Supporting Information for

Photocatalytic [2+2] Cycloadditions of Enones with Cleavable Redox Auxiliaries

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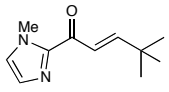
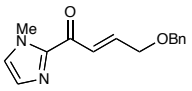
I. General information

A 23 W GE compact fluorescent light bulb was used for all photochemical reactions. *i*-Pr_2NEt was purified by distillation from CaH_2 immediately prior to use. $Ru(bpy)_3Cl_2{\cdot}6H_2O$ was purchased from commercial sources and used without further purification. Methyl acrylate was washed with aqueous NaOH, distilled water, brine, dried over $CaCl_2$, and fractionally distilled immediately prior to use. Benzene, CH_2Cl_2, THF, and MeCN were purified by elution through alumina. All other reagents were purchased from commercial sources and purified immediately prior to use. Chromatography was performed with Purasil 60Å silica gel (230–400 mesh). All glassware was oven-dried for at least 1 h before use. Diastereomer ratios for all compounds were determined by ^{1}H NMR analysis of the unpurified reaction mixtures. ^{1}H and ^{13}C NMR data for all previously uncharacterized compounds were obtained using Varian Inova-500 and Varian Unity-500 spectrometers and are referenced to TMS (0.00 ppm) and $CDCl_3$ (77 ppm), respectively. IR spectral data were obtained using a Bruker Vector 22 spectrometer (thin film on NaCl). Mass spectrometry was performed with a Micromass LCT (electrospray ionization, time-of-flight analyzer or electron impact). These facilities are funded by the NSF (CHE-9974839, CHE-9304546) and the University of Wisconsin. Cyclic voltammetry (CV) was conducted on a BAS Epsilon-EC instrument using MeCN with 0.1 M tetrabutylammonium hexafluorophosphate (nBu_4NPF_6) and 1 mM substrate. The electrodes were as follows: Glassy carbon (working), Pt wire (auxiliary) and $Ag/AgNO_3$ (0.1 M nBu_4NPF_6, 0.01 M $Ag/AgNO_3$) (reference). The potentials were referenced versus the ferrocene/ferrocenium redox couple by externally added ferrocene.

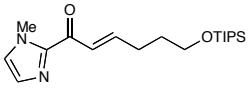
II. Synthesis of cyclization substrates



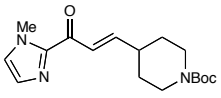
(*E*)-4,4-Dimethyl-1-(1-methyl-1*H*-imidazol-2-yl)pent-2-en-1-one: To a flame-dried 100 mL round bottomed flask was added 1-(1-methyl-1*H*-imidazole-2-yl)-2-(triphenylphosphoranylidene)-ethanone[1] (1.33 g, 3.45 mmol), benzene (18 mL), and freshly distilled trimethylacetaldehyde (2.97 g, 34.5 mmol). The reaction was heated to 75 °C and stirred for 105 h under N_2. After cooling to room temperature, the solvent was removed *in vacuo*, and the residue was purified by chromatography on a silica gel column (3:1 hexanes:EtOAc) to afford 0.563 g (2.93 mmol, 85 % yield) of a clear, viscous oil. IR (thin film): 2961, 1666, 1619, 1408 cm^{-1}; ^{1}H NMR: (499.9 MHz, $CDCl_3$) δ 7.34 (d, J = 16.0 Hz, 1H), 7.18 (d, J = 0.9 Hz, 1H), 7.13 (d, J = 16.0 Hz, 1H), 7.05 (s, 1H), 4.05 (s, 3H), 1.16 (s, 9H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 181.3, 158.5, 143.9, 129.1, 127.1, 121.2, 36.3, 34.1, 28.7; HRMS (EI) calc'd for $[C_{11}H_{16}N_2ONa]^+$ requires *m/z* 215.1160, found *m/z* 215.1155.



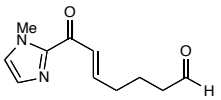
(*E*)-4-Benzyloxy-1-(1-methyl-1*H*-imidazol-2-yl)but-2-en-1-one: To a flame-dried 25 mL round bottomed flask was added 1-(1-methyl-1*H*-imidazole-2-yl)-2-(triphenylphosphoranylidene)-ethanone (0.782 g, 2.03 mmol), benzene (11 mL), and freshly distilled benzyloxyacetaldehyde (0.336 g, 2.24 mmol). The reaction was heated to 70 °C and stirred for 5 h under N_2. After cooling to room temperature the solvent was removed *in vacuo*, and the residue was purified by chromatography on a silica gel column (3:2 hexanes:EtOAc) to afford 0.485 g (1.89 mmol, 93% yield) of a clear, viscous oil that turned slightly yellow upon standing. IR (thin film): 2922, 1724, 1669, 1625, 1407 cm^{-1}; ^1H NMR: (500.2 MHz, $CDCl_3$) δ 7.65 (dt, J = 16.0, 2.0 Hz, 1H), 7.36 (m, 4H), 7.29 (m, 1H), 7.17 (d, J = 1.0 Hz, 1H), 7.14 (dt, J = 16.0, 4.5 Hz, 1H), 7.04 (s, 1H), 4.58 (s, 2H), 4.27 (dd, J = 5.0, 2.0 Hz, 2H), 4.03 (s, 3H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 180.3, 143.6, 143.3, 137.9, 129.4, 128.4, 127.8, 127.3, 126.3, 72.7, 69.3, 36.2. HRMS (EI) calc'd for $[C_{15}H_{16}N_2O_2Na]^+$ requires *m/z* 279.1109, found *m/z* 279.1104.



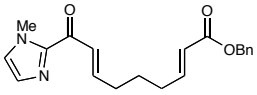
(*E*)-1-(1-Methyl-1*H*-imidazol-2-yl)-6-triisopropylsilanyloxy-hex-2-en-1-one: To a flame-dried 25 mL round bottomed flask was added 1-(1-methyl-1*H*-imidazole-2-yl)-2-(triphenylphosphoranylidene)-ethanone (0.612 g, 1.59 mmol), benzene (8 mL), and 4-((tris(1-methylethyl)silyl)oxy)-butanal[2] (0.389 g, 1.59 mmol). The reaction was warmed to 60 °C and stirred for 22 h under N_2. After cooling to room temperature the solvent was removed *in vacuo*, and the residue was purified by chromatography on a silica gel column (4:1 hexanes:EtOAc) to afford 0.318 g (0.907 mmol, 57% yield) of a clear, viscous oil. IR (thin film): 2943, 2866, 1668, 1622, 1409 cm^{-1}; ^1H NMR: (500.2 MHz, $CDCl_3$) δ 7.42 (dt, J = 15.7, 1.6 Hz, 1H), 7.17 (dt, J = 15.7, 7.1 Hz, 1H), 7.17 (d, J = 0.7 Hz, 1H), 7.04 (s, 1H), 4.05 (s, 3H), 3.74 (t, J = 6.3 Hz, 1H), 2.42 (ddt, J = 7.3, 7.2, 1.5 Hz, 2H), 1.77 (m, 2H), 1.06 (m, 21H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 180.8, 148.6, 143.8, 129.1, 127.1, 126.2, 62.7, 36.3, 31.6, 29.3, 18.0, 11.9; HRMS (EI) calc'd for $[C_{19}H_{34}N_2O_2SiNa]^+$ requires *m/z* 373.2287, found *m/z* 373.2282.



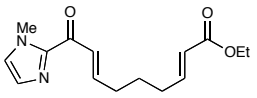
(*E*)-*tert*-Butyl 4-(3-(1-methyl-1H-imidazol-2-yl)-3-oxoprop-1-en-1-yl)piperidine-1-carboxylate: To a flame-dried 25 mL round-bottomed flask was added 1-(1-methyl-1*H*-imidazole-2-yl)-2-(triphenylphosphoranylidene)-ethanone (0.587 g, 1.53 mmol), benzene (8 mL), and 4-formylpiperidine-1-carboxylic acid tert-butyl ester[3] (0.391 g, 1.83 mmol). The reaction was heated to 75 °C and was stirred for 44 h under N_2. After cooling to room temperature the solvent was removed *in vacuo*, and the residue was purified by chromatography on a silica gel column (7:3 hexanes:EtOAc) to afford 0.354 g (1.11 mmol, 73% yield) of a clear, viscous oil. IR (thin film): 2934, 1690, 1688, 1621, 1410 cm^{-1}; ^1H NMR: (500.2 MHz, $CDCl_3$) δ 7.41 (dd, J = 15.8, 1.4 Hz, 1H), 7.18 (d, J = 0.8 Hz, 1H), 7.06 (s, 1H), 7.04 (dd, J = 15.8, 6.7 Hz, 1H), 4.13 (m, 2H), 4.05 (s, 3H), 2.79 (m, 2H), 2.41 (m, 1H), 1.80 (d, J = 12.5 Hz, 2H), 1.45 (m, 10H), 1.40 (ddd, J = 13.2, 5.1, 1.1 Hz, 1H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 180.7, 154.8, 150.8, 143.7, 129.3, 127.2, 124.7, 79.5, 43.5, 39.0, 36.3, 30.7, 28.4; HRMS (EI) calc'd for $[C_{17}H_{25}N_3O_3Na]^+$ requires *m/z* 342.1794, found *m/z* 342.1789.



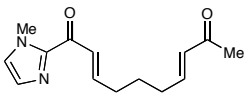
(*E*)-7-(1-Methyl-1H-imidazol-2-yl)-7-oxohept-5-enal (S-1): 1-(1-Methyl-1*H*-imidazole-2-yl)-2-(triphenylphosphoranylidene)-ethanone (5.6 g, 14.6 mmol) was placed in a 100 mL round-bottomed flask with 36 mL (0.4 M) CH_2Cl_2. 6 mL of a 50 wt. % in water solution of glutaraldehyde (3 g, 30.0 mmol) was added. The mixture was heated to 45 ºC and stirred overnight. Upon completion, the reaction was filtered over Celite and the filtrate was concentrated *in vacuo*. The residue was triturated three times with diethyl ether to remove triphenylphosphine oxide. The crude reaction mixture was then purified by chromatography on a silica gel column (2:1 hexanes:acetone) to afford 2.8 g (13.6 mmol, 93% yield) of a light yellow oil. IR (thin film) 2944, 2728, 1720, 1665, 1619, 1408 cm^{-1}; ^1H NMR: (499.9 MHz, $CDCl_3$) δ 9.78 (t, J = 1.4 Hz, 1H), 7.43 (dt, J = 15.7, 1.6 Hz, 1H), 7.18 (s, 1H), 7.07 (m, = 1H), 7.06 (s, 1H), 4.05 (s, 3H), 2.51 (td, J = 7.4, 1.4 Hz, 2H), 2.37 (qd, J = 7.4, 1.4 Hz, 2H), 1.88 (dq, J = 7.3, 7.4 Hz, 2H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 201.7, 180.4, 146.7, 129.2, 127.2, 127.0, 43.0, 42.7, 36.2, 31.7, 20.5; HRMS (EI) calc'd for $[C_{11}H_{14}N_2O_2Na]^+$ requires *m/z* 229.0948, found *m/z* 229.0958.



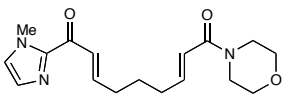
(2*E*,7*E*)-Benzyl 9-(1-methyl-1*H*-imidazol-2-yl)-9-oxonona-2,7-dienoate: A 25 mL round-bottomed flask was charged with **S-1** (950 mg, 4.6 mmol), benzyl 2-(triphenylphosphoranylidene)acetate[4] (2.5 g, 6.1 mmol) and 5 mL (0.9 M) CH_2Cl_2. The solution was stirred at room temperature for 18 h. The solvent was then removed *in vacuo* and the residue was triturated three times with diethyl ether to remove triphenylphosphine oxide. The crude reaction mixture was then purified by chromatography on a silica gel column (4:1 to 3:1 hexanes:acetone) to afford 1.4 g (4.1 mmol, 90 % yield) of a clear viscous oil. IR (thin film) 2950, 1732, 1670, 1409 cm^{-1}; ^1H NMR: (499.9 MHz, $CDCl_3$) δ 7.41 (dt, J = 15.6, 1.7 Hz, 1H), 7.37 (m, 5H), 7.17 (d, J = 0.9 Hz, 1H), 7.04 (s, 1H), 7.08 (dt, J = 15.7, 7.2 Hz, 1H), 7.00 (dt, J = 16.1, 6.8 Hz, 1H), 5.89 (dt, J = 15.6, 1.5 Hz, 1H), 5.17 (s, J = Hz, 2H), 4.04 (s, 3H), 2.35 (qd, J = 7.4, 1.7 Hz, 2H), 2.26 (qd, J = 7.4, 1.7 Hz, 2H), 1.69 (m, 2H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 180.5, 166.3, 148.9, 147.3, 143.6, 136.1, 129.2, 128.5, 128.2, 128.1, 127.1, 126.8, 121.6, 66.0, 36.3, 31.9, 31.6, 26.4; HRMS (EI) calc'd for $[C_{20}H_{21}N_2O_3+H]^+$ requires *m/z* 338.1625, found *m/z* 338.1621.



(2E,7E)-Ethyl 9-(1-methyl-1H-imidazol-2-yl)-9-oxonona-2,7-dienoate: A 25 mL round-bottomed flask was charged with **S-1** (750 mg, 3.42 mmol), ethyl 2-(triphenylphosphoranylidene)acetate[5] (2.03 g, 5.3 mmol) and 6 mL (0.6 M) CH_2Cl_2. The solution was stirred at room temperature for 6 h. The solvent was then removed *in vacuo* and the residue was triturated three times with diethyl ether to remove triphenylphosphine oxide. The crude reaction mixture was then purified by chromatography on a silica gel column (4:1 to 2:1 hexanes:acetone) to afford 810 mg (2.93 mmol, 86% yield) of a light yellow viscous oil. IR (thin film) 2936, 1716, 1667, 1408 cm^{-1}; ^1H NMR: (499.9 MHz, $CDCl_3$) δ 7.43 (dt, J = 15.5, 1.8 Hz, 1H), 7.17 (d, J = 0.9 Hz, 1H), 7.09 (dt, J = 15.5, 5.7 Hz, 1H), 7.05 (s, 1H), 6.94 (dt, J = 15.5, 6.7 Hz), 5.83 (dt, J = 15.5, 1.5 Hz, 1H), 4.19 (q, J = 7.4 Hz, 2H), 4.05 (s, 3H), 2.35 (qd, J = 7.4, 1.8 Hz, 2H), 2.26 (qd, J = 7.4, 1.8 Hz, 2H), 1.71 (dt, J = 7.4, 7.2 Hz, 2H), 1.29 (t, J = 7.1 Hz, 3H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 180.6, 166.6, 148.1, 147.3, 143.6, 129.2, 127.1, 126.8, 121.9, 60.2, 36.25, 31.9, 31.5, 26.5, 14.2; HRMS (EI) calc'd for $[C_{15}H_{19}N_2O_3+H]^+$ requires *m/z* 276.1469, found *m/z* 276.1458.

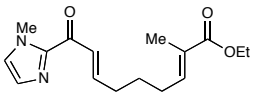


(2*E*,7*E*)-1-(1-Methyl-1*H*-imidazol-2-yl)deca-2,7-diene-1,9-dione: A 25 mL round-bottomed flask was charged with **S-1** (250 mg, 1.21 mmol), 1-(triphenylphosphoranylidene)propan-2-one[6] (660 mg, 2.1 mmol) and 10 mL (0.12 M) CH_2Cl_2. The solution was stirred at room temperature for 12 h. The solvent was then removed *in vacuo* and the crude reaction mixture was loaded directly onto a silica gel column and eluted with 2:1 hexanes:acetone to afford 180 mg (0.73 mmol, 60% yield) of a colorless viscous oil. IR (thin film) 2932, 1667, 1620, 1407, 1362 cm^{-1}; ^1H NMR: (499.9 MHz, $CDCl_3$) δ 7.43 (dt, J = 15.7, 1.4 Hz, 1H), 7.18 (d, J = 0.9 Hz, 1H), 7.09 (dt, J = 15.7, 6.8 Hz, 1H), 7.06 (s, 1H), 6.79 (dt, J = 15.7, 6.8 Hz, 1H), 6.09 (dt, J = 15.7, 1.4 Hz, 1H), 4.05 (s, 3H), 2.35 (qd, J = 7.2, 1.7 Hz, 2H), 2.28 (qd, J = 7.2, 1.3 Hz, 2H), 2.25 (s, 3H), 1.73 (m, 2H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 198.5, 180.4, 147.2, 147.1, 143.5, 131.6, 129.2, 127.1, 126.8, 36.2, 31.9, 31.7, 26. 8, 26.5; HRMS (EI) calc'd for $[C_{14}H_{18}N_2O_2+Na]^+$ requires *m/z* 269.1261, found *m/z* 269.1249.



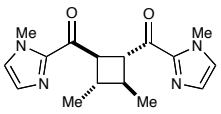
(2*E*,7*E*)-1-(1-Methyl-1*H*-imidazol-2-yl)-9-morpholinonona-2,7-diene-1,9-dione: A 25 mL round-bottomed flask was charged with 1.0 equivalent NaH dissolved in 3 mL dry THF at room temperature. Diethyl (2-morpholino-2-oxoethyl)phosphonate[7] was added to the stirring solution dropwise (60.5 mg, 0.228 mmol, 1.2 equiv), and the reaction was allowed to stir until solution became clear. A solution of **S-1** (40 mg, 0.194 mmol) in 0.5 mL dry THF was added dropwise, and the reaction was stirred for 15 min. The reaction was then diluted with 5 mL diethyl ether and washed with water. The organic layer was separated and dried over Na_2SO_4. After filtration, the solution was concentrated *in vacuo* and purified by chromatography on a silica gel column (1:1 acetone:hexanes) to afford 35 mg (0.11 mmol, 57% yield) of a colorless oil. IR (thin film) 2858, 1662, 1618, 1407 cm^{-1}; ^1H NMR: (499.9 MHz, $CDCl_3$) δ 7.42 (dt, J = 15.7, 1.5

Hz, 1H), 7.16 (d, J = 0.9 Hz, 1H), 7.09 (dt, J = 15.7, 6.7 Hz, 1H), 7.05 (s, 1H), 6.89 (dt, J = 15.1, 7.0 Hz, 1H), 6.23 (dt, J = 15.0, 1.5 Hz, 1H), 4.05 (s, 1H), 3.5-3.75 (m, 8H), 2.36 (qd, J = 7.4, 1.7 Hz, 2H), 2.27 (qd, J = 7.4, 1.6 Hz, 2H), 1.72 (m, 2H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 180.5, 165.5, 147.4, 146.0, 143.6, 129.2, 127.2, 126.8, 120.1, 66.8, 46.1, 42.2, 36.3, 31.9, 31.9, 26.7: HRMS (EI) calc'd for $[C_{17}H_{23}N_3O_3+H]^+$ requires *m/z* 318.1813, found *m/z* 318.1812.



(2*E*,7*E*)-Ethyl 2-methyl-9-(1-methyl-1*H*-imidazol-2-yl)-9-oxonona-2,7-dienoate: A 25 mL round-bottomed flask was charged with **S-1** (470 mg, 1.62 mmol), ethyl 2-(triphenylphosphoranylidene)propanoate[8] (1.17 g, 3.23 mmol) and 25 mL (0.06 M) CH_2Cl_2. The solution was stirred at room temperature overnight. The solvent was then removed *in vacuo* and the crude reaction mixture was loaded directly onto a silica gel column and eluted with 3:1 hexanes:acetone to afford 180 mg (0.619 mmol, 38% yield) as a colorless viscous oil. IR (thin film) 2360, 2254, 1700, 1666, 1621 cm^{-1}; ^{1}H NMR: (499.9 MHz, $CDCl_3$) δ 7.43 (dt, J = 15.5, 1.9 Hz, 1H), 7.17 (d, J = 1.0 Hz, 1H), 7.11 (dt, J = 15.6, 7.1 Hz, 1H), 7.05 (s, 1H), 6.75 (tq, J = 6.75, 1.7 Hz, 1H), 4.19 (q, J = 7.0 Hz, 2H), 4.05 (s, 3H), 2.36 (qd, J = 7.1, 1.5 Hz, 2H), 2.23 (q, J = 7.3 Hz, 2H), 1.83 (q, J = 1.0 Hz, 3H), 1.70 (dq, J = 7.6, 15.0 Hz, 2H), 1.30 (t, J = 7.0 Hz, 3H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 180.5, 168.1, 147.6, 143.6, 141.1, 129.2, 128.4, 127.1, 126.6, 60.4, 36.2, 32.1, 28.1, 27.1, 14.2, 12.4; HRMS (EI) calc'd for $[C_{16}H_{22}N_2O_3+H]^+$ requires *m/z* 291.1704, found *m/z* 291.1712.

III. Photocycloadditions

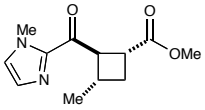


(3,4-Dimethylcyclobutane-1,2-diyl)bis((1-methyl-1H-imidazol-2-yl)methanone) (2). To an oven-dried 25 mL Schlenk tube was added 99.6 mg (0.663 mmol) (*E*)-1-(1-methyl-1*H*-imidazole-2-yl)-but-2-en-1-one, 12.7 mg (0.017 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 61.7 mg (0.663 mmol) $LiBF_4$, 120 μL (0.651 mmol) *i*-Pr_2NEt, and 3.3 mL acetonitrile. The solution was degassed using three freeze-pump-thaw cycles in the dark. The Schlenk tube was backfilled with nitrogen and irradiated for 4 h. Upon completion of the reaction, the reaction was diluted with acetone, passed through a silica plug, and the solvent was removed *in vacuo*. Purified by chromatography (2:1 hexanes:acetone) to afford 82.5 mg (0.275 mmol, 83% yield, >10:1 d.r.) of the cycloadduct. Experiment 2: 99.8 mg (0.664 mmol) enone, 12.6 mg (0.017 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 61.9 mg (0.666 mmol) $LiBF_4$, 120 μL (0.651 mmol) *i*-Pr_2NEt, and 3.3 mL acetonitrile. Isolated 80.7 mg (0.269 mmol, 81% yield, >10:1 d.r.). IR (thin film): 2954, 2921, 1665, 1405 cm^{-1}; ^{1}H NMR: (500.2 MHz, $CDCl_3$) δ 6.96 (d, J = 0.7 Hz, 2H), 6.94 (s, 2H), 4.14 (dd, J = 5.6, 3.2 Hz, 2H), 4.00 (s, 6H), 2.18 (m, 2H), 1.21 (m, 6H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 191.8, 143.1, 129.0, 126.6, 48.9, 38.5, 36.0, 19.1; HRMS (EI) calc'd for $[C_{16}H_{20}N_4O_2Na]^+$ requires *m/z* 323.1484, found *m/z* 323.1479.

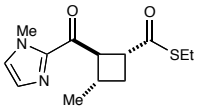
General procedure A: Intermolecular [2+2] cycloadditions (Figure 1): A dry 25 mL Schlenk tube was charged with the Michael acceptor (5 equiv) followed by a solution of $Ru(bpy)_3Cl_2{\cdot}6H_2O$ (0.025 equiv) and $LiBF_4$ (0.5–4.0 equiv) in dry MeCN (1.33 M with respect to the Michael acceptor). A second 25 mL Schlenk tube was charged a solution of imidazole enone (1 equiv) in dry MeCN (0.16 M with respect to the aryl enone). *i*-Pr_2NEt (2 equiv) was then added to the first tube. The solutions were degassed using three freeze-pump-thaw cycles under nitrogen in the dark. The Schlenk tubes were then placed in a water bath and irradiated using a 23 W compact fluorescent light bulb placed at a distance of 20 cm. The imidazole enone solution was added dropwise via syringe pump over a period of 45 min to the solution containing the Michael acceptor. Additional time was allowed for the reaction to reach completion (total reaction times listed in Table 3 refer to dropwise addition and subsequent irradiation combined). Upon completion of the reaction, the reaction was diluted with ethyl acetate, passed through a silica plug, and the solvent was removed *in vacuo*. The resulting residue was purified by chromatography on a silica gel column.

General procedure B: Intramolecular [2+2] cycloadditions (Figure 2): A dry 25 mL Schlenk tube was charged with a solution of the bisenone (1 equiv), $Ru(bpy)_3Cl_2{\cdot}6H_2O$ (0.025 equiv), $LiBF_4$ (0.5 equiv), and *i*-Pr_2NEt (2 or 0.5

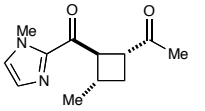
equiv) in acetonitrile (0.1 M). The solution was then degassed using three freeze-pump-thaw cycles under nitrogen in the dark. The Schlenk tube was then irradiated using a 23 W floodlight placed at a distance of 20 cm. Upon completion of the reaction, the reaction was passed through a silica plug using 100% ethyl acetate or acetone as the eluent, the solvent was removed *in vacuo*, and the residue was purified by chromatography on a silica gel column.



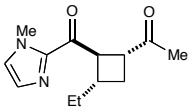
Methyl 3-methyl-2-(1-methyl-1H-imidazole-2-carbonyl)cyclobutanecarboxylate (3) (Figure 1): Prepared according to general procedure **A** using 104.2 mg (0.694 mmol) (*E*)-1-(1-methyl-1*H*-imidazole-2-yl)-but-2-en-1-one, 315 μL (3.50 mmol) methyl acrylate, 13.3 mg (0.018 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 130 mg (1.39 mmol) $LiBF_4$, 242 μL (1.39 mmol) *i*-Pr_2NEt, 6.94 mL acetonitrile and an irradiation time of 90 min. Purified by chromatography (23:2 hexanes:acetone) to afford 110 mg (0.466 mmol, 67% yield, >10:1 d.r.) of the cycloadduct as a clear oil. Experiment 2: 104.7 mg (0.697 mmol) enone, 315 μL (3.50 mmol) methyl acrylate, 13.1 mg (0.017 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 132 mg (1.41 mmol) $LiBF_4$, 245 μL (1.41 mmol) *i*-Pr_2NEt, and 6.97 mL acetonitrile. Isolated 111 mg (0.470 mmol, 67% yield, >10:1 d.r.). IR (thin film): 2954, 1733, 1668, 1411 cm^{-1}; ^1H NMR (499.9 MHz, $CDCl_3$) δ 7.14 (d, J = 0.9 Hz, 1H), 7.05 (s, 1H), 4.21 (dd, J = 8.9, 8.9 Hz, 1H), 4.02 (s, 3H), 3.66 (s, 3H), 3.46 (ddd, J = 9.0, 9.0, 9.0 Hz, 1H), 2.42 (m, 1H), 2.33 (ddd, J = 10.2, 8.8, 8.8 Hz, 1H), 1.92 (ddd, J = 10.3, 9.4, 9.4 Hz, 1H), 1.26 (d, J = 6.6 Hz, 3H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 190.9, 174.4, 142.6, 129.5, 127.3, 52.2, 51.7, 36.1, 35.1, 32.5, 29.3, 20.8; HRMS (EI) calc'd for $[C_{12}H_{16}N_2O_3Na]^+$ requires *m/z* 259.1059, found *m/z* 259.1054.



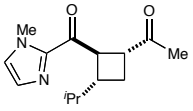
S-Ethyl 3-methyl-2-(1-methyl-1H-imidazole-2-carbonyl)cyclobutanecarbothioate (4) (Figure 1) : Prepared according to general procedure **A** using 109.4 mg (0.728 mmol) (*E*)-1-(1-methyl-1*H*-imidazole-2-yl)-but-2-en-1-one, 426 mg (3.67 mmol) ethyl prop-2-enethioate,[9] 13.5 mg (0.018 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 137 mg (1.46 mmol) $LiBF_4$, 255 μL (1.46 mmol) *i*-Pr_2NEt, 7.28 mL acetonitrile and an irradiation time of 90 min. Purified by chromatography (47:3 hexanes:acetone) to afford 150 mg (0.563 mmol, 77% yield, > 10:1 d.r.) of the cycloadduct as a clear oil. Experiment 2: 108.6 mg (0.723 mmol) enone, 421 mg (3.62 mmol) (S)-ethyl-prop-2-enethioate, 13.4 mg (0.018 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 136 mg (1.45 mmol) $LiBF_4$, 255 μL (1.46 mmol) *i*-Pr_2NEt, and 7.23 mL acetonitrile. Isolated 155 mg (0.582 mmol, 80% yield, >10:1 d.r.). IR (thin film): 2963, 2931, 1668, 1410 cm^{-1}; ^1H NMR: (500.2 MHz, $CDCl_3$) δ 7.15 (s, 1H), 7.06 (s, 1H), 4.26 (dd, J = 8.5, 8.5 Hz, 1H), 4.03 (s, 3H), 3.68 (ddd, J = 8.9, 8.9, 8.9 Hz 1H), 2.86 (q, J = 7.5 Hz, 2H), 2.39 (m, 1H), 2.33 (ddd, J = 10.1, 8.7, 8.7 Hz, 1H), 1.96 (ddd, J = 10.4, 9.3, 9.3 Hz, 1H), 1.26 (d, J = 6.6 Hz, 3H), 1.22 (t, J = 7.5 Hz, 3H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 200.0, 190.7, 142.6, 129.5, 127.3, 52.6, 43.1, 36.1, 32.5, 29.8, 23.0, 20.7, 14.7; HRMS (EI) calc'd for $[C_{13}H_{18}N_2O_2SNa]^+$ requires *m/z* 289.0987, found *m/z* 289.0982.



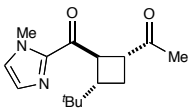
1-(3-Methyl-2-(1-methyl-1H-imidazole-2-carbonyl)cyclobutyl)ethanone (5) (Figure 1): Prepared according to general procedure **A** using 106.8 mg (0.711 mmol) (*E*)-1-(1-methyl-1*H*-imidazole-2-yl)-but-2-en-1-one, 290 μL (3.57 mmol) 3-buten-2-one, 13.4 mg (0.018 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 33.6 mg (0.358 mmol) $LiBF_4$, 250 μL (1.44 mmol) *i*-Pr_2NEt, 7.11 mL acetonitrile and an irradiation time of 90 min. Purified by chromatography (17:3 hexanes:acetone) to afford 119 mg (0.540 mmol, 76% yield, >10:1 d.r.) of the cycloadduct. Experiment 2: 103.4 mg (0.689 mmol) enone, 280 μL (3.45 mmol) 3-buten-2-one, 12.8 mg (0.0171 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 32.3 mg (0.344 mmol) $LiBF_4$, 240 μL (1.38 mmol) *i*-Pr_2NEt, and 6.89 mL acetonitrile. Isolated 111 mg (0.504 mmol, 73% yield, >10:1 d.r.) of the cycloadduct as a clear oil. IR (thin film): 2958, 1707, 1665, 1409 cm^{-1}; ^1H NMR: (500.2 MHz, $CDCl_3$) δ 7.15 (d, J = 0.6 Hz, 1H), 7.05 (s, 1H), 4.13 (dd, J = 8.8, 8.8 Hz, 1H), 4.03 (s, 3H), 3.56 (ddd, J = 9.1, 9.1, 9.1 Hz, 1H), 2.42 (app qq, J = 8.5, 6.8 Hz, 1H), 2.27 (ddd, J = 10.3, 8.9, 8.9 Hz, 1H), 2.08 (s, 3H), 1.84 (ddd, J = 10.4, 9.5, 9.5 Hz, 1H), 1.23 (d, J = 6.7 Hz, 3H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 208.4, 191.4, 142.6, 129.6, 127.3, 51.6, 42.9, 36.1, 32.0, 28.5, 27.5, 20.9; HRMS (EI) calc'd for $[C_{12}H_{16}N_2O_2Na]^+$ requires *m/z* 243.1109, found *m/z* 243.1104.



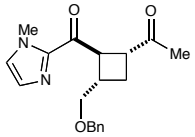
1-(3-Ethyl-2-(1-methyl-1H-imidazole-2-carbonyl)cyclobutyl)ethanone (6) (Figure 1): Prepared according to general procedure **A** using 103.5 mg (0.630 mmol) (*E*)-1-(1-methyl-1*H*-imidazole-2-yl)-pent-2-en-1-one, 260 μL (3.21 mmol) 3-buten-2-one, 11.7 mg (0.016 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 29.5 mg (0.315 mmol) $LiBF_4$, 220 μL (1.26 mmol) *i*-Pr_2NEt, 6.30 mL acetonitrile and an irradiation time of 90 min. Purified by chromatography (17:3 hexanes:acetone) to afford 115 mg (0.491 mmol, 78% yield, >10:1 d.r.) of the cycloadduct as a clear oil. Experiment 2: 103.4 mg (0.630 mmol) enone, 255 μL (3.15 mmol) 3-buten-2-one, 11.8 mg (0.016 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 29.5 mg (0.315 mmol) $LiBF_4$, 220 μL (1.26 mmol) *i*-Pr_2NEt, and 6.30 mL acetonitrile. Isolated 113 mg (0.482 mmol, 77% yield, >10:1 d.r.). IR (thin film): 2960, 2930, 2874, 1709, 1667, 1410 cm^{-1}; ^1H NMR: (500.2 MHz, $CDCl_3$) δ 7.14 (s, 1H), 7.07 (s, 1H), 4.22 (dd, J = 8.7, 8.7 Hz, 1H), 4.03 (s, 3H), 3.51 (ddd, J = 9.2, 9.2, 9.2 Hz, 1H), 2.31 (m, 1H), 2.23 (ddd, J = 10.2, 9.2, 9.2 Hz, 1H), 2.08 (s, 3H), 1.83 (ddd, J = 10.4, 9.4, 9.4 Hz, 1H), 1.69 (d of quint, J = 12.7, 7.3 Hz, 1H), 1.49 (ddq, J = 12.8, 7.4, 7.3 Hz, 1H), 0.79 (t, J = 7.4 Hz, 3H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 208.5, 191.7, 142.6, 129.5, 127.4, 49.9, 43.5, 37.9, 36.1, 28.5, 27.5, 26.3, 10.8; HRMS (EI) calc'd for $[C_{13}H_{18}N_2O_2Na]^+$ requires *m/z* 257.1266, found *m/z* 257.1261.



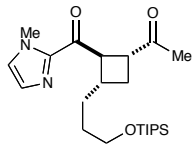
1-(3-*iso*-Propyl-2-(1-methyl-1H-imidazole-2-carbonyl)cyclobutyl)ethanone (7) (Figure 1): Prepared according to general procedure **A** using 108.6 mg (0.609 mmol) (*E*)-4-methyl-1-(1-methyl-1*H*-imidazol-2-yl)-pent-2-en-1-one, 250 μL (3.08 mmol) 3-buten-2-one, 11.5 mg (0.015 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 114 mg (1.22 mmol) $LiBF_4$, 212 μL (1.22 mmol) *i*-Pr_2NEt, 6.09 mL acetonitrile and an irradiation time of 2 h. Purified by chromatography (23:2 hexanes:acetone) to afford 111 mg (0.447 mmol, 73% yield, >10:1 d.r.) of the cycloadduct as a clear oil. Experiment 2: 106.7 mg (0.599 mmol) enone, 245 μL (3.02 mmol) 3-buten-2-one, 11.3 mg (0.015 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 112 mg (1.20 mmol) $LiBF_4$, 210 μL (1.20 mmol) *i*-Pr_2NEt, and 5.99 mL acetonitrile. Isolated 110 mg (0.443 mmol, 74% yield, >10:1 d.r.). IR (thin film): 2957, 2873, 1709, 1665, 1418 cm^{-1}; ^1H NMR: (500.2 MHz, $CDCl_3$) δ 7.15 (s, 1H), 7.06 (s, 1H), 4.36 (dd, J = 8.7, 8.7 Hz, 1H), 4.02 (s, 3H), 3.32 (ddd, J = 8.9, 8.9, 8.9 Hz, 1H), 2.24 (m, 1H), 2.17 (ddd, J = 10.3, 8.8, 8.8 Hz, 1H), 2.07 (s, 3H), 1.86 (ddd, J = 10.3, 9.4, 9.4 Hz, 1H), 1.64 (m, 1H), 0.83 (d, J = 6.5 Hz, 3H), 0.73 (d, J = 6.5 Hz, 3H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 208.3, 192.4, 142.7, 129.5, 127.5, 48.4, 44.5, 42.6, 36.2, 33.4, 27.7, 24.9, 19.6, 18.8; HRMS (EI) calc'd for $[C_{14}H_{20}N_2O_2Na]^+$ requires *m/z* 271.1422, found *m/z* 271.1417.



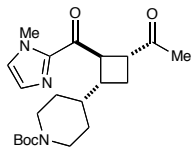
1-(3-(*tert*-Butyl)-2-(1-methyl-1H-imidazole-2-carbonyl)cyclobutyl)ethanone (8) (Figure 1): To a 25 mL Schlenk tube that had been evacuated and purged with N_2 was added 106.4 mg (0.553 mmol, 1.0 equiv) (*E*)-4,4-dimethyl-1-(1-methyl-1*H*-imidazol-2-yl)-pent-2-en-1-one, 112 μL (1.38 mmol, 2.5 equiv) 3-buten-2-one, 10.5 mg (0.014 mmol, 0.025 equiv) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 207 mg (2.21 mmol, 4.0 equiv) $LiBF_4$, 193 μL (1.11 mmol, 2.0 equiv) *i*-Pr_2NEt, and 5.53 mL acetonitrile. The solution was subsequently degassed using three freeze-pump-thaw cycles under nitrogen in the dark. The Schlenk tube was then placed in a water bath and irradiated for 14 h. Upon completion of the reaction, the reaction was diluted with ethyl acetate, passed through a silica plug, and the solvent was removed *in vacuo*. Purified by chromatography (4:1 hexanes:EtOAc) to afford 97 mg (0.370 mmol, 66% yield, >10:1 d.r.) of the cycloadduct as a clear oil. Experiment 2: 106.3 mg (0.553 mmol) enone, 112 μL (1.38 mmol) 3-buten-2-one, 10.3 mg (0.014 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 206 mg (2.20 mmol) $LiBF_4$, 195 μL (1.12 mmol) *i*-Pr_2NEt, and 5.53 mL acetonitrile. Isolated 93 mg (0.354 mmol, 65% yield, >10:1 d.r.). IR (thin film): 2957, 1709, 1666, 1409 cm^{-1}; ^1H NMR: (500.2 MHz, $CDCl_3$) δ 7.16 (d, J = 0.7 Hz, 1H), 7.06 (s, 1H), 4.53 (dd, J = 9.2 Hz, 1H), 4.02 (s, 3H), 3.25 (ddd, J = 9.2, 9.2, 9.2 Hz, 1H), 2.43 (ddd, J = 10.1, 9.0, 9.0 Hz, 1H), 2.06 (s, 3H), 1.99 (m, 2H), 0.80 (s, 9H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 208.1, 192.7, 142.8, 129.6, 127.6, 46.4, 45.2, 44.2, 36.1, 31.6, 27.6, 26.4, 21.8; HRMS (EI) calc'd for $[C_{15}H_{22}N_2O_2Na]^+$ requires *m/z* 285.1579, found *m/z* 285.1574.



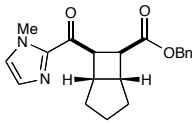
1-(3-((Benzyloxy)methyl)-2-(1-methyl-1H-imidazole-2-carbonyl)cyclobutyl)ethanone (9) (Figure 1)**:** Prepared according to general procedure **A** using 108.7 mg (0.424 mmol) (*E*)-4-benzyloxy-1-(1-methyl-1*H*-imidazol-2-yl)-but-2-en-1-one, 175 μL (2.16 mmol) 3-buten-2-one, 7.8 mg (0.010 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 79.9 mg (0.852 mmol) $LiBF_4$, 148 μL (0.848 mmol) *i*-Pr_2NEt, 4.24 mL acetonitrile and an irradiation time of 145 min. Purified by chromatography (7:3 hexanes:EtOAc) to afford 72 mg (0.221 mmol, 52% yield, >10:1 d.r.) of the cycloadduct as a clear oil. Experiment 2: 108.0 mg (0.723 mmol) enone, 170 μL (2.10 mmol) 3-buten-2-one, 7.9 mg (0.011 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 79.1 mg (0.844 mmol) $LiBF_4$, 147 μL (0.844 mmol) *i*-Pr_2NEt, and 4.21 mL acetonitrile. Isolated 70 mg (0.214 mmol, 51% yield, >10:1 d.r.). IR (thin film): 2927, 1708, 1666, 1410 cm^{-1}; ^1H NMR: (500.2 MHz, $CDCl_3$) δ 7.27 (m, 5H), 7.12 (d, J = 0.7 Hz, 1H), 7.03 (s, 1H), 4.51 (s, 2H), 4.36 (dd, J = 8.7 Hz, 1H), 4.00 (s, 3H), 3.67 (dd, J = 9.7, 5.4 Hz, 1H), 3.59 (dd, J = 9.6, 6.8 Hz, 1H), 3.55 (ddd, J = 9.2, 9.2, 9.2 Hz, 1H), 2.70 (m, 1H), 2.25 (ddd, J = 10.3, 9.0, 9.0 Hz, 1H), 2.11 (s, 3H), 2.10 (ddd, J =10.3, 9.4, 9.4 Hz, 1H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 208.2, 191.1, 142.4, 138.5, 129.5, 128.2, 127.5, 127.4, 127.3, 73.1, 72.8, 46.7, 43.6, 36.0, 35.5, 27.5, 24.3; HRMS (EI) calc'd for $[C_{19}H_{22}N_2O_3Na]^+$ requires *m/z* 349.1528, found *m/z* 349.1523.



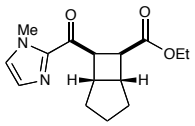
1-(2-(1-Methyl-1H-imidazole-2-carbonyl)-3-(3-((triisopropylsilyl)oxy)propyl)cyclobutyl) ethanone (10) (Figure 1)**:** Prepared according to general procedure **A** using 112.3 mg (0.320 mmol) (*E*)-1-(1-methyl-1*H*-imidazol-2-yl)-6-triisopropylsilanyloxy-hex-2-en-1-one, 130 μL (1.60 mmol) 3-buten-2-one, 6.0 mg (0.008 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 60.4 mg (0.644 mmol) $LiBF_4$, 112 μL (0.641 mmol) *i*-Pr_2NEt, 3.20 mL acetonitrile and an irradiation time of 90 min. Purified by chromatography (4:1 hexanes:EtOAc) to afford 92 mg (0.219 mmol, 68% yield, >10:1 d.r.) of the cycloadduct as a clear oil. Experiment 2: 113.2 mg (0.323 mmol) enone, 131 μL (1.61 mmol) 3-buten-2-one, 6.1 mg (0.008 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 60.7 mg (0.647 mmol) $LiBF_4$, 113 μL (0.649 mmol) *i*-Pr_2NEt, and 3.23 mL acetonitrile. Isolated 92 mg (0.219 mmol, 68% yield, >10:1 d.r.). IR (thin film): 2942, 2866, 2360, 1711, 1667, 1409 cm^{-1}; ^1H NMR: (500.2 MHz, $CDCl_3$) δ 7.13 (s, 1H), 7.04 (s, 1H), 4.22 (dd, J = 8.8, 8.8 Hz, 1H), 4.02 (s, 3H), 3.60 (t, J = 6.7 Hz, 2H), 3.51 (ddd, J = 9.2, 9.2, 9.2 Hz, 1H), 2.39 (m, 1H), 2.24 (ddd, J = 10.3, 9.4, 9.4 Hz, 1H), 2.08 (s, 3H), 1.84 (ddd, J = 10.1, 9.6, 9.6 Hz, 1H), 1.75 (m, 1H), 1.46 (m, 3H), 1.01 (m, 21H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 208.3, 191.6, 142.6, 129.6, 127.4, 63.2, 50.2, 43.5, 36.2, 36.1, 31.9, 30.1, 27.5, 26.8, 18.0, 11.9; HRMS (EI) calc'd for $[C_{23}H_{40}N_2O_3SiNa]^+$ requires *m/z* 443.2706, found *m/z* 443.2701.



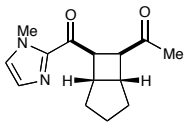
***tert*-Butyl 4-(3-acetyl-2-(1-methyl-1H-imidazole-2-carbonyl)cyclobutyl)piperidine-1-carboxylate (11)** (Figure 1)**:** Due to the hygroscopic nature of the piperdine enone, benzene (3 x 1 mL) was added to the enone, and the volatiles were removed *in vacuo* three times immediately prior to the reaction. Prepared according to general procedure **B** using 109.5 mg (0.343 mmol) 4-[(*E*)-3-(1-methyl-1*H*-imidazol-2-yl)-3-oxo-propenyl]-piperidine-1-carboxylic acid *tert*-butyl ester, 140 μL (1.73 mmol) 3-buten-2-one, 6.4 mg (0.009 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 64.2 mg (0.686 mmol) $LiBF_4$, 119 μL (0.686 mmol) *i*-Pr_2NEt, 3.43 mL acetonitrile and an irradiation time of 2 h. Purified by chromatography (2:3 hexanes:EtOAc) to afford 100 mg (0.257 mmol, 75% yield, >10:1 d.r.) of the cycloadduct as a clear oil. Experiment 2: 111.4 mg (0.349 mmol) enone, 143 μL (1.76 mmol) 3-buten-2-one, 6.5 mg (0.009 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 65.3 mg (0.697 mmol) $LiBF_4$, 121 μL (0.698 mmol) *i*-Pr_2NEt, and 3.49 mL acetonitrile. Isolated 105 mg (0.270 mmol, 77% yield, >10:1 d.r.). IR (thin film): 2976, 2930, 2853, 1688, 1667, 1410, 1366 cm^{-1}; ^1H NMR: (500.2 MHz, $CDCl_3$) δ 7.14 (s, 1H), 7.07 (s, 1H), 4.37 (dd, J = 8.7, 8.7 Hz, 1H), 4.02 (s, 3H), 4.02 (m, 2H), 3.37 (ddd, J = 9.4, 9.4, 9.4 Hz, 1H), 2.62 (m, 2H), 2.31 (pent, J = 8.9 Hz, 1H), 2.19 (ddd, J = 10.1, 9.5, 9.5 Hz, 1H), 2.07 (s, 3H), 1.90 (ddd, J = 10.2, 9.6, 9.6 Hz, 1H), 1.66 (m, 1H), 1.50 (m, 2H), 1.42 (s, 9H), 0.99 (qd, J = 12.5, 4.3 Hz, 1H), 0.88 (qd, J = 12.1, 4.2 Hz, 1H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 208.0, 191.9, 154.8, 142.4, 129.6, 127.7, 79.2, 47.9, 44.8, 41.3, 39.9, 36.2, 29.1, 28.5, 27.9, 24.7; HRMS (EI) calc'd for $[C_{21}H_{31}N_3O_4Na]^+$ requires *m/z* 412.2212, found *m/z* 412.2207.



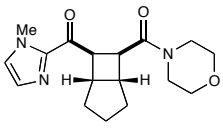
Benzyl 7-(1-methyl-1H-imidazole-2-carbonyl)bicyclo[3.2.0]heptane-6-carboxylate (12) (Figure 2): Prepared according to general procedure **B** using 203.3 mg (0.601 mmol) (2*E*,7*E*)-benzyl 9-(1-methyl-1H-imidazol-2-yl)-9-oxonona-2,7-dienoate, 11.3 mg (0.015 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 27.8 mg (0.299 mmol) $LiBF_4$, 220 μL (1.194 mmol) *i*-Pr_2NEt, 6.0 mL acetonitrile, and an irradiation time of 90 min. Purified by chromatography (3:1 hexanes:acetone) to afford 166.7 mg (0.493 mmol, 82% yield, >10:1 d.r.) of the cycloadduct as a clear oil. Experiment 2: 202.6 mg (0.600 mmol) bisenone, 11.5 mg (0.015 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 27.7 mg (0.300 mmol) $LiBF_4$, 220 μL (1.19 mmol) *i*-Pr_2NEt, and 6.0 mL acetonitrile. Isolated 160.8 mg (0.476 mmol, 79% yield, >10:1 d.r.). IR (thin film) 2930, 2361, 1667, 1623 cm^{-1}; ^1H NMR: (499.9 MHz, $CDCl_3$) δ 7.26 (m, 3H), 7.16 (m, 2H), 7.03 (d, J = 0.9 Hz, 1H), 6.90 (s, 1H), 4.93 (s, 2H), 4.01 (dd, J = 10.5, 5.7 Hz, 1H), 3.83 (s, 3H), 3.21 (m, 1H), 3.16 (dd, J = 10.5, 5.7 Hz, 1H), 3.02 (m, 1H), 1.90 (m, 2H), 1.73 (m, 2H), 1.58 (m, 2H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 191.5, 173.3, 142.6, 135.8, 128.6, 128.2, 127.8, 127.8, 126.5, 77.3, 77.0, 76.7, 65.9, 46.1, 44.8, 39.5, 37.9, 35.7, 32.3, 32.1, 25.0; HRMS (EI) calc'd for $[C_{20}H_{21}N_2O_3+H]^+$ requires *m/z* 338.1625, found *m/z* 338.1614.



Ethyl 7-(1-methyl-1H-imidazole-2-carbonyl)bicyclo[3.2.0]heptane-6-carboxylate (13) (Figure 2): Prepared according to general procedure **B** using 99.6 mg (0.361 mmol), (2*E*,7*E*)-ethyl 9-(1-methyl-1H-imidazol-2-yl)-9-oxonona-2,7-dienoate, 6.7 mg (0.009 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 16.8 mg (0.181 mmol) $LiBF_4$, 135 μL (0.733 mmol) *i*-Pr_2NEt, 3.6 mL acetonitrile, and an irradiation time of 135 min. Purified by chromatography (2:1 hexanes:acetone) to afford 84 mg (0.493 mmol, 85% yield, >10:1 d.r.) of the cycloadduct as a clear oil. Experiment 2: 101.1 mg (0.366 mmol) bisenone, 6.6 mg (0.009 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 16.7 mg (0.179 mmol) $LiBF_4$, 135 μL (0.733 mmol) *i*-Pr_2NEt, and 3.6 mL acetonitrile. Isolated 82 mg (0.476 mmol, 82% yield, >10:1 d.r.). IR (thin film) 2952, 2855, 1730, 1672, 1409 cm^{-1}; ^1H NMR: (499.9 MHz, $CDCl_3$) δ 7.08 (d, J = 0.9 Hz, 1H), 6.99 (s, 1H), 4.00 (s, 3H), 3.97 (ddd, J = 10.2, 5.2, 1.0 Hz, 1H), 3.93 (dq, J = 7.3, 0.9 Hz, 2H), 3.23 (m, 1H), 3.11 (ddd, J = 10.5, 5.6, 0.9 Hz, 1H), 2.97 (m, 1H), 1.90 (m, 2H), 1.73 (m, 2H), 1.60 (m, 2H), 1.03 (dd, J = 7.5, 7.5 Hz, 3H); ^{13}C NMR: (125.7 MHz, CDCl3) δ 191.7, 173.5, 142.8, 128.7, 126.4, 60.1, 46.2, 45.1, 39.5, 37.6, 35.9, 32.4, 32.1, 25.0, 13.9; HRMS (EI) calc'd for $[C_{15}H_{19}N_2O_3+H]^+$ requires *m/z* 276.1469, found *m/z* 276.1461.

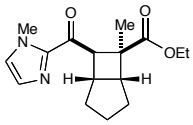


1-7-(1-Methyl-1*H*-imidazole-2-carbonyl)bicyclo[3.2.0]heptan-6-yl)ethanone (14) (Figure 2): Prepared according to general procedure **B** using 101.6 mg (0.413 mmol) (2*E*,7*E*)-1-(1-methyl-1H-imidazol-2-yl)deca-2,7-diene-1,9-dione, 17.5 mg (0.010 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 19.2 mg (0.206 mmol) $LiBF_4$, 37 μL (0.201 mmol) *i*-Pr_2NEt, 4.0 mL acetonitrile, and an irradiation time of 16.5 h. Purified by chromatography (3:1 hexanes:acetone) to afford 66.7 mg (0.271 mmol, 66% yield, >10:1 d.r.) of the cycloadduct as a clear oil. Experiment 2: 100.5 mg (0.408 mmol) bisenone, 7.4 mg (0.010 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 18.7 mg (0.201 mmol) $LiBF_4$, 37 μL (0.201 mmol) *i*-Pr_2NEt, and 4.0 mL acetonitrile. Isolated 64.5 mg (0.262 mmol, 65% yield, >10:1 d.r.). IR (thin film) 2949, 2862, 704, 1671, 1409 cm^{-1}; ^1H NMR: (499.9 MHz, $CDCl_3$) δ 7.02 (d, J = 0.9 Hz, 1H), 6.97 (s, 1H), 4.02 (s, 3H), 3.84 (ddd, J = 10.2, 5.5, 1.0 Hz, 1H), 3.33 (dd, J = 10.2, 5.5 Hz, 1H), 3.22 (m, 1H), 2.84 (m, 1H), 1.96 (s, 3H), 1.93 (m, 2H), 1.73 (m, 2H), 1.60 (m, 2H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 207.9, 192.2, 143.2, 128.4, 126.2, 53.8, 46.3, 39.9, 36.6, 35.8, 32.8, 32.0, 26.3, 25.0; HRMS (EI) calc'd for $[C_{14}H_{18}N_2O_2Na]^+$requires *m/z* 269.1261, found *m/z* 269.1266.



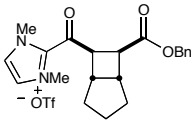
(1-Methyl-1H-imidazol-2-yl)-7-(morpholine-4-carbonyl)bicyclo[3.2.0]heptan-6-yl)methanone (15) (Figure 2): Prepared according to general procedure **B** using 98.5 mg (0.311 mmol), (2*E*,7*E*)-1-(1-methyl-1H-imidazol-2-yl)-9-morpholinonona-2,7-diene-1,9-dione, 5.8 mg (0.008 mmol) $Ru(bpy)_3Cl_2{\cdot}6H_2O$, 14.7 mg (0.158 mmol) $LiBF_4$, 110 μL (0.597 mmol) *i*-Pr_2NEt, 3.1 mL acetonitrile, and an irradiation time of 3 h. Purified by chromatography (1:3 hexanes:acetone) to afford 70.0 mg (0.220 mmol, 70% yield, >10:1 d.r.) of the cycloadduct as a light yellow oil.

Experiment 2: 100.6 mg (0.317 mmol) bisenone, 6.1 mg (0.008 mmol) $Ru(bpy)_3Cl_2 \cdot 6H_2O$, 14.5 mg (0.156 mmol) $LiBF_4$, 110 μL (0.597 mmol) *i*-Pr_2NEt, and 3.1 mL acetonitrile. Isolated 68.4 mg (0.216 mmol, 68% yield, >10:1 d.r.). IR (thin film) 3055, 2987, 1677, 1636, 1422 cm^{-1}; ^1H NMR: (499.9 MHz, $CDCl_3$) δ 7.02 (d, J = 0.9 Hz, 1H), 6.97 (s, 1H), 4.01 (s, 3H), 3.83 (ddd, J = 10.1, 5.9, 1.2 Hz, 1H), 3.53 (m, 2H), 3.25-3.47 (m, 8H), 2.91 (m, 1H), 1.92 (m, 2H), 1.53-1.76 (m, 4H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 191.4, 171.6, 143.2, 128.1, 126.0, 66.7, 66.3, 46.0, 45.8, 44.3, 41.9, 39.6, 37.1, 35.8, 32.3, 32.2, 25.2; HRMS (EI) calc'd for $[C_{17}H_{23}N_3O_3+H]^+$ requires *m/z* 318.1813, found *m/z* 318.1810.

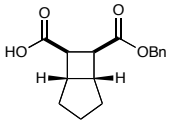


Ethyl 6-methyl-7-(1-methyl-1H-imidazole-2-carbonyl)bicyclo[3.2.0]heptane-6-carboxylate (16) (Figure 2): Prepared according to general procedure **B** using 88.7 mg (0.305 mmol) (2*E*,7*E*)-ethyl 2-methyl-9-(1-methyl-1*H*-imidazol-2-yl)-9-oxonona-2,7-dienoate, 6.0 mg (0.008 mmol) $Ru(bpy)_3Cl_2 \cdot 6H_2O$, 14.3 mg (0.154 mmol) $LiBF_4$, 115 μL (0.624 mmol) *i*-Pr_2NEt, 3.1 mL acetonitrile, and an irradiation time of 24 h. Purified by chromatography (5:1 hexanes:acetone) to afford 59.0 mg (0.203 mmol, 66% yield, >10:1 d.r.) of the cycloadduct as a light yellow oil. Experiment 2: 84.9 mg (0.294 mmol) bisenone, 5.7 mg (0.008 mmol) $Ru(bpy)_3Cl_2 \cdot 6H_2O$, 14.5 mg (0.156 mmol) $LiBF_4$, 115 μL (0.624 mmol) *i*-Pr_2NEt, and 3.1 mL acetonitrile. Isolated 56.7 mg (0.195 mmol, 66% yield, >10:1 d.r.). IR (thin film) 2956, 2254,1719, 1669, 1410 cm^{-1}; ^1H NMR: (499.9 MHz, $CDCl_3$) δ 7.06 (s, 1H), 6.97 (s, 1H), 3.98 (s, 3H), 3.96 (m, 2H), 3.41 (m, 1H), 3.38 (m, 1H), 2.86 (dd, J = 7.7, 7.7 Hz, 1H), 1.89 (m, 2H), 1.79 (m, 1H), 1.64 (dd, J = 13.4, 6.7 Hz, 1H), 1.52 (m, 2H), 1.48 (s, 3H), 1.05 (t, J = 7.2 Hz, 3H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 191.8, 176.2, 143.4, 128.6, 126.1, 60.3, 54.2, 48.9, 42.2, 36.4, 35.7, 31.4, 27.0, 26.3, 18.7, 13.9; HRMS (EI) calc'd for $[C_{16}H_{22}N_2O_3+Na]^+$ requires *m/z* 313.1523, found *m/z* 313.1520.

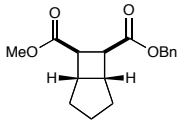
IV. Cleavage of the auxiliary group



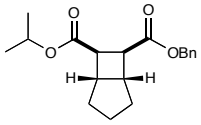
2-(7-((Benzyloxy)carbonyl)bicyclo[3.2.0]heptane-6-carbonyl)-1,3-dimethyl-1H-imidazol-3-ium triflate: In an oven-dried round-bottomed flask was placed a solution of cyclobutane **12** (405 mg, 1.20 mmol) in dry dichloromethane (12 mL, 0.1 M). Neat methyl trifluoromethanesulfonate (135 μL, 1.193 mmol) was added dropwise, and the solution was stirred for 16 h. Upon completion, the reaction was quenched with acetone and the volatiles were removed *in vacuo*. The triflate salt was recrystallized from acetone/diethyl ether to afford 282.5 mg (0.800 mmol, 67%) of a colorless solid (m.p.: 135–140 ºC). IR (thin film) 3629, 3093, 2262, 1634 cm^{-1}; ^1H NMR: (500.2 MHz, CD_3CN) δ 7.30 (m, 5H), 7.19 (m, 2H), 5.00, 4.92 (ABq, 2H, J_{AB} = 12 Hz), 3.87 (s, 6H), 3.54 (ddd, J = 10.0, 4.4, 0.9 Hz, 1H), 3.35 (ddd, J = 10.0, 5.6, 1.0 Hz, 1H), 3.13 (m, 1H), 3.05 (m, 1H), 1.88 (m, 2H), 1.71 (m, 2H), 1.59 (m, 2H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 191.4, 171.6, 143.2, 128.1, 126.0, 66.7, 66.3, 46.0, 45.8, 44.3, 41.9, 39.6, 37.1, 35.8, 32.3, 32.2, 25.2; HRMS (EI) calc'd for $[C_{21}H_{25}N_2O_6]^+$ requires *m/z* 353.1860, found *m/z* 353.1851.



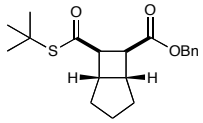
7-((Benzyloxy)carbonyl)bicyclo[3.2.0]heptane-6-carboxylic acid (Table 3, entry 1): In an oven-dried 1.5 dram vial was placed 49.6 mg (0.099 mmol) of the imidazolium triflate salt in 1.5 mL dry diethyl ether. The vial was sealed with a septum and flushed with a nitrogen atmosphere. 50 μL (0.33 mmol, 3.5 equiv) of DBU was added, and the reaction was stirred for several minutes before addition of 0.2 mL (11.1 mmol, 111 equiv) H_2O. After 2 h, the solution was acidified with 1 mL acetic acid, concentrated *in vacuo* and purified by chromatography on a silica column (3:1 Hex:EtOAc + 1% AcOH) to afford 14 mg (0.051 mmol, 52% yield, >10:1 d.r.) of a clear oil. IR(thin film) 3629, 3093, 2262, 1634 cm^{-1}; ^1H NMR: (499.9 MHz, $CDCl_3$) δ 7.33 (m, 5H), 5.08, 5.11 (ABq, 2H, J_{AB} = 12.6 Hz), 3.07 (m, 2H), 2.90 (m, 2H), 1.89 (m, 1H), 1.52-1.79 (m, 5H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 173.0, 135.8, 128.5, 128.3, 128.2, 66.6, 43.2, 39.5, 39.2, 32.1, 24.9; HRMS (EI) calc'd for $[C_{16}H_{18}O_4\text{-}H^+]^-$ requires *m/z* 273.1132, found *m/z* 273.1130.



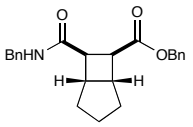
6-Benzyl 7-methyl bicyclo[3.2.0]heptane-6,7-dicarboxylate (Table 3, entry 2): In an oven-dried 1.5 dram vial was placed 51.4 mg (0.102 mmol) of the imidazolium triflate salt in 1.5 mL dry diethyl ether. The vial was sealed with a septum and flushed with a nitrogen atmosphere. 50 μL (0.33 mmol, 3.5 equiv) of DBU was added, and the reaction was stirred for several minutes before addition of 0.5 mL (12.3 mmol, 111 equiv) methanol. After 2 h, the solution was concentrated *in vacuo* and purified by chromatography on a silica column (10:1 Hex:EtOAc) to afford 25.4 mg (0.088 mmol, 86% yield, >10:1 d.r.) of a clear oil. IR (thin film) 2953, 1731,1647, 1309 cm^{-1}; ^1H NMR: (499.9 MHz, $CDCl_3$) δ 7.34 (m, 5H), 5.13 (s, 2H), 3.69 (s, 3H), 3.59 (dd, J = 10.4, 8.6 Hz, 1H), 3.09 (dd, J = 6.5, 0.8 Hz, 1H), 2.98 (m, 1H), 2.88 (m, 1H), 1.76 (m, 3H), 1.62-1.42 (m, 3H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 174.1, 172.4, 136.0, 128.5, 128.1, 128.0, 66.3, 51.5, 41.6, 40.8, 39.7, 38.2, 32.4, 28.5, 25.3; HRMS (EI) calc'd for $[C_{17}H_{20}O_4Na]^+$ requires *m/z* 311.1254, found *m/z* 311.1259.



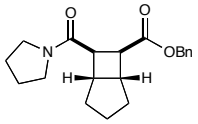
6-Benzyl 7-*iso*-propyl bicyclo[3.2.0]heptane-6,7-dicarboxylate (Table 3, entry 3): In a 1.5 dram vial was placed 49.2 mg (0.098 mmol) of the imidazolium triflate salt in 1.5 mL dichloromethane. The vial was sealed with a septum and flushed with a nitrogen atmosphere. 50 μL (0.33 mmol, 3.5 equiv) of DBU were added, and the reaction was stirred for several minutes before addition of 0.2 mL (2.59 mmol, 26 equiv) isopropanol. After 2 h, the solution was concentrated *in vacuo* and purified by column chromatography (9:1 Hex:EtOAc) to afford 27.6 mg (0.087 mmol, 88% yield) of a clear oil. IR (thin film) 2957, 1725, 1263, 1194 cm^{-1}; ^1H NMR: (499.9 MHz, $CDCl_3$) δ 7.34 (m, 5H), 5.13 (s, 2H), 5.04 (sept, J = 6.3 Hz, 1H), 3.52 (dd, J = 8.8, 10.1 Hz, 1H), 3.09 (dd, J = 6.5, 8.7 Hz, 1H), 2.98 (m, 1H), 2.87 (m, 1H), 1.82-1.62 (m, 4H), 1.48 (m, 2H), 1.24 (t, J = 5.5 Hz, 6H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 174.2, 171.4, 136.1, 128.5, 128.1, 127.9, 77.3, 77.0, 76.7, 67.9, 66.2, 41.6, 40.7, 39.9, 38.3, 32.4, 28.2, 25.3, 22.0, 21.9; HRMS (EI) calc'd for $[C_{19}H_{24}O_4Na]^+$ requires *m/z* 339.1567, found *m/z* 339.1562.



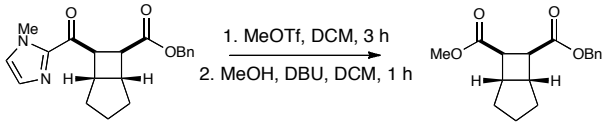
Benzyl 7-((tert-butylthio)carbonyl)bicyclo[3.2.0]heptane-6-carboxylate (Table 3, entry 5)**:** In a 1.5 dram vial was placed 51.2 mg (0.1023 mmol) of the imidazolium triflate salt in 1.5 mL dichloromethane. The vial was sealed with a septum and flushed with a nitrogen atmosphere. 50 μL (0.33 mmol, 3.5 equiv) of DBU were added and let stir for several minutes before addition of 0.150 mL (1.33 mmol, 13 equiv) *tert*-butylthiol. After 3.5 h, the reaction was concentrated *in vacuo* and purified by column chromatography (10:1 Hex:EtOAc) to afford 35.1 mg (0.101 mmol, 88% yield, >10:1 d.r.) of a clear oil. IR (thin film) 3629, 3093, 2262, 1634 cm^{-1}; ^1H NMR: (500.2 MHz, $CDCl_3$) δ 7.33 (m, 5H), 5.12 (m, 2H), 3.66 (dd, J = 10.1, 8.7 Hz, 1H), 3.19 (dd, J = 8.7, 6.3 Hz, 1H), 2.99 (m, 1H), 2.86 (m, 1H), 1.78 (m, 4H), 1.47 (s, 9H), 1.44 (m, 2H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 198.1, 173.9, 136.1, 128.5, 128.1, 127.9, 66.2, 48.2, 47.6, 40.6, 40.6, 40.1, 32.2, 30.0, 27.3, 25.3; HRMS (EI) calc'd for $[C_{21}H_{24}N_2O_6+H]^+$ requires *m/z* 353.1860, found *m/z* 353.1851.



Benzyl 7-(benzylcarbamoyl)bicyclo[3.2.0]heptane-6-carboxylate (Table 3, entry 6)**:** In a 1.5 dram vial was placed 47.0 mg (0.099 mmol) of the imidazolium triflate salt in 1.5 mL dichloromethane. The vial was sealed with a septum and flushed with a nitrogen atmosphere. 200 μL (1.83 mmol, 20 equiv) benzylamine were added. After 2 h, the solution was concentrated *in vacuo* and purified by chromatography on a silica column (5:1 Hex:EtOAc) to afford 33.3 mg of a colorless oil (0.916 mmol, >10:1 d.r.). IR (thin film) 3305, 2954, 1729, 1649 cm^{-1}; ^1H NMR: (500.2 MHz, $CDCl_3$) δ 7.29 (m, 10H), 5.73 (t, J = 5.6 Hz, 1H), 5.11 (ABq, 2H), 4.43 (d, J = 5.6 Hz, 2H), 3.37 (t, J = 9.8 Hz, 1H), 3.17 (dd, J = 8.4, 5.6 Hz, 1H), 2.89 (m, 2H), 1.80 (m, 4H), 1.48 (m, 2H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 174.3, 170.7, 138.3, 135.9, 128.7, 128.5, 128.2, 128.0, 127.8, 127.5, 66.4, 43.5, 41.7, 41.0, 40.5, 38.1, 32.3, 27.9, 25.7; HRMS (EI) calc'd for $[C_{21}H_{24}N_2O_6+H]^+$ requires *m/z* 353.1860, found *m/z* 353.1851.

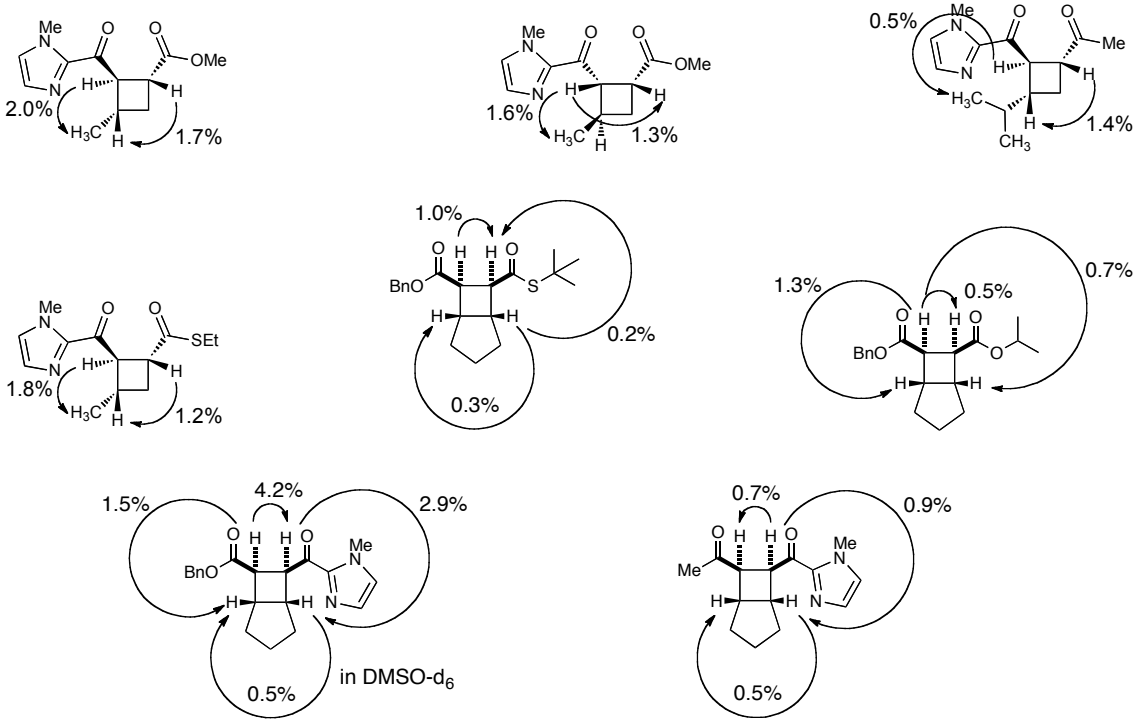


Benzyl 7-(pyrrolidine-1-carbonyl)bicyclo[3.2.0]heptane-6-carboxylate (Table 3, entry 7)**:** In an oven-dried 1.5 dram vial was placed 50.1 mg (0.099 mmol) of the imidazolium triflate salt in 1.5 mL dichloromethane. 100 μL (1.20 mmol, 12.0 equiv) distilled pyrrolidine were added After 12 h, the solution was concentrated *in vacuo* and purified by column chromatography (1:1 Hex/EtOAc with 2% TEA) to afford 24 mg to afford (0.741 mmol, >10:1 d.r.) of a colorless oil. IR (thin film) 3629, 3093, 2262, 1634 cm^{-1}; ^1H NMR: (500.2 MHz, $CDCl_3$) δ 7.35 (m, 5H), 5.12 (s, 2H), 3.56-3.42 (m, 3H), 3.38 (dd, J = 6.8, 6.8 Hz, 1H), 3.36 (dd, J = 6.8, 6.8 Hz, 1H), 3.22 (m, 1H), 2.94 (m, 1H), 2.86 (m, 1H), 1.97-1.73 (m, 7H), 1.45 (m, 3H); ^{13}C NMR: (125.7 MHz, $CDCl_3$) δ 175.0, 169.0, 136.2, 128.5, 128.4, 128.0, 128.0, 77.3, 77.0, 76.8, 66.2, 45.6, 45.5, 40.7, 40.5, 40.3, 37.4, 32.4, 28.2, 26.1, 25.4, 24.3; HRMS (EI) calc'd for $[C_{20}H_{24}NO_3+H]^+$ requires *m/z* 327.1829, found *m/z* 327.1833.



One-pot methylation-esterification procedure: An oven-dried Schlenk flask containing 99.8 mg (0.295 mmol) cyclobutane **12** and 1.5 mL dry dichloromethane (0.2 M) was stirred under N_2 before careful addition of freshly distilled MeOTf (40 μL, 0.341 mmol, 1.1 equiv). The flask was sealed, and the reaction was stirred at room temperature for 90 min. The volatiles were removed *in vacuo*, and the residue was dissolved in 1.5 mL dichloromethane. To this solution were added 100 μL DBU (0.669 mmol, 2.3 equiv) and 500 μL (12.5 mmol, 44 equiv) freshly distilled methanol. The vessel was sealed and the reaction was stirred for 90 min under N_2. The reaction was then concentrated and the crude residue loaded directly onto silica for purification by chromatography (4:1 Hex:EtOAc) to afford 49.5 mg of a colorless oil (0.1717 mmol, 58% yield).

V. Stereochemical determinations by NOE



VI. References

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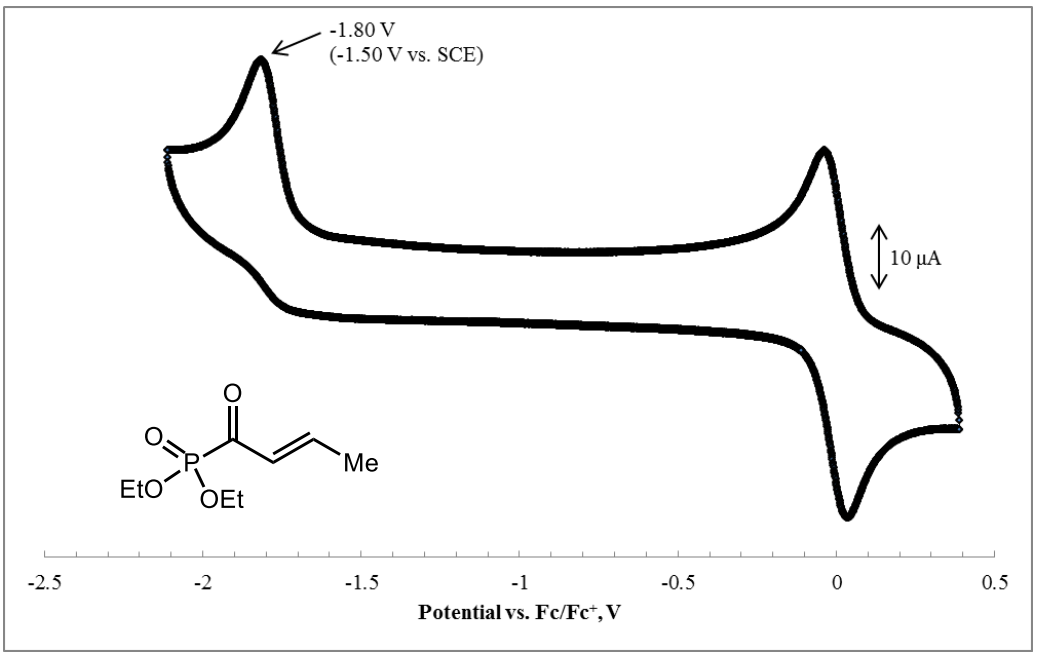
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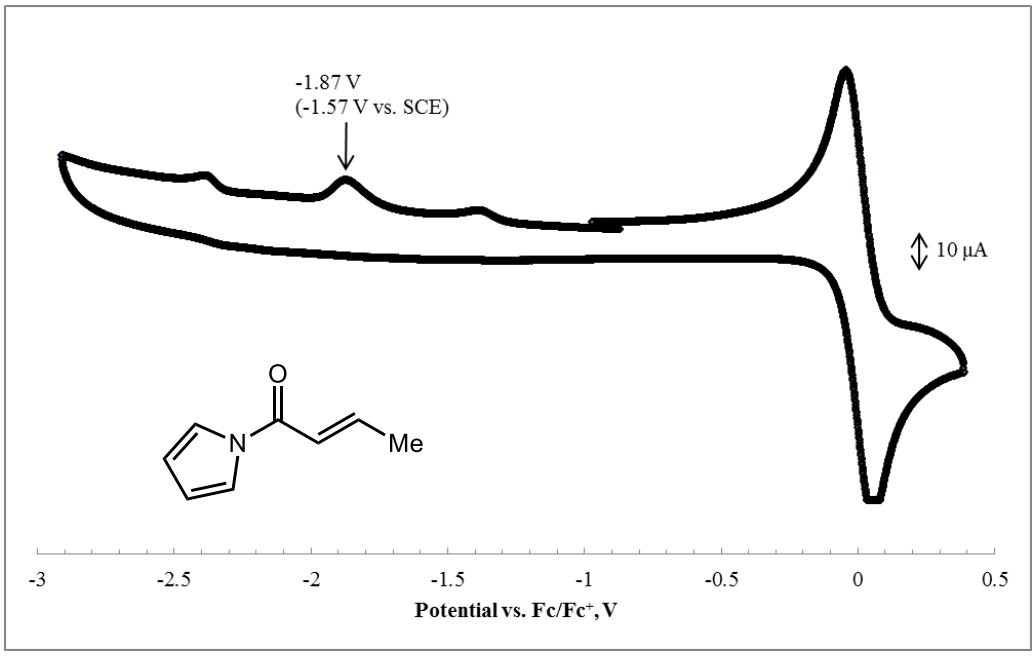
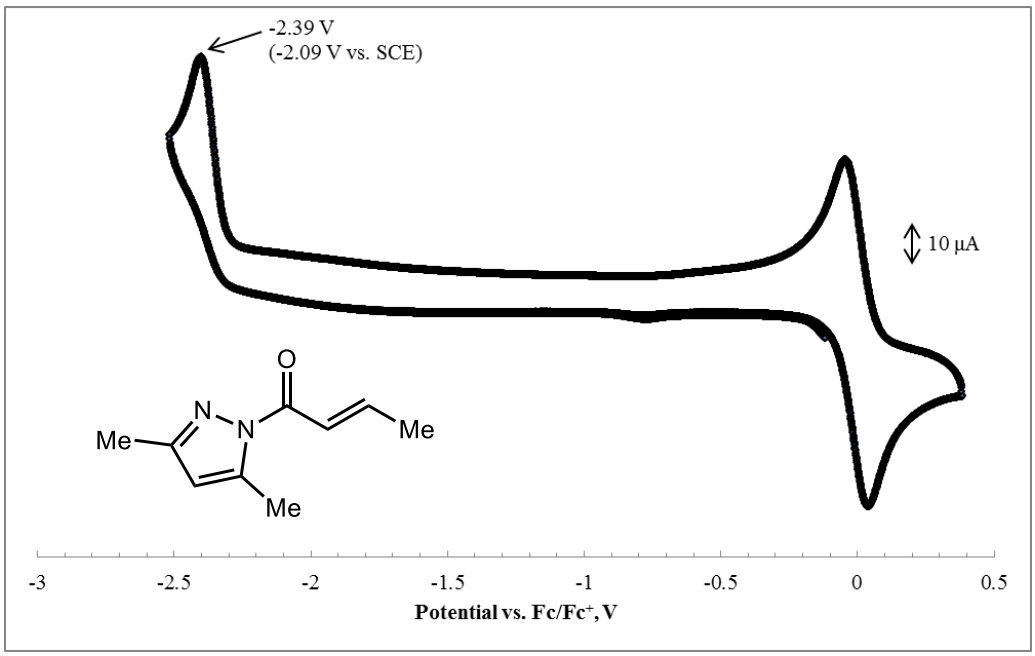
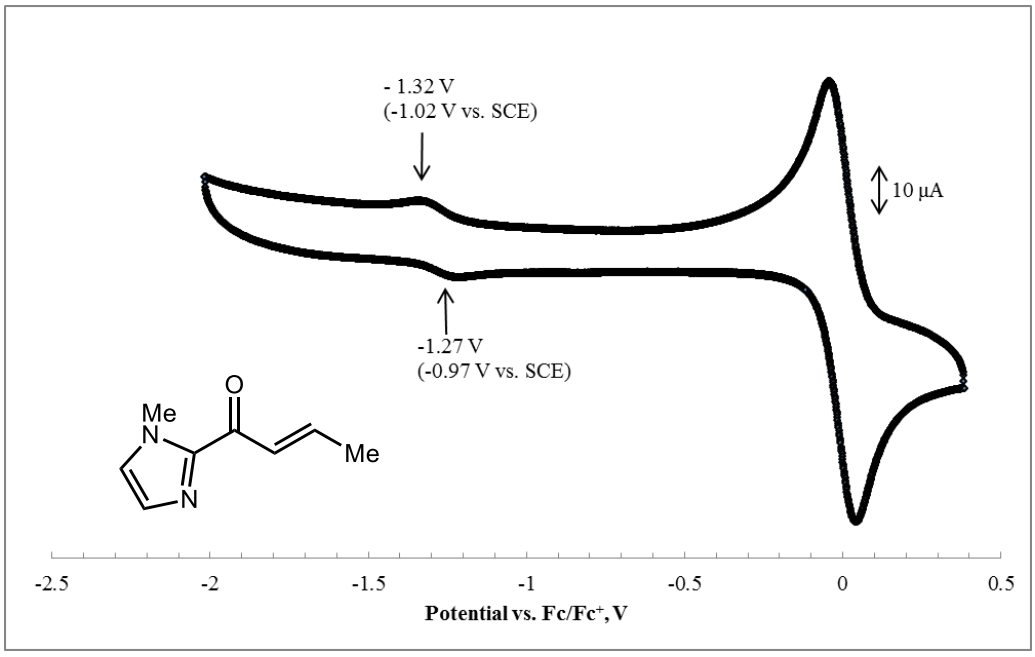
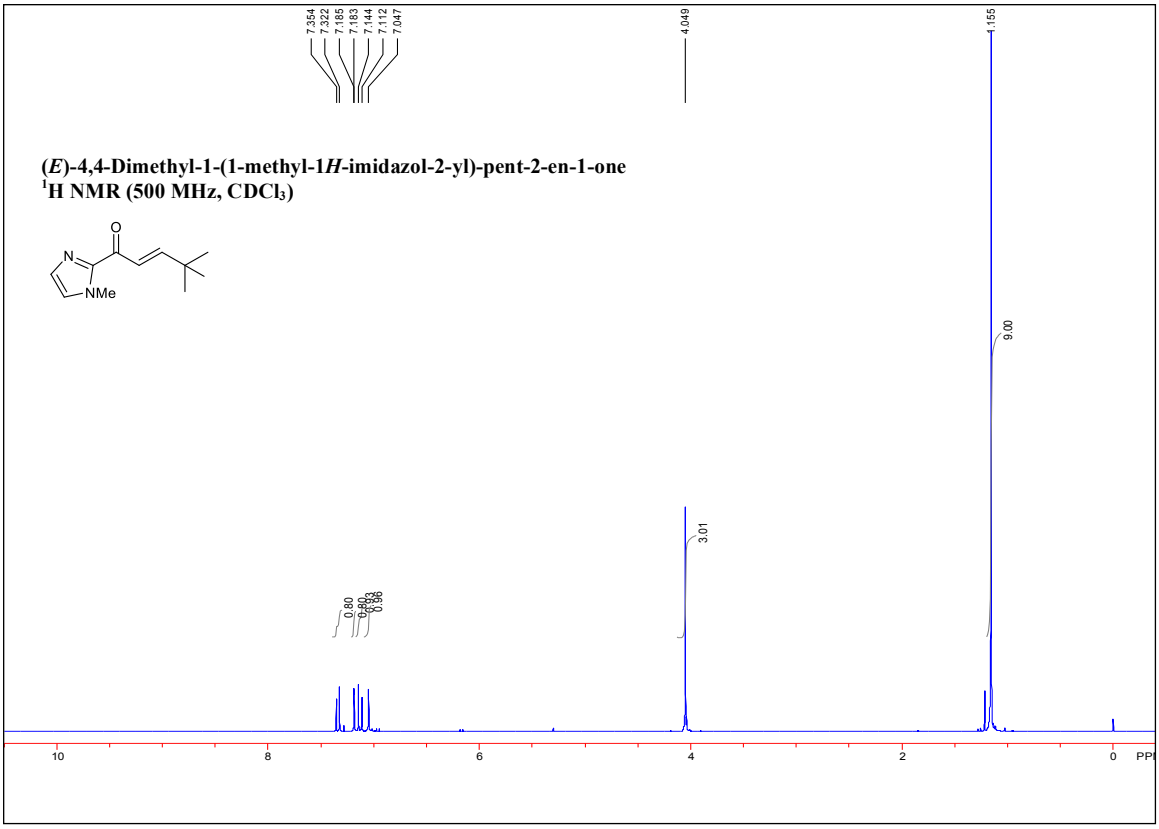
(-1.50 V vs. SCE)
10 µA
O
O
P
EtO
OEt
Me
-2.5
-2
-1.5
-1
-0.5
0
0.5
Potential vs. Fc/Fc+, V

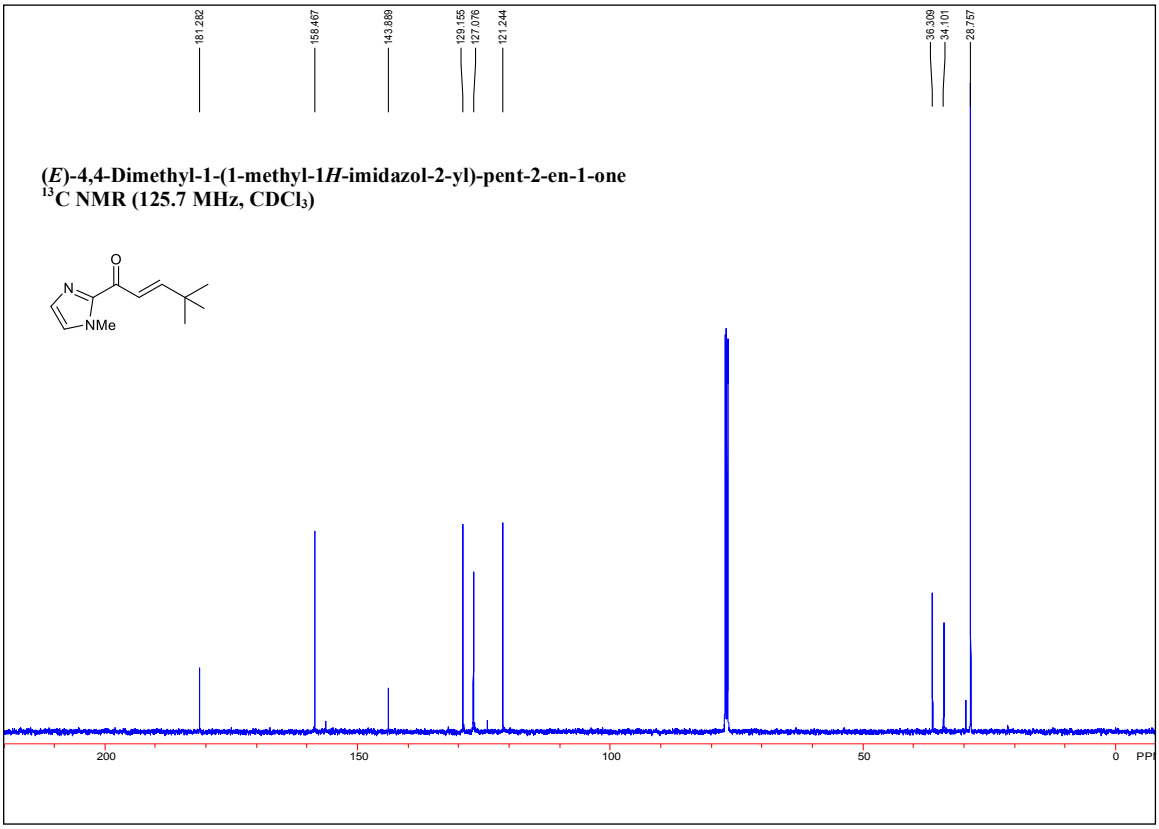

-1.87 V
(-1.57 V vs. SCE)
10 µA
O
N
Me
-3
-2.5
-2
-1.5
-1
-0.5
0
0.5
Potential vs. Fc/Fc+, V


-2.39 V
(-2.09 V vs. SCE)
10 µA
O
N
N
Me
Me
Me
-3
-2.5
-2
-1.5
-1
-0.5
0
0.5
Potential vs. Fc/Fc+, V

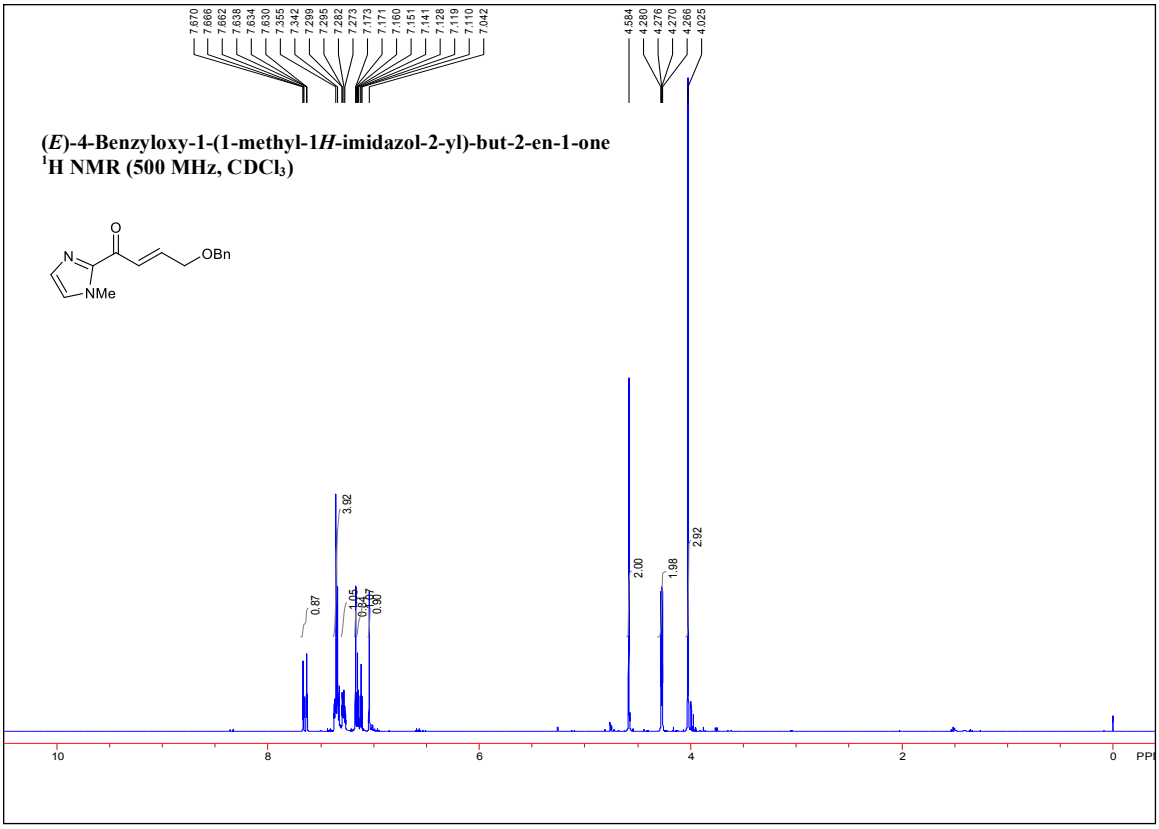

- 1.32 V
(-1.02 V vs. SCE)
10 µA
-1.27 V
(-0.97 V vs. SCE)
O
Me
N
Me
N
-2.5
-2
-1.5
-1
-0.5
0
0.5
Potential vs. Fc/Fc+, V



(*E*)-4,4-Dimethyl-1-(1-methyl-1*H*-imidazol-2-yl)-pent-2-en-1-one
^{1}H NMR (500 MHz, $CDCl_3$)



(*E*)-4,4-Dimethyl-1-(1-methyl-1*H*-imidazol-2-yl)-pent-2-en-1-one
^{13}C NMR (125.7 MHz, $CDCl_3$)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7.630
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7.342
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7.295
7.282
7.273
7.173
7.171
7.160
7.151
7.141
7.128
7.119
7.110
7.042
4.584
4.280
4.276
4.270
4.266
4.025
(E)-4-Benzyloxy-1-(1-methyl-1H-imidazol-2-yl)-but-2-en-1-one
1H NMR (500 MHz, CDCl3)
O
N
NMe
OBn
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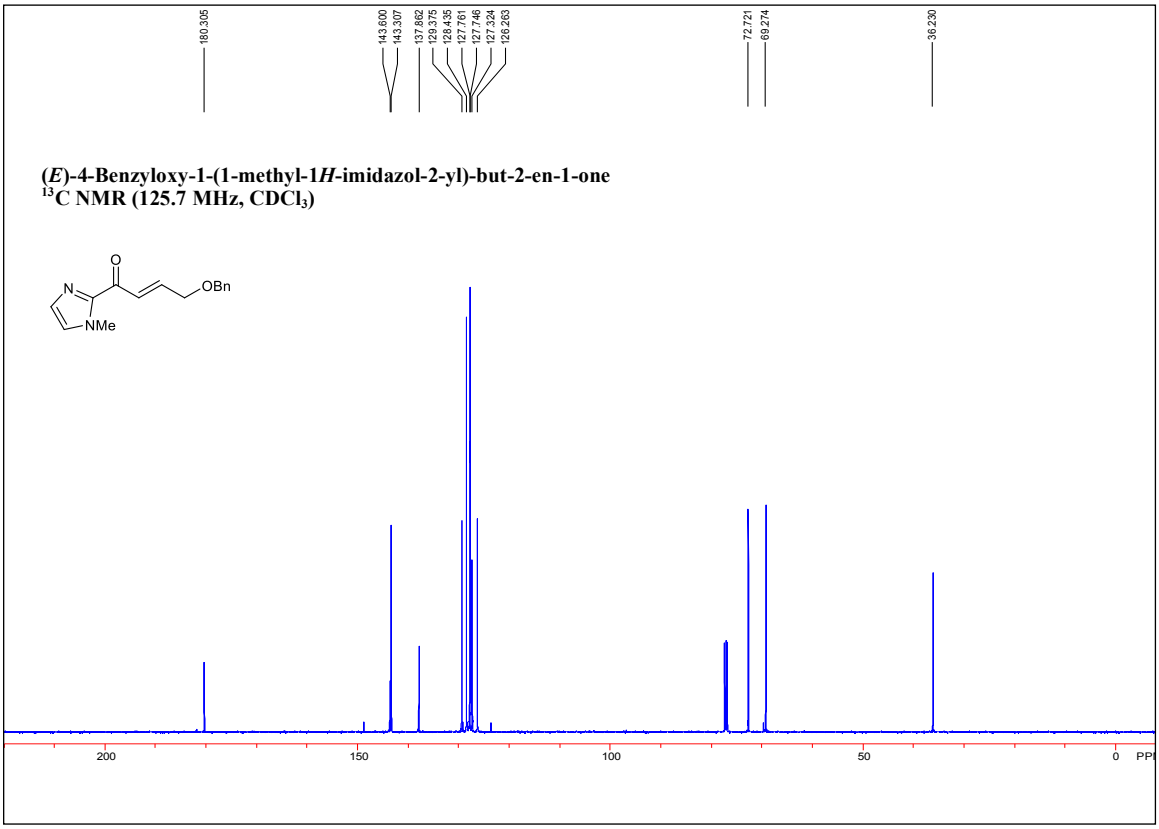
2.92
2.00
1.98
0.87
1.05
0.90
10
8
6
4
2
0
PPM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129.375
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127.746
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126.263
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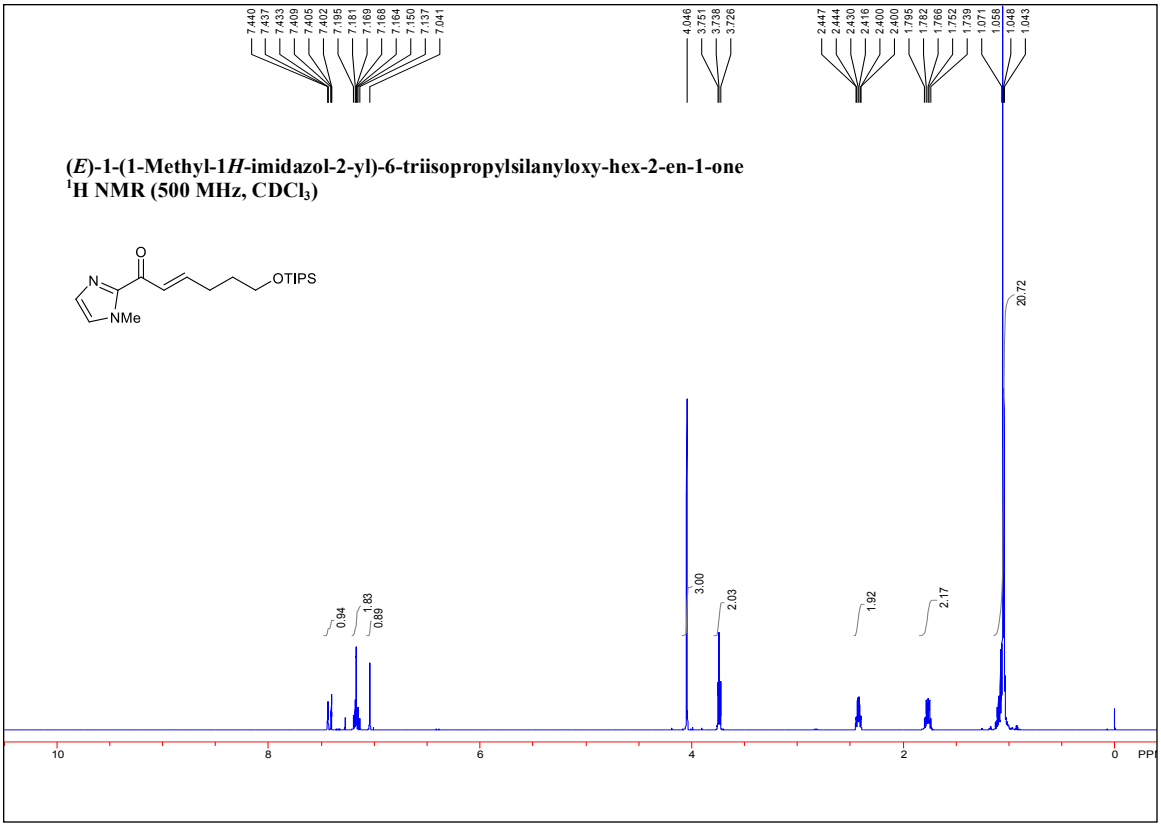
69.274
36.230
(E)-4-Benzyloxy-1-(1-methyl-1H-imidazol-2-yl)-but-2-en-1-one
13C NMR (125.7 MHz, CDCl3)
O
N
NMe
OBn
200
150
100
50
0
PPM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7.402
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7.181
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7.137
7.041
4.046
3.751
3.738
3.726
2.447
2.444
2.430
2.416
2.400
2.400
1.795
1.782
1.766
1.752
1.739
1.071
1.058
1.048
1.043
(E)-1-(1-Methyl-1H-imidazol-2-yl)-6-triisopropylsilanyloxy-hex-2-en-1-one
1H NMR (500 MHz, CDCl3)
O
N
NMe
OTIPS
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1.92
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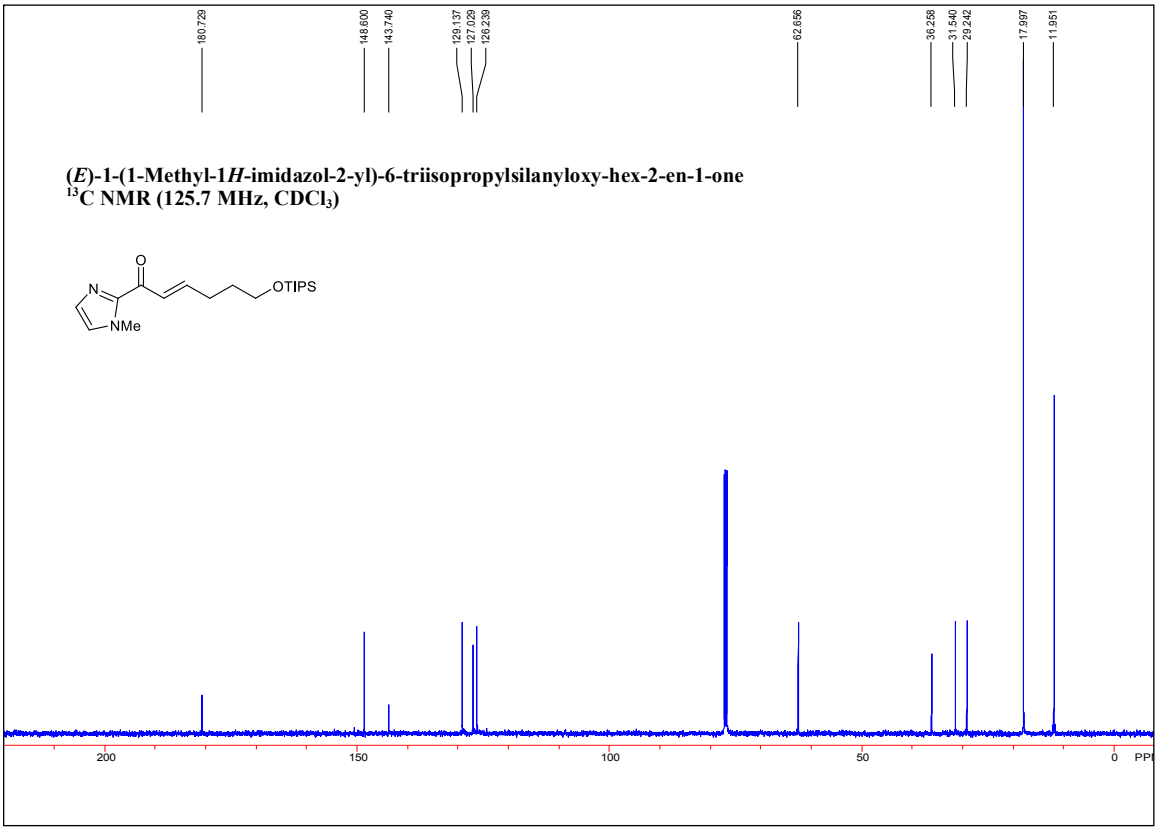
10
8
6
4
2
0
PPM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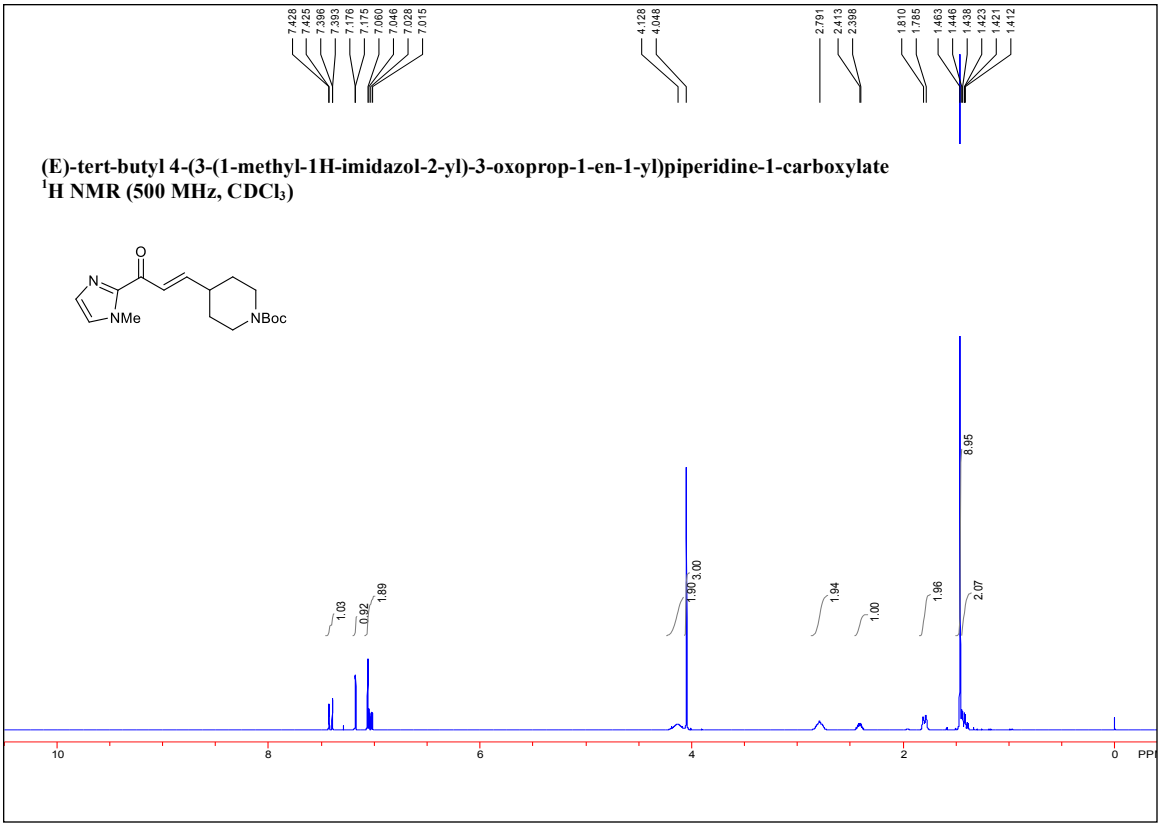
31.540
29.242
17.997
11.951
(E)-1-(1-Methyl-1H-imidazol-2-yl)-6-triisopropylsilanyloxy-hex-2-en-1-one
13C NMR (125.7 MHz, CDCl3)
O
N
NMe
OTIPS
200
150
100
50
0
PPM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7.175
7.060
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7.015
4.128
4.048
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2.413
2.398
1.810
1.785
1.463
1.446
1.438
1.423
1.421
1.412
(E)-tert-butyl 4-(3-(1-methyl-1H-imidazol-2-yl)-3-oxoprop-1-en-1-yl)piperidine-1-carboxylate
1H NMR (500 MHz, CDCl3)
O
N
NMe
NBoc
8.95
1.03
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10
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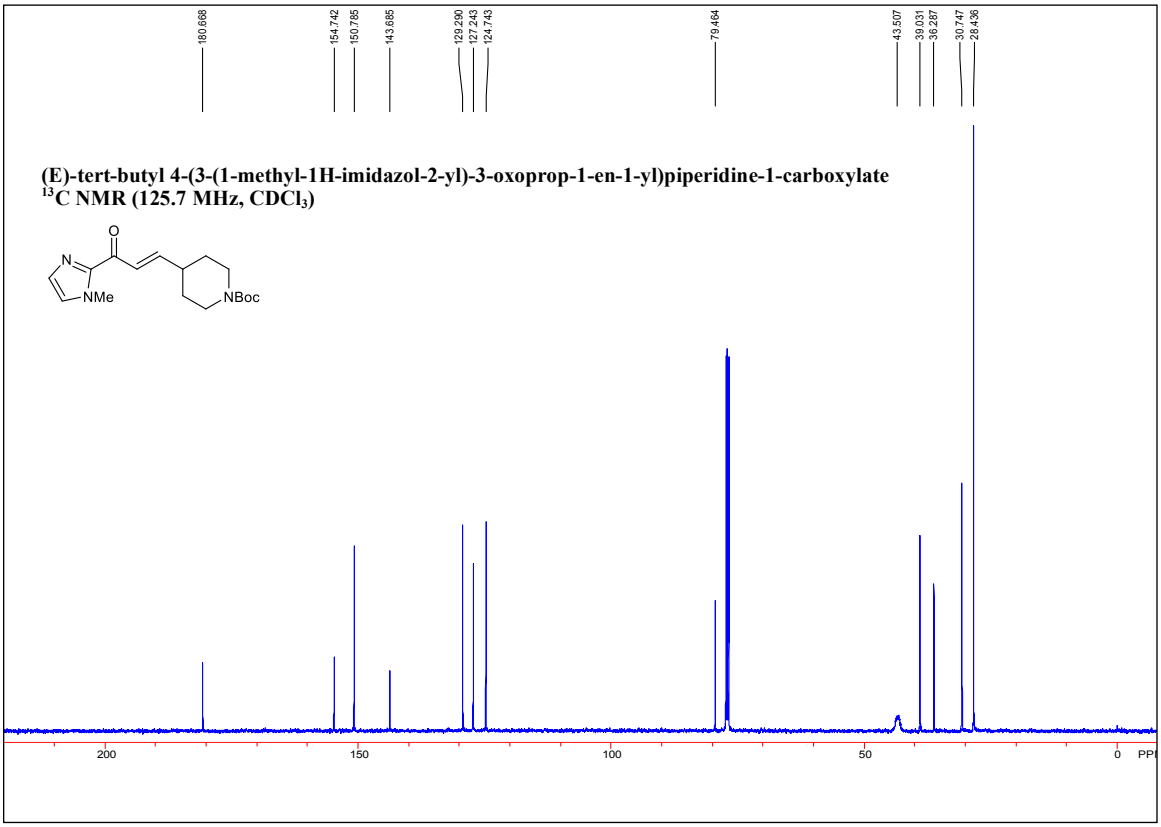
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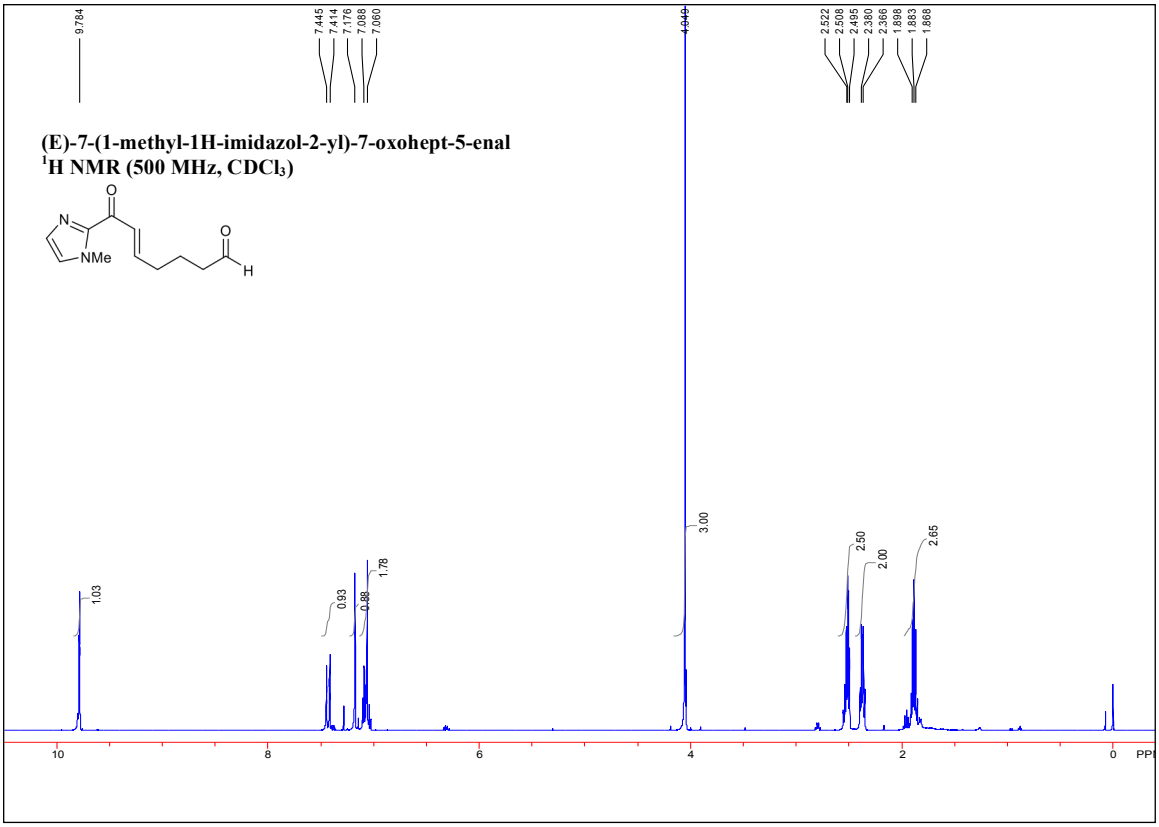
129.290
127.243
124.743
79.464
43.507
39.031
36.287
30.747
28.436
(E)-tert-butyl 4-(3-(1-methyl-1H-imidazol-2-yl)-3-oxoprop-1-en-1-yl)piperidine-1-carboxylate
13C NMR (125.7 MHz, CDCl3)
O
N
NMe
NBoc
200
150
100
50
0
PPM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7.060
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2.522
2.508
2.495
2.380
2.366
1.898
1.883
1.868
(E)-7-(1-methyl-1H-imidazol-2-yl)-7-oxohept-5-enal
1H NMR (500 MHz, CDCl3)
O
N
NMe
O
H
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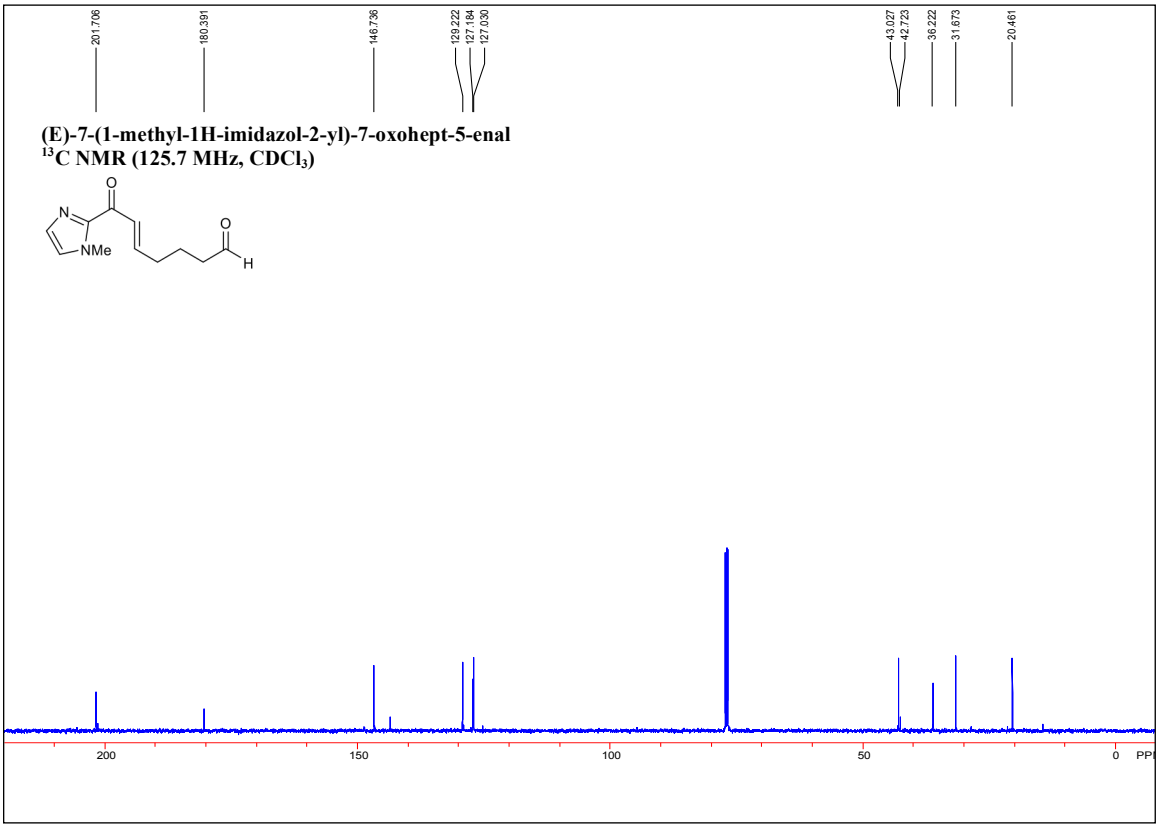
2.50
2.00
2.65
10
8
6
4
2
0
PPM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127.030
43.027
42.723
36.222
31.673
20.461
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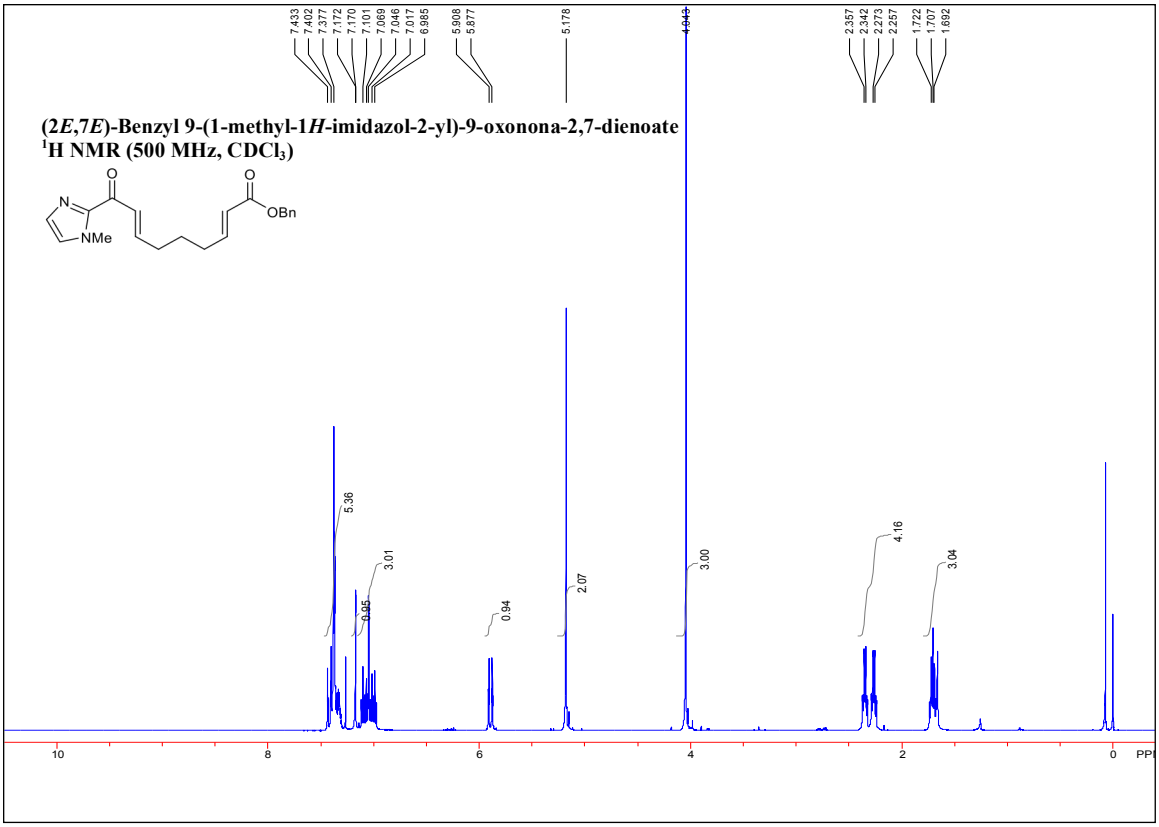
13C NMR (125.7 MHz, CDCl3)
O
N
NMe
O
H
200
150
100
50
0
PPM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7.069
7.046
7.017
6.985
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5.178
4.913
2.357
2.342
2.273
2.257
1.722
1.707
1.692
(2E,7E)-Benzyl 9-(1-methyl-1H-imidazol-2-yl)-9-oxonona-2,7-dienoate
1H NMR (500 MHz, CDCl3)
O
N
NMe
O
OBn
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3.01
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0.94
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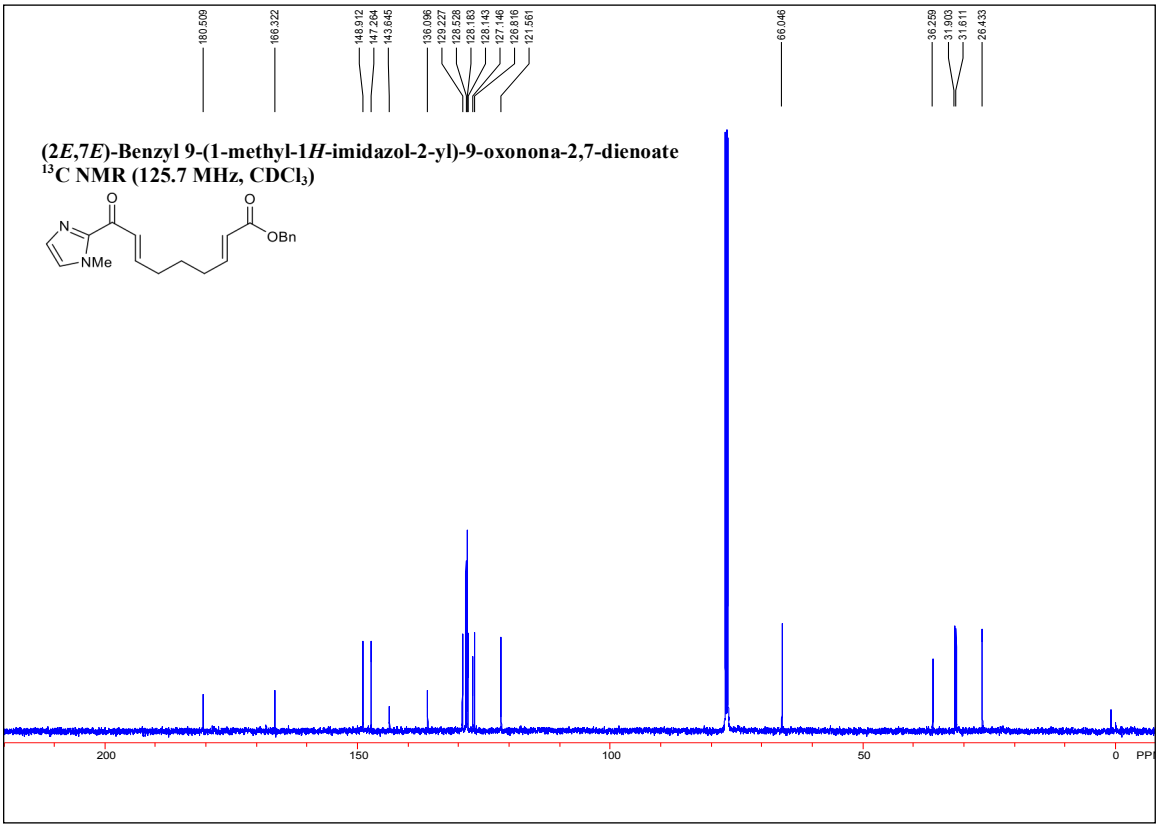
3.00
4.16
3.04
10
8
6
4
2
0
PPM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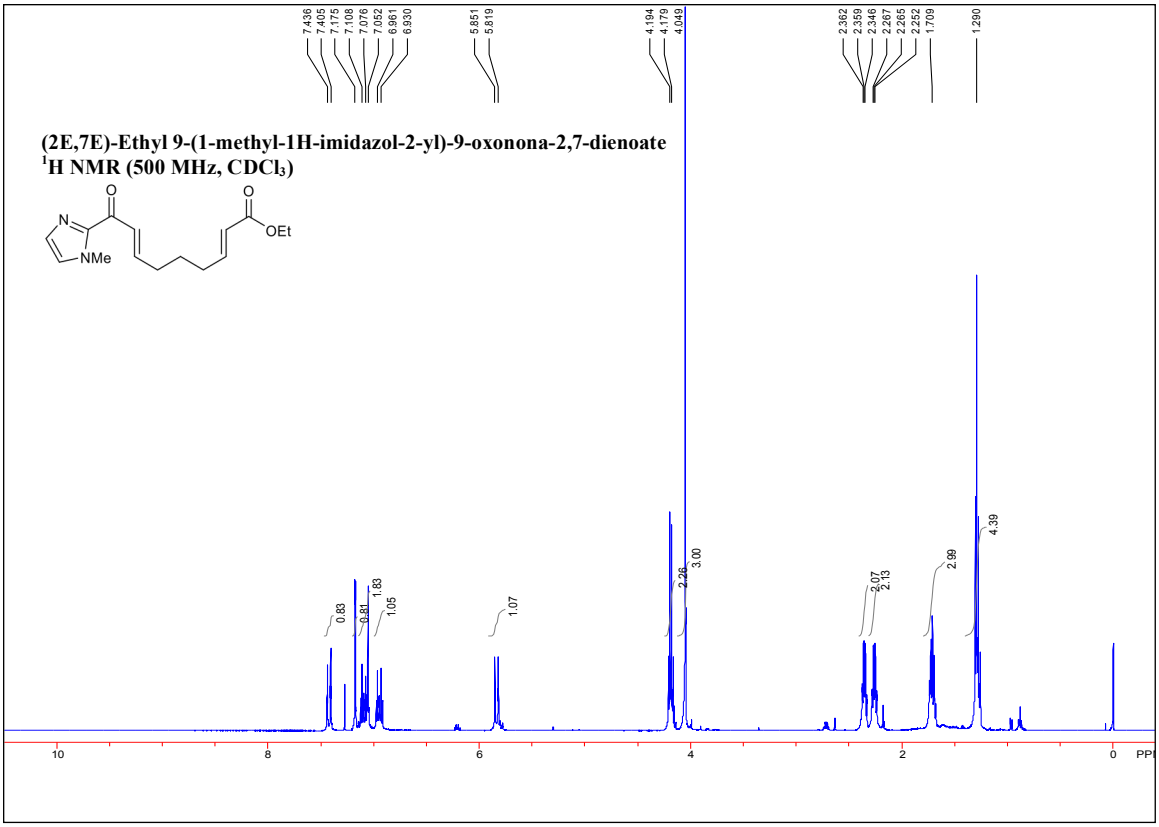
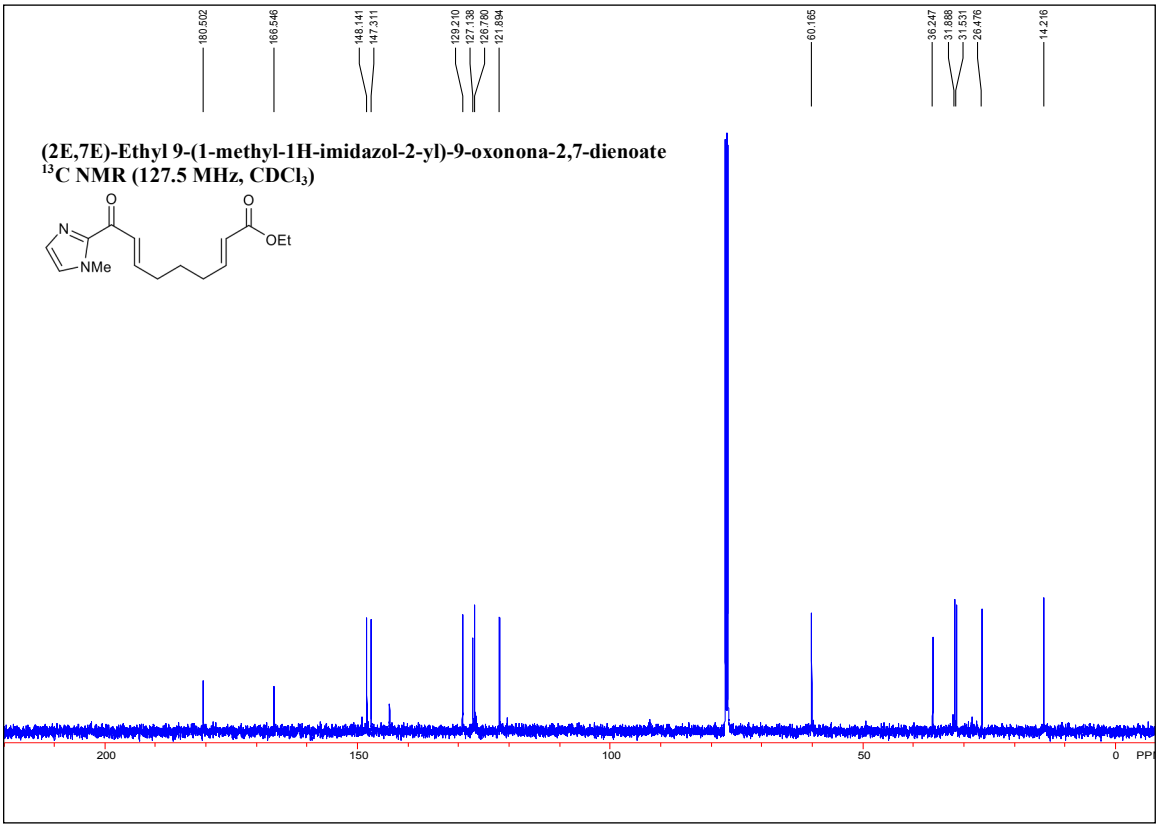
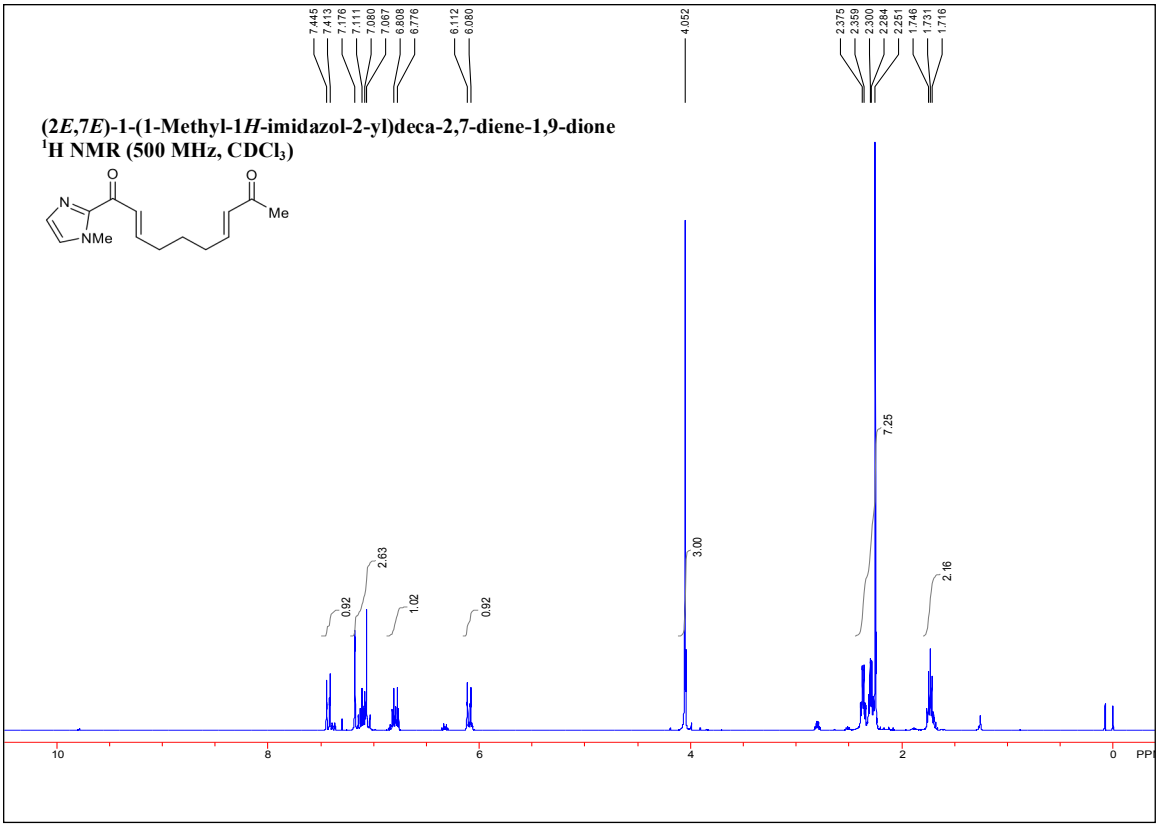
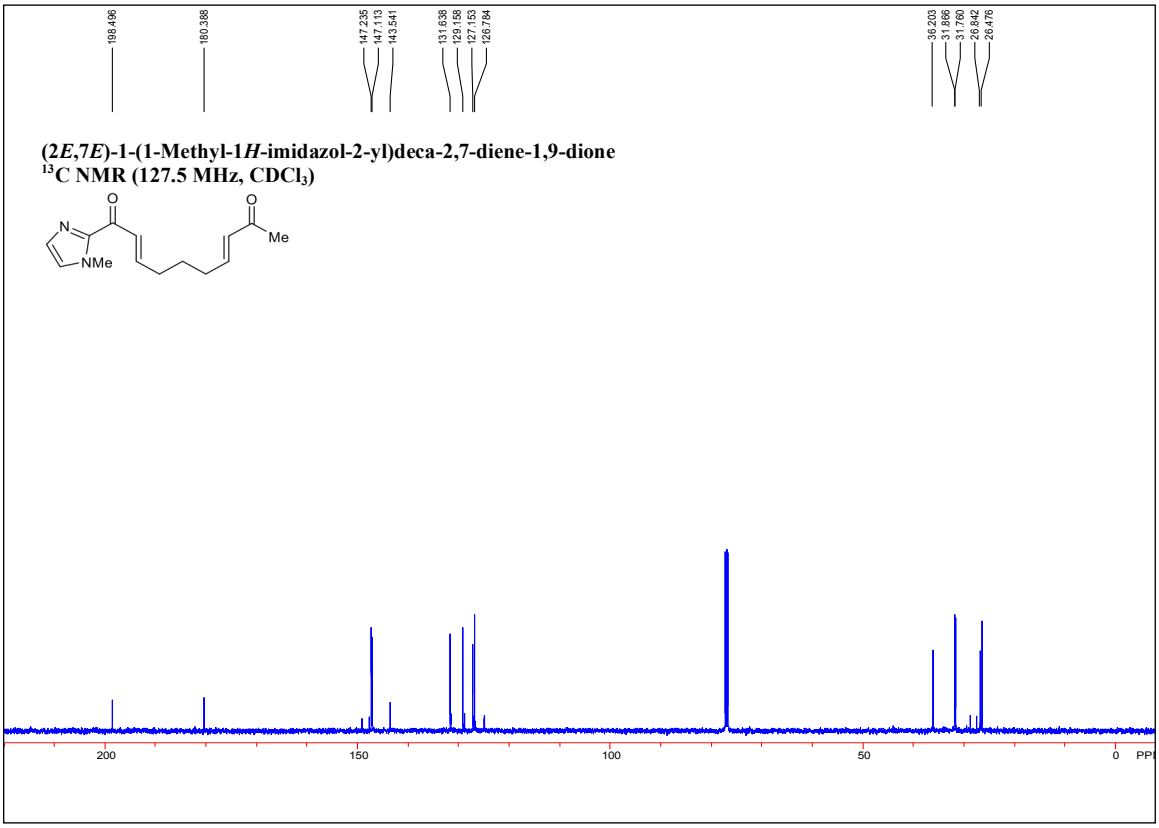
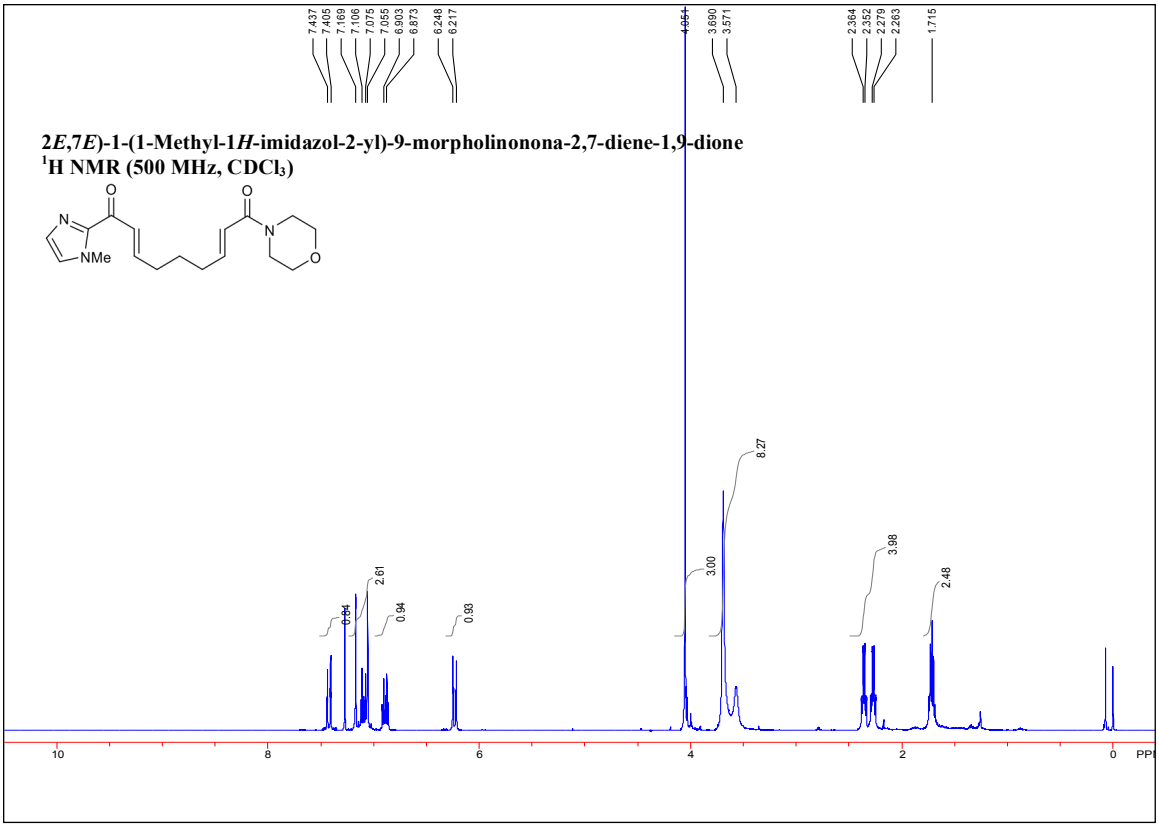
136.096
129.227
128.528
128.183
128.143
127.146
126.816
121.561
66.046
36.259
31.903
31.611
26.433
(2E,7E)-Benzyl 9-(1-methyl-1H-imidazol-2-yl)-9-oxonona-2,7-dienoate
13C NMR (125.7 MHz, CDCl3)
O
N
NMe
O
OBn
200
150
100
50
0
PPM


7.436
7.405
7.175
7.108
7.076
7.052
6.961
6.930
5.851
5.819
4.194
4.179
4.049
2.362
2.359
2.346
2.267
2.265
2.252
1.709
1.290
(2E,7E)-Ethyl 9-(1-methyl-1H-imidazol-2-yl)-9-oxonona-2,7-dienoate
1H NMR (500 MHz, CDCl3)
O
N
NMe
O
OEt
0.83
0.81
1.83
1.05
1.07
2.26
3.00
2.07
2.13
2.99
4.39
10
8
6
4
2
0
PPM

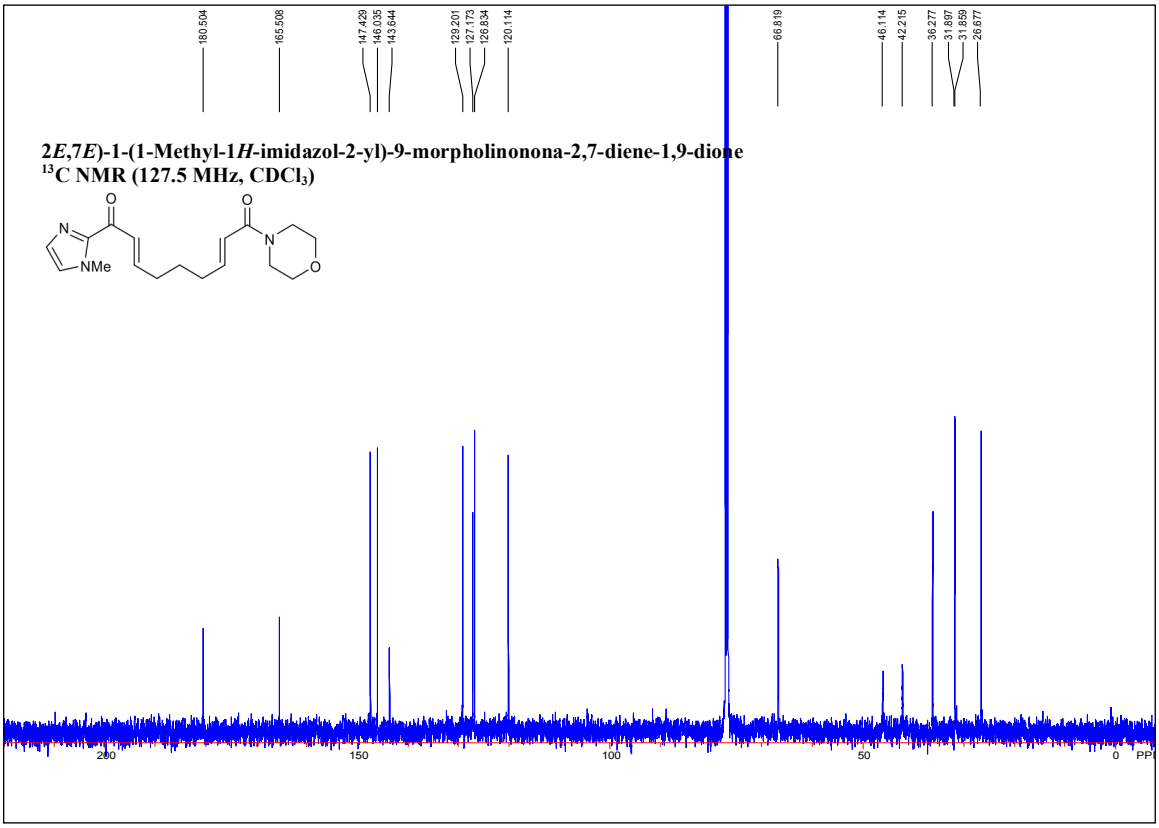

180.502
166.546
148.141
147.311
129.210
127.138
126.780
121.894
60.165
36.247
31.888
31.531
26.476
14.216
(2E,7E)-Ethyl 9-(1-methyl-1H-imidazol-2-yl)-9-oxonona-2,7-dienoate
13C NMR (127.5 MHz, CDCl3)
O
N
NMe
O
OEt
200
150
100
50
0
PPM


7.445
7.413
7.176
7.111
7.080
7.067
6.808
6.776
6.112
6.080
4.052
2.375
2.359
2.300
2.284
2.251
1.746
1.731
1.716
(2E,7E)-1-(1-Methyl-1H-imidazol-2-yl)deca-2,7-diene-1,9-dione
1H NMR (500 MHz, CDCl3)
O
N
NMe
O
Me
7.25
3.00
2.63
2.16
1.02
0.92
0.92
10
8
6
4
2
0
PPM

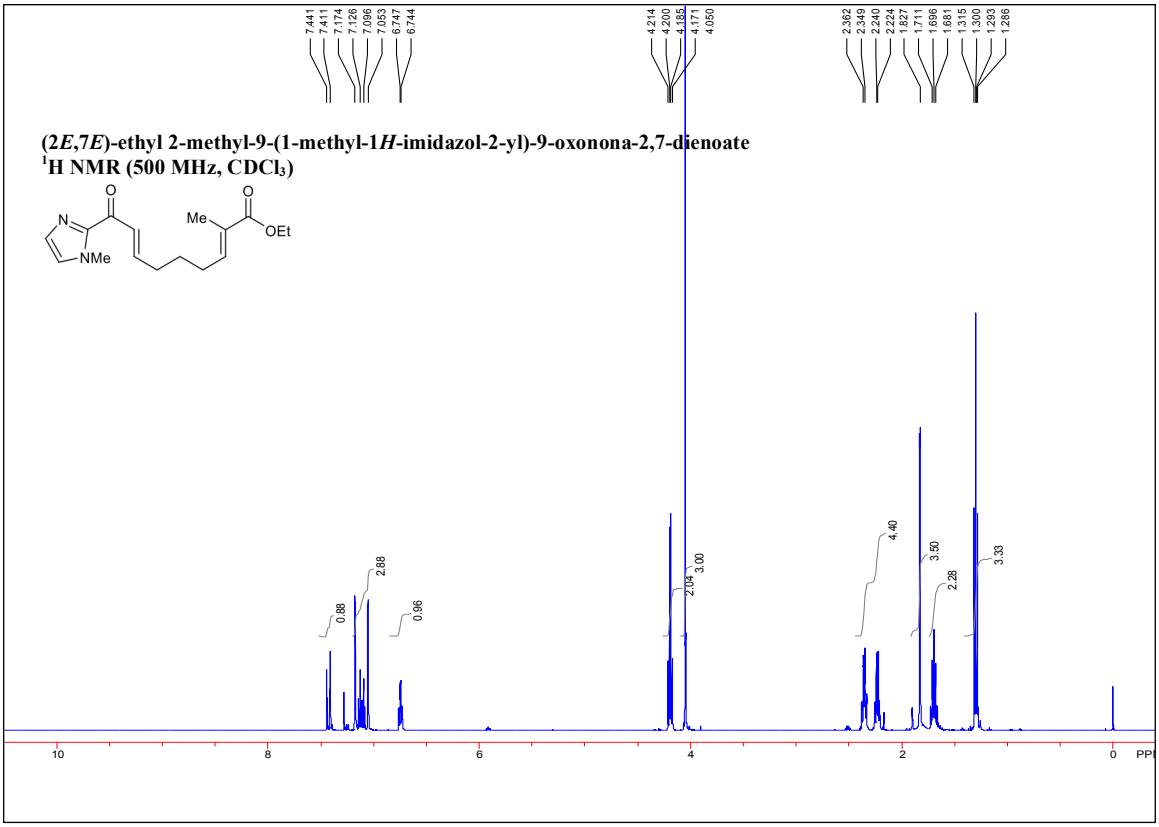

198.496
180.388
147.235
147.113
143.541
131.638
129.158
127.153
126.784
36.203
31.866
31.760
26.642
26.476
(2E,7E)-1-(1-Methyl-1H-imidazol-2-yl)deca-2,7-diene-1,9-dione
13C NMR (127.5 MHz, CDCl3)
O
N
NMe
O
Me
200
150
100
50
0
PPM

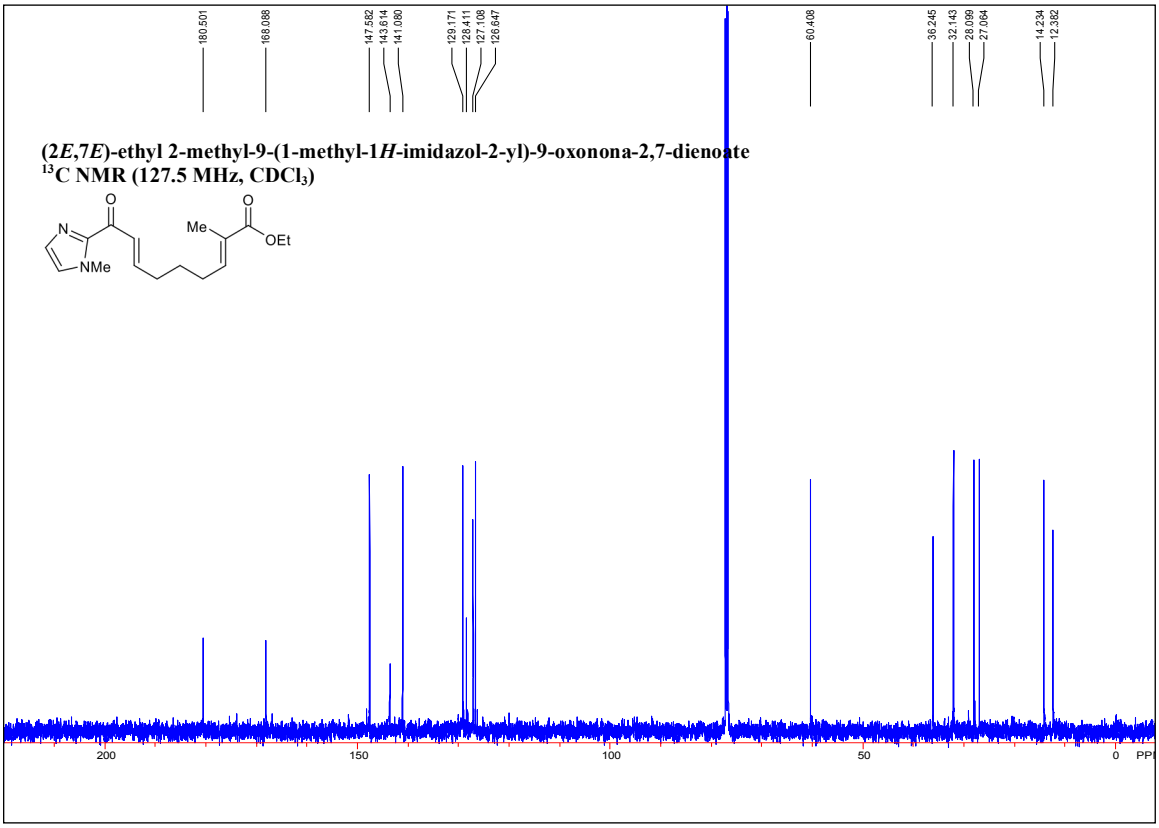


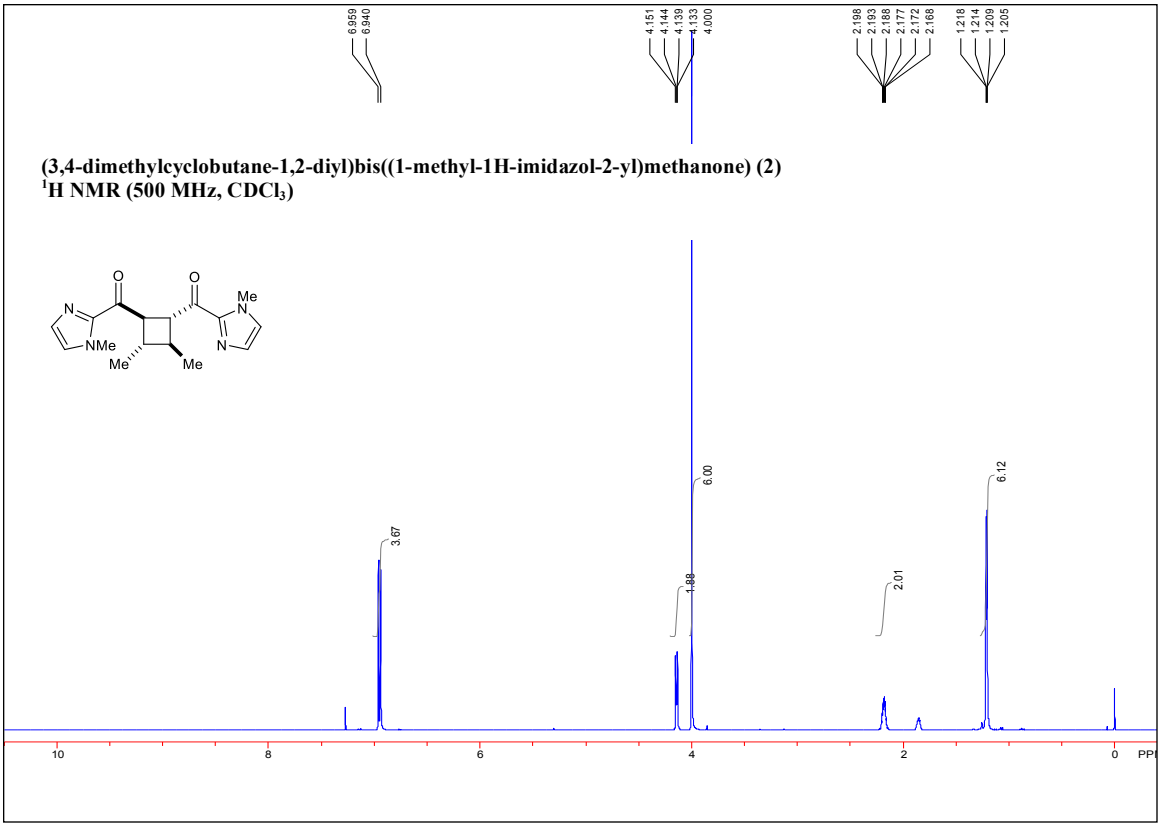
2*E*,7*E*)-1-(1-Methyl-1*H*-imidazol-2-yl)-9-morpholinonona-2,7-diene-1,9-dione
^{1}H NMR (500 MHz, $CDCl_3$)

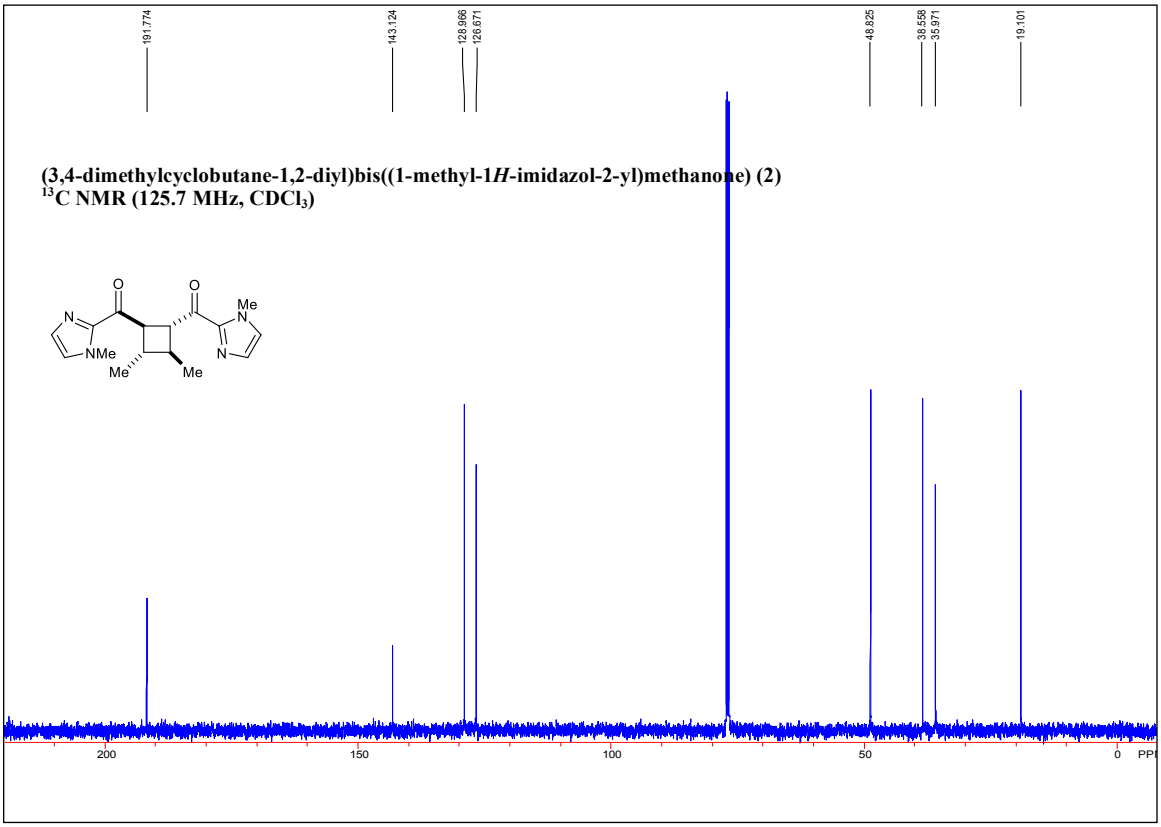


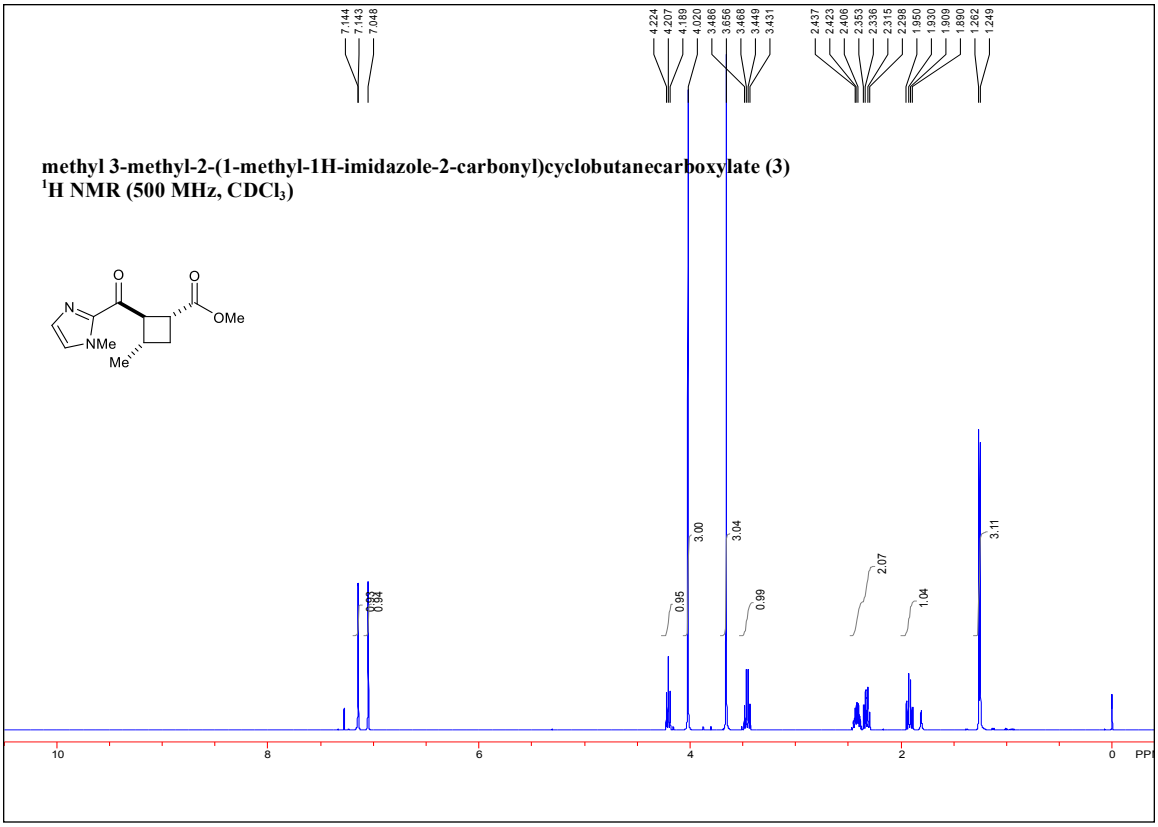
2*E*,7*E*)-1-(1-Methyl-1*H*-imidazol-2-yl)-9-morpholinonona-2,7-diene-1,9-dione
^{13}C NMR (127.5 MHz, $CDCl_3$)

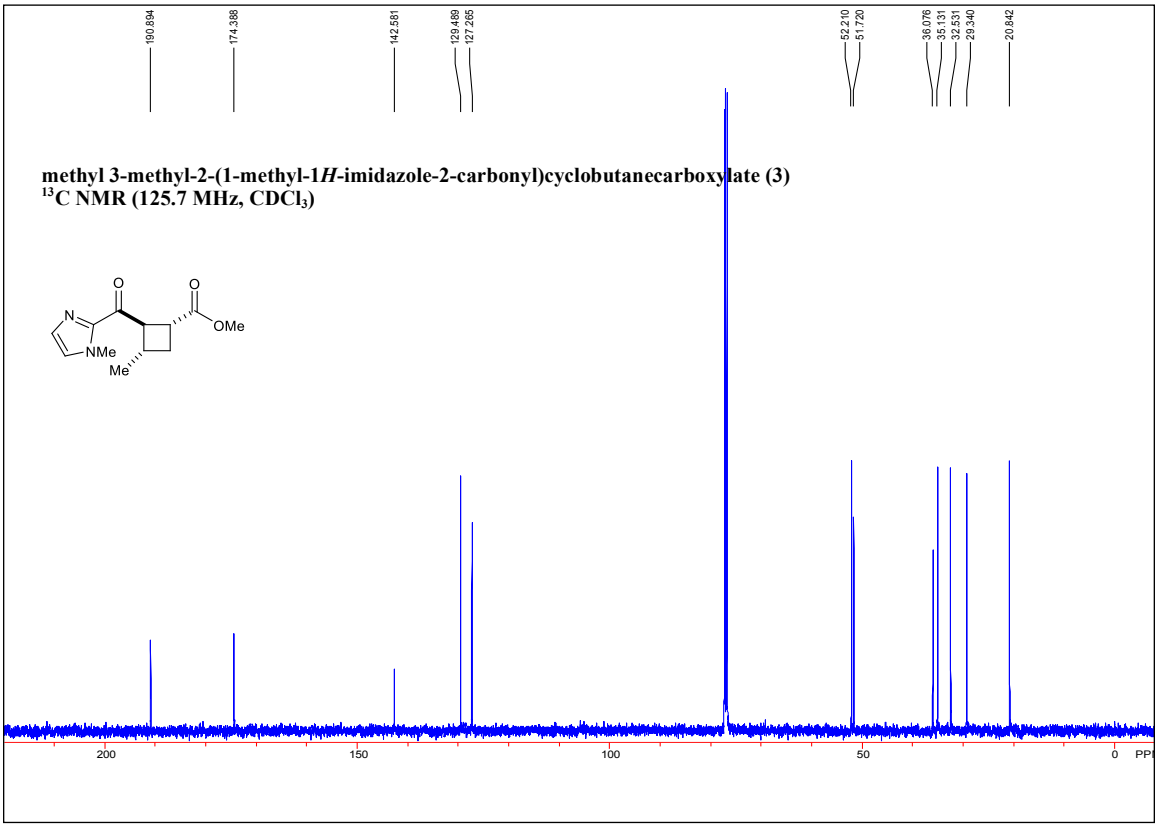

7.441
7.411
7.174
7.126
7.096
7.053
6.747
6.744
4.214
4.200
4.185
4.171
4.050
2.362
2.349
2.240
2.224
1.827
1.711
1.696
1.681
1.315
1.300
1.293
1.286
(2E,7E)-ethyl 2-methyl-9-(1-methyl-1H-imidazol-2-yl)-9-oxonona-2,7-dienoate
1H NMR (500 MHz, CDCl3)
O
N
NMe
O
Me
OEt
0.88
2.88
0.96
2.04
3.00
4.40
3.50
2.28
3.33
10
8
6
4
2
0
PPM

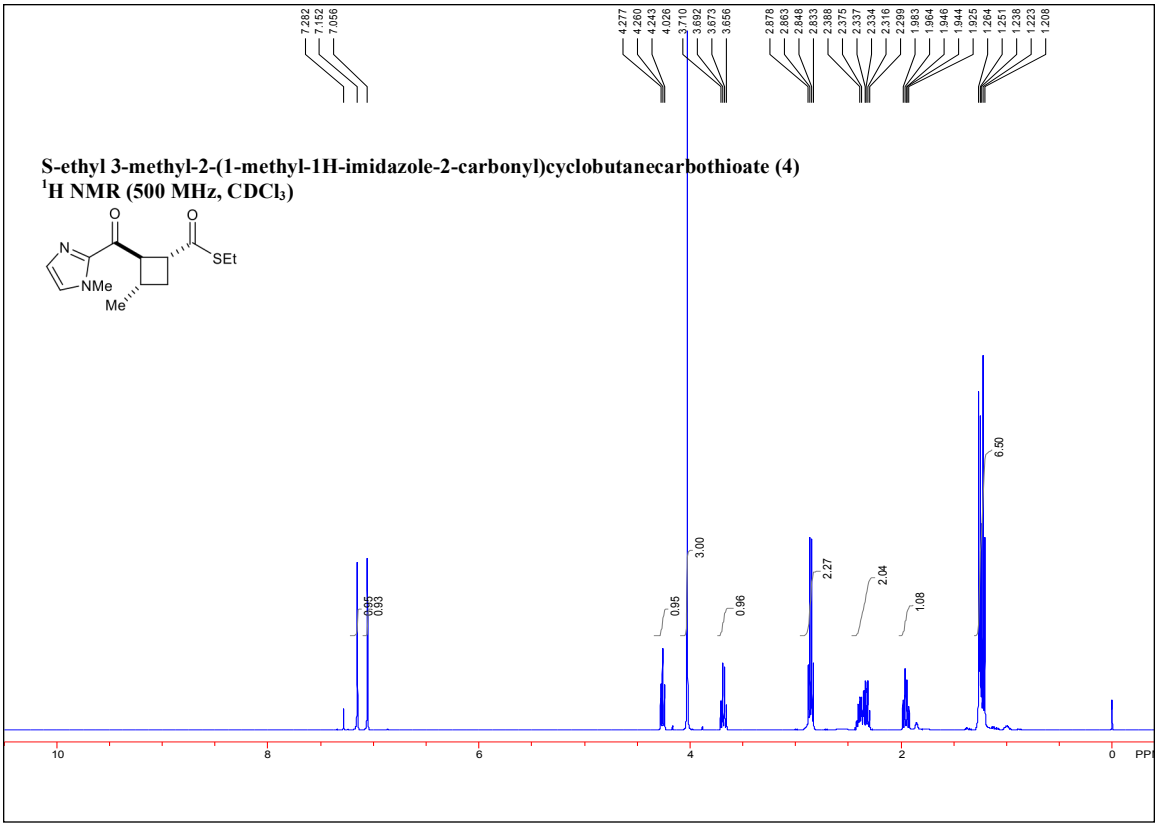

180.501
168.088
147.582
143.614
141.080
129.171
128.411
127.108
126.647
60.408
36.245
32.143
28.099
27.064
14.234
12.382
(2E,7E)-ethyl 2-methyl-9-(1-methyl-1H-imidazol-2-yl)-9-oxonona-2,7-dienoate
13C NMR (127.5 MHz, CDCl3)
O
N
NMe
O
Me
OEt
200
150
100
50
0
PPM

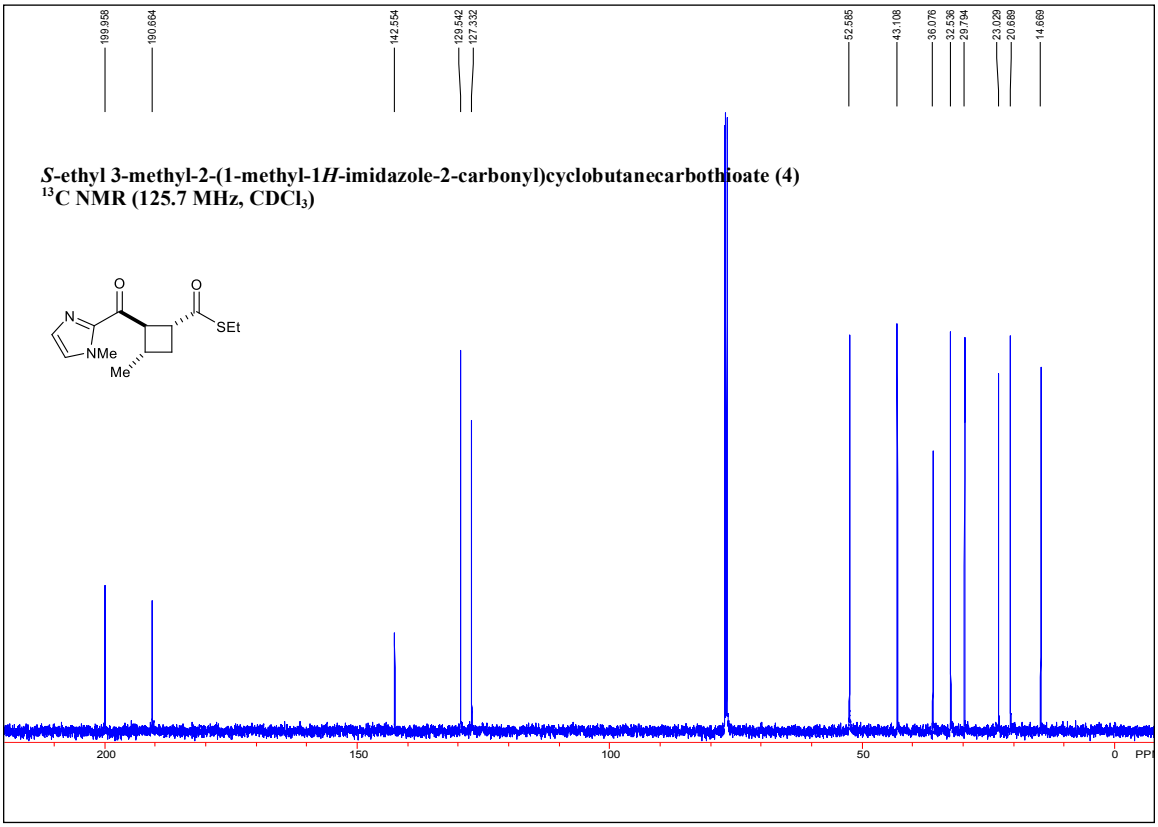

6.959
6.940
4.151
4.144
4.139
4.133
4.000
2.198
2.193
2.188
2.177
2.172
2.168
1.218
1.214
1.209
1.205
(3,4-dimethylcyclobutane-1,2-diyl)bis((1-methyl-1H-imidazol-2-yl)methanone) (2)
1H NMR (500 MHz, CDCl3)
O
O
N
NMe
Me
Me
Me
N
N
3.67
1.88
6.00
2.01
6.12
10
8
6
4
2
0
PPM

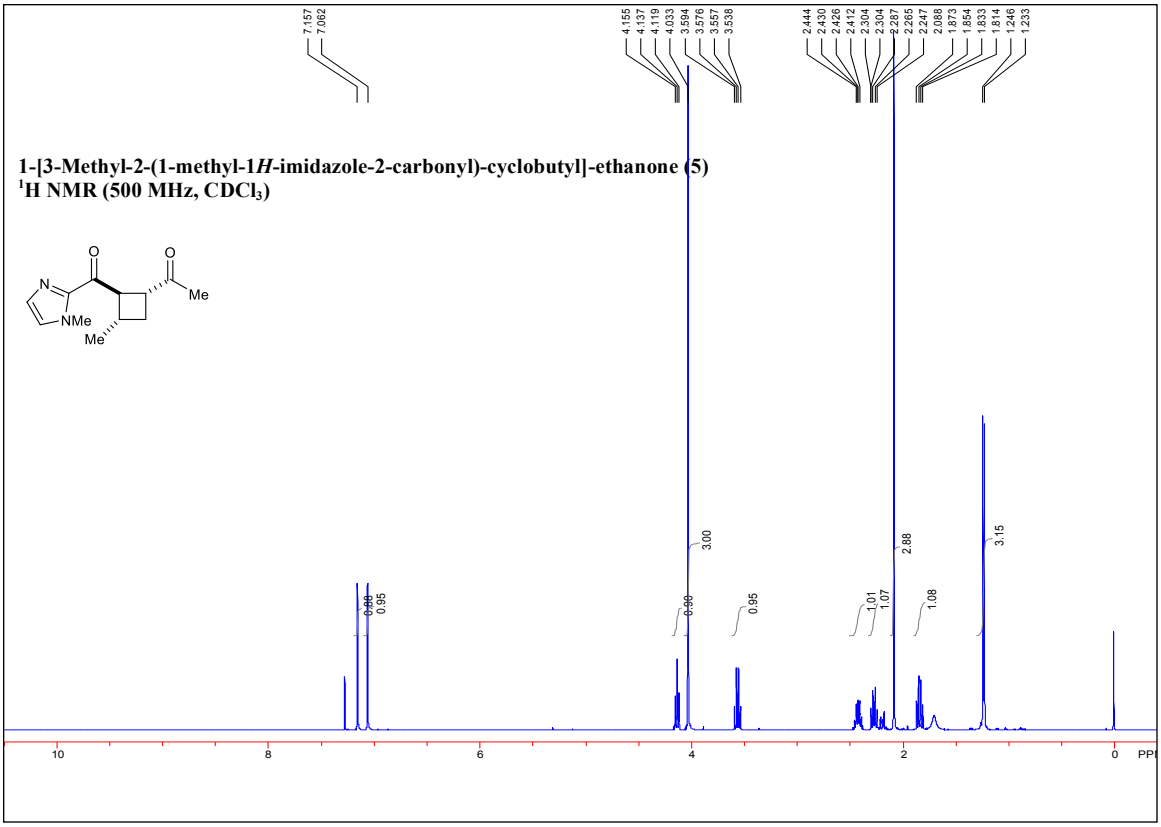

191.774
143.124
128.966
126.671
48.825
38.558
35.971
19.101
(3,4-dimethylcyclobutane-1,2-diyl)bis((1-methyl-1H-imidazol-2-yl)methanone) (2)
13C NMR (125.7 MHz, CDCl3)
O
O
N
NMe
Me
Me
Me
N
N
200
150
100
50
0
PPM

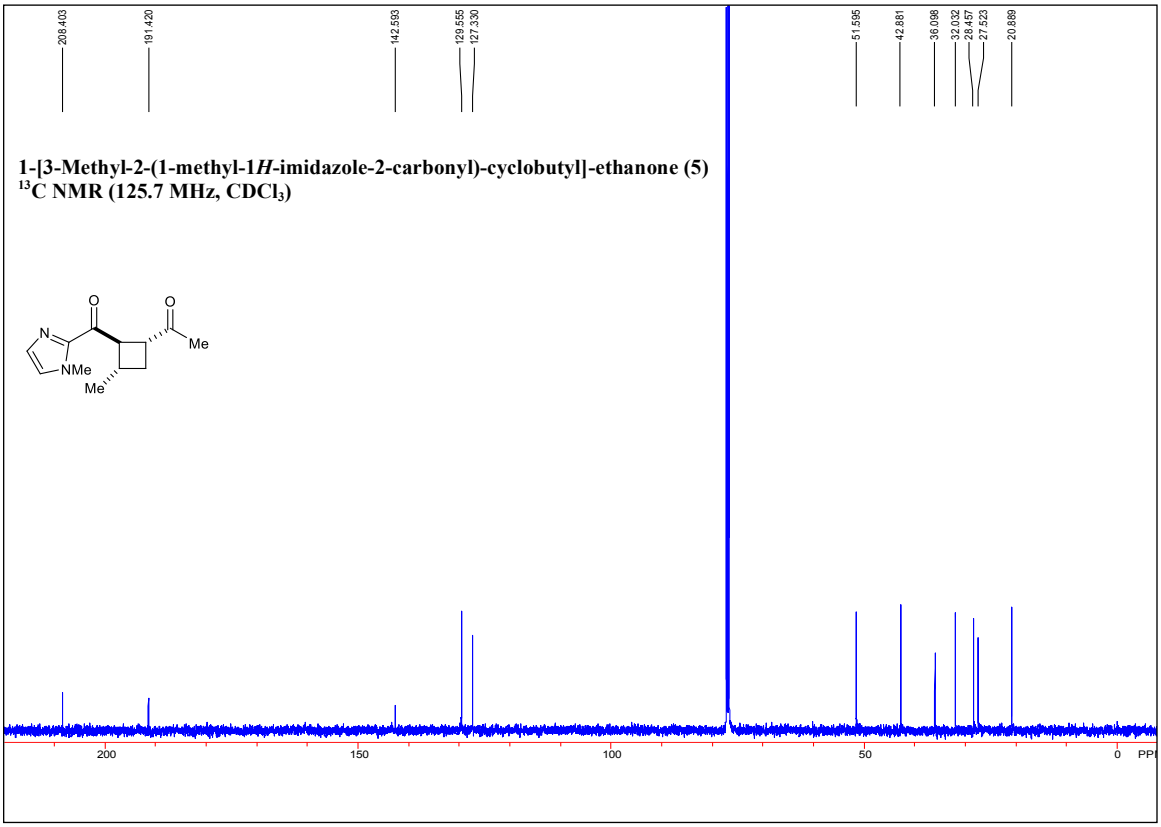

7.144
7.143
7.048
4.224
4.207
4.189
4.020
3.466
3.656
3.468
3.449
3.431
2.437
2.423
2.406
2.353
2.336
2.315
2.298
1.950
1.930
1.909
1.890
1.262
1.249
methyl 3-methyl-2-(1-methyl-1H-imidazole-2-carbonyl)cyclobutanecarboxylate (3)
1H NMR (500 MHz, CDCl3)
O
O
N
OMe
NMe
Me
3.00
3.04
3.11
2.07
0.95
0.99
1.04
0.93
0.94
10
8
6
4
2
0
PPM

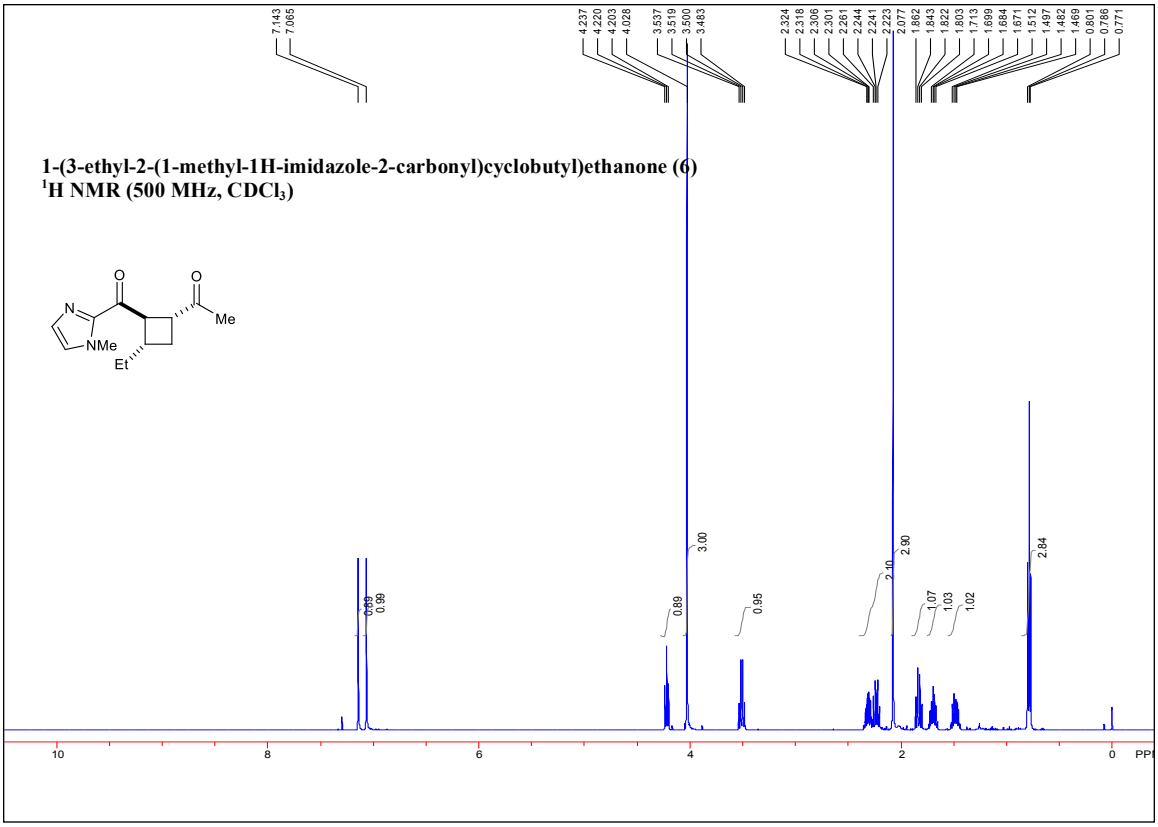

190.894
174.388
142.581
129.489
127.265
52.210
51.720
36.076
35.131
32.531
29.340
20.842
methyl 3-methyl-2-(1-methyl-1H-imidazole-2-carbonyl)cyclobutanecarboxylate (3)
13C NMR (125.7 MHz, CDCl3)
O
O
N
OMe
NMe
Me
200
150
100
50
0
PPM

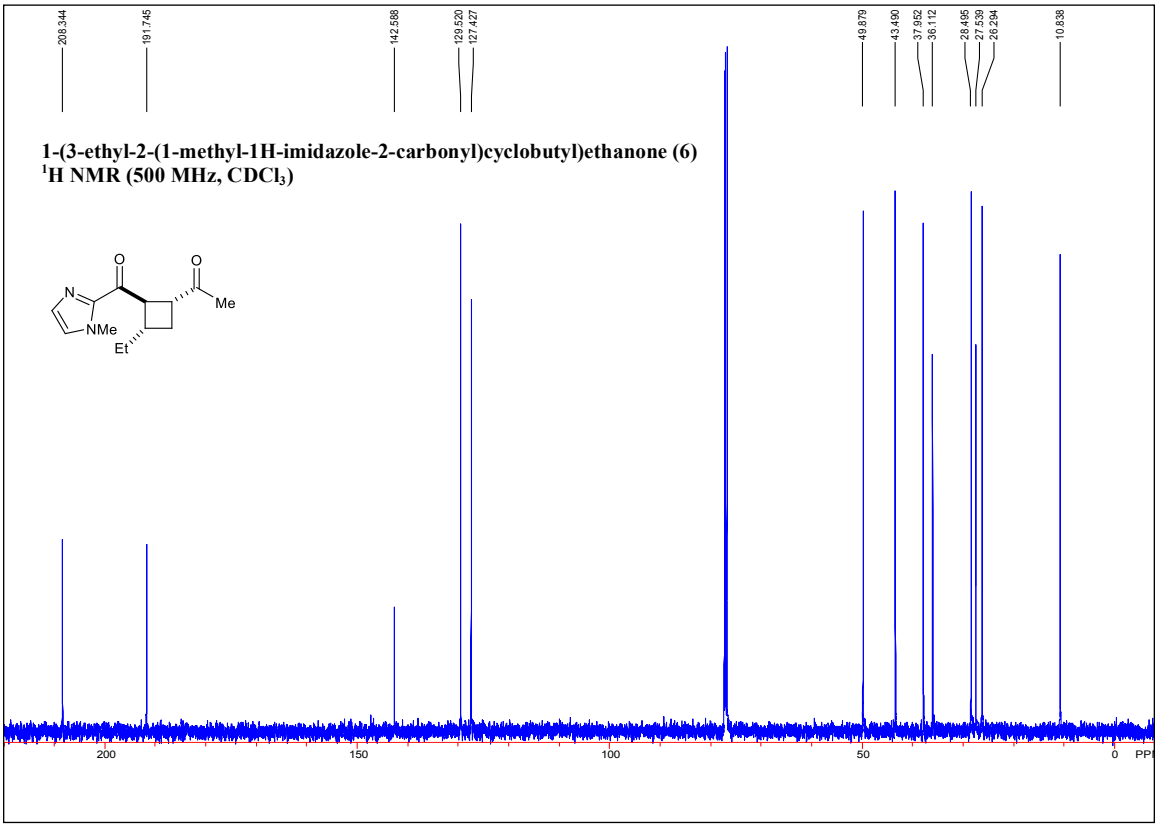

7.282
7.152
7.056
4.277
4.260
4.243
4.026
3.710
3.692
3.673
3.656
2.878
2.863
2.848
2.833
2.388
2.375
2.337
2.334
2.316
2.299
1.983
1.964
1.946
1.944
1.925
1.264
1.251
1.238
1.223
1.208
S-ethyl 3-methyl-2-(1-methyl-1H-imidazole-2-carbonyl)cyclobutanecarbothioate (4)
1H NMR (500 MHz, CDCl3)
O
O
N
SEt
NMe
Me
0.95
0.93
0.95
3.00
0.96
2.27
2.04
1.08
6.50
10
8
6
4
2
0
PPI

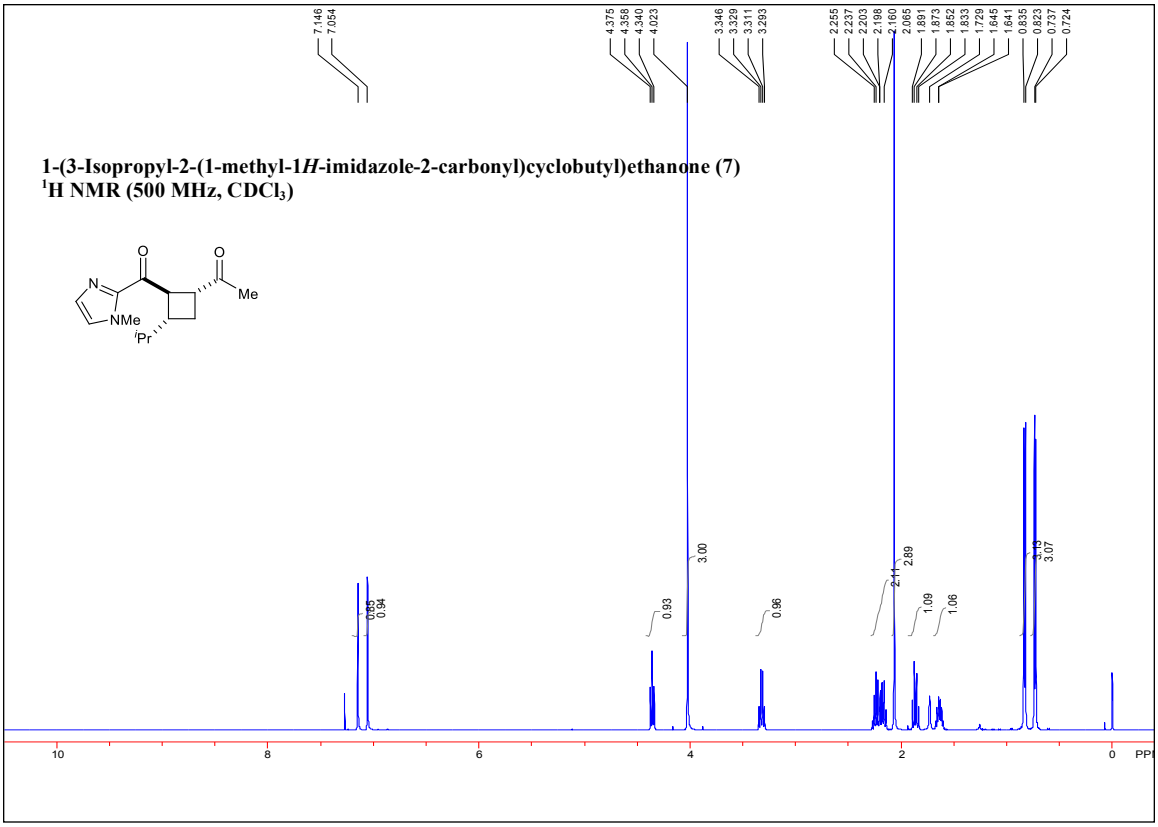

199.958
190.664
142.554
129.542
127.332
52.585
43.108
36.076
32.536
29.794
23.029
20.689
14.669
S-ethyl 3-methyl-2-(1-methyl-1H-imidazole-2-carbonyl)cyclobutanecarbothioate (4)
13C NMR (125.7 MHz, CDCl3)
O
O
N
SEt
NMe
Me
200
150
100
50
0
PPI


7.157
7.062
4.155
4.137
4.119
4.033
3.594
3.576
3.557
3.538
2.444
2.430
2.426
2.412
2.304
2.304
2.287
2.265
2.247
2.088
1.873
1.854
1.833
1.814
1.246
1.233
1-[3-Methyl-2-(1-methyl-1H-imidazole-2-carbonyl)-cyclobutyl]-ethanone (5)
1H NMR (500 MHz, CDCl3)
O
O
N
Me
NMe
Me
0.88
0.95
3.00
0.99
0.95
1.01
1.07
2.88
1.08
3.15
10
8
6
4
2
0
PPM

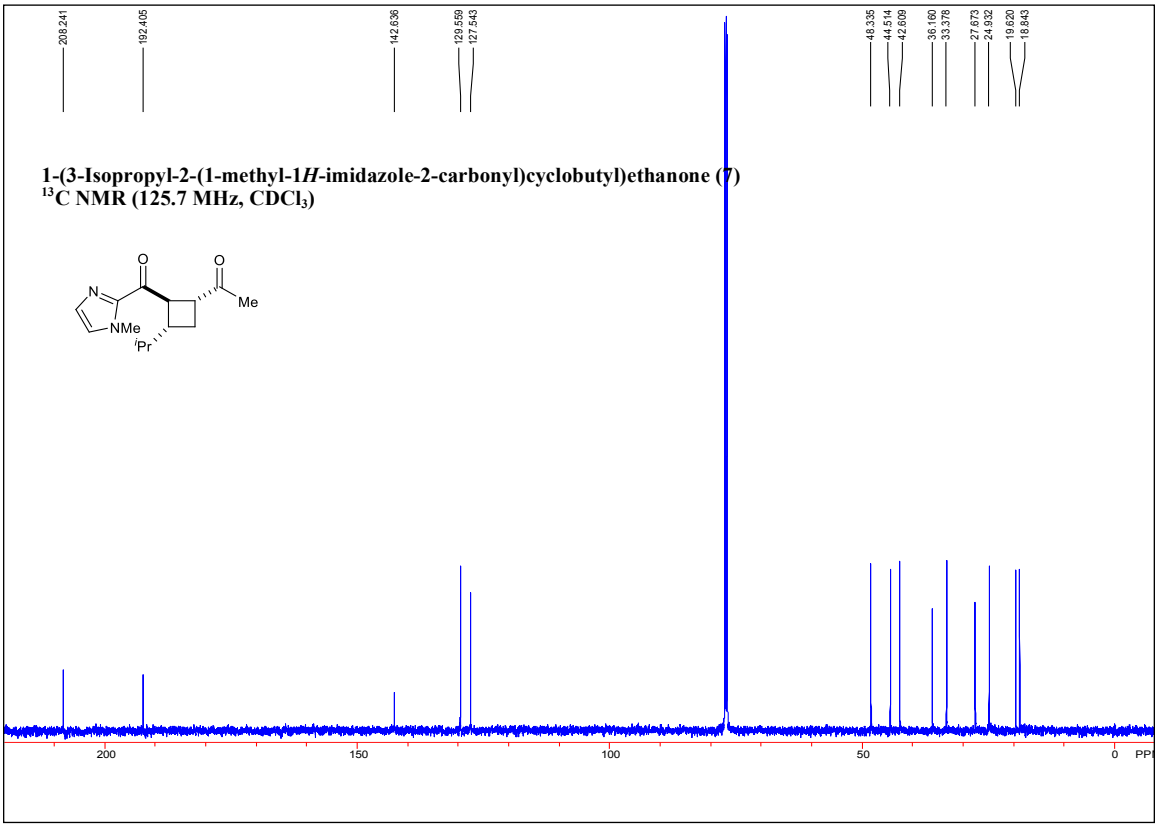

208.403
191.420
142.593
129.555
127.300
51.595
42.881
36.098
32.032
28.457
27.523
20.889
1-[3-Methyl-2-(1-methyl-1H-imidazole-2-carbonyl)-cyclobutyl]-ethanone (5)
13C NMR (125.7 MHz, CDCl3)
O
O
N
Me
NMe
Me
200
150
100
50
0
PPM


7.143
7.065
4.237
4.220
4.203
4.028
3.537
3.519
3.500
3.483
2.324
2.318
2.306
2.301
2.261
2.244
2.241
2.223
2.077
1.862
1.843
1.822
1.803
1.713
1.699
1.684
1.671
1.512
1.497
1.492
1.469
0.801
0.786
0.771
1-(3-ethyl-2-(1-methyl-1H-imidazole-2-carbonyl)cyclobutyl)ethanone (6)
1H NMR (500 MHz, CDCl3)
O
O
N
Me
NMe
Et
0.89
0.99
0.89
3.00
0.95
2.10
2.90
1.07
1.03
1.02
2.84
10
8
6
4
2
0
PPM

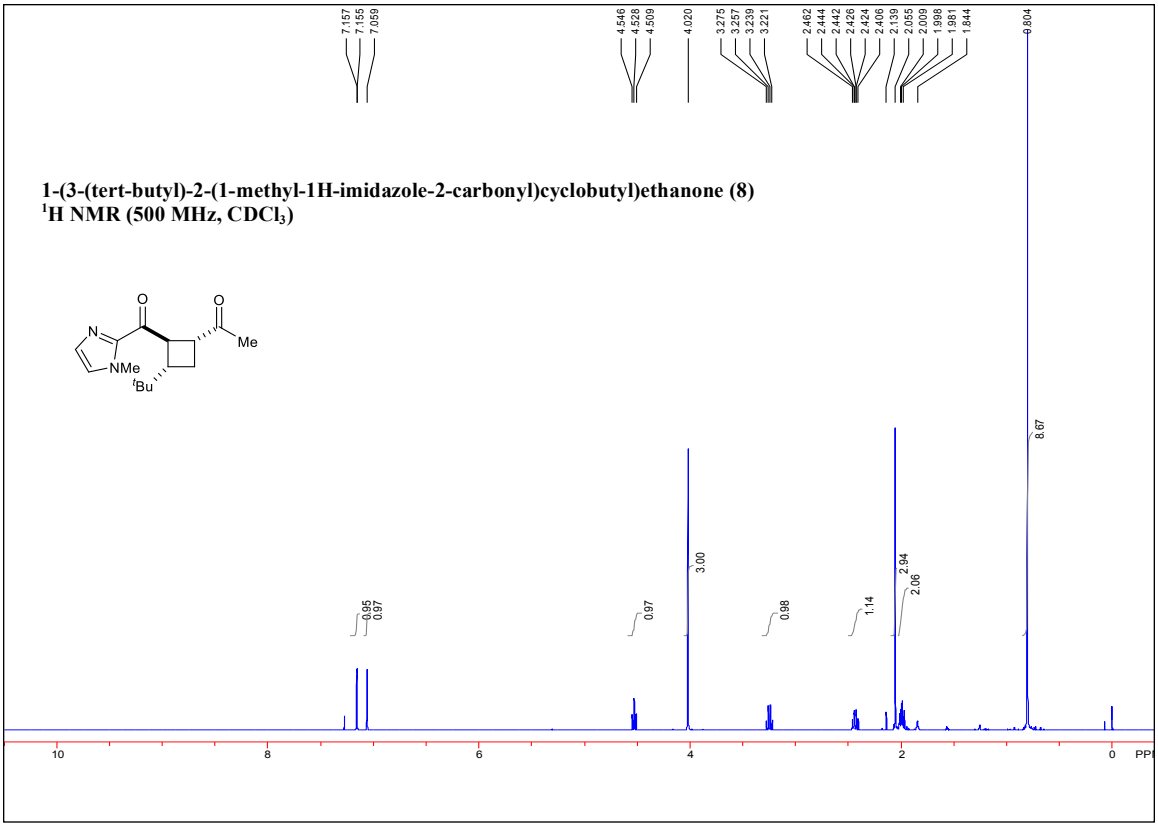

208.344
191.745
142.588
129.520
127.427
49.879
43.490
37.852
36.112
28.495
27.539
26.294
10.838
1-(3-ethyl-2-(1-methyl-1H-imidazole-2-carbonyl)cyclobutyl)ethanone (6)
1H NMR (500 MHz, CDCl3)
O
O
N
Me
NMe
Et
200
150
100
50
0
PPM



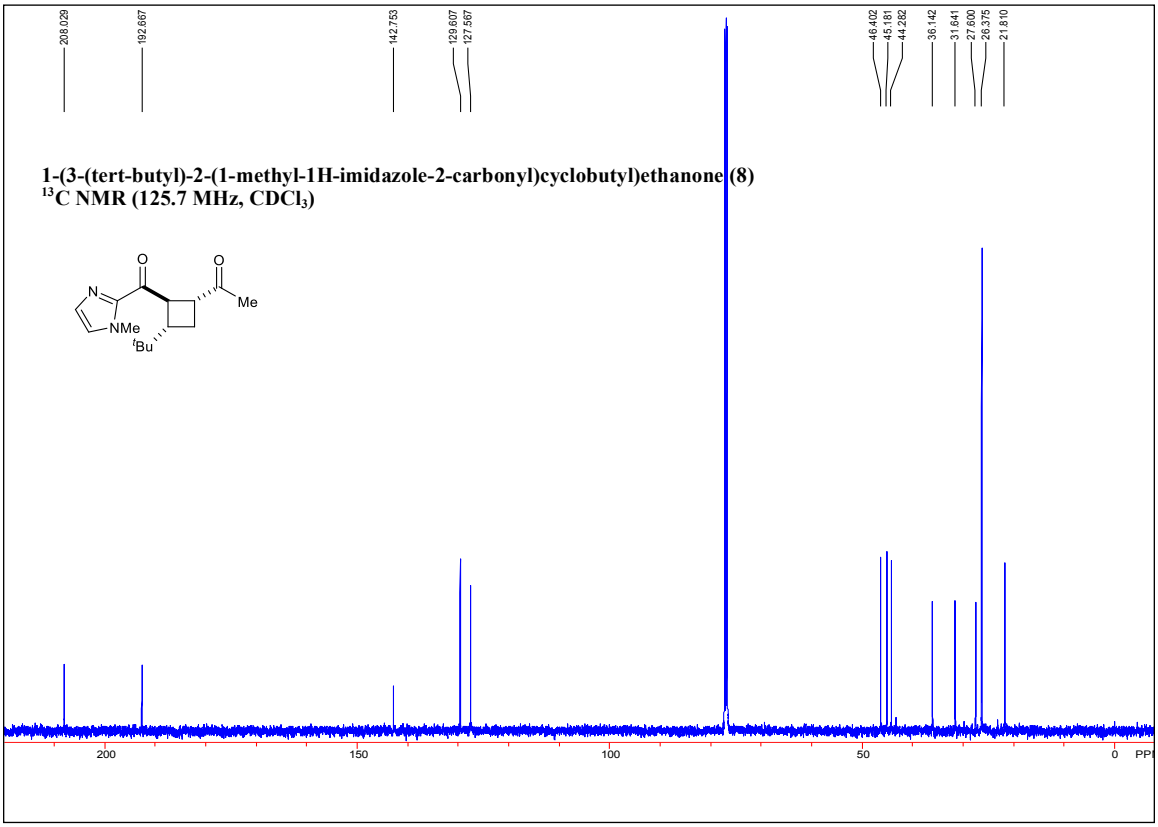
1-(3-Isopropyl-2-(1-methyl-1*H*-imidazole-2-carbonyl)cyclobutyl)ethanone (7)
^{1}H NMR (500 MHz, $CDCl_3$)



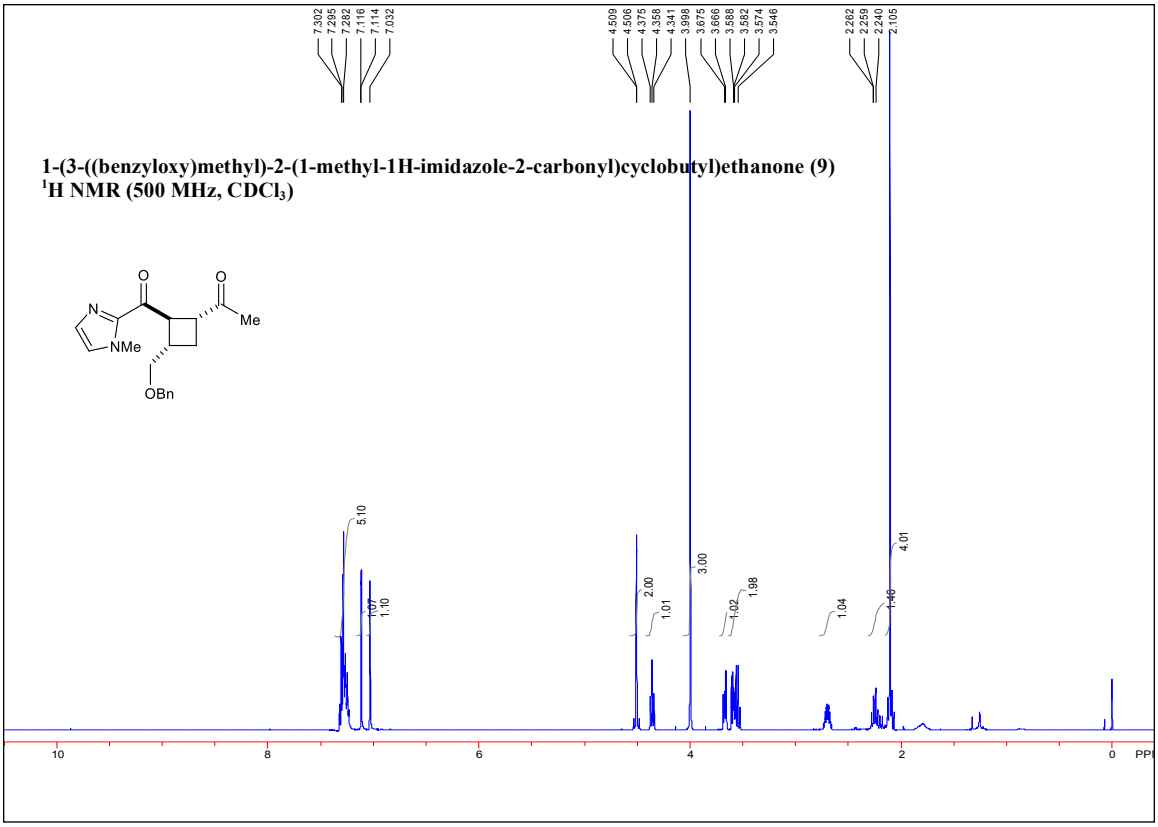
1-(3-Isopropyl-2-(1-methyl-1*H*-imidazole-2-carbonyl)cyclobutyl)ethanone (7)
^{13}C NMR (125.7 MHz, $CDCl_3$)

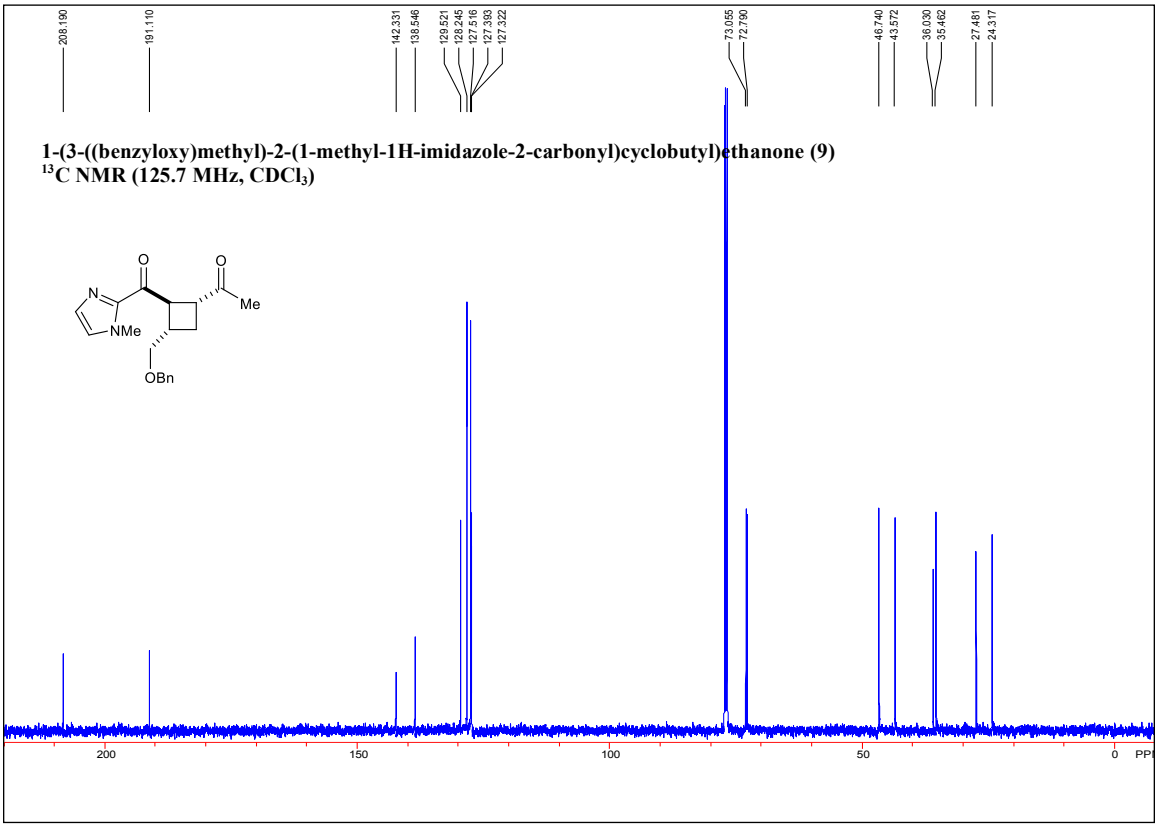


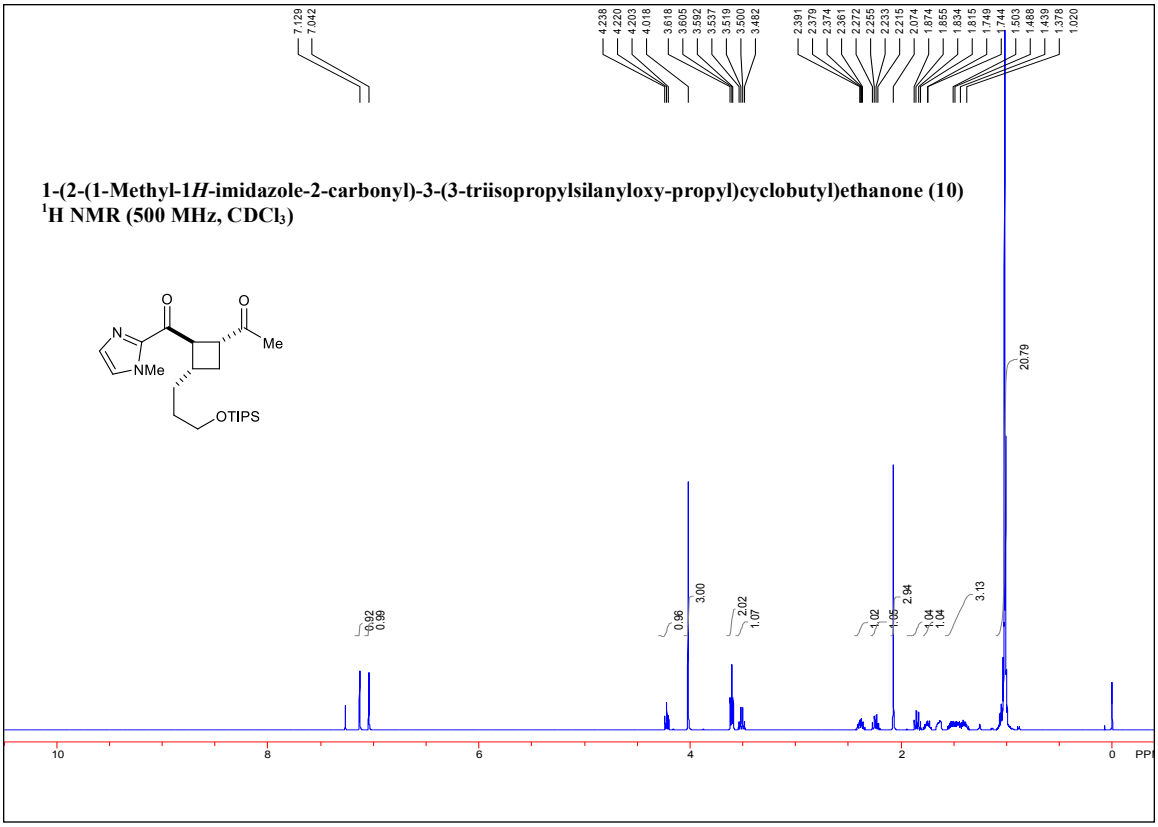
1-(3-(tert-butyl)-2-(1-methyl-1H-imidazole-2-carbonyl)cyclobutyl)ethanone (8)
^{1}H NMR (500 MHz, $CDCl_3$)



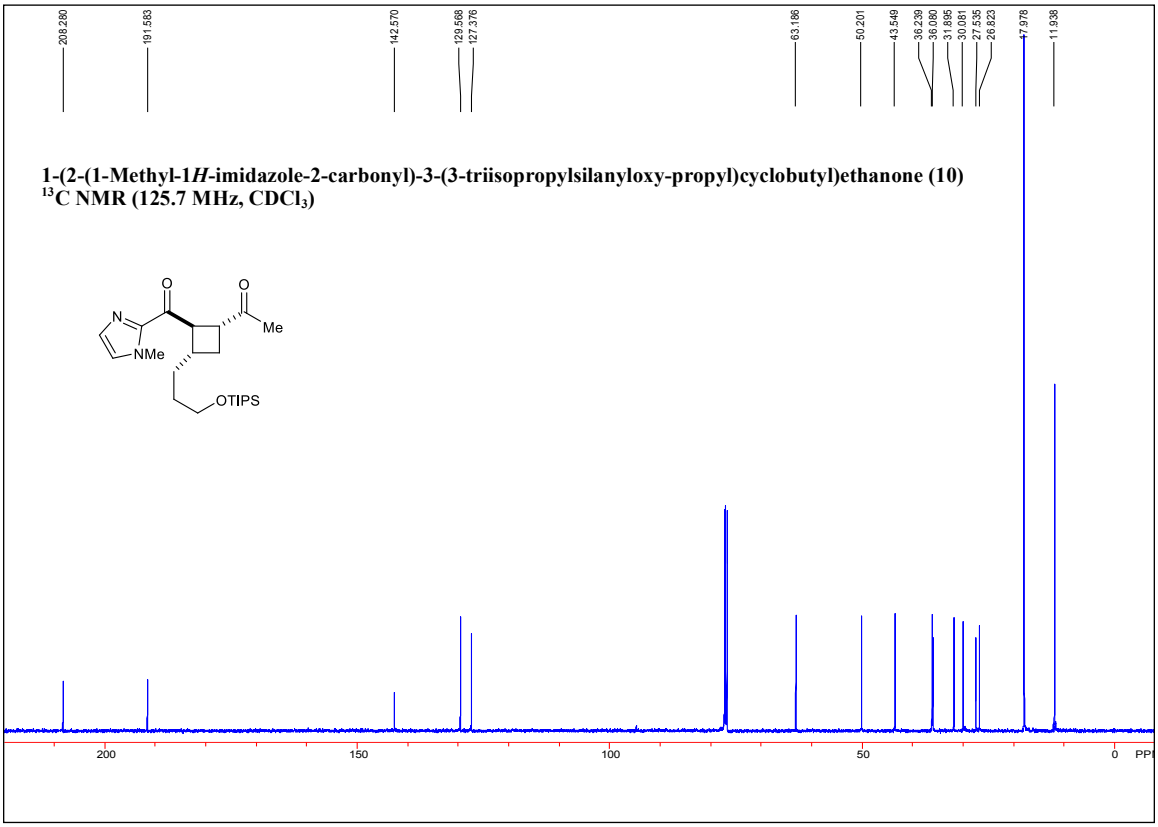
1-(3-(tert-butyl)-2-(1-methyl-1H-imidazole-2-carbonyl)cyclobutyl)ethanone (8)
^{13}C NMR (125.7 MHz, $CDCl_3$)


7.302
7.295
7.282
7.116
7.114
7.032
4.509
4.506
4.375
4.358
4.341
3.998
3.675
3.666
3.588
3.582
3.574
3.546
2.262
2.259
2.240
2.105
1-(3-((benzyloxy)methyl)-2-(1-methyl-1H-imidazole-2-carbonyl)cyclobutyl)ethanone (9)
1H NMR (500 MHz, CDCl3)
O
O
N
Me
NMe
OBn
5.10
1.07
1.10
2.00
1.01
3.00
1.02
1.98
1.04
1.46
4.01
10
8
6
4
2
0
PPM

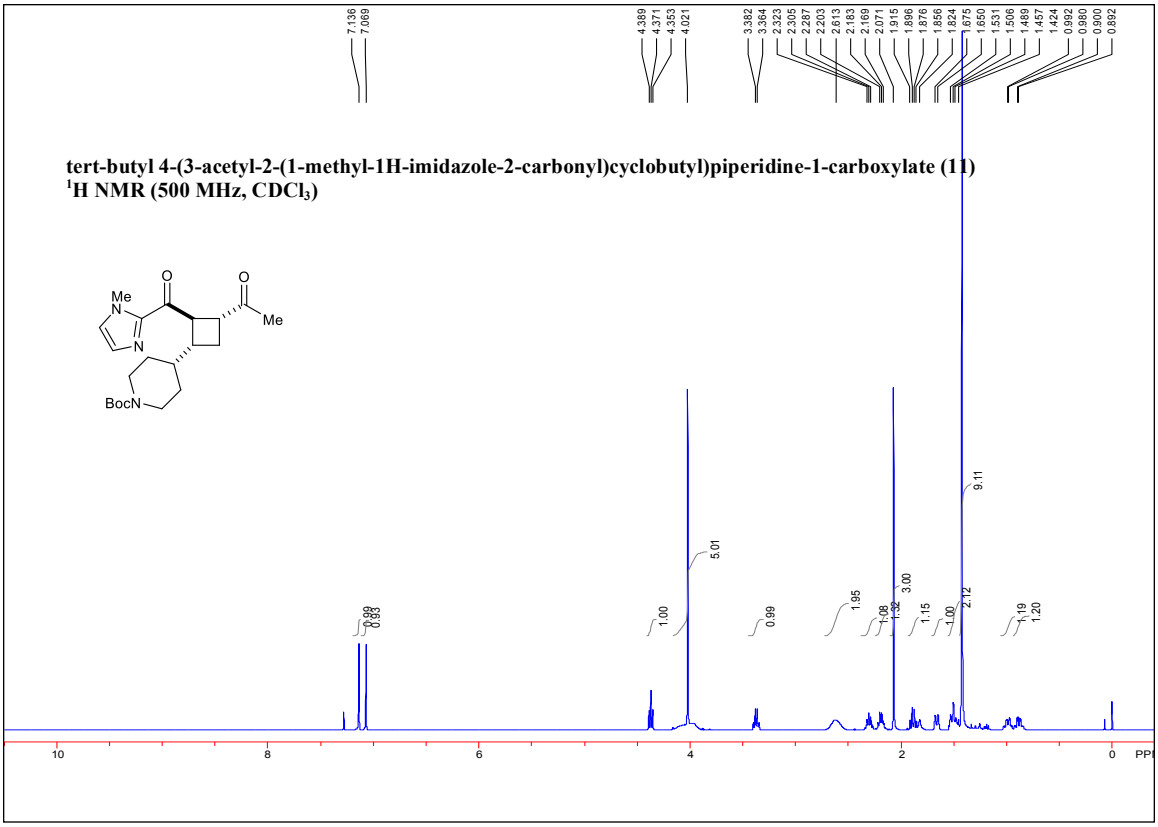

208.190
191.110
142.331
138.546
129.521
128.245
127.516
127.393
127.322
73.055
72.790
46.740
43.572
36.030
35.462
27.481
24.317
1-(3-((benzyloxy)methyl)-2-(1-methyl-1H-imidazole-2-carbonyl)cyclobutyl)ethanone (9)
13C NMR (125.7 MHz, CDCl3)
O
O
N
Me
NMe
OBn
200
150
100
50
0
PPM

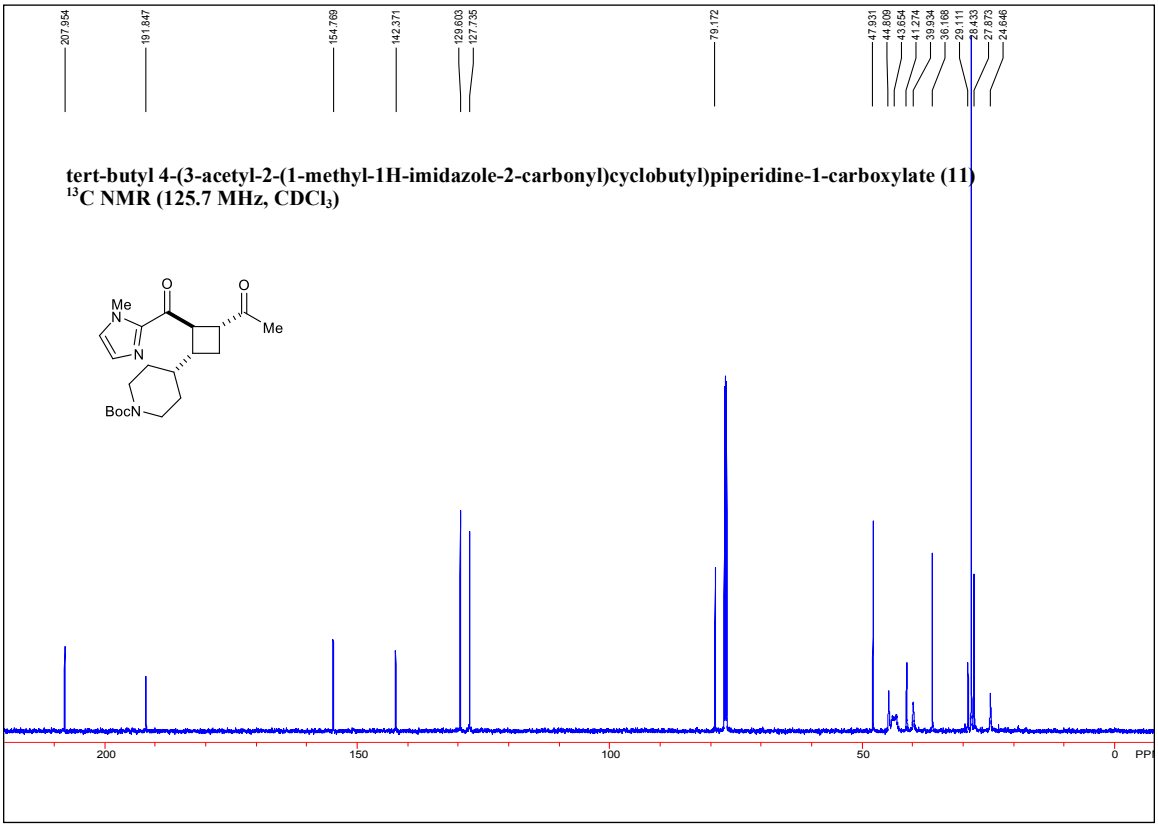


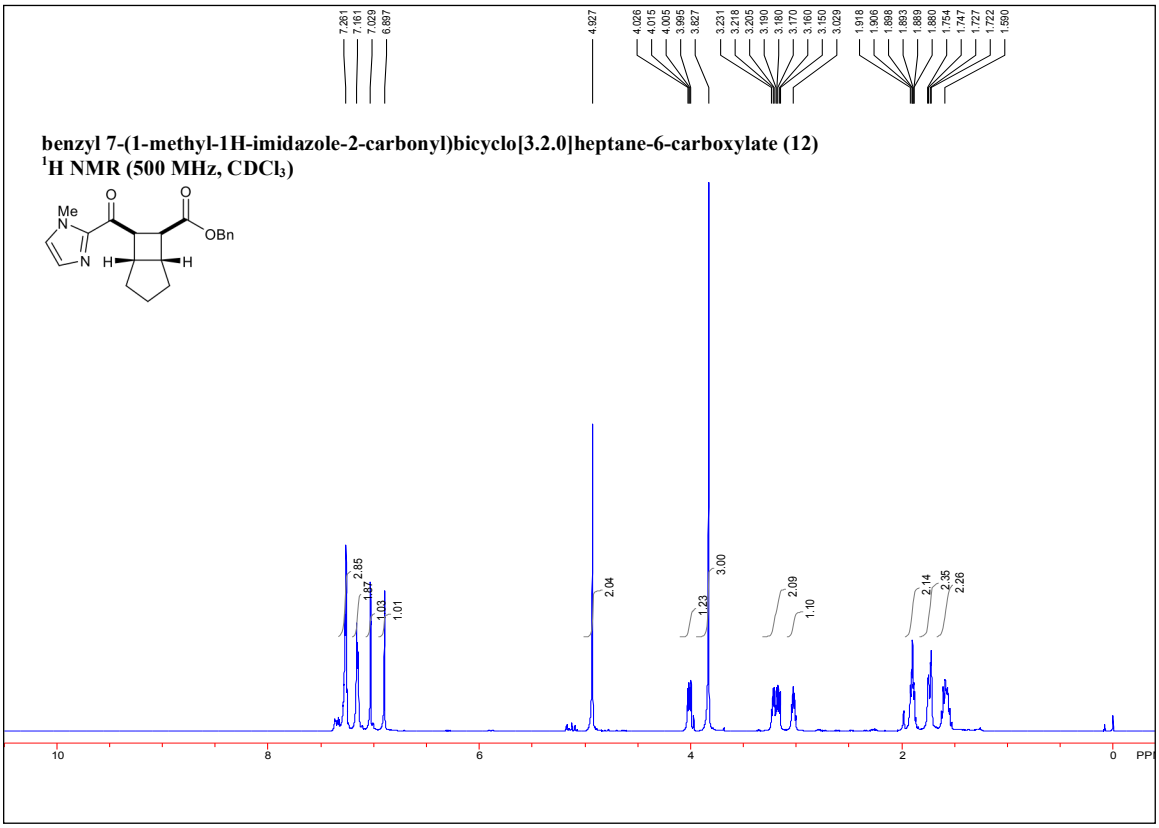
1-(2-(1-Methyl-1*H*-imidazole-2-carbonyl)-3-(3-triisopropylsilanyloxy-propyl)cyclobutyl)ethanone (10)
^{1}H NMR (500 MHz, $CDCl_3$)

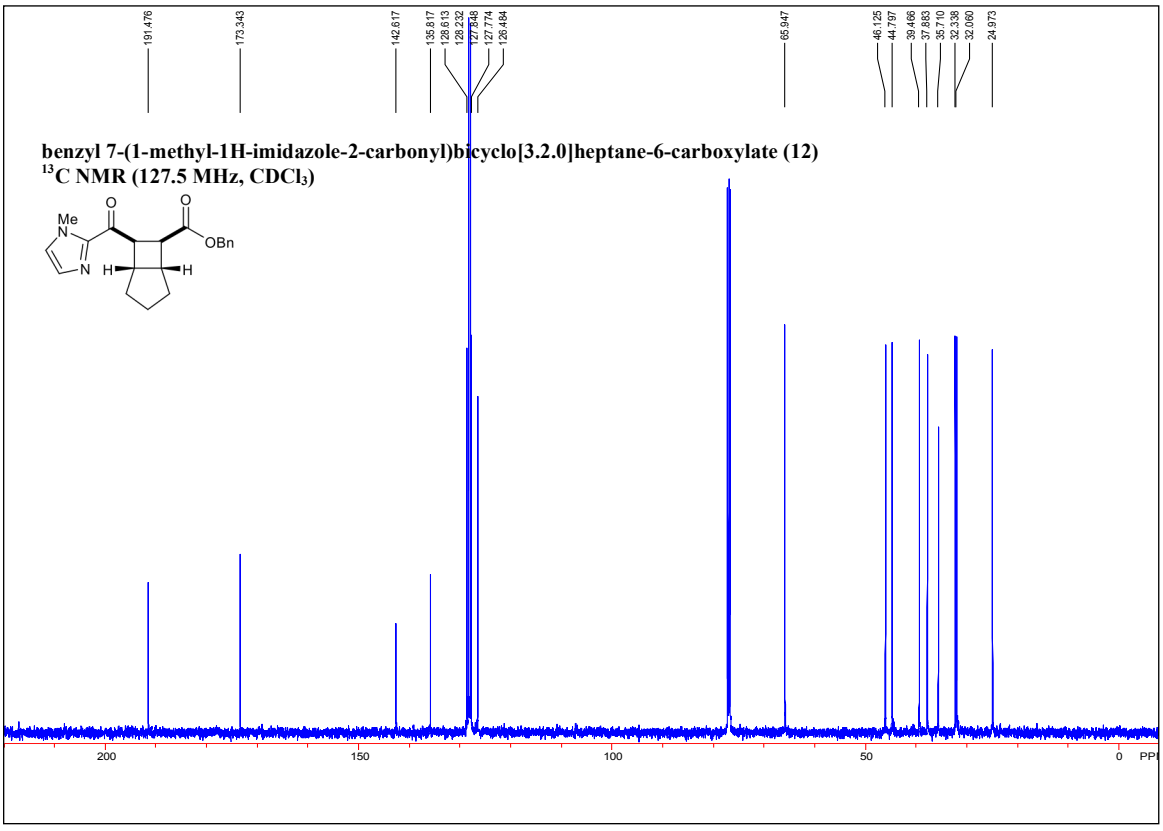


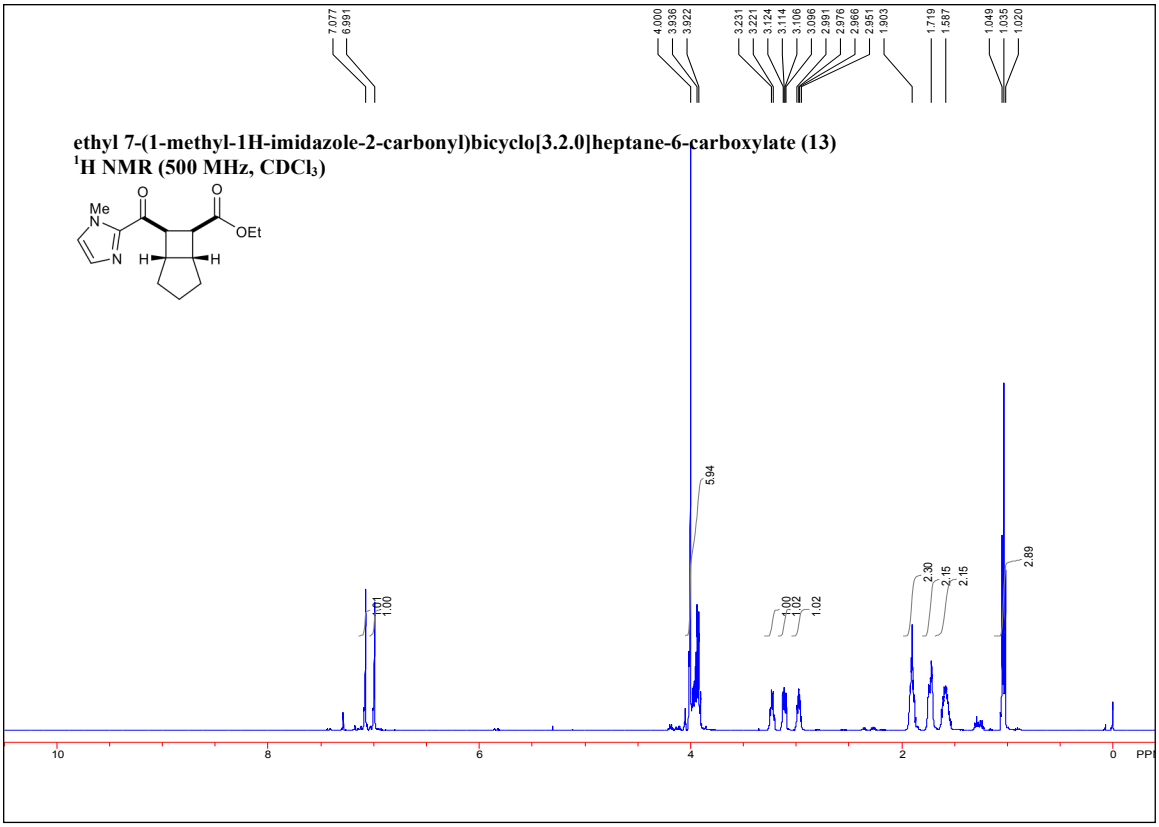
1-(2-(1-Methyl-1*H*-imidazole-2-carbonyl)-3-(3-triisopropylsilanyloxy-propyl)cyclobutyl)ethanone (10)
^{13}C NMR (125.7 MHz, $CDCl_3$)

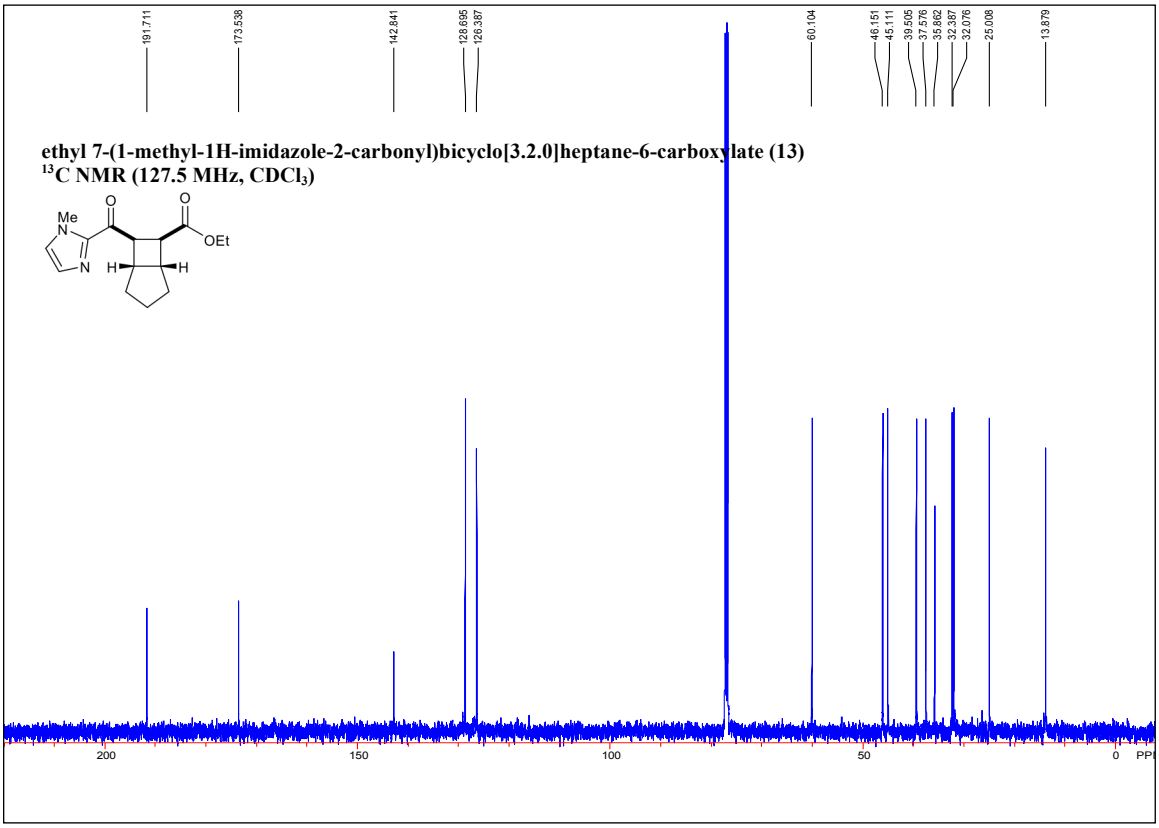

7.136
7.069
4.389
4.371
4.353
4.021
3.382
3.364
2.323
2.305
2.287
2.203
2.183
2.169
2.071
1.915
1.896
1.876
1.856
1.824
1.675
1.650
1.531
1.506
1.489
1.457
1.424
0.992
0.980
0.900
0.892
tert-butyl 4-(3-acetyl-2-(1-methyl-1H-imidazole-2-carbonyl)cyclobutyl)piperidine-1-carboxylate (11)
1H NMR (500 MHz, CDCl3)
Me
N
N
O
O
Me
BocN
9.11
5.01
3.00
2.12
1.95
1.20
1.19
1.15
1.00
1.00
0.99
1.00
0.93
0.89
10
8
6
4
2
0
PPM

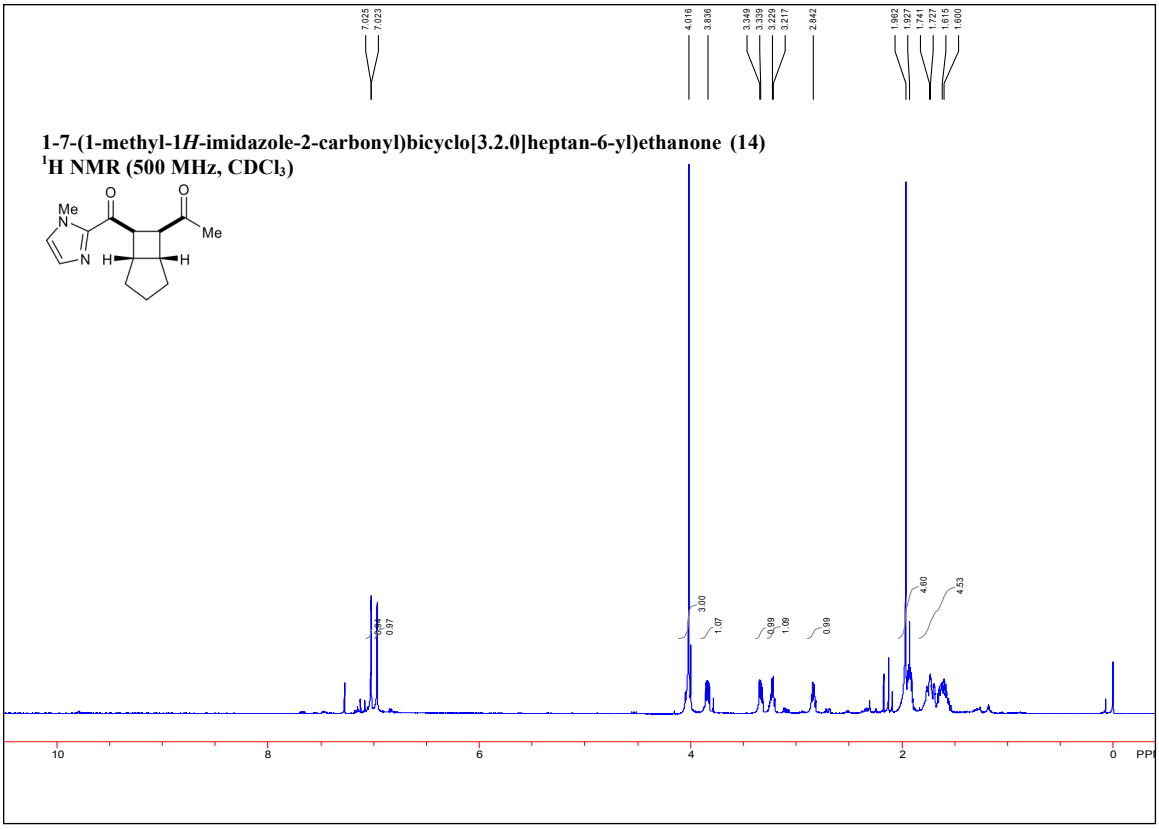

207.954
191.847
154.769
142.371
129.603
127.735
79.172
47.931
44.809
43.654
41.274
39.934
36.168
29.111
28.433
27.873
24.646
tert-butyl 4-(3-acetyl-2-(1-methyl-1H-imidazole-2-carbonyl)cyclobutyl)piperidine-1-carboxylate (11)
13C NMR (125.7 MHz, CDCl3)
Me
N
N
O
O
Me
BocN
200
150
100
50
0
PPM

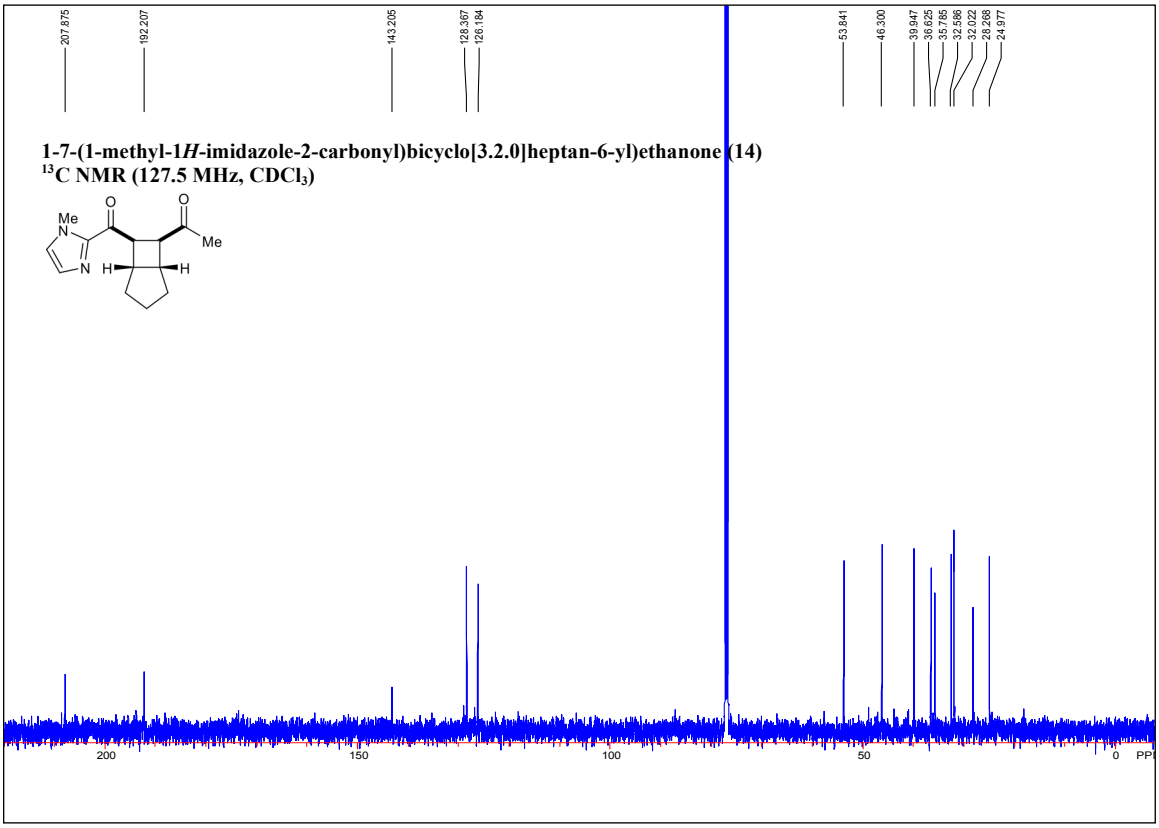

7.281
7.161
7.029
6.897
4.927
4.026
4.015
4.005
3.995
3.827
3.231
3.218
3.205
3.190
3.180
3.170
3.160
3.150
3.029
1.918
1.906
1.898
1.893
1.889
1.880
1.754
1.747
1.727
1.722
1.590
benzyl 7-(1-methyl-1H-imidazole-2-carbonyl)bicyclo[3.2.0]heptane-6-carboxylate (12)
1H NMR (500 MHz, CDCl3)
Me
N
N
O
O
OBn
H
H
2.85
1.87
1.03
1.01
2.04
1.23
3.00
2.09
1.10
2.14
2.35
2.26
10
8
6
4
2
0
PPM

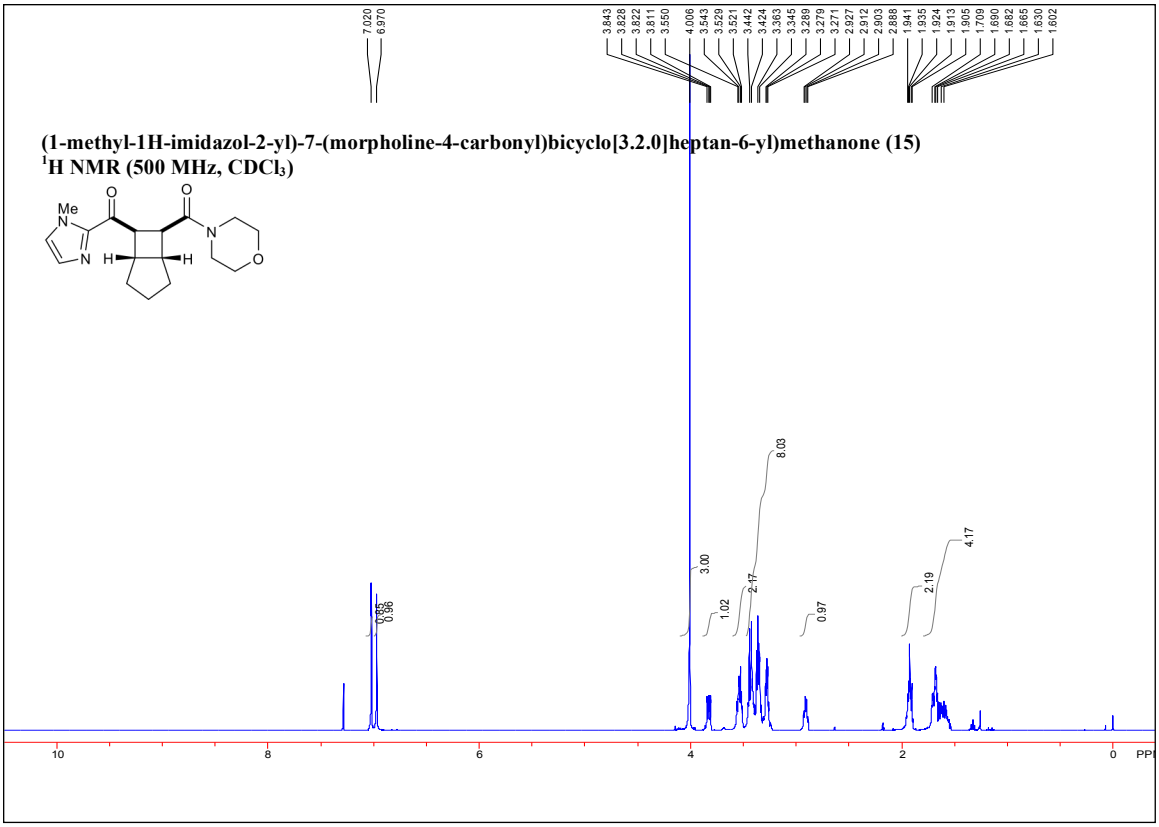

191.476
173.343
142.617
135.817
128.613
128.232
127.948
127.774
126.484
65.947
46.125
44.797
39.466
37.883
35.710
32.338
32.060
24.973
benzyl 7-(1-methyl-1H-imidazole-2-carbonyl)bicyclo[3.2.0]heptane-6-carboxylate (12)
13C NMR (127.5 MHz, CDCl3)
Me
N
N
O
O
OBn
H
H
200
150
100
50
0
PPM

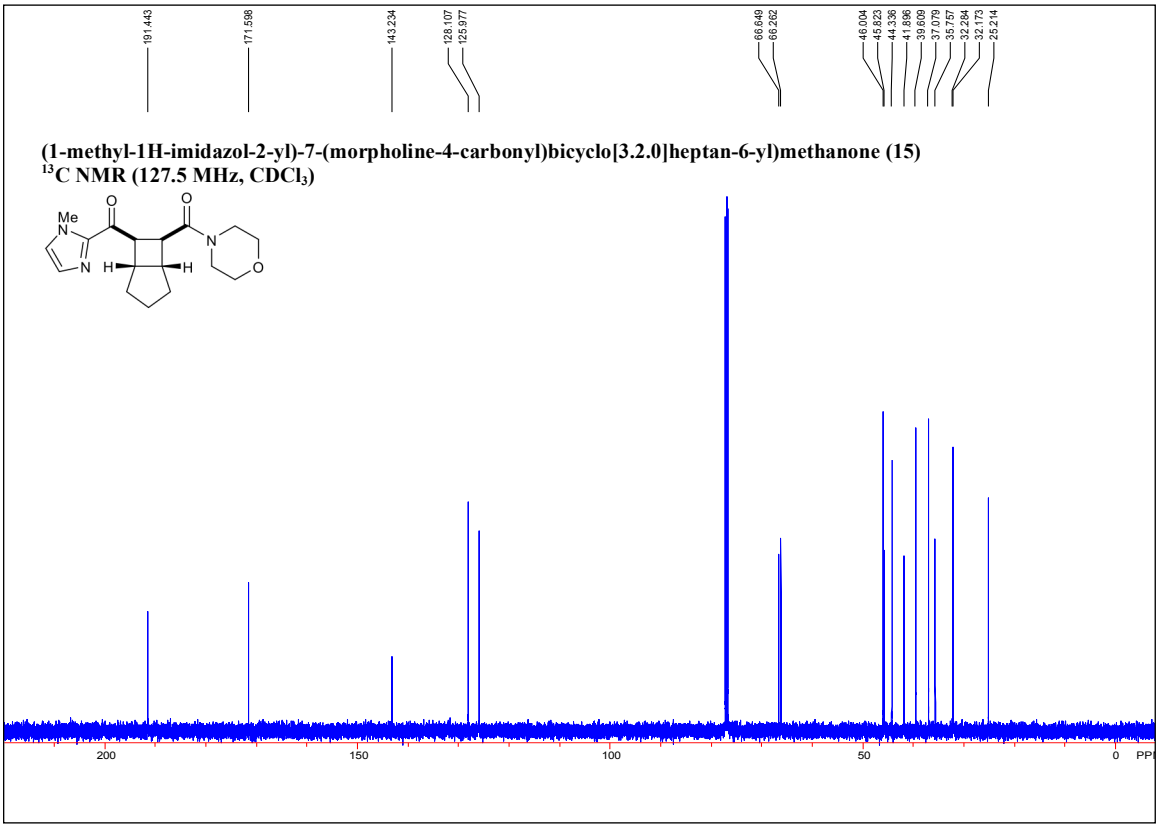

7.077
6.991
4.000
3.936
3.922
3.231
3.221
3.124
3.114
3.106
3.096
2.991
2.976
2.966
2.951
1.903
1.719
1.587
1.049
1.035
1.020
ethyl 7-(1-methyl-1H-imidazole-2-carbonyl)bicyclo[3.2.0]heptane-6-carboxylate (13)
1H NMR (500 MHz, CDCl3)
Me
N
N
O
O
OEt
H
H
5.94
1.01
1.00
1.00
1.02
1.02
2.30
2.15
2.15
2.89
10
8
6
4
2
0
PPM

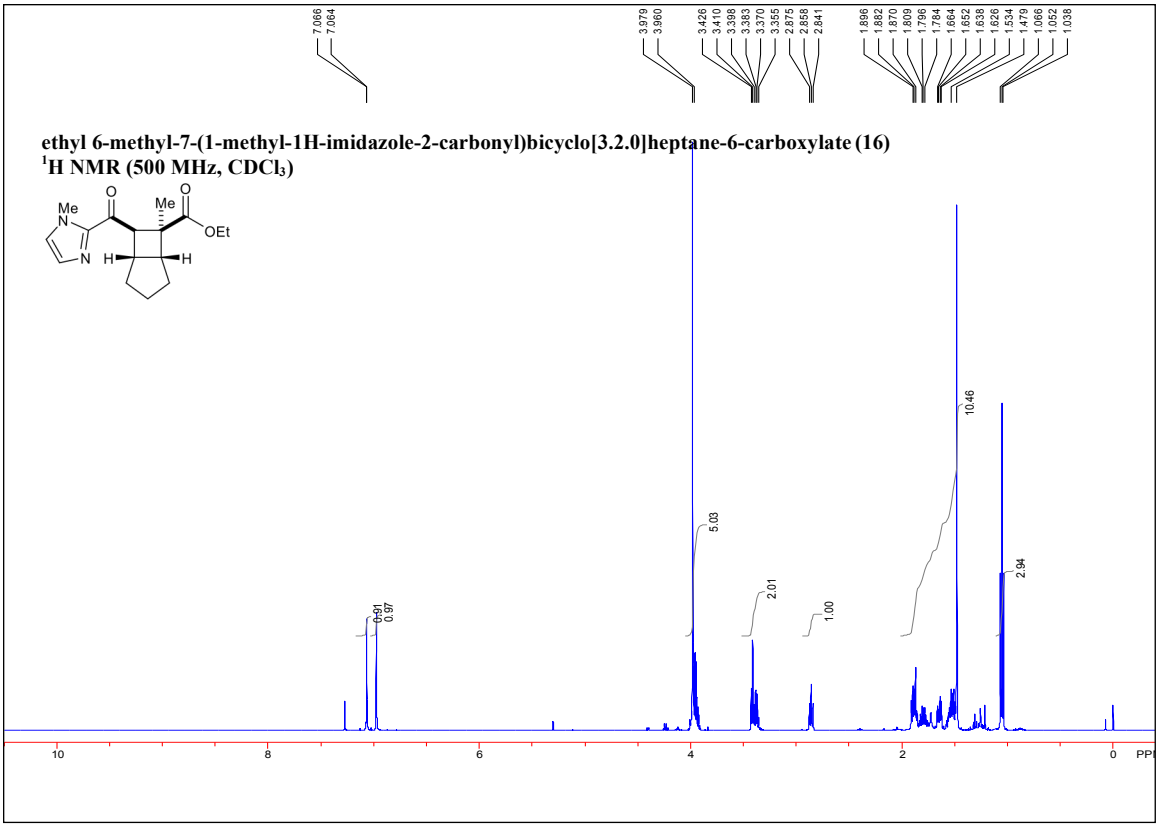

191.711
173.538
142.841
128.695
126.387
60.104
46.151
45.111
39.505
37.576
35.862
32.387
32.076
25.008
13.879
ethyl 7-(1-methyl-1H-imidazole-2-carbonyl)bicyclo[3.2.0]heptane-6-carboxylate (13)
13C NMR (127.5 MHz, CDCl3)
Me
N
N
O
O
OEt
H
H
200
150
100
50
0
PPM

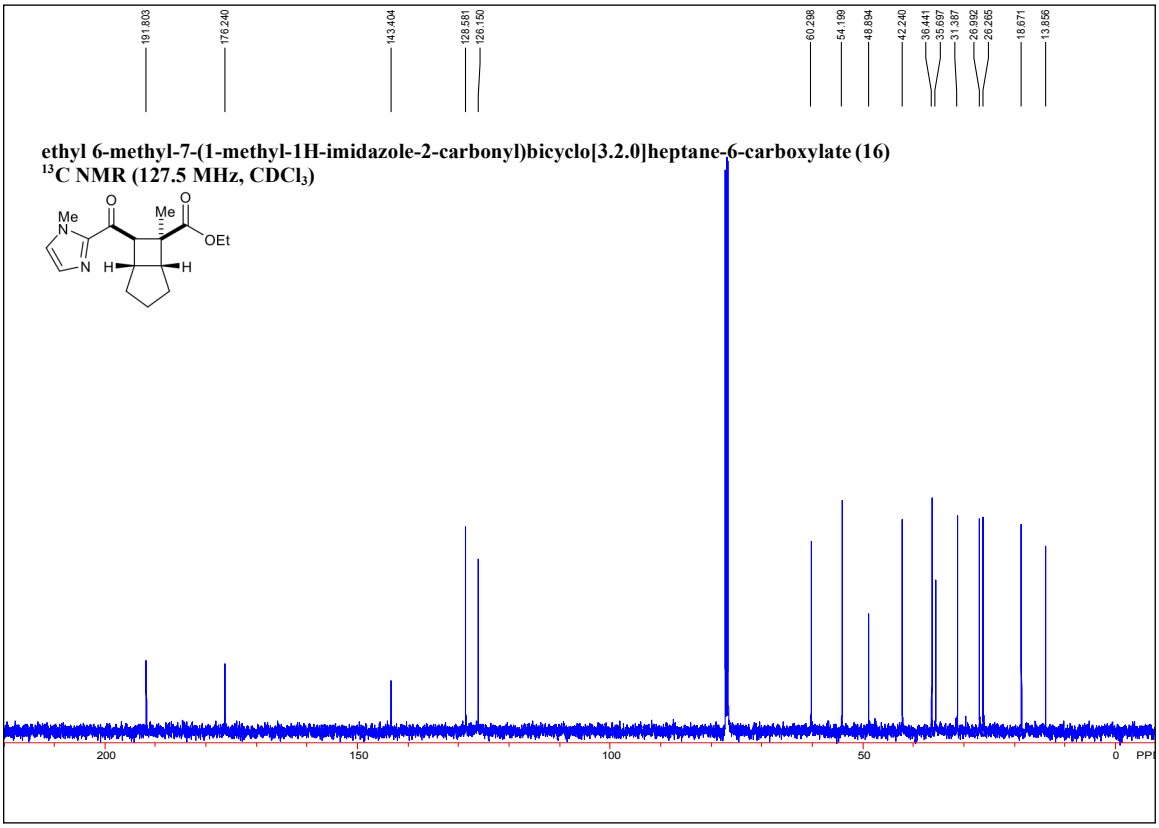

7.025
7.023
4.016
3.836
3.349
3.339
3.229
3.217
2.842
1.962
1.927
1.741
1.727
1.615
1.600
1-7-(1-methyl-1H-imidazole-2-carbonyl)bicyclo[3.2.0]heptan-6-yl)ethanone (14)
1H NMR (500 MHz, CDCl3)
Me
N
N
O
O
Me
H
H
3.00
1.07
0.99
1.09
0.99
4.60
4.53
10
8
6
4
2
0
PPM

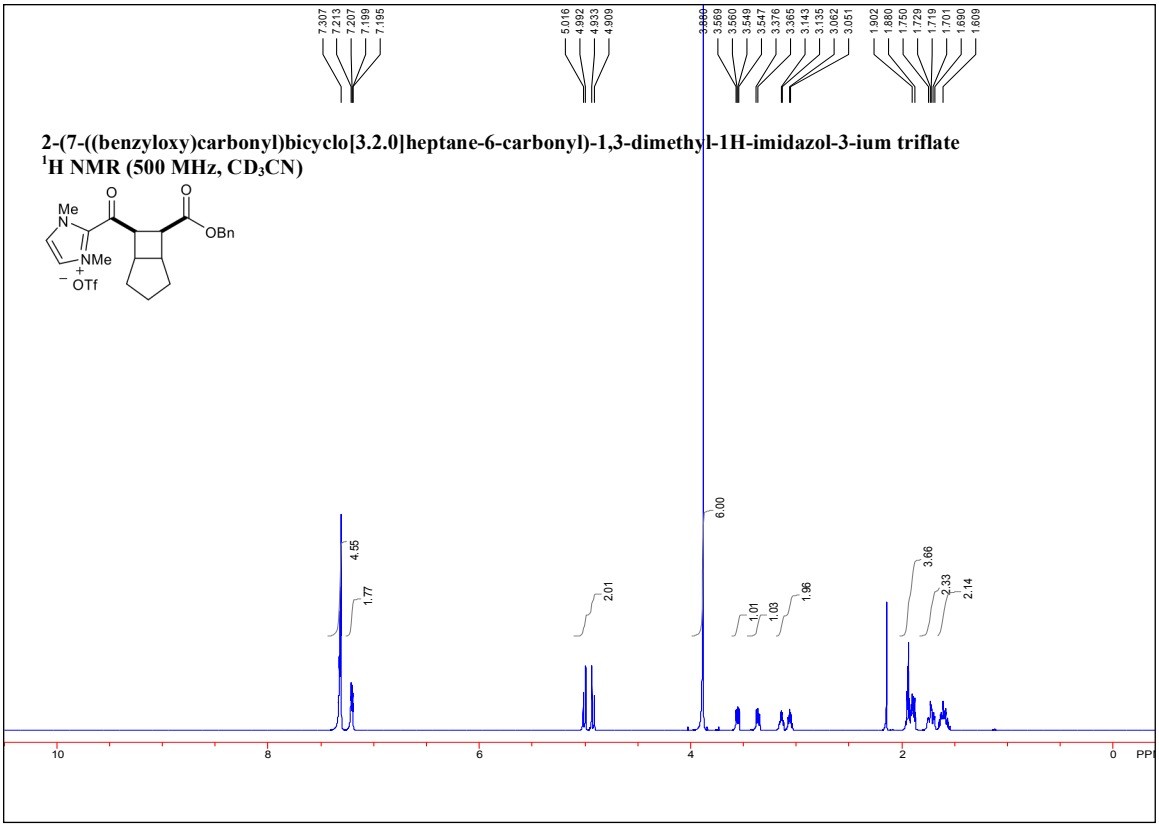

207.875
192.207
143.205
128.367
126.184
53.841
46.300
39.947
36.625
35.785
32.586
32.022
28.268
24.977
1-7-(1-methyl-1H-imidazole-2-carbonyl)bicyclo[3.2.0]heptan-6-yl)ethanone (14)
13C NMR (127.5 MHz, CDCl3)
Me
N
N
O
O
Me
H
H
200
150
100
50
0
PPM

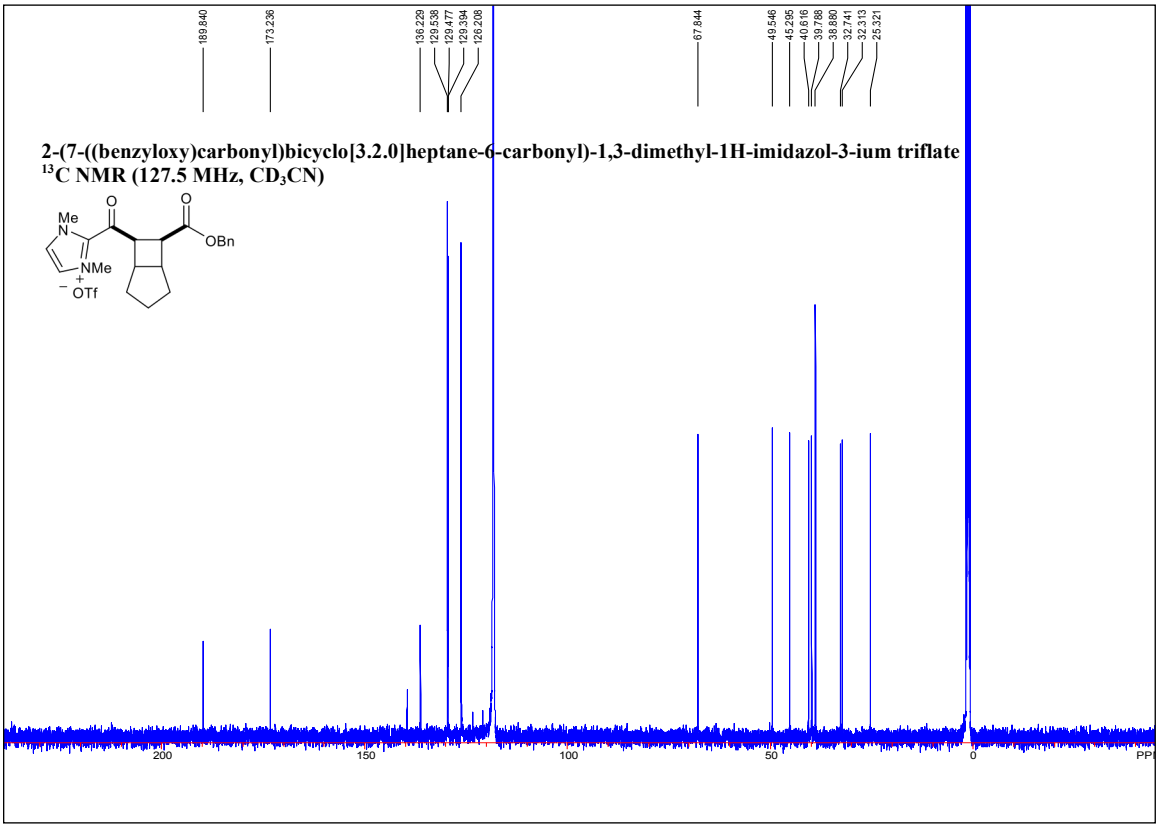

7.020
6.970
3.843
3.828
3.822
3.811
3.550
4.006
3.543
3.529
3.521
3.442
3.424
3.363
3.345
3.289
3.279
3.271
2.927
2.912
2.903
2.888
1.941
1.935
1.924
1.913
1.905
1.709
1.690
1.682
1.665
1.630
1.602
(1-methyl-1H-imidazol-2-yl)-7-(morpholine-4-carbonyl)bicyclo[3.2.0]heptan-6-yl)methanone (15)
1H NMR (500 MHz, CDCl3)
Me
N
N
O
O
N
O
H
H
8.03
4.17
3.00
2.17
2.19
1.02
0.97
0.86
0.86
10
8
6
4
2
0
PPM

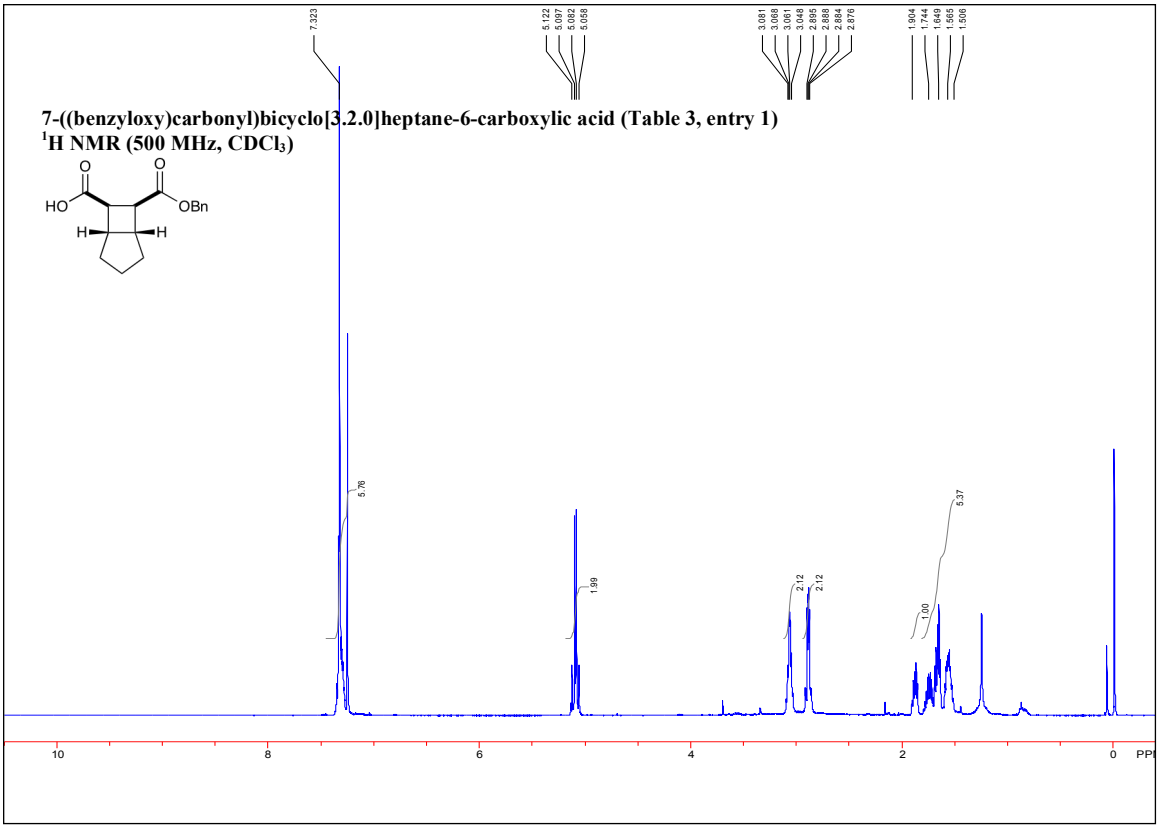

191.443
171.598
143.234
128.107
125.977
66.649
66.262
46.004
45.823
44.336
41.896
39.609
37.079
35.757
32.284
32.173
25.214
(1-methyl-1H-imidazol-2-yl)-7-(morpholine-4-carbonyl)bicyclo[3.2.0]heptan-6-yl)methanone (15)
13C NMR (127.5 MHz, CDCl3)
Me
N
N
O
O
N
O
H
H
200
150
100
50
0
PPM


7.066
7.064
3.979
3.960
3.426
3.410
3.398
3.383
3.370
3.355
2.875
2.858
2.841
1.896
1.882
1.870
1.809
1.796
1.784
1.664
1.652
1.638
1.626
1.534
1.479
1.066
1.052
1.038
ethyl 6-methyl-7-(1-methyl-1H-imidazole-2-carbonyl)bicyclo[3.2.0]heptane-6-carboxylate (16)
1H NMR (500 MHz, CDCl3)
Me
O
Me
O
N
N
OEt
H
H
10.46
5.03
2.94
2.01
1.00
0.91
0.97
10
8
6
4
2
0
PPM

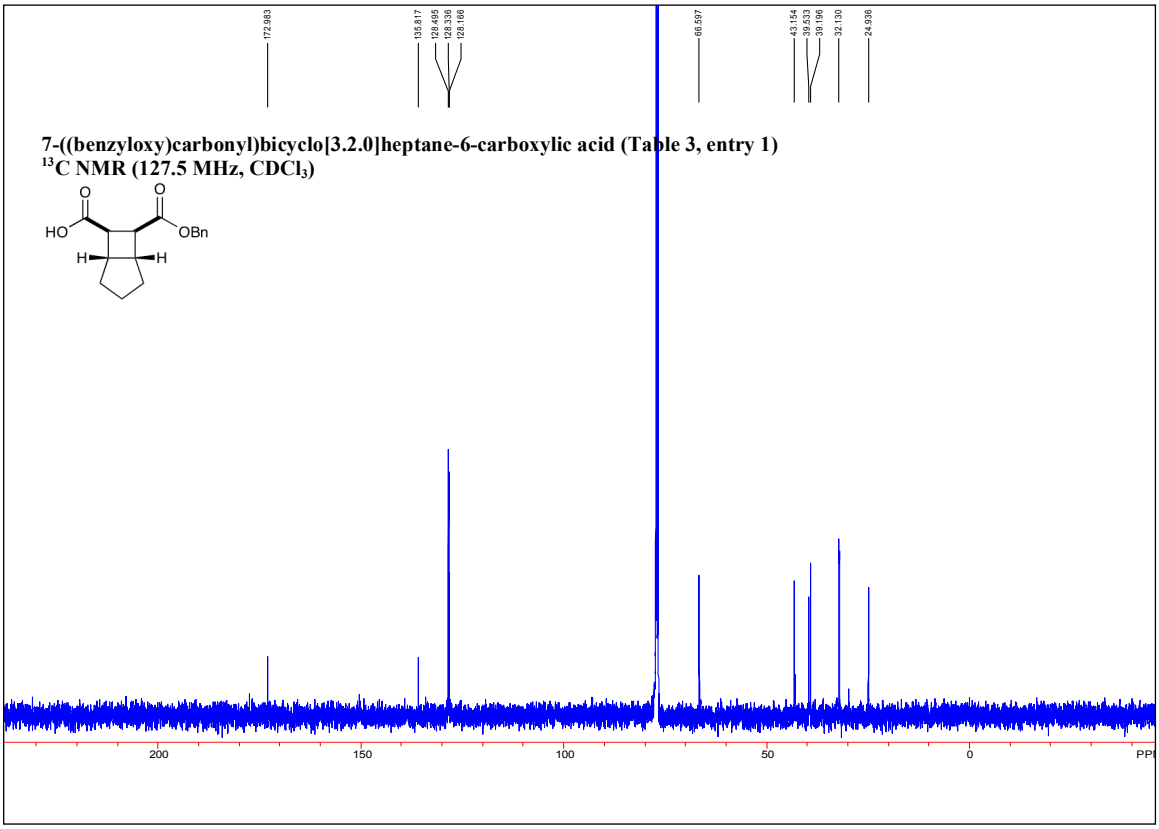

191.803
176.240
143.404
128.581
126.150
60.298
54.199
48.894
42.240
36.441
35.697
31.387
26.932
26.265
18.671
13.856
ethyl 6-methyl-7-(1-methyl-1H-imidazole-2-carbonyl)bicyclo[3.2.0]heptane-6-carboxylate (16)
13C NMR (127.5 MHz, CDCl3)
Me
O
Me
O
N
N
OEt
H
H
200
150
100
50
0
PPM


7.307
7.213
7.207
7.199
7.195
5.016
4.992
4.933
4.909
3.880
3.569
3.560
3.549
3.547
3.376
3.365
3.143
3.135
3.062
3.051
1.902
1.880
1.750
1.729
1.719
1.701
1.690
1.609
2-(7-((benzyloxy)carbonyl)bicyclo[3.2.0]heptane-6-carbonyl)-1,3-dimethyl-1H-imidazol-3-ium triflate
1H NMR (500 MHz, CD3CN)
Me
N
NMe
OTf
OBn
4.55
1.77
2.01
6.00
1.01
1.03
1.96
3.66
2.33
2.14
10
8
6
4
2
0
PPM

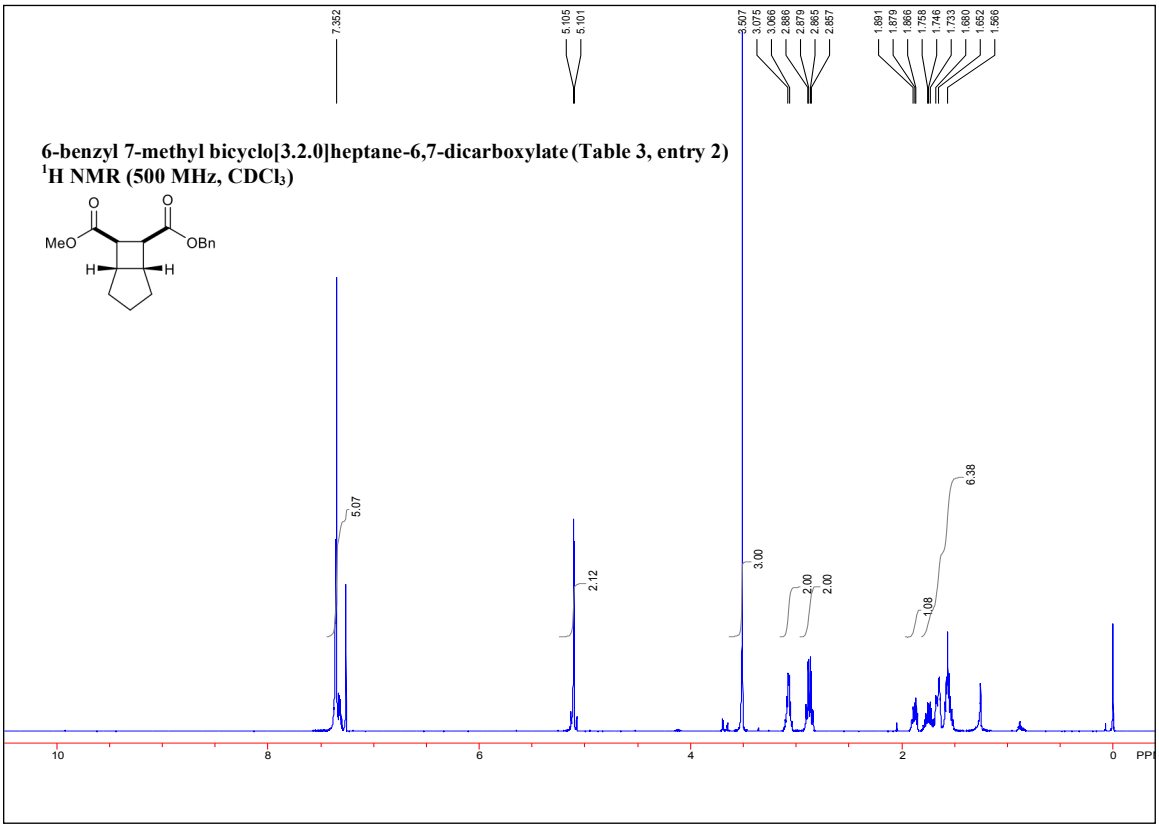

189.840
173.236
136.229
129.538
129.477
129.394
126.208
67.844
49.546
45.295
40.616
39.788
38.880
32.741
32.213
25.321
2-(7-((benzyloxy)carbonyl)bicyclo[3.2.0]heptane-6-carbonyl)-1,3-dimethyl-1H-imidazol-3-ium triflate
13C NMR (127.5 MHz, CD3CN)
Me
N
NMe
OTf
OBn
200
150
100
50
0
PPM

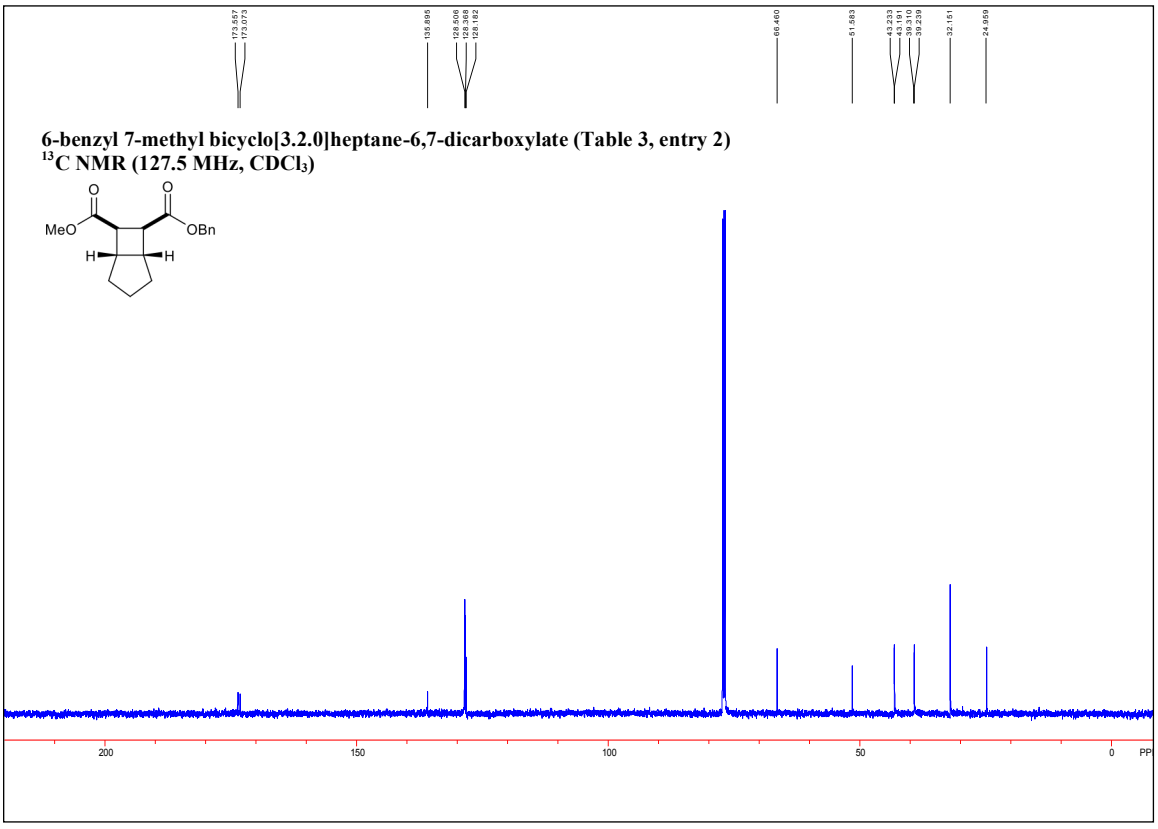


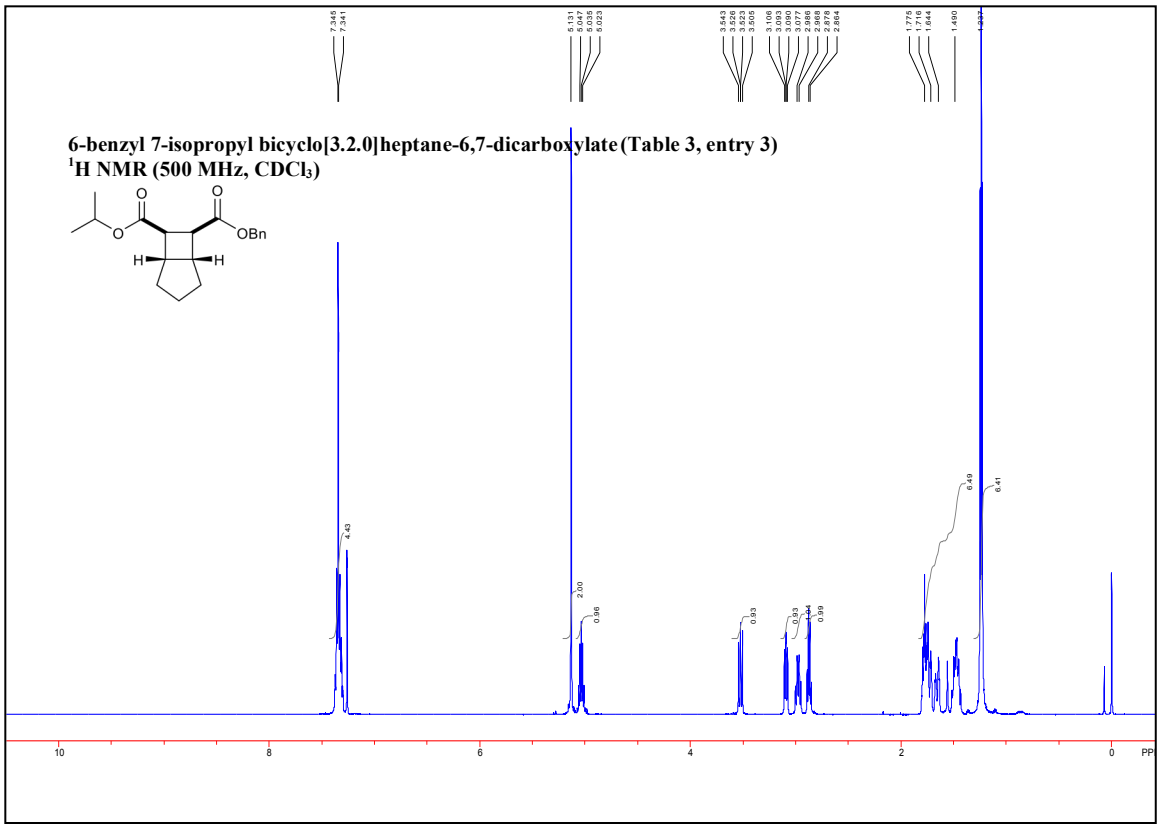
7-((benzyloxy)carbonyl)bicyclo[3.2.0]heptane-6-carboxylic acid (Table 3, entry 1)
^{1}H NMR (500 MHz, $CDCl_3$)

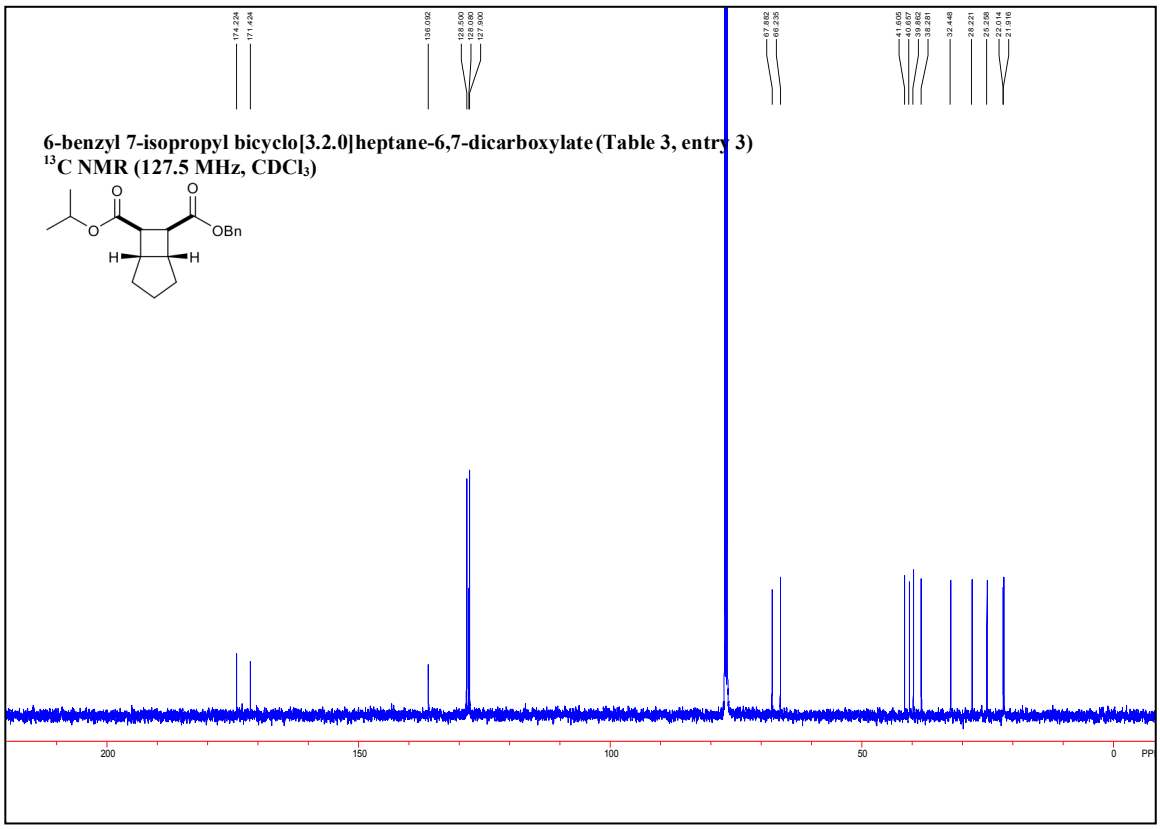


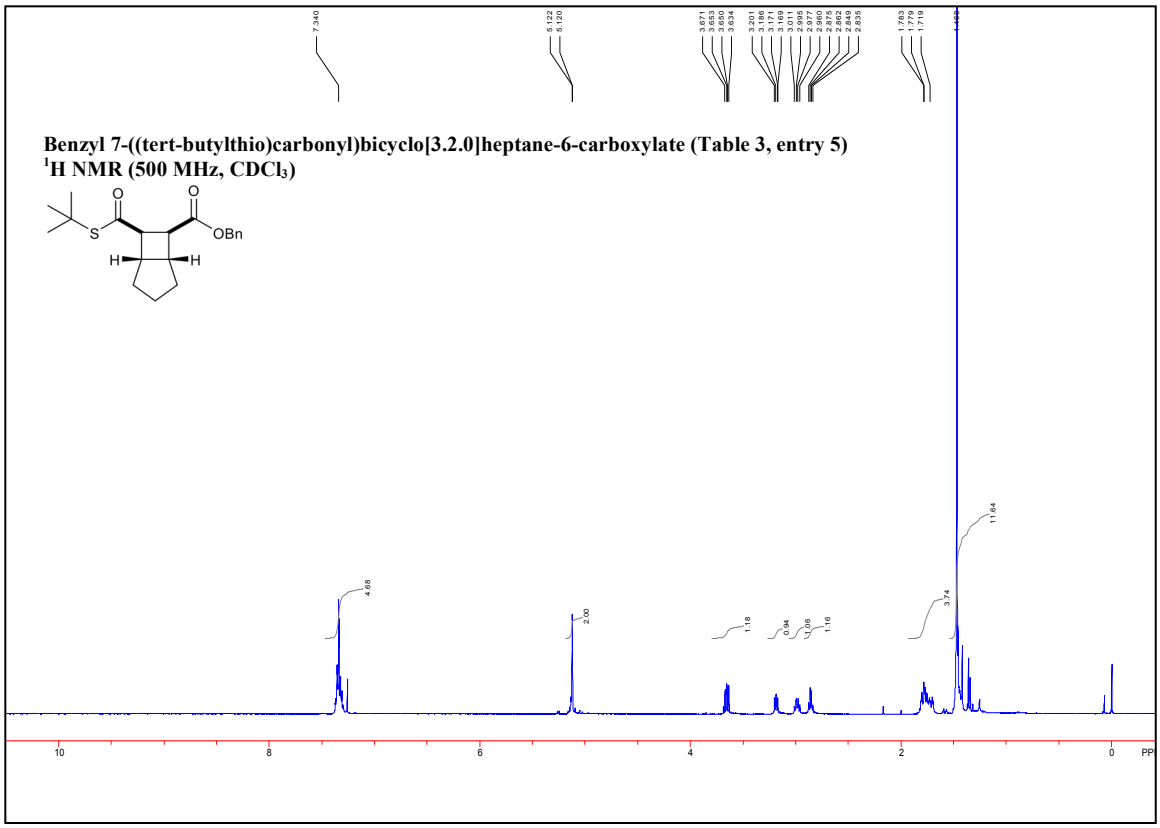
7-((benzyloxy)carbonyl)bicyclo[3.2.0]heptane-6-carboxylic acid (Table 3, entry 1)
^{13}C NMR (127.5 MHz, $CDCl_3$)

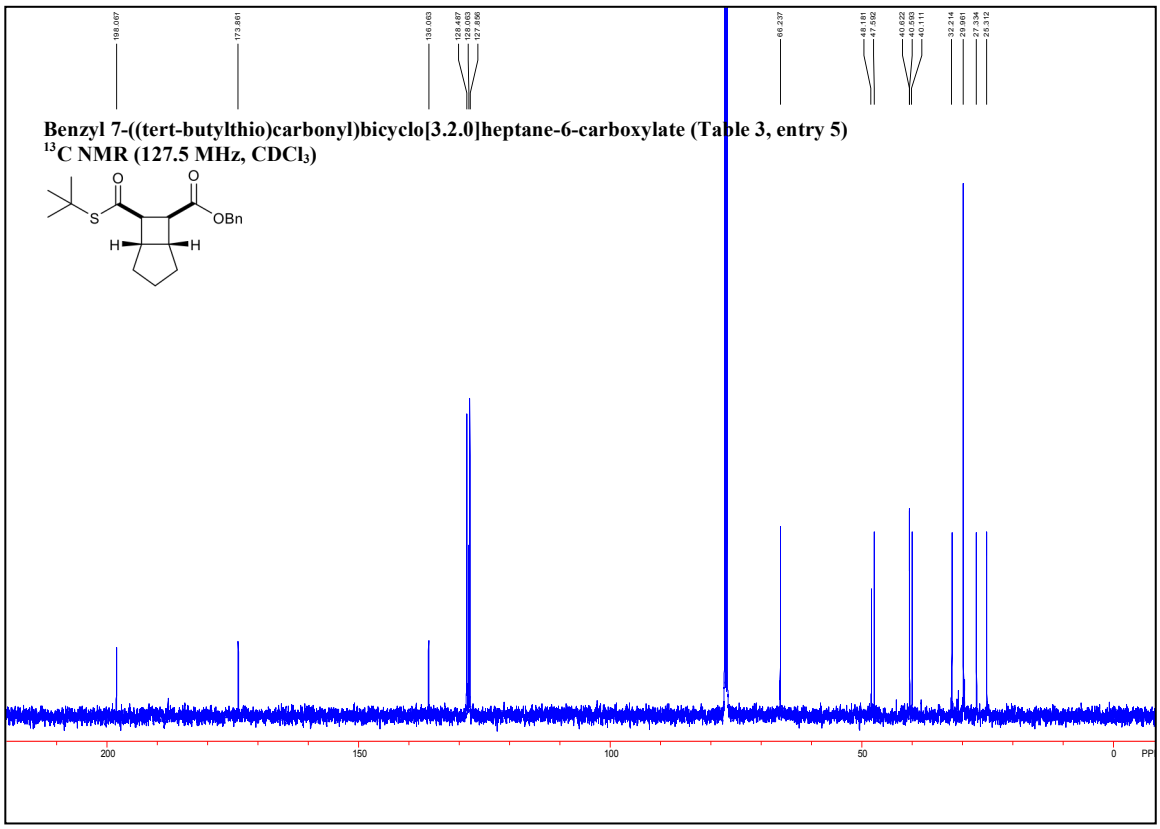

7.352
5.105
5.101
3.507
3.075
3.066
2.886
2.879
2.865
2.857
1.891
1.879
1.866
1.758
1.746
1.733
1.680
1.652
1.566
6-benzyl 7-methyl bicyclo[3.2.0]heptane-6,7-dicarboxylate (Table 3, entry 2)
1H NMR (500 MHz, CDCl3)
MeO
OBn
H
H
5.07
2.12
3.00
2.00
2.00
1.08
6.38
10
8
6
4
2
0
PPM

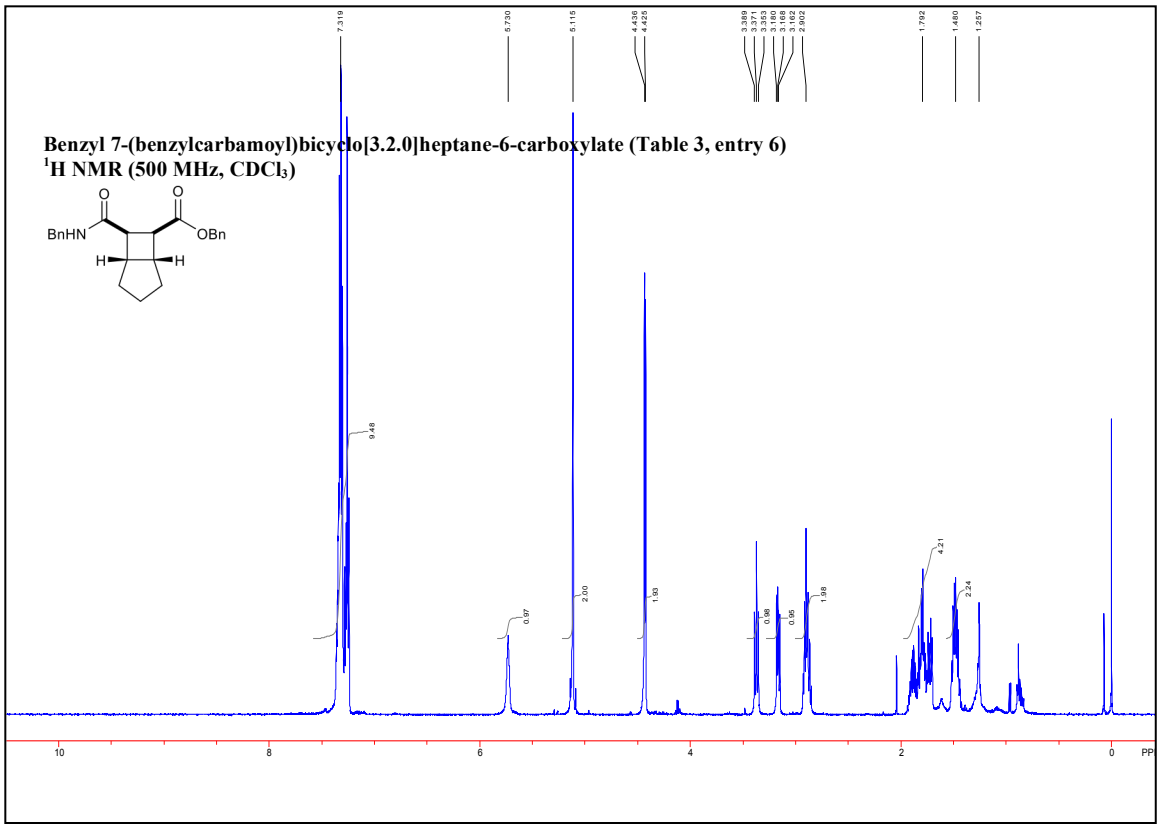

173.657
173.073
135.895
128.506
128.368
128.182
66.460
51.863
43.233
43.191
39.310
39.229
32.151
24.959
6-benzyl 7-methyl bicyclo[3.2.0]heptane-6,7-dicarboxylate (Table 3, entry 2)
13C NMR (127.5 MHz, CDCl3)
MeO
OBn
H
H
200
150
100
50
0
PPM

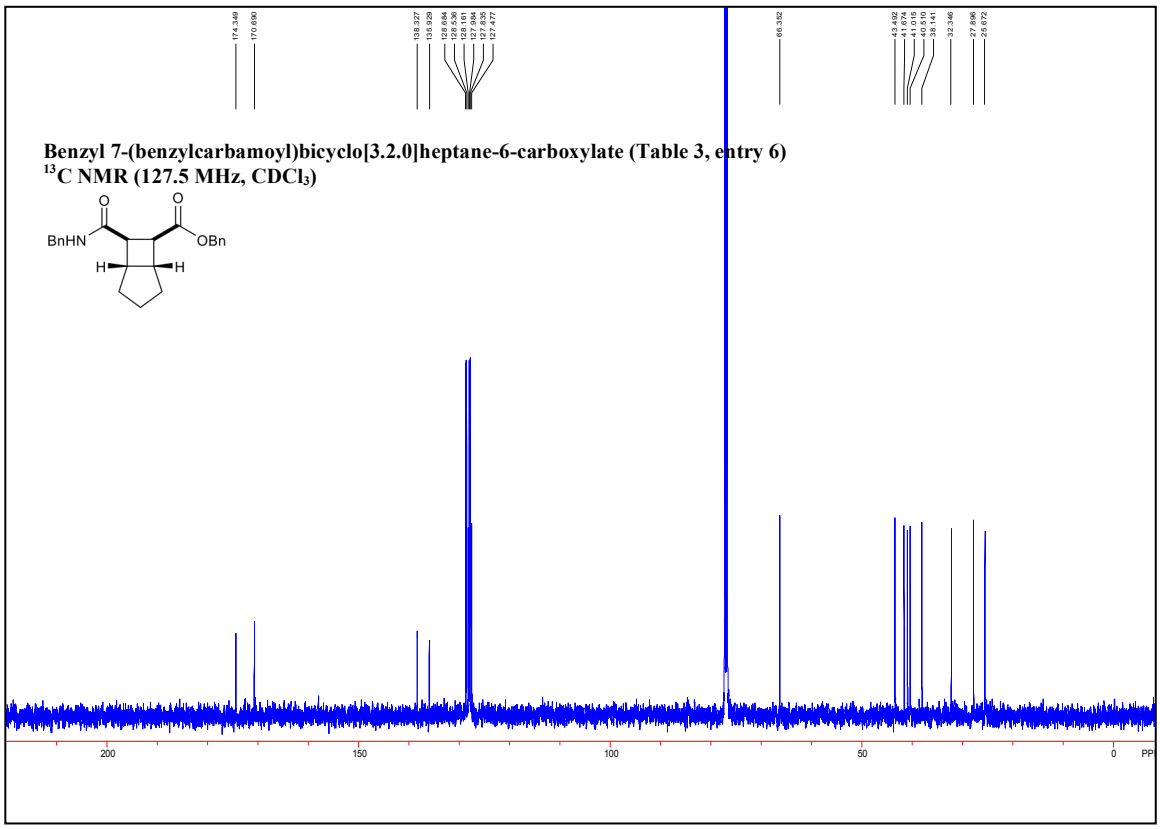

6-benzyl 7-isopropyl bicyclo[3.2.0]heptane-6,7-dicarboxylate (Table 3, entry 3)
^{1}H NMR (500 MHz, CDCl$_3$)
O
O
O
OBn
H
H
7.345
7.341
5.131
5.047
5.035
5.023
3.543
3.526
3.508
3.503
3.108
3.093
3.090
3.077
2.968
2.879
2.864
1.775
1.716
1.644
1.490
1.262
2.49
2.00
0.96
0.93
0.93
0.94
0.99
6.49
6.41
10
8
6
4
2
0
PPM

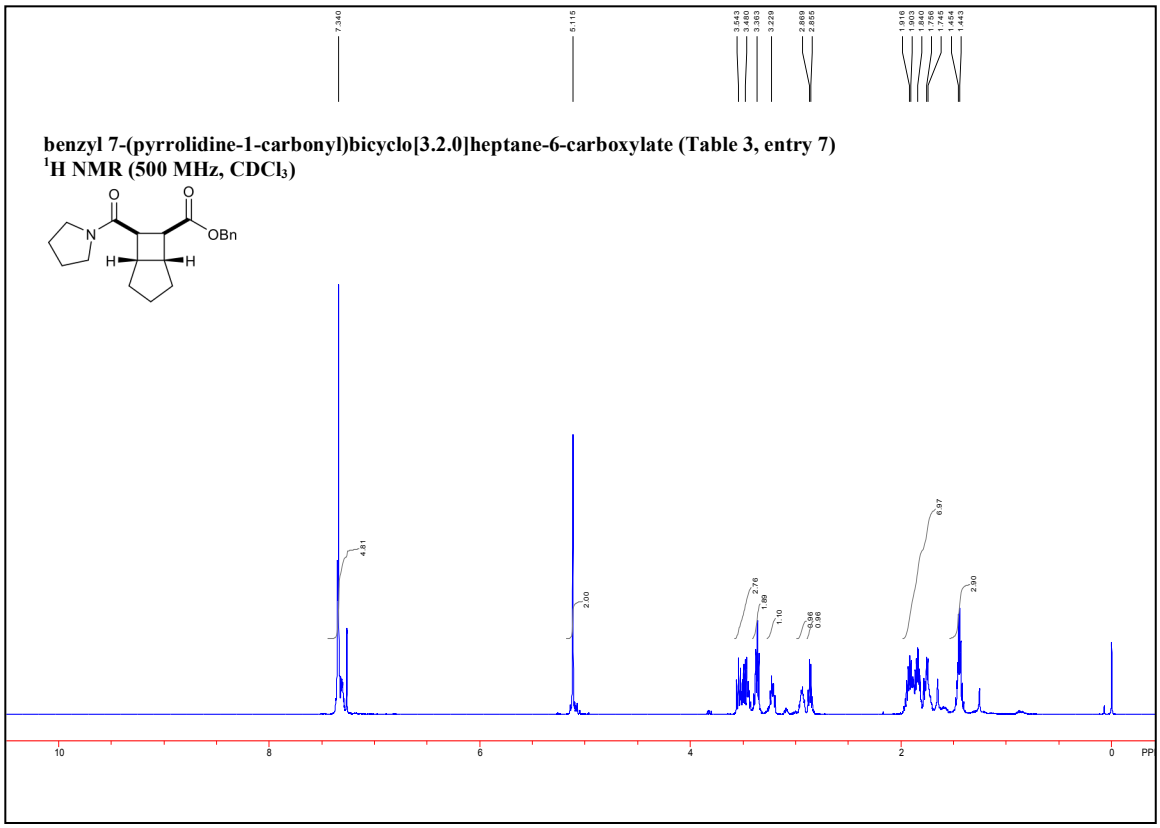

6-benzyl 7-isopropyl bicyclo[3.2.0]heptane-6,7-dicarboxylate (Table 3, entry 3)
^{13}C NMR (127.5 MHz, CDCl$_3$)
O
O
O
OBn
H
H
174.224
171.424
136.092
128.500
128.090
127.900
67.882
66.235
41.605
40.657
39.882
38.281
32.448
28.221
25.298
22.014
21.916
200
150
100
50
0
PPM

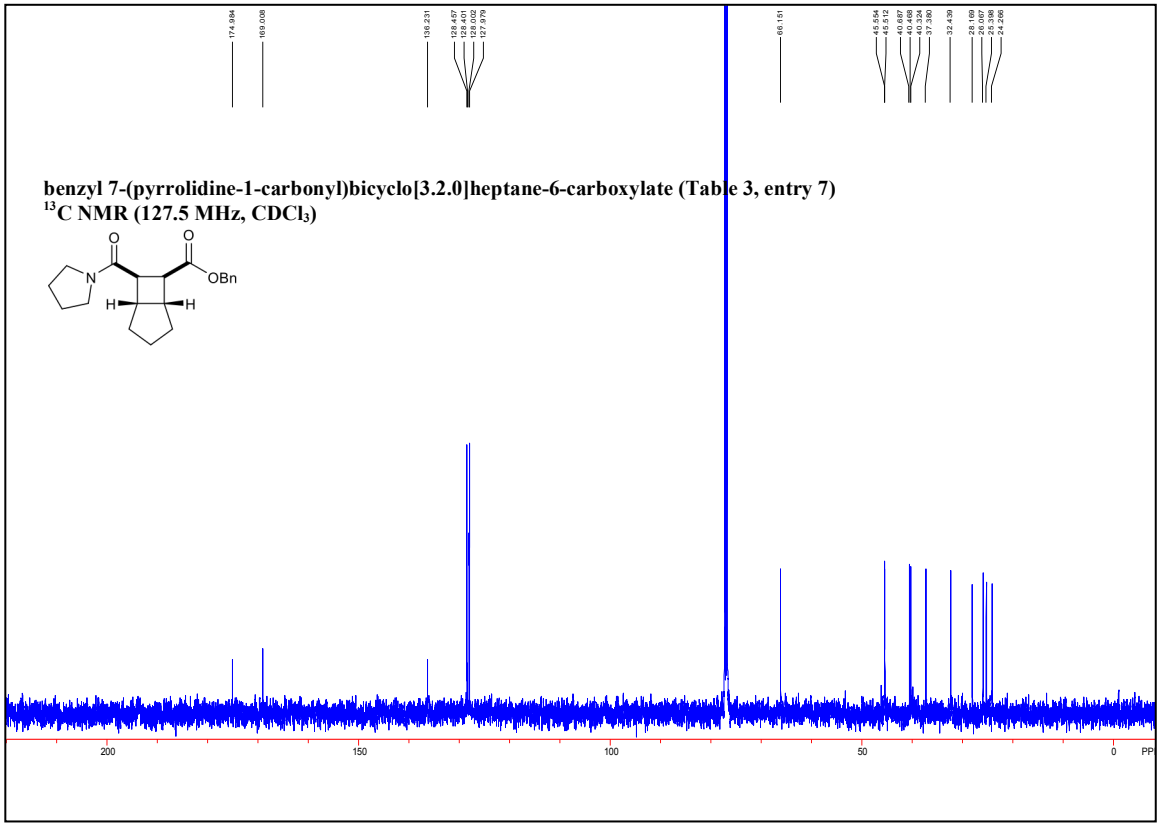

Benzyl 7-((tert-butylthio)carbonyl)bicyclo[3.2.0]heptane-6-carboxylate (Table 3, entry 5)
1H NMR (500 MHz, CDCl3)
O
O
S
OBn
H
H
7.340
5.122
5.120
3.671
3.653
3.650
3.634
3.201
3.186
3.171
3.156
3.011
2.995
2.977
2.960
2.875
2.857
2.849
2.835
1.793
1.779
1.719
1.464
4.69
2.00
1.18
0.94
1.06
1.16
3.74
11.64
10
8
6
4
2
0
PPM


Benzyl 7-((tert-butylthio)carbonyl)bicyclo[3.2.0]heptane-6-carboxylate (Table 3, entry 5)
13C NMR (127.5 MHz, CDCl3)
O
O
S
OBn
H
H
198.087
173.861
136.083
128.487
128.083
127.896
66.237
48.181
47.592
40.622
40.500
40.111
32.214
29.991
27.334
25.312
200
150
100
50
0
PPM


Benzyl 7-(benzylcarbamoyl)bicyclo[3.2.0]heptane-6-carboxylate (Table 3, entry 6)
^{1}H NMR (500 MHz, CDCl$_3$)
BnHN
OBn
H
H
7.319
5.730
5.116
4.436
4.425
3.389
3.371
3.353
3.190
3.168
3.162
2.902
1.792
1.490
1.257
9.48
0.97
2.00
1.93
0.98
0.95
1.98
4.21
2.24
10
8
6
4
2
0
PPM


Benzyl 7-(benzylcarbamoyl)bicyclo[3.2.0]heptane-6-carboxylate (Table 3, entry 6)
^{13}C NMR (127.5 MHz, CDCl$_3$)
BnHN
OBn
H
H
174.349
170.690
138.327
135.929
128.684
128.536
128.161
127.984
127.835
127.477
66.352
43.492
41.674
41.016
40.858
38.141
32.346
27.896
25.672
200
150
100
50
0
PPM


benzyl 7-(pyrrolidine-1-carbonyl)bicyclo[3.2.0]heptane-6-carboxylate (Table 3, entry 7)
^{1}H NMR (500 MHz, CDCl$_3$)
OBn
H
H
7.340
5.116
3.543
3.480
3.363
3.229
2.869
2.855
1.916
1.903
1.850
1.756
1.745
1.454
1.443
4.91
2.00
2.76
1.89
1.10
0.98
0.96
6.97
2.90
10
8
6
4
2
0
PPM


benzyl 7-(pyrrolidine-1-carbonyl)bicyclo[3.2.0]heptane-6-carboxylate (Table 3, entry 7)
^{13}C NMR (127.5 MHz, CDCl$_3$)
OBn
H
H
174.984
169.008
136.231
128.457
128.401
128.052
127.979
66.151
45.554
45.512
40.687
40.498
40.334
37.380
32.439
28.169
26.097
25.398
24.266
200
150
100
50
0
PPM