Supporting Information

Rhodium(III)-Catalyzed Decarboxylative C-H Functionalization of Isoxazoles with Alkynes, Alkenes and Sulfoxonium Ylides

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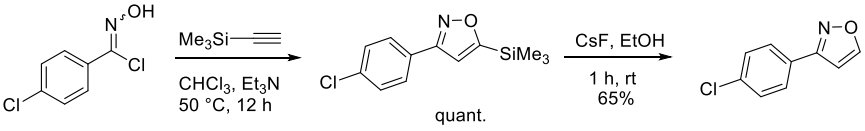
1. General Techniques

NMR spectra were recorded on a Bruker biospin AVANCE Ⅱ (400 MHz for ^{1}H, 100 MHz for ^{13}C) or a Bruker biospin AVANCE Ⅲ (500 MHz for ^{1}H, 125 MHz for ^{13}C) instrument in the indicated solvent. Chemical shifts are reported in units parts per million (ppm) relative to the signal (0.00 ppm) for internal tetramethylsilane for solutions in $CDCl_3$ (7.26 ppm for ^{1}H, 77.16 ppm for ^{13}C) or DMSO-d_6 (2.50 ppm for ^{1}H) or CD_3OD (3.31 ppm for ^{1}H, 49.0 ppm for ^{13}C). Multiplicities are reported using the following abbreviations: s; singlet, d; doublet, dd; doublet of doublets, t; triplet, q; quartet, m; multiplet, br; broad, *J*; coupling constants in Hertz. IR spectra were recorded on a JASCO FT/IR-4200 spectrometer. Only the strongest and/or structurally important peaks are reported as IR data given in $cm^{-1.}$ Mass spectra were measured using a JMS-700 Mstation and Bruker microTOF II. HRMS (EI, 70 eV) was calibrated as perfluorokerosene and HRMS (ESI-TOF) was calibrated as sodium formate. All reactions were monitored by thin-layer chromatography carried out on 0.2 mm E. Merck silica gel plates (60F-254) with UV light (254 nm), and were visualized using an aqueous alkaline $KMnO_4$ solution. Column chromatography was performed on Silica Gel 60 N, purchased from Fuji Silysia Chemical Ltd. Sulfoxonium ylides **4a-f** were prepared prepared according to reported procedures.[1]

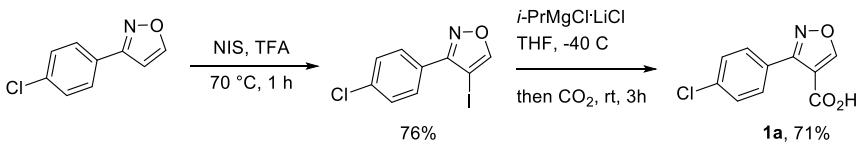
2. Preparation of 4-isoxazolyl carboxylic acids

3-(4-Chlorophenyl)isoxazole-4-carboxylic acid (1a)



To a stirred solution of 4-chloro-*N*-hydroxybenzimidoyl chloride[3] (1.89 g, 0.01 mol) and trimethylsilylacetylene (1.56 mL, 0.011 mol) in $CHCl_3$ (20.0 mL), NEt_3 (3.48 mL, 0.025 mol, 2.50 equiv.) was added at 0 °C under an argon atmosphere. After being stirred for 1 h at 50 °C, the reaction mixture was diluted with CH_2Cl_2. The organic phase was washed with water and brine and dried over $MgSO_4$ and concentrated *in vacuo*. The residue was dissolved in ethanol (25.0 ml) and CsF (3.03 g, 0.02 mol, 2.00 equiv.) was added under an argon atmosphere. After being stirred for 1 h at room temperature, the reaction mixture was poured into water. The aqueous layer was extracted with two portions of CH_2Cl_2. The combined extract was washed with brine, dried over $MgSO_4$ and concentrated *in vacuo*. The residue was purified by column chromatography on silica gel with hexane : ethyl acetate = 95 : 5 to afford 3-(4-chlorophenyl)isoxazole as a pale yellow solid. Spectral properties were identical to those previously reported.[2]

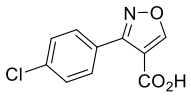
3-(4-Chlorophenyl)isoxazole. Obtained as a white solid (1.16 g, 65%): Mp 74-76 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.47 (d, J = 1.2 Hz, 1H), 7.77 (d, J = 8.4 Hz, 2H), 7.44 (d, J = 8.4 Hz, 2H), 6.64 (d, J = 1.6 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 160.6, 159.2, 136.2, 129.3, 128.2, 127.4, 102.4; FT-IR (neat, cm^{-1}) 3151, 3128, 1546, 1504, 1429, 1274, 1121, 781.



To a stirred solution of 3-(4-chlorophenyl)isoxazole (1.00 equiv.) in TFA (1.00 mL), *N*-iodosuccinimide (2.0 equiv.) was added under an argon atmosphere. After being stirred at 70 °C for 1 h, saturated aq. $NaHCO_3$ was added. The mixture was poured into diethyl ether, the aqueous layer was extracted with two portions of Et_2O. The combined extract was washed with 10% aq. $Na_2S_2O_3$ and brine, dried over $MgSO_4$ and concentrated *in vacuo* to get 3-(4-chlorophenyl)-4-iodo-isoxazole[5] in 76% yield. To this 3-(4-chlorophenyl)-4-iodo-isoxazole (3.75 g, 0.012 mol) in THF (20 mL), 0.8 M solution of *i*PrMgCl·LiCl in THF (16.3 mL, 0.013 mol, 1.10 equiv.) was added dropwise at -78 °C under an argon atmosphere. After being stirred at the same temperature for 30 min, the vessel was filled with CO_2 gas that was collected in a balloon by sublimation of dry ice. After being stirred at room temperature for 3 h, the reaction mixture was acidified with 1 M aq. HCl. The aqueous layer was extracted with two portions of chloroform. The combined extract was washed with brine, dried over $MgSO_4$ and concentrated *in vacuo* to give crude **1a,** which was purified by column chromatography using EtOAc/hexane (1:1) as eluent.

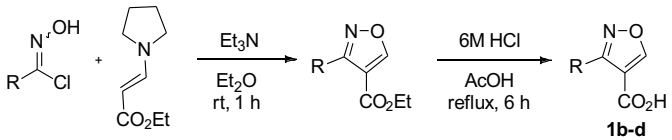
3-(4-Chlorophenyl)isoxazole-4-carboxylic acid (1a)[6]

Obtained as a white solid (1.94 g, 71%): mp 180-182 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.11 (s, 1H), 7.73 (d, J = 8.8 Hz, 2H), 7.46 (d, J = 8.4 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.9, 165.4, 160.5, 136.9, 130.9, 128.8, 125.4, 112.1; FT-IR (neat, cm^{-1}) 3094, 1714, 1602, 1557, 1464, 1309, 778; HRMS (ESI) *m/z* calcd for $C_{10}H_5ClNO_3$ $[M-H]^-$ 221.9958, found 221.9963.



3-Substituted-isoxazolyl-4-carboxylic acids (1b-d)[6]

To a solution of *N*-hydroxybenzimidoyl chloride (0.01 mol)[3] and (*E*) ethyl-3-(pyrrolidin-1-yl)acrylate (1.69 g, 0.01 mol) in diethyl ether, triethylamine (3.48 mL, 0.025 mol) was added at room temperature. The reaction mixture was stirred at room temperature for 1h, and then reaction mixture was poured into saturated NH_4Cl solution, extracted with EtOAc, washed with brine, dried over Na_2SO_4 and the solvent was evaporated under reduced pressure to give crude 3-substituted-isoxazolyl-4-carboxylates. Now, these crude isoxazolyl-4-carboxylates were treated with 6M HCl and AcOH (3:2) and the reaction mixture was refluxed for 6 h (monitored by TLC). The reaction mixture cooled to room temperature, and quenched with saturated aq. $NaHCO_3$, extracted with EtOAc, washed with brine, dried over Na_2SO_4 and the solvent was evaporated under reduced pressure to give crude **1b-d** which were purified by column chromatography using EtOAc/hexane as eluent. Spectral properties were identical to those in our previous report.[6]

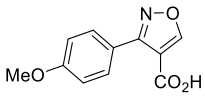


1b-d

3-(4-Methoxyphenyl)isoxazole-4-carboxylic acid (1b)

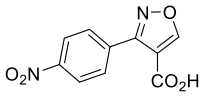
Obtained as a white solid (0.29 g, 67%): mp 195-197 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.07 (s, 1H), 7.75 (d, J = 8.4 Hz, 2H), 7.00 (d, J = 8.8 Hz, 2H), 3.86 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.6, 164.8, 161.4,

161.0, 131.0, 119.1, 113.9, 111.9, 55.4; FT-IR (neat, cm^{-1}) 2954, 2916, 1712, 1613, 1466, 1253, 827; HRMS (ESI) *m/z* calcd for $C_{11}H_8NO_4$ $[M-H]^-$ 218.0453 found 218.0447



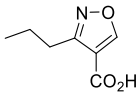
3-(4-Nitrophenyl)isoxazole-4-carboxylic acid (1c)

Obtained as a white solid (0.32 g, 73%): Mp 218-220 °C; ^1H NMR (400 MHz, DMSO-d_6) δ 9.72 (s, 1H), 8.33 (d, J = 8.8 Hz, 2H), 8.01 (d, J = 8.8 Hz, 2H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 166.3, 161.6, 159.5, 148.4, 133.7, 130.8, 123.3, 113.4; FT-IR (neat, cm^{-1}) 3108, 1714, 1529, 1348, 1162, 851; HRMS (ESI) *m/z* calcd for $C_{10}H_5N_2O_5$ $[M-H]^+$ 233.0198, found 233.0206.



3-Propylisoxazole-4-carboxylic acid (1d)

Obtained as a white solid (0.29 g, 70%): Mp 83-85 °C; ^1H NMR (400 MHz, $CDCl_3$) δ 8.95 (s, 1H), 2.91 (t, J = 7.6 Hz, 2H), 1.77 (sextet, J = 7.2 Hz, 2H), 1.01 (t, J = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.8, 164.5, 162.5, 112.4, 27.1, 21.1, 13.9; FT-IR (neat, cm^{-1}) 3120, 2963, 1730, 1579, 1427, 1242, 1109; HRMS (FAB) *m/z* calcd for $C_7H_{10}NO_3$ $[M-H]^-$ 156.0661, found 156.0659.

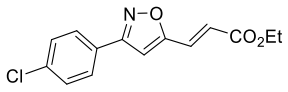


3. General procedures for alkenylation with alkenes

In an oven dried vial tube 4-isoxazolyl carboxylic acid **1** (0.1 mmol), alkene **2** (0.15 mmol), $[Cp^*RhCl_2]_2$ (1.6 mg, 0.025 mmol, 0.025 equiv.), Ag_2CO_3 (27.6 mg, 0.1 mmol, 1.0 equiv.) were taken in DMF (0.5 mL) under an argon atmosphere and then the reaction mixture was heated at 100 °C for 6-8 h (monitored by TLC). After completion of the reaction, the residue was filtered through a pad of celite using ethyl acetate. The solvent was evaporated under reduced pressure, and the residue was purified by column chromatography with hexane : ethyl acetate (90 : 10) to afford desired alkenyl isoxazoles **3**.

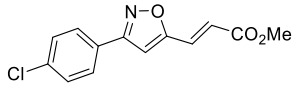
Ethyl (*E*)-3-(3-(4-chlorophenyl)isoxazol-5-yl)acrylate (3a)

Following the general procedure using carboxylic acid **1a** (22.4 mg, 0.1 mmol) and ethyl acrylate (**2a**) (12.0 mg, 0.12 mmol), purification by column chromatography on silica gel (hexane : ethyl acetate = 90 : 10) afforded **3a** (24.2 mg, 87%) as a white solid: mp 155-156°C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, J = 8.8 Hz, 2H), 7.53 (d, J = 16.0 Hz, 1H), 7.45 (d, J = 8.4 Hz, 2H), 6.73 (s, 1H), 6.68 (d, J = 16.0 Hz, 1H), 4.30 (q, J = 7.2 Hz, 2H), 1.35 (t, J = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.5, 165.7, 162.1, 136.5, 129.4, 128.2, 127.3, 127.0, 124.4, 103.8, 61.3, 14.3; FT-IR (neat, cm^{-1}) 2991, 1713, 1428, 1303, 807; HRMS (ESI) *m/z* calcd for $C_{14}H_{12}ClNO_3Na$ $[M+Na]^+$ 300.0403, found 300.040.



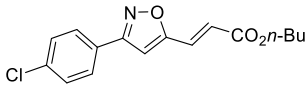
Methyl (*E*)-3-(3-(4-chlorophenyl)isoxazol-5-yl)acrylate (3b)

Following the general procedure using carboxylic acid **1a** (22.4 mg, 0.1 mmol) and methyl acrylate (**2b**) (10.3 mg, 0.12 mmol), purification by column chromatography on silica gel (hexane : ethyl acetate = 90 : 10) afforded **3b** (20.0 mg, 76%) as a white solid: mp 147-148°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, J = 8.4 Hz, 2H), 7.53 (d, J = 16.0 Hz, 1H), 7.45 (d, J = 8.8 Hz, 2H), 6.73 (s, 1H), 6.68 (d, J = 16.0 Hz, 1H), 3.84 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.4, 166.2, 162.1, 136.5, 129.4, 128.2, 127.5, 126.9, 123.8, 103.9, 52.3; FT-IR (neat, cm^{-1}) 3064, 1718, 1427, 1313, 1197, 969; HRMS (ESI) *m/z* calcd for $C_{13}H_{11}ClNO_3$ $[M+H]^+$ 264.0427, found 264.0425.



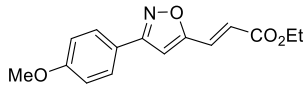
Butyl (*E*)-3-(3-(4-chlorophenyl)isoxazol-5-yl)acrylate (3c)

Following the general procedure using carboxylic acid **1a** (22.4 mg, 0.1 mmol) and butyl acrylate (**2c**) (15.3 mg, 0.12 mmol), purification by column chromatography on silica gel (hexane : ethyl acetate = 90 : 10) afforded **3c** (25.0 mg, 82%) as a white solid: mp 124-125°C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.75 (d, J = 8.5 Hz, 2H), 7.52 (d, J = 16.0 Hz, 1H), 7.45 (d, J = 8.5 Hz, 2H), 6.72 (s, 1H), 6.67 (d, J = 16.0 Hz, 1H), 4.23 (q, J = 6.5 Hz, 2H), 1.70 (sextet, J = 7.0 Hz, 2H), 1.43 (sextet, J = 7.5 Hz, 2H), 0.96 (t, J = 7.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.6, 165.8, 162.1, 136.5, 129.4, 128.1, 127.2, 127.0, 124.4, 103.8, 65.2, 30.7, 19.2, 13.8; FT-IR (neat, cm^{-1}) 2960, 1713, 1505, 1428, 1186, 781; HRMS (ESI) *m/z* calcd for $C_{16}H_{16}ClNNaO_3$ $[M+H]^+$ 328.0716, found 328.0704.



Ethyl (*E*)-3-(3-(4-methoxyphenyl)isoxazol-5-yl)acrylate (3d)

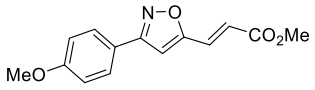
Following the general procedure using carboxylic acid **1b** (21.9 mg, 0.1 mmol) and ethyl acrylate (**2a**) (12.0 mg, 0.12 mmol), purification by column chromatography on silica gel (hexane : ethyl acetate = 90 : 10) afforded **3d** (22.4 mg, 82%) as a white solid: mp 94-95°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (d, J = 8.8 Hz, 2H), 7.52 (d, J = 16.0 Hz, 1H), 6.98 (d, J = 8.8 Hz, 2H), 6.69 (s, 1H), 6.65 (d, J = 16.4 Hz, 1H), 4.29 (q, J = 7.2 Hz, 2H), 1.34 (t, J = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.0, 165.9, 162.6, 161.3, 128.3, 127.5, 123.9, 121.0, 114.5, 103.9, 61.2, 55.5, 14.3; FT-IR (neat, cm^{-1}) 3105, 2982, 1705, 1652, 1453, 1266, 808; HRMS (ESI) *m/z* calcd for $C_{15}H_{15}NO_4Na$ $[M+Na]^+$ 296.0899 found 296.0885.



Methyl (*E*)-3-(3-(4-methoxyphenyl)isoxazol-5-yl)acrylate (3e)

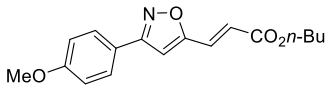
Following the general procedure using carboxylic acid **1b** (21.9 mg, 0.1 mmol) and methyl acrylate (**2b**) (10.3 mg, 0.12 mmol), purification by column chromatography on silica gel (hexane : ethyl acetate = 90 : 10) afforded

3e (20.2 mg, 78%) as a white solid: mp 140-141°C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.75 (d, J = 8.4 Hz, 2H), 7.53 (d, J = 16.0 Hz, 1H), 6.98 (d, J = 8.8 Hz, 2H), 6.70 (s, 1H), 6.66 (d, J = 16.0 Hz, 1H), 3.86 (s, 3H), 3.83 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.3, 165.8, 162.7, 161.3, 128.3, 127.8, 123.3, 120.9, 114.5, 104.0, 55.5, 52.2; FT-IR (neat, cm^{-1}) 2922, 1724, 1435, 1282, 1030, 810; HRMS (ESI) *m/z* calcd for $C_{14}H_{13}NO_4Na$ $[M+Na]^+$ 282.0742 found 282.0737.



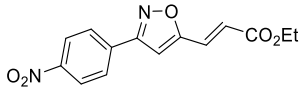
Butyl (*E*)-3-(3-(4-methoxyphenyl)isoxazol-5-yl)acrylate (3f)

Following the general procedure using carboxylic acid **1b** (21.9 mg, 0.1 mmol) and butyl acrylate (**2c**) (15.3 mg, 0.12 mmol), purification by column chromatography on silica gel (hexane : ethyl acetate = 90 : 10) afforded **3f** (20.2 mg, 67%) as a white solid: mp 92-93°C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.75 (d, J = 9.0 Hz, 2H), 7.51 (d, J = 16.0 Hz, 1H), 6.98 (d, J = 8.5 Hz, 2H), 6.69 (s, 1H), 6.65 (d, J = 16.0 Hz, 1H), 4.23 (t, J = 7.0 Hz, 2H), 3.85 (s, 3H), 1.70 (sextet, J = 7.0 Hz, 2H), 1.43 (sextet, J = 7.5 Hz, 2H), 0.96 (t, J = 7.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.0, 165.9, 162.6, 161.3, 128.3, 127.5, 123.9, 121.0, 114.5, 103.9, 65.1, 55.4, 30.7, 19.2, 13.8; FT-IR (neat, cm^{-1}) 2959, 1709, 1652, 1432, 970, 808; HRMS (ESI) *m/z* calcd for $C_{17}H_{19}NO_4Na$ $[M+Na]^+$ 324.1212 found 324.1204.



Ethyl (*E*)-3-(3-(4-nitrophenyl)isoxazol-5-yl)acrylate (3g)

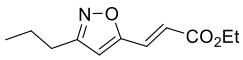
Following the general procedure using carboxylic acid **1c** (23.4 mg, 0.1 mmol) and ethyl acrylate (**2a**) (12.0 mg, 0.12 mmol), purification by column chromatography on silica gel (hexane : ethyl acetate = 95 : 5) afforded **3g** (21.3 mg, 74%) as a white solid: mp 145-146°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.34 (d, J = 8.8 Hz, 2H), 8.00 (d, J = 8.8 Hz, 2H), 7.55 (d, J = 16.0 Hz, 1H), 6.82 (s, 1H), 6.71 (d, J = 16.0 Hz, 1H), 4.30 (q, J = 7.2 Hz, 2H), 1.35 (t, J = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.3, 165.6, 161.3, 149.0, 134.5, 127.8, 126.9, 124.9, 124.4, 103.8, 61.4, 14.3; FT-IR (neat, cm^{-1}) 2956, 1710, 1522, 1304, 970; HRMS (ESI) *m/z* calcd for $C_{14}H_{12}N_2O_5Na$ $[M+Na]^+$ 311.0644 found 311.0632.



Ethyl (*E*)-3-(3-(4-propylphenyl)isoxazol-5-yl)acrylate (3h)

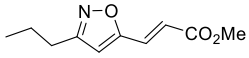
Following the general procedure using carboxylic acid **1d** (15.5 mg, 0.1 mmol) and ethyl acrylate (**2a**) (12.0 mg, 0.12 mmol), purification by column chromatography on silica gel (hexane : ethyl acetate = 95 : 5) afforded **3h** (11.3 mg, 55%) as a colorless liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, J = 16.0 Hz, 1H), 6.58 (d, J = 16.4 Hz, 1H), 6.30 (s, 1H), 4.27 (q, J = 7.2 Hz, 2H), 2.66 (t, J = 7.2 Hz, 2H), 1.70 (sextet, J = 7.2 Hz, 2H), 1.33 (t, J = 7.2 Hz, 3H), 0.98 (t, J = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.9, 165.4, 164.6, 127.7,

123.6, 105.9, 61.1, 28.0, 21.7, 14.3, 13.8; FT-IR (neat, cm^{-1}) 2964, 1718, 1654, 1570, 1304, 1175, 972; HRMS (ESI) *m/z* calcd for $C_{11}H_{15}NO_3Na$ $[M+Na]^+$ 232.0944 found 232.0949.



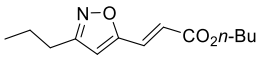
Methyl (*E*)-3-(3-propylisoxazol-5-yl)acrylate (3i)

Following the general procedure using carboxylic acid **1d** (15.5 mg, 0.1 mmol) and methyl acrylate (**2b**) (10.3 mg, 0.12 mmol), purification by column chromatography on silica gel (hexane : ethyl acetate = 95 : 5) afforded **3i** (16.9 mg, 87%) as a white solid: mp 59-60°C; ^1H NMR (400 MHz, $CDCl_3$) δ 7.45 (d, J = 16.0 Hz, 1H), 6.58 (d, J = 16.0 Hz, 1H), 6.30 (s, 1H), 3.81 (s, 3H), 2.66 (t, J = 7.2 Hz, 2H), 1.70 (sextet, J = 7.2 Hz, 2H), 0.98 (t, J = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.4, 165.3, 164.6, 127.9, 123.1, 106.0, 52.2, 28.0, 21.7, 13.8; FT-IR (neat, cm^{-1}) 2930, 1714, 1569, 1308, 1179, 822; HRMS (ESI) *m/z* calcd for $C_{10}H_{13}NO_3$ $[M+H]^+$ 196.0974, found 196.0970.



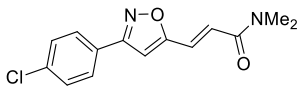
Butyl (*E*)-3-(3-propylisoxazol-5-yl)acrylate (3j)

Following the general procedure using carboxylic acid **1d** (15.5 mg, 0.1 mmol) and butyl acrylate (**2c**) (15.3 mg, 0.12 mmol), purification by column chromatography on silica gel (hexane : ethyl acetate = 95 : 5) afforded **3j** (13.0 mg, 55%) as amorphous solid. ^1H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, J = 16.0 Hz, 1H), 6.58 (d, J = 16.0 Hz, 1H), 6.30 (s, 1H), 4.21 (t, J = 6.8 Hz, 3H), 2.66 (t, J = 7.6 Hz, 2H), 1.74-1.64 (m, 4H), 1.42 (sextet, J = 7.2 Hz, 2H), 0.98 (t, J = 7.2 Hz, 3H), 0.95 (t, J = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 166.0, 165.4, 164.6, 127.6, 105.9, 65.0, 30.7, 28.0, 21.7, 19.2, 13.83, 13.81; FT-IR (neat, cm^{-1}) 2962, 1719, 1570, 1421, 1304, 1172; HRMS (ESI) *m/z* calcd for $C_{13}H_{19}NO_3Na$ $[M+Na]^+$ 260.1263, found 260.1259.



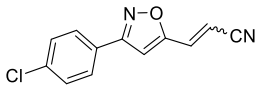
(*E*)-3-(3-(4-Chlorophenyl)isoxazol-5-yl)-*N*,*N*-dimethylacrylamide (3k)

Following the general procedure using carboxylic acid **1a** (22.4 mg, 0.1 mmol), *N*,*N*-dimethylacrylamide (**2d**) (11.9 mg, 0.12 mmol), purification by column chromatography on silica gel (hexane : ethyl acetate = 60 : 40) afforded **3k** (19.9 mg, 72%) as a white solid: mp 214-215°C; ^1H NMR (500 MHz, $CDCl_3$) δ 7.75 (d, J = 8.5 Hz, 2H), 7.52 (d, J = 15.5 Hz, 1H), 7.45 (d, J = 8.5 Hz, 2H), 7.19 (d, J = 15.5 Hz, 1H), 6.66 (s, 1H), 3.20 (s, 3H), 3.08 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.2, 165.1, 162.1, 136.4, 129.4, 128.2, 127.1, 125.4, 123.3, 103.5, 37.5, 36.1; FT-IR (neat, cm^{-1}) 2921, 1658, 1618, 1560, 1428, 971; HRMS (ESI) *m/z* calcd for $C_{14}H_{13}ClN_2O_2Na$ $[M+Na]^+$ 299.0563 and 301.0534 found 299.0552 and 310.528.



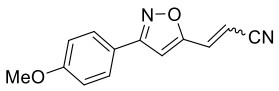
(*E*/*Z*)-3-(3-(4-Chlorophenyl)isoxazol-5-yl)acrylonitrile (3l).

Following the general procedure using carboxylic acid **1a** (22.4 mg, 0.1 mmol), acrylonitrile (**2e**) (6.4 mg, 0.12 mmol), purification by column chromatography on silica gel (hexane : ethyl acetate = 10 : 90) afforded **3l** (19.8 mg, 83%) as a pale yellow solid (E/Z = 4:1): mp 120-121°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, J = 8.8 Hz, 0.5H), 7.74 (d, J = 8.4 Hz, 1.5H), 7.46 (d, J = 8.8 Hz, 2H), 7.37 (s, 0.25H), 7.27 (d, J = 16.0 Hz, 0.75H), 7.23 (d, J = 12.4 Hz, 0.25H), 6.76 (s, 0.75H), 6.21 (d, J = 16.8 Hz, 0.75H), 5.72 (d, J = 12.0 Hz, 0.25H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.2, 164.7, 162.39, 162.36, 136.9, 136.7, 133.0, 132.8, 129.57, 129.52, 128.3, 128.2, 126.7, 126.5, 116.5, 115.7, 104.7, 103.9, 102.7, 100.5; FT-IR (neat, cm^{-1}) 3074, 2224, 1556, 1425, 1097, 948; HRMS (ESI) *m/z* calcd for $C_{12}H_7ClN_2ONa$ $[M+Na]^+$ 253.0145, found 253.0144.



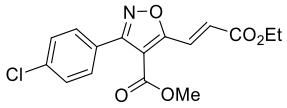
(*E*/*Z*)-3-(3-(4-Chlorophenyl)isoxazol-5-yl)acrylonitrile (3m)

Following the general procedure using carboxylic acid **1b** (21.9 mg, 0.1 mmol), acrylonitrile (**2e**) (6.4 mg, 0.12 mmol), purification by column chromatography on silica gel (hexane : ethyl acetate = 10 : 90) afforded **3m** (20.3 mg, 90%) as a white solid (*E*/*Z* = 4:1): mp 125-126°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, J = 8.4 Hz, 0.5H), 7.74 (d, J = 8.8 Hz, 1.5H), 7.34 (s, 0.25H), 7.26 (d, J = 16.4 Hz, 0.75H), 7.21 (d, J = 12.0 Hz, 0.25H), 6.99 (d, J = 8.8 Hz, 2H), 6.73 (s, 0.75H), 6.18 (d, J = 16.4 Hz, 0.75H), 5.70 (d, J = 12.0 Hz, 0.25H), 3.86 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.2, 162.9, 161.6, 133.2, 128.5, 128.4, 120.4, 116.7, 114.65, 114.60, 104.8, 103.9, 102.2, 55.5; FT-IR (neat, cm^{-1}) 2917, 2220, 1609, 1524, 1431, 1254, 835; HRMS (ESI) *m/z* calcd for $C_{13}H_{10}N_2O_2Na$ $[M+Na]^+$ 249.0640, found 249.0635.

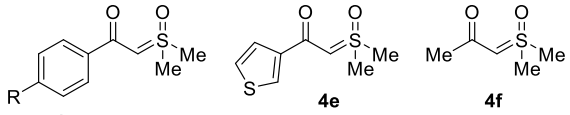


Methyl (*E*)-3-(4-chlorophenyl)-5-(3-ethoxy-3-oxoprop-1-en-1-yl)isoxazole-4-carboxylate (6)

In an oven dried vial tube, carboxylic acid **1a** (22.4 mg, 0.1 mmol), ethyl acrylate (**2a**) (12.0 mg, 0.12 mmol, 1.2 equiv.), $[Cp^*RhCl_2]_2$ (1.6 mg, 0.025 mmol, 0.025 equiv.), Ag_2CO_3 (27.6 mg, 0.1 mmol, 1.0 equiv.) were taken in DMF (0.5 mL) under an argon atmosphere and then the reaction mixture was heated at 100 °C for 30 min. To the reaction mixture, MeI (19 μL, 0.3 mmol, 3.0 equiv.) and K_2CO_3 (27.6 mg, 0.2 mmol, 2.0 equiv.) were added. After being stirred at room temperature for 3 h, the residue was filtered through a pad of celite using ethyl acetate. The solvent was evaporated under reduced pressure, and the residue were purified by column chromatography with hexane : ethyl acetate (90 : 10) to give corresponding methyl ester **6** (4.0 mg, 0.087 mmol, 12% yield) as a white solid. Mp 86-87 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, J = 16.0 Hz, 1H), 7.60 (d, J = 8.4 Hz, 2H), 7.45 (d, J = 8.4 Hz, 2H), 6.91 (d, J = 16.4 Hz, 1H), 4.33 (q, J = 7.2 Hz, 2H), 3.85 (s, 3H), 1.36 (t, J = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.0, 165.4, 162.5, 161.4, 136.6, 130.9, 128.7, 127.9, 126.9, 126.2, 111.0, 61.6, 52.5, 14.3; FT-IR (neat, cm^{-1}) 2955, 1721, 1557, 1440, 1243, 1078; HRMS (ESI) *m/z* calcd for $C_{16}H_{14}ClNO_5Na$ $[M+Na]^+$ 358.0458, found 358.0452.



4. Structure of sulfoxonium ylides (4)



4a (R = H)
4b (R = OMe)
4c (R = CF_3)
4d (R = Br)

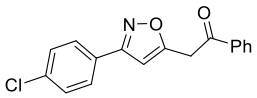
4e **4f**

5. General procedures for acylmethylation with sulfoxonium ylides

In an oven dried vial tube 4-isoxazolyl carboxylic acid **1** (0.1 mmol), sulfoxonium ylide **4** (0.12 mmol), $[Cp^*RhCl_2]_2$ (1.6 mg, 0.025 mmol, 0.025 equiv.), $AgSbF_6$ (6.2 mg, 0.020 mmol, 0.2 equiv.), NaOAc (1.6 mg, 0.020 mmol, 0.2 equiv.) were taken in 1,2-dichloroethane (0.5 mL) under an argon atmosphere and then the reaction mixture was heated at 100 °C for 6-8 h (monitored by TLC). After completion of the reaction, the residue was filtered through a pad of celite using ethyl acetate. The solvent was evaporated under reduced pressure, and the residue was purified by column chromatography to afford desired alkyl isoxazoles **5**.

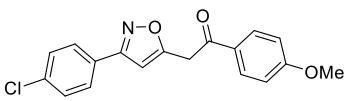
2-(3-(4-Chlorophenyl)isoxazol-5-yl)-1-phenylethan-1-one (5a)

Following the general procedure using carboxylic acid **1a** (22.4 mg, 0.1 mmol), sulfoxonium ylide **4a** (23.5 mg, 0.12 mmol), purification by column chromatography on silica gel (hexane : ethyl acetate = 10 : 90) afforded **5a** (20.8 mg, 70%) as a white solid: mp 129-130°C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.03-8.02 (m, 2H), 7.74 (d, J = 8.5 Hz, 2H), 7.64-7.61 (m, 1H), 7.53-7.50 (m, 2H), 7.42 (d, J = 8.5 Hz, 2H), 6.61 (s, 1H), 4.51 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 192.8, 166.7, 161.8, 136.1, 135.8, 134.1, 129.3, 129.0, 128.6, 128.2, 127.6, 101.8, 36.8; FT-IR (neat, cm^{-1}) 3127, 1692, 1449, 1007, 843; HRMS (ESI) *m/z* calcd for $C_{17}H_{12}ClNNaO_2$ $[M+Na]^+$ 320.0454 , found 320.0456.



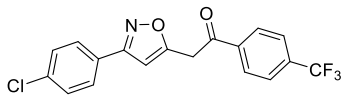
2-(3-(4-Chlorophenyl)isoxazol-5-yl)-1-(4-methoxyphenyl)ethan-1-one (5b)

Following the general procedure using carboxylic acid **1a** (22.4 mg, 0.1 mmol), sulfoxonium ylide **4b** (25.3 mg, 0.12 mmol), purification by column chromatography on silica gel (hexane : ethyl acetate = 10 : 90) afforded **5b** (15.0 mg, 46%) as a white solid: mp 119-120°C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.00 (d, J = 8.5 Hz, 2H), 7.73 (d, J = 8.5 Hz, 2H), 7.41 (d, J = 8.5 Hz, 2H), 6.97 (d, J = 8.5 Hz, 2H), 6.58 (s, 1H), 4.45 (s, 2H), 3.88 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 191.3, 167.1, 164.3, 161.8, 136.1, 131.0, 129.2, 128.8, 128.2, 127.7, 114.2, 101.6, 55.7, 36.6; FT-IR (neat, cm^{-1}) 2924, 1718, 1600, 1489, 1259, 1029; HRMS (ESI) *m/z* calcd for $C_{18}H_{14}ClNNaO_3[M+Na]^+$ 350.0560, found 350.0554.



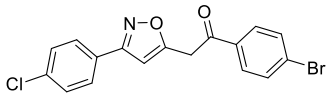
2-(3-(4-Chlorophenyl)isoxazol-5-yl)-1-(4-(trifluoromethyl)phenyl)ethan-1-one (5c)

Following the general procedure using carboxylic acid **1a** (22.4 mg, 0.1 mmol), sulfoxonium ylide **4c** (31.7 mg, 0.12 mmol), purification by column chromatography on silica gel (hexane : ethyl acetate = 10 : 90) afforded **5c** (14.9 mg, 41%) as a white solid: mp 152-153°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, J = 8.0 Hz, 2H), 7.79 (d, J = 8.4 Hz, 2H), 7.74 (d, J = 8.8 Hz, 2H), 7.43 (d, J = 8.4 Hz, 2H), 6.63 (s, 1H), 4.54 (d, J = 0.4 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 191.9, 165.8, 161.9, 138.4, 136.2, 135.4 (q, $J_{C\text{-}F}$ = 33.5 Hz), 129.3, 129.0, 128.2, 127.4, 126.4 (q, $J_{C\text{-}F}$ = 3.8 Hz), 123.5 (q, $J_{C\text{-}F}$ = 271.0 Hz), 37.1; FT-IR (neat, cm^{-1}) 2920, 1697, 1410, 1328, 1119, 827; HRMS (ESI) *m/z* calcd for $C_{18}H_{11}ClF_3NNaO_2$[M+Na]$^+$ 388.0328, found 388.0326.



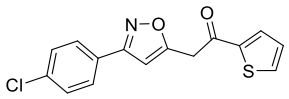
1-(4-Bromophenyl)-2-(3-(4-chlorophenyl)isoxazol-5-yl)ethan-1-one (5d)

Following the general procedure using carboxylic acid **1a** (22.4 mg, 0.1 mmol), sulfoxonium ylide **4d** (35.3 mg, 0.12 mmol), purification by column chromatography on silica gel (hexane : ethyl acetate = 20 : 80) afforded **5d** (24.7 mg, 66%) as a white solid: mp 150-151°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, J = 8.4 Hz, 2H), 7.73 (d, J = 8.8 Hz, 2H), 7.66 (d, J = 8.4 Hz, 2H), 7.43 (d, J = 8.8 Hz, 2H), 6.06 (s, 1H), 4.76 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 191.8, 166.2, 161.9, 136.2, 132.4, 130,1, 129.5, 129.3. 128.2, 127.5, 101.9, 36.8; FT-IR (neat, cm^{-1}) 2918, 1692, 1583, 1396, 1006, 810; HRMS (ESI) *m/z* calcd for $C_{17}H_{11}BrClNNaO_2$[M+Na]$^+$ 397.9559, found 397.9559.



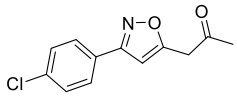
2-(3-(4-Chlorophenyl)isoxazol-5-yl)-1-(thiophen-2-yl)ethan-1-one (5e)

Following the general procedure using carboxylic acid **1a** (22.4 mg, 0.1 mmol), sulfoxonium ylide **4e** (24.2 mg, 0.12 mmol), purification by column chromatography on silica gel (hexane : ethyl acetate = 10 : 90) afforded **5e** (24.2 mg, 80%) as a pale yellow solid: mp 126-127°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (dd, J = 4.0 Hz, 1.2 Hz, 1H), 7.74-7.71 (m, 3H), 7.41 (d, J = 8.8 Hz, 2H), 7.18 (dd, J = 4.8 Hz, 3.6 Hz, 1H), 6.62 (s, 1H), 4.43 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 185.5, 166.2, 161.9, 142.9, 136.1, 135.4, 133.4, 129.3, 128.6, 128.2, 127.5, 101.8, 37.4; FT-IR (neat, cm^{-1}) 3128, 1670, 1455, 1220, 839; HRMS (ESI) *m/z* calcd for $C_{15}H_{10}ClNNaO_2S$[M+Na]$^+$ 326.0018, found 326.0007.



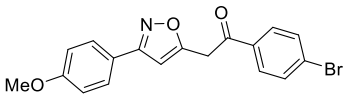
1-(3-(4-Chlorophenyl)isoxazol-5-yl)propan-2-one (5f)

Following the general procedure using carboxylic acid **1a** (22.4 mg, 0.1 mmol), sulfoxonium ylide **4f** (16.1 mg, 0.12 mmol), purification by column chromatography on silica gel (hexane : ethyl acetate = 5 : 95) afforded **5f** (12.0 mg, 51%) as a white solid: mp 95-96°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, J = 8.8 Hz, 2H), 7.43 (d, J = 8.8 Hz, 2H), 6.55 (s, 1H), 3.95 (s, 2H), 2.30 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 200.9, 166.2, 161.9, 136.2, 129.3, 128.2, 127.5, 101.7, 41.4, 30.0; FT-IR (neat, cm^{-1}) 2913, 1716, 1431, 1327, 1098, 834; HRMS (ESI) *m/z* calcd for $C_{12}H_{10}ClNNaO_2[M+Na]^+$ 258.0298, found 258.0291.



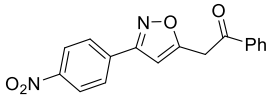
1-(4-Bromophenyl)-2-(3-(4-methoxyphenyl)isoxazol-5-yl)ethan-1-one (5g)

Following the general procedure using carboxylic acid **1b** (21.9 mg, 0.1 mmol), sulfoxonium ylide **4d** (35.3 mg, 0.12 mmol), purification by column chromatography on silica gel (hexane : ethyl acetate = 10 : 90) afforded **5g** (19.7 mg, 53%) as a white solid: mp 145-146°C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.89 (d, J = 8.5 Hz, 2H), 7.73 (d, J = 8.5 Hz, 2H), 7.65 (d, J = 8.5 Hz, 2H), 6.96 (d, J = 8.5 Hz, 2H), 6.55 (s, 1H), 4.45 (s, 2H), 3.85 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 192.0, 165.5, 162.5, 161.1, 134.5, 132.4, 130.1, 129.4, 128.3, 121.5, 114.4, 101.7, 55.4, 36.9; FT-IR (neat, cm^{-1}) 2917, 1698, 1546, 1257, 813; HRMS (ESI) *m/z* calcd for $C_{18}H_{14}BrNNaO_3[M+Na]^+$ 394.0055, found 394.0050.



2-(3-(4-Nitrophenyl)isoxazol-5-yl)-1-phenylethan-1-one (5h)

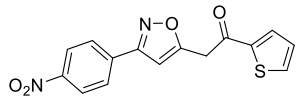
Following the general procedure using carboxylic acid **1c** (23.4 mg, 0.1 mmol), sulfoxonium ylide **4a** (23.5 mg, 0.12 mmol), purification by column chromatography on silica gel (hexane : ethyl acetate = 20 : 80) afforded **5h** (20.9 mg, 68%) as a white solid: mp 194-195°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (d, J = 8.8 Hz, 2H), 8.04 (d, J = 7.6 Hz, 2H), 7.99 (d, J = 8.8 Hz, 2H), 7.67-7.63 (m, 1H), 7.55-7.51 (m, 2H), 6.74 (s, 1H), 4.56 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 192.5, 167.5, 161.0, 148.8, 135.7, 135.2, 134.3, 129.1, 128.6, 127.8, 124.3, 102.2, 36.7; FT-IR (neat, cm^{-1}) 2915, 1690, 1516, 1356, 858; HRMS (ESI) *m/z* calcd for $C_{17}H_{12}N_2NaO_4$ $[M+Na]^+$ 331.0695, found 331.0692.



2-(3-(4-Nitrophenyl)isoxazol-5-yl)-1-(thiophen-2-yl)ethan-1-one (5i)

Following the general procedure using carboxylic acid **1c** (23.4 mg, 0.1 mmol), sulfoxonium ylide **4e** (24.2 mg, 0.12 mmol), purification by column chromatography on silica gel (hexane : ethyl acetate = 10 : 90) afforded **5i** (14.1 mg, 45%) as a pale yellow solid: mp 148-149°C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (d, J = 8.8 Hz, 2H), 7.99 (d, J = 8.8 Hz, 2H), 7.86 (dd, J = 2.8 Hz, 2.0 Hz, 1H), 7.76 (dd, J = 4.0 Hz, 0.8 Hz, 1H), 7.20 (dd, J = 4.0

Hz, 3.2 Hz, 1H), 6.75 (s, 1H), 4.48 (s, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 185.2, 167.1, 161.1, 148.8, 142.8, 135.5, 135.2, 133.4, 128.7, 127.8, 124.3, 102.1, 37.3; FT-IR (neat, cm^{-1}) 2916, 1642, 1518, 1414, 1352, 859; HRMS (ESI) *m/z* calcd for $C_{15}H_{10}N_2NaO_4S[M+Na]^+$ 337.0259, found 337.0250.



6. References

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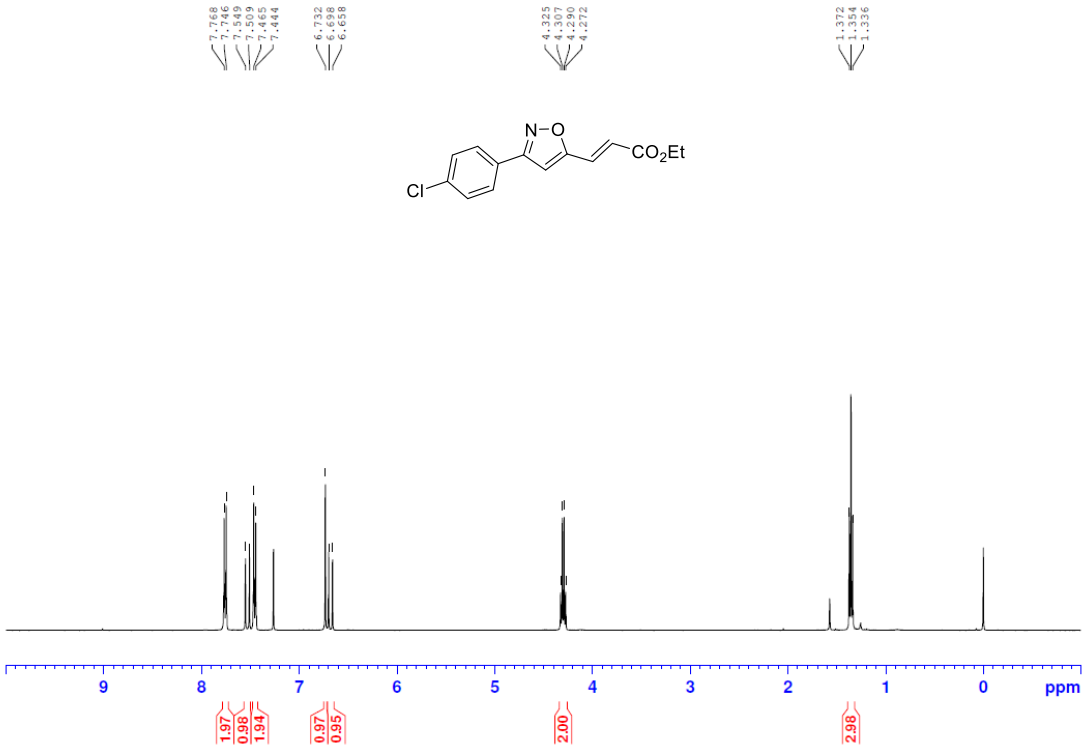
2. X. Z. Wang, J. Jia, Y. Zhang, W. R.Xu, W. Liu, F. N. Shi, J. W. Wang, *J. Chin. Chem. Soc*. **2007**, *54*, 643-652.
3. M. Shigenobu, K. Takenaka and H. Sasai, *Angew. Chem. Int. Ed.* 2015, **54**, 9572-9576.
4. T. Morita, S. Fuse, H. Nakamura, *Angew. Chem. Int. Ed.* **2016**, *55*, 13580-13584.
5. S. Yugandar, H. Nakamura, *Chem. Commun.* **2019**, *55*, 8382-8385.

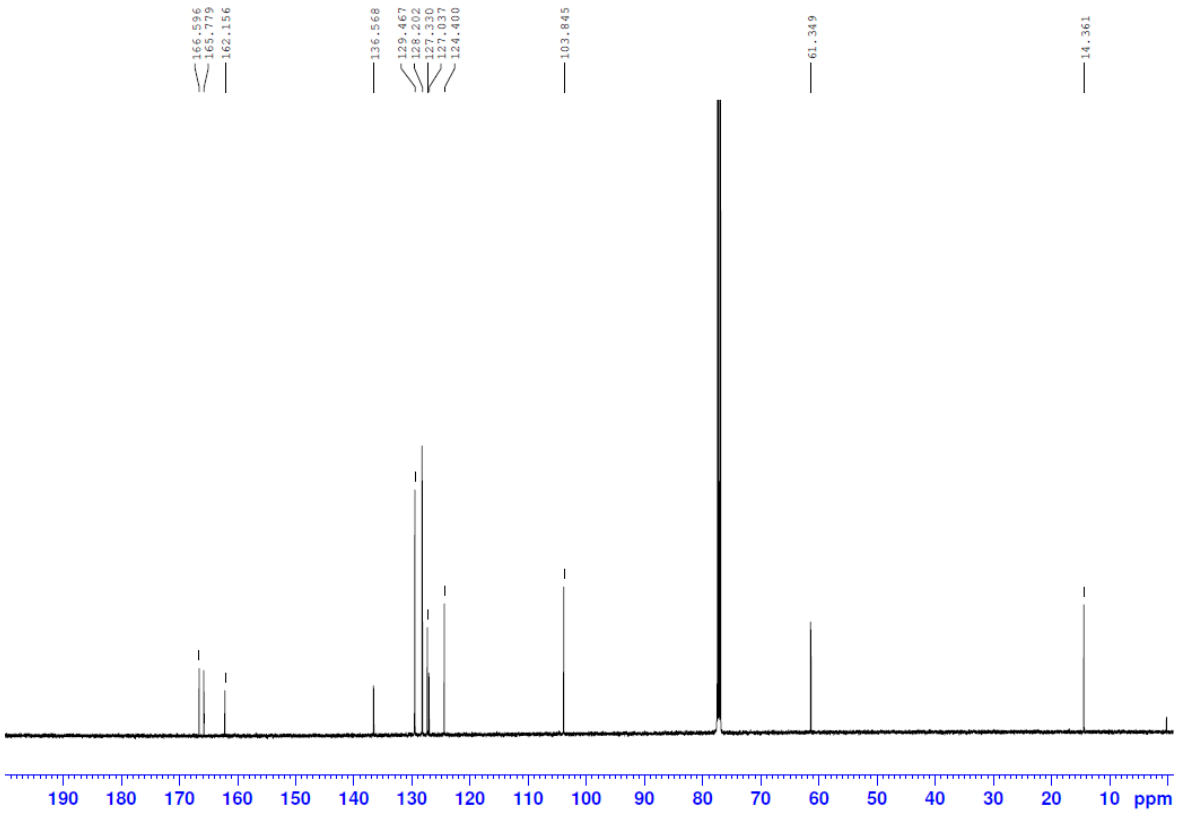
7. NMR Spectra

Ethyl (*E*)-3-(3-(4-chlorophenyl)isoxazol-5-yl)acrylate (3a)

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

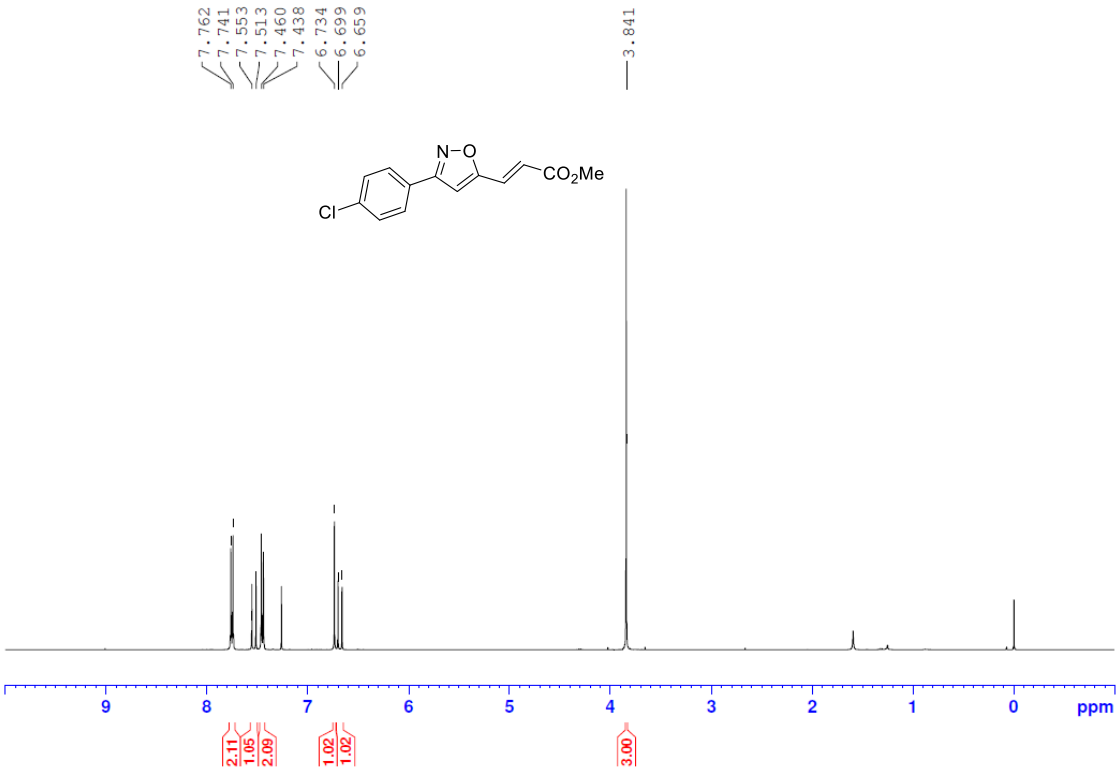


^{13}C NMR (125 MHz, $CDCl_3$)

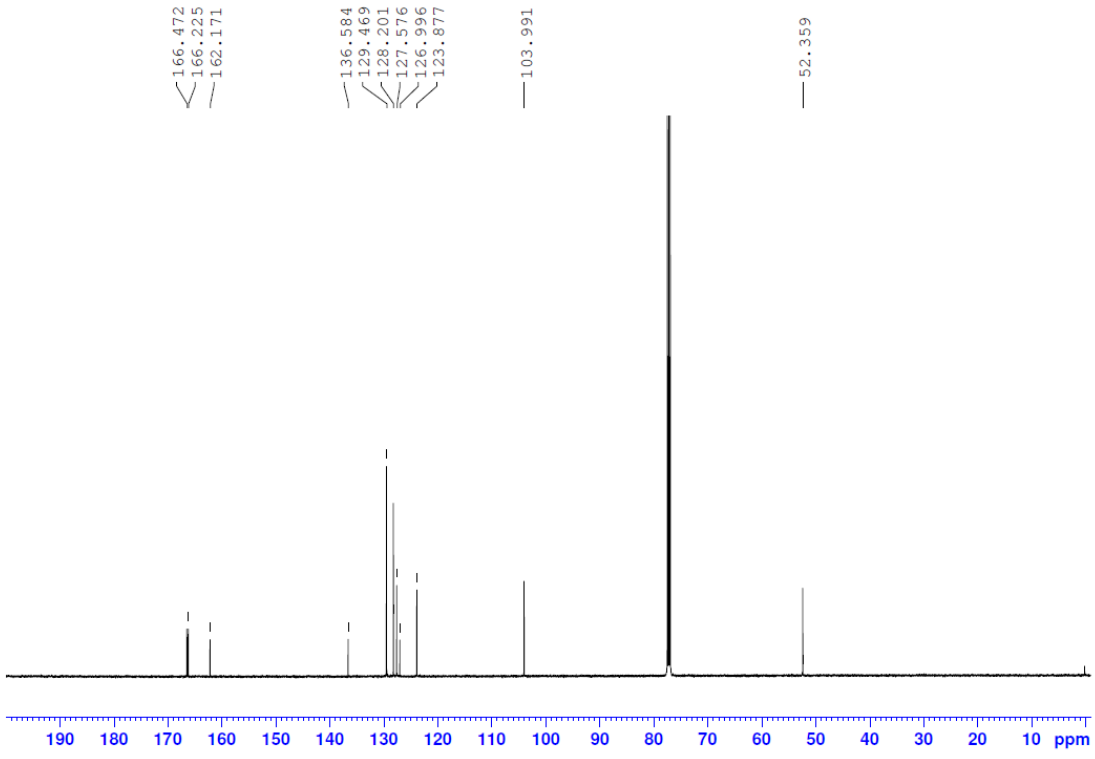


Methyl (*E*)-3-(3-(4-chlorophenyl)isoxazol-5-yl)acrylate (3b)

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

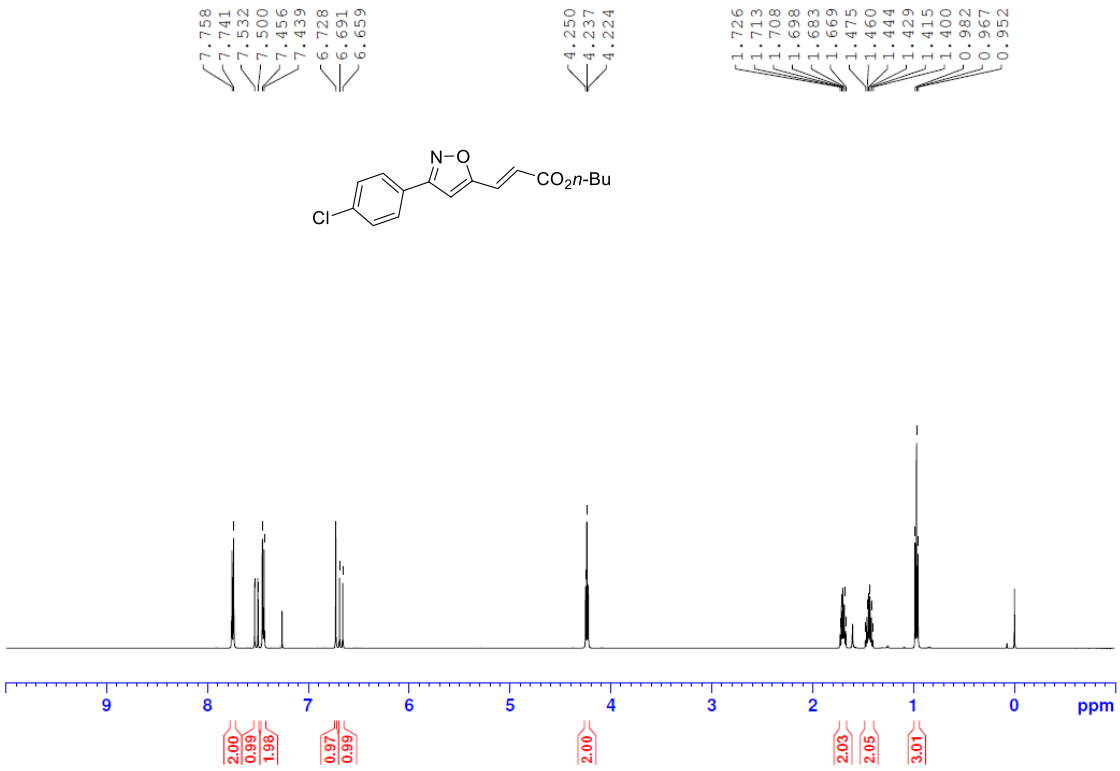


^{13}C NMR (125 MHz, $CDCl_3$)

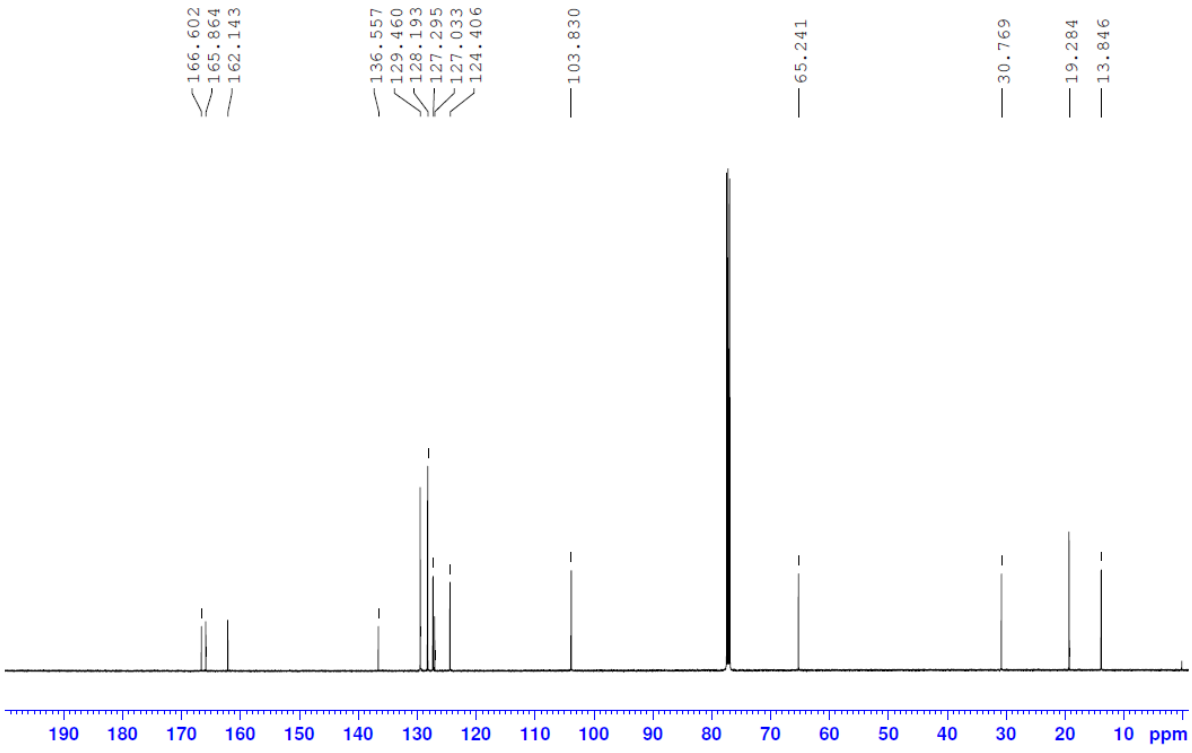


Butyl (*E*)-3-(3-(4-chlorophenyl)isoxazol-5-yl)acrylate (3c)

^{1}H NMR (500 MHz, $CDCl_3$)

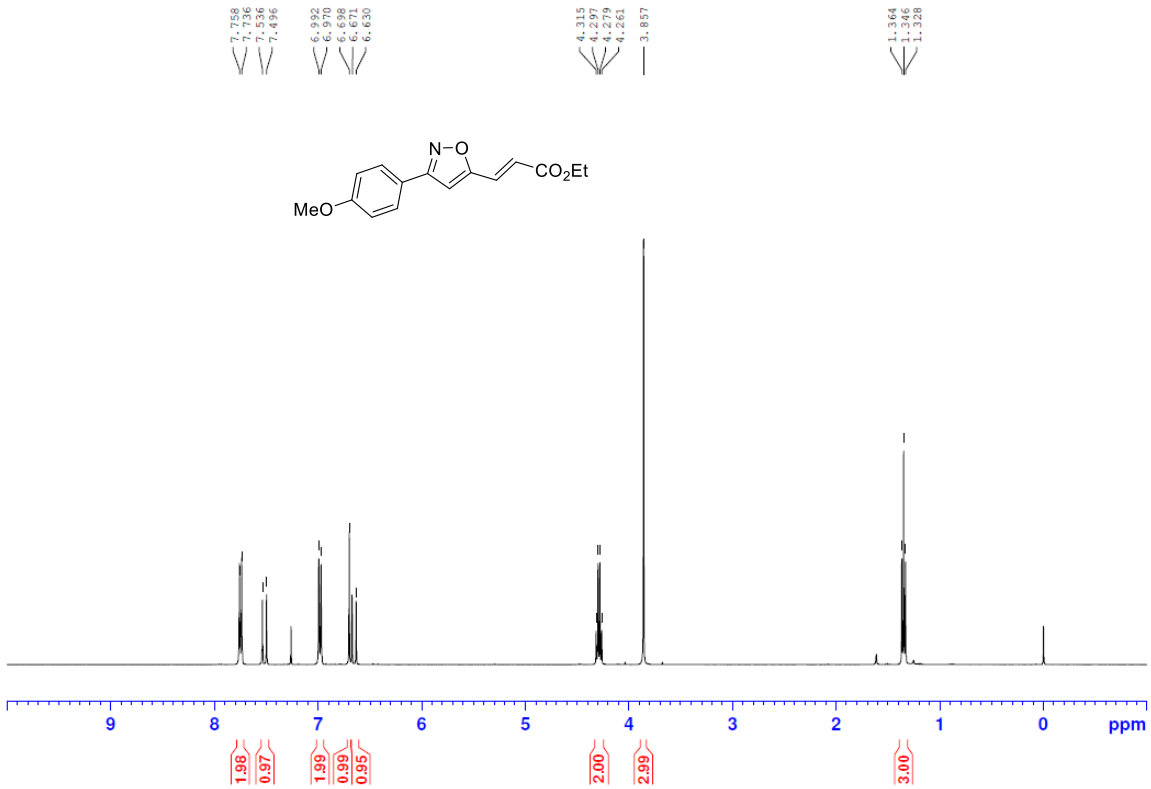


^{13}C NMR (125 MHz, $CDCl_3$)

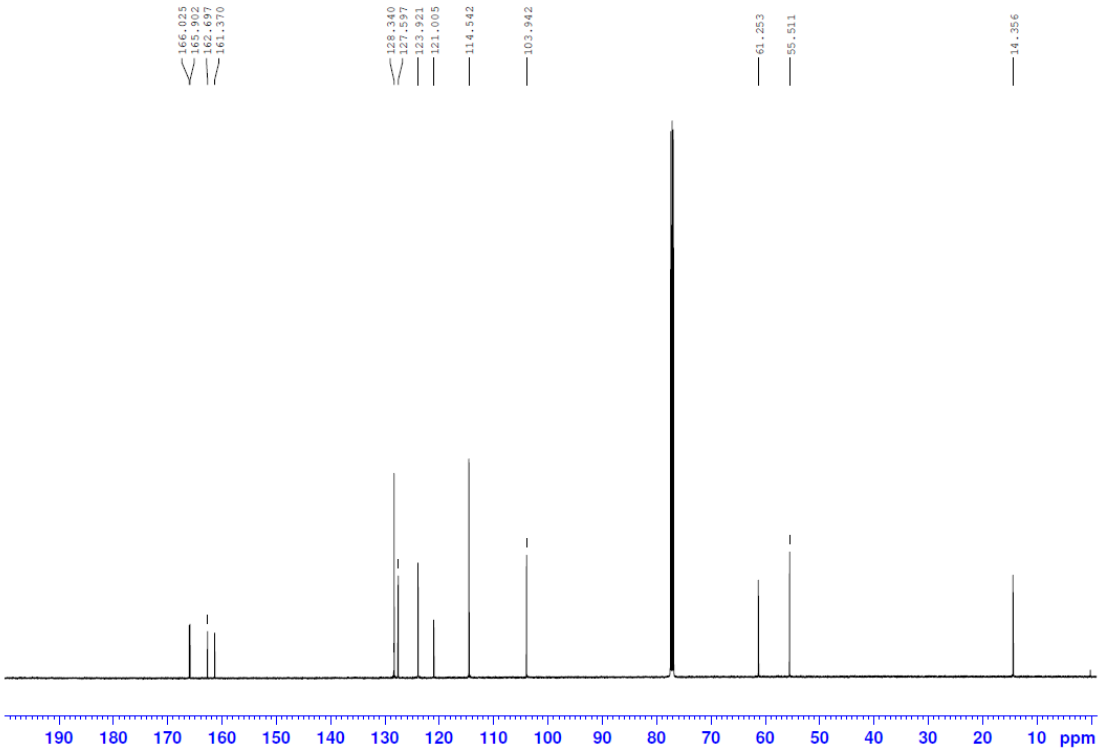


Ethyl (*E*)-3-(3-(4-methoxyphenyl)isoxazol-5-yl)acrylate (3d)

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

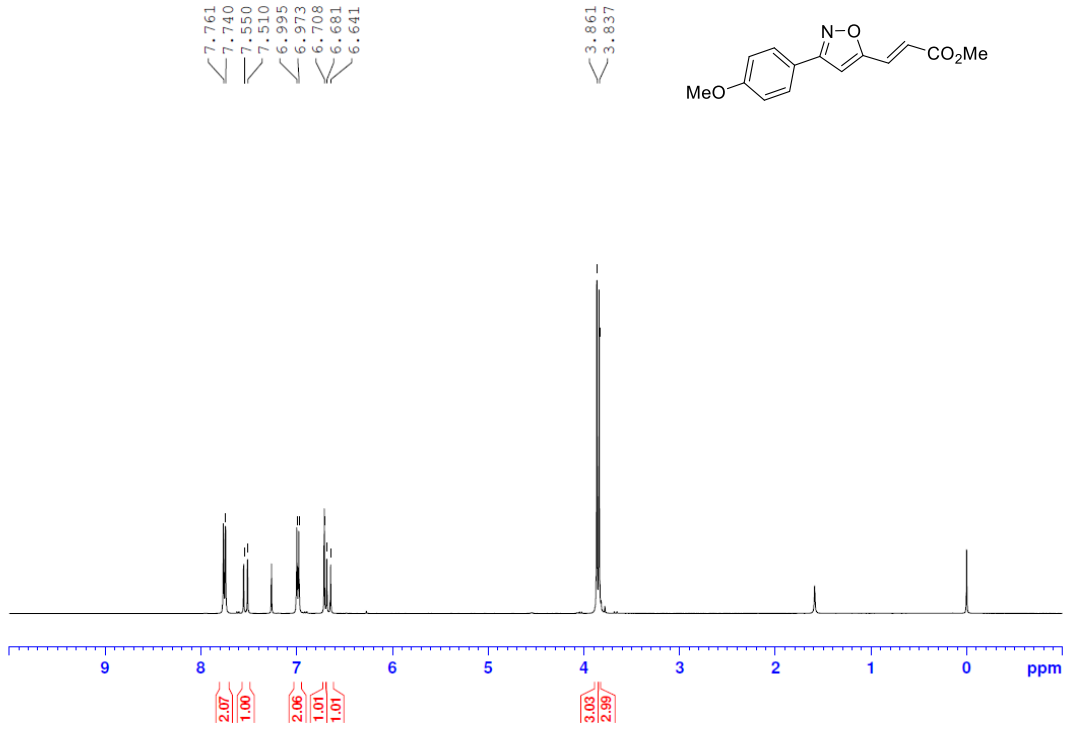


^{13}C NMR (125 MHz, $CDCl_3$)

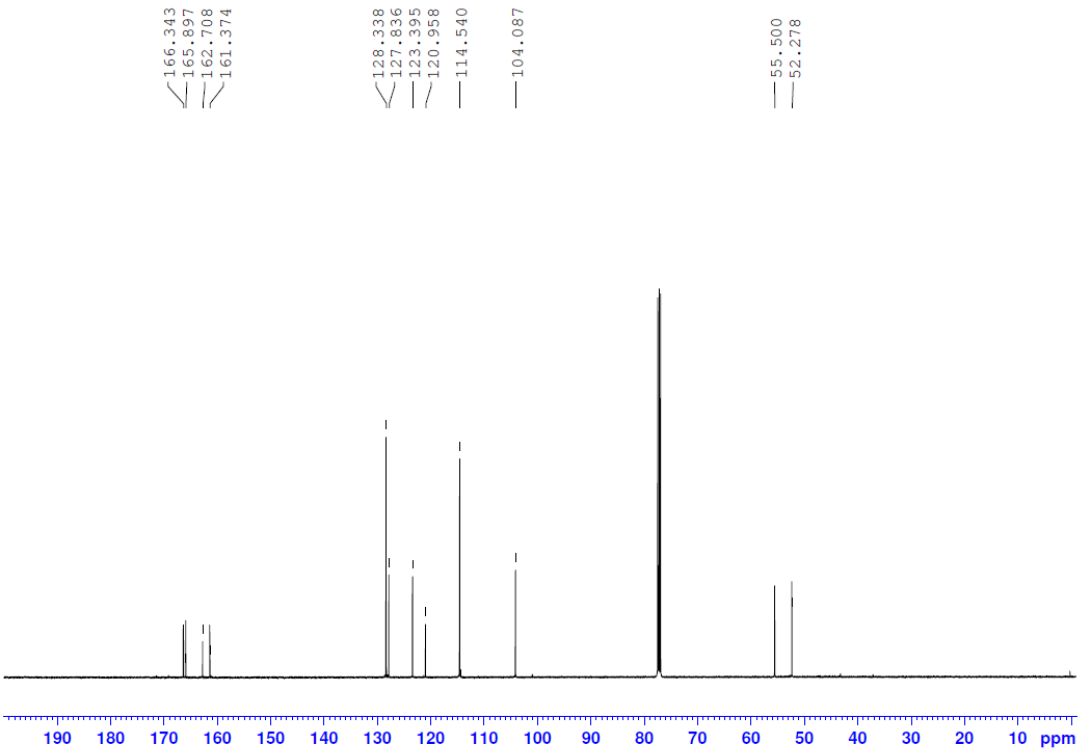


Methyl (*E*)-3-(3-(4-methoxyphenyl)isoxazol-5-yl)acrylate (3e)

^{1}H NMR (500 MHz, $CDCl_3$)

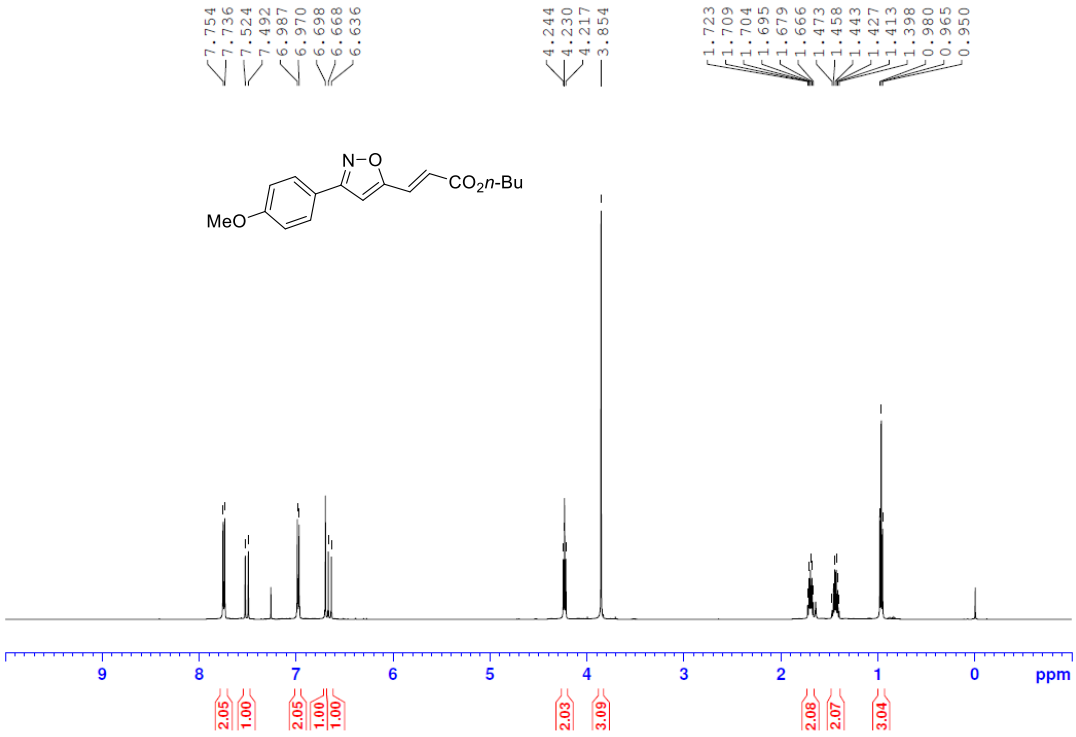


^{13}C NMR (125 MHz, $CDCl_3$)

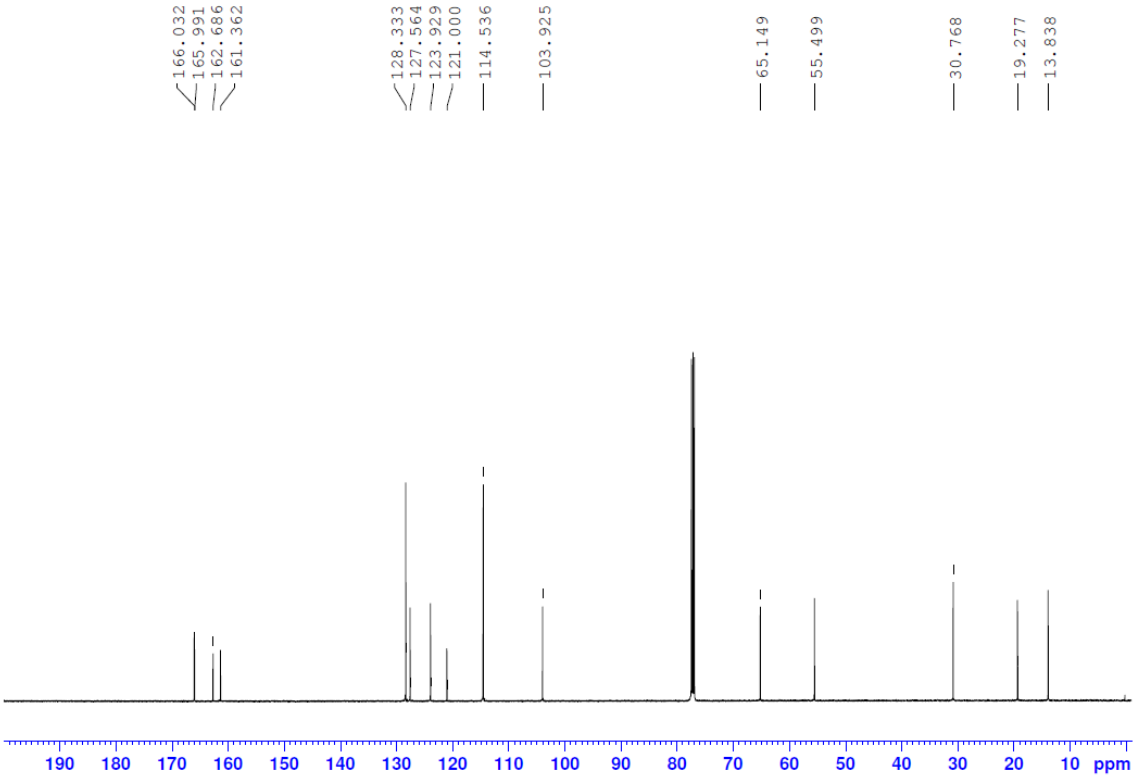


Butyl (*E*)-3-(3-(4-methoxyphenyl)isoxazol-5-yl)acrylate (3f)

^{1}H NMR (500 MHz, $CDCl_3$)

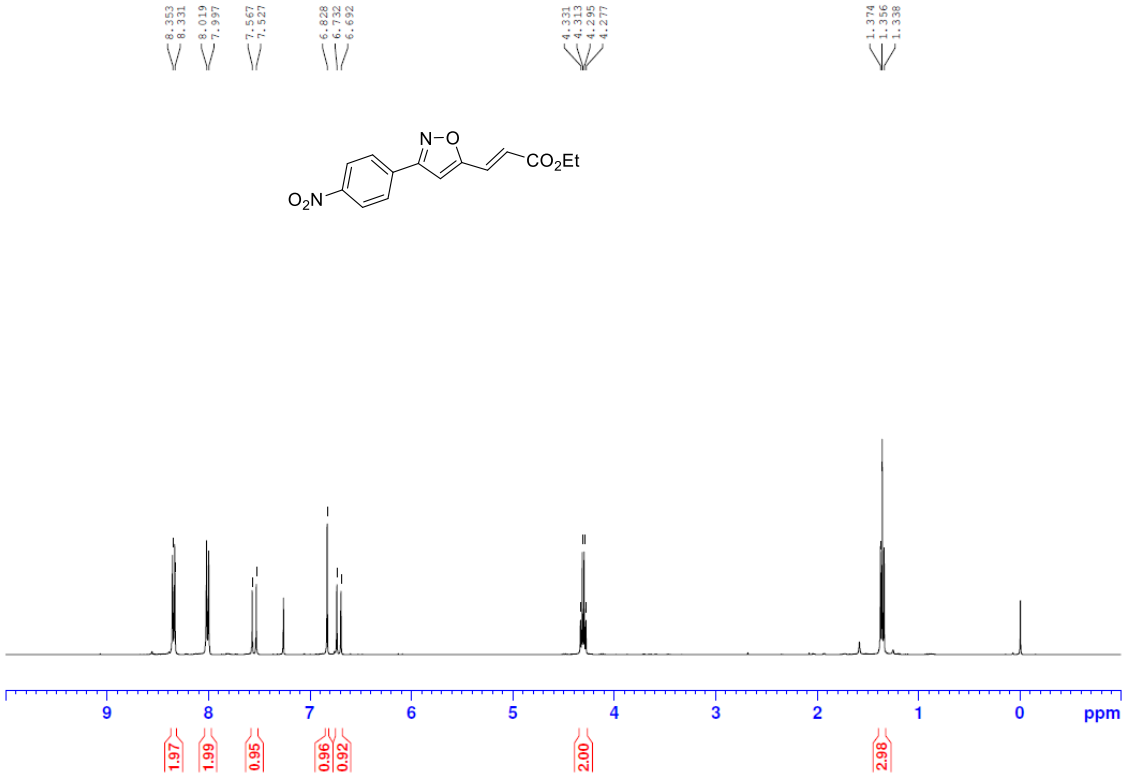


^{13}C NMR (125 MHz, $CDCl_3$)

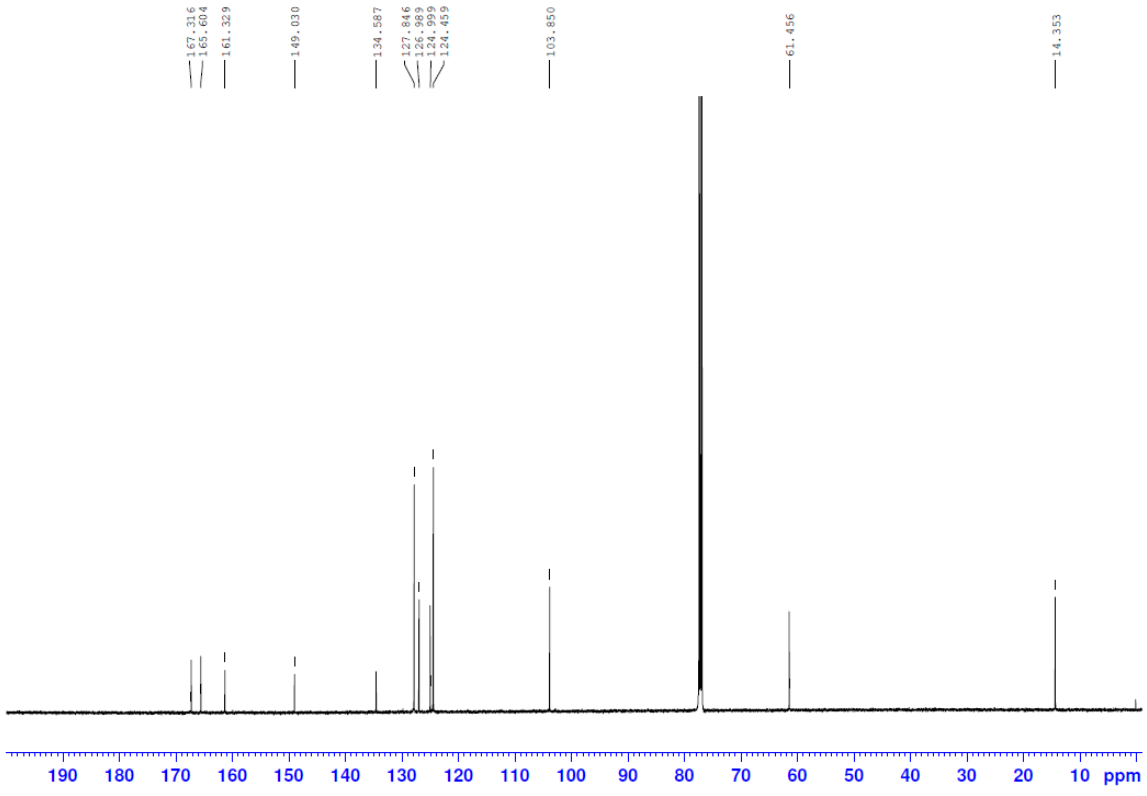


Ethyl (*E*)-3-(3-(4-nitrophenyl)isoxazol-5-yl)acrylate (3g)

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

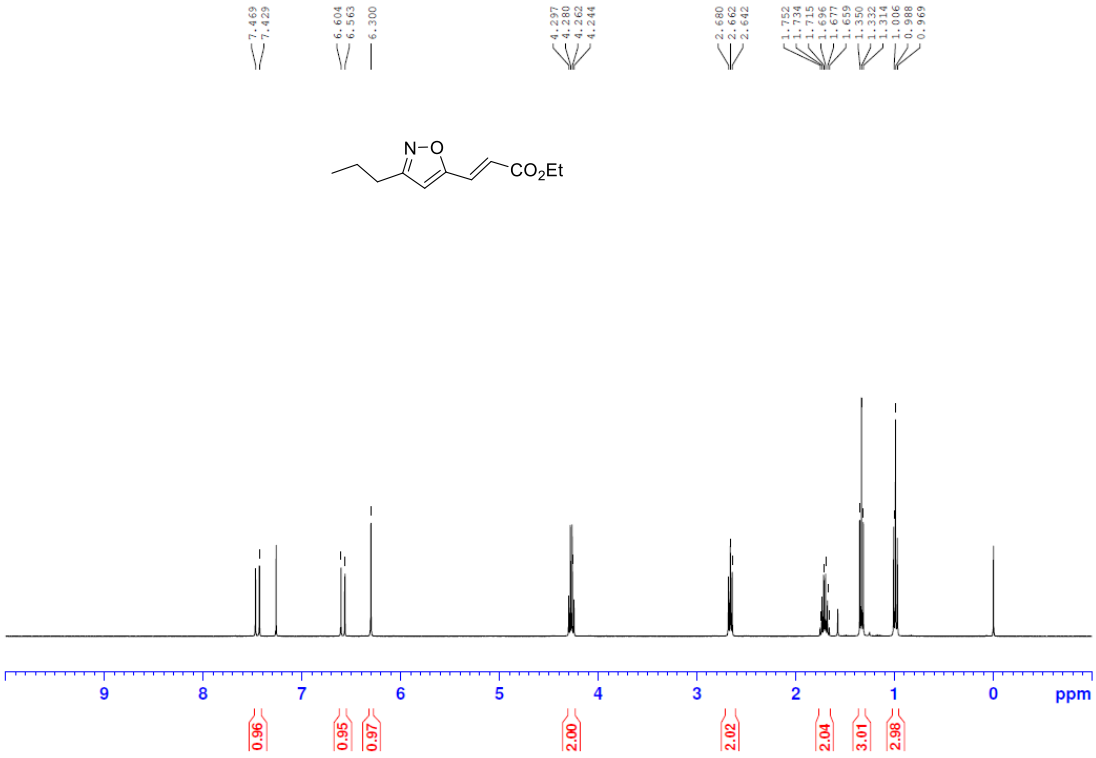


^{13}C NMR (125 MHz, $CDCl_3$)

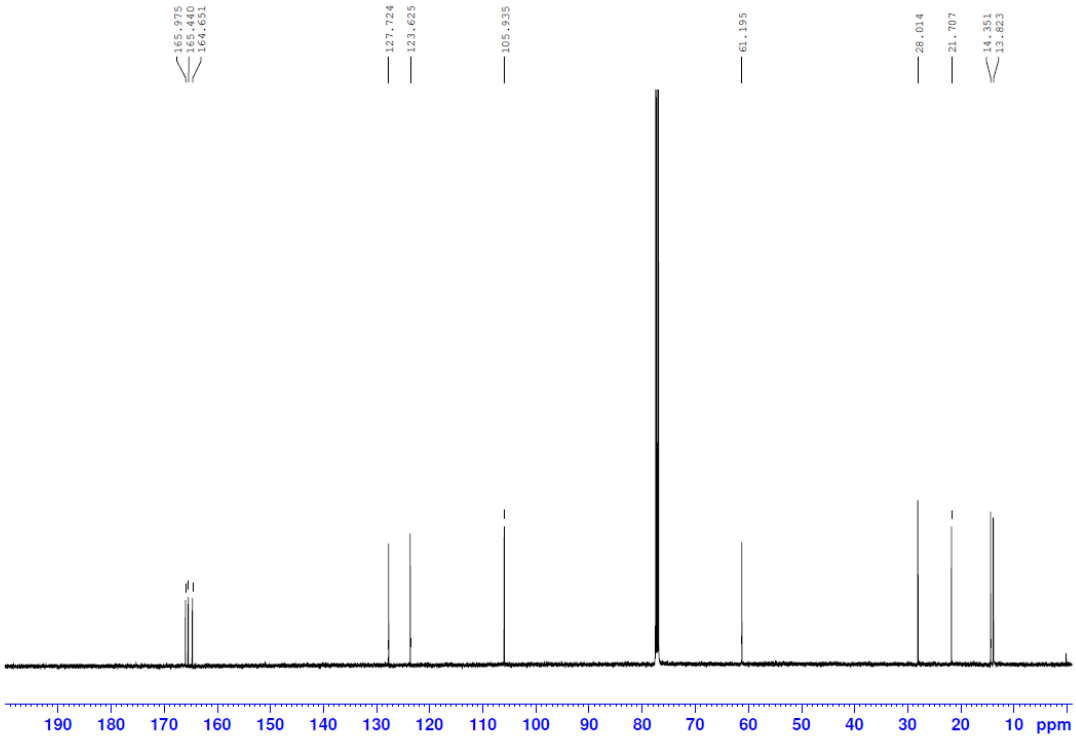


Ethyl (*E*)-3-(3-(4-propylphenyl)isoxazol-5-yl)acrylate (3h)

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

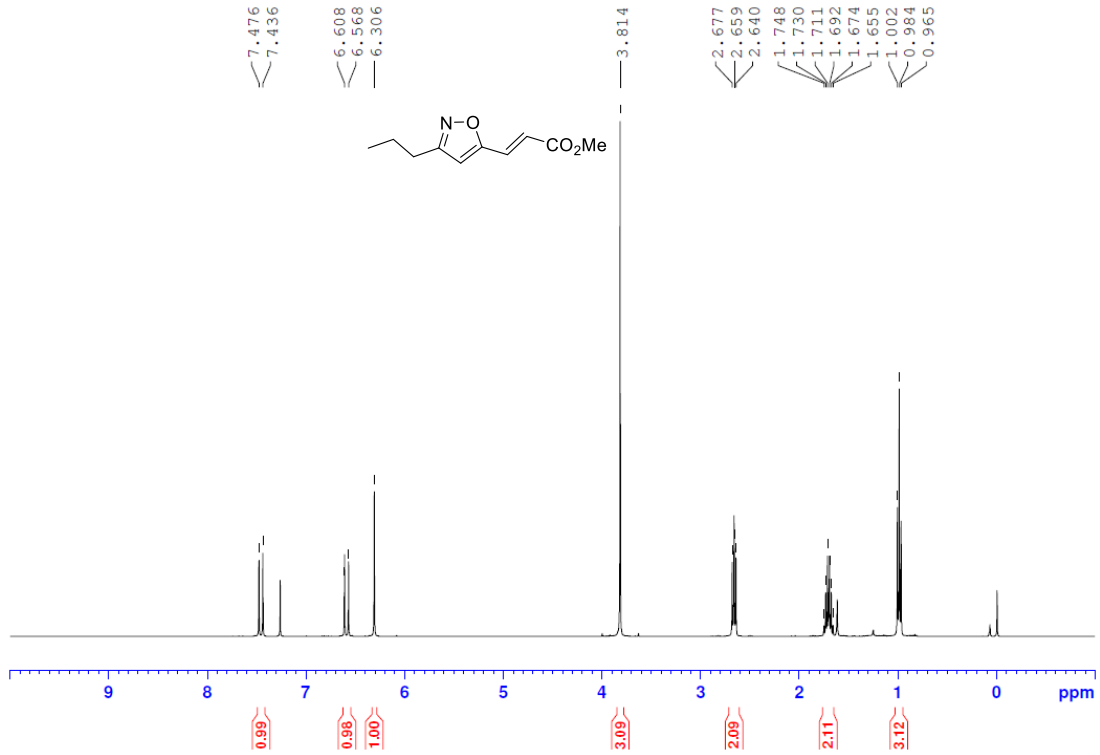


^{13}C NMR (125 MHz, $CDCl_3$)

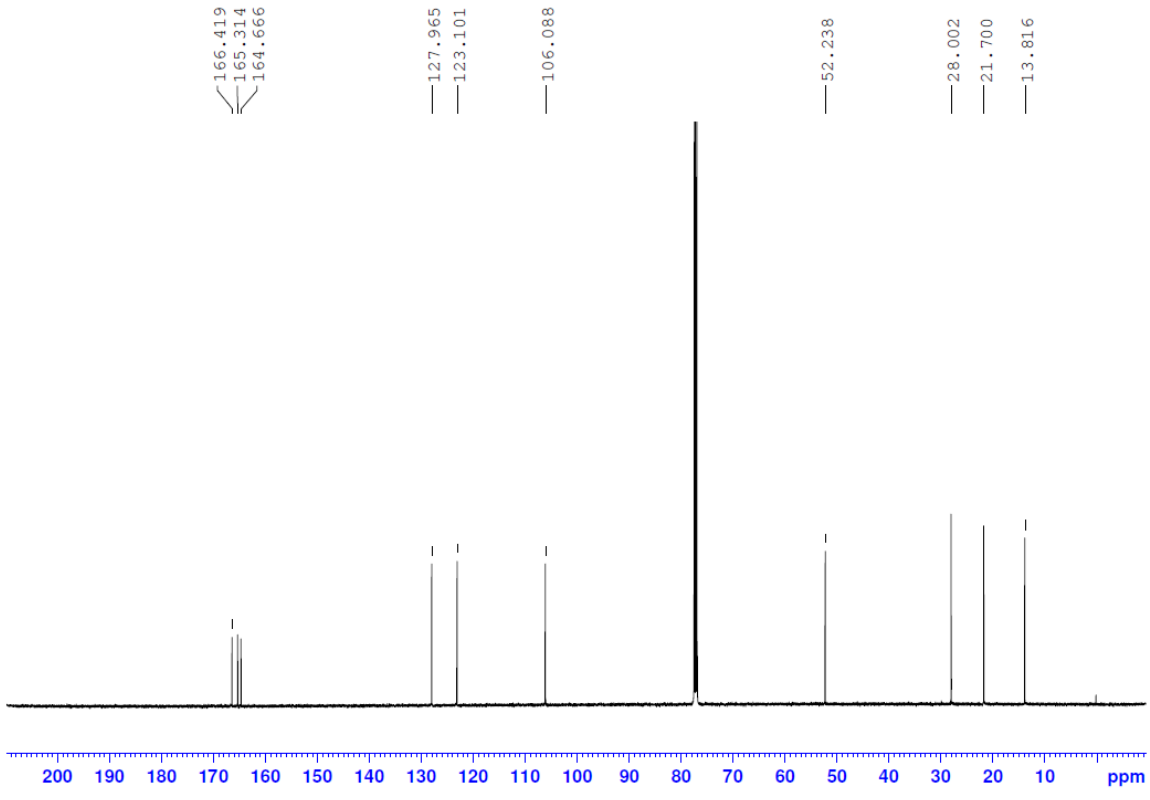


Methyl (*E*)-3-(3-propylisoxazol-5-yl)acrylate (3i)

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

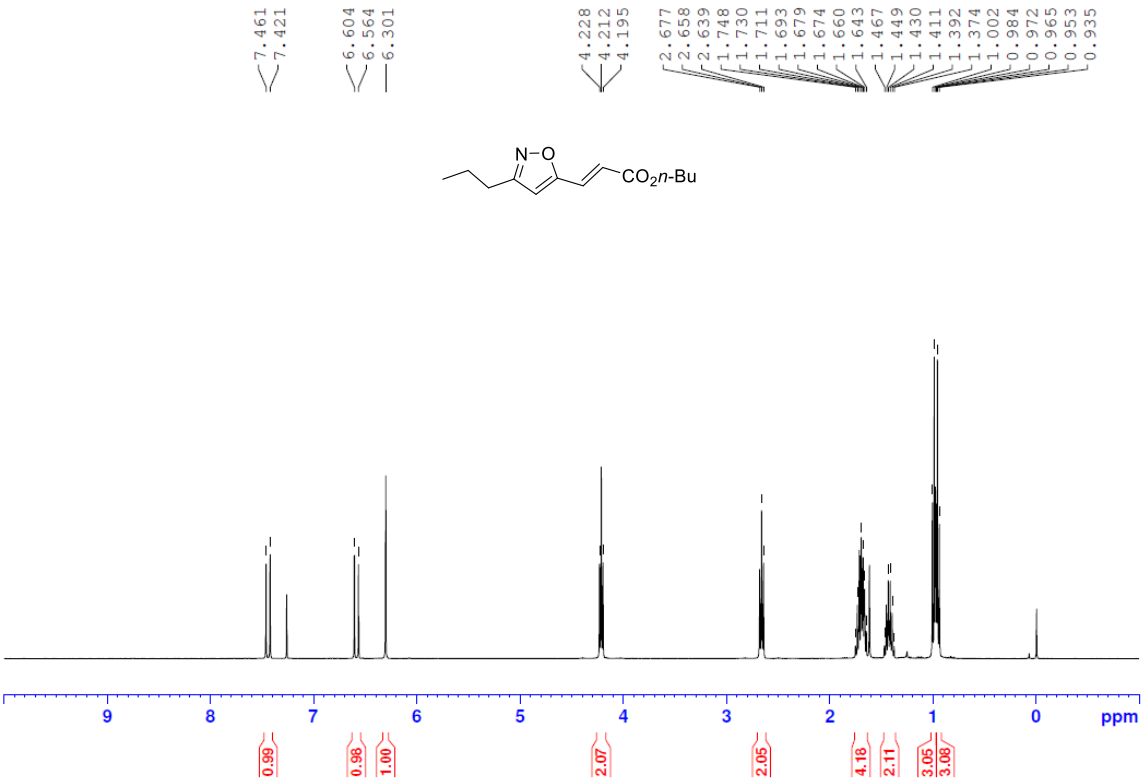


^{13}C NMR (125 MHz, $CDCl_3$)

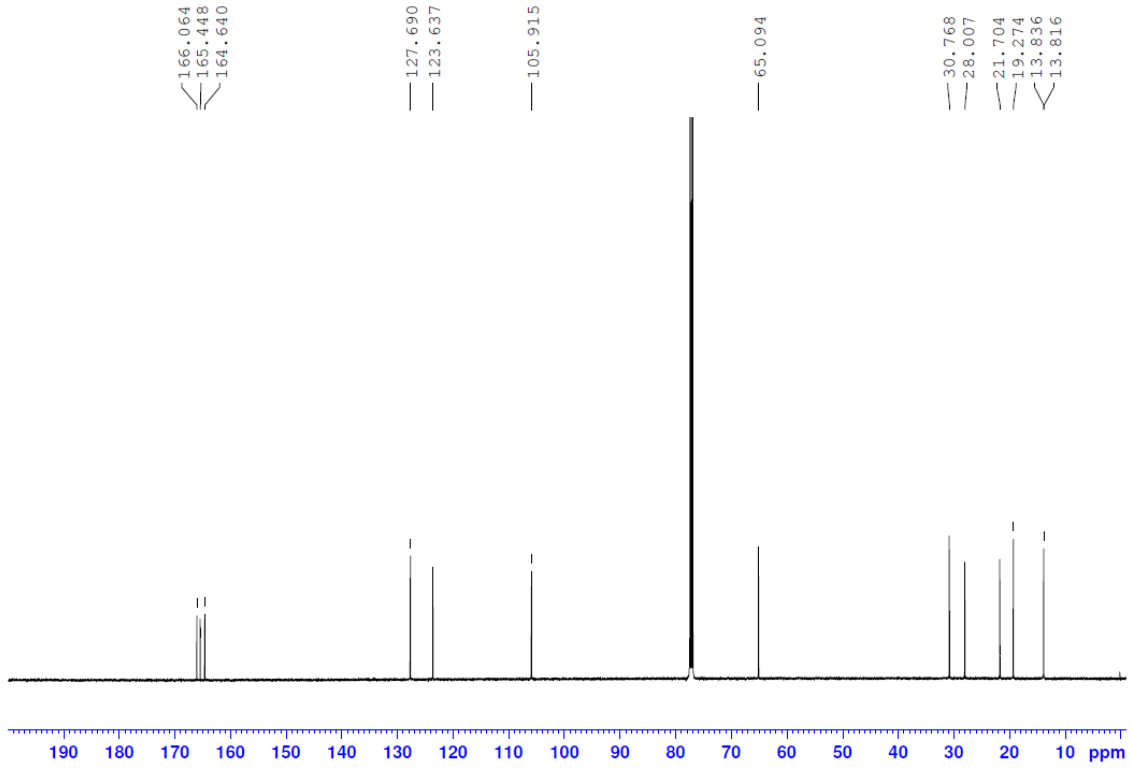


Butyl (*E*)-3-(3-propylisoxazol-5-yl)acrylate (3j)

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

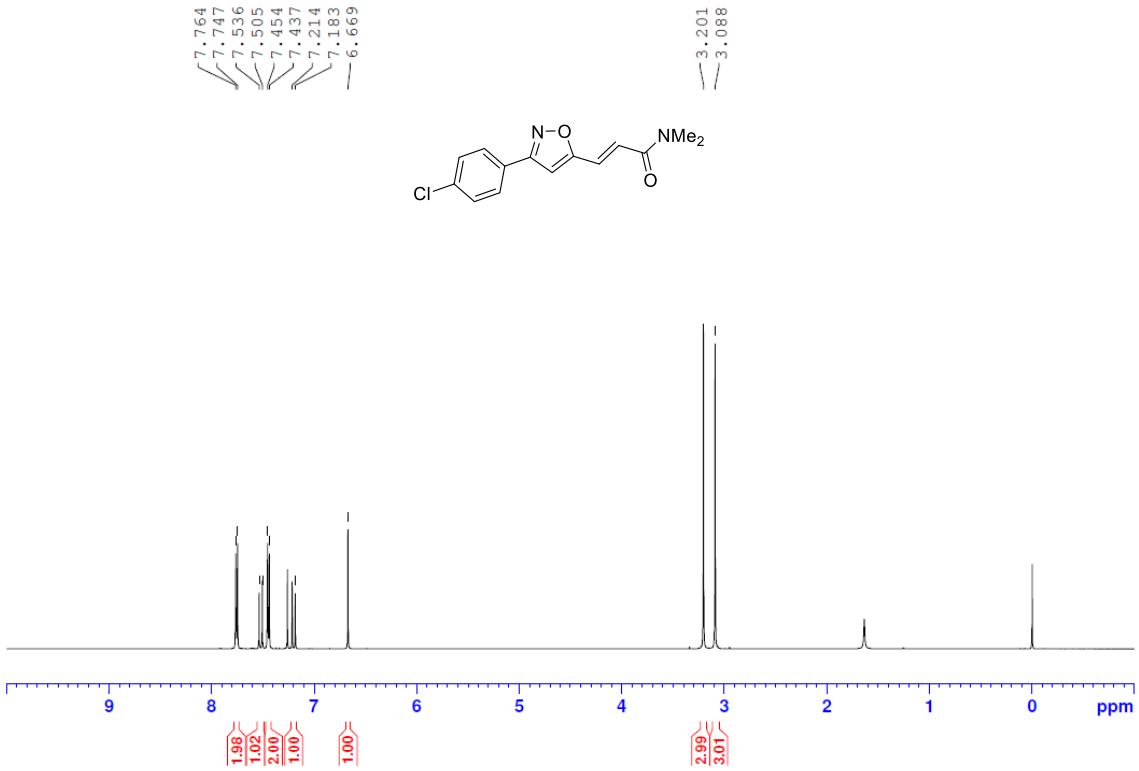


^{13}C NMR (125 MHz, $CDCl_3$)

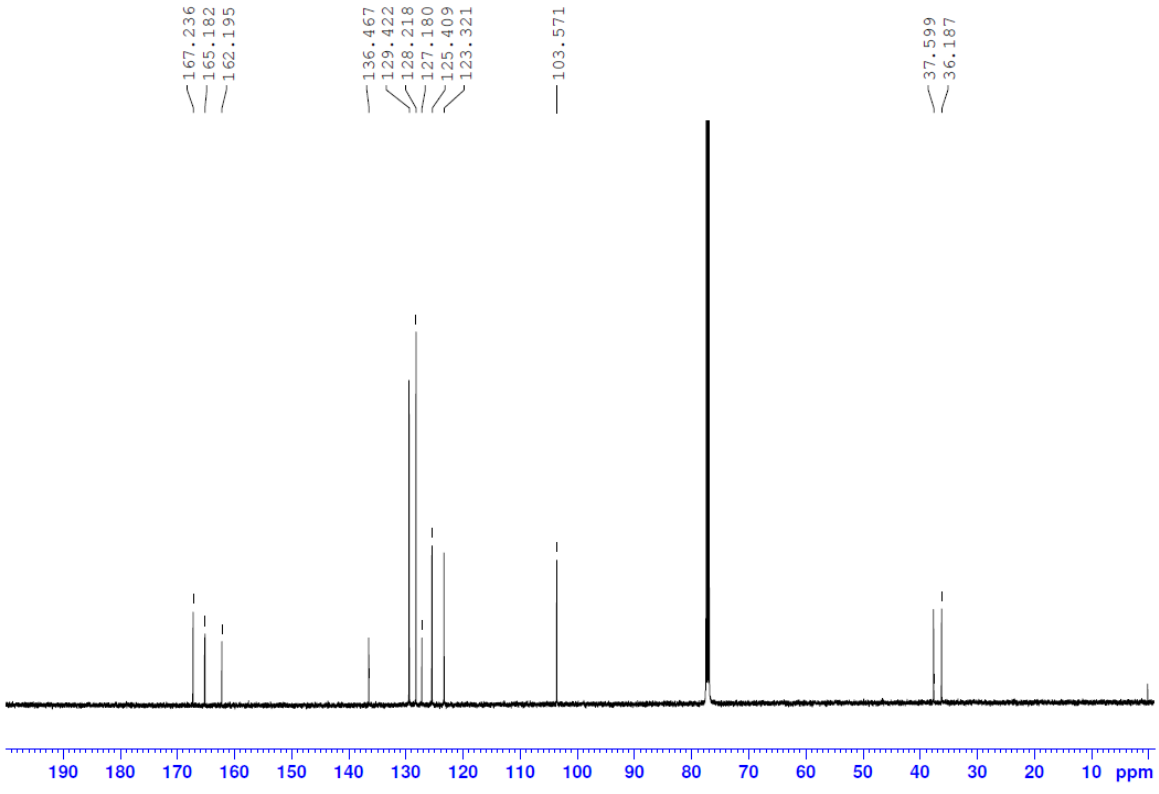


(*E*)-3-(3-(4-Chlorophenyl)isoxazol-5-yl)-*N*,*N*-dimethylacrylamide (3k)

^{1}H NMR (500 MHz, $CDCl_3$)

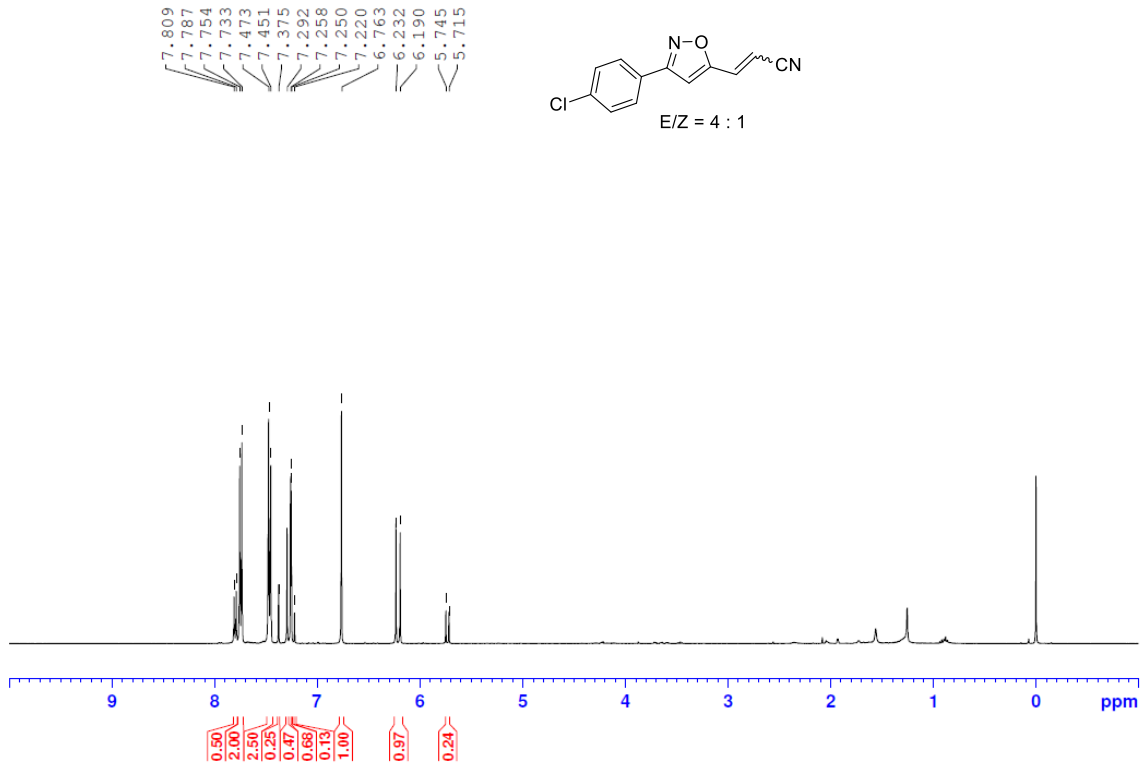


^{13}C NMR (125 MHz, $CDCl_3$)

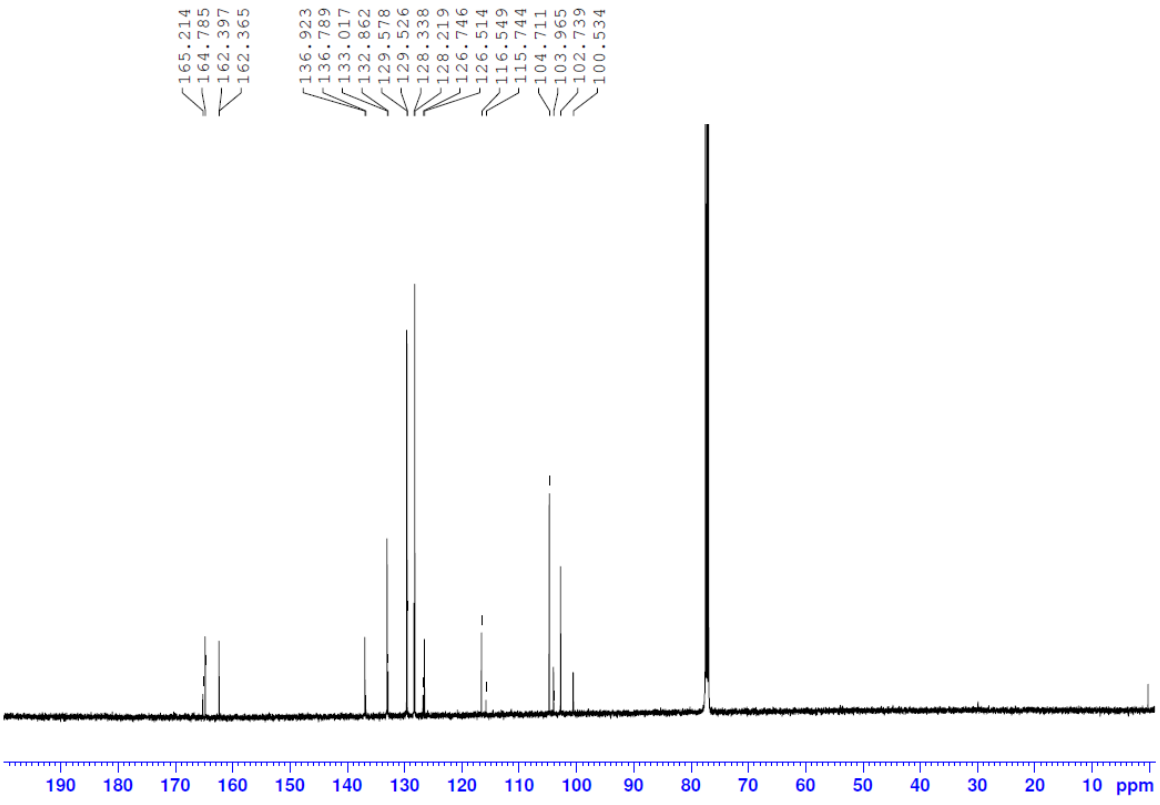


(*E*/*Z*)-3-(3-(4-Chlorophenyl)isoxazol-5-yl)acrylonitrile (3l)

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

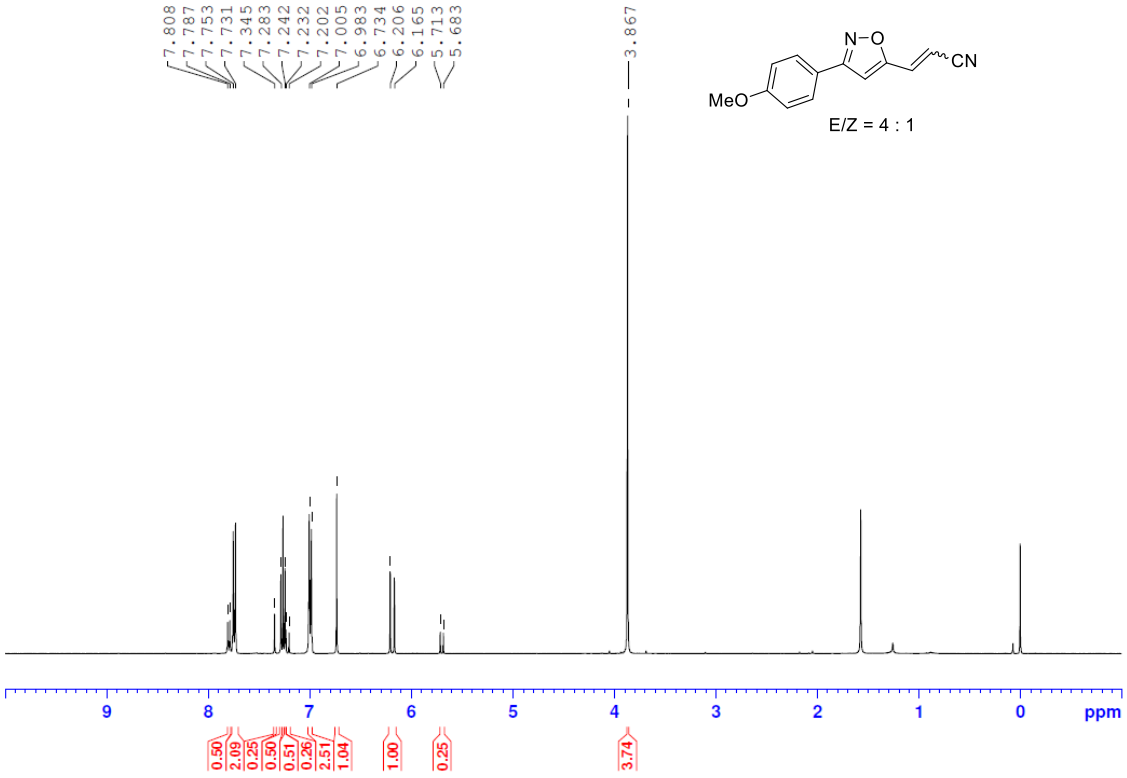


^{13}C NMR (125 MHz, $CDCl_3$)

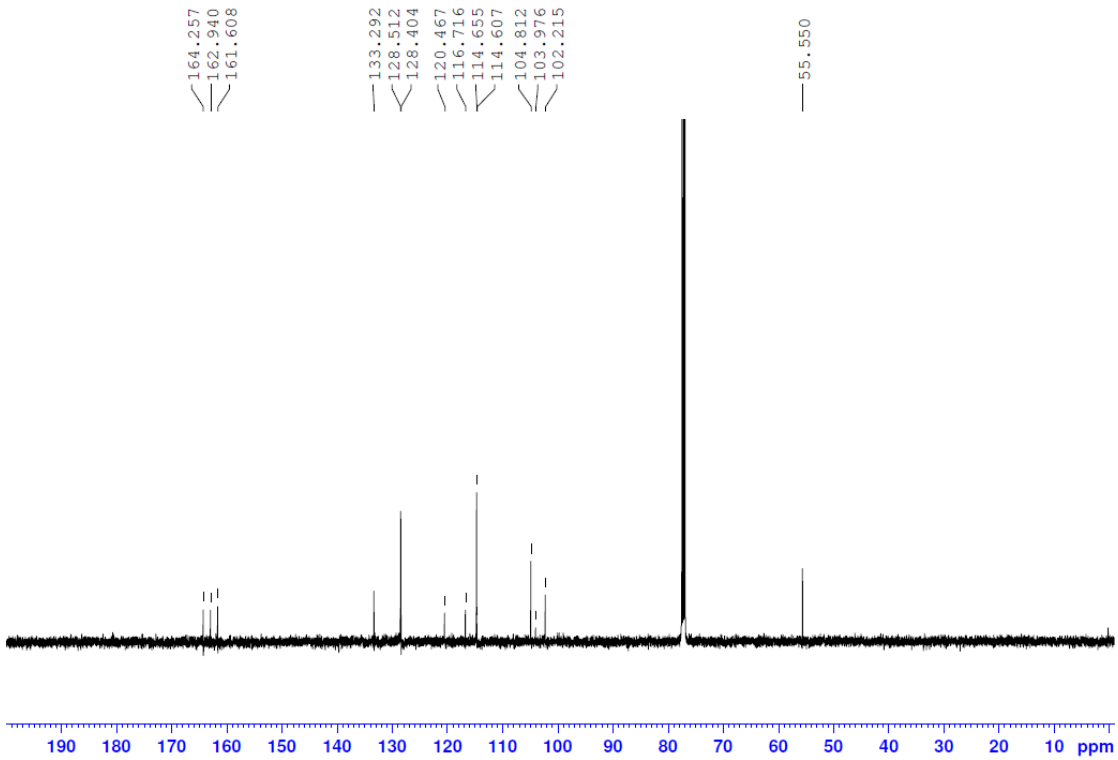


(*E*/*Z*)-3-(3-(4-Chlorophenyl)isoxazol-5-yl)acrylonitrile (3m)

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

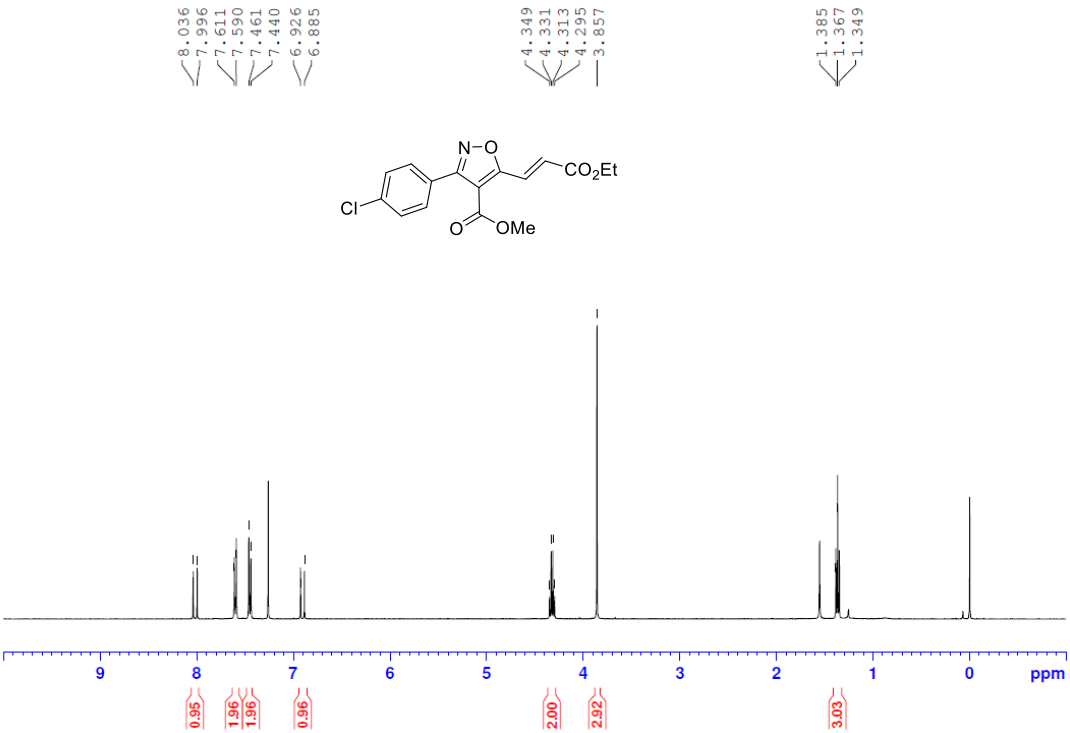


^{13}C NMR (125 MHz, $CDCl_3$)

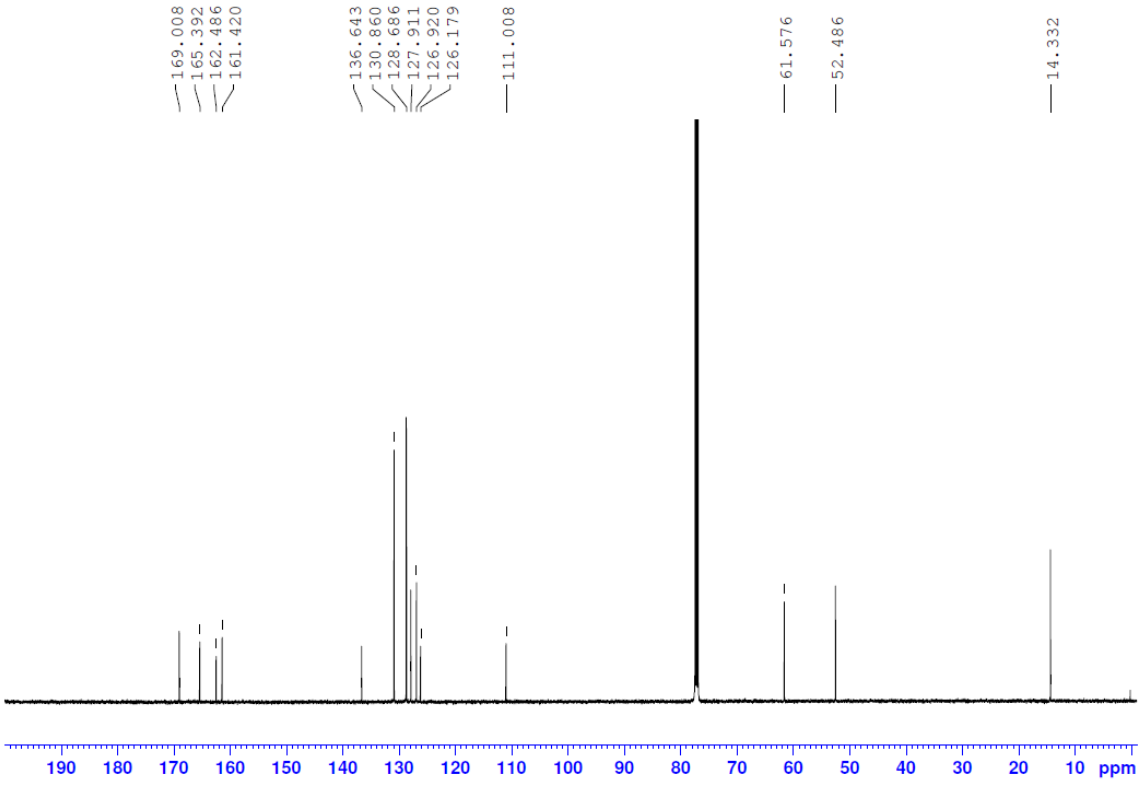


Methyl (*E*)-3-(4-chlorophenyl)-5-(3-ethoxy-3-oxoprop-1-en-1-yl)isoxazole-4-carboxylate (6)

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

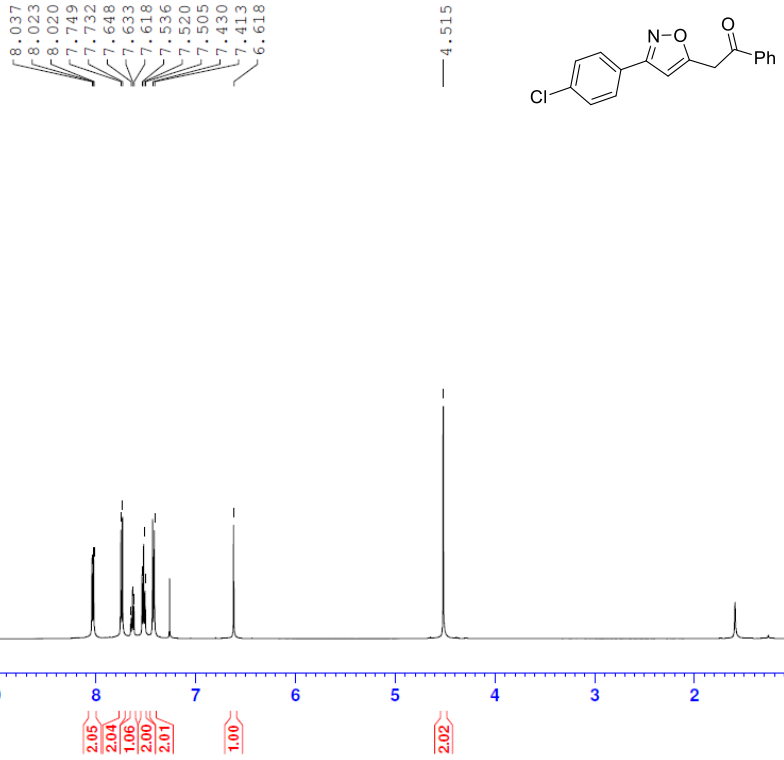


^{13}C NMR (125 MHz, $CDCl_3$)

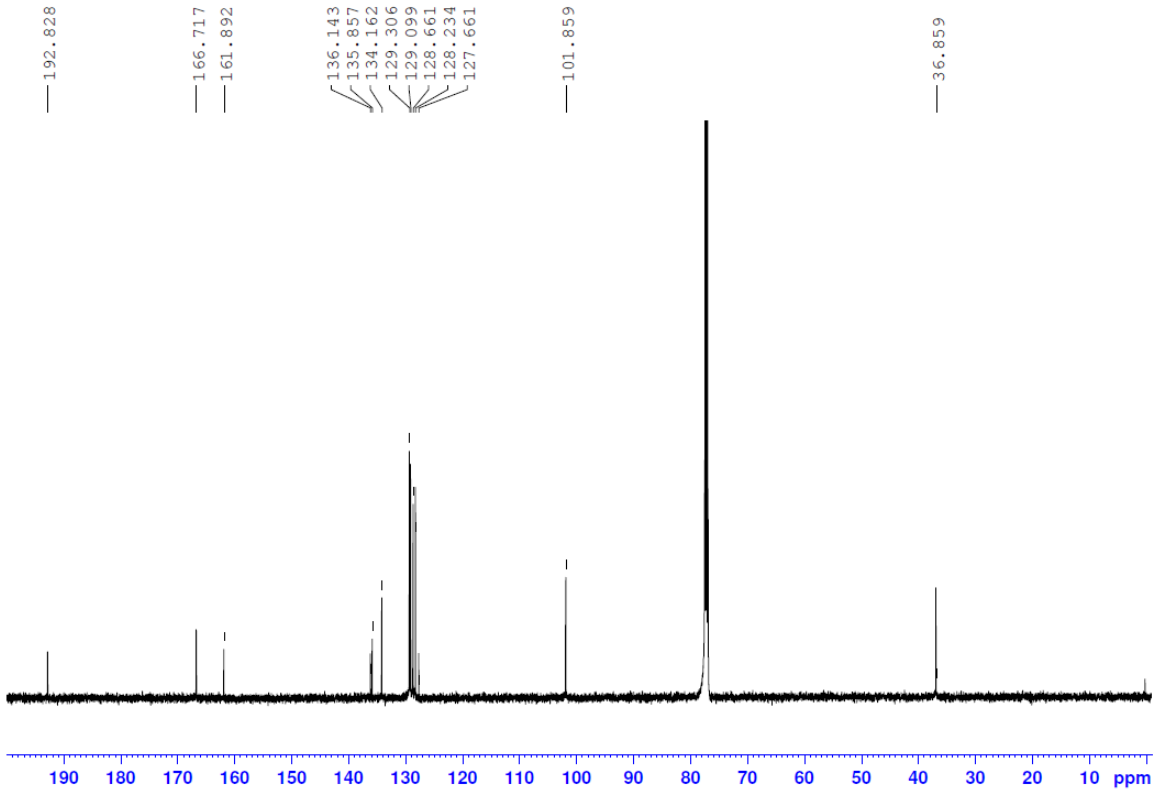


2-(3-(4-Chlorophenyl)isoxazol-5-yl)-1-phenylethan-1-one (5a)

^{1}H NMR (500 MHz, $CDCl_3$)

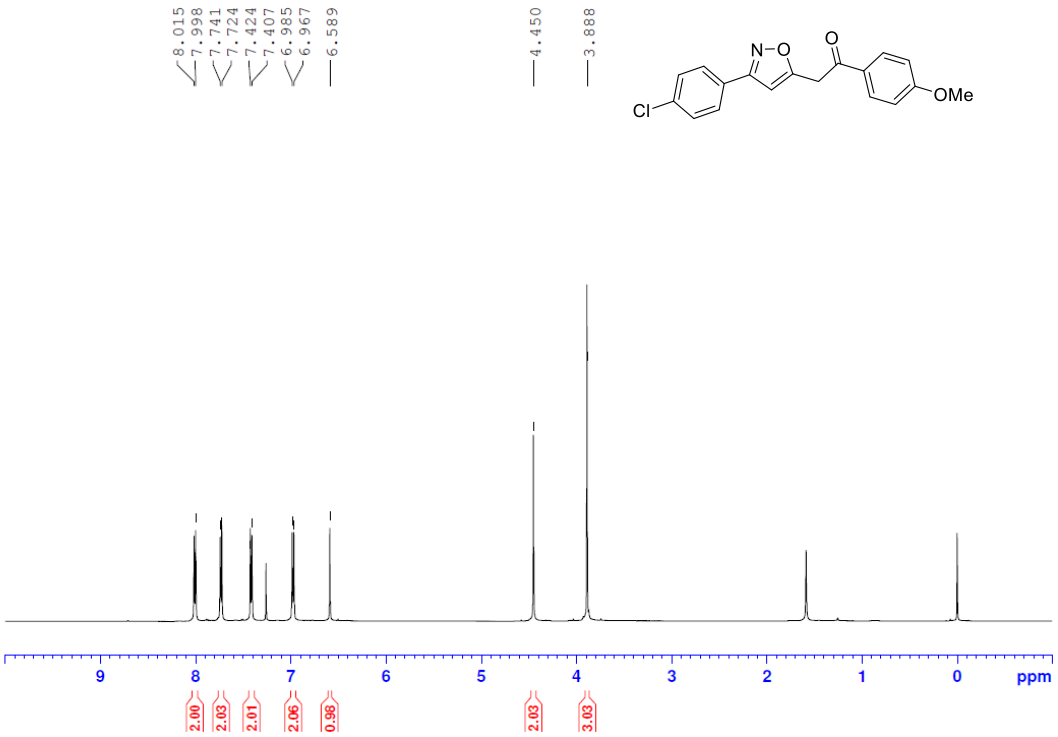


^{13}C NMR (125 MHz, $CDCl_3$)

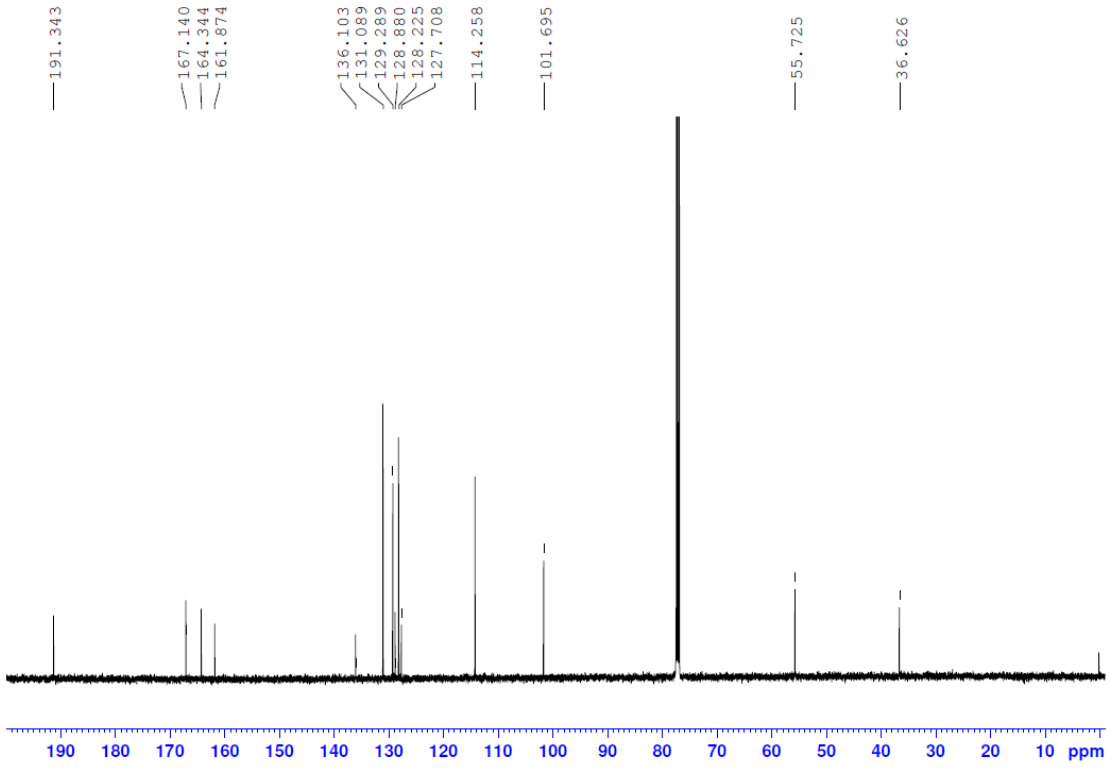


2-(3-(4-Chlorophenyl)isoxazol-5-yl)-1-(4-methoxyphenyl)ethan-1-one (5b)

^{1}H NMR (500 MHz, $CDCl_3$)

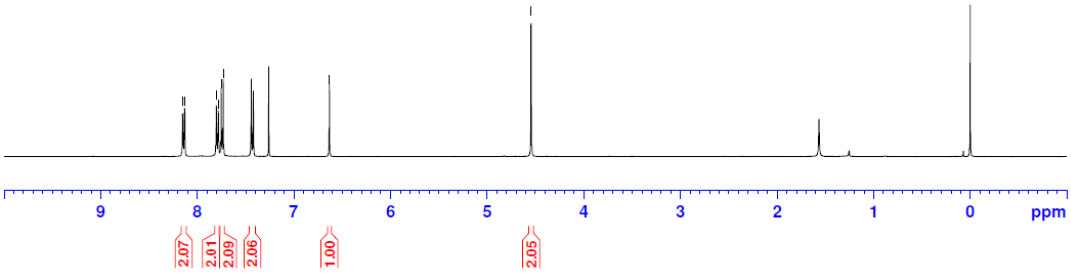


^{13}C NMR (125 MHz, $CDCl_3$)

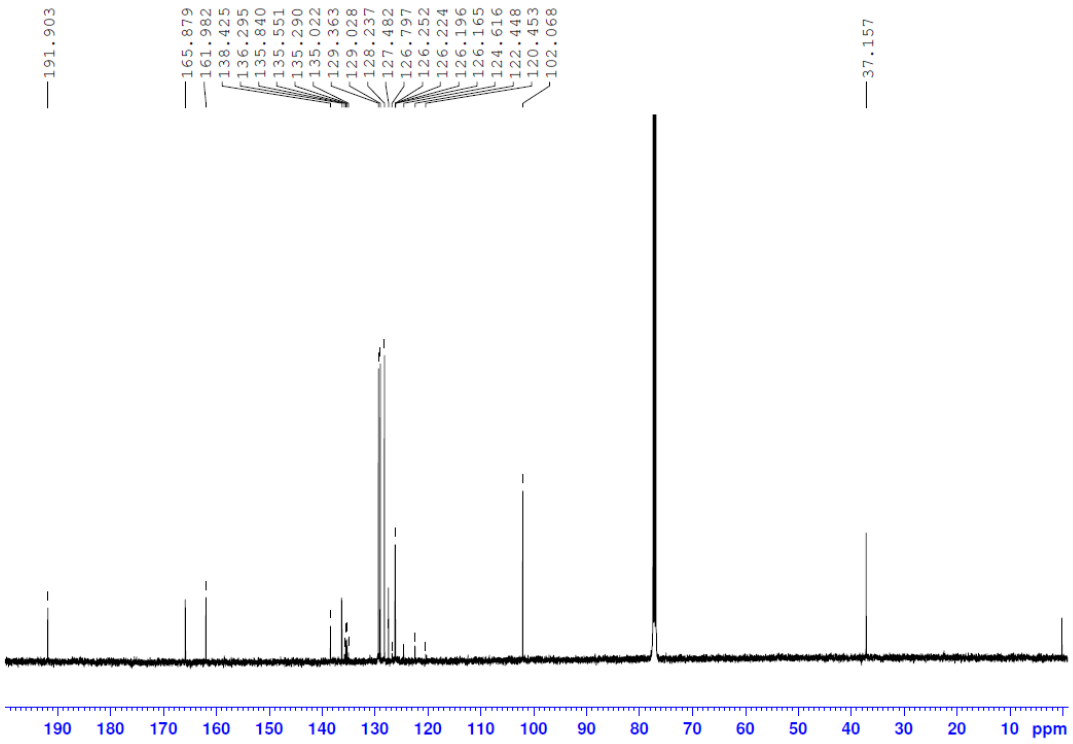


2-(3-(4-Chlorophenyl)isoxazol-5-yl)-1-(4-(trifluoromethyl)phenyl)ethan-1-one (5c)

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

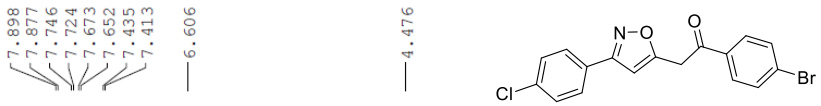


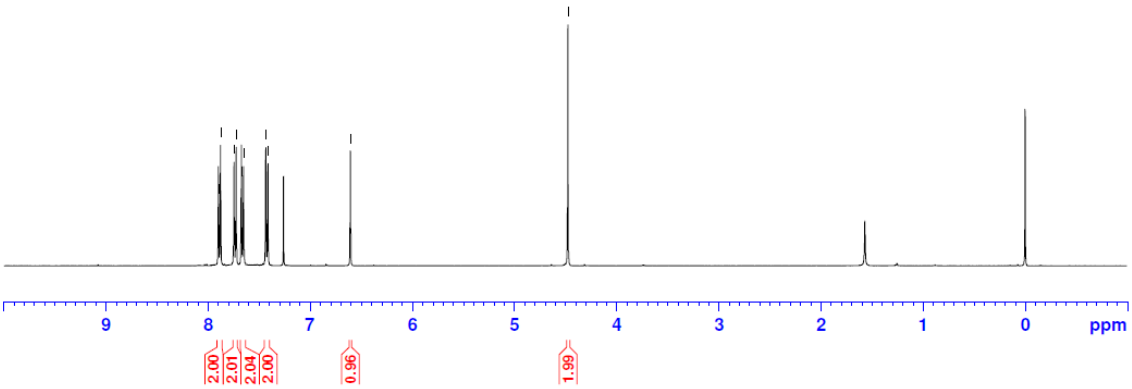
^{13}C NMR (125 MHz, $CDCl_3$)



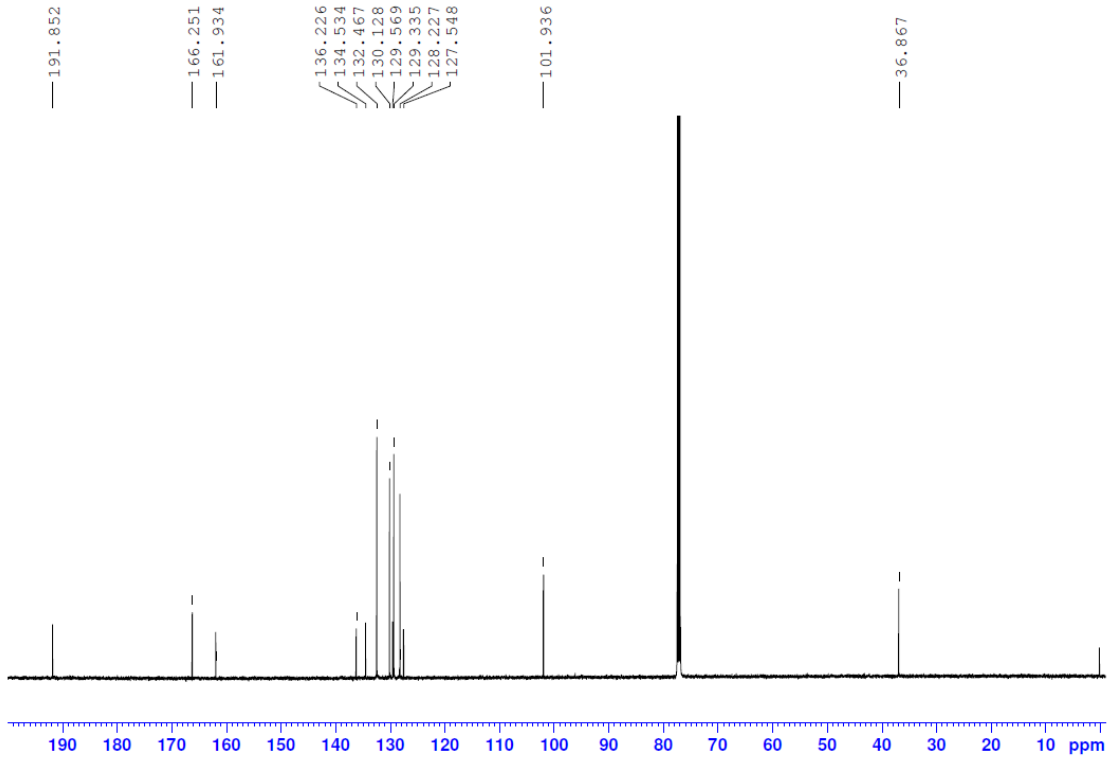
1-(4-Bromophenyl)-2-(3-(4-chlorophenyl)isoxazol-5-yl)ethan-1-one (5d)

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



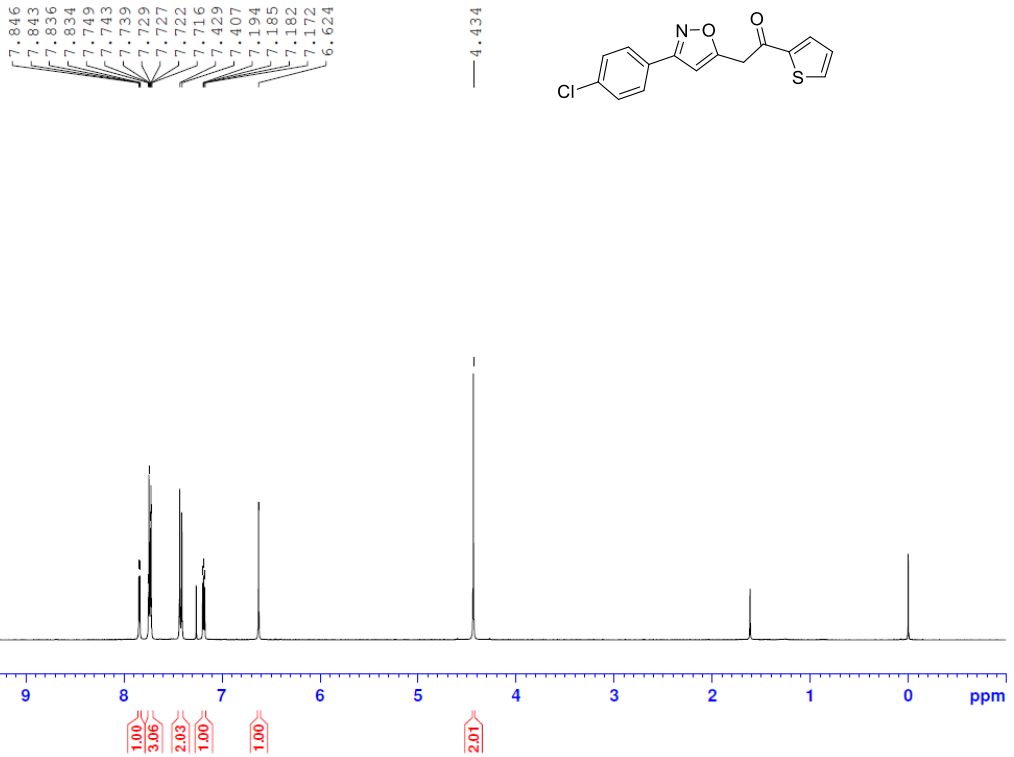


^{13}C NMR (125 MHz, $CDCl_3$)

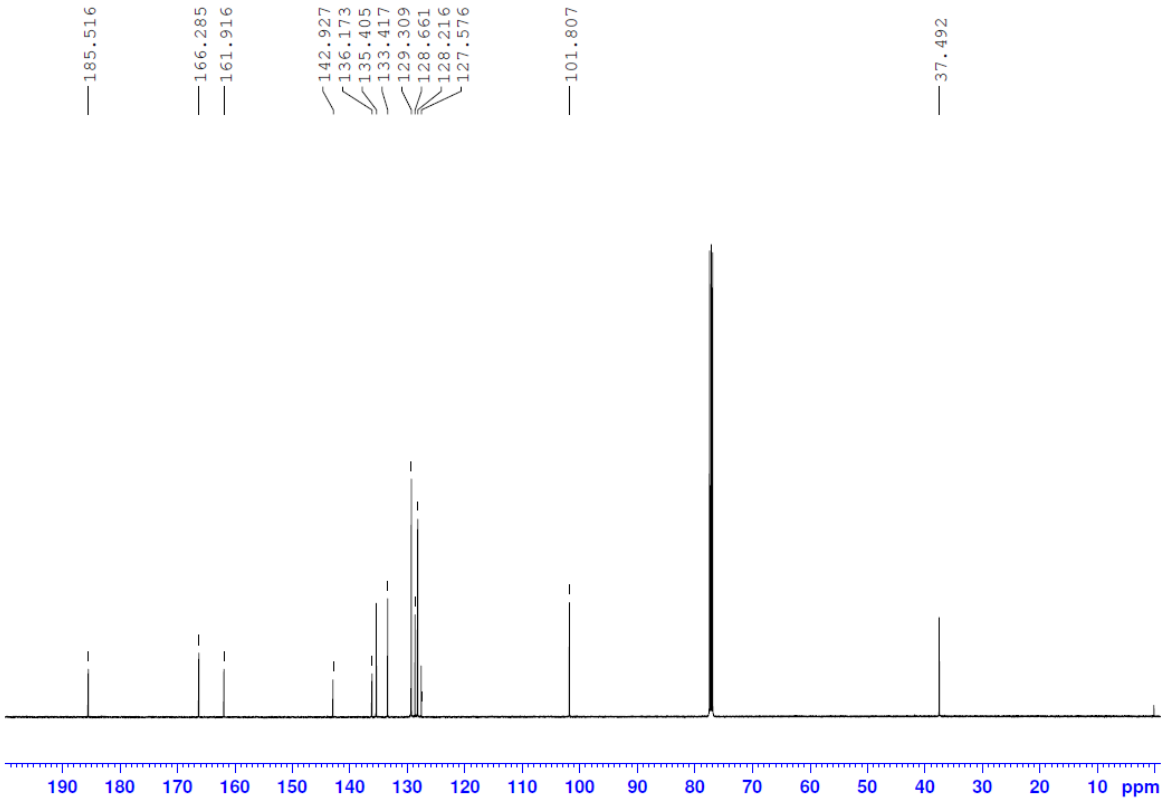


2-(3-(4-Chlorophenyl)isoxazol-5-yl)-1-(thiophen-2-yl)ethan-1-one (5e)

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

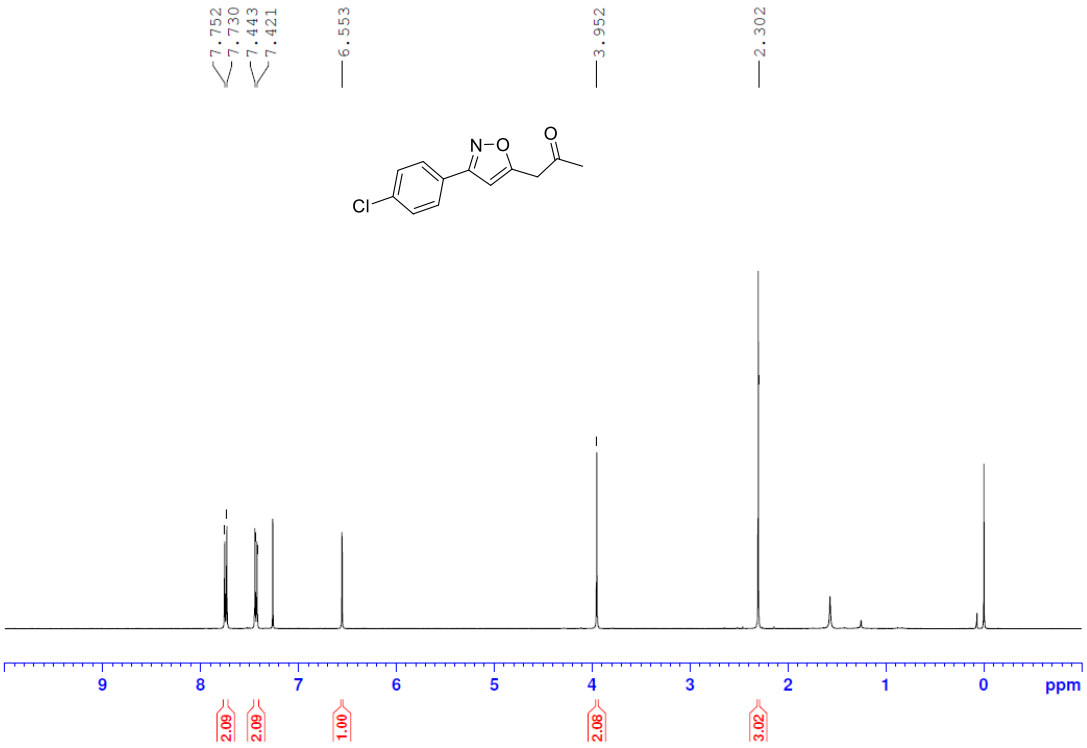


^{13}C NMR (125 MHz, $CDCl_3$)

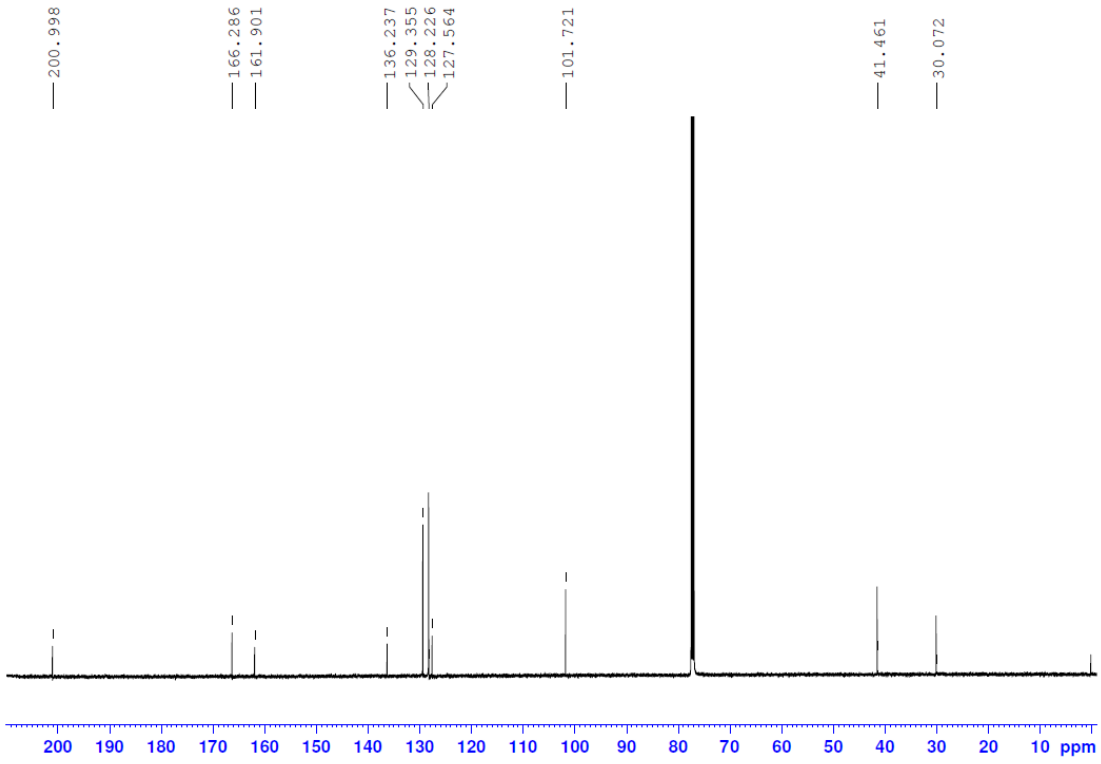


1-(3-(4-Chlorophenyl)isoxazol-5-yl)propan-2-one (5f)

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

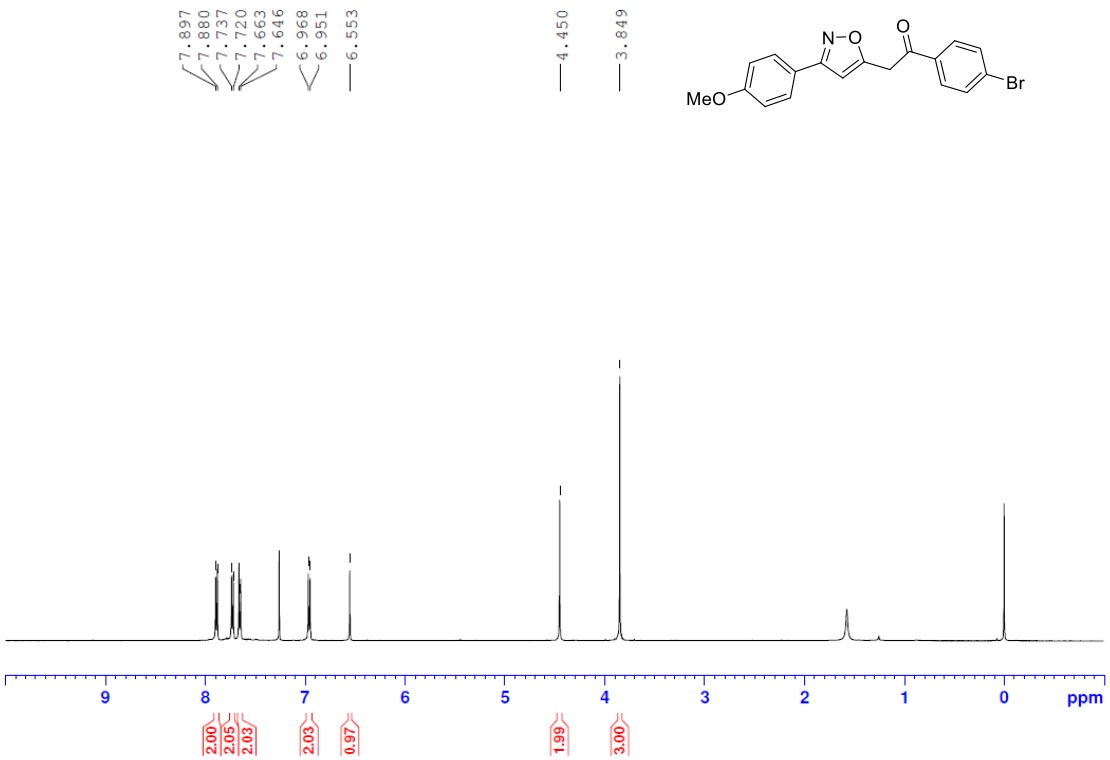


^{13}C NMR (125 MHz, $CDCl_3$)

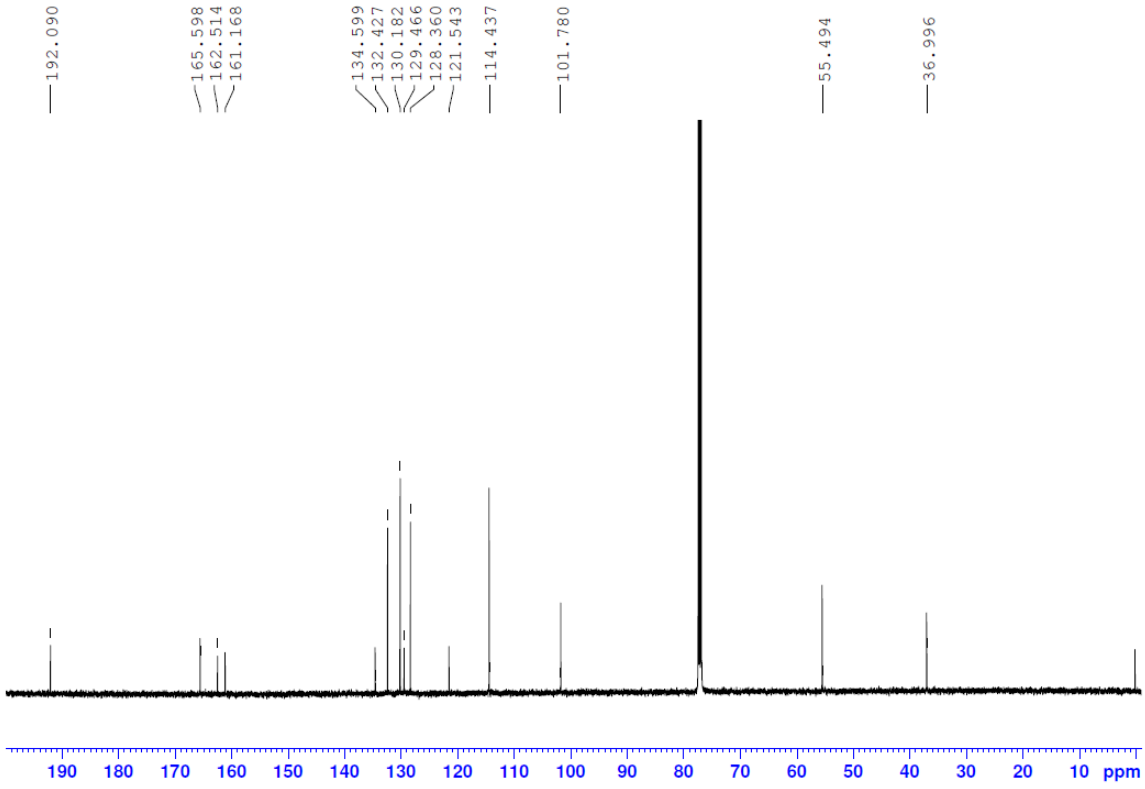


1-(4-Bromophenyl)-2-(3-(4-methoxyphenyl)isoxazol-5-yl)ethan-1-one (5g)

^{1}H NMR (500 MHz, $CDCl_3$)

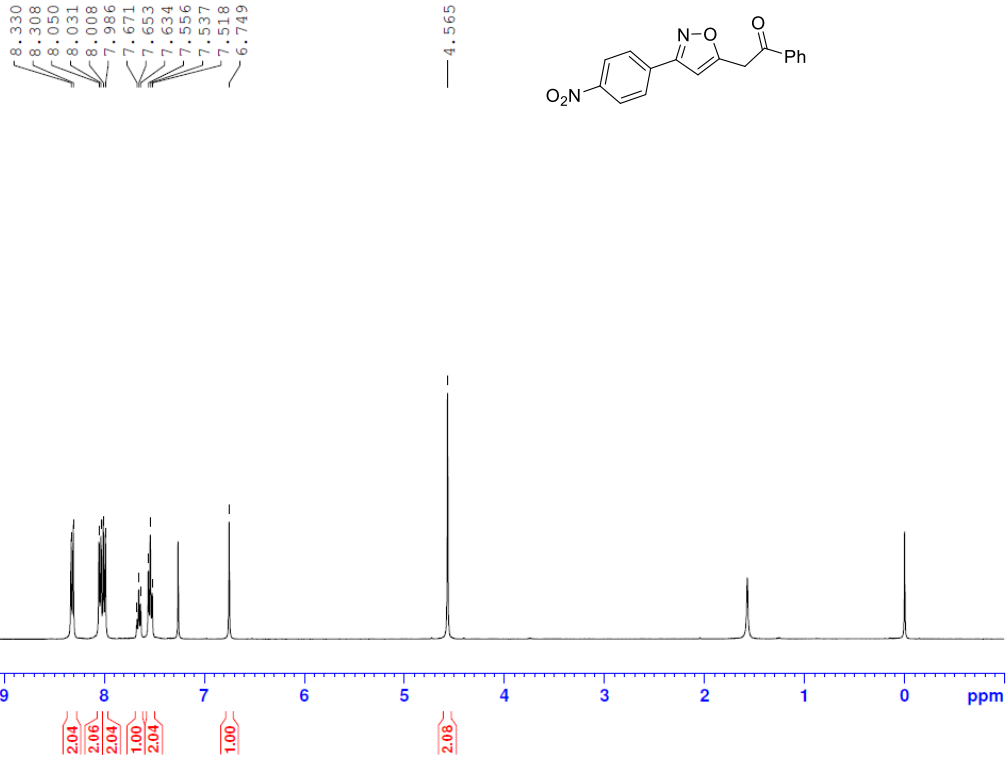


^{13}C NMR (125 MHz, $CDCl_3$)

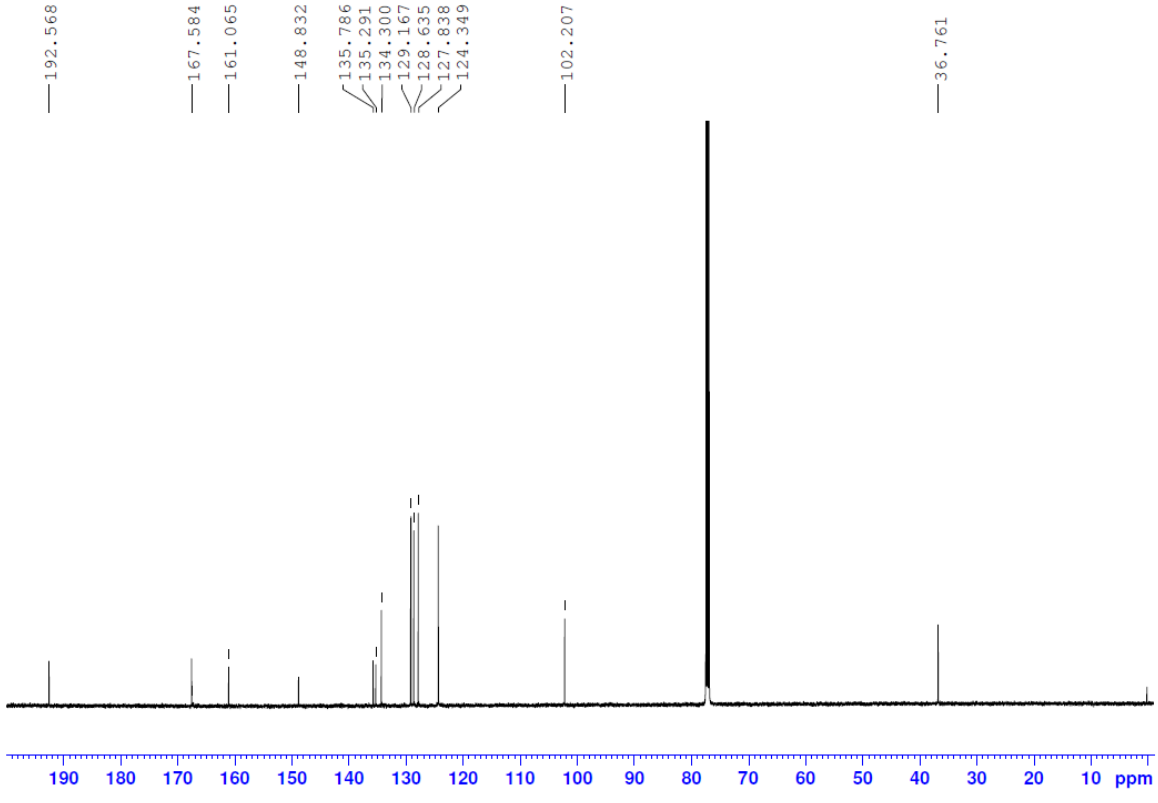


2-(3-(4-Nitrophenyl)isoxazol-5-yl)-1-phenylethan-1-one (5h)

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

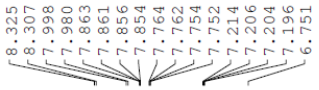


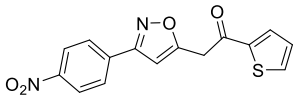
^{13}C NMR (125 MHz, $CDCl_3$)

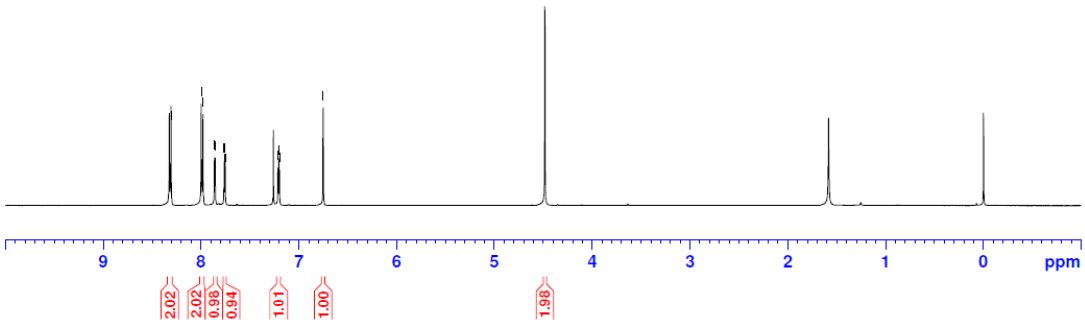


2-(3-(4-Nitrophenyl)isoxazol-5-yl)-1-(thiophen-2-yl)ethan-1-one (5i)

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







^{13}C NMR (125 MHz, $CDCl_3$)