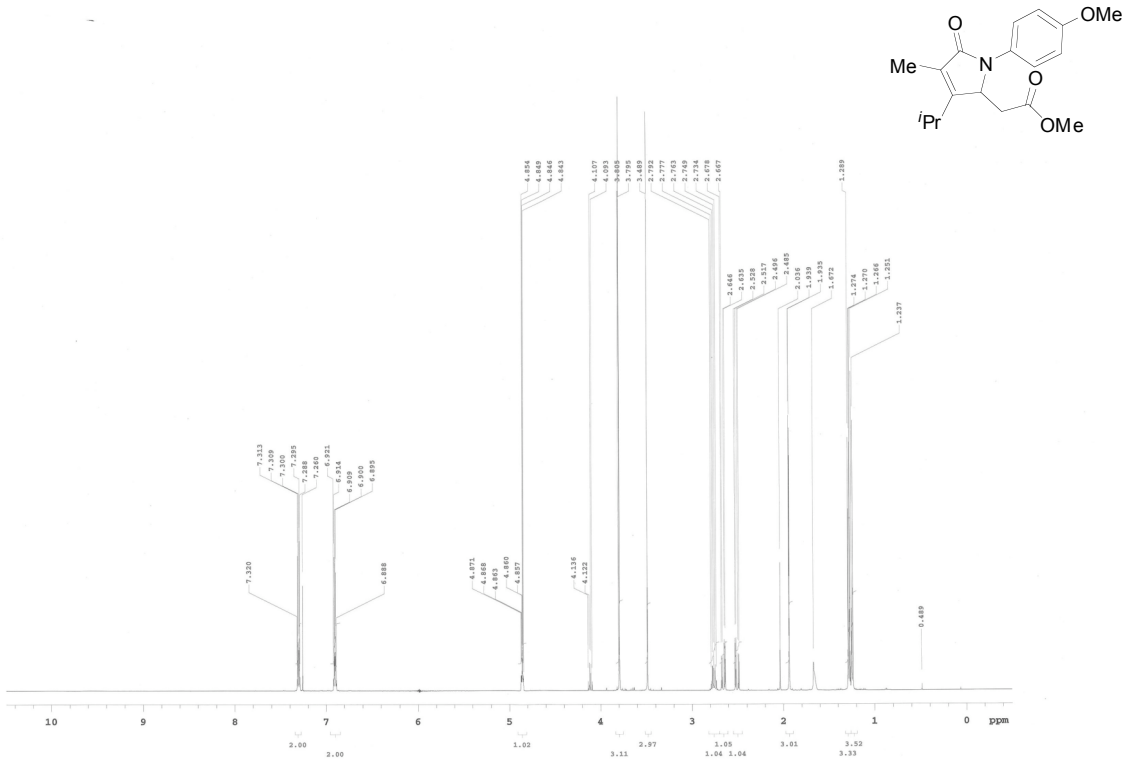

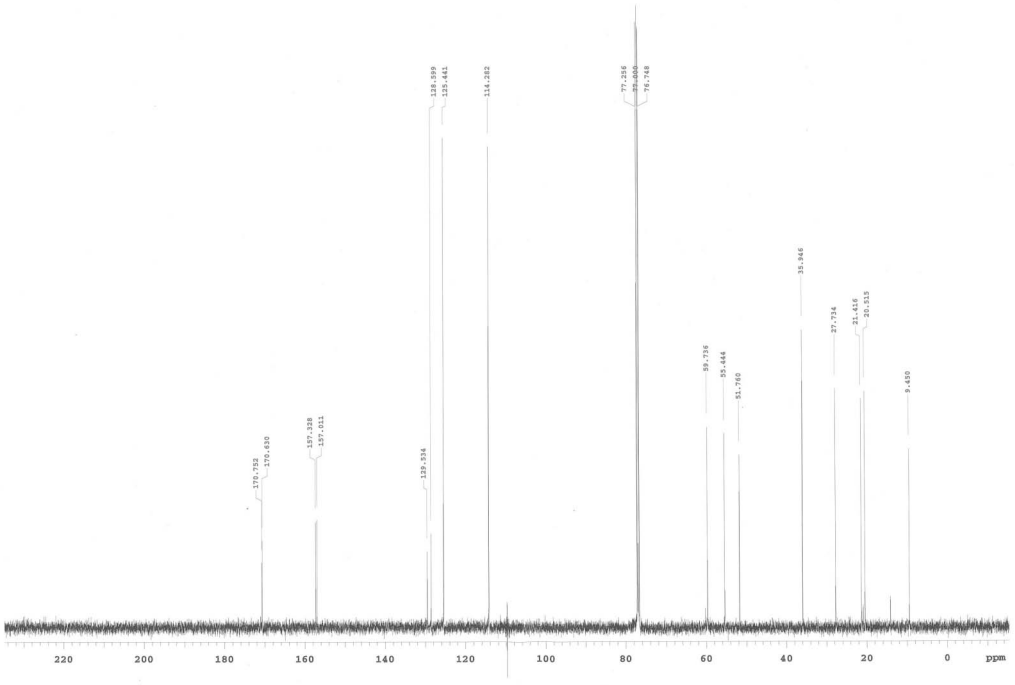

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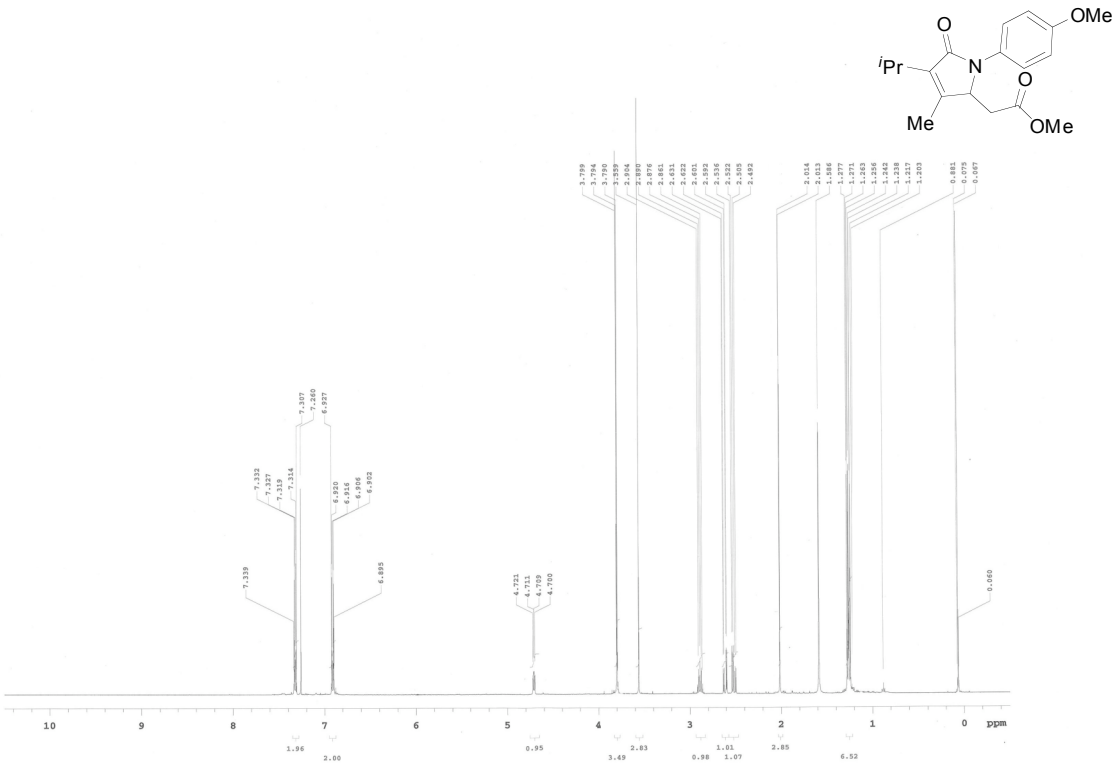

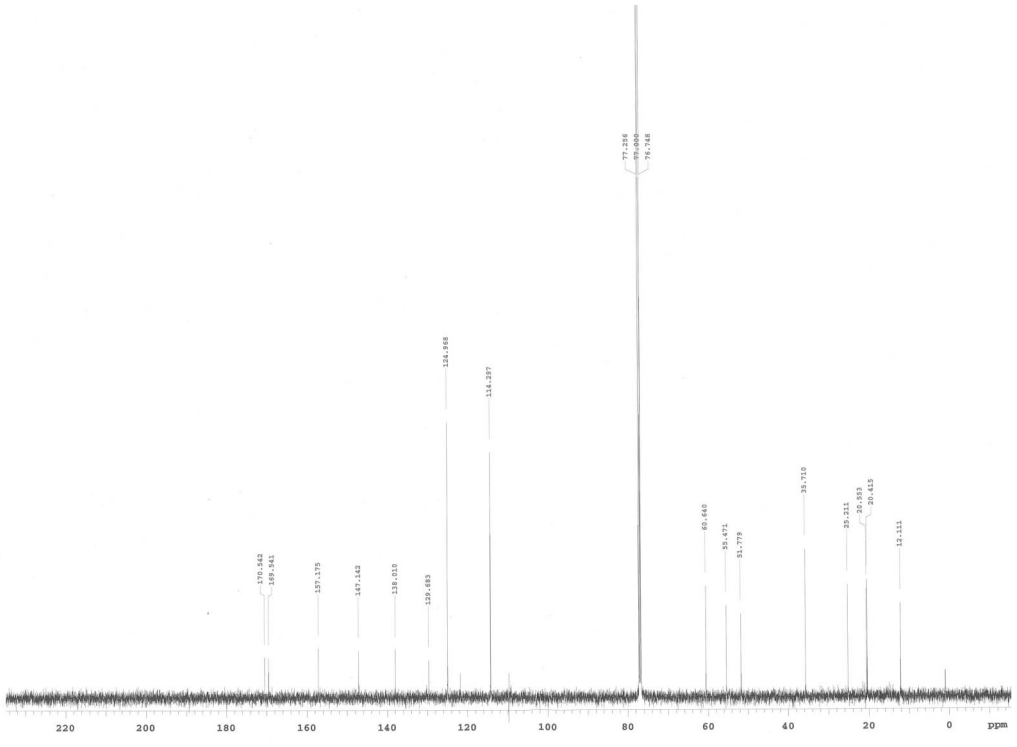

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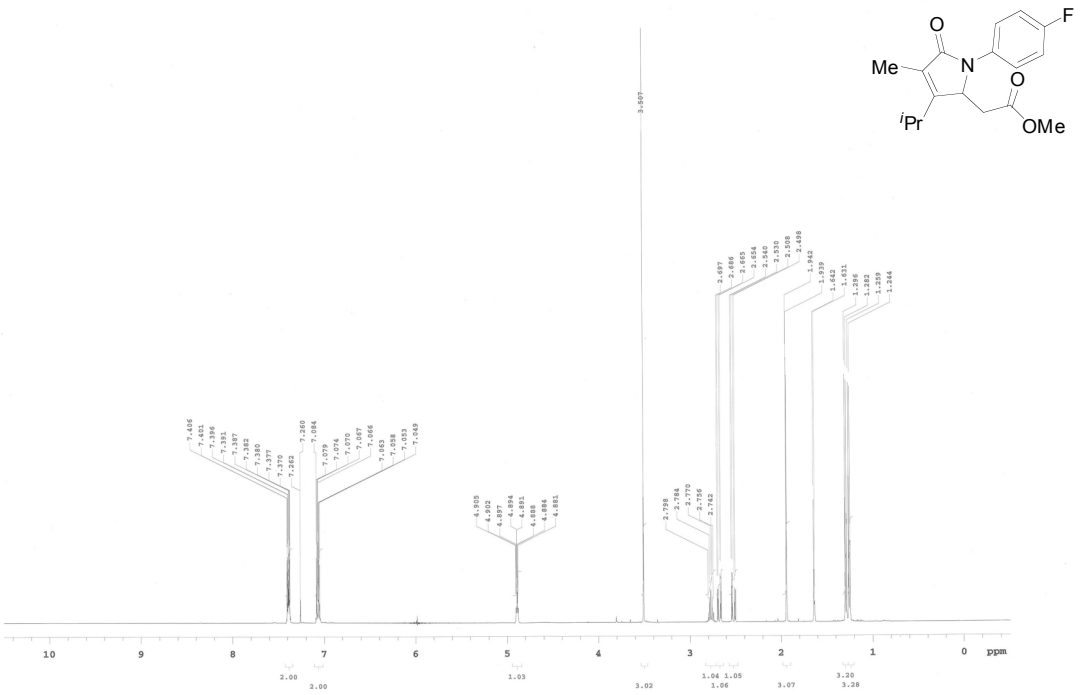

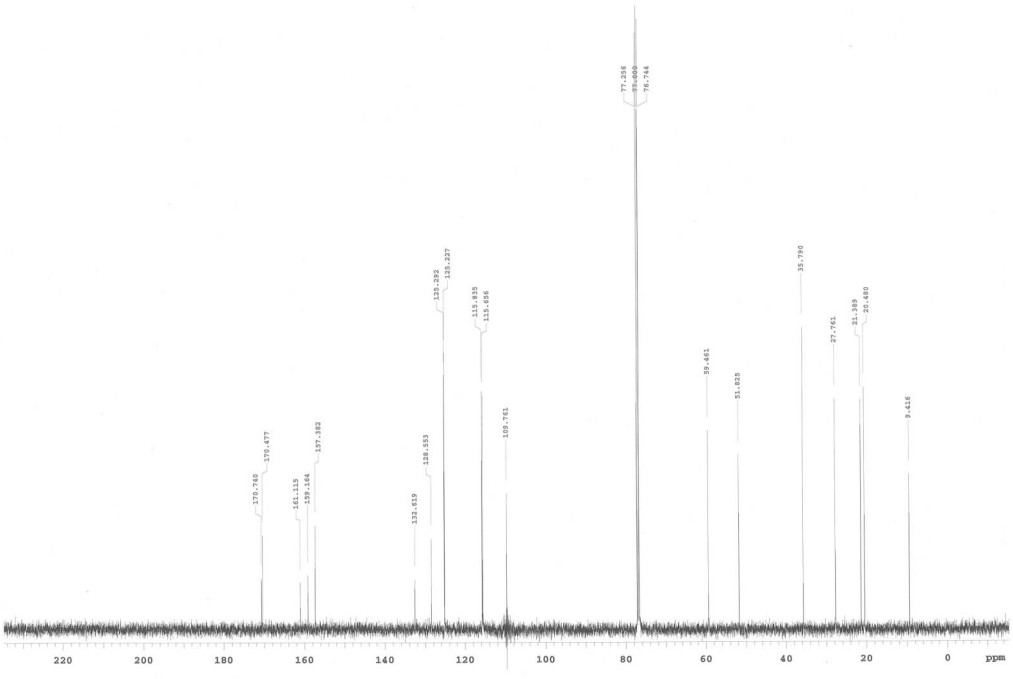

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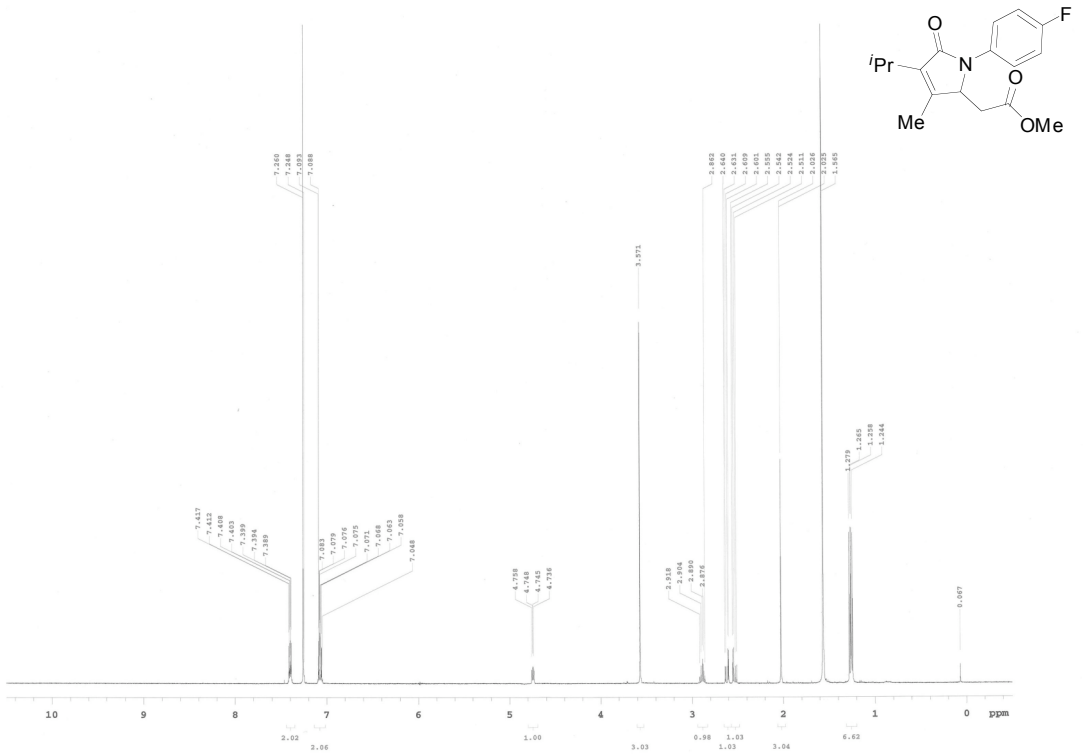

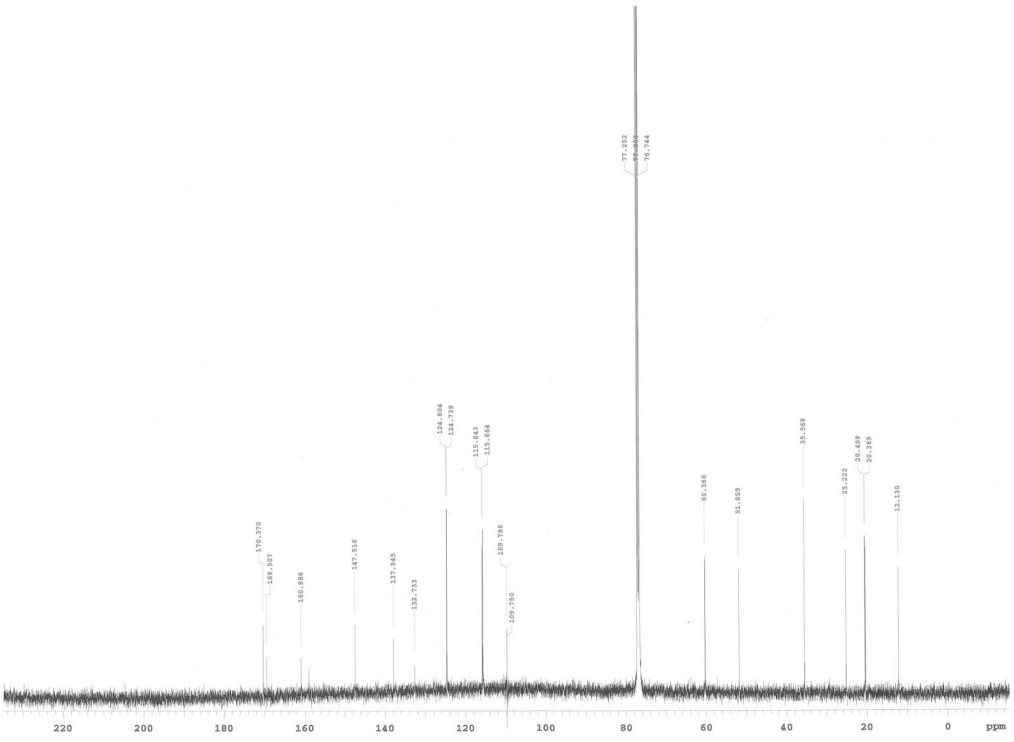

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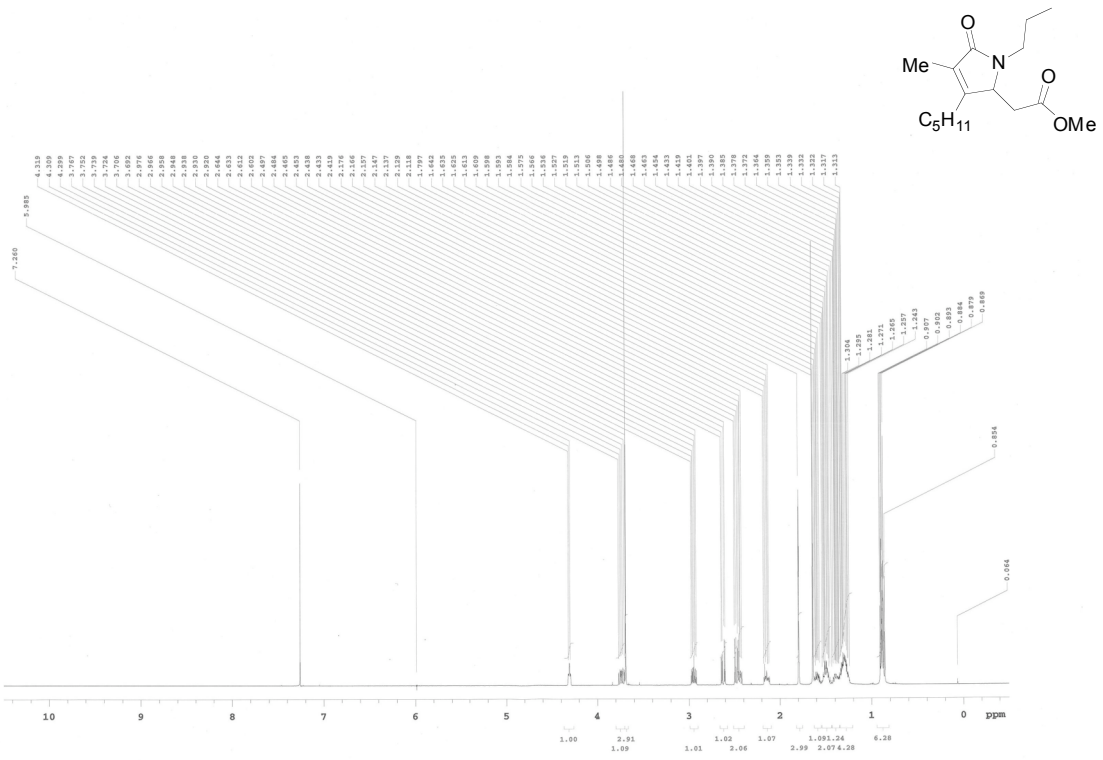

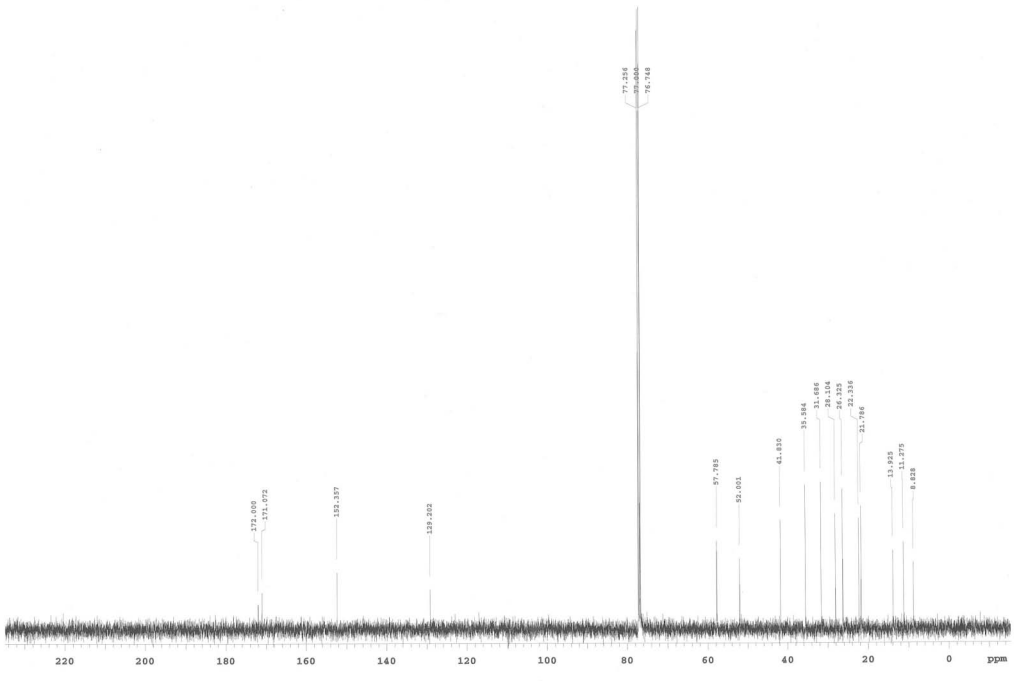

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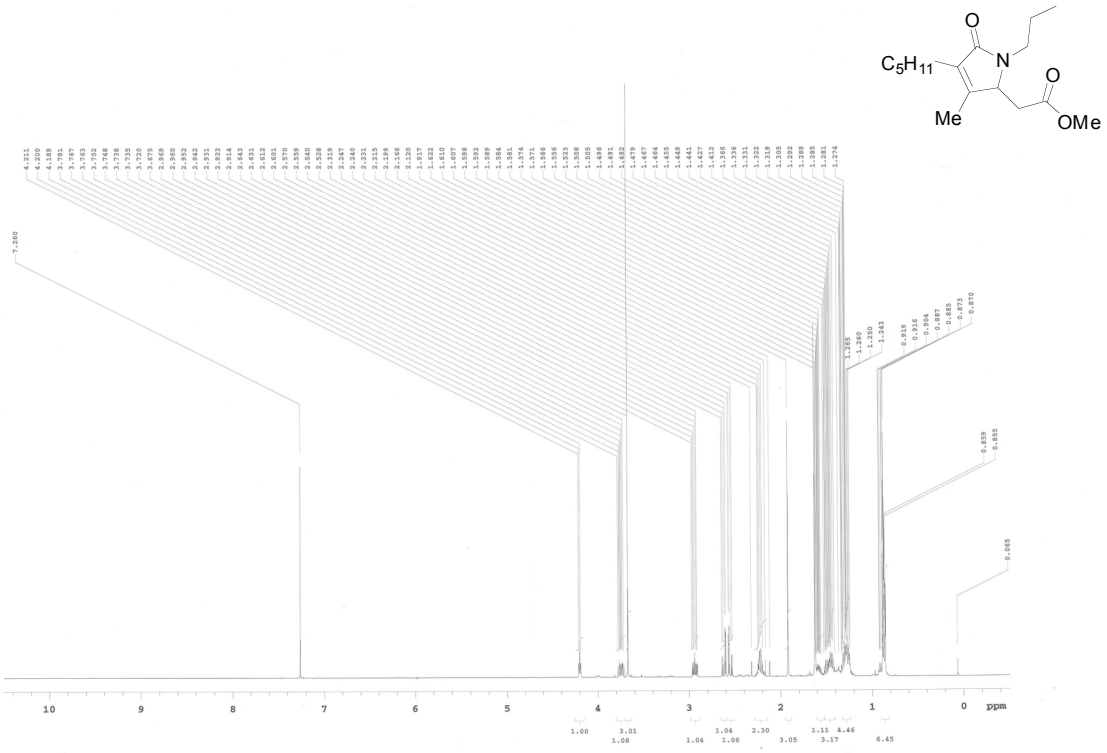

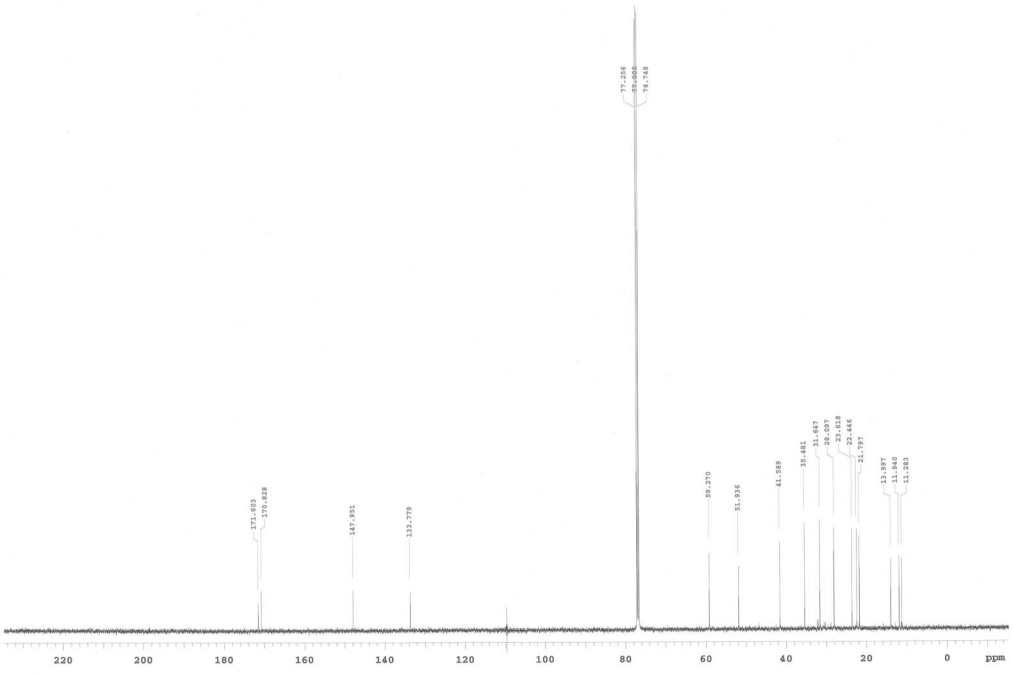

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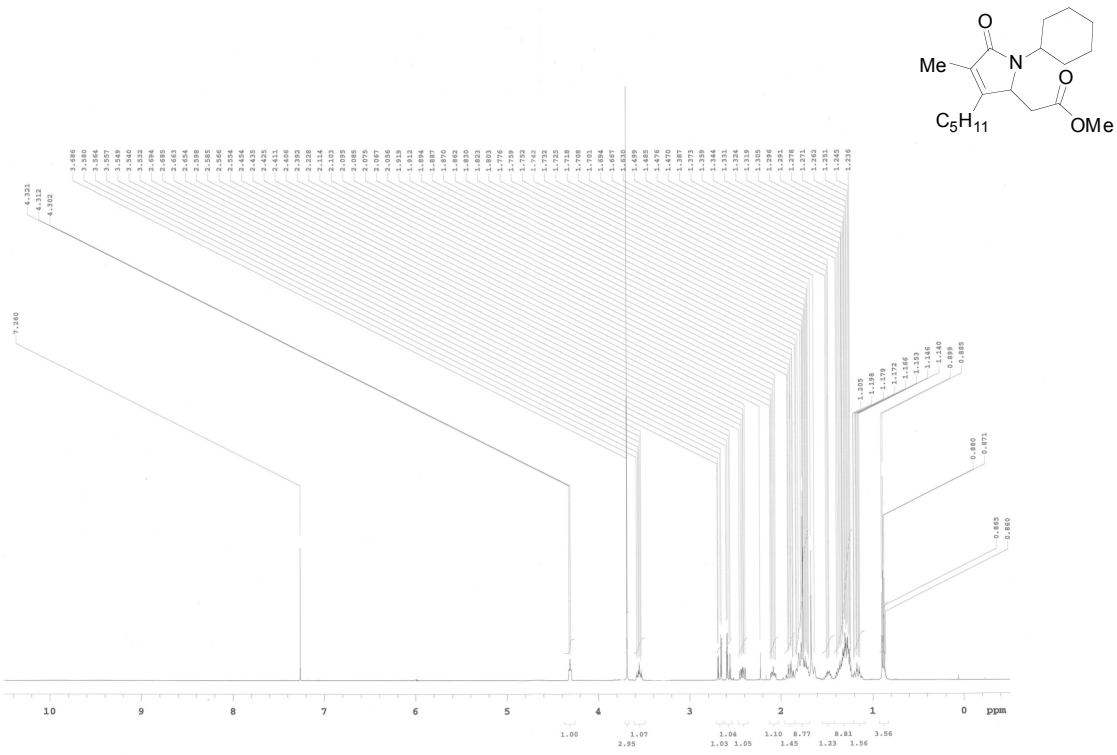

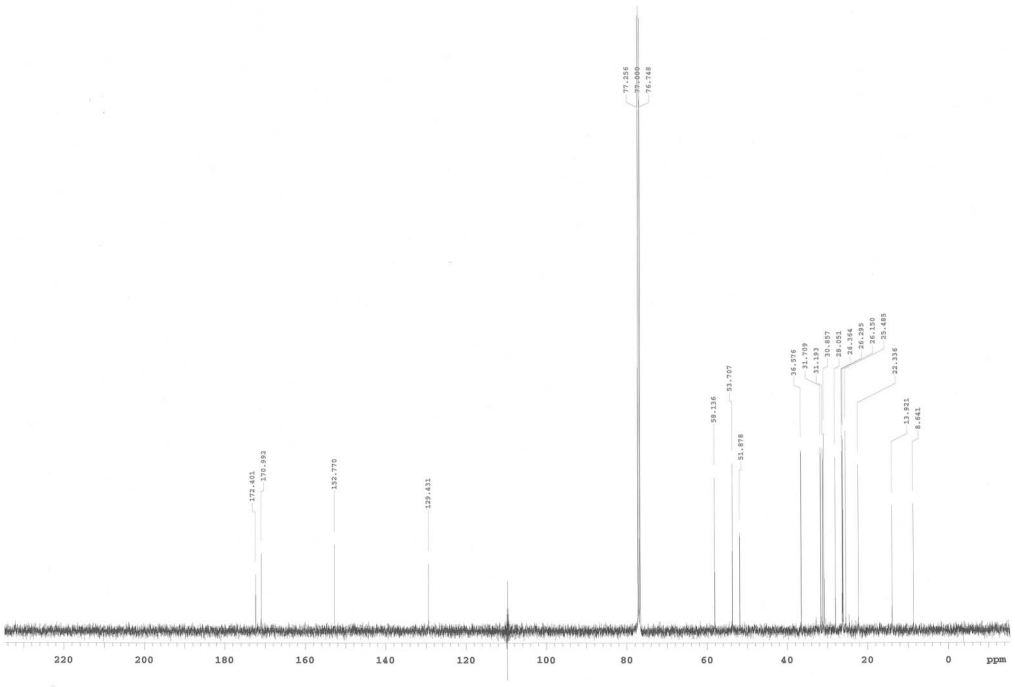

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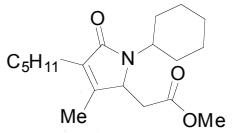

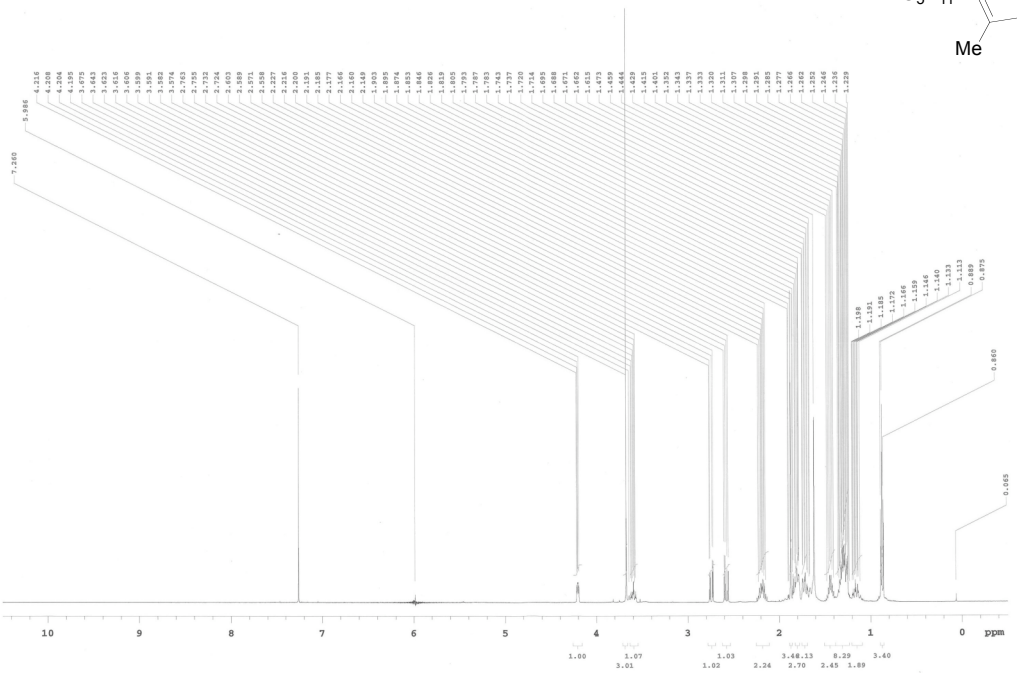

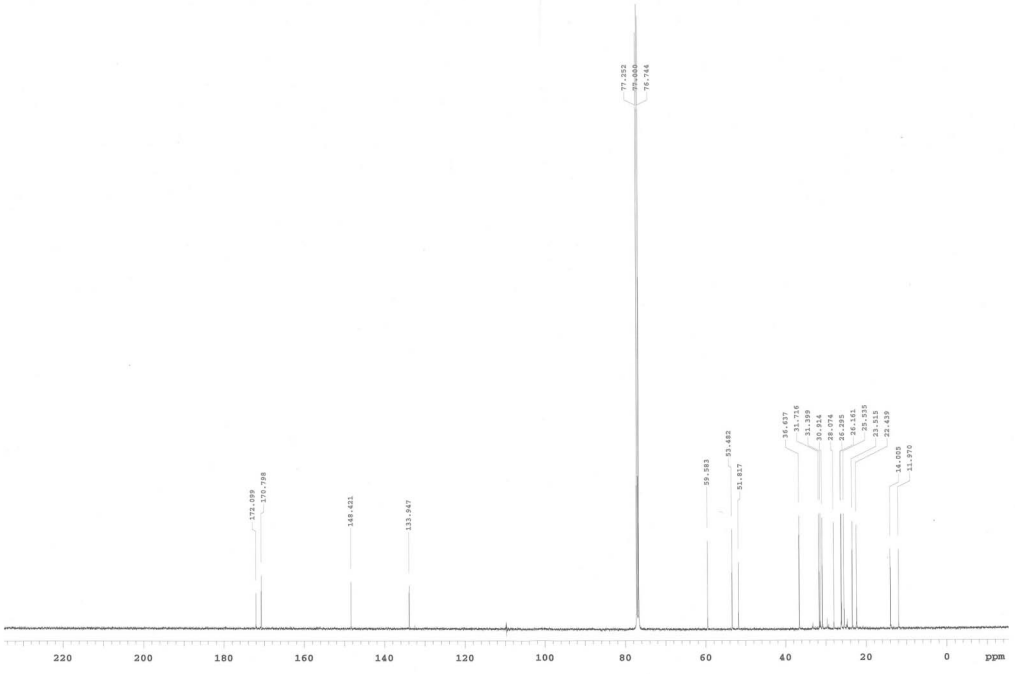

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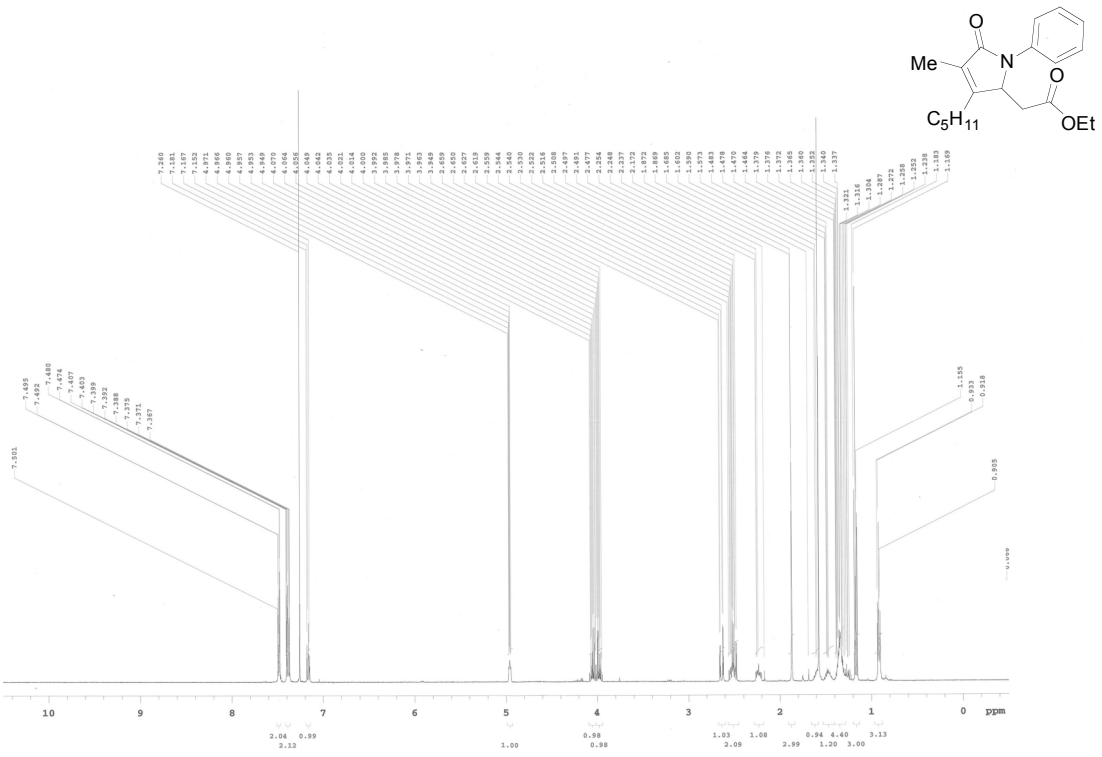

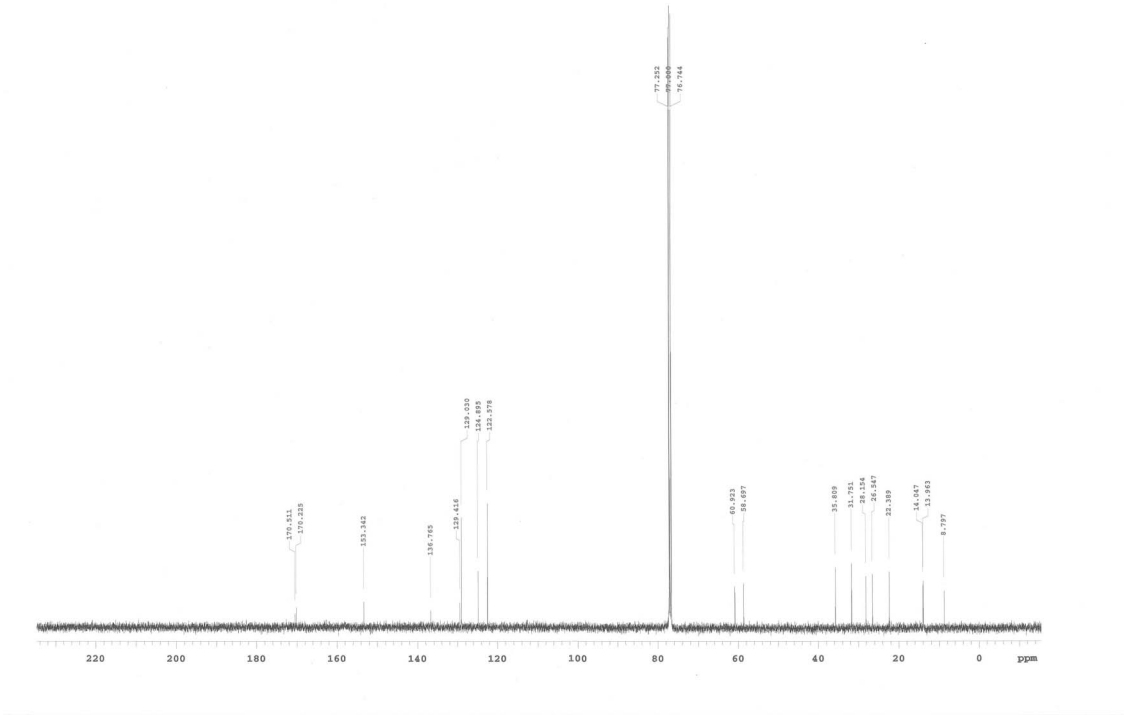

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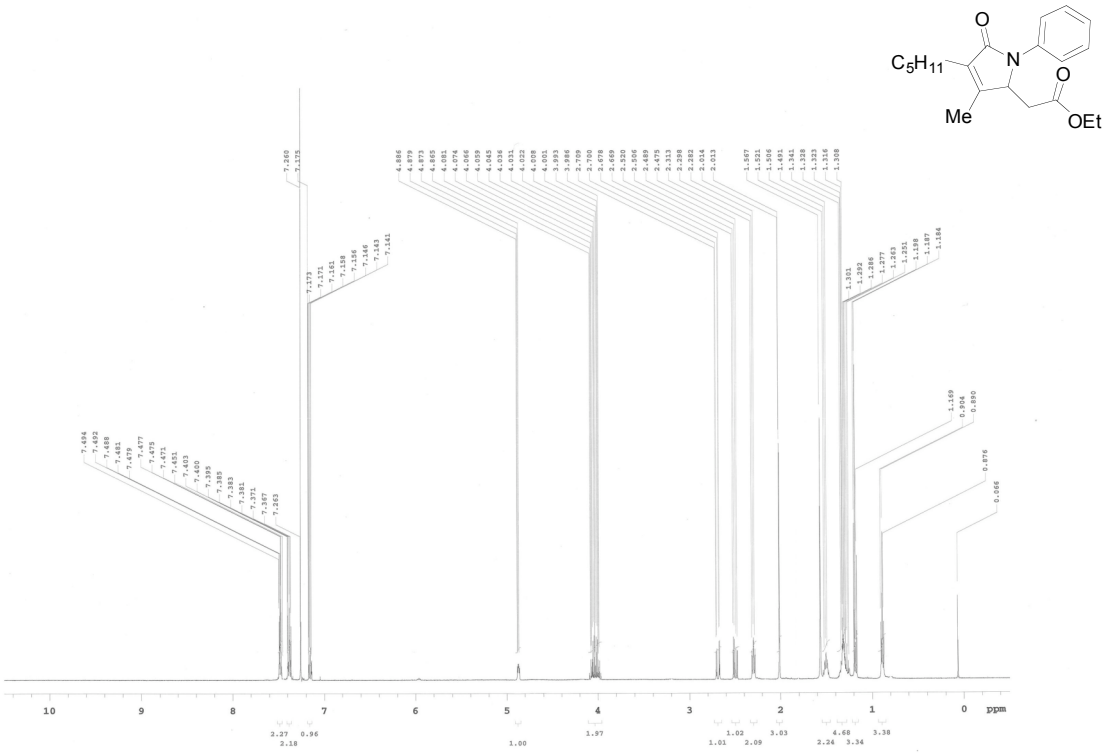

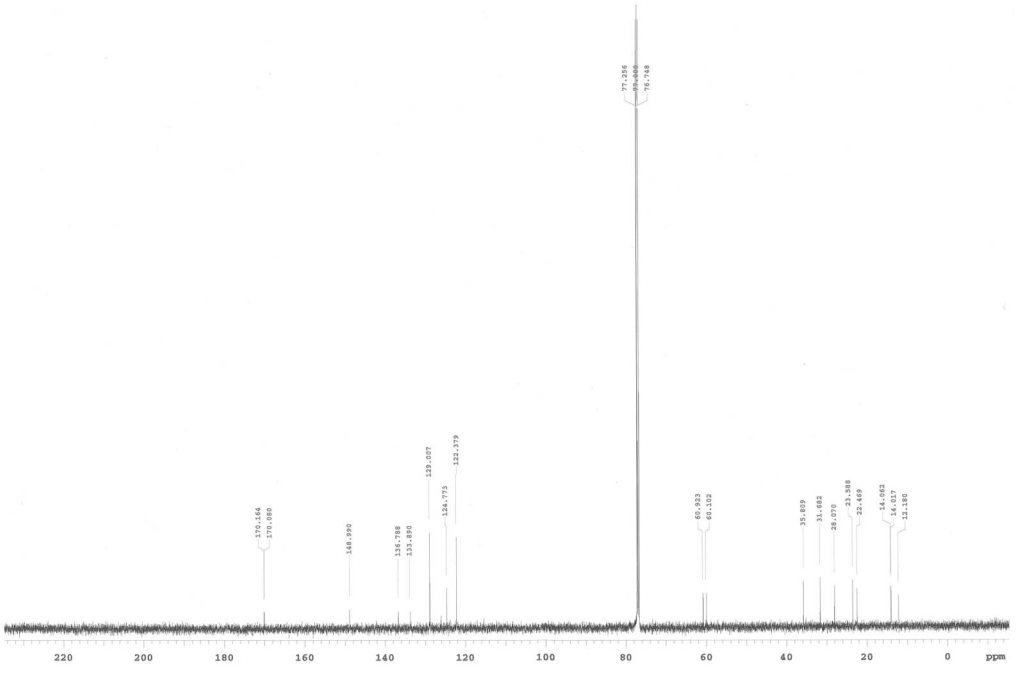

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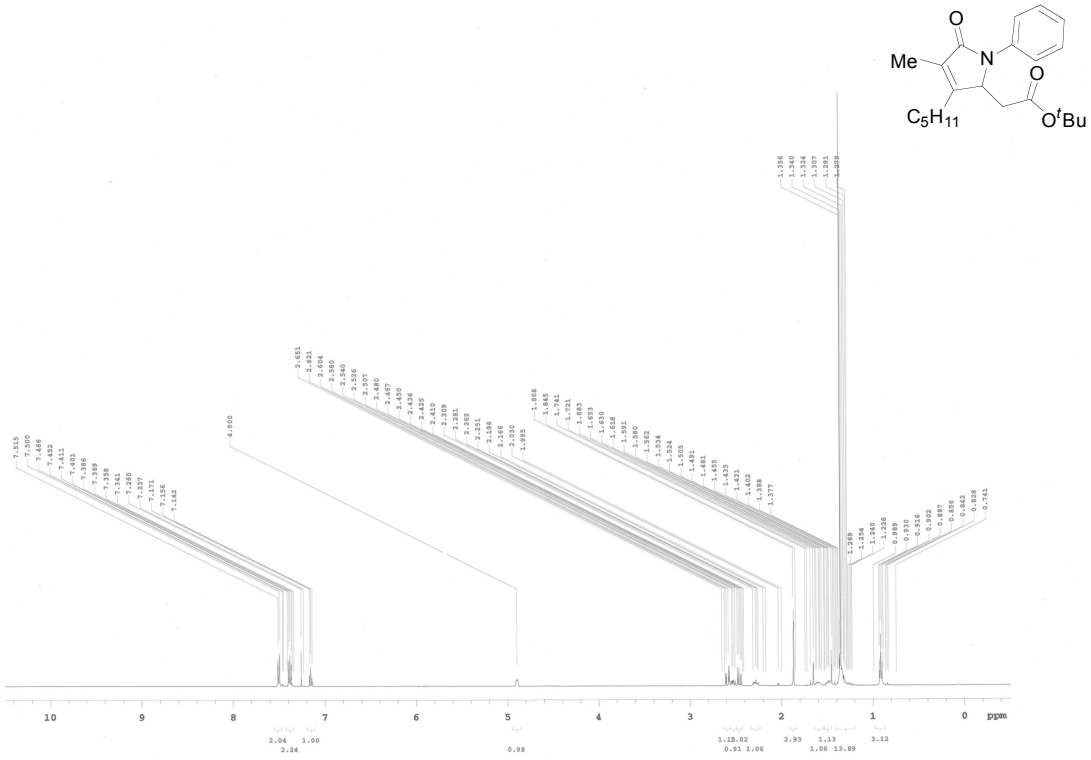

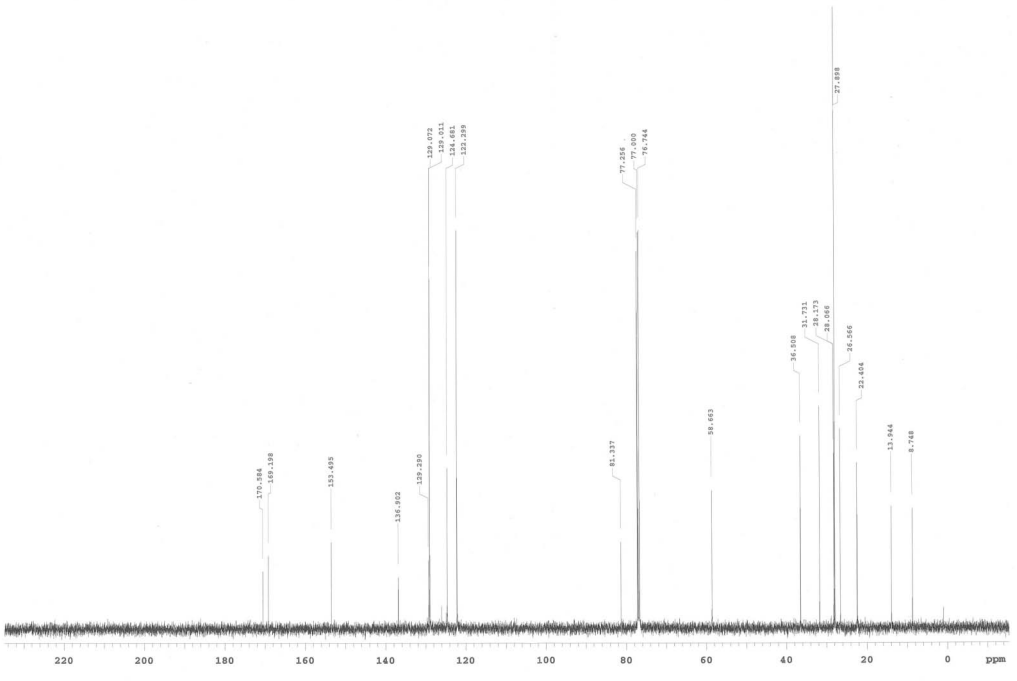

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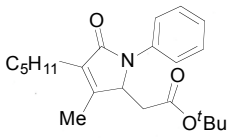

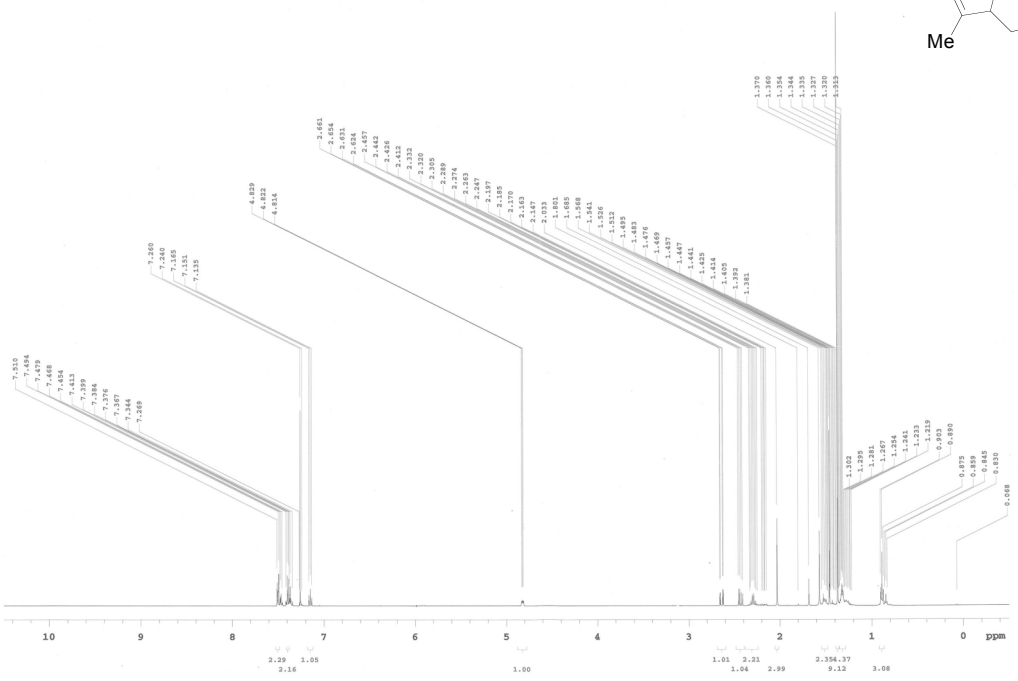

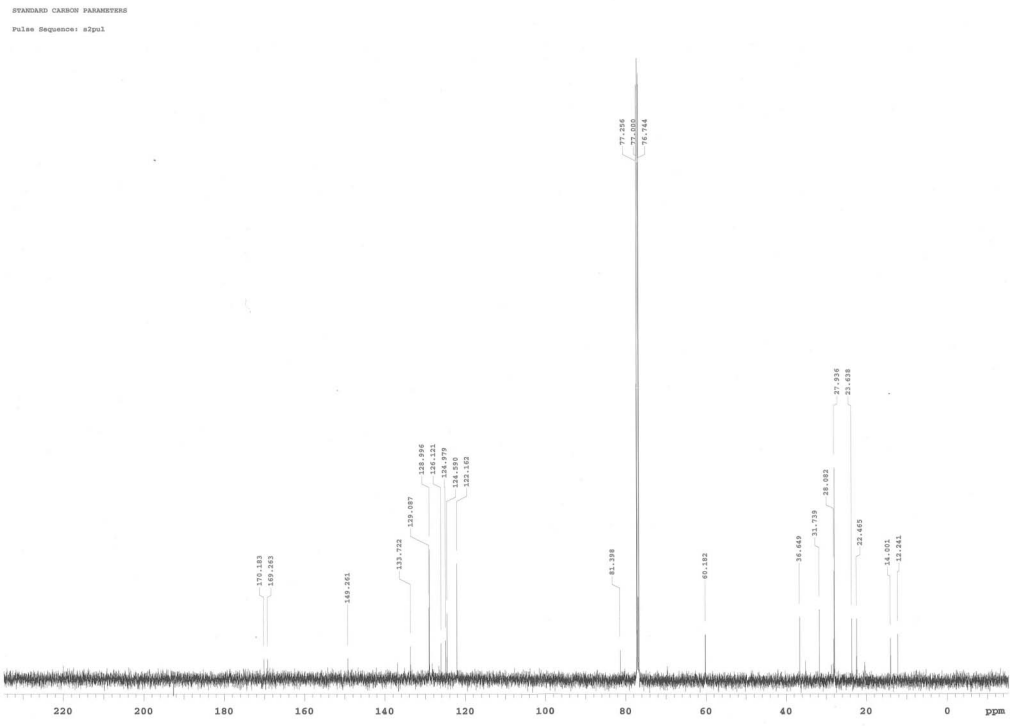

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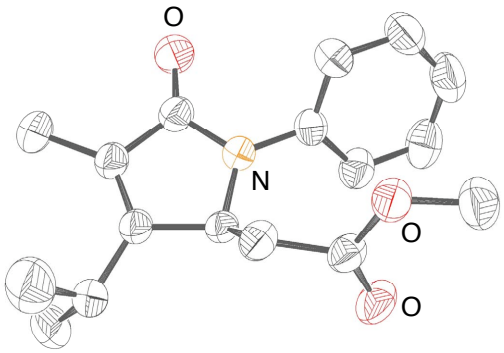
ORTEP Drawing of 4faa



Empirical formula	C17 H21 N O3	
Formula weight	287.35	
Temperature	299(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 7.5585(9) Å	⟨= 81.294(2)°.
	b = 9.6085(11) Å	®= 73.303(2)°.
	c = 11.6562(14) Å	© = 75.162(2)°.
Volume	781.04(16) Å^3	
Z	2	
Density (calculated)	1.222 Mg/m^3	
Absorption coefficient	0.083 mm^{-1}	
F(000)	308	
Crystal size	1.00 x 1.00 x 1.00 mm^3	
Theta range for data collection	2.20 to 26.99°.	
Index ranges	-9<=h<=9, -10<=k<=12, -14<=l<=12	
Reflections collected	4726	
Independent reflections	3303 [R(int) = 0.0202]	
Completeness to theta = 26.99°	96.7 %	
Absorption correction	None	
Max. and min. transmission	0.9212 and 0.9212	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3303 / 0 / 194	
Goodness-of-fit on F^2	0.894	
Final R indices [I>2sigma(I)]	R1 = 0.0556, wR2 = 0.1916	
R indices (all data)	R1 = 0.0632, wR2 = 0.2090	
Largest diff. peak and hole	0.342 and -0.216 e.Å^{-3}	

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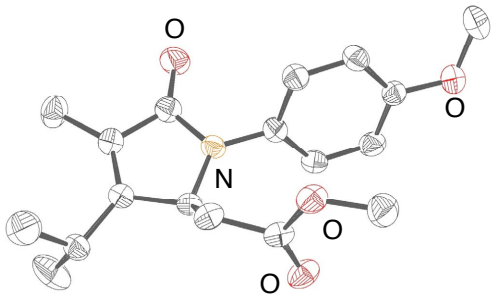
ORTEP Drawing of 4fab



Empirical formula	C18 H23 N O4	
Formula weight	317.37	
Temperature	273(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 13.318(2) Å	⟨= 90°.
	b = 9.6924(15) Å	®= 114.026(2)°.
	c = 14.726(2) Å	© = 90°.
Volume	1736.3(5) Å^3	
Z	4	
Density (calculated)	1.214 Mg/m^3	
Absorption coefficient	0.085 mm^{-1}	
F(000)	680	
Crystal size	1.00 x 0.50 x 0.10 mm^3	
Theta range for data collection	1.51 to 27.04°.	
Index ranges	-17<=h<=16, -12<=k<=12, -12<=l<=18	
Reflections collected	10596	
Independent reflections	7345 [R(int) = 0.0178]	
Completeness to theta = 27.04°	96.6 %	
Absorption correction	None	
Max. and min. transmission	0.9915 and 0.9194	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7345 / 0 / 425	
Goodness-of-fit on F^2	1.038	
Final R indices [I>2sigma(I)]	R1 = 0.0563, wR2 = 0.1535	
R indices (all data)	R1 = 0.0833, wR2 = 0.1734	
Largest diff. peak and hole	0.205 and -0.159 e.Å^{-3}	

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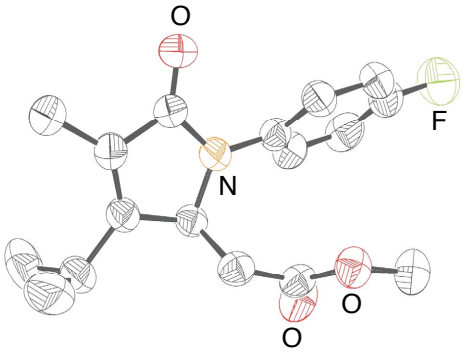
ORTEP Drawing of 4fac



Empirical formula	C17 H20 F N O3	
Formula weight	305.34	
Temperature	273(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 7.551(2) Å	⟨= 83.585(5)°.
	b = 9.516(3) Å	®= 73.507(5)°.
	c = 12.088(4) Å	© = 77.383(5)°.
Volume	811.6(4) Å^3	
Z	2	
Density (calculated)	1.250 Mg/m^3	
Absorption coefficient	0.093 mm^{-1}	
F(000)	324	
Crystal size	1.00 x 1.00 x 1.00 mm^3	
Theta range for data collection	2.20 to 27.22°.	
Index ranges	-9<=h<=9, -11<=k<=12, -6<=l<=15	
Reflections collected	5019	
Independent reflections	3478 [R(int) = 0.0145]	
Completeness to theta = 27.22°	95.8 %	
Absorption correction	None	
Max. and min. transmission	0.9129 and 0.9129	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3478 / 0 / 203	
Goodness-of-fit on F^2	1.074	
Final R indices [I>2sigma(I)]	R1 = 0.0506, wR2 = 0.1442	
R indices (all data)	R1 = 0.0585, wR2 = 0.1509	
Largest diff. peak and hole	0.233 and -0.190 e.Å^{-3}	

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