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Supporting Information

Tetra-Substituted Furans by a Gold-Catalysed Tandem C(sp^3)–H Alkynylation/Oxy-Alkynylation Reaction

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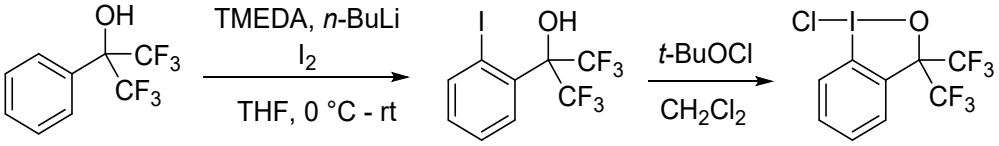
1. General Methods

Reactions were performed in oven-dried glassware unless otherwise noted, chemicals were obtained from commercial suppliers (Sigma-Aldrich, Alfa Aesar and TCI) and used without further purification. Deuterated solvents were bought from Euriso-Top. NMR spectra were, if not mentioned otherwise, recorded at room temperature on the following spectrometers: Bruker Avance-III-300, Bruker Avance III 400, and Bruker Avance-III-500. ^{1}H NMR spectra were recorded in $CDCl_3$ and referenced to residual $CHCl_3$ at 7.26 ppm. Multiplicities were reported using the following abbreviations: s (singlet), brs (broad singlet), d (doublet), t (triplet), q (quartet), m (multiple). All ^{13}C NMR spectra were measured with ^{1}H-decoupling. The multiplicities mentioned in these spectra [s (singlet, quaternary carbon), d (doublet, CH-group), t (triplet, CH_2-group), q (quartet, CH_3-group)] were determined by DEPT135 spectra. (MS and HRMS) were determined at the chemistry department of the University of Heidelberg under the direction of Dr. J. Gross. EI^+-spectra were measured on a JOEL JMS-700 spectrometer. For ESI^+-spectra a Bruker ApexQu FT-ICR-MS spectrometer was applied. Infrared Spectroscopy (IR) was processed on an FT-IR Bruker (IF528), IR Perkin Elmer (283) or FT-IR Bruker Vector 22. The solvent or matrix is denoted in brackets. For the most significant bands the wave number ν (cm^{-1}) is given. X-ray crystal structure analyses were measured at the chemistry department of the University of Heidelberg under the direction of Dr. F. Rominger on a Bruker Smart CCD or Bruker APEX-II CCD instrument using Mo-Kα-radiation. Diffraction intensities were corrected for Lorentz and polarization effects. An empirical absorption correction was applied using SADABS based on the Laue symmetry of reciprocal space. Hydrogen atoms were either isotropically refined or calculated. The structures were solved and refined by Dr. F. Rominger using the SHELXTL software package. Melting Points were measured in open glass capillaries in a Büchi melting point apparatus (according to Dr. Tottoli) and were not calibrated. Flash Column Chromatography was accomplished using Silica gel 60 (0.04 - 0.063 mm / 230 - 400 mesh ASTM) purchased from Macherey-Nagel or Aluminium oxide (neutral or basic) purchased from Macherey-Nagel. As eluents, mixtures of petroleum ether (PE), ethyl acetate (EA) were used. Analytical Thin Layer Chromatography (TLC) was carried out on precoated Macherey-Nagel POLYGRAM® SIL G/UV254 or POLYGRAM® ALOX N/UV254 plastic sheets. Detection was accomplished using UV-light (254 nm), $KMnO_4$ (in 1.5 M Na_2CO_3 (aq.)). IUPAC names of the compounds described in the experimental section were determined with the program ACDLabs 12.0®.

2. Experiment Procedures

Procedure A: Preparation of **2**

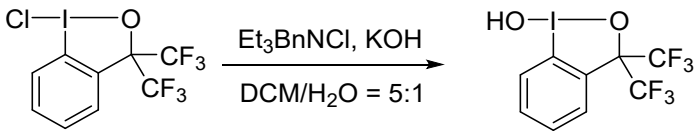


Under argon, TMEDA (4 mmol, 0.2 equiv) was added to a solution of *n*-BuLi (2.5 M in hexane, 44 mmol, 2.2 equiv). After 15 min, the cloudy solution was cooled to 0 °C and 1,1,1,3,3,3-hexafluoro-2-phenylpropan-2-ol (20 mmol, 1 equiv) in THF (3 mL) was added dropwise. The reaction was stirred at 0 °C for 30 min and then at room temperature overnight. I_2 (22 mmol, 1.1 equiv) in THF (10 mL) was added at 0 °C and the mixture was stirred at 0 °C for 30 min and room temperature for 4 h. The reaction was quenched with saturated NH_4Cl (aq). Ethyl acetate was added and the layers were separated. The aqueous layer was then extracted twice with ethyl acetate. The organic layers were combined, washed twice with saturated $Na_2S_2O_3$ (aq), dried over Na_2SO_4, and filtered. The resulting solvent was evaporated under the reduced pressure to afford 1,1,1,3,3,3-hexafluoro-2-(2-iodophenyl)propan-2-ol as a brown oil which was used without further purification.

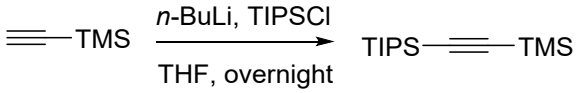
The crude product was dissolved in CH_2Cl_2 (20 mL) under air. *t*-BuOCl (21 mmol, 1.05 equiv) was then added dropwise at 0 °C. The resulting suspension was stirred under room temperature for 30 min. Then, the reaction mixture was filtered and washed with CH_2Cl_2 to afford in 45% yield as a yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09 (d, J = 8.5 Hz, 1H), 7.89 – 7.80 (m, 1H), 7.77 – 7.70 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 133.8 (d), 132.1 (s), 131.6 (d), 129.7 (m), 128.5 (d), 122.9 (q, $^1J_{C\text{-}F}$ = 289.6 Hz), 113.4 (s), 85.2 (m). IR (reflection) $\tilde{\nu}$ = 3100, 1738, 1593, 1564, 1462, 1442, 1289, 1263, 1237, 1193, 1155, 1136, 1119, 1102, 1043, 1007, 969, 950, 765, 757, 727, 690, 682, 667 cm^{-1}. The spectroscopic data is in agreement with that previously reported.[1]

***t*-BuOCl**

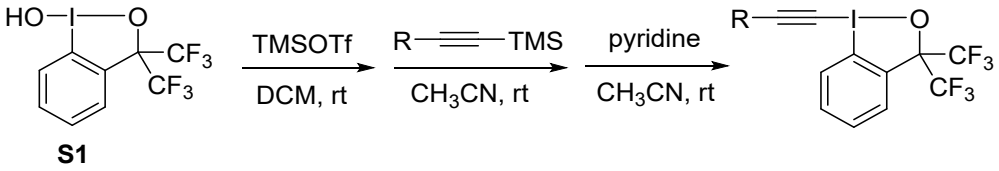
tert-Butyl alcohol (100 mmol) was dissolved in AcOH (6 mL) and cooled to 0 °C. To this reaction mixture an 12 % aqueous solution of sodium hypochlorite (130 mL) was added. After 10 min the organic phase was separated, washed with sat. $NaHCO_3$ (3 x 10 mL) and brine (10 mL) and dried over $CaCl_2$. The product was obtained as a yellow liquid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.33 (s, 9H). The spectroscopic data is in agreement with that previously reported.[2]



Under air, to a stirred solution of previous chemical (10 mmol, 1 equiv) in CH_2Cl_2 (20 mL) were added Et_3BnNCl (0.5 mmol, 0.05 equiv) and KOH (10 mmol, 1 equiv) in water (4 mL). After stirring at room temperature for 12 h, the resulting suspension was filtered and washed with CH_2Cl_2 to afford desirable product in 74% yield as a white solid. ^{1}H NMR (300 MHz, DMSO-d_6) δ 8.03 – 7.85 (m, 2H), 7.78 – 7.69 (m, 2H). The spectroscopic data is in agreement with that previously reported.[1]



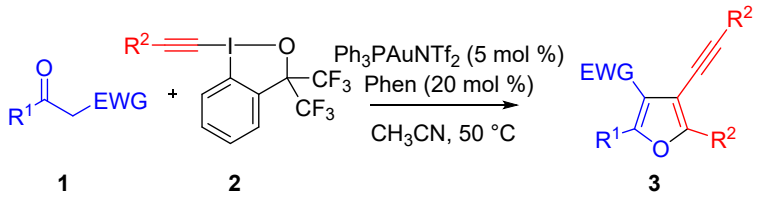
To a solution of trimethysilylacetylene (11 mmol) in THF (15 mL) was added *n*-BuLi (2.5 M in hexane, 10 mmol, 1 equiv) at -78 °C. After being stirred at -78 °C for 15 min, the reaction was further stirred at 0 °C for 10 min. After being cooled down to -78 °C again, TIPSCl (10 mmol, 1 equiv) was added. The reaction mixture was then allowed to warm to room temperature and stirred overnight. The reaction was quenched with saturated NH_4Cl solution. The resulting mixture was extracted with Et_2O (2 × 20 mL), the organic layers were combined, washed with saturated brine (20 mL) and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure, and the crude product was afforded as a yellow oil (87% yield); ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.12 – 1.08 (m, 21H), 0.20 (s, 9H). The spectroscopic data is in agreement with that previously reported.[1]



S1

Under argon, TMSOTf (1.1 equiv) was added dropwise to a suspension of **S1** (1 mmol, 1.0 equiv) in CH_2Cl_2 (2 mL) at room temperature. After 30 min, the solvent was removed at 0 °C under vacuum, and then CH_3CN (3 mL) was added. Trimethyl(phenylethynyl)silane (1.3 equiv) was added to the mixture dropwise at 0 °C. Then, the resulting solution was warmed up to room temperature and stirred for 12 h. After that, a solution of pyridine (1.1 equiv) was added slowly, and the resulting mixture was stirred at room temperature for 3 h. The solvent was then evaporated under the reduced pressure and the residue was purified by column chromatography on silica gel to afford **2a** in 86% yield as a white solid.

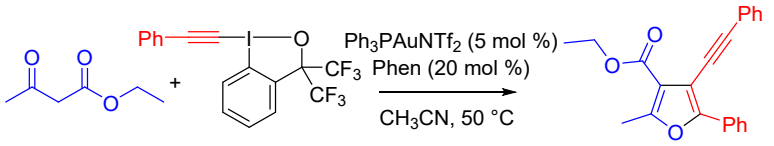
Procedure B: Synthesis of chemical **3**



1 **2** **3**

A mixture of **1** (0.10 mmol) and **2** (0.22 mmol) in 1.0 mL CH_3CN was treated with $Ph_3PAuNTf_2$ (5 mol %), Phen (20 mol%) and then heated to 50 °C in an oil bath. The reactions were monitored by TLC analysis and the chemical **1** were consumed completely. The solvent was removed under vacuum and the crude residue was purified by silica gel column chromatography to give the desired products.

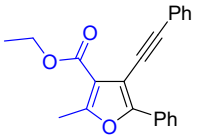
Procedure C: Gram-Scale Synthesis **3aa**



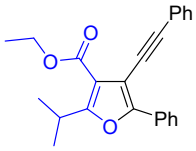
1a (5.0 mmol) **2a** (11.0 mmol) **3aa** (1.16 g, 70% yield)

A mixture of **1a** (5.0 mmol) and **2** (11.0 mmol) in 15.0 mL CH_3CN was treated with $Ph_3PAuNTf_2$ (5 mol %), Phen (20 mol%) and then heated to 50 °C in an oil bath. The reactions were monitored by TLC analysis and the chemical **1a** were consumed completely. The solvent was removed under vacuum and the crude residue was purified by silica gel column chromatography to give the desired products **3aa** in 70% yield (1.16 g).

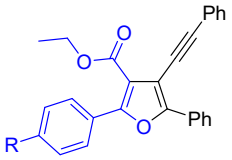
S1. Direct comparison of You's method and our method



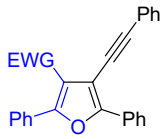
3aa, 76% (66%)[b]



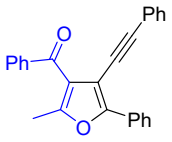
3da, 65% (60%)[b]



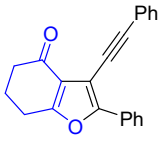
3ga, R = H, 54% (59%)[b]
3ja, R = OMe, 67% (50%)[b]



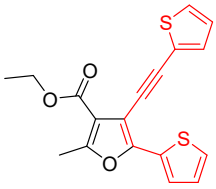
3ta, EWG = CO_2Me, 64% (57%)[b]
3ua, EWG = CN, 81% (trace)[c]
3va, EWG = NO_2, 44% (trace)[c]



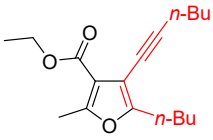
3wa, 78% (54%)[b]



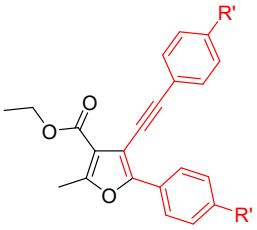
3xa, 84% (trace)[c]



3al, 73% (58%)[b]



3an, 62% (46%)[b]



3ab, R' = Me, 76% (50%)[b]
3ac, R' = OMe, 70% (56%)[b]
3ae, R' = Cl, 81% (56%)[b]

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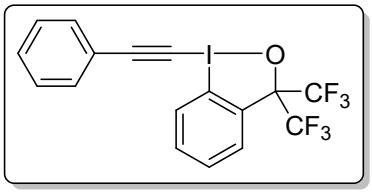
[a]Product yield in our manuscript. [b]Product yield reported by You et al. [c]Product yield under the standard conditions in You's paper (*Angew. Chem., Int. Ed.*, 2014, **53**, 7870).

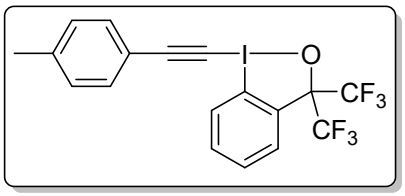
3. Characterization Data

1-(phenylethynyl)-3,3-bis(trifluoromethyl)-1,3-dihydro-1λ^3-benzo[*d*][1,2] iodaoxole (2a)



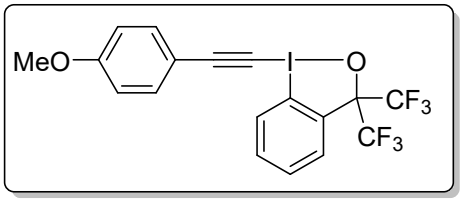
Yield: 405 mg, 86%; white solid, mp 132-133 °C; R_f = 0.46 (PE/EA = 10/1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.35 – 8.23 (m, 1H), 7.86 (m, 1H), 7.77 – 7.65 (m, 2H), 7.63 – 7.52 (m, 2H), 7.49 – 7.35 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 133.1 (d), 132.8 (d, 2C), 131.4 (d), 130.3 (d), 130.1 (s), 130.0 (m), 128.8 (d, 2C), 128.5 (d), 123.7 (q, $^1J_{C\text{-}F}$ = 290.8 Hz), 121.4 (s), 111.5 (s), 105.4 (s), 81.8 (m), 54.5 (s). IR (reflection) $\tilde{\nu}$ = 2139, 1738, 1595, 1567, 1488, 1466, 1442, 1290, 1259, 1182, 1151, 1137, 1071, 1048, 1026, 964, 947, 873, 794, 754, 728, 691, 664, 641 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{17}H_{10}F_6IO$ $[M+H]^+$: 470.9675, found: 470.9680.

1-(*p*-tolylethynyl)-3,3-bis(trifluoromethyl)-1,3-dihydro-1λ^3-benzo[*d*][1,2] iodaoxole (2b)



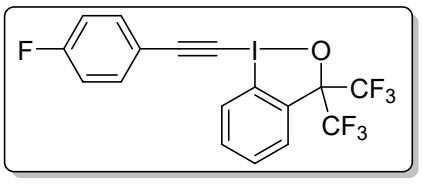
Yield: 364 mg, 75%; white solid, mp 124-125 °C; R_f = 0.70 (PE/EA = 5/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 – 8.25 (m, 1H), 7.90 – 7.82 (m, 1H), 7.73 – 7.64 (m, 2H), 7.45 (m, 2H), 7.21 (m, 2H), 2.41 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 140.8 (s), 132.9 (d), 132.6 (d, 2C), 131.2 (d), 130.1 (s) 129.9 (m), 129.4 (d, 2C), 128.3 (d), 123.6 (q, $^1J_{C\text{-}F}$ = 290.6 Hz), 118.2 (s), 111.5 (s), 105.7 (s), 81.7 (m), 53.5 (s), 21.6 (q). IR (reflection) $\tilde{\nu}$ = 3079, 3030, 2930, 2139, 1738, 1606, 1565, 1505, 1464, 1440, 1379, 1289, 1255, 1183, 1148, 1046, 1019, 963, 949, 814, 761, 753, 727, 691, 662, 640 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{18}H_{12}F_6IO$ $[M+H]^+$: 484.9832, found: 484.9826.

1-((4-methoxyphenyl)ethynyl)-3,3-bis(trifluoromethyl)-1,3-dihydro-1λ^3-benzo[*d*][1,2]iodaoxole (2c)



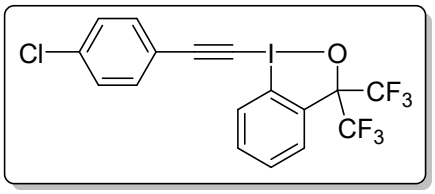
Yield: 165 mg, 33%; pale yellow solid, mp 91-92 °C; R_f = 0.42 (PE/EA = 5/1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.33 – 8.24 (m, 1H), 7.89 – 7.80 (m, 1H), 7.74 – 7.63 (m, 2H), 7.55 – 7.46 (m, 2H), 6.97 – 6.87 (m, 2H), 3.85 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 161.1 (s), 134.4 (d, 2C), 132.9 (d), 131.1 (d), 130.1 (s), 129.9 (m), 128.3 (d), 123.6 (q, $^1J_{C\text{-}F}$ = 290.6 Hz), 114.3 (d, 2C), 113.2 (s), 111.6 (s), 105.9 (s), 81.6 (m), 55.4 (q), 52.7 (s). IR (reflection) $\tilde{\nu}$ = 3076, 2966, 2843, 2135, 1738, 1603, 1565, 1508, 1464, 1440, 1295, 1253, 1219, 1183, 1165, 1150, 1138, 1029, 965, 946, 832, 762, 728, 690, 663, 641 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{18}H_{12}F_6IO_2$ $[M+H]^+$: 500.9781, found: 500.9787.

1-((4-fluorophenyl)ethynyl)-3,3-bis(trifluoromethyl)-1,3-dihydro-1λ^3-benzo[*d*][1,2]iodaoxole (2d)



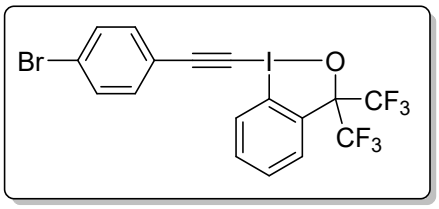
Yield: 321 mg, 64%; white solid, mp 136-137 °C; R_f = 0.62 (PE/EA = 5/1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.31 – 8.21 (m, 1H), 7.85 (m, 1H), 7.76 – 7.64 (m, 2H), 7.61 – 7.49 (m, 2H), 7.16 – 7.04 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 163.5 (d, $^1J_{C\text{-}F}$ = 252.8 Hz), 134.7 (d, $^3J_{C\text{-}F}$ = 8.7 Hz), 132.9 (d), 131.2 (d), 129.94 (s), 129.86 (m), 128.2 (d), 123.5 (q, $^1J_{C\text{-}F}$ = 290.5 Hz), 117.4 (d, $^4J_{C\text{-}F}$ = 3.6 Hz), 116.0 (d, $^2J_{C\text{-}F}$ = 22.3 Hz), 111.3 (s), 104.0 (s), 81.6 (m), 54.3 (s). IR (reflection) $\tilde{\nu}$ = 2144, 1748, 1599, 1566, 1505, 1465, 1441, 1289, 1266, 1205, 1184, 1166, 1146, 1118, 1094, 1048, 1017, 968, 949, 837, 763, 739, 728, 691, 663, 641 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{17}H_9F_7IO$ $[M+H]^+$: 488.9581, found: 488.9584.

1-((4-chlorophenyl)ethynyl)-3,3-bis(trifluoromethyl)-1,3-dihydro-1λ^3-benzo[*d*][1,2]iodaoxole (2e)



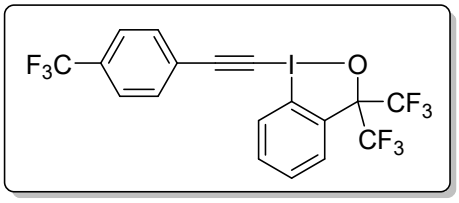
Yield: 479 mg, 95%; pale yellow solid, mp 118-119 °C; R_f = 0.55 (PE/EA = 5/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.28 – 8.21 (m, 1H), 7.89 – 7.81 (m, 1H), 7.74 – 7.66 (m, 2H), 7.52 – 7.45 (m, 2H), 7.41 – 7.35 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 136.4 (s), 133.8 (d, 2C), 133.0 (d), 131.3 (d), 130.02(s), 129.97 (m), 129.1 (d, 2C), 128.3 (d), 123.6 (q, $^1J_{C\text{-}F}$ = 290.6 Hz), 119.8 (s), 111.4 (s), 103.9 (s), 81.7 (m), 55.9 (s). IR (reflection) $\tilde{\nu}$ = 2133, 1738, 1563, 1487, 1462, 1439, 1399, 1295, 1262, 1219, 1185, 1164, 1148, 1118, 1095, 1045, 1015, 964, 947, 827, 800, 761, 729, 691, 664, 645 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{17}H_9{}^{35}ClF_6IO$ $[M+H]^+$: 504.9285, found: 504.9280.

1-((4-bromophenyl)ethynyl)-3,3-bis(trifluoromethyl)-1,3-dihydro-1λ^3-benzo[*d*][1,2]iodaoxole (2f)



Yield: 467 mg, 85%; pale yellow solid, mp 149-150 °C; R_f = 0.54 (PE/EA = 5/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.28 – 8.20 (m, 1H), 7.85 (m, 1H), 7.75 – 7.65 (m, 2H), 7.58 – 7.50 (m, 2H), 7.45 – 7.37 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 133.9 (d, 2C), 133.0 (d), 132.0 (d, 2C), 131.3 (d), 130.0 (s), 129.9 (m), 128.3 (d), 124.7 (s), 123.6 (q, $^1J_{C\text{-}F}$ = 291.0 Hz), 120.2 (s), 111.3 (s), 103.9 (s), 81.5 (m), 56.1 (s). IR (reflection) $\tilde{\nu}$ = 2131, 1738, 1564, 1484, 1462, 1440, 1394, 1263, 1219, 1185, 1165, 1148, 1132, 1070, 1045, 1012, 964, 947, 824, 796, 762, 729, 691, 664, 641, 612 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{17}H_9{}^{79}BrF_6IO$ $[M+H]^+$: 548.8780, found: 548.8778.

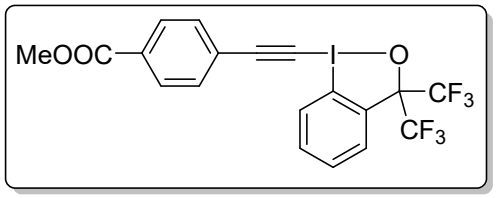
3,3-bis(trifluoromethyl)-1-((4-(trifluoromethyl)phenyl)ethynyl)-1,3-dihydro-1λ^3-benzo[*d*][1,2]iodaoxole (2g)



Yield: 422 mg, 78%; white solid, mp 124-125 °C; R_f = 0.60 (PE/EA = 5/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 – 8.22 (m, 1H), 7.87 (m, 1H), 7.76 – 7.68 (m, 2H), 7.67 (s, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 133.1 (d), 132.8 (d, 2C), 131.7 (q, $^2J_{C\text{-}F}$ = 33.0 Hz), 131.4 (d), 130.04 (m), 130.01 (s), 125.6 (q, $^3J_{C\text{-}F}$ = 3.7 Hz), 125.12 (s), 125.11 (s) 123.6 (q, $^1J_{C\text{-}F}$ = 272.4 Hz), 123.5 (q, $^1J_{C\text{-}F}$ = 291.5 Hz), 111.3 (s), 103.1 (s), 81.7 (m), 57.8 (s). IR (reflection) $\tilde{\nu}$ = 3072, 2146, 1747, 1615, 1566, 1465, 1440, 1405, 1321, 1296, 1264, 1183, 1148, 1123, 1106, 1066, 1047, 1017, 964, 948, 841, 820,

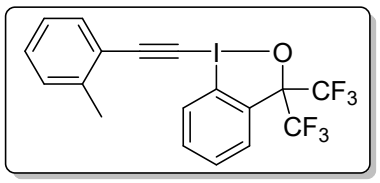
755, 728, 691, 664, 641, 608 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{18}H_9F_9IO$ $[M+H]^+$: 538.9549, found: 538.9546.

4-((3,3-bis(trifluoromethyl)-1λ³-benzo[*d*][1,2]iodaoxol-1(3H)-yl)ethynyl)benzoate (2h)



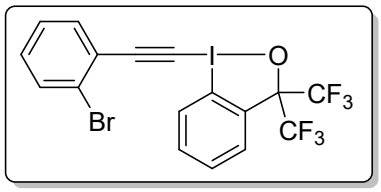
Yield: 386 mg, 73%; white solid, mp 195-196 °C; R_f = 0.58 (PE/EA = 5/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 – 8.22 (m, 1H), 8.10 – 8.04 (m, 2H), 7.89 – 7.83 (m, 1H), 7.75 – 7.67 (m, 2H), 7.64 – 7.58 (m, 2H), 3.94 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 166.1 (s), 133.1 (d), 132.5 (d, 2C), 131.4 (d), 131.2 (s), 130.0 (m), 129.7 (d, 2C), 128.4 (d), 125.8 (s), 123.5 (q, ^1J = 290.6 Hz), 111.3 (s), 103.9 (s), 81.7 (m), 58.0 (s), 52.4 (q). IR (reflection) $\tilde{\nu}$ = 3066, 3005, 2953, 2847, 2148, 1702, 1605, 1563, 1466, 1435, 1405, 1314, 1286, 1267, 1180, 1150, 1133, 1118, 1048, 1019, 970, 952, 881, 862, 843, 776, 766, 754, 732, 693, 683, 664, 644, 620 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{19}H_{12}F_6IO_3$ $[M+H]^+$: 528.9730, found: 528.9738.

1-(*o*-tolylethynyl)-3,3-bis(trifluoromethyl)-1,3-dihydro-1λ³-benzo[*d*][1,2] iodaoxole (2i)



Yield: 382 mg, 79%; white solid, mp 147-148 °C; R_f = 0.52 (PE/EA = 5/1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.37 – 8.28 (m, 1H), 7.91 – 7.81 (m, 1H), 7.76 – 7.64 (m, 2H), 7.53 (dd, J = 7.6, 0.9 Hz, 1H), 7.38 – 7.17 (m, 3H), 2.52 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 141.5 (s), 133.1 (d), 132.8 (d), 131.1 (d), 130.1 (d), 129.99 (s), 129.86 (m), 129.8 (d), 128.2 (d), 125.8 (d), 123.5 (q, $^1J_{C\text{-}F}$ = 291.0 Hz), 121.1 (s), 111.5 (s), 104.3 (s), 81.6 (m), 57.4 (s), 20.8 (q). IR (reflection) $\tilde{\nu}$ = 3075, 2928, 2136, 1739, 1562, 1481, 1464, 1438, 1381, 1287, 1264, 1219, 1176, 1149, 1132, 1043, 1017, 962, 952, 943, 833, 759, 750, 728, 712, 692, 681, 660, 641 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{18}H_{12}F_6IO$ $[M+H]^+$: 484.9832, found: 484.9832.

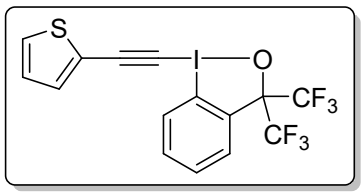
1-((2-bromophenyl)ethynyl)-3,3-bis(trifluoromethyl)-1,3-dihydro-1λ³-benzo[*d*][1,2]iodaoxole (2j)



Yield: 452 mg, 83%; white solid, mp 140-141 °C; R_f = 0.53 (PE/EA = 5/1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.54 – 8.42 (m, 1H), 7.86 (m, 1H), 7.78 – 7.61 (m, 3H), 7.57 (dd, J = 7.6, 1.9 Hz, 1H), 7.40 – 7.23 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 134.3 (d), 133.0 (d), 132.6 (d), 131.2 (d), 131.0 (d), 129.8 (m), 128.7 (d), 127.2 (d), 126.0 (s), 123.7 (s), 123.5 (q, $^1J_{C\text{-}F}$ = 291.0 Hz), 111.4 (s),

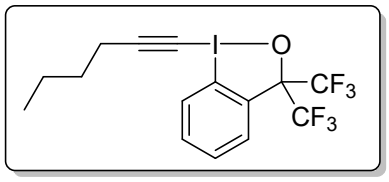
102.8 (s), 81.6 (m), 59.4 (s). IR (reflection) $\tilde{\nu}$ = 3077, 2145, 1738, 1563, 1465, 1438, 1287, 1262, 1251, 1220, 1194, 1176, 1150, 1135, 1045, 1027, 962, 952, 944, 808, 752, 728, 691, 681, 660, 641 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{17}H_9{}^{79}BrF_6IO$ $[M+H]^+$: 548.8780, found: 548.8781.

1-(thiophen-2-ylethynyl)-3,3-bis(trifluoromethyl)-1,3-dihydro-1λ³-benzo[*d*][1,2]iodaoxole (2l)



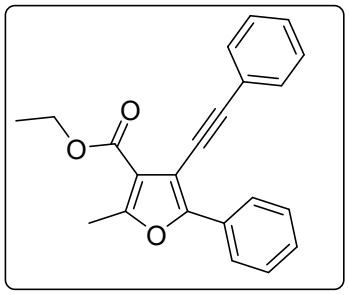
Yield: 123 mg, 26%; yellow solid, mp 112-113 °C; R_f = 0.42 (PE/EA = 5/1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.29 – 8.19 (m, 1H), 7.85 (m, 1H), 7.76 – 7.64 (m, 2H), 7.45 – 7.38 (m, 2H), 7.10 – 7.03 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ = 135.0 (d), 132.9 (d), 131.2 (d), 129.92 (s), 129.85 (m), 129.7 (d), 128.3 (d), 127.2 (d), 123.5 (q, $^1J_{C\text{-}F}$ = 291.0 Hz), 121.1 (s), 111.6 (s), 98.2 (s), 81.6 (m), 59.5 (s). IR (reflection) $\tilde{\nu}$ = 2123, 1564, 1462, 1439, 1421, 1258, 1221, 1178, 1164, 1148, 1132, 1117, 1077, 1044, 1021, 964, 947, 855, 835, 763, 728, 706, 689, 663, 640 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{15}H_8F_6IOS$ $[M+H]^+$: 476.9239, found: 476.9231.

1-(hex-1-yn-1-yl)-3,3-bis(trifluoromethyl)-1,3-dihydro-1λ³-benzo[*d*][1,2]iodaoxole (2n)



Yield: 266 mg, 60%; white solid, mp 106-107 °C; R_f = 0.52 (PE/EA = 5/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27 – 8.18 (m, 1H), 7.87 – 7.78 (m, 1H), 7.73 – 7.61 (m, 2H), 2.53 (t, J = 7.1 Hz, 2H), 1.64 – 1.58 (m, 2H), 1.47 (dt, J = 14.3, 7.3 Hz, 2H), 0.96 (t, J = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ = 132.7 (d), 131.0 (d), 130.1 (s), 129.8 (m), 128.2 (d), 123.65 (q, $^1J_{C\text{-}F}$ = 290.8 Hz), 110.9 (s), 107.9 (s), 81.6 (m), 43.4 (s), 30.4 (t), 22.0 (t), 20.0 (t), 13.5 (q). IR (reflection) $\tilde{\nu}$ = 3076, 2968, 2944, 2879, 2160, 1739, 1566, 1464, 1440, 1382, 1263, 1215, 1181, 1167, 1155, 1135, 1048, 1009, 963, 950, 868, 760, 731, 691, 663, 642 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{15}H_{14}F_6IO$ $[M+H]^+$: 450.9988, found: 450.9987.

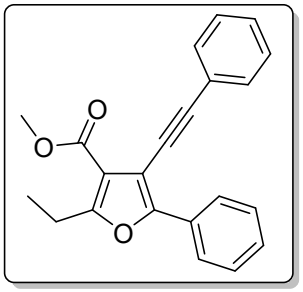
ethyl 2-methyl-5-phenyl-4-(phenylethynyl)furan-3-carboxylate (3aa)



Yield: 25 mg, 76%; yellow solid, mp 70-71 °C; R_f = 0.72 (PE/EA = 10/1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.14 (dt, J = 8.2, 1.6 Hz, 2H), 7.61 – 7.54 (m, 2H), 7.48 – 7.41 (m, 2H), 7.40 – 7.31 (m, 4H), 4.38 (q, J = 7.1 Hz, 2H), 2.67 (s, 3H), 1.41 (t, J = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.3 (s), 158.4 (s), 153.5 (s), 131.3 (d, 2C), 129.7 (s), 128.5 (d, 2C), 128.32 (d, 2C), 128.28 (d), 128.2 (d), 124.9 (d, 2C), 123.6 (s),

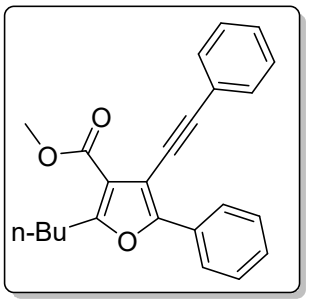
115.6 (s), 102.7 (s), 95.6 (s), 82.2 (s), 60.3 (t), 14.3 (q), 14.1 (q). IR (reflection) $\tilde{\nu}$ = 3338, 3061, 2982, 2928, 2216, 1950, 1880, 1708, 1605, 1499, 1485, 1444, 1422, 1370, 1336, 1248, 1212, 1148, 1098, 1072, 1044, 1014, 964, 947, 927, 830, 783, 757, 730, 692, 667, 647 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{22}H_{19}O_3$ $[M+H]^+$: 331.1329, found: 331.1327.

methyl 2-ethyl-5-phenyl-4-(phenylethynyl)furan-3-carboxylate (3ba)



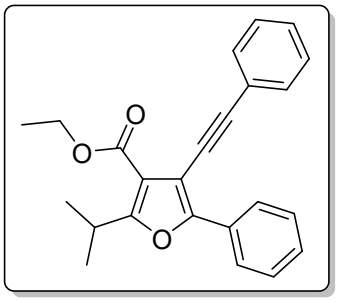
Yield: 26 mg, 79%; yellow solid, mp 73-75 °C; R_f = 0.61 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 – 8.10 (m, 2H), 7.60 – 7.54 (m, 2H), 7.49 – 7.43 (m, 2H), 7.41 – 7.31 (m, 4H), 3.92 (s, 3H), 3.09 (q, J = 7.6 Hz, 2H), 1.34 (t, J = 7.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.7 (s), 163.3 (s), 153.5 (s), 131.4 (d, 2C), 129.9 (s), 128.6 (d, 2C), 128.40 (d, 2C), 128.39 (d), 128.3 (d), 125.0 (d, 2C), 123.6 (s), 114.7 (s), 102.8 (s), 95.9 (s), 82.2 (s), 51.5 (q), 21.5 (t), 12.2 (q). IR (reflection) $\tilde{\nu}$ = 3059, 2977, 2948, 2879, 2215, 1950, 1881, 1713, 1604, 1557, 1499, 1484, 1440, 1412, 1348, 1322, 1247, 1228, 1200, 1118, 1100, 1072, 1029, 1019, 965, 945, 913, 837, 812, 787, 755, 730, 689, 656 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{22}H_{19}O_3$ $[M+H]^+$: 331.1329, found: 331.1327.

methyl 2-butyl-5-phenyl-4-(phenylethynyl)furan-3-carboxylate (3ca)



Yield: 27 mg, 76%; yellow liquid; R_f = 0.60 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (dt, J = 8.3, 1.7 Hz, 2H), 7.59 – 7.53 (m, 2H), 7.49 – 7.42 (m, 2H), 7.41 – 7.32 (m, 4H), 3.92 (s, 3H), 3.10 – 3.04 (m, 2H), 1.80 – 1.69 (m, 2H), 1.43 (dq, J = 14.7, 7.4 Hz, 2H), 0.97 (t, J = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.8 (s), 162.6 (s), 153.5 (s), 131.4 (d, 2C), 129.9 (s), 128.6 (d, 2C), 128.40 (d, 2C), 128.38 (d), 128.3 (d), 125.0 (d, 2C), 123.6 (s), 115.2 (s), 102.7 (s), 95.9 (s), 82.2 (s), 51.4 (q), 30.1 (t), 27.6 (t), 22.3 (t), 13.8 (q). IR (reflection) $\tilde{\nu}$ = 3059, 2955, 2931, 2871, 2216, 1949, 1879, 1800, 1714, 1603, 1558, 1498, 1484, 1439, 1379, 1346, 1325, 1244, 1200, 1110, 1071, 1034, 962, 912, 848, 814, 784, 755, 688 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{24}H_{23}O_3$ $[M+H]^+$: 359.1642, found: 359.1646.

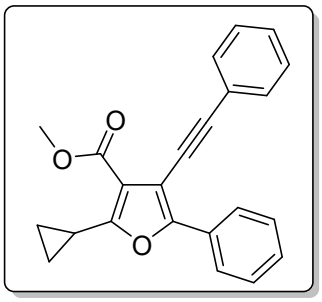
ethyl 2-isopropyl-5-phenyl-4-(phenylethynyl)furan-3-carboxylate (3da)



Yield: 23 mg, 65%; yellow solid, mp 67-68 °C; R_f = 0.64 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (dt, J = 8.3, 1.7 Hz, 2H), 7.62 – 7.55 (m, 2H), 7.51 – 7.44 (m,

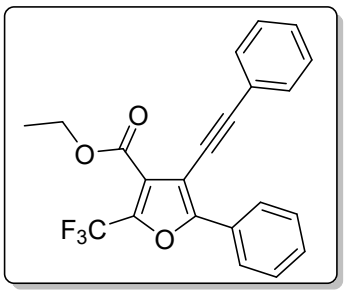
2H), 7.43 – 7.33 (m, 4H), 4.40 (q, J = 7.1 Hz, 2H), 3.89 – 3.80 (m, 1H), 1.43 (t, J = 7.1 Hz, 3H), 1.39 (s, 3H), 1.38 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.2 (s), 163.3 (s), 153.3 (s), 131.4 (d, 2C), 130.0 (s), 128.6 (d, 2C), 128.4 (d, 2C), 128.3 (d), 128.2 (d), 125.0 (d, 2C), 123.7 (s), 113.9 (s), 102.7 (s), 95.6 (s), 82.3 (s), 60.4 (t), 27.5 (d), 20.7 (q, 2C), 14.4 (q). IR (reflection) $\tilde{\nu}$ = 3064, 2982, 2937, 2873, 2215, 1703, 1602, 1556, 1498, 1484, 1443, 1413, 1368, 1340, 1310, 1271, 1248, 1211, 1161, 1116, 1089, 1058, 1025, 1007, 913, 841, 788, 766, 754, 689, 650 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{24}H_{23}O_3$ $[M+H]^+$: 359.1642, found: 359.1638.

methyl 2-cyclopropyl-5-phenyl-4-(phenylethynyl)furan-3-carboxylate (3ea)



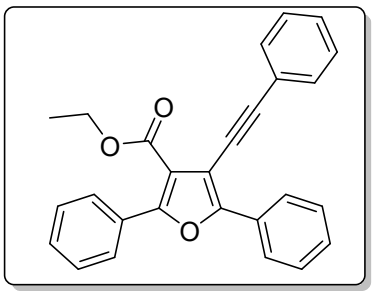
Yield: 22 mg, 65%; yellow solid, mp 109-110 °C; R_f = 0.60 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (dd, J = 8.4, 1.2 Hz, 2H), 7.60 – 7.54 (m, 2H), 7.46 – 7.29 (m, 6H), 3.94 (s, 3H), 2.85 (m, 1H), 1.21 – 1.09 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.1 (s), 162.5 (s), 152.1 (s), 131.4 (d, 2C), 129.8 (s), 128.5 (d, 2C), 128.4 (d, 2C), 128.3 (d, 2C), 124.8 (d, 2C), 123.6 (s), 115.0 (s), 103.1 (s), 95.8 (s), 82.2 (s), 51.4 (q), 9.4 (d), 9.0 (t, 2C). IR (reflection) $\tilde{\nu}$ = 3007, 2945, 1705, 1594, 1561, 1498, 1485, 1443, 1404, 1371, 1281, 1246, 1209, 1184, 1119, 1084, 1058, 1027, 1015, 956, 905, 881, 831, 816, 782, 755, 681, 650 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{23}H_{19}O_3$ $[M+H]^+$: 343.1329, found: 343.1322.

ethyl 5-phenyl-4-(phenylethynyl)-2-(trifluoromethyl)furan-3-carboxylate (3fa)



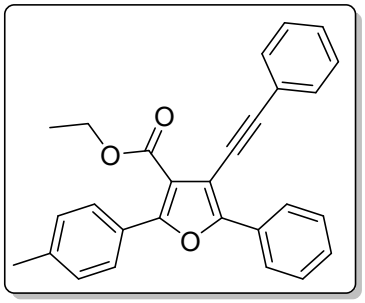
Yield: 22 mg, 58%; white solid, mp 94-95 °C; R_f = 0.65 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 – 8.14 (m, 2H), 7.57 (tdd, J = 5.2, 3.2, 2.0 Hz, 2H), 7.52 – 7.35 (m, 6H), 4.43 (q, J = 7.1 Hz, 2H), 1.40 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.2 (s), 155.8 (s), 141.4 (d, $^2J_{C\text{-}F}$ = 43.0 Hz), 131.6 (d, 2C), 129.9 (d), 128.9 (d), 128.8 (d, 2C), 128.5 (d, 2C), 128.4 (s), 125.7 (d, 2C), 122.7 (s), 122.0 (d, $^3J_{C\text{-}F}$ = 2.4 Hz), 118.5 (q, $^1J_{C\text{-}F}$ = 269.6 Hz), 104.4 (s), 97.0 (s), 79.5 (s), 61.7 (t), 14.0 (q). IR (reflection) $\tilde{\nu}$ = 2992, 2940, 2905, 1958, 1886, 1727, 1613, 1552, 1499, 1483, 1445, 1412, 1367, 1350, 1306, 1246, 1228, 1163, 1142, 1079, 1020, 999, 981, 914, 872, 842, 788, 758, 719, 687, 626 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{22}H_{16}F_3O_3$ $[M+H]^+$: 385.1046, found: 385.1040.

ethyl 2,5-diphenyl-4-(phenylethynyl)furan-3-carboxylate (3ga)



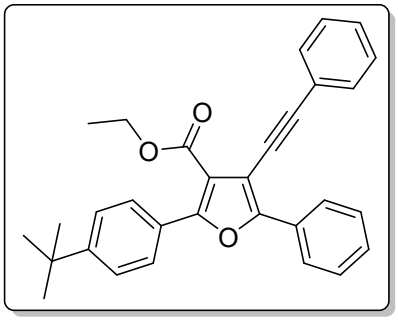
Yield: 21 mg, 54%; yellow solid, mp 80-81 °C; R_f = 0.60 (PE/EA = 10/1); ^{1}H NMR (700 MHz, $CDCl_3$) δ 8.22 (d, J = 8.0 Hz, 2H), 7.94 (d, J = 7.8 Hz, 2H), 7.58 (d, J = 7.5 Hz, 2H), 7.46 (dt, J = 14.0, 7.6 Hz, 5H), 7.41 – 7.35 (m, 4H), 4.40 (q, J = 7.1 Hz, 2H), 1.37 (t, J = 7.1 Hz, 3H). ^{13}C NMR (175 MHz, $CDCl_3$) δ 163.3 (s), 155.4 (s), 154.3 (s), 131.5 (d, 2C), 129.7 (d), 129.6 (s), 129.3 (s), 128.8 (d), 128.7 (d, 2C), 128.49 (d, 2C), 128.46(d), 128.41 (d, 2C), 128.3 (d, 2C), 125.4 (d, 2C), 123.4 (s), 116.4 (s), 104.5 (s), 96.0 (s), 81.8 (s), 61.0 (t), 14.2 (q). IR (reflection) $\tilde{\nu}$ = 3057, 2984, 2935, 2902, 2219, 1952, 1883, 1712, 1599, 1571, 1552, 1483, 1445, 1390, 1367, 1337, 1319, 1296, 1239, 1158, 1130, 1112, 1070, 1026, 965, 914, 838, 789, 769, 757, 688, 610 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{27}H_{21}O_3$ $[M+H]^+$: 393.1485, found: 393.1492.

ethyl 5-phenyl-4-(phenylethynyl)-2-(*p*-tolyl)furan-3-carboxylate (3ha)



Yield: 22 mg, 55%; yellow solid, mp 83-84 °C; R_f = 0.61 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (dd, J = 5.3, 3.3 Hz, 2H), 7.84 (d, J = 8.2 Hz, 2H), 7.62 – 7.55 (m, 2H), 7.51 – 7.44 (m, 2H), 7.43 – 7.34 (m, 4H), 7.28 (d, J = 8.0 Hz, 2H), 4.39 (q, J = 7.1 Hz, 2H), 2.42 (s, 3H), 1.37 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.3 (s), 155.8 (s), 154.0 (s), 139.9 (s), 131.4 (d, 2C), 129.7 (s), 128.9 (d, 2C), 128.7 (d), 128.6 (d, 2C), 128.5 (d, 2C), 128.4 (d), 128.3 (d, 2C), 126.5 (s), 125.3 (d, 2C), 123.5 (s), 115.9 (s), 104.5 (s), 95.9 (s), 81.9 (s), 60.9 (t), 21.5 (q), 14.3 (q). IR (reflection) $\tilde{\nu}$ = 3058, 2987, 2928, 2217, 1713, 1601, 1505, 1483, 1442, 1412, 1366, 1336, 1314, 1292, 1234, 1219, 1188, 1159, 1107, 1070, 1021, 965, 911, 842, 824, 783, 757, 717, 686, 666, 646 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{28}H_{23}O_3$ $[M+H]^+$: 407.1642, found: 407.1638.

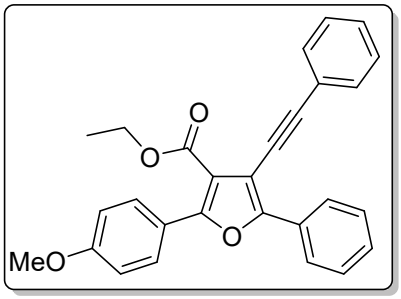
ethyl 2-(4-(tert-butyl)phenyl)-5-phenyl-4-(phenylethynyl)furan-3-carboxylate (3ia)



Yield: 20 mg, 45%; yellow liquid; R_f = 0.62 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (dd, J = 5.3, 3.3 Hz, 2H), 7.86 – 7.78 (m, 2H), 7.54 – 7.47 (m, 2H), 7.44 – 7.37 (m, 4H), 7.34 – 7.25 (m, 4H), 4.33 (q, J = 7.1 Hz, 2H), 1.32 – 1.26 (m, 12H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.3 (s), 155.8 (s), 154.0 (s), 153.0 (s), 131.4 (d, 2C), 129.7 (s), 128.7 (d), 128.6 (d,

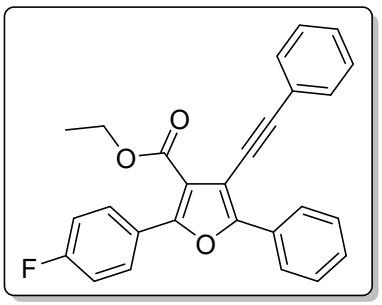
2C), 128.5 (d, 2C), 128.4 (d), 128.2 (d, 2C), 126.5 (s), 125.3 (d, 2C), 125.2 (d, 2C), 123.5 (s), 115.9 (s), 104.4 (s), 95.9 (s), 82.0 (s), 60.9 (t), 34.9 (s), 31.2 (q, 3C), 14.3 (q). IR (reflection) $\tilde{\nu}$ = 3060, 2963, 2904, 2868, 1950, 1879, 1718, 1601, 1578, 1501, 1483, 1463, 1444, 1413, 1367, 1337, 1317, 1293, 1269, 1236, 1201, 1122, 1098, 1071, 1020, 967, 913, 839, 787, 756, 730, 690 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{31}H_{29}O_3$ $[M+H]^+$: 449.2111, found: 449.2113.

ethyl 2-(4-methoxyphenyl)-5-phenyl-4-(phenylethynyl)furan-3-carboxylate (3ja)



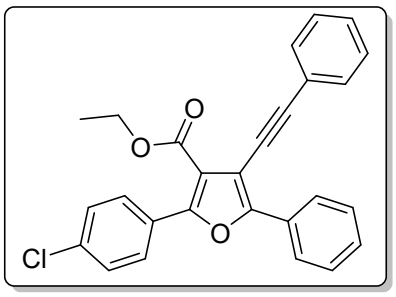
Yield: 28 mg, 67%; yellow solid, mp 138-139 °C; R_f = 0.44 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 – 8.18 (m, 2H), 7.96 – 7.89 (m, 2H), 7.61 – 7.55 (m, 2H), 7.50 – 7.44 (m, 2H), 7.42 – 7.33 (m, 4H), 7.01 – 6.96 (m, 2H), 4.39 (q, J = 7.1 Hz, 2H), 3.88 (s, 3H), 1.38 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.4 (s), 160.7 (s), 155.9 (s), 153.8 (s), 131.4 (d, 2C), 130.1 (d, 2C), 129.7 (s), 128.61 (d, 2C), 128.59 (d), 128.5 (d, 2C), 128.4 (d), 125.3 (d, 2C), 123.6 (s), 121.9 (s), 115.1 (s), 113.7 (d, 2C), 104.4 (s), 95.9 (s), 82.1 (s), 60.8 (t), 55.4 (q), 14.3 (q). IR (reflection) $\tilde{\nu}$ = 3063, 2976, 2934, 1707, 1610, 1581, 1503, 1484, 1461, 1439, 1403, 1390, 1365, 1339, 1303, 1263, 1237, 1178, 1125, 1111, 1071, 1022, 964, 910, 837, 816, 785, 763, 753, 683, 666, 645, 617 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{28}H_{23}O_4$ $[M+H]^+$: 423.1591, found: 423.1591.

ethyl 2-(4-fluorophenyl)-5-phenyl-4-(phenylethynyl)furan-3-carboxylate (3ka)



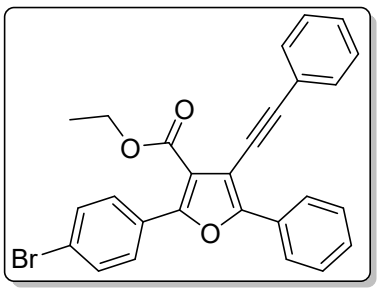
Yield: 32 mg, 78%; yellow solid, mp 120-121 °C; R_f = 0.60 (PE/EA = 10/1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.21 (d, J = 7.9 Hz, 2H), 8.01 – 7.93 (m, 2H), 7.58 (dd, J = 7.4, 1.9 Hz, 2H), 7.48 (t, J = 7.7 Hz, 2H), 7.39 (q, J = 5.8 Hz, 4H), 7.16 (t, J = 8.7 Hz, 2H), 4.40 (q, J = 7.1 Hz, 2H), 1.38 (t, J = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.5 (d, $^1J_{C\text{-}F}$ = 250.3 Hz), 163.2 (s), 154.7 (s), 154.3 (s), 131.4 (d, 2C), 130.6 (d, $^3J_{C\text{-}F}$ = 8.6 Hz, 2C), 129.5 (s), 128.9 (d), 128.7 (d, 2C), 128.5 (d, 3C), 125.5 (d, $^4J_{C\text{-}F}$ = 3.4 Hz), 125.4 (d, 2C), 123.4 (s), 116.2 (s), 115.39 (d, $^2J_{C\text{-}F}$ = 21.9 Hz, 2C), 104.5 (s), 96.1 (s), 81.8 (s), 61.0 (t), 14.3 (q). IR (reflection) $\tilde{\nu}$ = 2988, 1710, 1600, 1503, 1482, 1443, 1413, 1366, 1334, 1292, 1235, 1161, 1127, 1112, 1099, 1070, 1024, 963, 910, 840, 805, 785, 757, 686, 665, 644 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{27}H_{20}FO_3$ $[M+H]^+$: 411.1391, found: 411.1388.

ethyl 2-(4-chlorophenyl)-5-phenyl-4-(phenylethynyl)furan-3-carboxylate (3la)



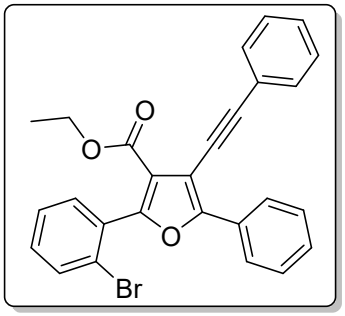
Yield: 29 mg, 68%; white solid, mp 100-101 °C; R_f = 0.61 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (dd, J = 5.3, 3.3 Hz, 2H), 7.95 – 7.88 (m, 2H), 7.61 – 7.54 (m, 2H), 7.51 – 7.37 (m, 8H), 4.40 (q, J = 7.1 Hz, 2H), 1.38 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.1 (s), 154.4 (s), 154.2 (s), 135.7 (s), 131.4 (d, 2C), 129.7 (d, 2C), 129.5 (s), 128.9 (d), 128.7 (d, 2C), 128.53 (d, 2C), 128.49 (d, 3C), 127.7 (s), 125.4 (d, 2C), 123.4 (s), 116.8 (s), 104.7 (s), 96.1 (s), 81.6 (s), 61.1 (t), 14.3 (q). IR (reflection) $\tilde{\nu}$ = 3062, 2989, 2925, 1711, 1601, 1579, 1549, 1481, 1440, 1406, 1367, 1333, 1305, 1281, 1235, 1184, 1129, 1114, 1104, 1092, 1071, 1014, 963, 910, 834, 784, 756, 685, 664, 633, 623 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{27}H_{20}{}^{35}ClO_3$ $[M+H]^+$: 427.1095, found: 427.1092.

ethyl 2-(4-bromophenyl)-5-phenyl-4-(phenylethynyl)furan-3-carboxylate (3ma)



Yield: 33 mg, 71%; yellow solid, mp 92-93 °C; R_f = 0.62 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 – 8.16 (m, 2H), 7.89 – 7.80 (m, 2H), 7.63 – 7.54 (m, 4H), 7.51 – 7.45 (m, 2H), 7.42 – 7.33 (m, 4H), 4.40 (q, J = 7.1 Hz, 2H), 1.38 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.1 (s), 154.5 (s), 154.2 (s), 131.5 (d, 2C), 131.4 (d, 2C), 129.8 (d, 2C), 129.4 (s), 128.9 (d), 128.7 (d, 2C), 128.5 (d, 3C), 128.2 (s), 125.4 (d, 2C), 124.0 (s), 123.4 (s), 116.9 (s), 104.7 (s), 96.2 (s), 81.6 (s), 61.1 (t), 14.3 (q). IR (reflection) $\tilde{\nu}$ = 3058, 2985, 1714, 1602, 1577, 1549, 1479, 1440, 1406, 1367, 1334, 1304, 1279, 1235, 1184, 1113, 1076, 1024, 1010, 964, 910, 831, 784, 756, 717, 685, 663, 615 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{27}H_{20}{}^{79}BrO_3$ $[M+H]^+$: 471.0590, found: 471.0593.

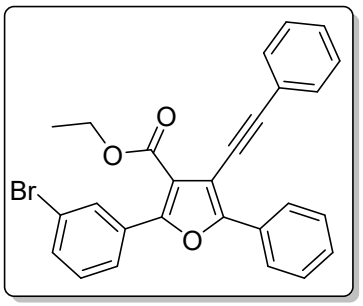
ethyl 2-(2-bromophenyl)-5-phenyl-4-(phenylethynyl)furan-3-carboxylate (3na)



Yield: 37 mg, 79%; yellow solid, mp 108-109 °C; R_f = 0.50 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (dt, J = 8.3, 1.7 Hz, 2H), 7.71 (dd, J = 8.0, 1.1 Hz, 1H), 7.64 – 7.58 (m, 2H), 7.55 (dd, J = 7.6, 1.7 Hz, 1H), 7.50 – 7.31 (m, 8H), 4.26 (q, J = 7.1 Hz, 2H), 1.19 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.2 (s), 155.1 (s), 155.0 (s), 132.9 (d), 132.4 (d), 131.5 (d, 2C), 131.4 (s), 131.1 (d), 129.6 (s), 128.9 (d), 128.7 (d, 2C), 128.4 (d, 3C), 126.9 (d), 125.4 (d, 2C), 124.0 (s), 123.5 (s), 118.6 (s), 103.5 (s), 96.3 (s), 81.5 (s), 60.7 (t), 14.0 (q). IR

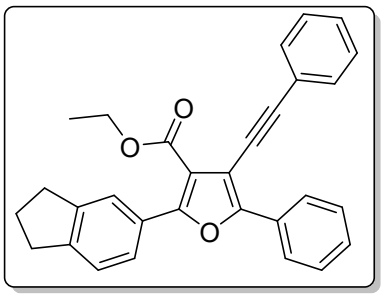
(reflection) $\tilde{\nu}$ = 2978, 1713, 1622, 1567, 1469, 1432, 1365, 1336, 1277, 1239, 1156, 1112, 1076, 1047, 1026, 966, 945, 919, 865, 838, 785, 766, 756, 728, 684, 657, 646, 610 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{27}H_{20}{}^{79}BrO_3$ $[M+H]^+$: 471.0590, found: 471.0593.

ethyl 2-(3-bromophenyl)-5-phenyl-4-(phenylethynyl)furan-3-carboxylate (3oa)



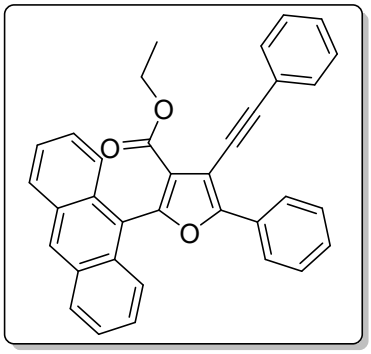
Yield: 30 mg, 64%; yellow solid, mp 91-92 °C; R_f = 0.60 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 – 8.19 (m, 2H), 8.10 (t, J = 1.8 Hz, 1H), 7.90 (m, 1H), 7.64 – 7.54 (m, 3H), 7.52 – 7.46 (m, 2H), 7.42 – 7.31 (m, 5H), 4.41 (q, J = 7.1 Hz, 2H), 1.38 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.9 (s), 154.7 (s), 153.4 (s), 132.4 (d), 131.5 (d, 2C), 131.2 (s), 131.1 (d), 129.7 (d), 129.4 (s), 129.0 (d), 128.7 (d, 2C), 128.5 (d), 128.48 (d, 2C), 126.9 (d), 125.4 (d, 2C), 123.3 (s), 122.3 (s), 117.3 (s), 104.8 (s), 96.3 (s), 81.5 (s), 61.2 (t), 14.2 (q). IR (reflection) $\tilde{\nu}$ = 3069, 2975, 1709, 1598, 1574, 1558, 1498, 1469, 1441, 1426, 1390, 1368, 1330, 1243, 1131, 1117, 1069, 1027, 997, 972, 903, 893, 844, 776, 749, 715, 687, 662, 610 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{27}H_{20}{}^{79}BrO_3$ $[M+H]^+$: 471.0590, found: 471.0593.

ethyl 2-(2,3-dihydro-1*H*-inden-5-yl)-5-phenyl-4-(phenylethynyl)furan-3-carboxylate (3pa)



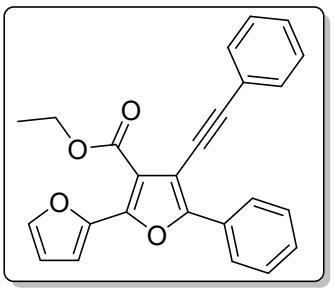
Yield: 19 mg, 44%; yellow liquid; R_f = 0.60 (PE/EA = 10/1); ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.25 – 8.19 (m, 2H), 7.77 (s, 1H), 7.70 (d, J = 7.8 Hz, 1H), 7.58 (dd, J = 7.8, 1.6 Hz, 2H), 7.47 (t, J = 7.7 Hz, 2H), 7.42 – 7.35 (m, 4H), 7.32 (d, J = 7.8 Hz, 1H), 4.39 (q, J = 7.1 Hz, 2H), 2.98 (m, 4H), 2.17 – 2.09 (m, 2H), 1.37 (t, J = 7.1 Hz, 3H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 163.4 (s), 156.3 (s), 153.9 (s), 146.4 (s), 144.3 (s), 131.4 (d, 2C), 129.7 (s), 128.6 (d, 3C), 128.5 (d, 2C), 128.4 (d), 127.2 (s), 126.7 (d), 125.3 (d, 2C), 124.4 (d), 124.2 (d), 123.5 (s), 115.7 (s), 104.4 (s), 95.9 (s), 82.0 (s), 60.9 (t), 33.0 (t), 32.9 (t), 25.5 (t), 14.3 (q). IR (reflection) $\tilde{\nu}$ = 3060, 2954, 2842, 2217, 1951, 1888, 1716, 1600, 1553, 1483, 1443, 1367, 1337, 1235, 1219, 1087, 1070, 1026, 979, 913, 888, 824, 787, 756, 690 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{30}H_{25}O_3$ $[M+H]^+$: 433.1798, found: 433.1796.

ethyl 2-(anthracen-9-yl)-5-phenyl-4-(phenylethynyl)furan-3-carboxylate (3qa)



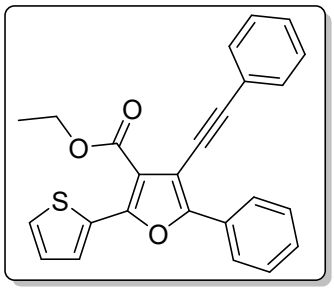
Yield: 30 mg, 61%; yellow liquid; R_f = 0.44 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.62 (s, 1H), 8.25 (dd, J = 5.3, 3.4 Hz, 2H), 8.11 – 8.05 (m, 2H), 7.82 – 7.74 (m, 2H), 7.72 – 7.64 (m, 2H), 7.53 – 7.36 (m, 10H), 3.89 (q, J = 7.1 Hz, 2H), 0.57 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.1 (s), 155.8 (s), 154.7 (s), 131.61 (s, 2C), 131.58 (d, 2C), 131.1 (s, 2C), 129.7 (s), 129.5 (d), 128.9 (d), 128.7 (d, 2C), 128.54 (d, 2C), 128.49 (d), 128.47 (d, 2C), 126.6 (d, 2C), 125.7 (d, 2C), 125.4 (d, 4C), 124.0 (s), 123.5 (s), 121.1 (s), 103.6 (s), 96.7 (s), 81.6 (s), 60.2 (t), 13.3 (q). IR (reflection) $\tilde{\nu}$ = 3054, 2981, 2250, 2198, 1951, 1720, 1603, 1554, 1522, 1483, 1444, 1425, 1371, 1322, 1205, 1117, 1080, 1045, 1014, 909, 894, 845, 790, 757, 737, 692, 607 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{35}H_{25}O_3$ $[M+H]^+$: 493.1798, found: 493.1800.

ethyl 5-phenyl-4-(phenylethynyl)-[2,2'-bifuran]-3-carboxylate (3ra)



Yield: 26 mg, 68%; yellow solid, mp 90-91 °C; R_f = 0.60 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.30 – 8.19 (m, 2H), 7.64 – 7.54 (m, 3H), 7.52 – 7.44 (m, 3H), 7.43 – 7.34 (m, 4H), 6.58 (dd, J = 3.5, 1.8 Hz, 1H), 4.43 (q, J = 7.1 Hz, 2H), 1.43 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.6 (s), 154.1 (s), 147.4 (s), 144.0 (s), 143.8 (d), 131.4 (d, 2C), 129.5 (s), 128.8 (d), 128.6 (d, 2C), 128.48 (d, 2C), 128.45 (d), 125.5 (d, 2C), 123.4 (s), 114.8 (s), 113.9 (d), 112.0 (d), 104.1 (s), 96.1 (s), 81.8 (s), 60.9 (t), 14.4 (q). IR (reflection) $\tilde{\nu}$ = 3161, 3116, 3058, 2981, 2905, 1701, 1598, 1539, 1498, 1478, 1443, 1367, 1326, 1255, 1164, 1130, 1080, 1022, 971, 905, 886, 834, 783, 766, 750, 688, 668, 629 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{25}H_{19}O_4$ $[M+H]^+$: 383.1278, found: 383.1275.

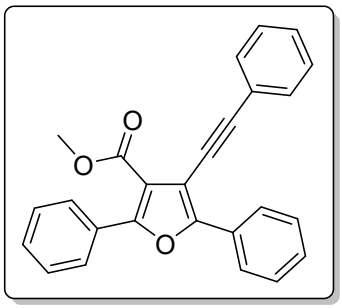
ethyl 5-phenyl-4-(phenylethynyl)-2-(thiophen-2-yl)furan-3-carboxylate (3sa)



Yield: 22 mg, 56%; yellow solid, mp 106-107 °C; R_f = 0.60 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 – 8.19 (m, 2H), 8.06 (m, 1H), 7.57 (m, 2H), 7.48 (m, 3H), 7.43 – 7.34 (m, 4H), 7.15 (dd, J = 5.0, 3.8 Hz, 1H), 4.45 (q, J = 7.1 Hz, 2H), 1.44 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.0 (s), 153.6 (s), 151.5 (s), 131.4 (d, 2C), 131.0 (s), 129.4 (s), 129.3 (d), 128.8 (d), 128.7 (d, 2C), 128.51 (d), 128.48 (d, 2C), 128.4 (d), 127.5 (d), 125.4 (d, 2C), 123.5 (s), 114.4 (s), 104.4 (s), 96.1 (s), 81.9 (s), 60.9

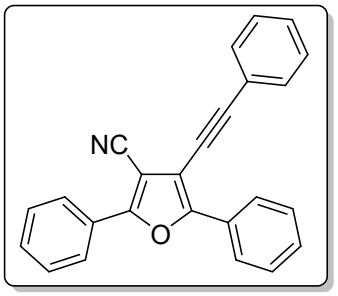
(t), 14.4 (q). IR (reflection) $\tilde{\nu}$ = 2981, 2211, 1703, 1598, 1572, 1482, 1429, 1371, 1314, 1248, 1211, 1129, 1104, 1073, 1048, 1026, 913, 855, 784, 755, 706, 687, 637, 609 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{25}H_{19}O_3S$ $[M+H]^+$: 399.1049, found: 399.1046.

methyl 2,5-diphenyl-4-(phenylethynyl)furan-3-carboxylate (3ta)



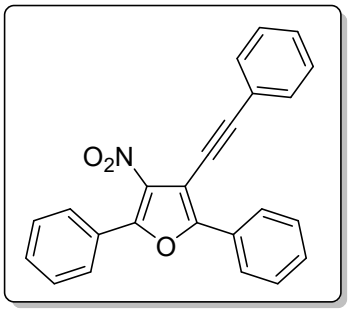
Yield: 24 mg, 64%; yellow solid, mp 121-122 °C; R_f = 0.60 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.26 – 8.20 (m, 2H), 7.96 – 7.90 (m, 2H), 7.62 – 7.56 (m, 2H), 7.52 – 7.43 (m, 5H), 7.42 – 7.35 (m, 4H), 3.93 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.6 (s), 155.6 (s), 154.2 (s), 131.5 (d), 129.7 (d), 129.6 (s), 129.3 (s), 128.8 (d), 128.7 (d, 2C), 128.5 (d, 4C), 128.4 (d, 2C), 128.3 (d, 2C), 125.3 (d, 2C), 123.4 (s), 116.3 (s), 104.5 (s), 96.2 (s), 81.7 (s), 51.8 (q). IR (reflection) $\tilde{\nu}$ = 3076, 2997, 2949, 2213, 1709, 1598, 1583, 1569, 1483, 1433, 1341, 1321, 1294, 1238, 1190, 1132, 1116, 1070, 1027, 987, 940, 924, 816, 788, 762, 687, 610 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{26}H_{19}O_3$ $[M+H]^+$: 379.1329, found: 379.1330.

2,5-diphenyl-4-(phenylethynyl)furan-3-carbonitrile (3ua)



Yield: 28 mg, 81%; yellow solid, mp 177-178 °C; R_f = 0.64 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 – 8.14 (m, 2H), 8.11 – 8.05 (m, 2H), 7.66 – 7.60 (m, 2H), 7.56 – 7.47 (m, 5H), 7.44 – 7.37 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 157.6 (s), 153.7 (s), 131.8 (d, 2C), 130.6 (d), 129.4 (d), 129.2 (d, 2C), 129.1 (d), 128.9 (d, 2C), 128.7 (s), 128.5 (d, 2C), 127.5 (s), 125.6 (d, 2C), 125.2 (d, 2C), 122.4 (s), 113.6 (s), 105.9 (s), 98.1 (s), 96.6 (s), 78.7 (s). IR (reflection) $\tilde{\nu}$ = 3060, 2229, 1956, 1888, 1808, 1600, 1559, 1484, 1444, 1345, 1231, 1153, 1119, 1101, 1070, 1027, 999, 965, 923, 841, 771, 758, 686, 638 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{25}H_{16}NO$ $[M+H]^+$: 346.1226, found: 346.1224.

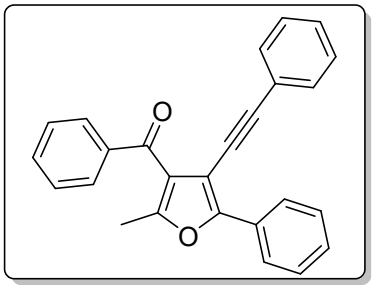
3-nitro-2,5-diphenyl-4-(phenylethynyl)furan (3va)



Yield: 16 mg, 44%; yellow solid, mp 188-189 °C; R_f = 0.61 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 – 8.18 (m, 2H), 7.95 – 7.88 (m, 2H), 7.66 – 7.59 (m, 2H), 7.56 – 7.49 (m, 5H), 7.46 – 7.36 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.6 (s), 150.9 (s), 131.7 (d, 2C), 131.0 (d), 129.7 (d), 129.0 (d), 128.9 (d, 2C), 128.7 (d, 6C), 128.5 (d, 2C), 127.1 (s), 125.4 (d, 2C), 122.6 (s), 100.9

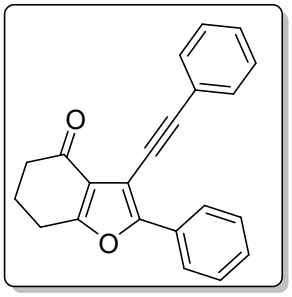
(s), 98.1 (s), 78.6 (s). IR (reflection) $\tilde{\nu}$ = 3064, 2220, 1987, 1963, 1605, 1572, 1553, 1509, 1483, 1446, 1410, 1357, 1233, 1189, 1145, 1119, 1072, 1028, 999, 967, 925, 833, 761, 688, 622 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{24}H_{16}NO_3$ $[M+H]^+$: 366.1125, found: 366.1128.

(2-methyl-5-phenyl-4-(phenylethynyl)furan-3-yl)(phenyl)methanone (3wa)



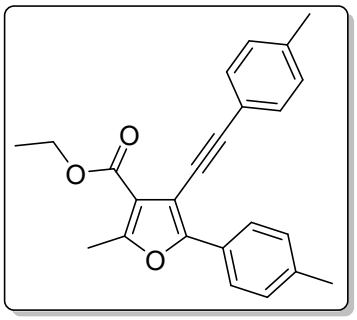
Yield: 28 mg, 78%; yellow solid, mp 88-89 °C; R_f = 0.62 (PE/EA = 10/1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.12 – 8.03 (m, 2H), 7.89 – 7.83 (m, 2H), 7.54 – 7.46 (m, 1H), 7.44 – 7.35 (m, 4H), 7.31 – 7.23 (m, 1H), 7.19 – 7.12 (m, 3H), 6.99 – 6.91 (m, 2H), 2.45 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.3 (s), 156.6 (s), 153.1 (s), 138.4 (s), 132.7 (d), 131.0 (d, 2C), 129.8 (d, 2C), 129.7 (s), 128.5 (d, 2C), 128.4 (d), 128.1 (d, 3C), 128.0 (d, 2C), 124.9 (d, 2C), 123.6 (s), 122.9 (s), 102.9 (s), 97.2 (s), 81.9 (s), 13.6 (q). IR (reflection) $\tilde{\nu}$ = 3060, 1650, 1598, 1578, 1498, 1484, 1451, 1440, 1396, 1379, 1341, 1265, 1243, 1212, 1183, 1164, 1152, 1133, 1115, 1096, 1068, 1023, 999, 933, 910, 858, 838, 803, 762, 753, 729, 687, 673, 626 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{26}H_{19}O_2$ $[M+H]^+$: 363.1380, found: 363.1378.

2-phenyl-3-(phenylethynyl)-6,7-dihydrobenzofuran-4(5*H*)-one (3xa)



Yield: 26 mg, 84%; white solid, mp 110-111 °C; R_f = 0.26 (PE/EA = 5/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (dd, J = 5.3, 3.3 Hz, 2H), 7.62 (m, 2H), 7.46 (m 2H), 7.41 – 7.31 (m, 4H), 2.96 (t, J = 6.3 Hz, 2H), 2.59 – 2.52 (m, 2H), 2.29 – 2.17 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.2 (s), 165.5 (s), 154.8 (s), 131.7 (d, 2C), 129.6 (s), 128.7 (d, 3C), 128.4 (d), 128.3 (d, 2C), 125.1 (d, 2C), 123.4 (s), 122.1 (s), 100.0 (s), 96.3 (s), 81.4 (s), 38.1 (t), 23.6 (t), 22.3 (t). IR (reflection) $\tilde{\nu}$ = 3062, 2935, 1676, 1600, 1558, 1499, 1482, 1454, 1435, 1415, 1357, 1223, 1175, 1157, 1145, 1091, 1063, 1023, 1010, 905, 884, 766, 752, 688, 652 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{22}H_{17}O_2$ $[M+H]^+$: 313.1223, found: 313.1222.

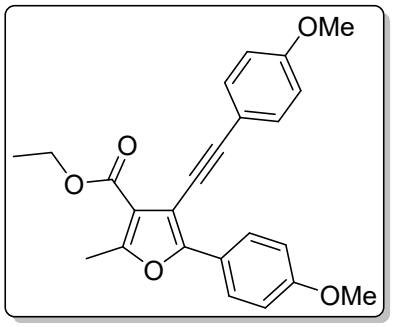
ethyl 2-methyl-5-(*p*-tolyl)-4-(*p*-tolylethynyl)furan-3-carboxylate (3ab)



Yield: 27 mg, 76%; yellow solid, mp 132-133 °C; R_f = 0.60 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, J = 8.3 Hz, 2H), 7.48 (d, J = 8.1 Hz, 2H), 7.29 – 7.25 (m, 2H), 7.20 (d, J = 7.9 Hz, 2H), 4.40 (q, J = 7.1 Hz, 2H), 2.68 (s, 3H), 2.42 (s, 3H), 2.41 (s, 3H), 1.43 (t, J =

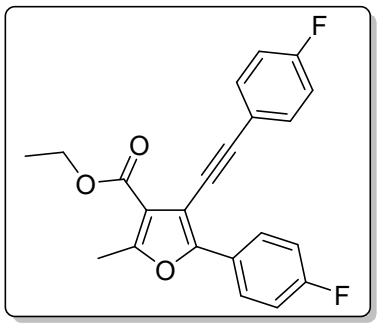
7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.5 (s), 158.1 (s), 153.7 (s), 138.3 (s, 2C), 131.2 (d, 2C), 129.23 (d, 2C), 129.15 (d, 2C), 127.2 (s), 125.0 (d, 2C), 120.7 (s), 115.5 (s), 102.2 (s), 95.7 (s), 81.7 (s), 60.3 (t), 21.5 (q), 21.4 (q), 14.4 (q), 14.1 (q). IR (reflection) $\tilde{\nu}$ = 2989, 2923, 1704, 1602, 1517, 1498, 1441, 1417, 1368, 1332, 1269, 1248, 1207, 1182, 1159, 1098, 1038, 975, 842, 811, 777, 717, 686, 656 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{24}H_{23}O_3$ $[M+H]^+$: 359.1642, found: 359.1643.

ethyl 5-(4-methoxyphenyl)-4-((4-methoxyphenyl)ethynyl)-2-methylfuran-3-carboxylate (3ac)



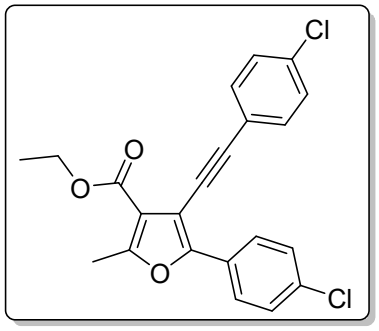
Yield: 27 mg, 70%; yellow solid, mp 93-94 °C; R_f = 0.31 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 – 8.03 (m, 2H), 7.53 – 7.45 (m, 2H), 6.99 – 6.93 (m, 2H), 6.93 – 6.87 (m, 2H), 4.36 (q, J = 7.1 Hz, 2H), 3.85 (s, 3H), 3.84 (s, 3H), 2.64 (s, 3H), 1.40 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.6 (s), 159.6 (s), 159.6 (s), 157.8 (s), 153.4 (s), 132.8 (d, 2C), 126.5 (d, 2C), 122.9 (s), 116.0 (s), 115.4 (s), 114.1 (d, 2C), 114.0 (d, 2C), 101.3 (s), 95.1 (s), 81.1 (s), 60.3 (t), 55.34 (q), 55.32 (q), 14.4 (q), 14.1 (q). IR (reflection) $\tilde{\nu}$ = 2971, 2840, 1887, 1696, 1602, 1567, 1516, 1499, 1462, 1444, 1404, 1370, 1329, 1289, 1244, 1210, 1173, 1095, 1025, 979, 829, 811, 783, 724, 684, 654 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{24}H_{23}O_5$ $[M+H]^+$: 391.1540, found: 391.1533.

ethyl 5-(4-fluorophenyl)-4-((4-fluorophenyl)ethynyl)-2-methylfuran-3-carboxylate (3ad)



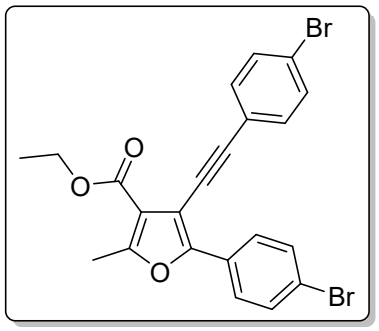
Yield: 32 mg, 87%; yellow solid, mp 94-95 °C; R_f = 0.61 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12 – 8.03 (m, 2H), 7.57 – 7.48 (m, 2H), 7.17 – 7.02 (m, 4H), 4.36 (q, J = 7.1 Hz, 2H), 2.65 (s, 3H), 1.39 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.2 (s), 162.59 (d, $^1J_{C\text{-}F}$ = 249.8 Hz), 162.58 (d, $^1J_{C\text{-}F}$ = 249.8 Hz), 158.4 (s), 152.7 (s), 133.2 (d, $^3J_{C\text{-}F}$ = 8.4 Hz, 2C), 126.9 (d, $^3J_{C\text{-}F}$ = 8.1 Hz, 2C), 126.1 (d, $^4J_{C\text{-}F}$ = 3.3 Hz), 119.6 (d, $^4J_{C\text{-}F}$ = 3.5 Hz), 115.8 (d, $^2J_{C\text{-}F}$ = 22.6 Hz, 2C), 115.7 (d, $^2J_{C\text{-}F}$ = 21.7 Hz, 2C), 115.6 (s), 102.4 (s), 94.5 (s), 81.7 (d, $^5J_{C\text{-}F}$ = 1.2 Hz), 60.4 (t), 14.4 (q), 14.1 (q). IR (reflection) $\tilde{\nu}$ = 3069, 2989, 2910, 1884, 1704, 1599, 1513, 1496, 1449, 1419, 1368, 1332, 1269, 1226, 1160, 1116, 1100, 1035, 1018, 974, 828, 779, 717, 681, 655, 624 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{22}H_{17}F_2O_3$ $[M+H]^+$: 367.1140, found: 367.1135.

ethyl 5-(4-chlorophenyl)-4-((4-chlorophenyl)ethynyl)-2-methylfuran-3-carboxylate (3ae)



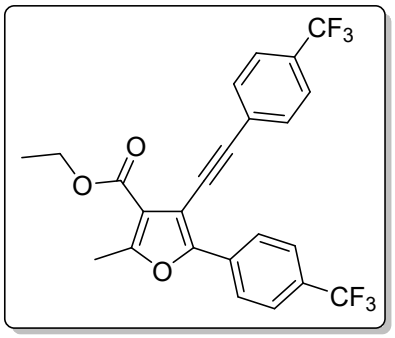
Yield: 32 mg, 81%; yellow solid, mp 127-128 °C; R_f = 0.62 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 – 8.00 (m, 2H), 7.50 – 7.44 (m, 2H), 7.43 – 7.38 (m, 2H), 7.37 – 7.32 (m, 2H), 4.36 (q, J = 7.1 Hz, 2H), 2.65 (s, 3H), 1.39 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.1 (s), 158.7 (s), 152.6 (s), 134.5 (s), 134.2 (s), 132.5 (d, 2C), 128.8 (d, 4C), 128.2 (s), 126.2 (d, 2C), 121.9 (s), 115.7 (s), 103.1 (s), 95.0 (s), 82.9 (s), 60.5 (t), 14.4 (q), 14.2 (q). IR (reflection) $\tilde{\nu}$ = 2986, 2907, 1896, 1707, 1604, 1496, 1480, 1449, 1416, 1398, 1366, 1331, 1264, 1247, 1210, 1176, 1092, 1034, 1013, 973, 854, 823, 771, 713, 681, 637, 618 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{22}H_{17}Cl_2O_3$ $[M+H]^+$: 399.0549, found: 399.0545.

ethyl 5-(4-bromophenyl)-4-((4-bromophenyl)ethynyl)-2-methylfuran-3-carboxylate (3af)



Yield: 29 mg, 60%; yellow solid, mp 126-127 °C; R_f = 0.63 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 – 7.93 (m, 2H), 7.59 – 7.54 (m, 2H), 7.54 – 7.48 (m, 2H), 7.42 – 7.37 (m, 2H), 4.36 (q, J = 7.1 Hz, 2H), 2.65 (s, 3H), 1.38 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.1 (s), 158.8 (s), 152.7 (s), 132.7 (d, 2C), 131.79 (d, 2C), 131.76 (d, 2C), 128.6 (s), 126.4 (d, 2C), 122.7 (s), 122.5 (s), 122.3 (s), 115.8 (s), 103.2 (s), 95.2 (s), 83.1 (s), 60.5 (t), 14.4 (q), 14.2 (q). IR (reflection) $\tilde{\nu}$ = 3090, 3054, 2984, 2905 ,1897, 1708, 1605, 1589, 1492, 1479, 1419, 1393, 1367, 1330, 1263, 1246, 1211, 1178, 1102, 1071, 1034, 1009, 973, 821, 779, 765, 711, 697, 681, 666, 636 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{22}H_{17}{}^{79}Br_2O_3$ $[M+H]^+$: 486.9539, found: 486.9537.

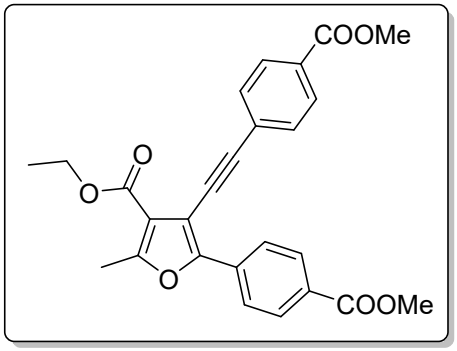
ethyl 2-methyl-5-(4-(trifluoromethyl)phenyl)-4-((4-(trifluoromethyl)phenyl) ethynyl)furan-3-carboxylate (3ag)



Yield: 30 mg, 65%; white solid, mp 110-111 °C; R_f = 0.61 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22 (d, J = 8.2 Hz, 2H), 7.74 – 7.60 (m, 6H), 4.39 (q, J = 7.1 Hz, 2H), 2.69 (s, 3H), 1.40 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.9 (s), 159.5 (s),

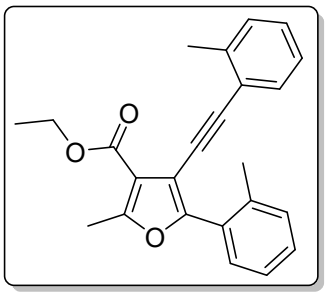
152.3 (s), 132.7 (s), 131.62 (d, 2C), 130.31 (q, $^{2}J_{C\text{-}F}$= 33.0 Hz), 130.09 (q, $^{2}J_{C\text{-}F}$= 33.0 Hz), 126.95 (s), 125.64 (q, $^{3}J_{C\text{-}F}$= 3.8 Hz, 2C), 125.46 (q, $^{3}J_{C\text{-}F}$ = 3.8 Hz, 2C), 125.02 (d, 2C), 123.96 (q, $^{1}J_{C\text{-}F}$= 271.9 Hz), 123.88 (q, $^{1}J_{C\text{-}F}$= 272.0 Hz), 116.0 (s), 104.3 (s), 95.2 (s), 84.0 (s), 60.6 (t), 14.4 (q), 14.2 (q). IR (reflection) $\tilde{\nu}$ = 2980, 2933, 2908, 2218, 1923, 1710, 1615, 1503, 1480, 1409, 1370, 1322, 1244, 1212, 1173, 1160, 1116, 1100, 1067, 1015, 971, 839, 781, 766, 739, 687 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{24}H_{17}F_6O_3$ $[M+H]^+$: 467.1076, found: 467.1075.

ethyl 5-(4-(methoxycarbonyl)phenyl)-4-((4-(methoxycarbonyl)phenyl)ethynyl)-2-methylfuran-3-carboxylate (3ah)



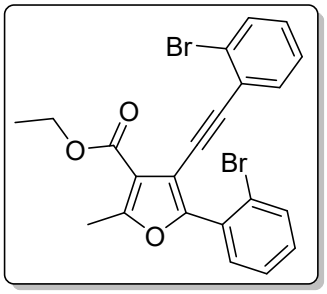
Yield: 33 mg, 74%; white solid, mp 149-150 °C; R_f = 0.46 (PE/EA = 5/1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.21 – 8.14 (m, 2H), 8.12 – 8.01 (m, 4H), 7.60 (d, J = 8.5 Hz, 2H), 4.37 (q, J = 7.1 Hz, 2H), 3.94 (s, 3H), 3.93 (s, 3H), 2.67 (s, 3H), 1.39 (t, J = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.6 (s), 166.5 (s), 162.9 (s), 159.5 (s), 152.7 (s), 133.5 (s), 131.3 (d, 2C), 129.9 (d, 2C), 129.8 (s), 129.7 (d, 2C), 129.5 (s), 127.9 (s), 124.6 (d, 2C), 116.0 (s), 104.6 (s), 96.0 (s), 84.9 (s), 60.6 (t), 52.3 (q), 52.2 (q), 14.4 (q), 14.2 (q). IR (reflection) $\tilde{\nu}$ = 2992, 2954, 2916, 2843, 2213, 1925, 1726, 1706, 1606, 1574, 1488, 1434, 1405, 1367, 1333, 1308, 1270, 1249, 1211, 1193, 1180, 1117, 1098, 1038, 1015, 967, 850, 825, 809, 782, 763, 697, 668 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{26}H_{23}O_7$ $[M+H]^+$: 447.1438, found: 447.1442

ethyl 2-methyl-5-(*o*-tolyl)-4-(*o*-tolylethynyl)furan-3-carboxylate (3ai)



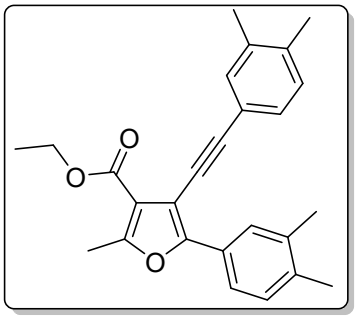
Yield: 28 mg, 79%; yellow liquid; R_f = 0.61 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (d, J = 7.6 Hz, 1H), 7.40 (d, J = 7.5 Hz, 1H), 7.34 – 7.22 (m, 3H), 7.21 – 7.14 (m, 2H), 7.14 – 7.08 (m, 1H), 4.39 (q, J = 7.1 Hz, 2H), 2.65 (s, 3H), 2.45 (s, 3H), 2.37 (s, 3H), 1.39 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.5 (s), 158.6 (s), 155.5 (s), 140.1 (s), 137.2 (s), 131.8 (d), 130.8 (d), 130.0 (d), 129.4 (d), 129.2 (d), 128.9 (s), 128.1 (d), 125.5 (d), 125.4 (d), 123.4 (s), 114.8 (s), 105.2 (s), 93.1 (s), 85.4 (s), 60.4 (t), 20.8 (q), 20.6 (q), 14.5 (q), 14.2 (q). IR (reflection) $\tilde{\nu}$ = 3059, 3019, 2979, 2926, 2867, 2216, 1953, 1919, 1707, 1602, 1477, 1456, 1416, 1367, 1330, 1287, 1240, 1211, 1191, 1120, 1089, 1030, 973, 943, 841, 783, 755, 721, 657 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{24}H_{23}O_3$ $[M+H]^+$: 359.1642, found: 359.1642.

ethyl 5-(2-bromophenyl)-4-((2-bromophenyl)ethynyl)-2-methylfuran-3-carboxylate (3aj)



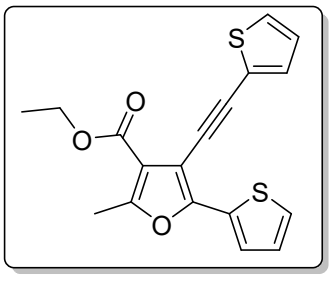
Yield: 21 mg, 43%; yellow liquid; R_f = 0.59 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (dd, J = 7.7, 1.7 Hz, 1H), 7.68 (dd, J = 8.0, 1.1 Hz, 1H), 7.54 (dd, J = 8.1, 1.0 Hz, 1H), 7.48 (dd, J = 7.7, 1.7 Hz, 1H), 7.38 (td, J = 7.6, 1.2 Hz, 1H), 7.30 – 7.26 (m, 1H), 7.25 – 7.20 (m, 1H), 7.16 – 7.09 (m, 1H), 4.39 (q, J = 7.1 Hz, 2H), 2.68 (s, 3H), 1.40 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.2 (s), 159.3 (s), 154.1 (s), 133.6 (d), 133.5 (d), 132.39 (d), 132.38 (d), 130.6 (d), 130.3 (s), 129.3 (d), 127.1 (d), 126.9 (d), 125.7 (s), 125.1 (s), 122.6 (s), 114.9 (s), 106.0 (s), 92.8 (s), 85.5 (s), 60.5 (t), 14.5 (q), 14.2 (q). IR (reflection) $\tilde{\nu}$ = 2977, 2219, 1699, 1595, 1567, 1477, 1457, 1428, 1369, 1331, 1257, 1235, 1210, 1095, 1065, 1023, 975, 835, 781, 751, 722, 685, 665, 638 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{22}H_{17}{}^{79}Br_2O_3$ $[M+H]^+$: 486.9539, found: 486.9522.

ethyl 5-(3,4-dimethylphenyl)-4-((3,4-dimethylphenyl)ethynyl)-2-methylfuran-3-carboxylate (3ak)



Yield: 25 mg, 65%; white solid, mp 152-153 °C; R_f = 0.60 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (s, 1H), 7.86 (dd, J = 7.9, 1.7 Hz, 1H), 7.35 (s, 1H), 7.30 (dd, J = 7.8, 1.2 Hz, 1H), 7.20 (d, J = 8.0 Hz, 1H), 7.13 (d, J = 7.7 Hz, 1H), 4.37 (q, J = 7.1 Hz, 2H), 2.65 (s, 3H), 2.33 (s, 3H), 2.30 (s, 3H), 2.29 (s, 3H), 2.28 (s, 3H), 1.41 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.6 (s), 158.0 (s), 153.8(s), 137.1 (s), 137.0 (s), 136.7 (s), 136.6 (s), 132.4 (d), 129.8 (d), 129.7 (d), 128.7 (d), 127.6 (s), 126.2 (d), 122.6 (d), 121.1 (s), 115.4 (s), 102.2 (s), 95.8 (s), 81.6 (s), 60.3 (t), 20.0 (q), 19.8 (q), 19.7 (q), 19.6 (q), 14.4 (q), 14.1 (q). IR (reflection) $\tilde{\nu}$ = 2971, 2920, 1718, 1597, 1503, 1487, 1446, 1407, 1382, 1337, 1287, 1233, 1207, 1179, 1165, 1126, 1098, 1022, 976, 884, 816, 780, 713, 688, 650, 628 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{26}H_{27}O_3$ $[M+H]^+$: 387.1955, found: 387.1951.

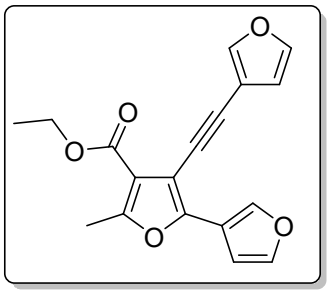
ethyl 2-methyl-5-(thiophen-2-yl)-4-(thiophen-2-ylethynyl)furan-3-carboxylate (3al)



Yield: 25 mg, 73%; yellow solid, mp 73-74 °C; R_f = 0.56 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.65 (dd, J = 3.7, 1.1 Hz, 1H), 7.34 (dd, J = 7.6, 2.7 Hz, 3H), 7.10 (dd,

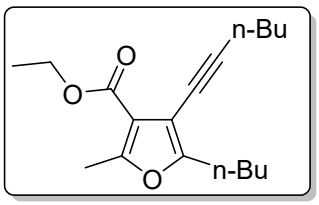
J = 5.0, 3.7 Hz, 1H), 7.07 – 7.02 (m, 1H), 4.36 (q, J = 7.1 Hz, 2H), 2.64 (s, 3H), 1.42 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.1 (s), 158.3 (s), 150.7 (s), 131.7 (s), 131.65 (d), 127.5 (d), 127.4 (d), 127.2 (d), 125.8 (d), 124.7 (d), 123.6 (s), 115.0 (s), 101.5 (s), 90.3 (s), 85.5 (s), 60.5 (t), 14.3 (q), 14.0 (q). IR (reflection) $\tilde{\nu}$ = 3116, 2982, 2904, 1704, 1605, 1480, 1436, 1406, 1377, 1350, 1315, 1244, 1227, 1173, 1098, 1041, 855, 820, 778, 701, 612 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{18}H_{15}O_3S_2$ $[M+H]^+$: 343.0457, found: 343.0456.

ethyl 3-(furan-3-ylethynyl)-5-methyl-[2,3'-bifuran]-4-carboxylate (3am)



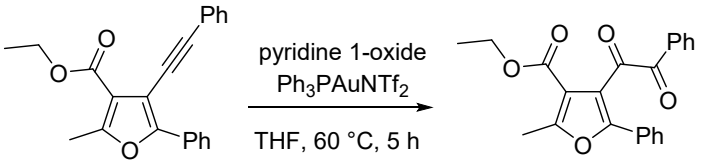
Yield: 25 mg, 81%; white solid, mp 62-63 °C; R_f = 0.55 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (d, J = 0.6 Hz, 1H), 7.73 – 7.68 (m, 1H), 7.46 (t, J = 1.7 Hz, 1H), 7.42 (t, J = 1.7 Hz, 1H), 6.94 (dd, J = 1.8, 0.6 Hz, 1H), 6.54 (dd, J = 1.7, 0.5 Hz, 1H), 4.34 (q, J = 7.1 Hz, 2H), 2.61 (s, 3H), 1.38 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.3 (s), 158.0 (s) 149.0 (s), 145.3 (d), 143.3 (d), 143.0 (d), 139.9 (d), 116.7 (s), 114.8 (s), 112.4 (d), 107.9 (s), 107.6 (d), 102.1 (s), 86.9 (s), 82.9 (s), 60.4 (t), 14.2 (q), 14.0 (q). IR (reflection) $\tilde{\nu}$ = 3137, 2994, 2227, 1700, 1597, 1512, 1475, 1444, 1414, 1377, 1333, 1260, 1236, 1161, 1148, 1103, 1085, 1056, 1017, 977, 937, 873, 840, 803, 782, 735, 695, 634 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{18}H_{15}O_5$ $[M+H]^+$: 311.0914, found: 311.0918.

ethyl 5-butyl-4-(hex-1-yn-1-yl)-2-methylfuran-3-carboxylate (3an)



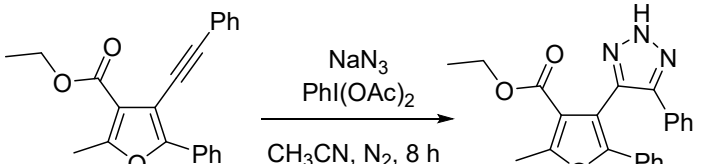
Yield: 18 mg, 62%; yellow liquid; R_f = 0.82 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.29 (q, J = 7.1 Hz, 2H), 2.66 (t, J = 7.4 Hz, 2H), 2.50 (s, 3H), 2.43 (t, J = 7.0 Hz, 2H), 1.66 – 1.56 (m, 4H), 1.52 – 1.44 (m, 2H), 1.38 – 1.31 (m, 5H), 0.93 (dd, J = 13.6, 7.3 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.8 (s), 158.4 (s), 157.2 (s), 113.8 (s), 103.6 (s), 94.2 (s), 71.4 (s), 60.0 (t), 31.0 (t), 29.9 (t), 26.3 (t), 22.1 (t), 22.0 (t), 19.4 (t), 14.3 (q), 14.0 (q), 13.7 (q), 13.6 (q). IR (reflection) $\tilde{\nu}$ = 2959, 2933, 2873, 2222, 1711, 1610, 1585, 1465, 1429, 1378, 1296, 1228, 1192, 1144, 1085 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{18}H_{27}O_3$ $[M+H]^+$: 291.1955, found: 291.1960.

4. Diverse Transformations of 3aa



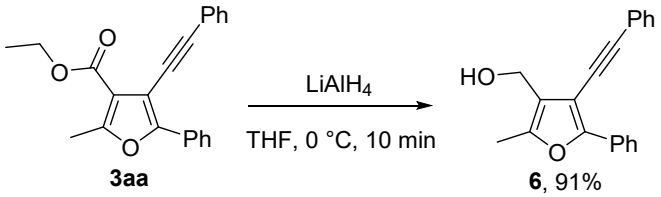
3aa **4**, 70%

A mixture of **3aa** (0.1 mmol), pyridine 1-oxide (0.2 mmol) and $Ph_3PAuNTf_2$ (5 mol %) in 1.0 mL THF and then heated to 60 °C in an oil bath. The reactions were monitored by TLC analysis and the **3aa** was consumed completely (about 5 h). The solvent was removed under vacuum and the crude residue was purified by silica gel column chromatography to give the desired products **4**. Yield: 27.0 mg, 70%; yellow liquid; R_f = 0.45 (PE/EA = 5/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 – 8.11 (m, 2H), 7.87 – 7.80 (m, 2H), 7.66 – 7.59 (m, 1H), 7.56 – 7.48 (m, 2H), 7.44 – 7.36 (m, 3H), 3.99 (q, J = 7.1 Hz, 2H), 2.66 (s, 3H), 1.07 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 189.3 (s), 188.9 (s), 163.1 (s), 158.2 (s), 155.5 (s), 133.8 (d), 133.0 (s), 130.7 (d, 2C), 129.9 (d), 128.7 (s), 128.45 (d, 2C), 128.43 (d, 2C), 127.7 (d, 2C), 118.9 (s), 114.9 (s), 60.9 (t), 14.1 (q), 13.8 (q). IR (reflection) $\tilde{\nu}$ = 2982, 1701, 1676, 1597, 1580, 1559, 1489, 1449, 1426, 1331, 1265, 1232, 1154, 1100, 1067, 1025, 974, 914, 849, 787, 756, 696 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{22}H_{19}O_5$ $[M+H]^+$: 363.1227, found: 363.1224.

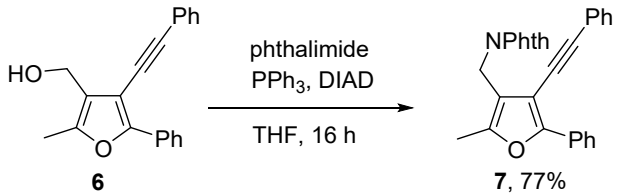


3aa **5**, 65%

The mixture of **3aa** (0.1 mmol), NaN_3 (1.5 equiv), and $PhI(OAc)_2$ (1.5 equiv) in MeCN (2.0 mL) was stirred at room temperature under ambient nitrogen for 8 h, The solvent was removed under vacuum and the crude residue was purified by silica gel column chromatography to give the desired product **5**. Yield: 24 mg, 65%; yellow liquid; R_f = 0.40 (PE/EA = 3/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.08 (brs, 1H), 7.66 – 7.60 (m, 2H), 7.38 – 7.33 (m, 2H), 7.31 – 7.27 (m, 1H), 7.26 – 7.24 (m, 2H), 7.23 – 7.17 (m, 3H), 3.95 (q, J = 7.1 Hz, 2H), 2.72 (s, 3H), 0.93 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.3 (s), 159.6 (s), 150.3 (s), 145.43 (s), 145.37 (s), 130.3 (s), 129.4 (s), 128.64 (d, 2C), 128.59 (d, 2C), 128.28 (d), 128.27 (d), 126.6 (d, 2C), 125.4 (d, 2C), 115.6 (s), 119.9 (s), 60.1 (t), 14.2 (q), 13.6 (q). IR (reflection) $\tilde{\nu}$ = 2983, 2927, 2250, 2113, 1714, 1597, 1446, 1382, 1322, 1239, 1212, 1180, 1101, 984, 948, 912, 768, 734, 694, 665, 648 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{22}H_{20}N_3O_3$ $[M+H]^+$: 374.1488, found: 374.1488.



The solution of **3aa** (0.1 mmol) in 1.0 mL THF at 0 °C was slowly added $LiAlH_4$ (0.2 mmol). The resulting solution was warmed to room temperature and stirred for 30 min. The solvent was diluted with water (2.0 mL) and extracted with ethyl acetate and dried over anhydrous $MgSO_4$. After the solvent was evaporated, the crude product was purified by column chromatography give **6**. Yield: 26 mg, 91%; white solid, mp 99-100 °C; R_f = 0.23 (PE/EA = 5/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.08 (dt, J = 8.2, 1.6 Hz, 2H), 7.55 (m, 2H), 7.46 – 7.34 (m, 5H), 7.33 – 7.28 (m, 1H), 4.63 (s, 2H), 2.40 (s, 3H), 1.77 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.9 (s), 148.9 (s), 131.4 (d, 2C), 130.4 (s), 128.6 (d, 2C), 128.5 (d, 2C), 128.4 (d), 127.8 (d), 124.5 (d, 2C), 123.3 (s), 122.2 (s), 103.5 (s), 96.0 (s), 81.6 (s), 55.6 (t), 11.8 (q). IR (reflection) $\tilde{\nu}$ = 3237, 3058, 2924, 2871, 2215, 1629, 1603, 1561, 1485, 1443, 1372, 1324, 1256, 1125, 1073, 993, 910, 797, 765, 747, 725, 682, 645 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{20}H_{17}O_2$ $[M+H]^+$: 289.1223, found: 289.1217.

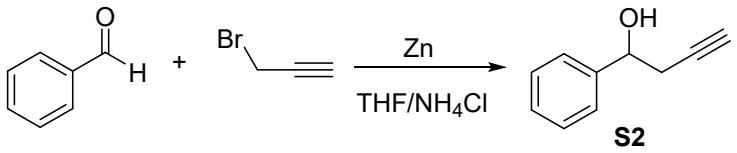


Alcohol **6** (0.1 mmol) was dissolved in anhydrous THF (1.0 mL) and the solution was cooled in an ice-bath. Triphenylphosphane (0.2 mmol), diisopropylazodicarboxylate (DIAD, 0.2 mmol) and phthalimide (0.2 mmol) were added sequentially into the solution. The reaction was stirred at 0 °C for 3 h and then warmed to room temperature. After stirring at room temperature for 12 h, the solution was extracted by EtOAc and washed with water. The organic phase was combined, dried over Na_2SO_4, and concentrated in vacuum. The residue was purified by flash chromatography. Yield: 32 mg, 77%; white solid, mp 193-194 °C; R_f = 0.38 (PE/EA = 5/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (dd, J = 8.4, 1.1 Hz, 2H), 7.81 – 7.75 (m, 2H), 7.67 – 7.61 (m, 2H), 7.57 (dt, J = 8.3, 2.2 Hz, 2H), 7.42 – 7.31 (m, 5H), 7.29 – 7.24 (m, 1H), 4.79 (s, 2H), 2.53 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.8 (s, 2C), 152.9 (s), 150.2 (s), 133.8 (d, 2C), 132.2 (s, 2C), 131.5 (d, 2C), 130.4 (s), 128.4 (d, 2C), 128.2 (d, 2C), 128.1 (d), 127.7 (d), 124.5 (d, 2C), 123.6 (s), 123.2 (d, 2C), 117.2 (s), 103.8 (s), 96.3 (s), 81.8 (s), 32.1 (t), 12.1 (q). IR (reflection) $\tilde{\nu}$ = 3472, 3082, 2921, 1774, 1713,

1633, 1597, 1498, 1485, 1469, 1436, 1396, 1359, 1313, 1252, 1189, 1149, 1112, 1088, 1071, 1040, 1025, 938, 912, 870, 793, 761, 727, 715, 688, 660, 643, 615 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{28}H_{20}NO_3$ $[M+H]^+$: 418.1438, found: 418.1439.

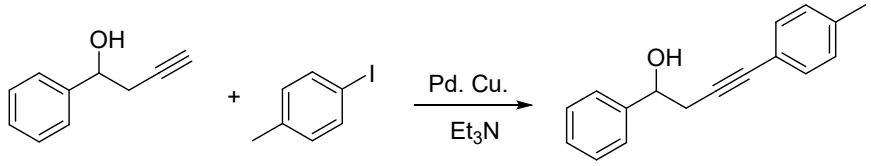
5. Mechanistic Experiments

Preparation of substrates 8:



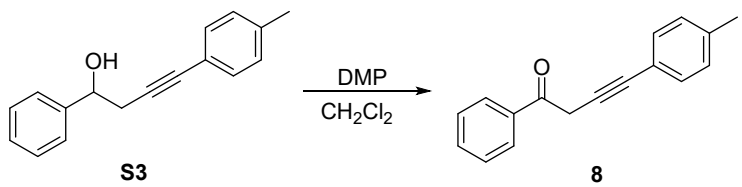
S2

Propargyl bromide (2 equiv) was added to a mixture of the zinc dust (2 equiv) and the aldehydes (1 mmol) in THF/NH_4Cl (1:1) (6 mL) at room temperature. Then, the mixture was stirred at room temperature and monitored by TLC analysis. When the start material was disappeared, the mixture was extracted with diethyl ether (3 × 5 mL) and the organic extract was washed with brine, dried ($MgSO_4$) and concentrated under reduced pressure. The crude residue was purified by silica gel column chromatography to obtain **S2**. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40 – 7.30 (m, 5H), 4.86 (td, *J* = 6.4, 3.5 Hz, 1H), 2.64 (dd, *J* = 6.4, 2.6 Hz, 2H), 2.58 (d, *J* = 3.6 Hz, 1H), 2.07 (t, *J* = 2.6 Hz, 1H). The spectroscopic data is in agreement with that previously reported.[3]



S2 **S3**

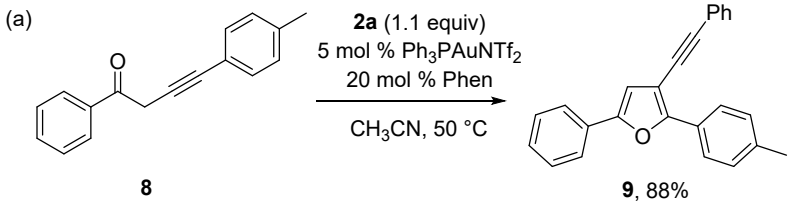
To a dried schlenk flask was added $Pd(PPh_3)_2Cl_2$ (5 mol %), CuI (10 mol %), 4-Iodotoluene (1.1 mmol), **S2** (1.0 mmol) and Et_3N under argon. The resulting mixture was stirred for 16 h at rt. EtOAc were added and the mixture filtered. After removal of solvent using rotary evaporator, the crude compound was purified by silica gel column chromatography to obtain **S3**. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 – 7.27 (m, 7H), 7.10 (d, *J* = 7.9 Hz, 2H), 4.95 (t, *J* = 6.4 Hz, 1H), 2.91 – 2.80 (m, 2H), 2.51 (dd, *J* = 9.2, 5.0 Hz, 1H), 2.34 (s, 3H). The spectroscopic data is in agreement with that previously reported.[3]



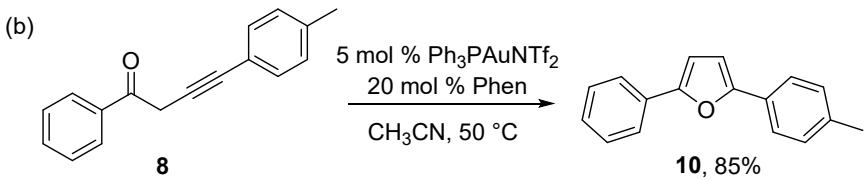
S3 **8**

A solution of the above prepared alcohol **S3** (1.0 mmol) in dichloromethane (5.0 mL) was added to Dess-Martin periodinane (DMP) (1.5 mmol) stirring at room

temperature. After disappearance of the starting material (TLC), the reaction mixture was poured into a saturated aqueous $Na_2S_2O_3$ solution and neutralized with saturated with Na_2CO_3 solution. The combined organic layers were washed with brine, dried over $MgSO_4$ and concentrated. The crude extracts were purified by silica gel column chromatography to obtain **8**. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.12 – 8.03 (m, 2H), 7.61 (dt, *J* = 2.7, 1.8 Hz, 1H), 7.53 (dd, *J* = 6.3, 1.4 Hz, 2H), 7.33 (d, *J* = 8.1 Hz, 2H), 7.11 (d, *J* = 7.9 Hz, 2H), 4.09 (s, 2H), 2.35 (s, 3H). The spectroscopic data is in agreement with that previously reported.[4]



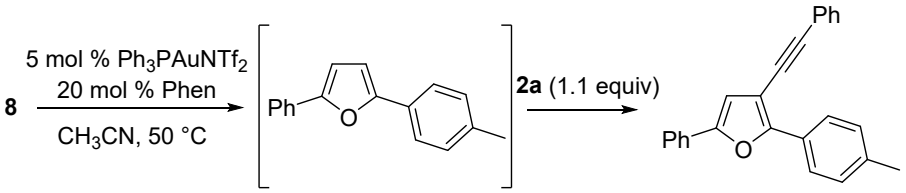
A mixture of **8** (0.1 mmol) and **2a** (1.1 equiv) in 1.0 mL CH_3CN was treated with $Ph_3PAuNTf_2$ (5 mol %), Phen (20 mol%) and then heated to 50 °C in an oil bath. The reactions were monitored by TLC analysis and the chemical **8** was consumed completely. The solvent was removed under vacuum and the crude residue was purified by silica gel column chromatography to give the desired products **9**. Yield: 28 mg, 84%; white solid, mp 120-121 °C; R_f = 0.78 (PE/EA = 10/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 – 8.08 (m, 2H), 7.75 (dd, *J* = 5.2, 3.3 Hz, 2H), 7.62 – 7.56 (m, 2H), 7.48 – 7.35 (m, 5H), 7.34 – 7.27 (m, 3H), 6.84 (s, 1H), 2.42 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.4 (s), 152.0 (s), 138.2 (s), 131.4 (d, 2C), 130.1 (s), 129.3 (d, 2C), 128.8 (d, 2C), 128.4 (d, 2C), 128.2 (d), 127.82 (d), 127.75 (s), 124.9 (d, 2C), 123.9 (d, 2C), 123.5 (s), 109.9 (d), 104.1 (s), 93.8 (s), 83.0 (s), 21.4 (q). IR (reflection) $\tilde{\nu}$ = 3031, 2917, 2855, 2207, 1598, 1509, 1482, 1442, 1262, 1157, 1113, 1056, 1028, 930, 915, 818, 799, 754, 714, 691, 684, 656, 644, 614 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{25}H_{19}O$ $[M+H]^+$: 335.1430, found: 335.1429.



A chemical of **8** (0.1 mmol) in 1.0 mL CH_3CN was treated with $Ph_3PAuNTf_2$ (5 mol %), Phen (20 mol%) and then heated to 50 °C in an oil bath. The reactions were monitored by TLC analysis and the chemical **8** was consumed completely. The solvent was removed under vacuum and the crude residue was purified by silica gel column chromatography to give the desired products **10**. Yield: 22 mg, 94%; white solid, mp

98-99 °C; R_f = 0.80 (PE/EA = 10/1); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.70 – 7.62 (m, 2H), 7.62 – 7.52 (m, 2H), 7.37 – 7.27 (m, 2H), 7.24 – 7.08 (m, 3H), 6.64 (d, J = 3.5 Hz, 1H), 6.59 (d, J = 3.5 Hz, 1H), 2.29 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.6 (s), 153.0 (s), 137.3 (s), 130.9 (s), 129.4 (d, 2C), 128.7 (d, 2C), 128.1 (s), 127.2 (d), 123.72 (d, 2C), 123.66 (d, 2C), 107.2 (d), 106.5 (d), 21.3 (q). IR (reflection) $\tilde{\nu}$ = 3039, 3023, 2912, 2856, 2722, 1891, 1811, 1605, 1567, 1545, 1497, 1482, 1446, 1375, 1310, 1289, 1210, 1177, 1156, 1114, 1065, 1024, 967, 928, 910, 821, 794, 758, 715, 691, 672, 639, 619 cm^{-1}. HRMS (ESI, m/z) calc'd for $C_{17}H_{15}O$ $[M+H]^+$: 235.1117, found: 235.1119.

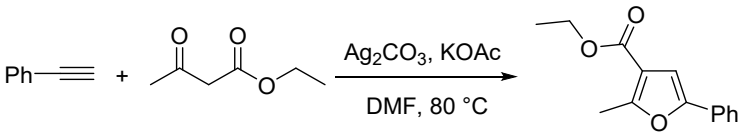
(c)



10 **9**, not observed

A mixture of **8** (0.1 mmol) in 1.0 mL CH_3CN was treated with $Ph_3PAuNTf_2$ (5 mol %), Phen (20 mol%) and then heated to 50 °C in an oil bath. The reactions were monitored by TLC analysis and the furan **10** was generated then added **2a**. The trisubstituted furan **9** not observed.

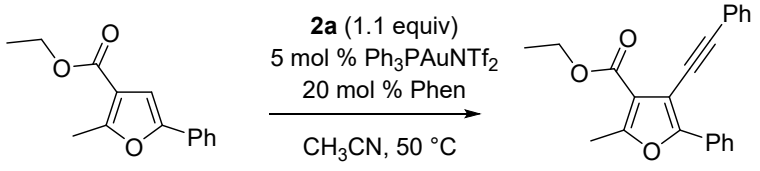
Preparation of substrates 11:



11, 80%

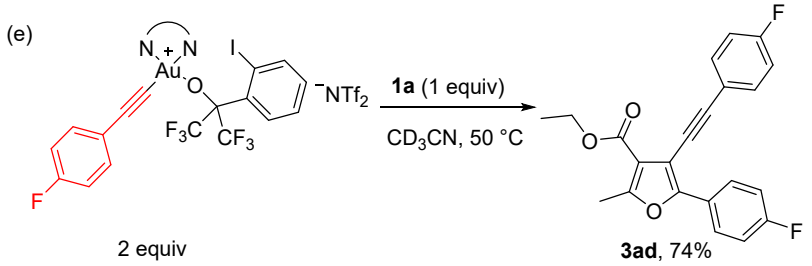
A mixture of phenylacetylene (0.25 mmol), ethyl acetoacetate (0.75 mmol), Ag_2CO_3 (0.50 mmol), and KOAc (0.50 mmol) in DMF was stirred in N_2 at 80 °C in an oil bath for 12 h. After completion of the reaction, the mixture was quenched with diluted hydrochloride, the solution was extracted with ethyl acetate. The organic layers were combined, and dried over sodium sulfate. The pure product was obtained by flash column chromatography on silica gel to afford **11** in 80% yield. ^{1}H NMR (301 MHz, $CDCl_3$) δ 7.57 – 7.41 (m, 2H), 7.21 (dd, J = 10.8, 4.2 Hz, 2H), 7.10 (dd, J = 9.1, 5.6 Hz, 1H), 6.74 (s, 1H), 4.20 (q, J = 7.0 Hz, 2H), 2.49 (s, 3H), 1.27 (t, J = 7.1 Hz, 3H). The spectroscopic data is in agreement with that previously reported.[5]

(d)



11 **3aa**, not observed

A mixture of **11** (0.1 mmol) and **2a** (1.1 equiv) in 1.0 mL CH_3CN was treated with $Ph_3PAuNTf_2$ (5 mol %), Phen (20 mol%) and then heated to 50 °C in an oil bath. The reactions were monitored by TLC analysis and not observed chemical **3aa**.



A J. Young tube was charge with $Ph_3PAuNTf_2$ and Phen in CD_3CN. Then alkynyliodonium reagents was added. The reaction was monitored by ^1H and ^{19}F NMR.[6] After determining the formation of **A**, **1a** were added. The reaction mixture was stirred at 50 °C in an oil bath. The reactions were monitored by TLC analysis and the desired products **3ad** was obtain.

6. References

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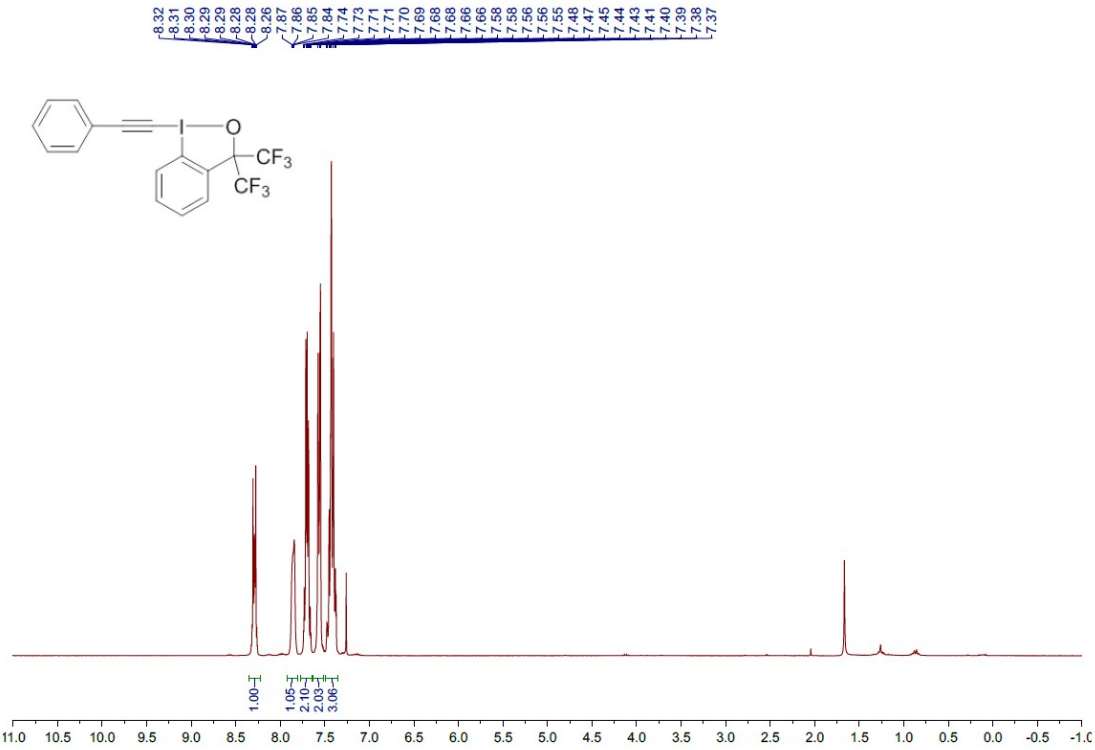
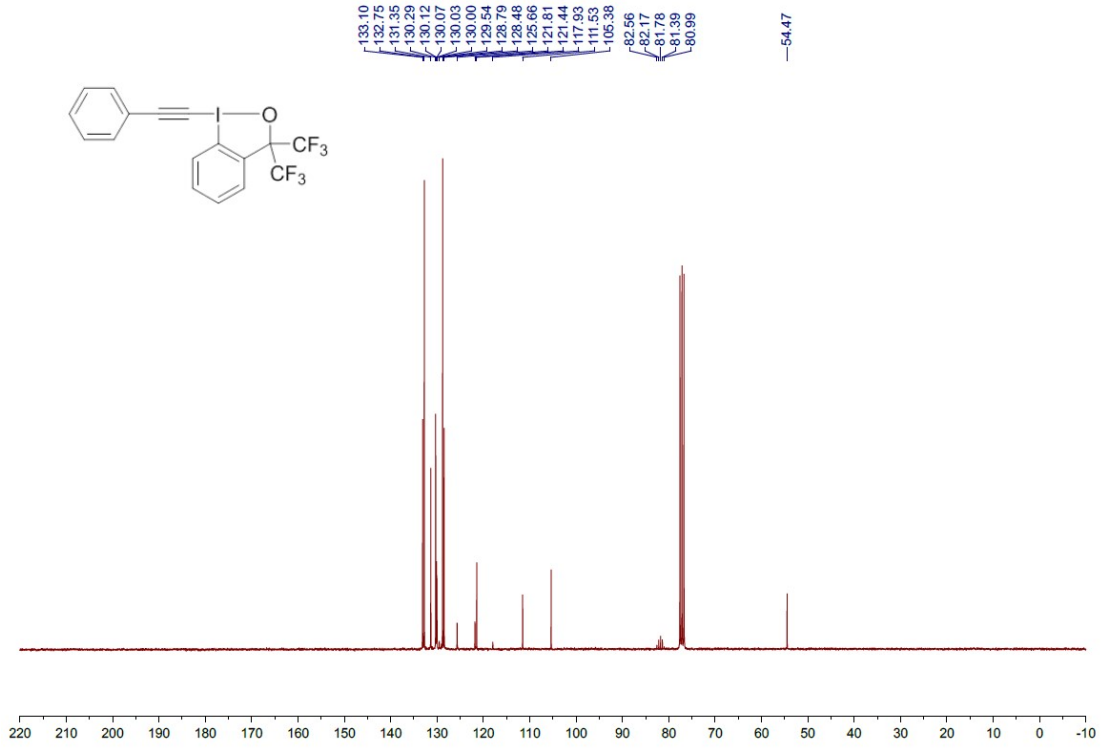
7. NMR Spectra

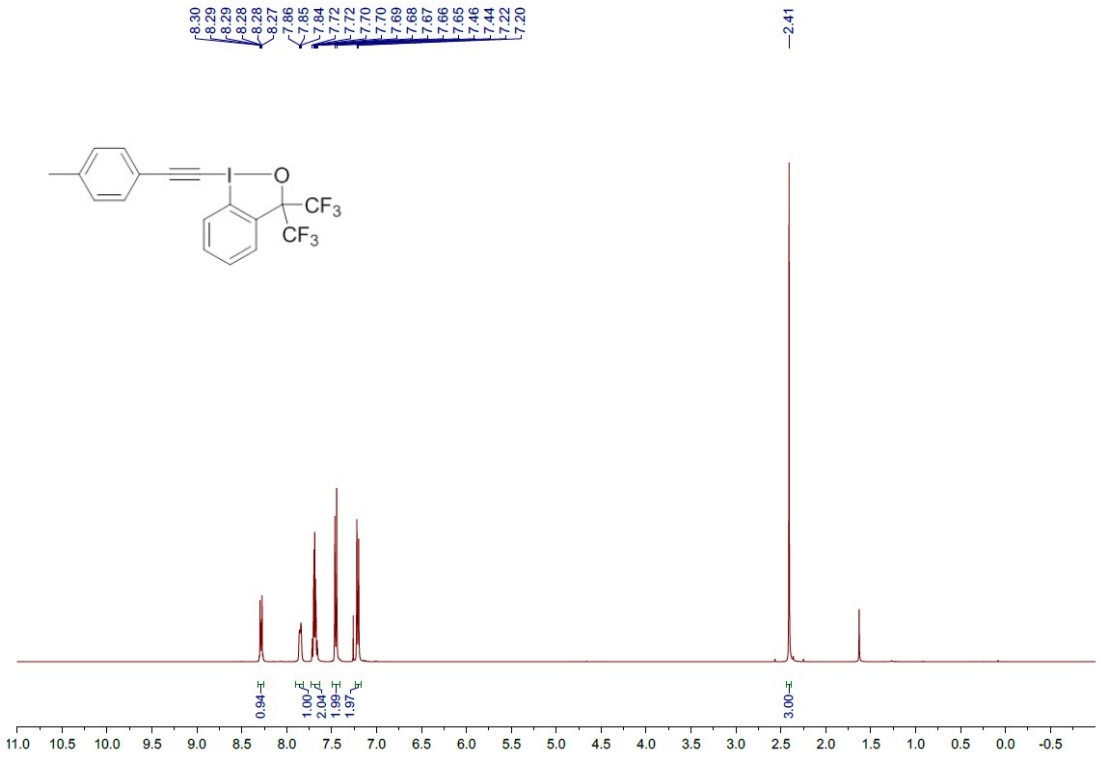
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of Compound **2a**



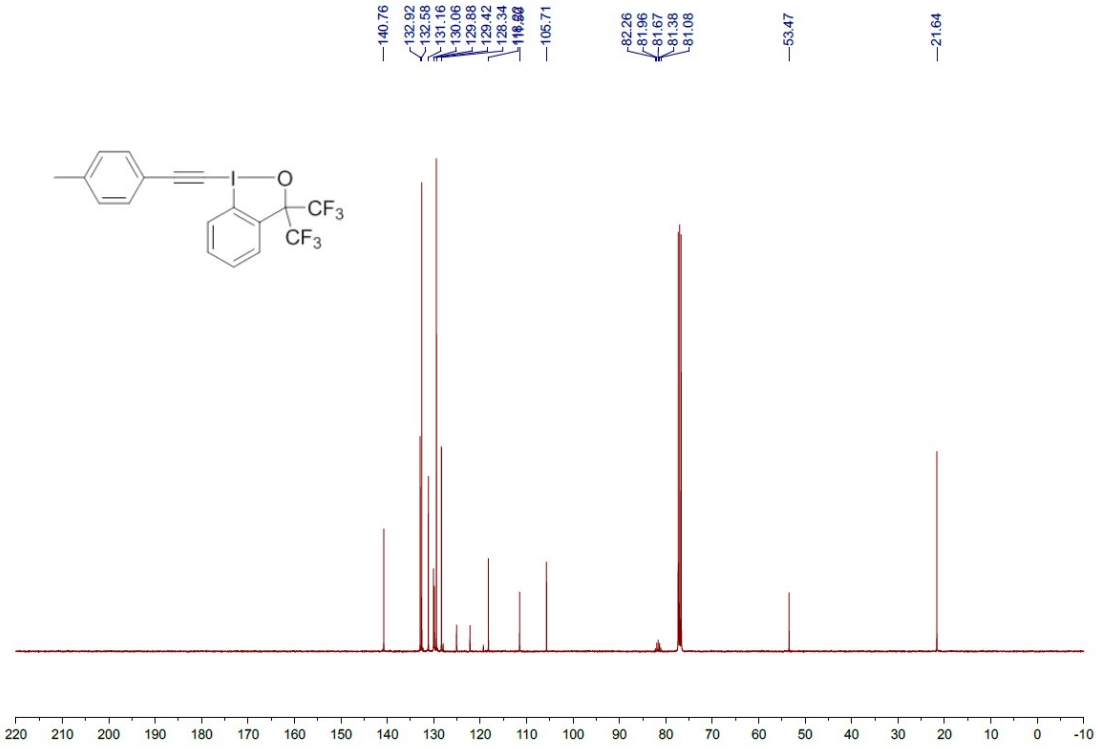
^{13}C NMR (75 MHz, $CDCl_3$) Spectrum of Compound **2a**



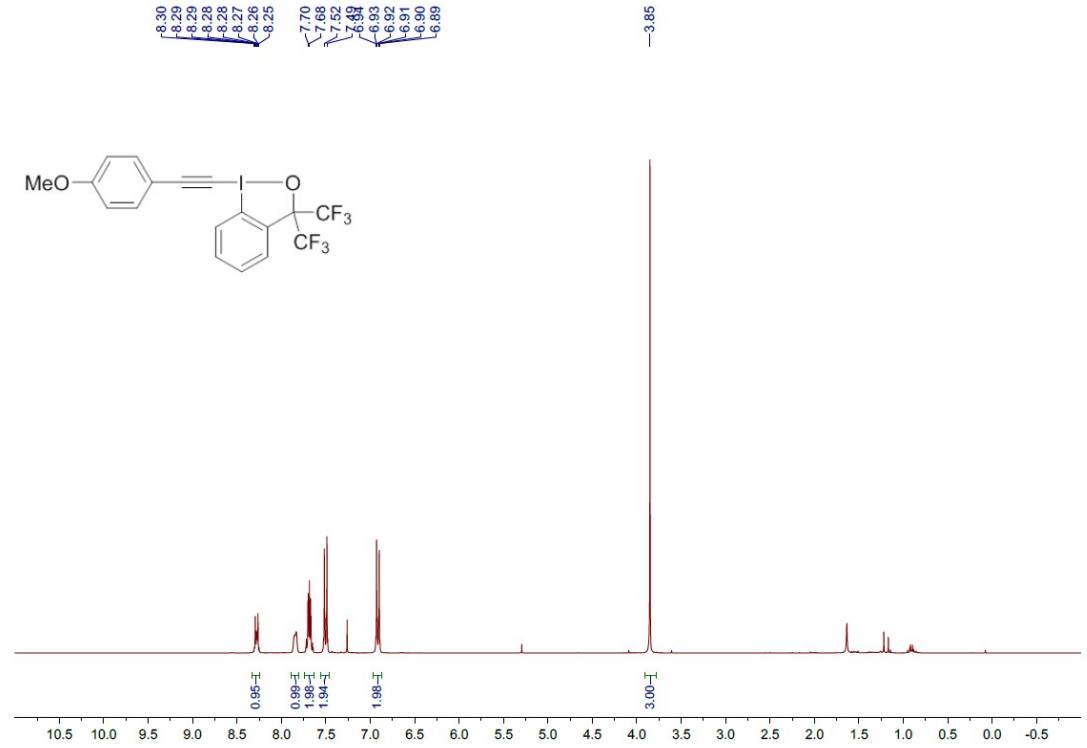
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **2b**



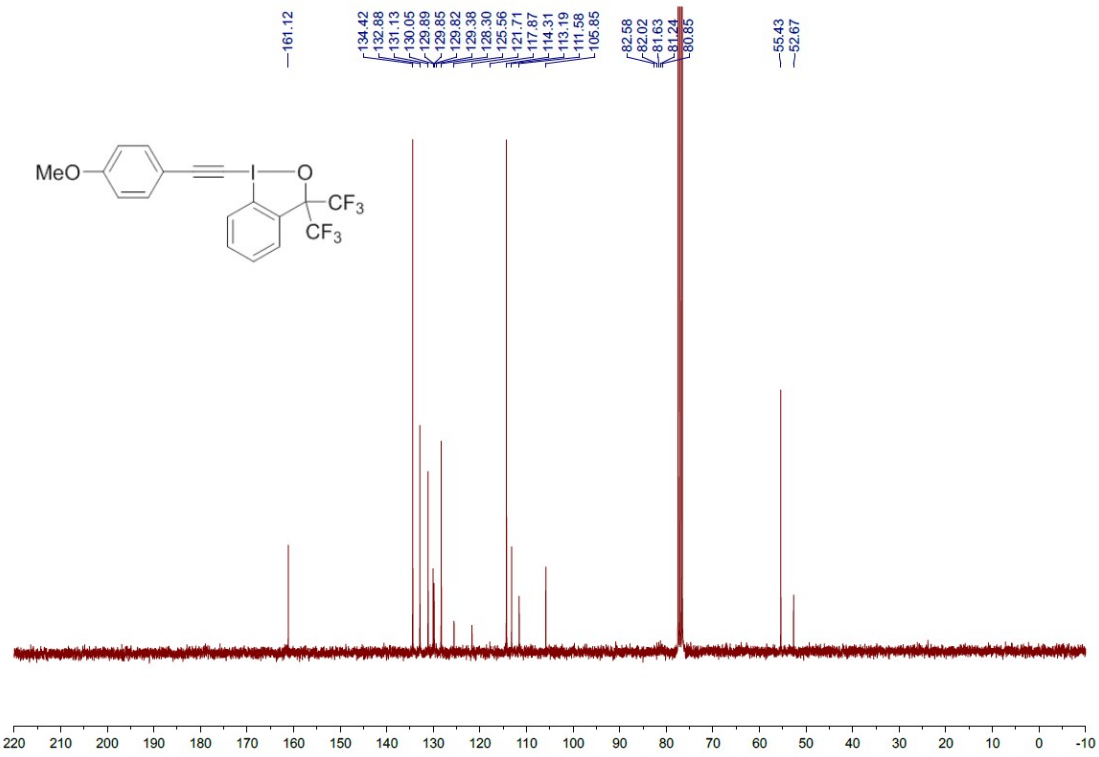
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **2b**



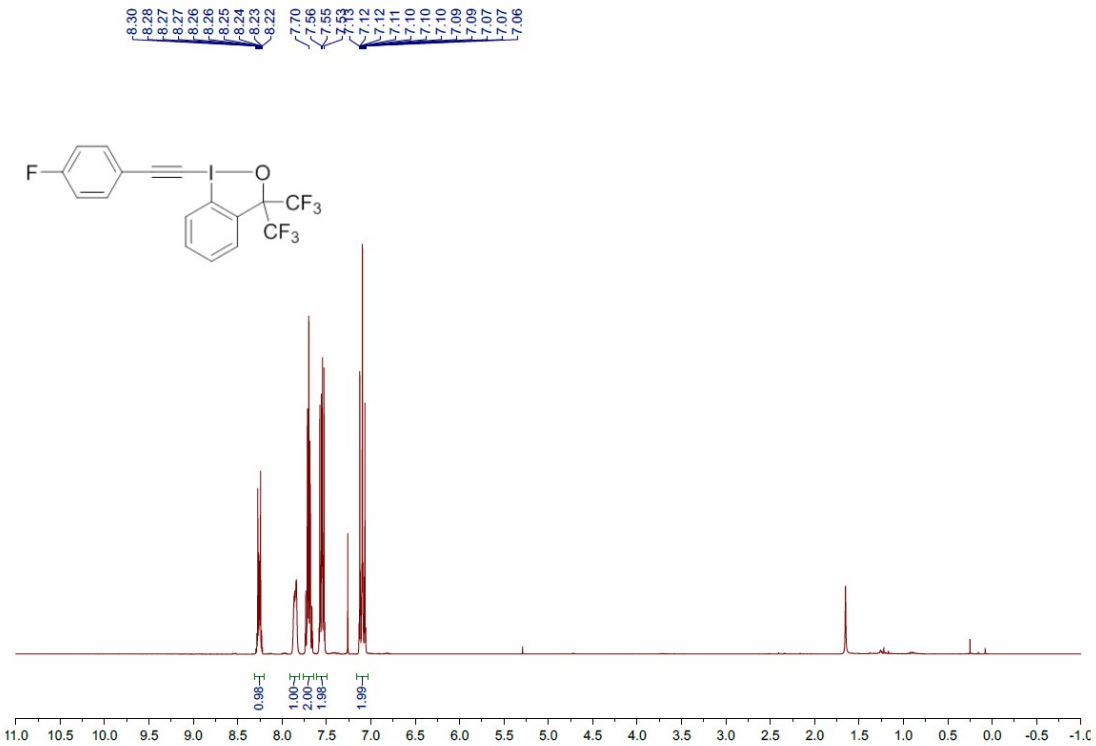
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of Compound **2c**



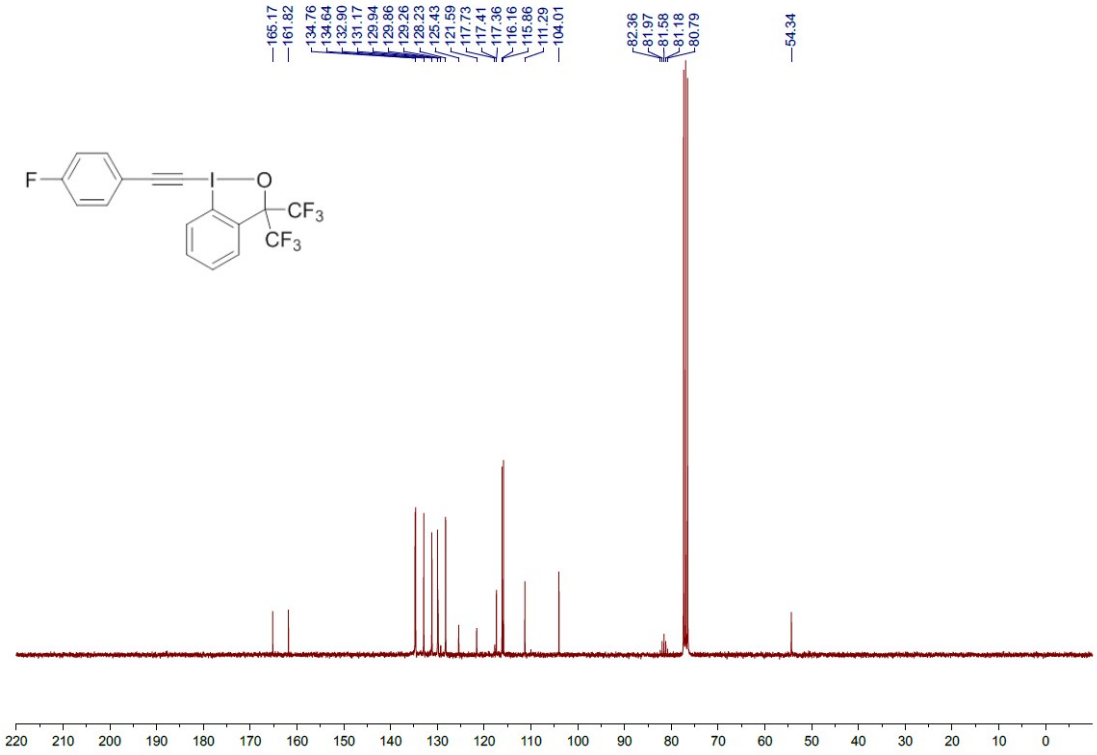
^{13}C NMR (75 MHz, $CDCl_3$) Spectrum of Compound **2c**



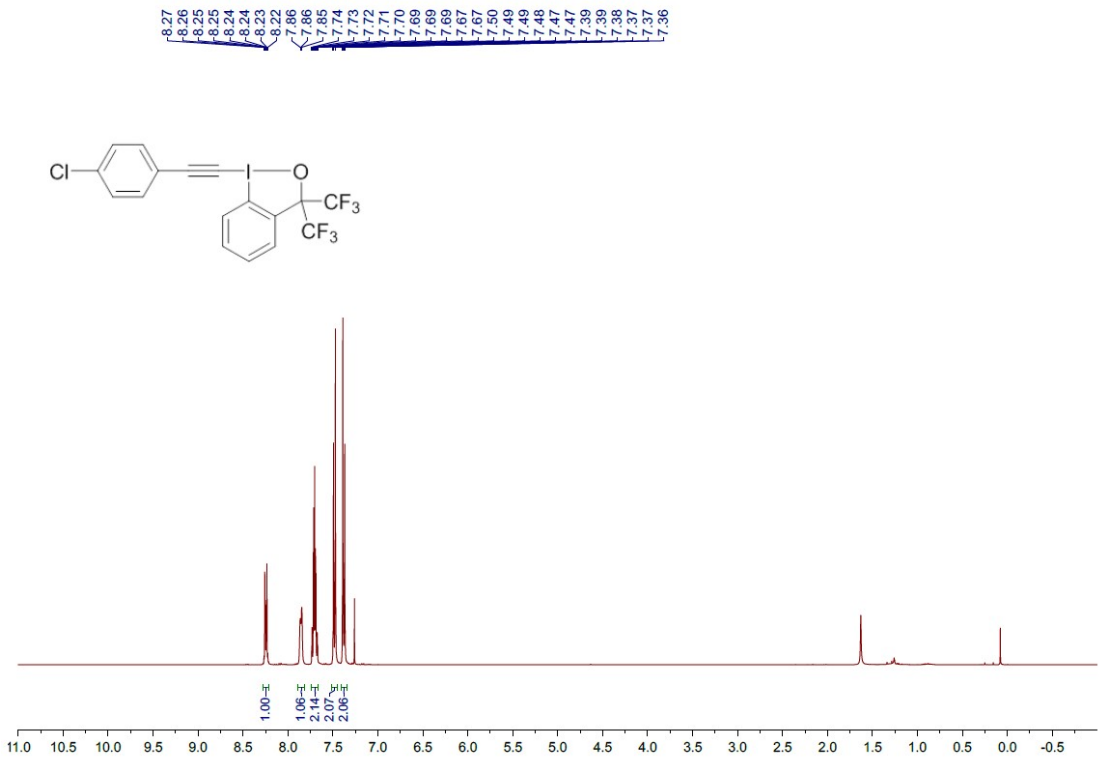
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of Compound **2d**



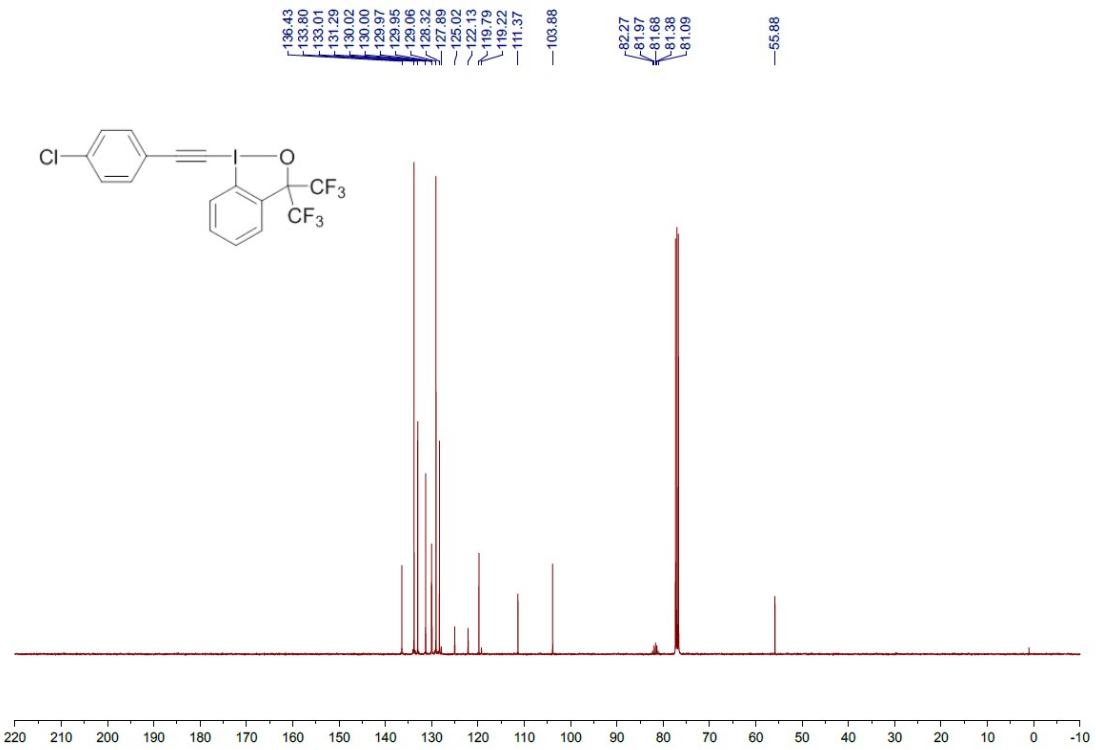
^{13}C NMR (75 MHz, $CDCl_3$) Spectrum of Compound **2d**



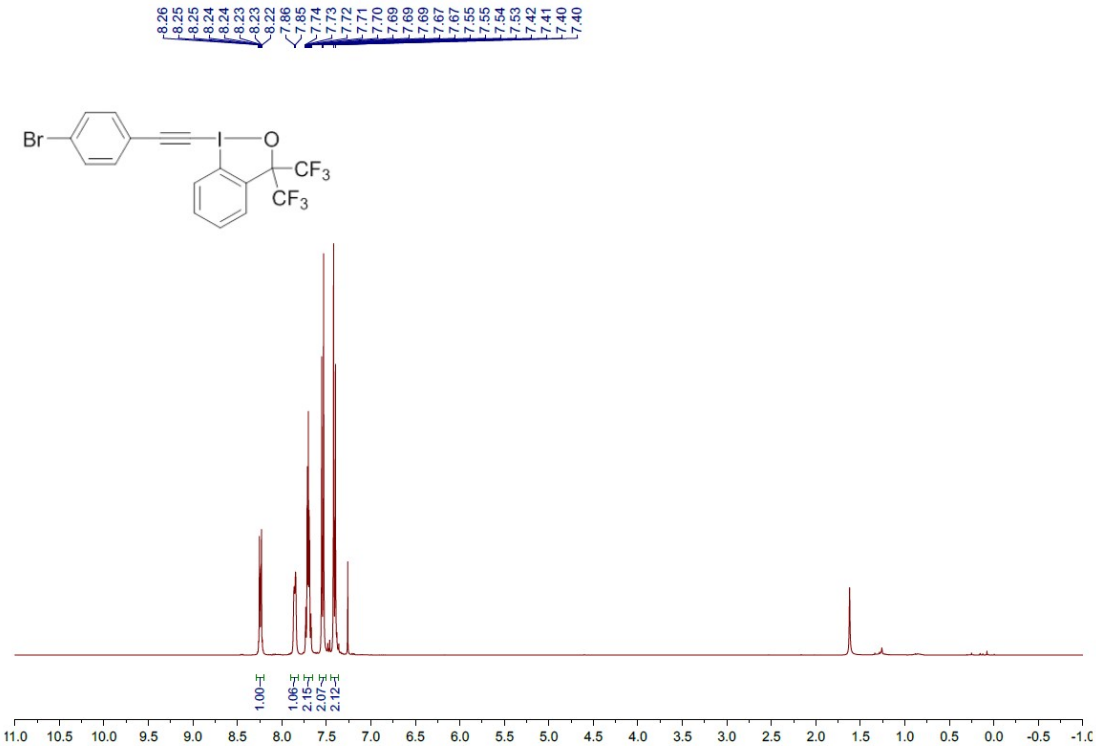
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **2e**



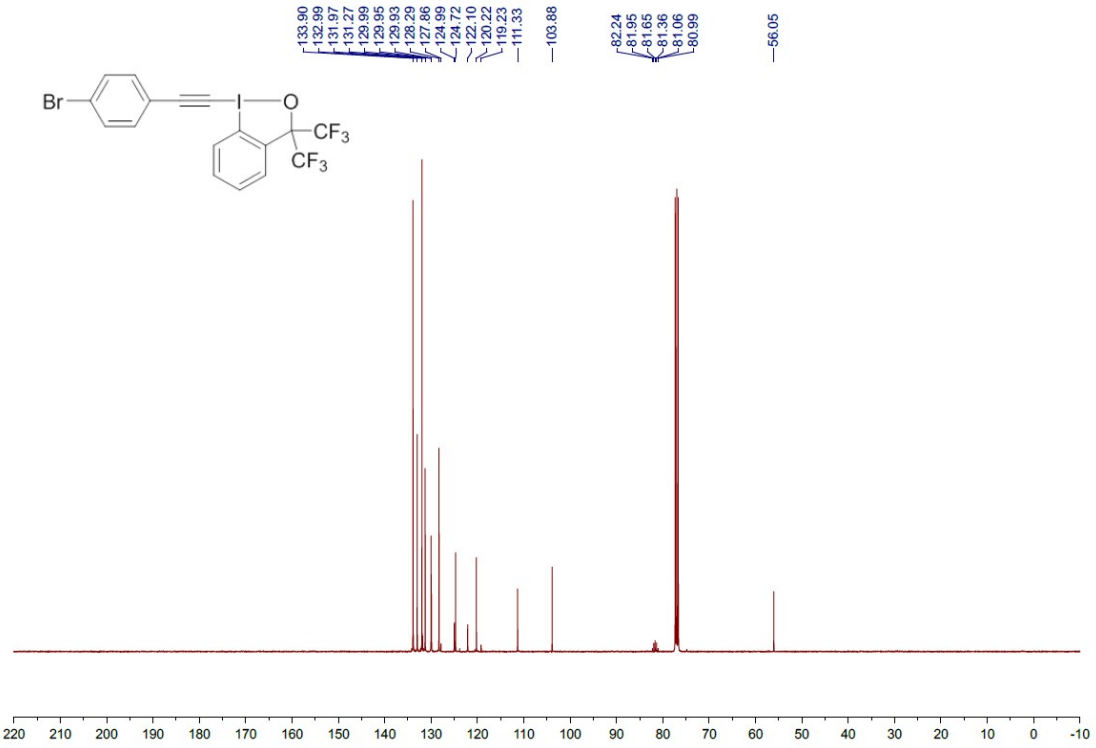
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **2e**



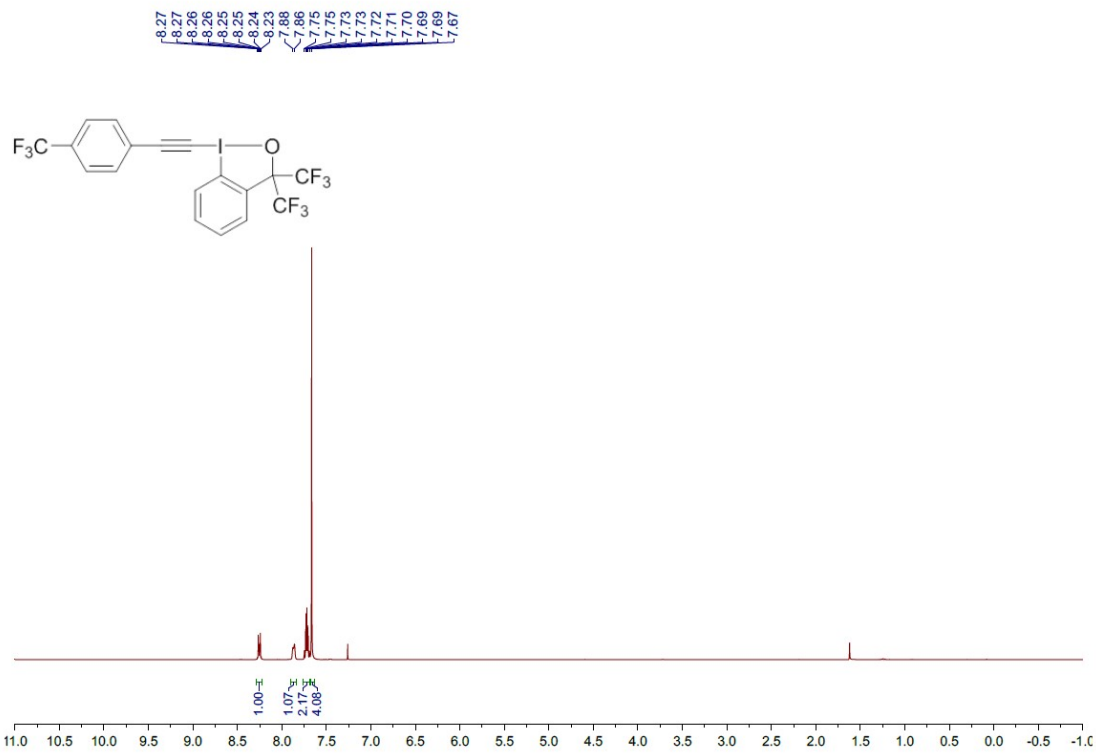
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **2f**



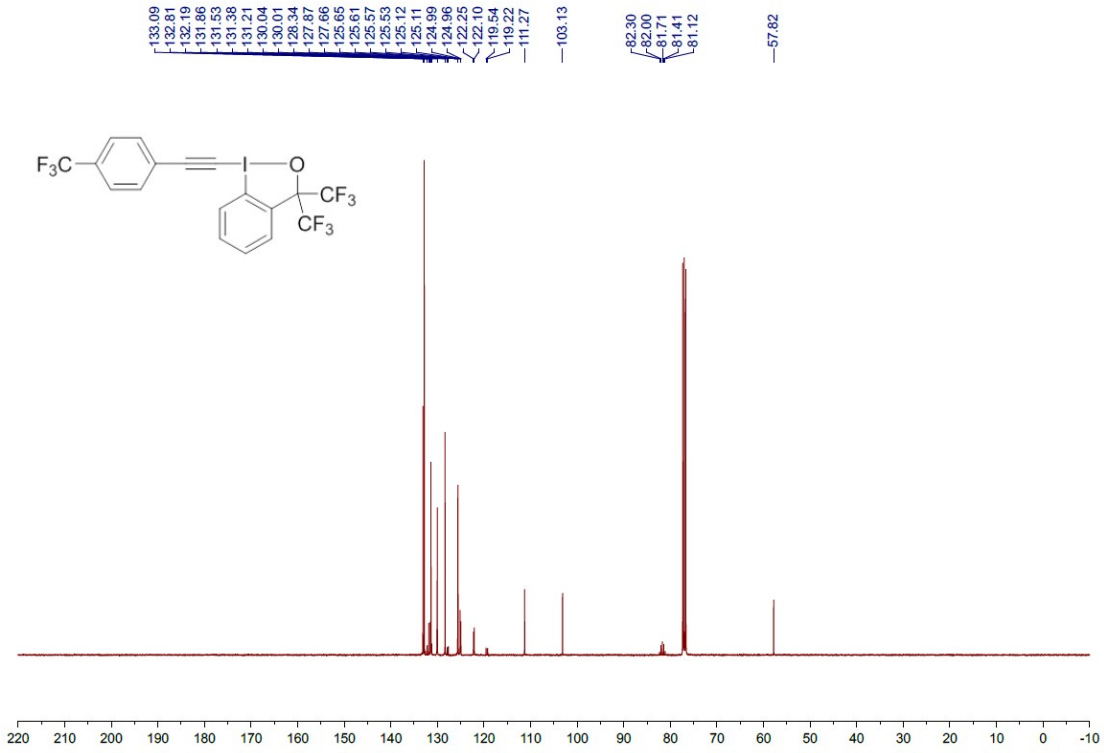
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **2f**



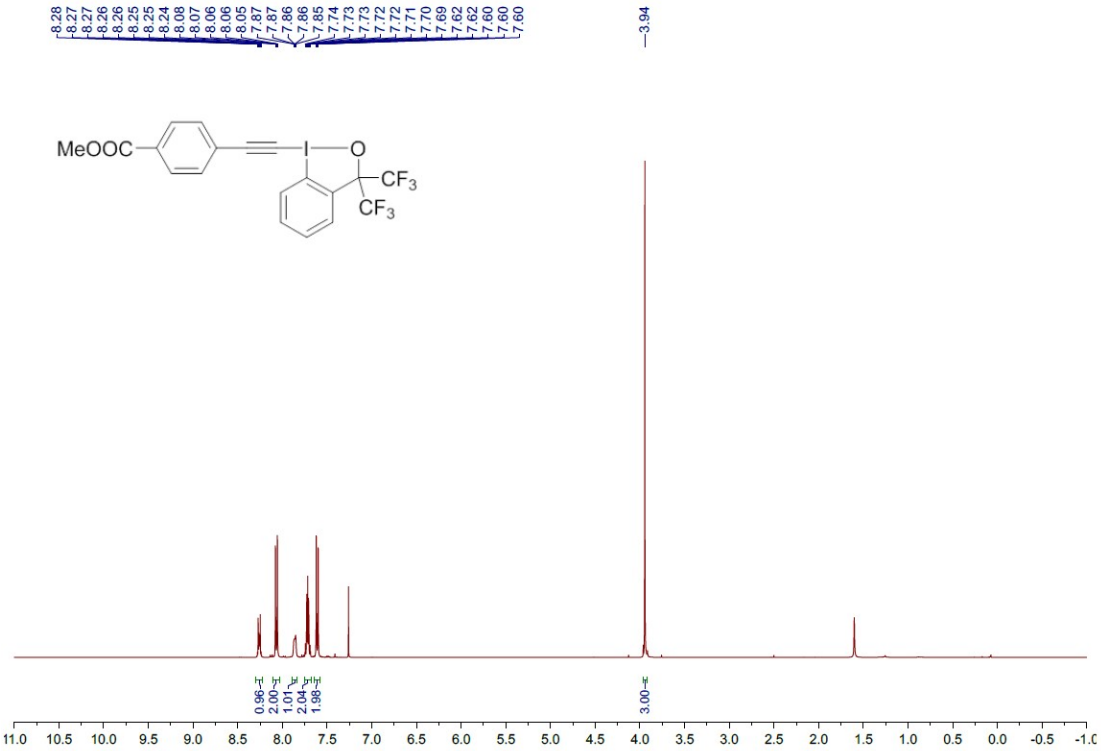
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **2g**



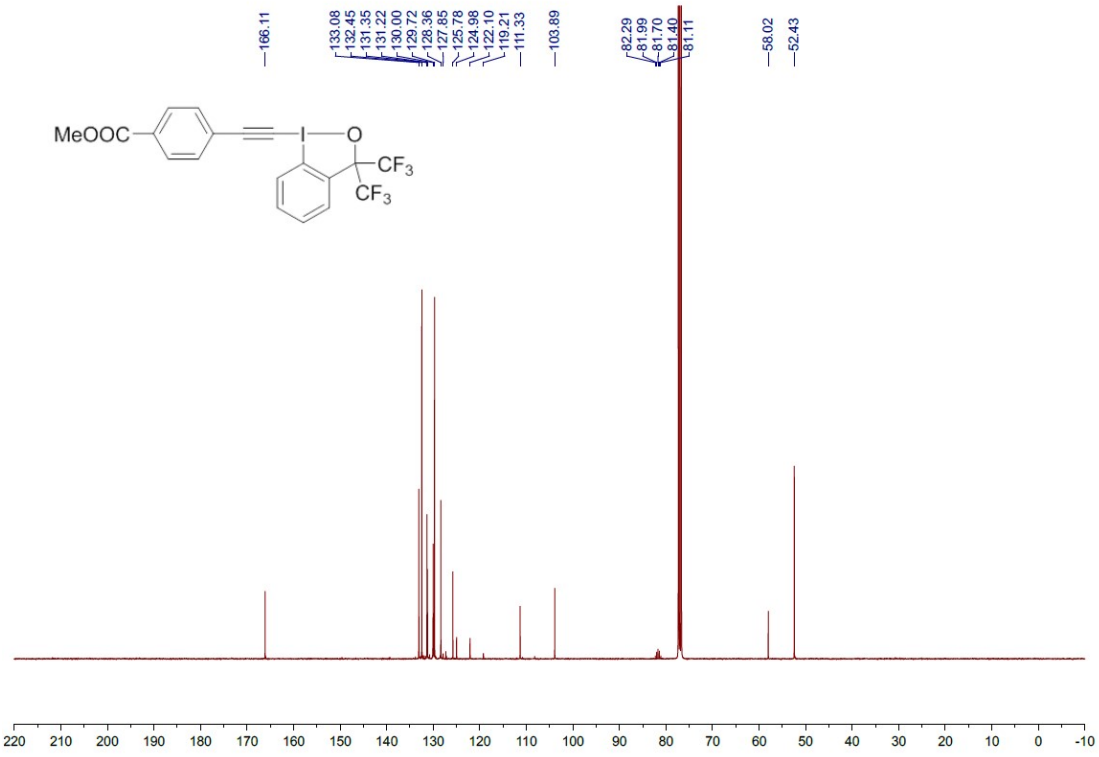
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **2g**



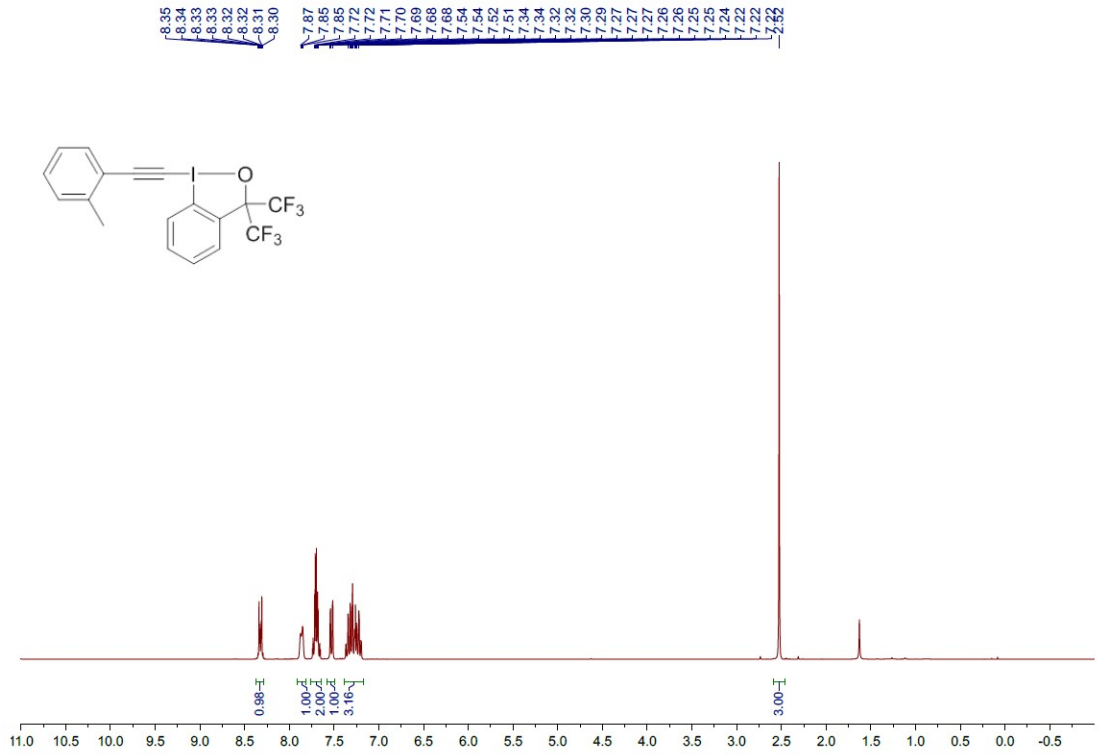
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **2h**



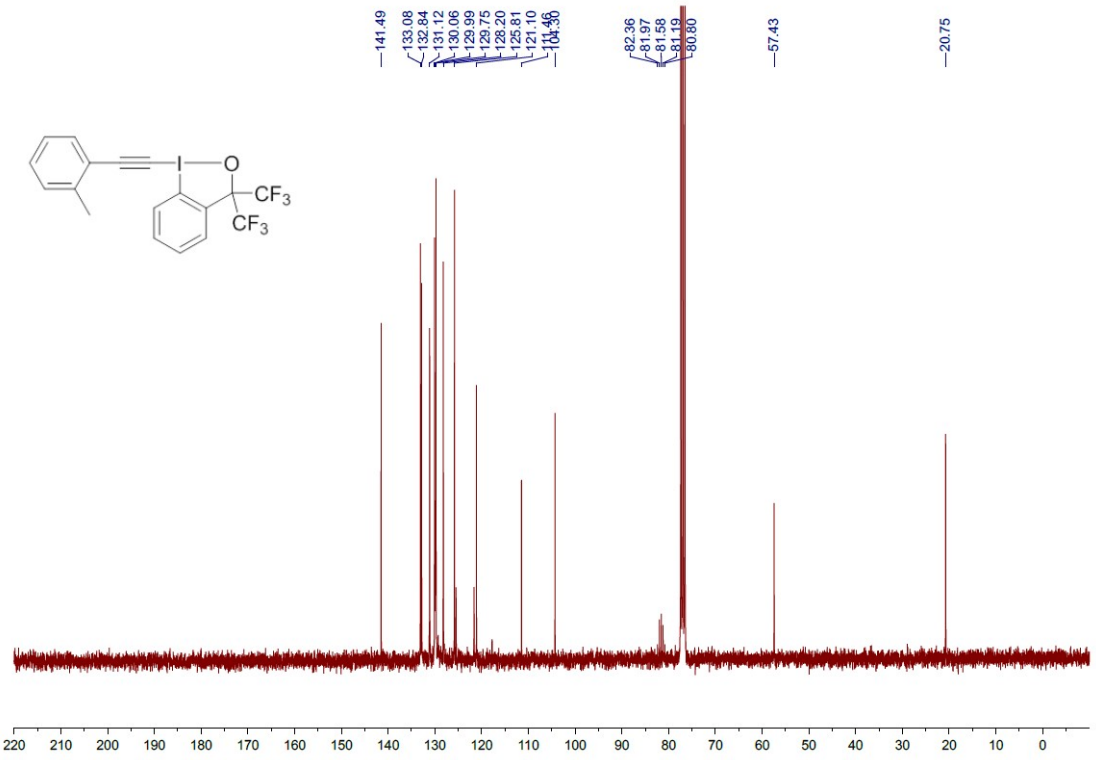
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **2h**



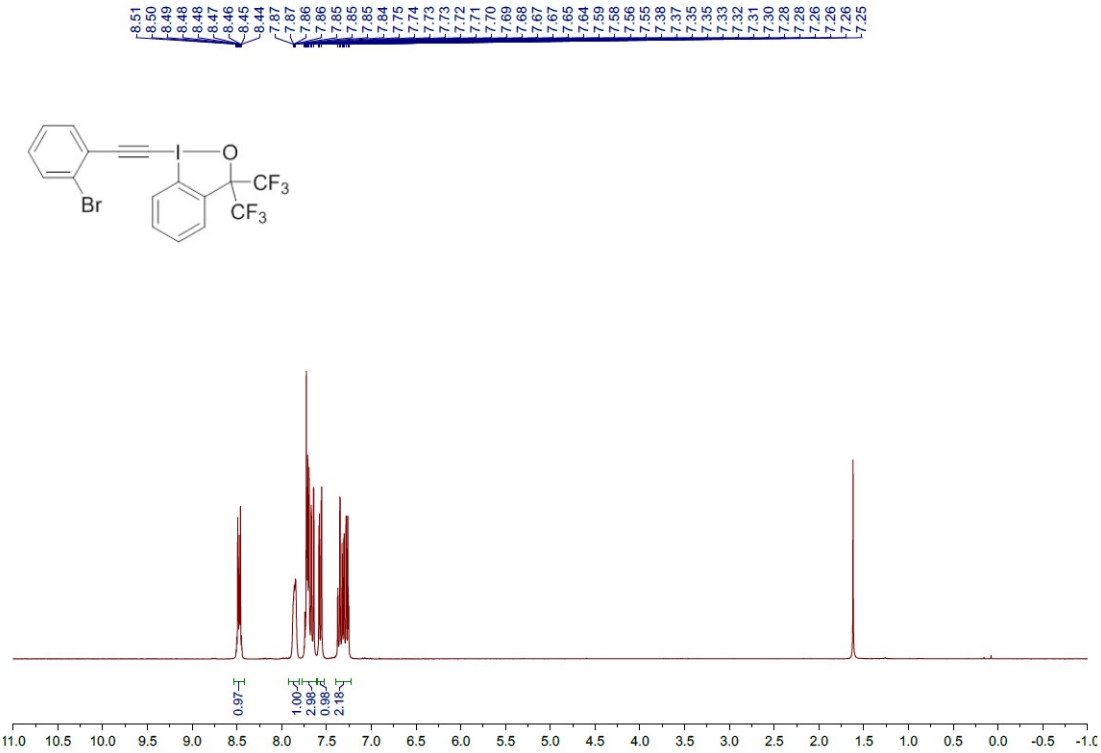
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of Compound **2i**



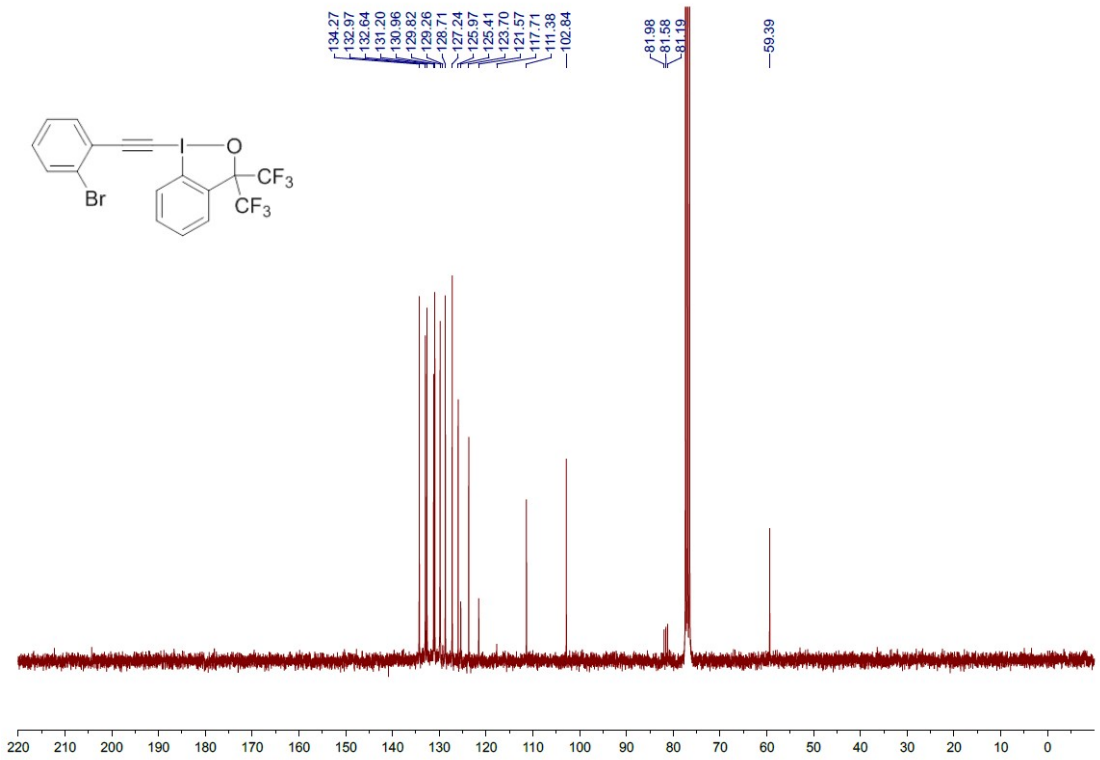
^{13}C NMR (75 MHz, $CDCl_3$) Spectrum of Compound **2i**



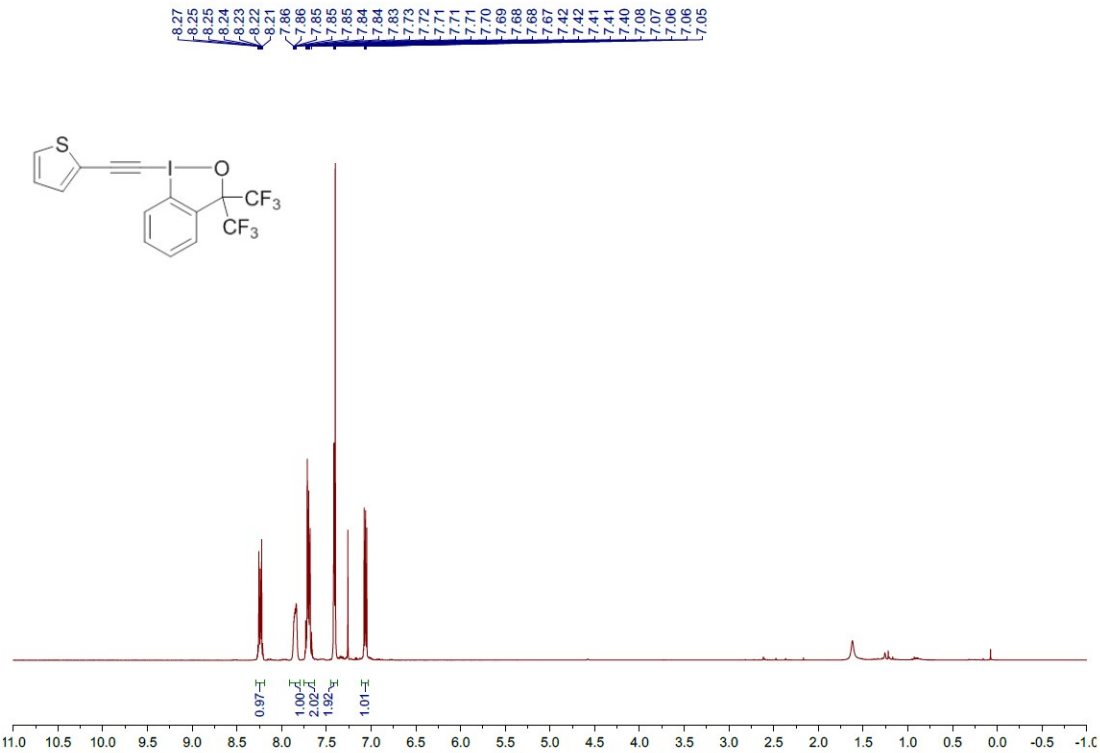
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of Compound **2j**



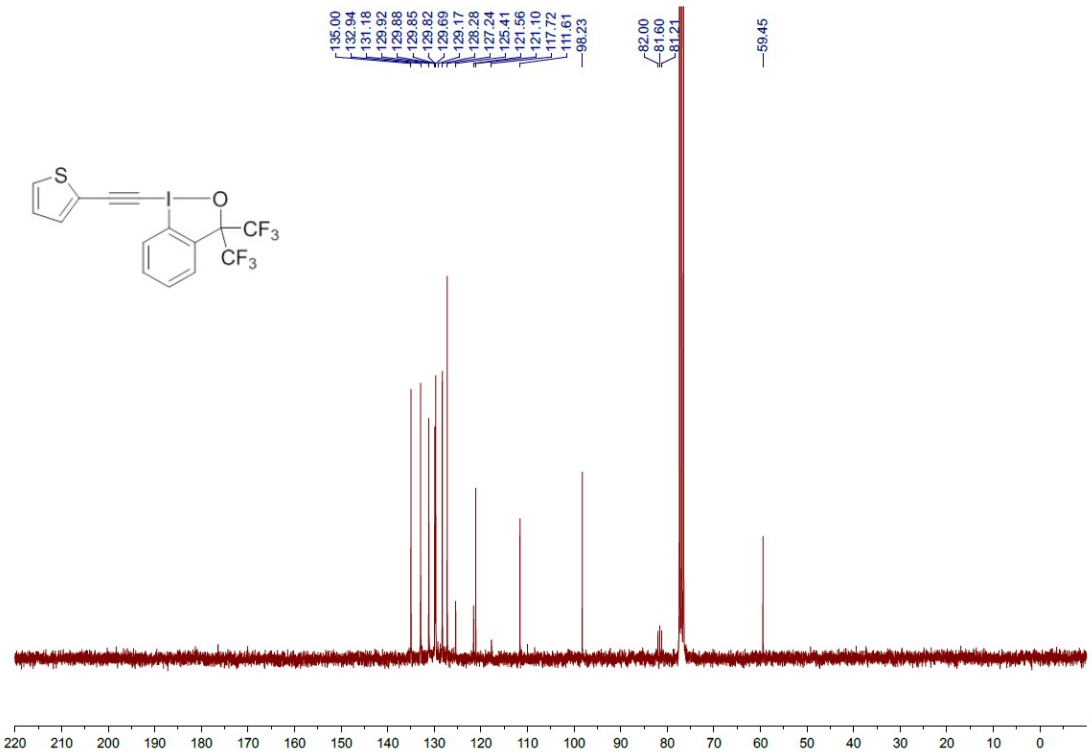
^{13}C NMR (75 MHz, $CDCl_3$) Spectrum of Compound **2j**



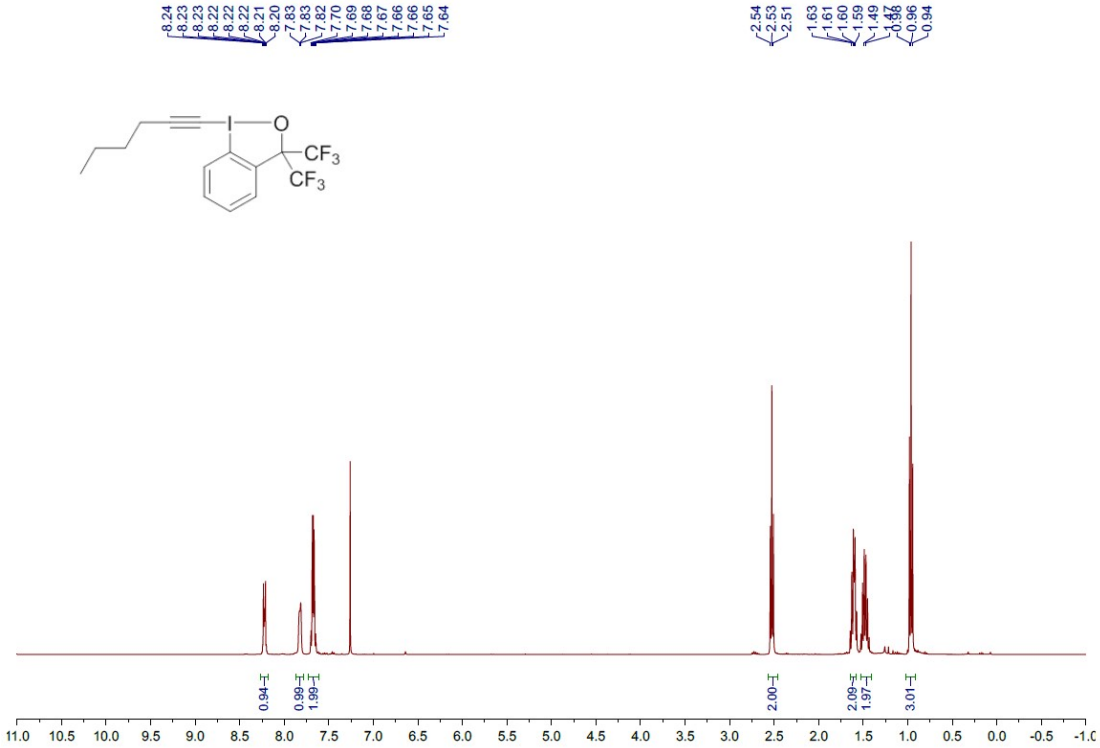
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of Compound **2l**



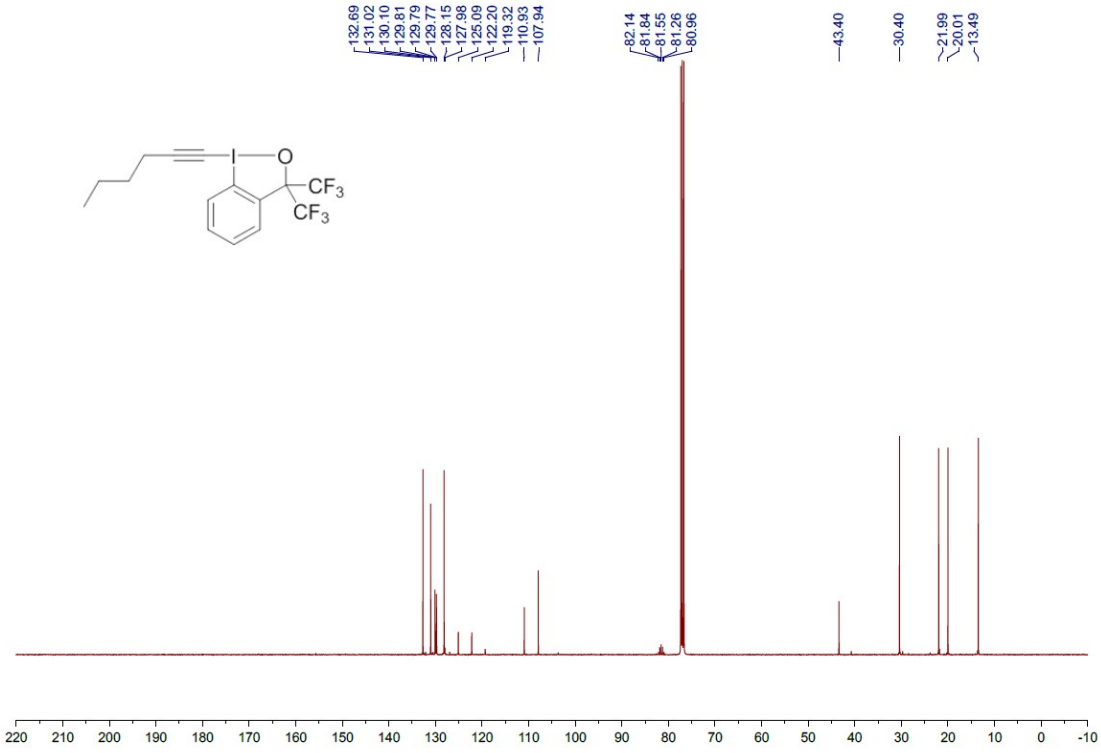
^{13}C NMR (75 MHz, $CDCl_3$) Spectrum of Compound **2l**



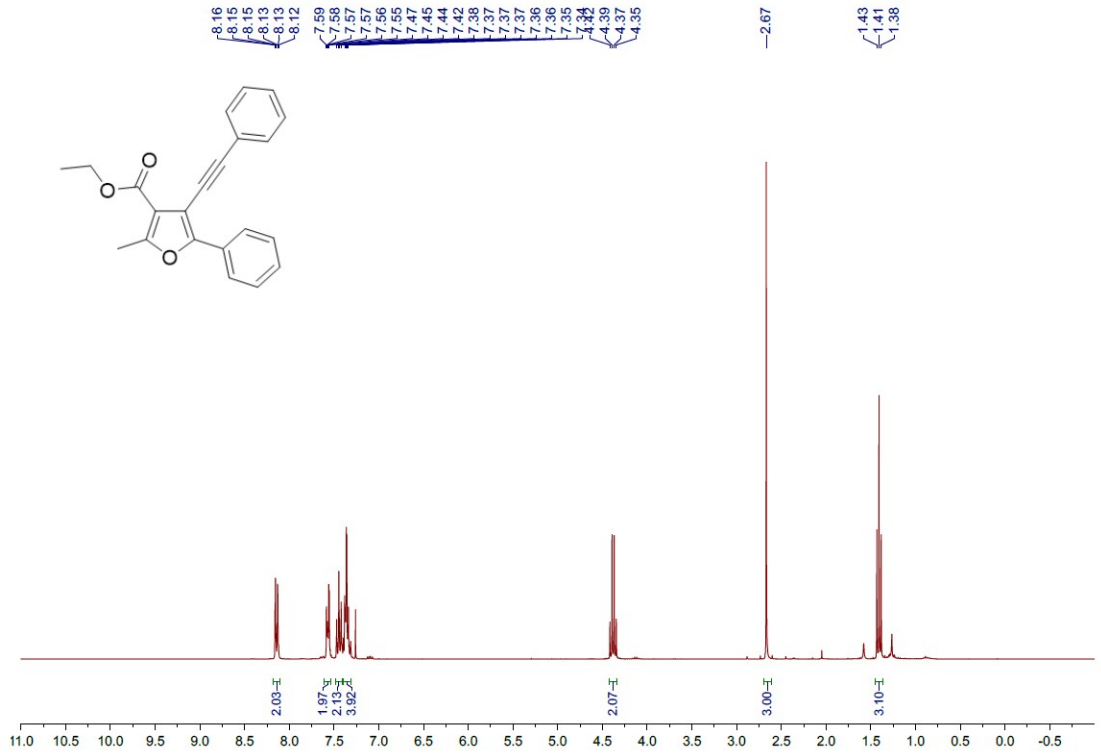
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **2n**



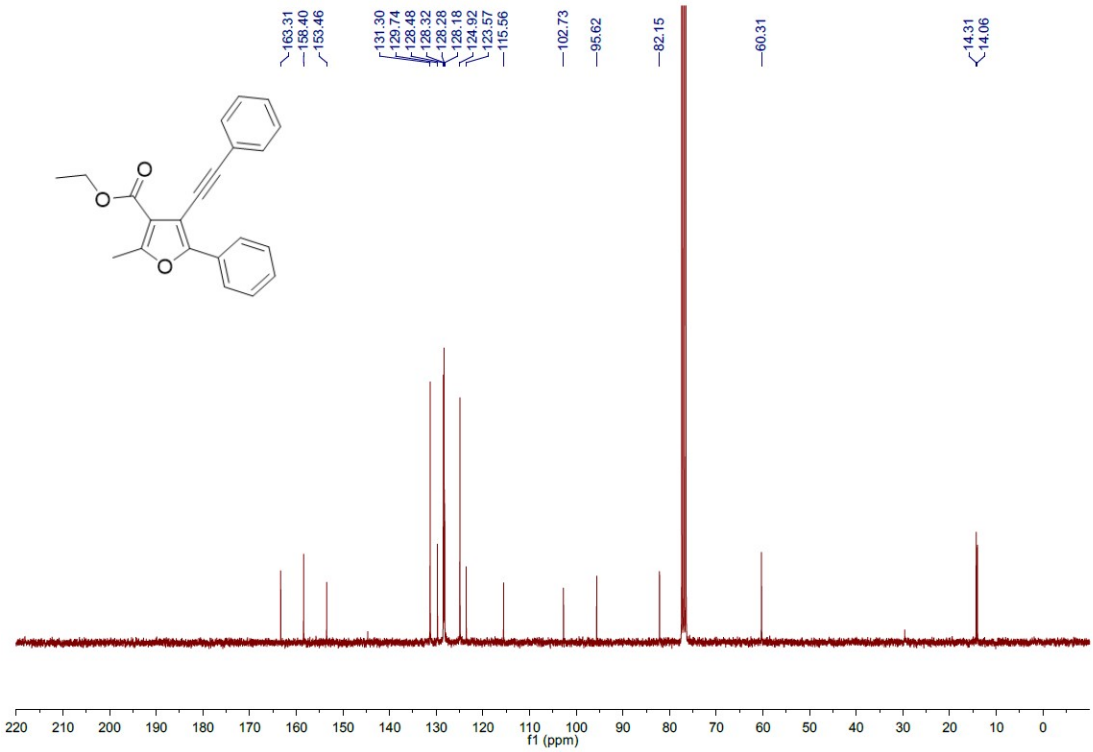
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **2n**



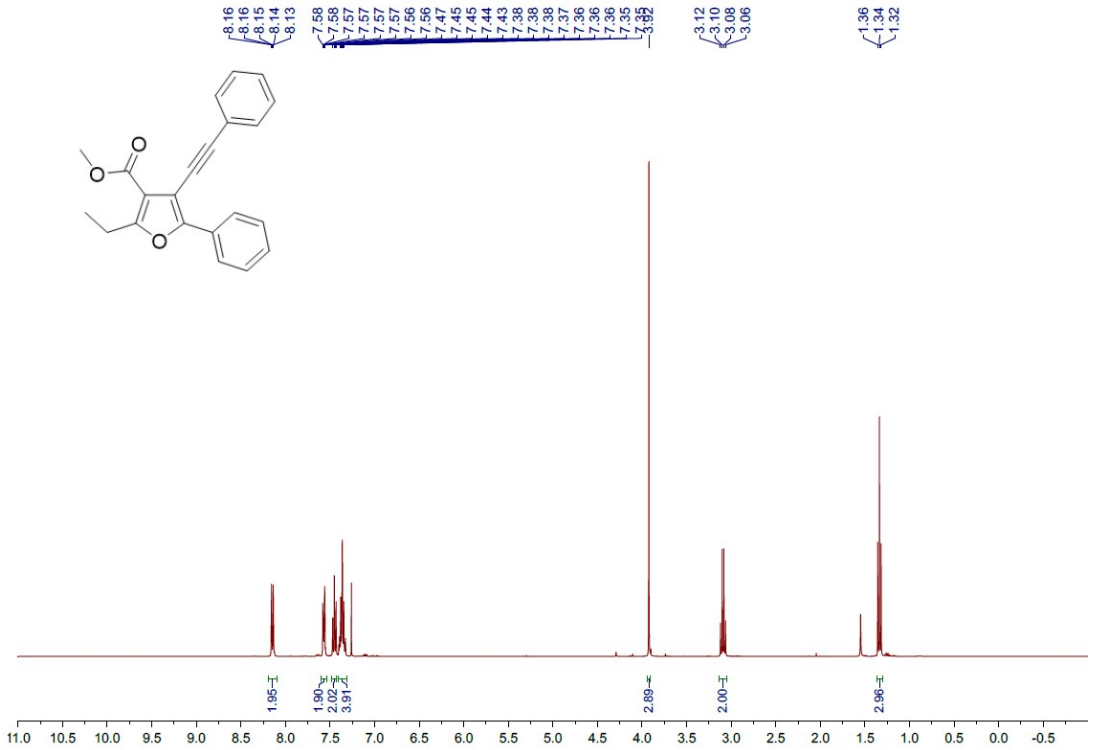
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of Compound **3aa**



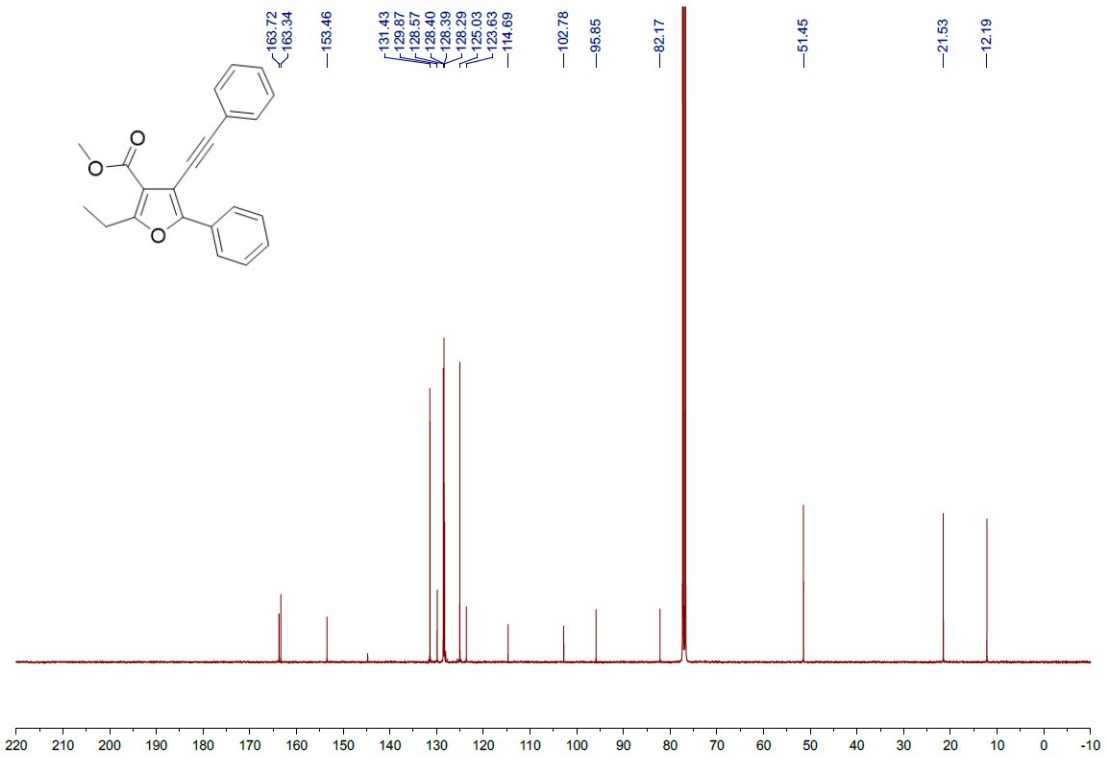
^{13}C NMR (75 MHz, $CDCl_3$) Spectrum of Compound **3aa**



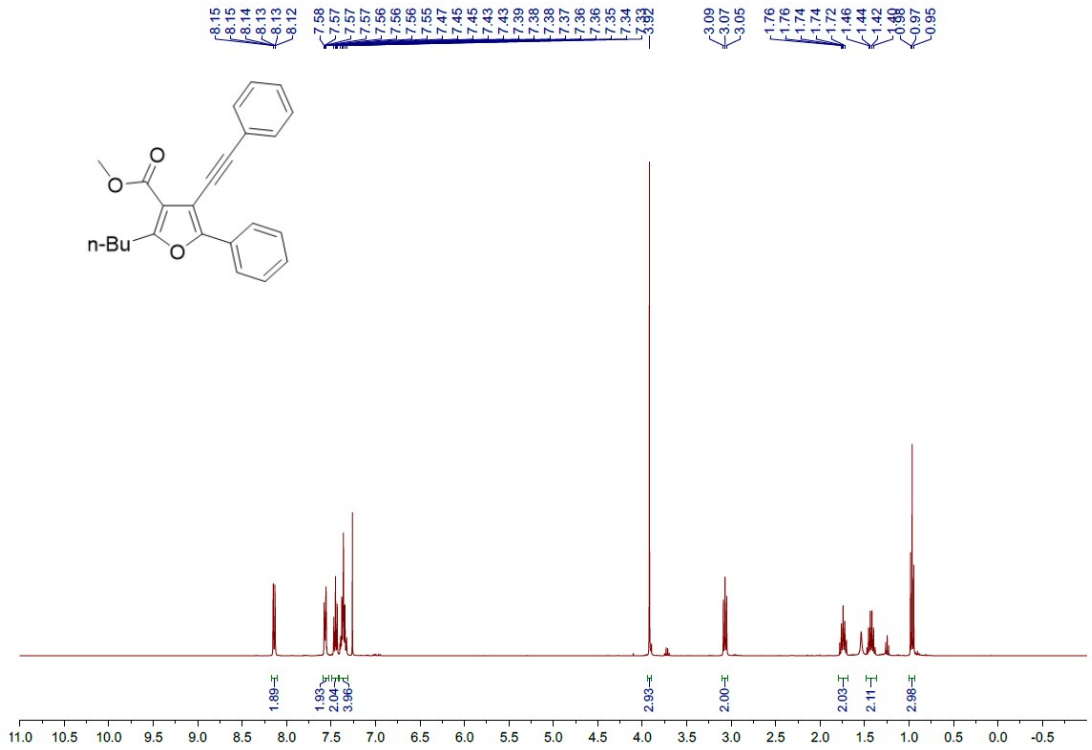
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3ba**



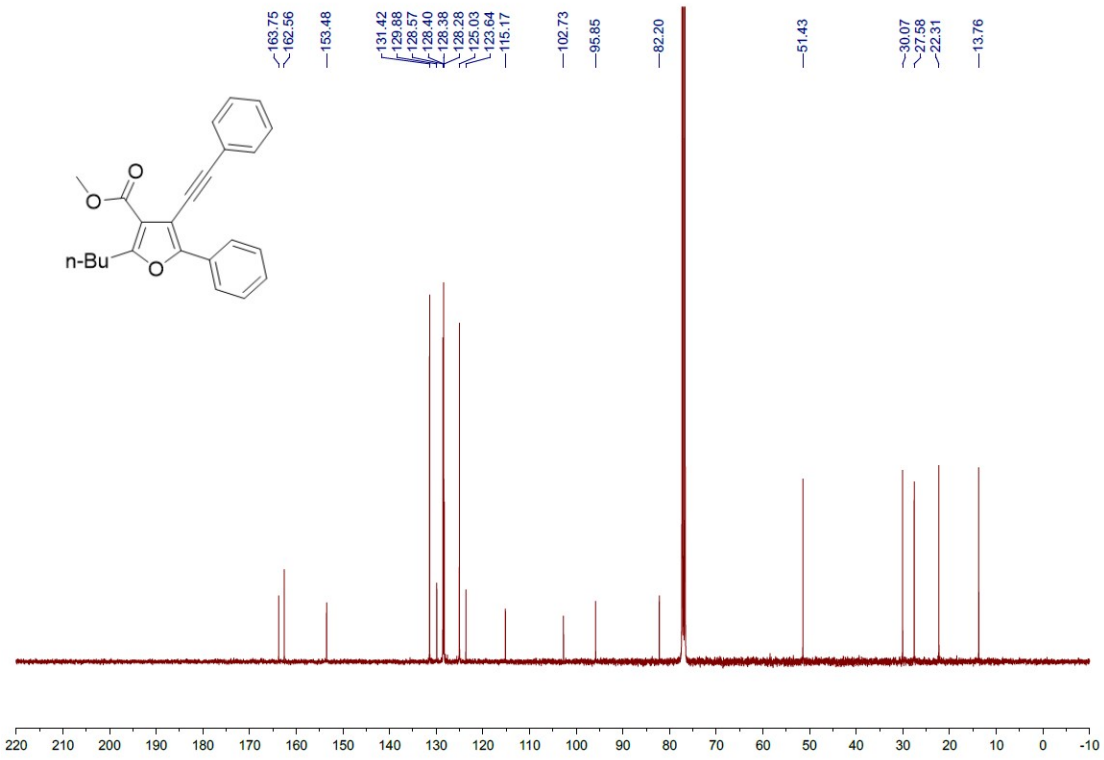
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3ba**



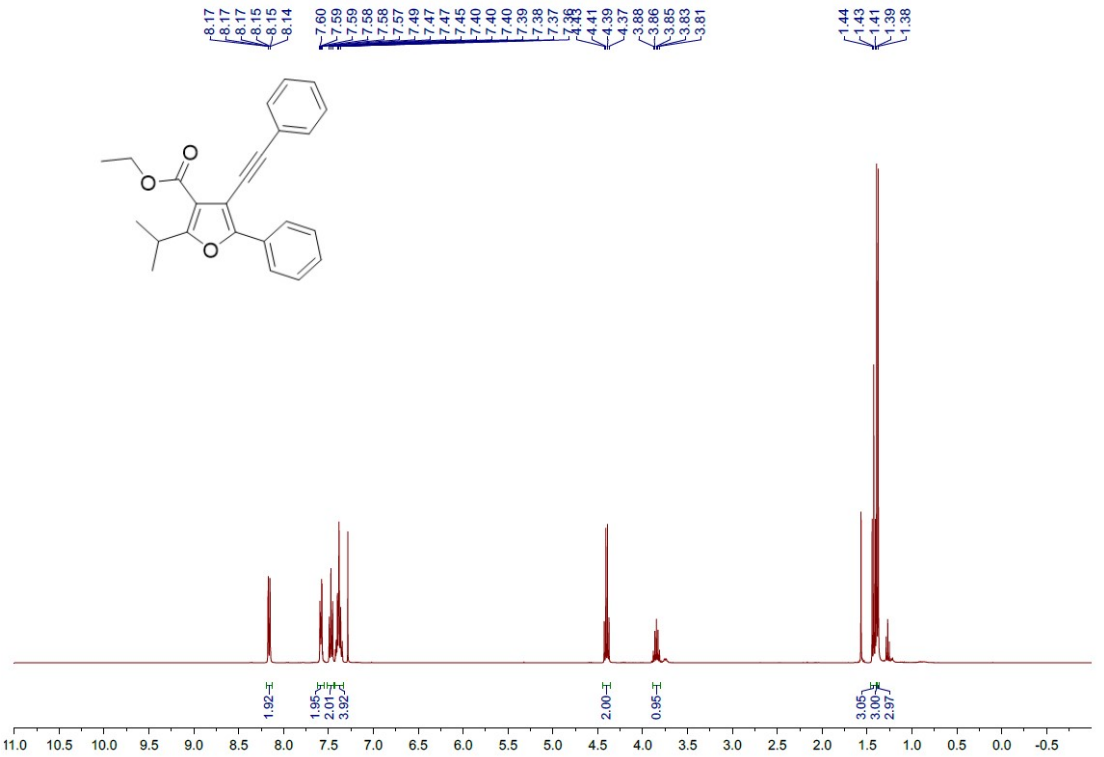
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3ca**



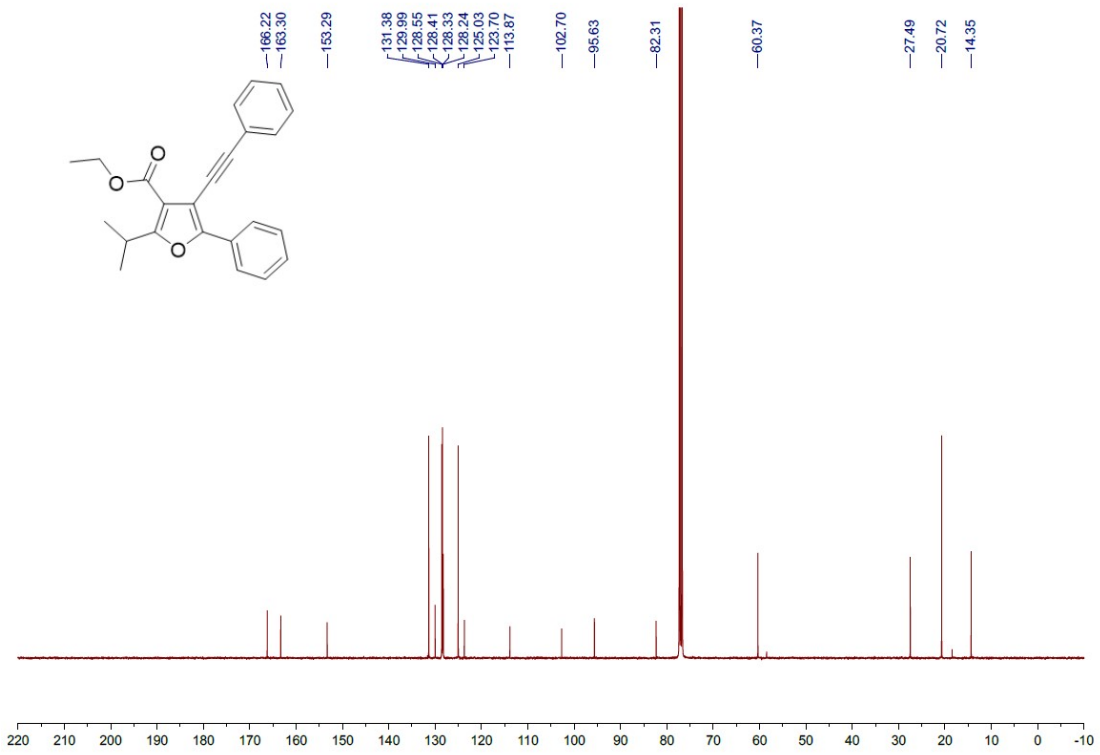
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3ca**



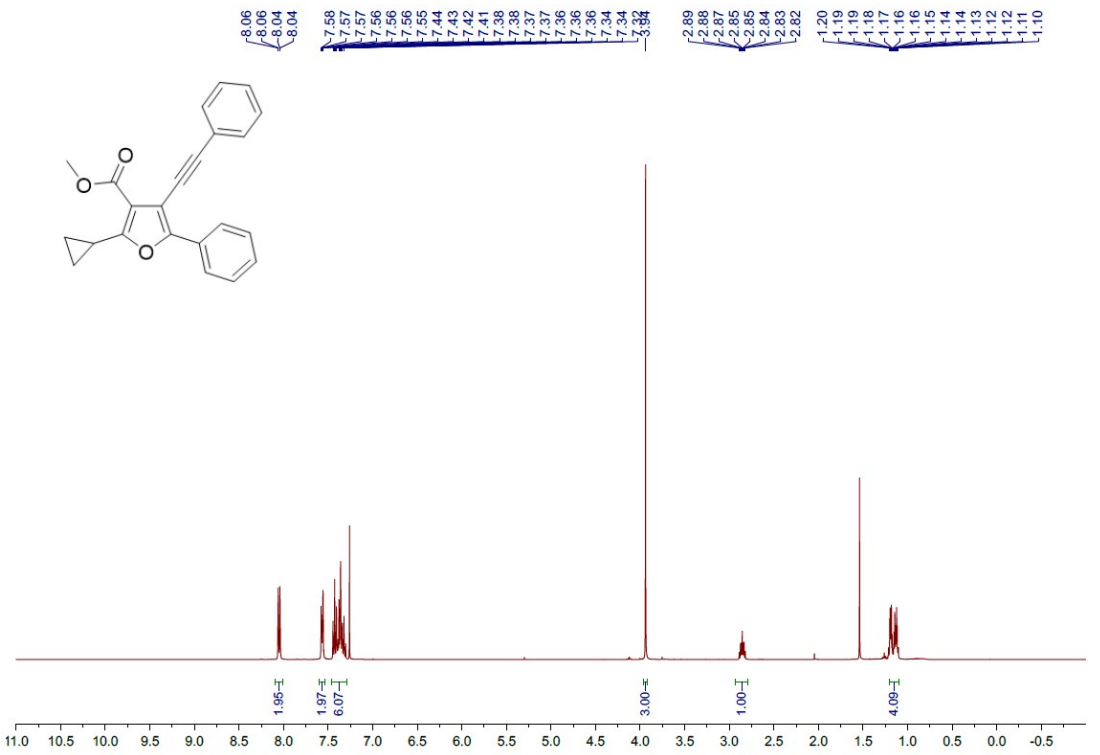
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3da**



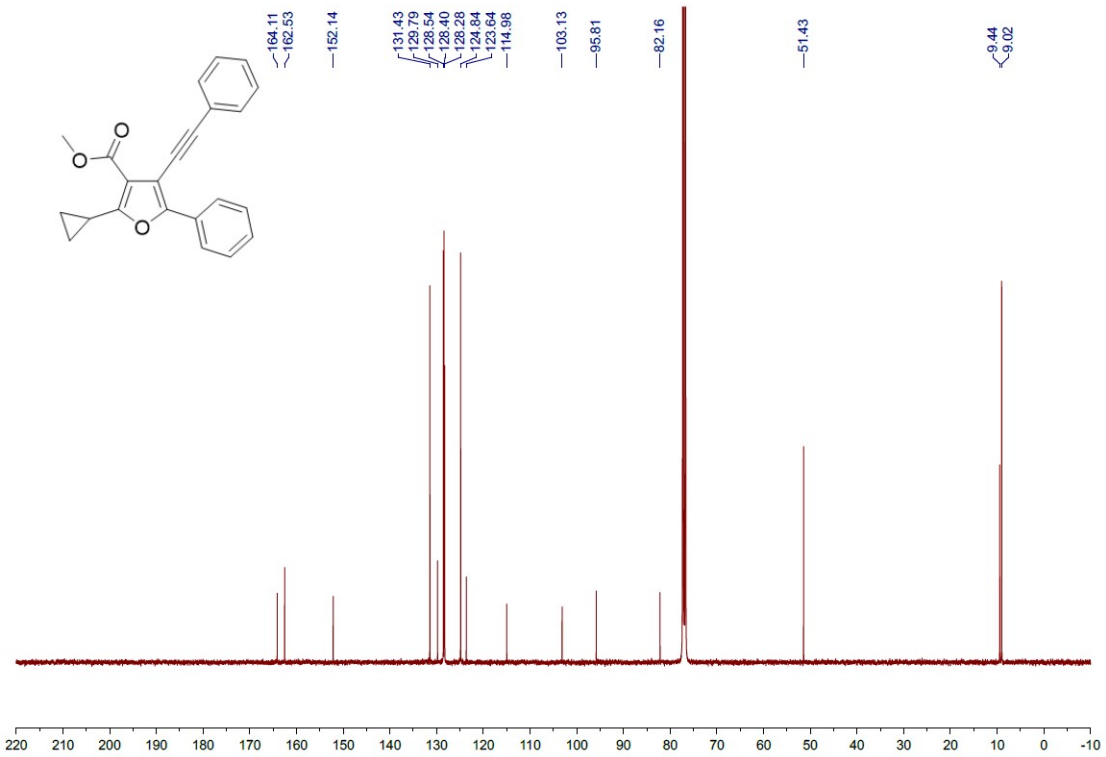
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3da**



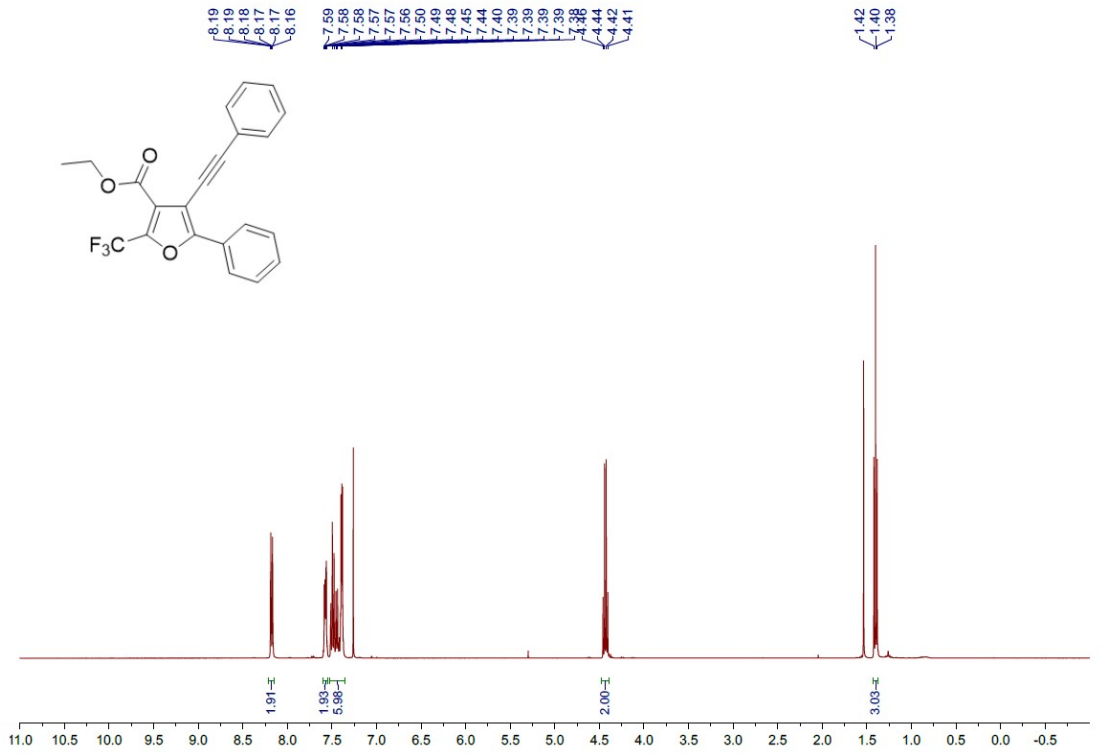
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3ea**



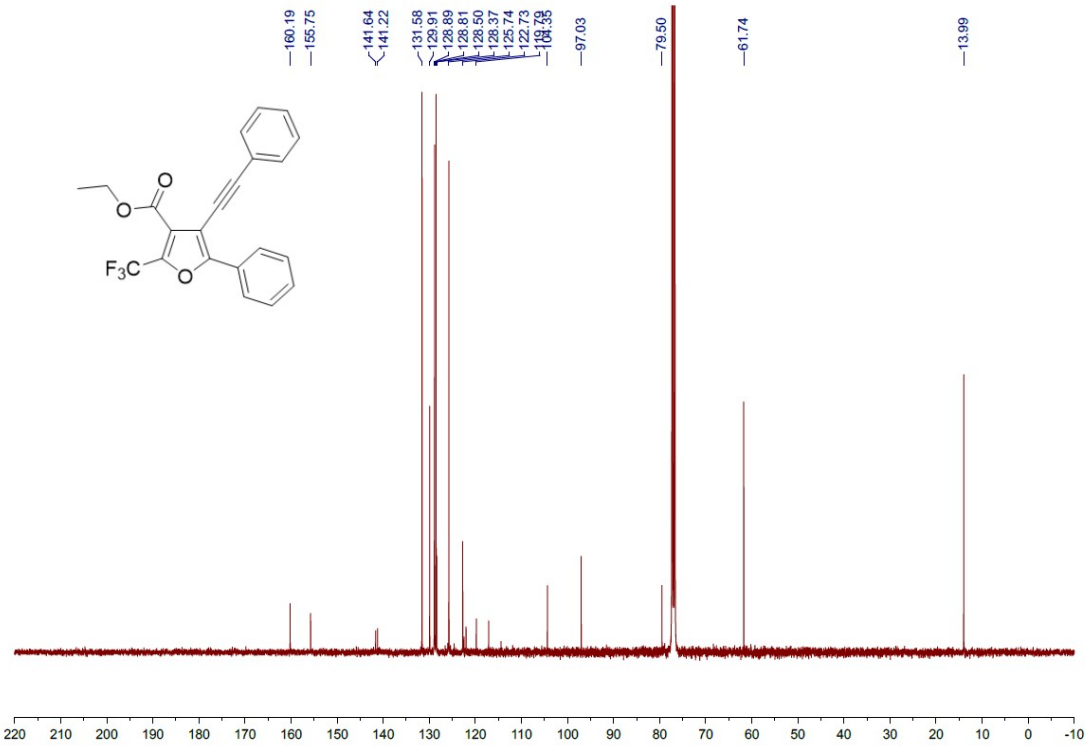
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3ea**



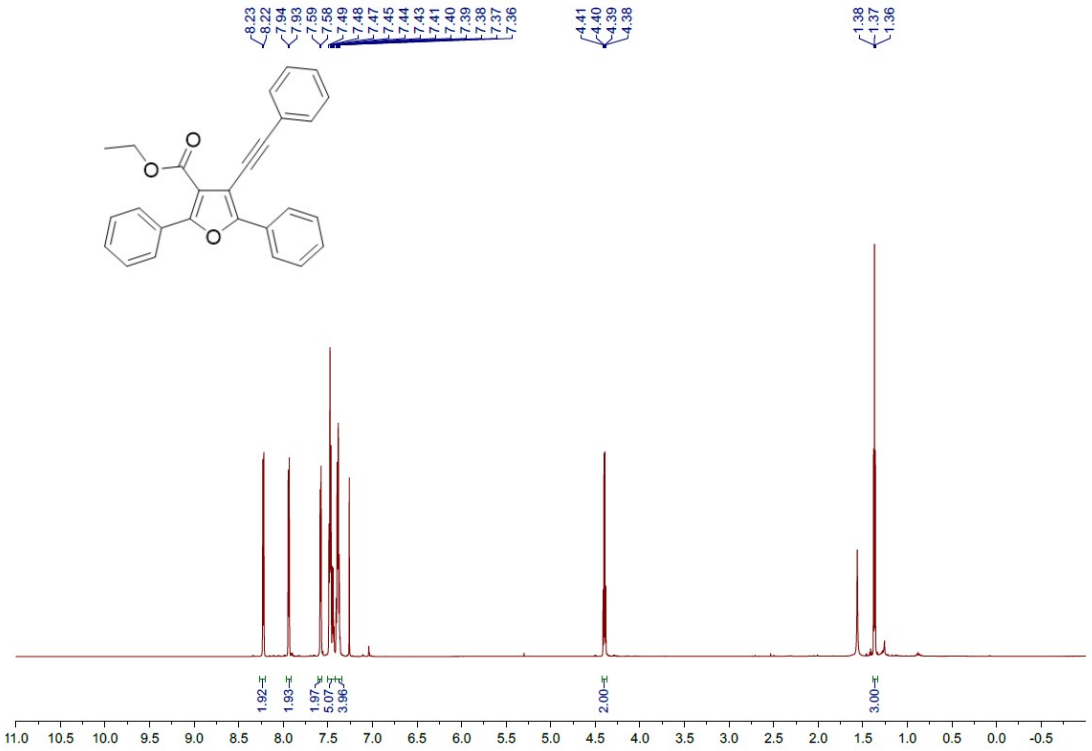
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3fa**



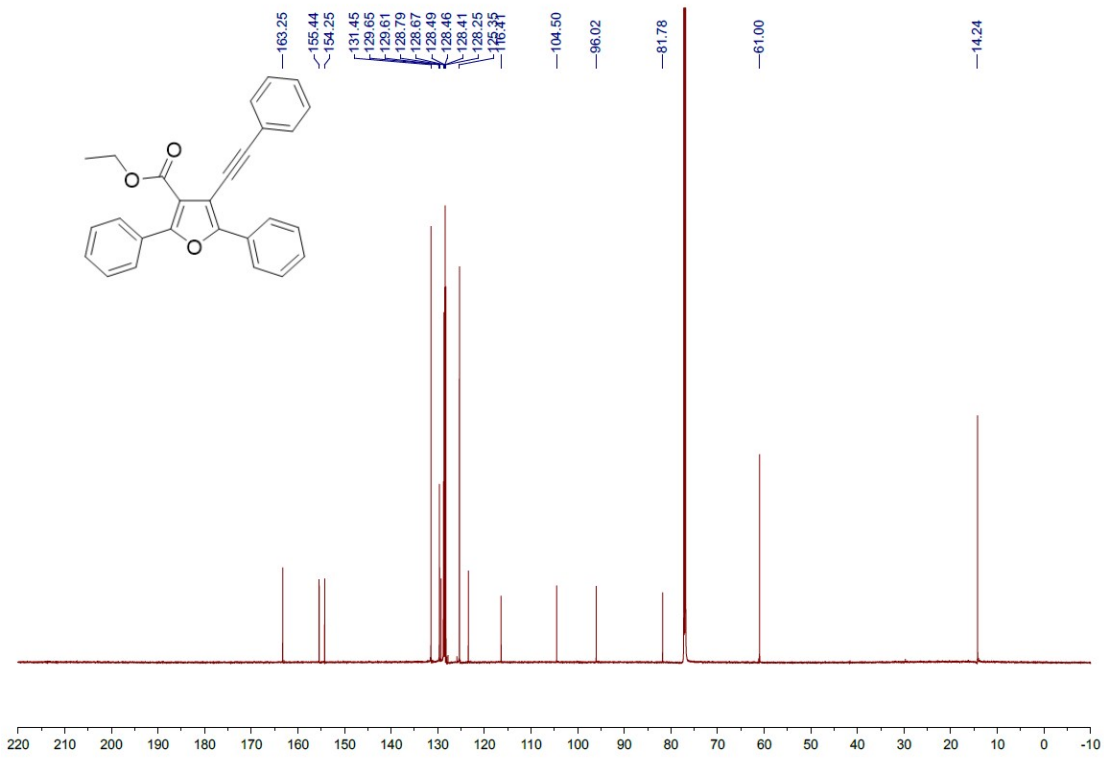
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3fa**



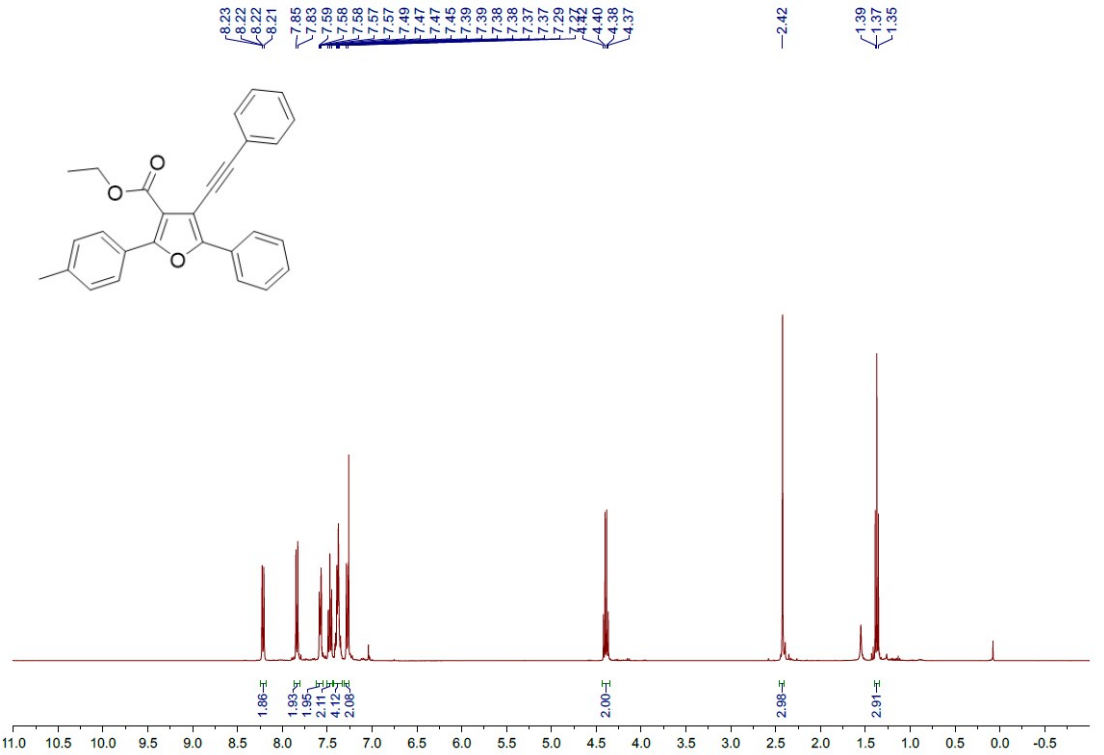
^{1}H NMR (700 MHz, $CDCl_3$) Spectrum of Compound **3ga**



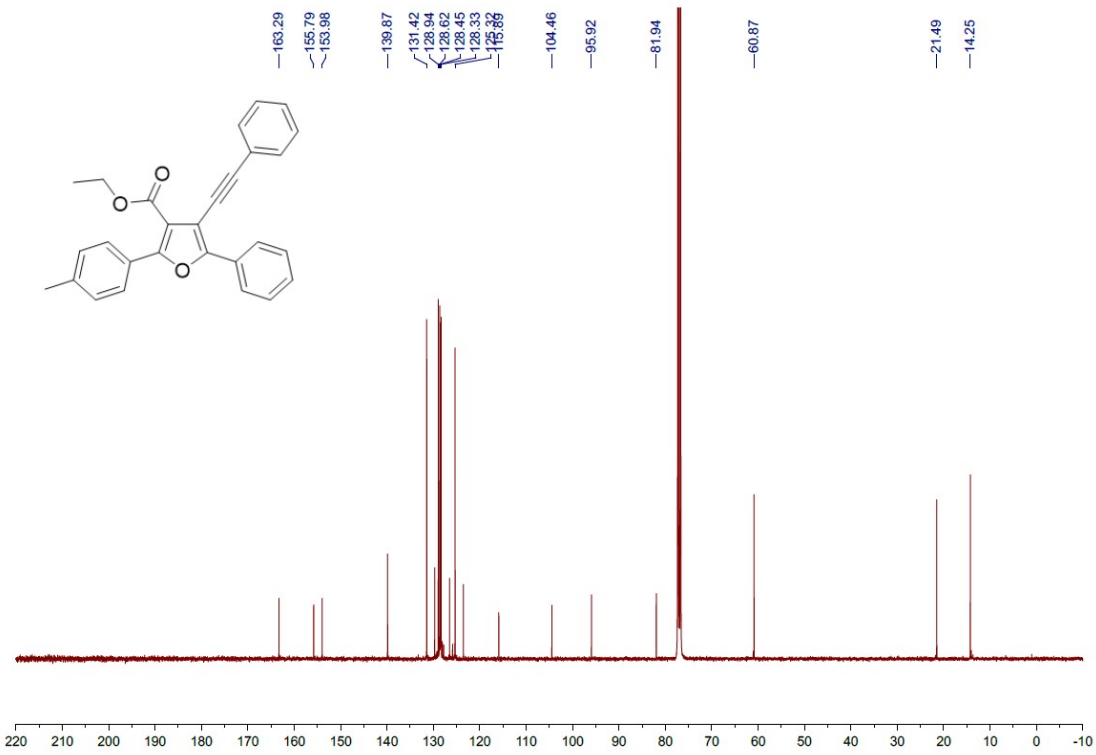
^{13}C NMR (175 MHz, $CDCl_3$) Spectrum of Compound **3ga**



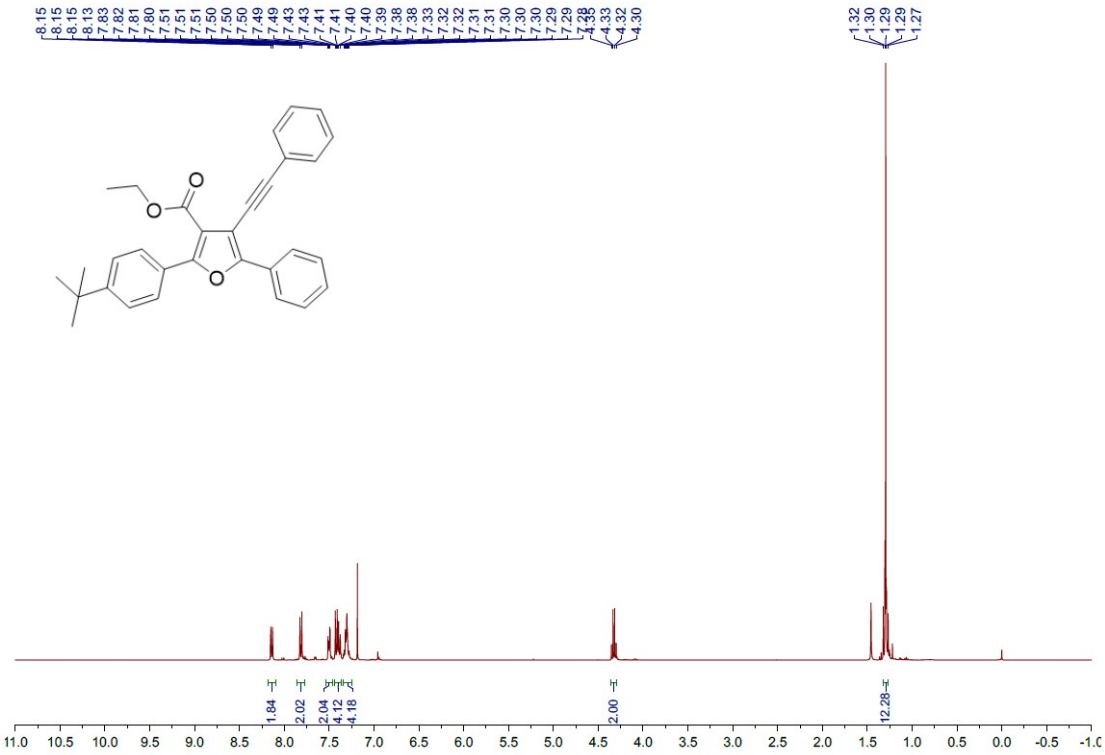
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3ha**



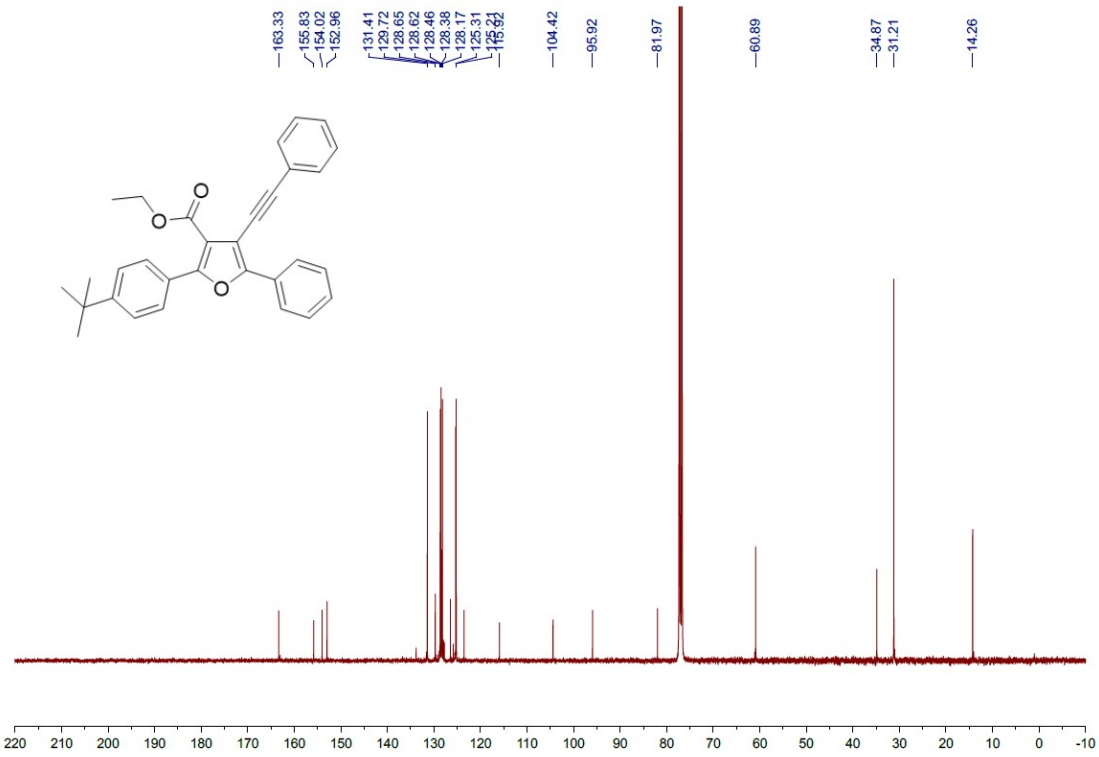
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3ha**



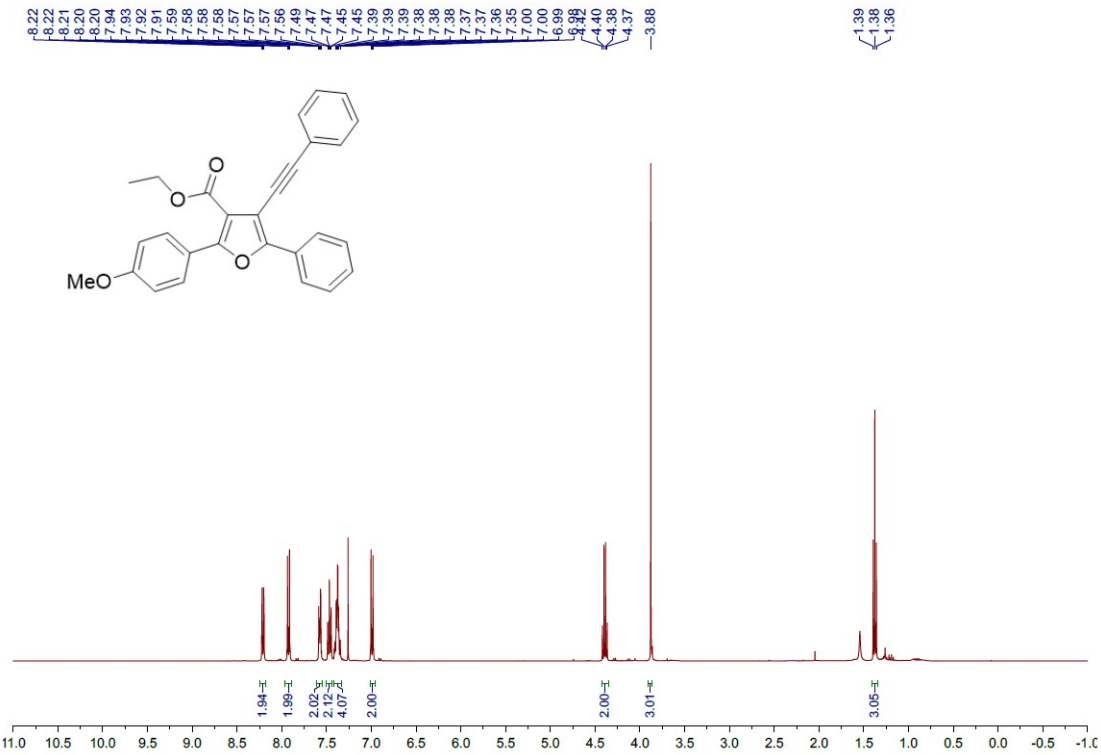
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3ia**



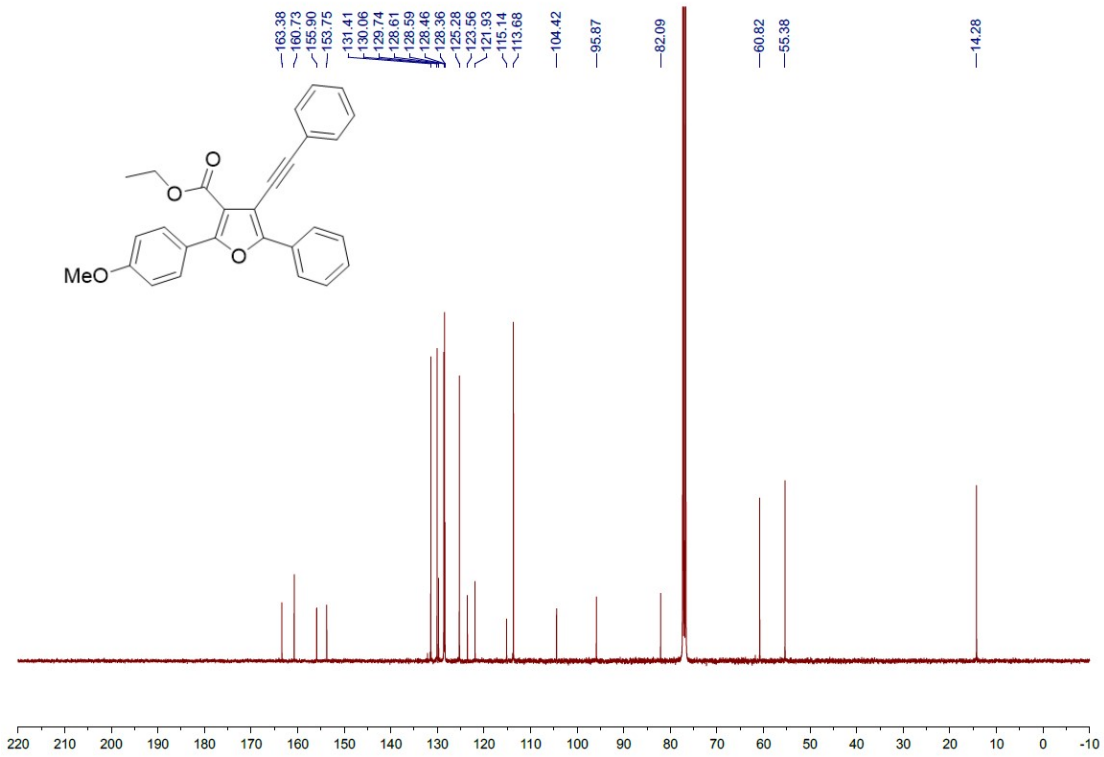
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3ia**



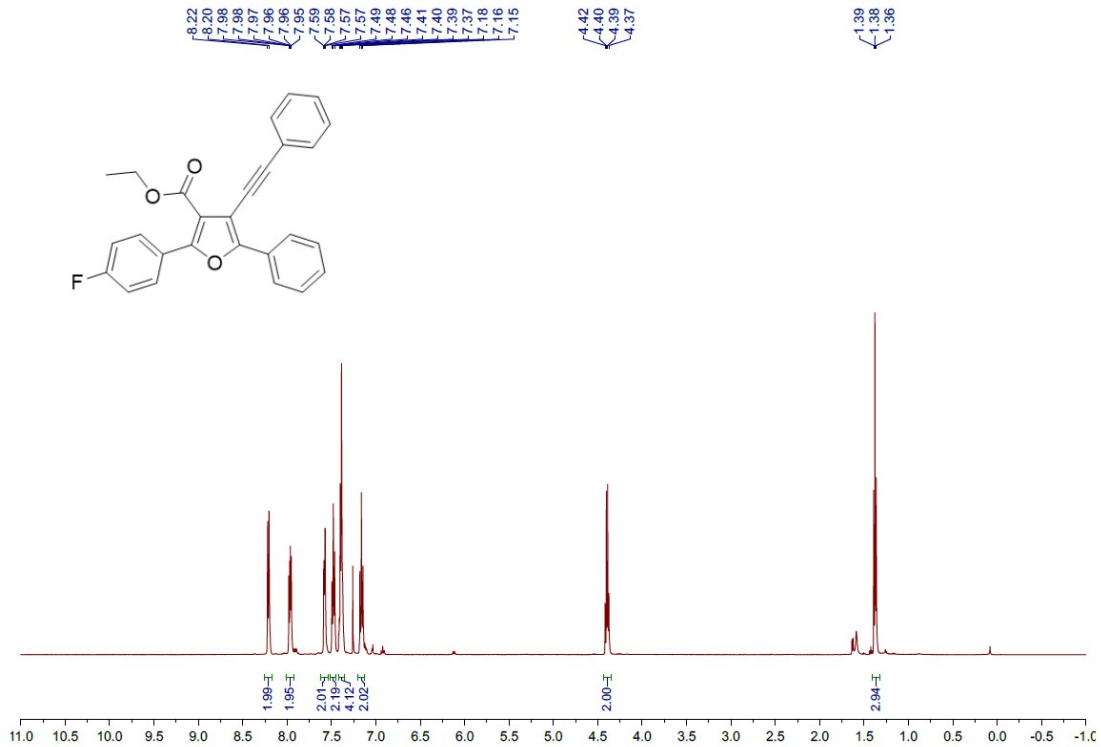
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3ja**



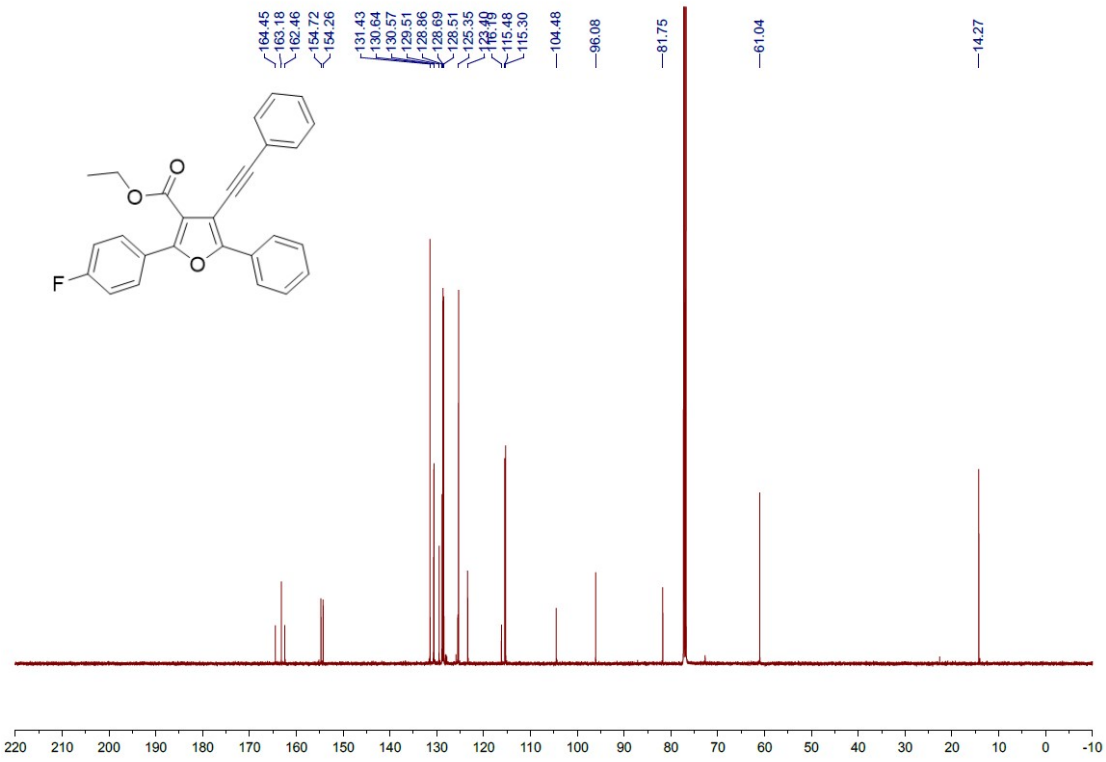
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3ja**



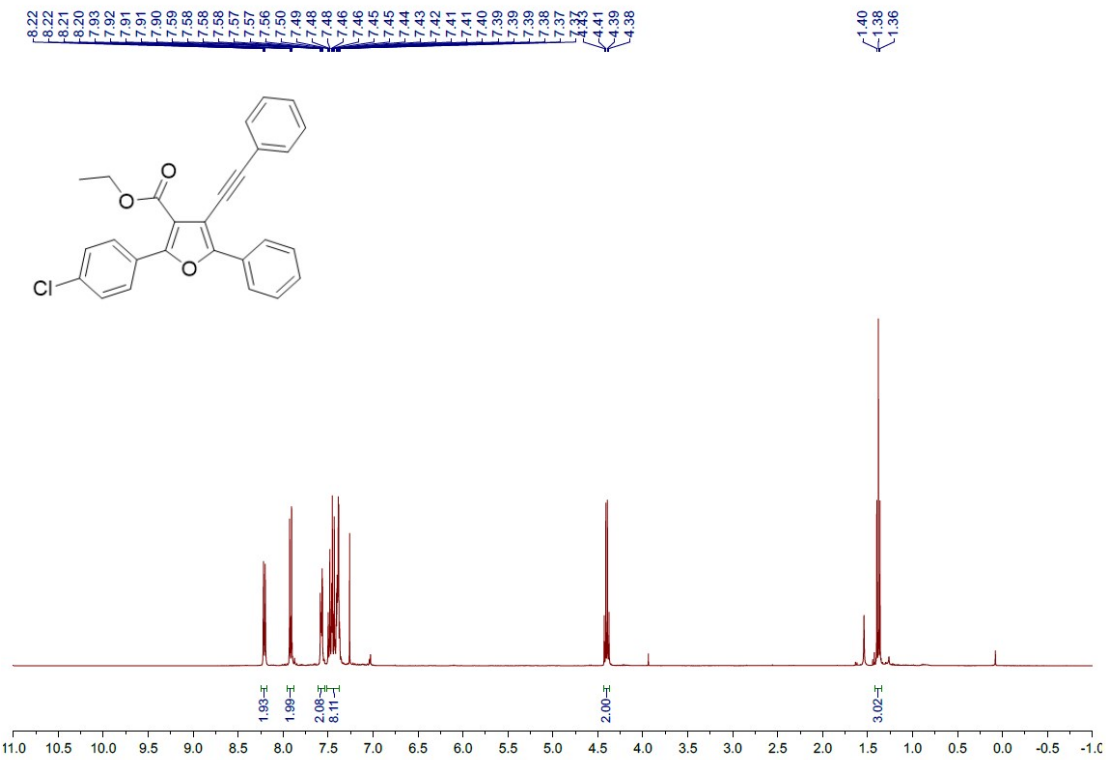
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of Compound **3ka**



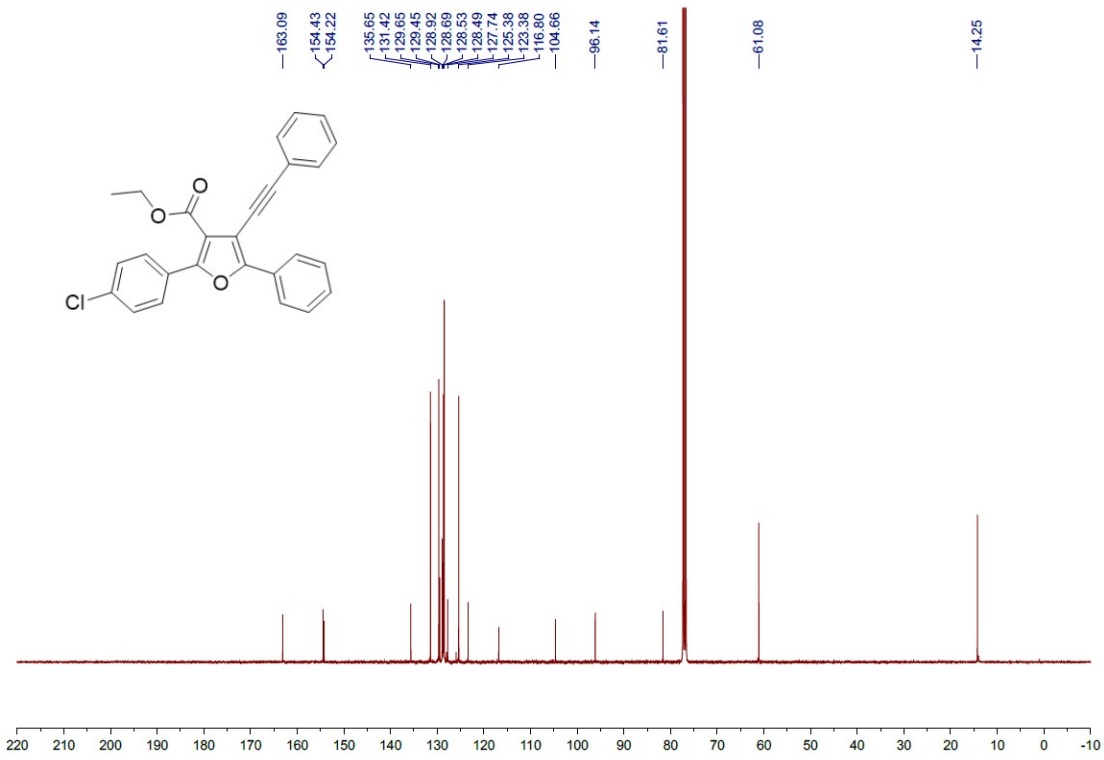
^{13}C NMR (125 MHz, $CDCl_3$) Spectrum of Compound **3ka**



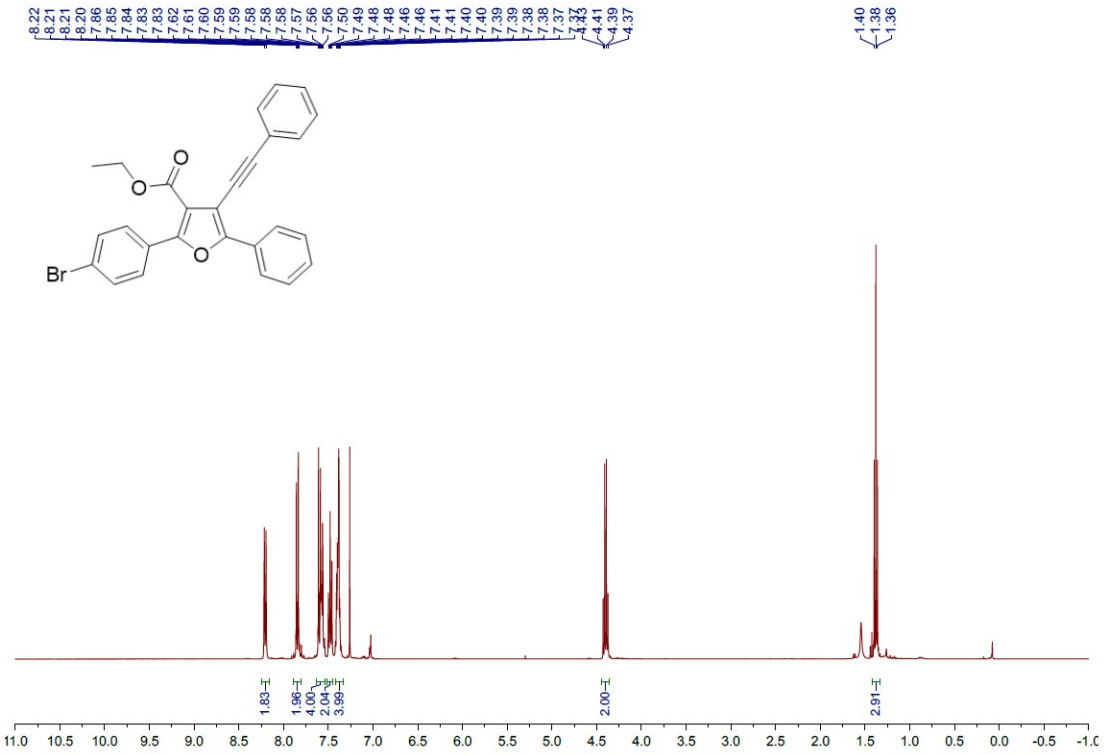
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3la**



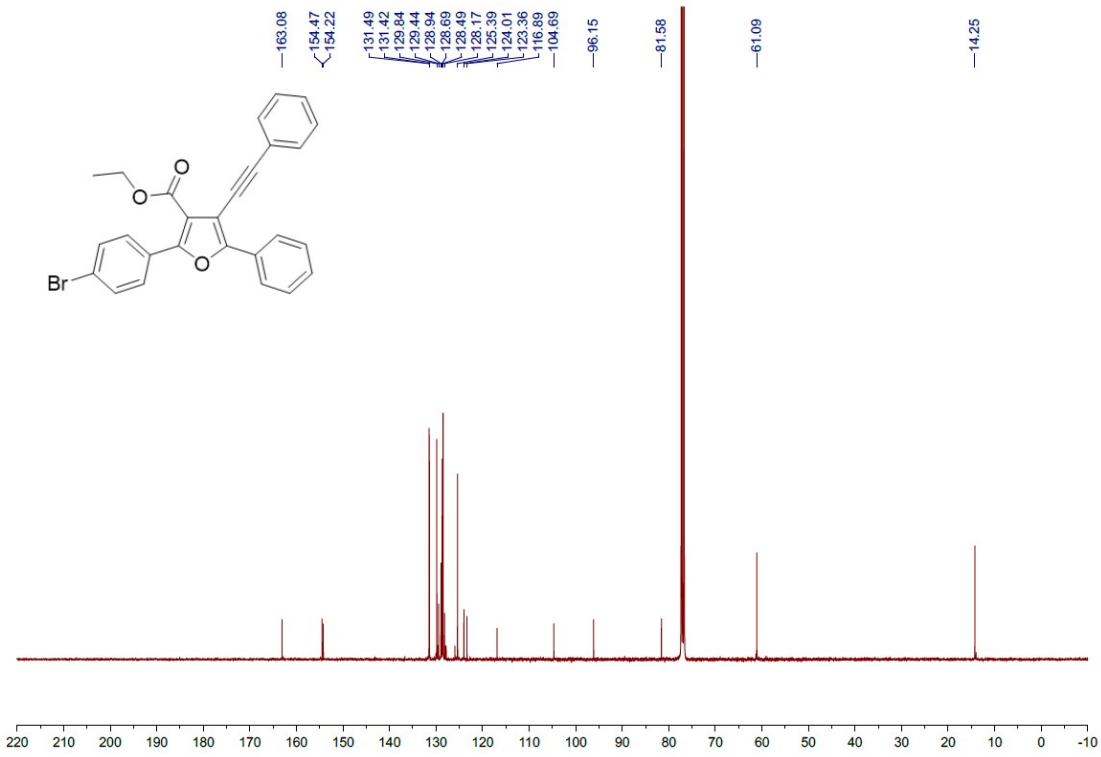
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3la**



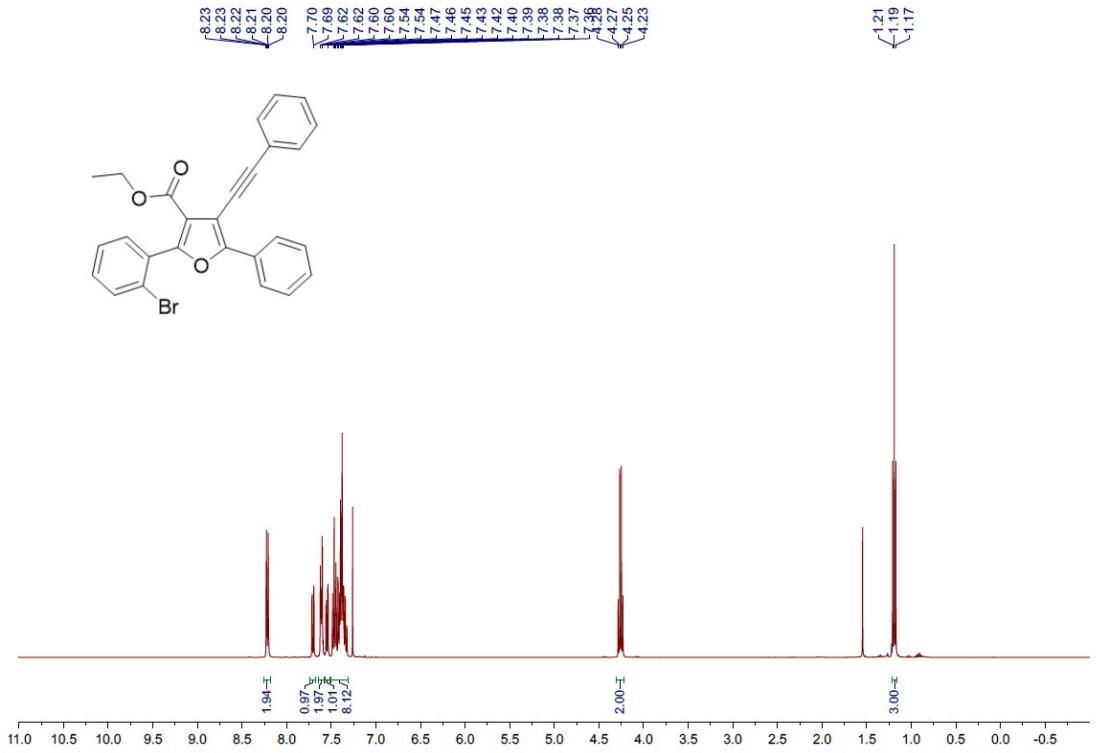
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3ma**



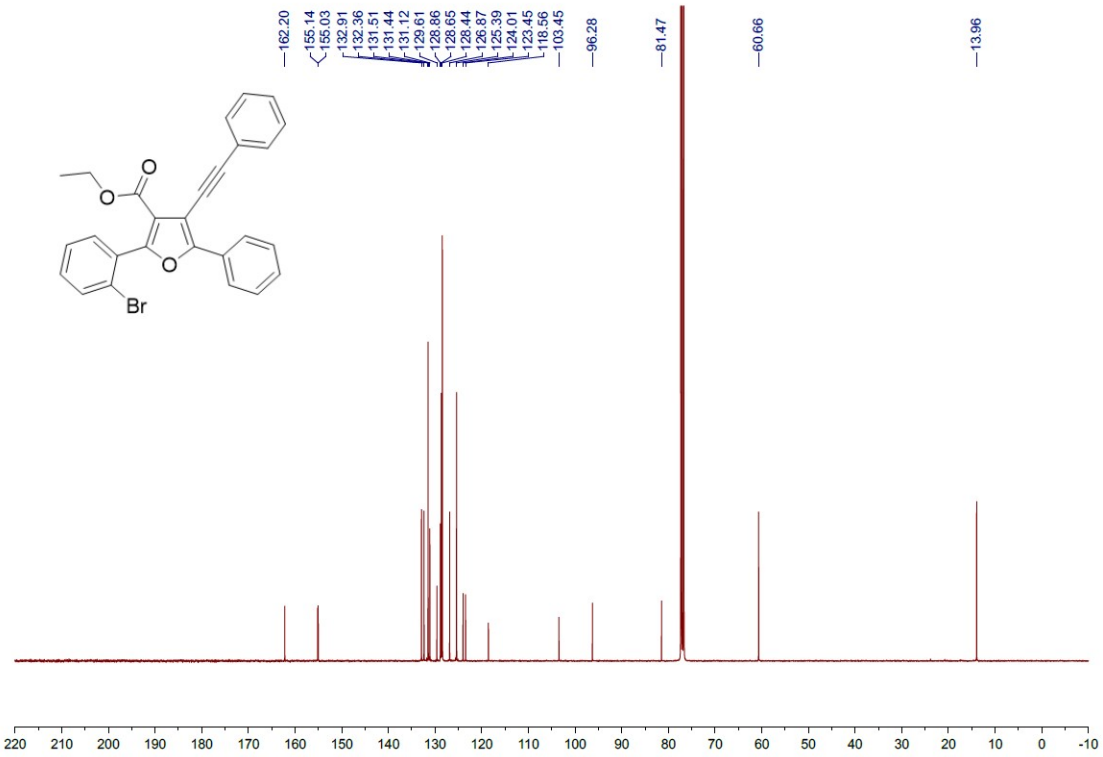
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3ma**



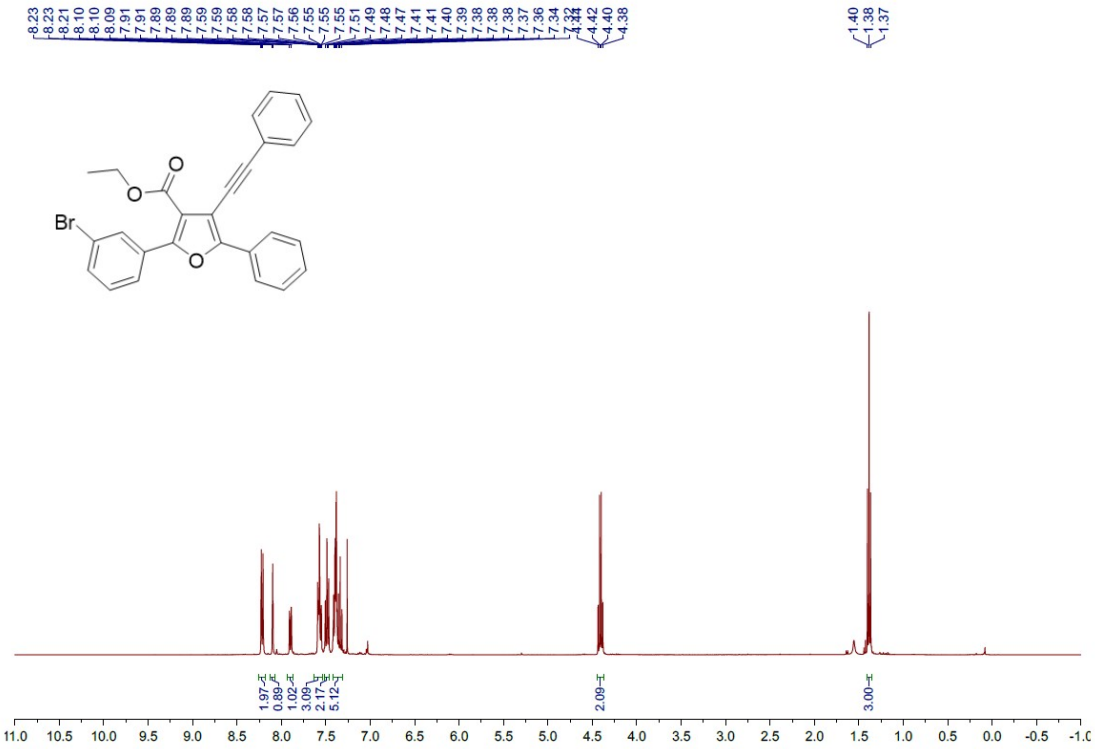
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3na**



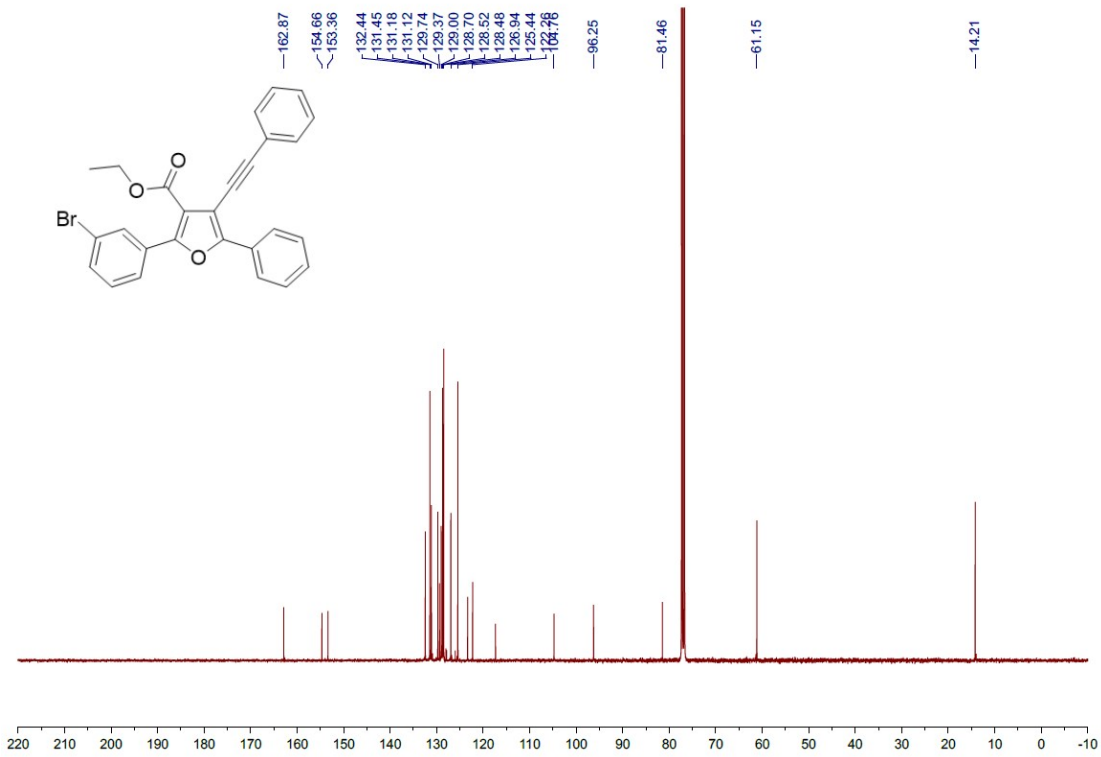
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3na**



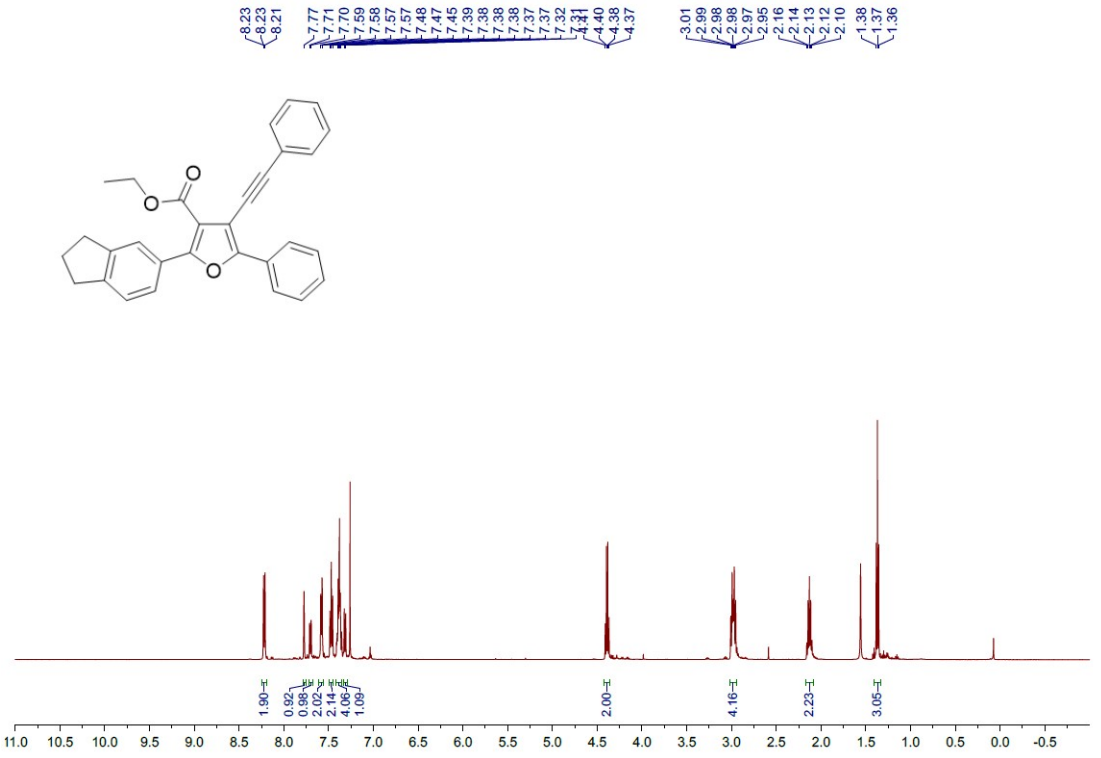
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3oa**



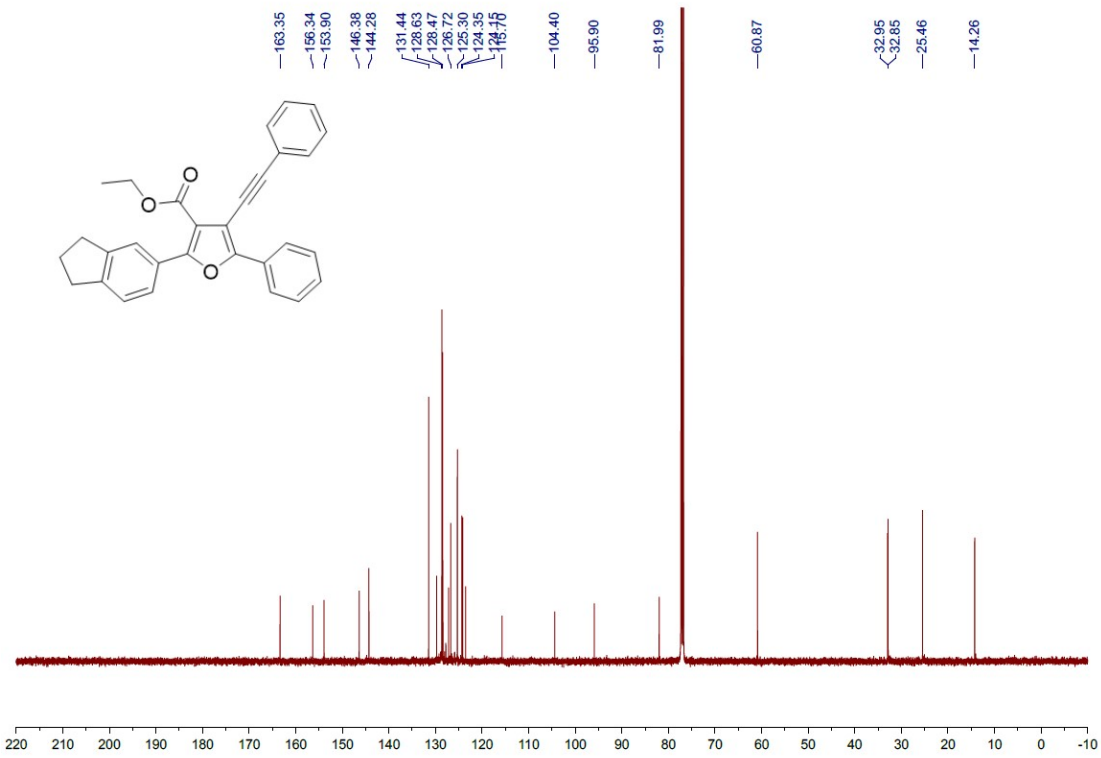
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3oa**



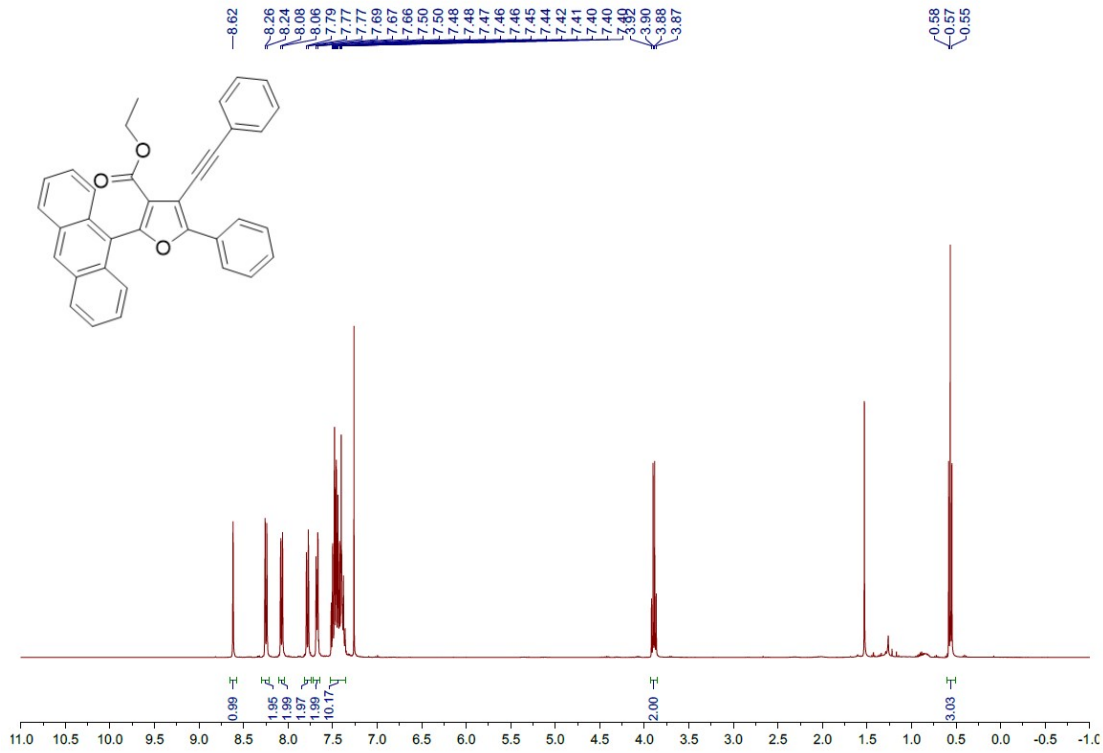
^{1}H NMR (500 MHz, $CDCl_3$) Spectrum of Compound **3pa**



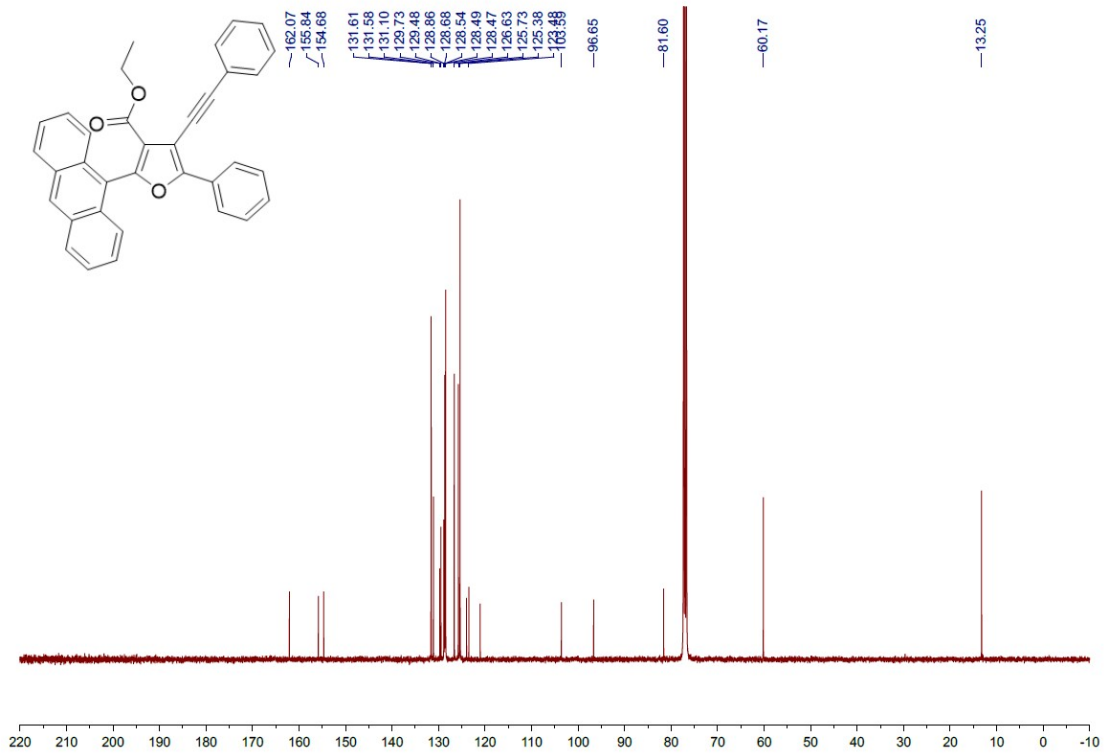
^{13}C NMR (125 MHz, $CDCl_3$) Spectrum of Compound **3pa**



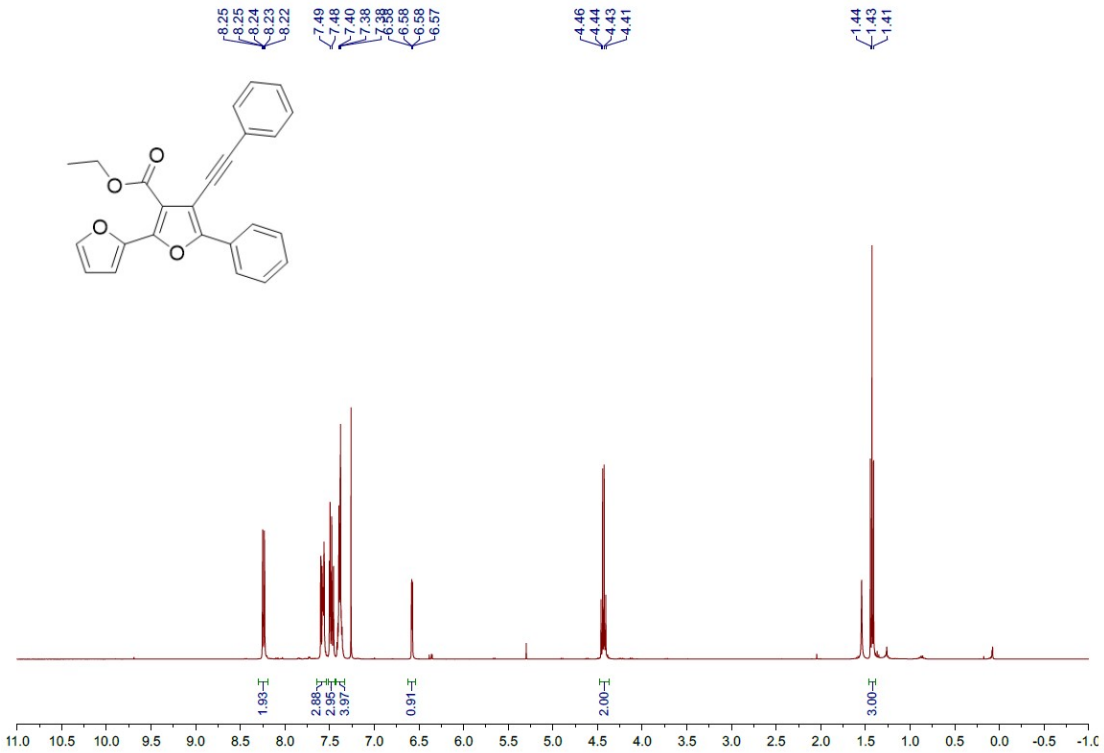
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3qa**



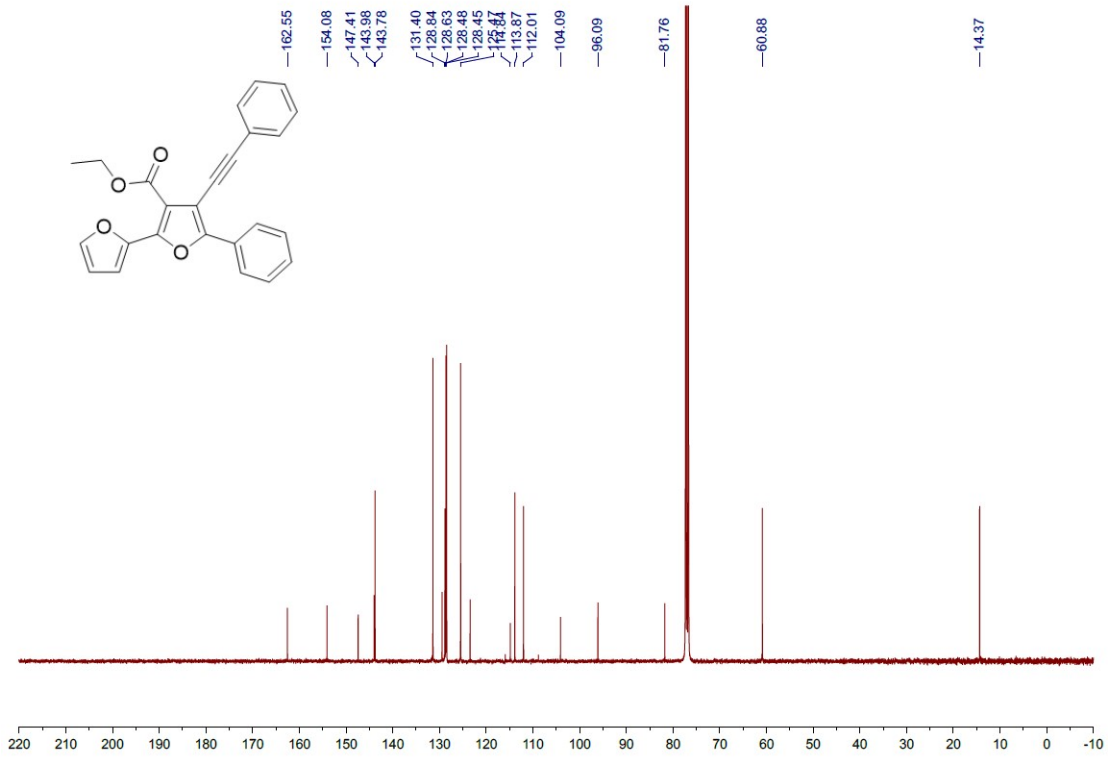
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3qa**



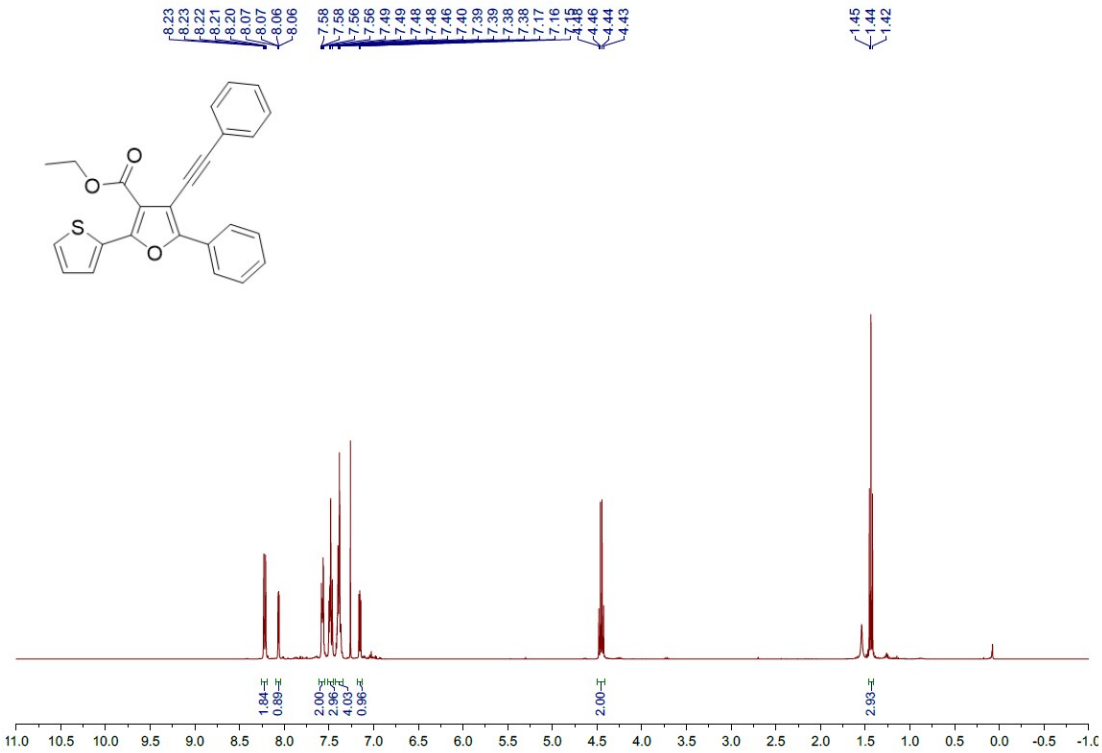
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3ra**



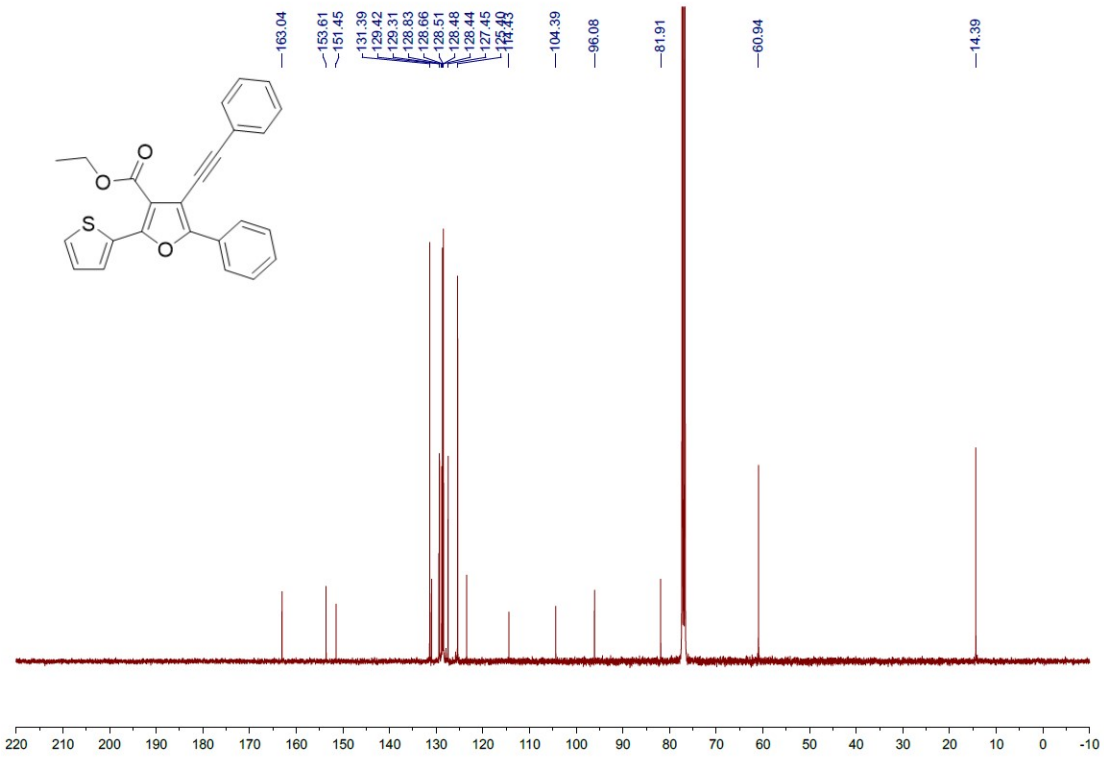
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3ra**



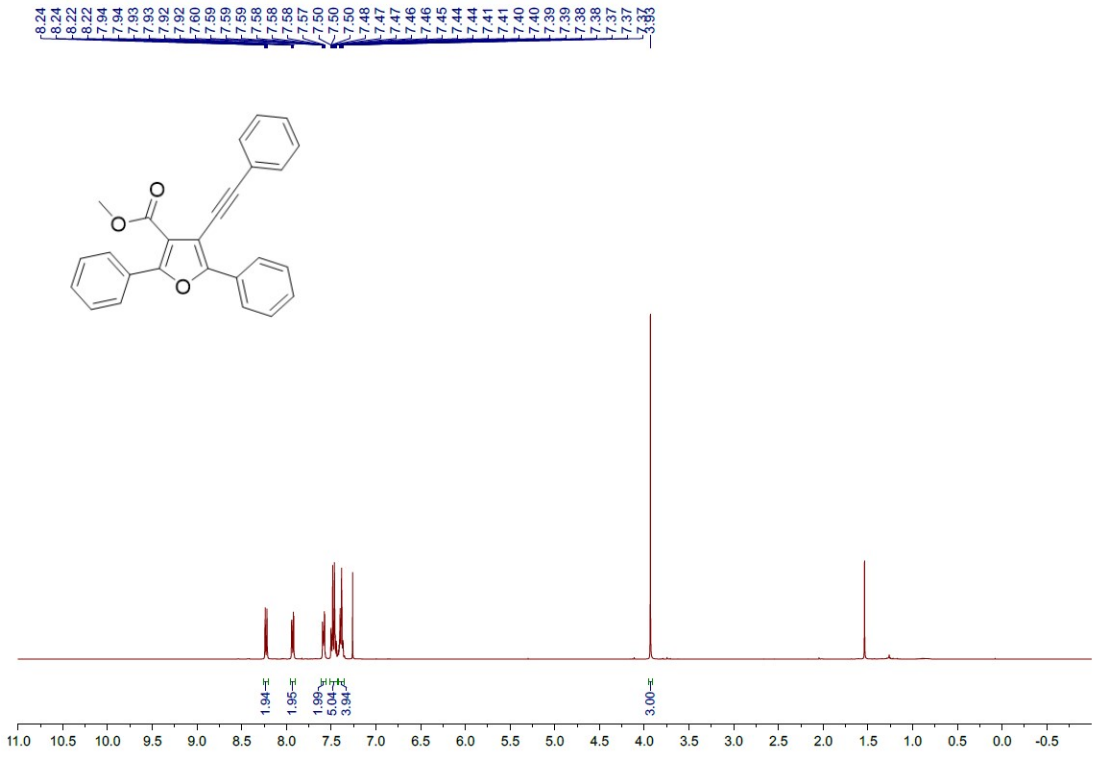
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3sa**



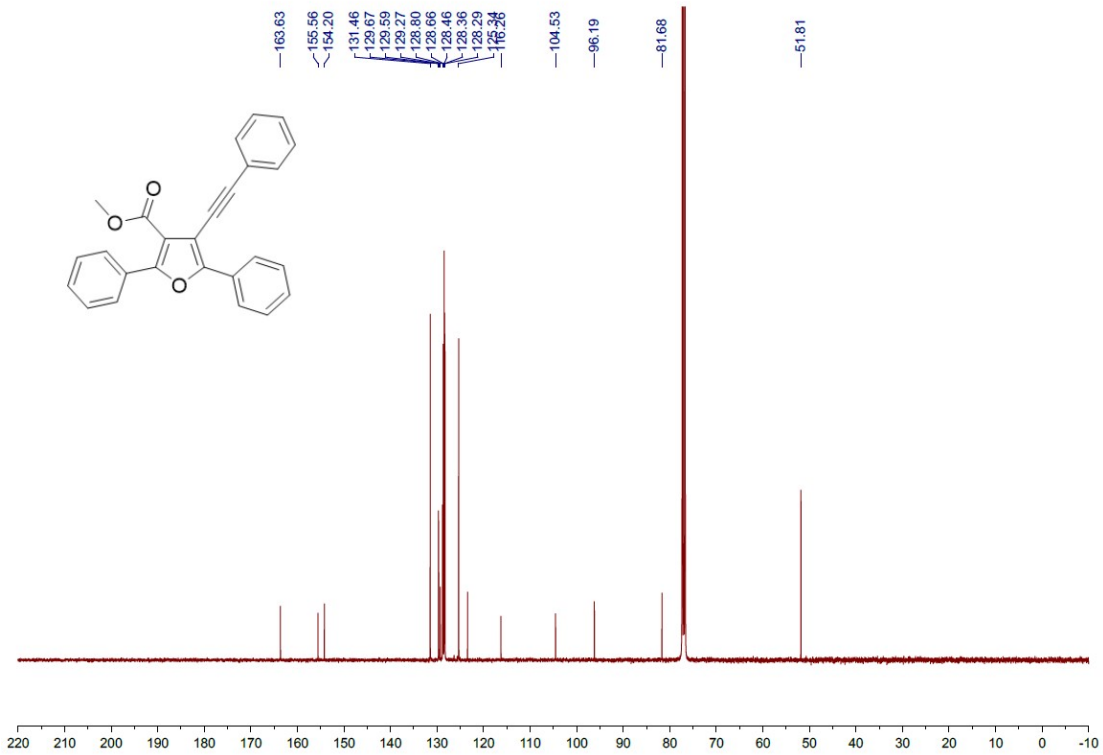
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3sa**



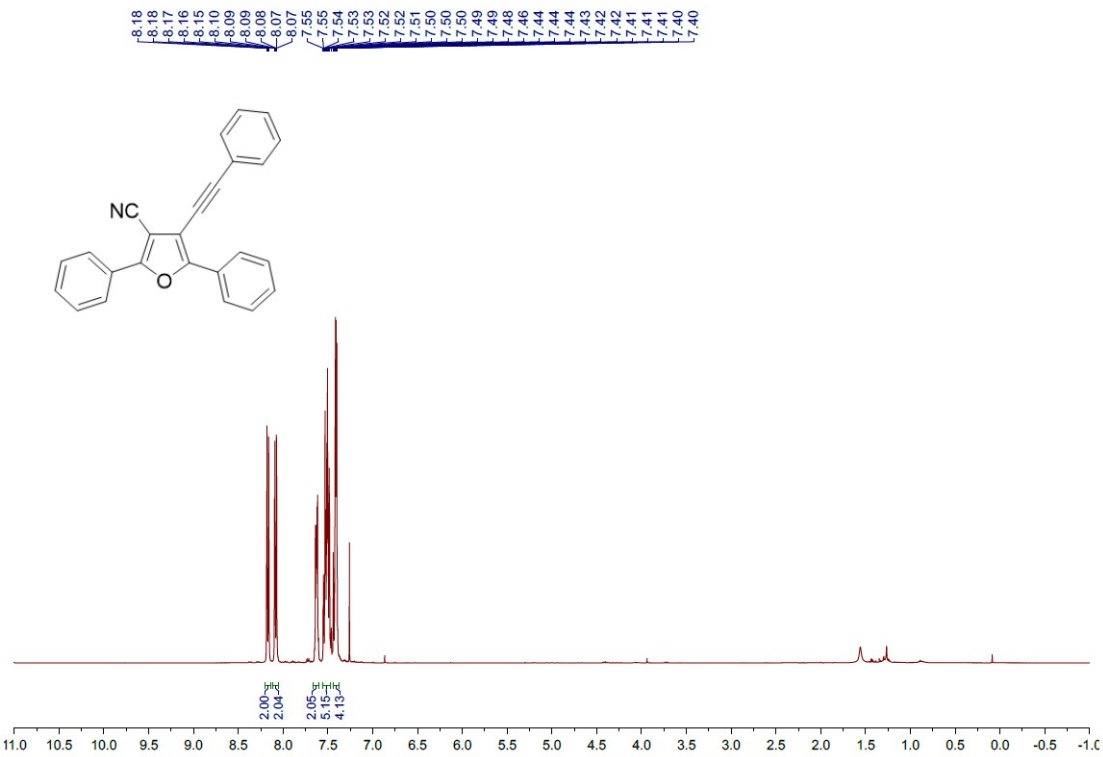
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3ta**



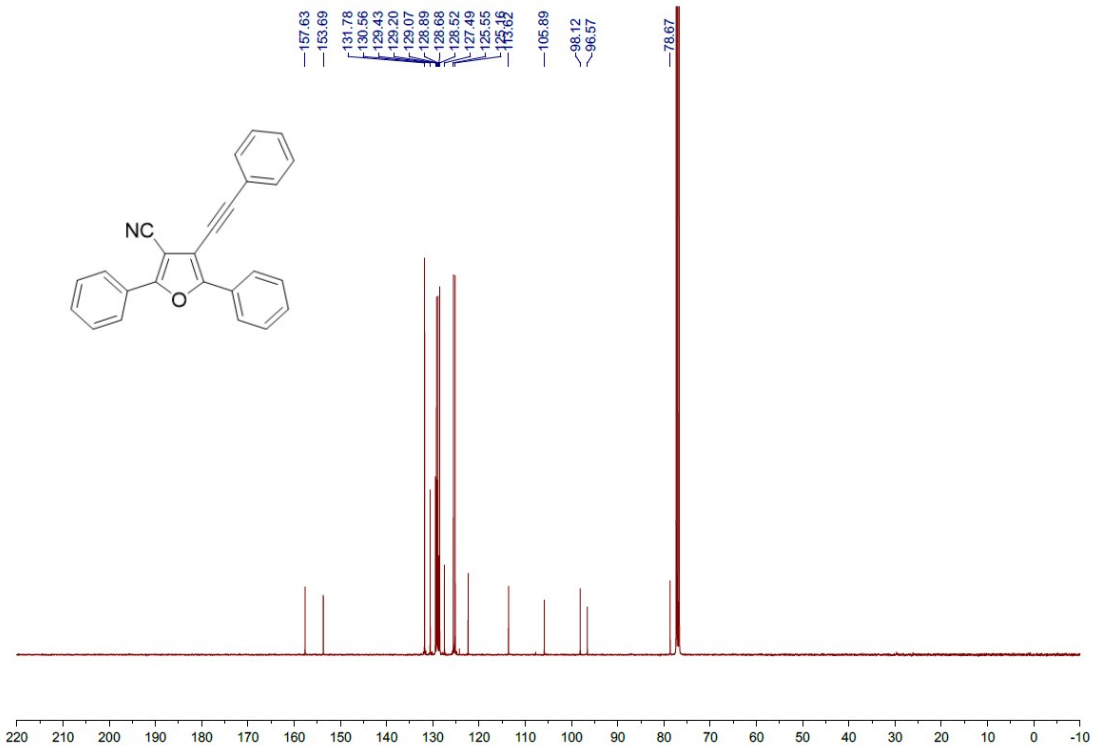
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3ta**



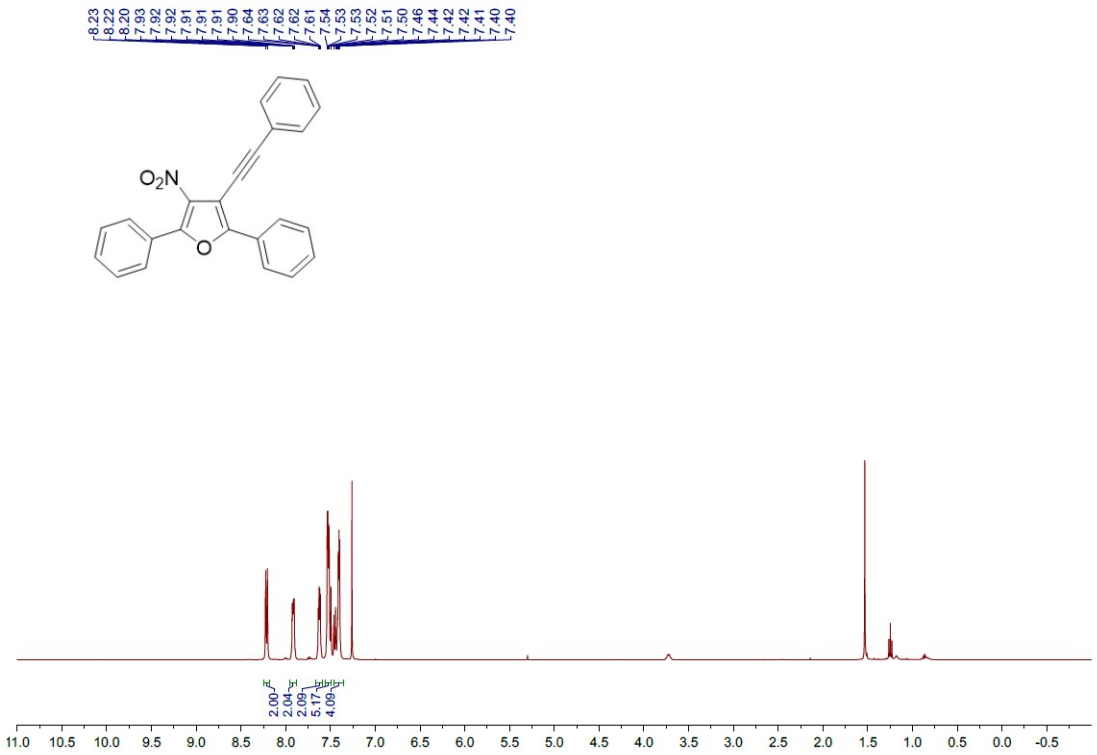
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3ua**



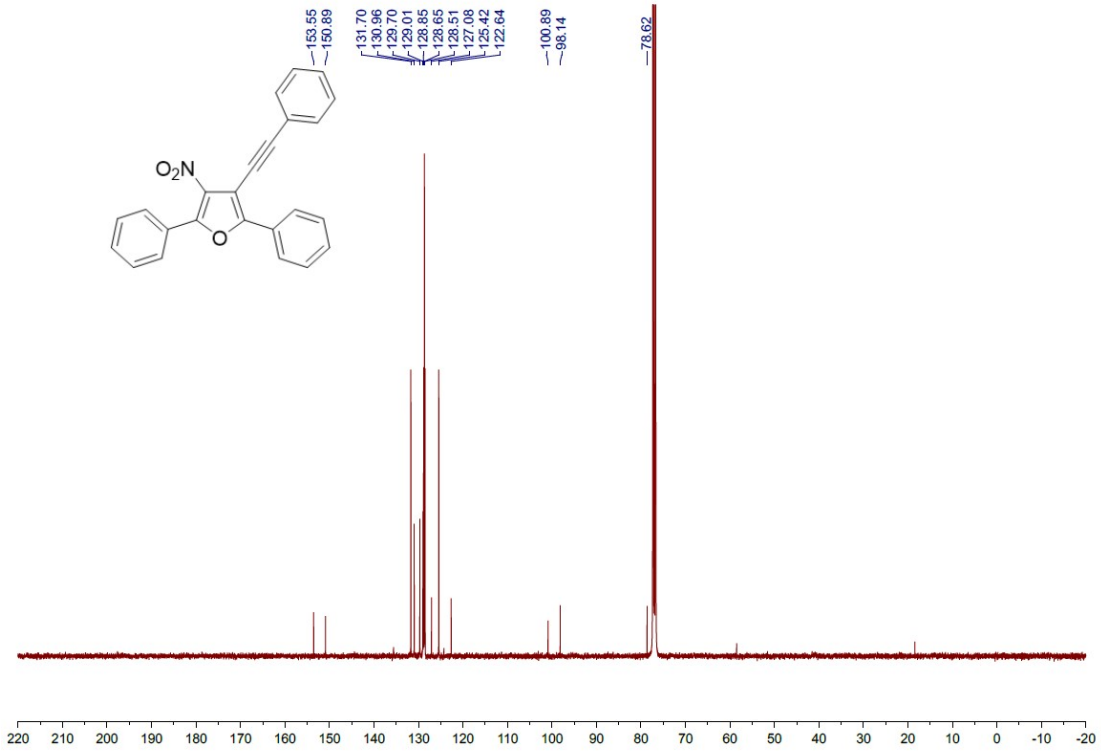
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3ua**



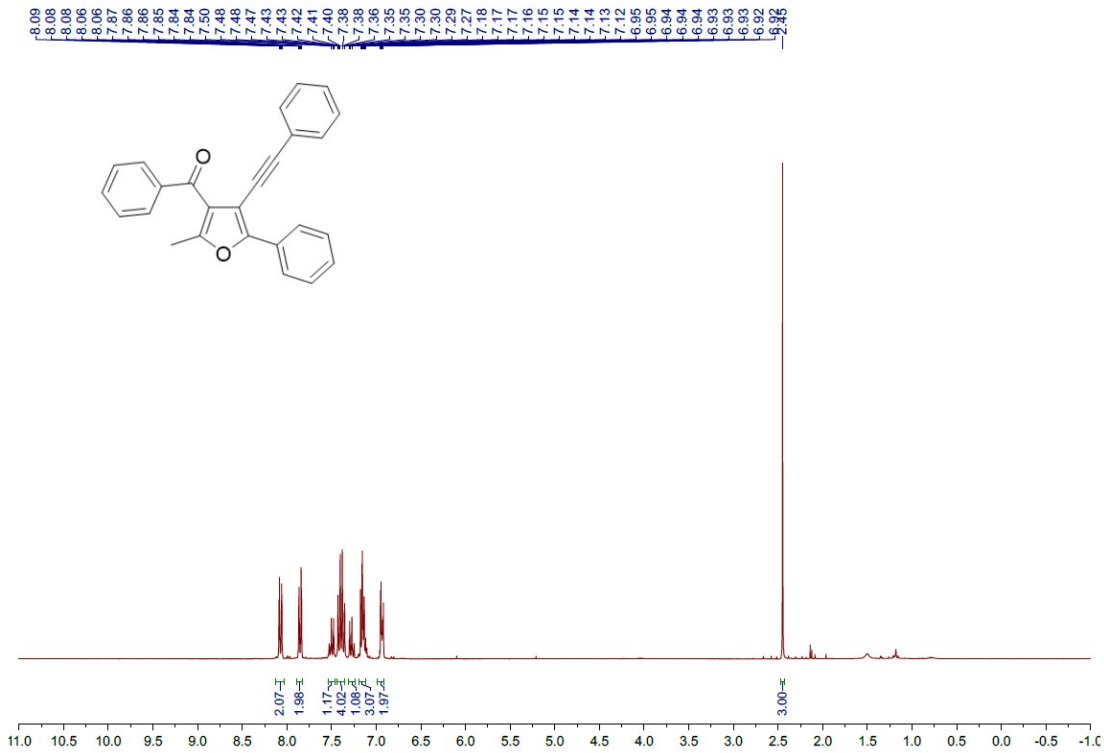
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3va**



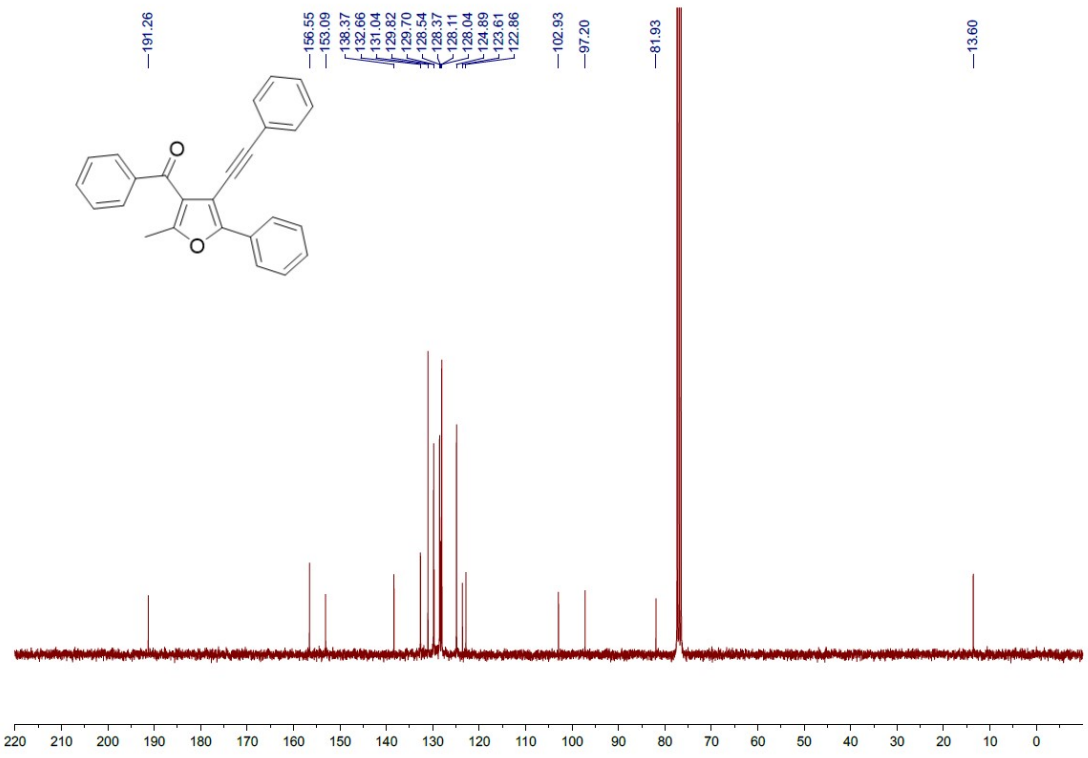
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3va**



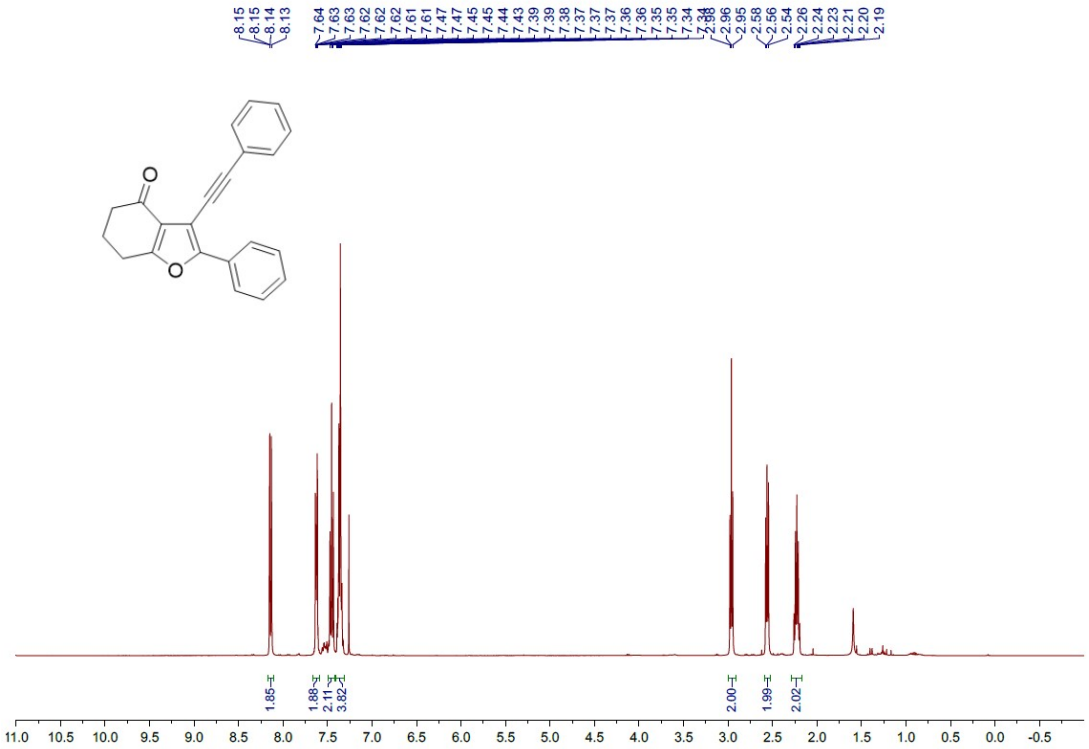
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of Compound **3wa**



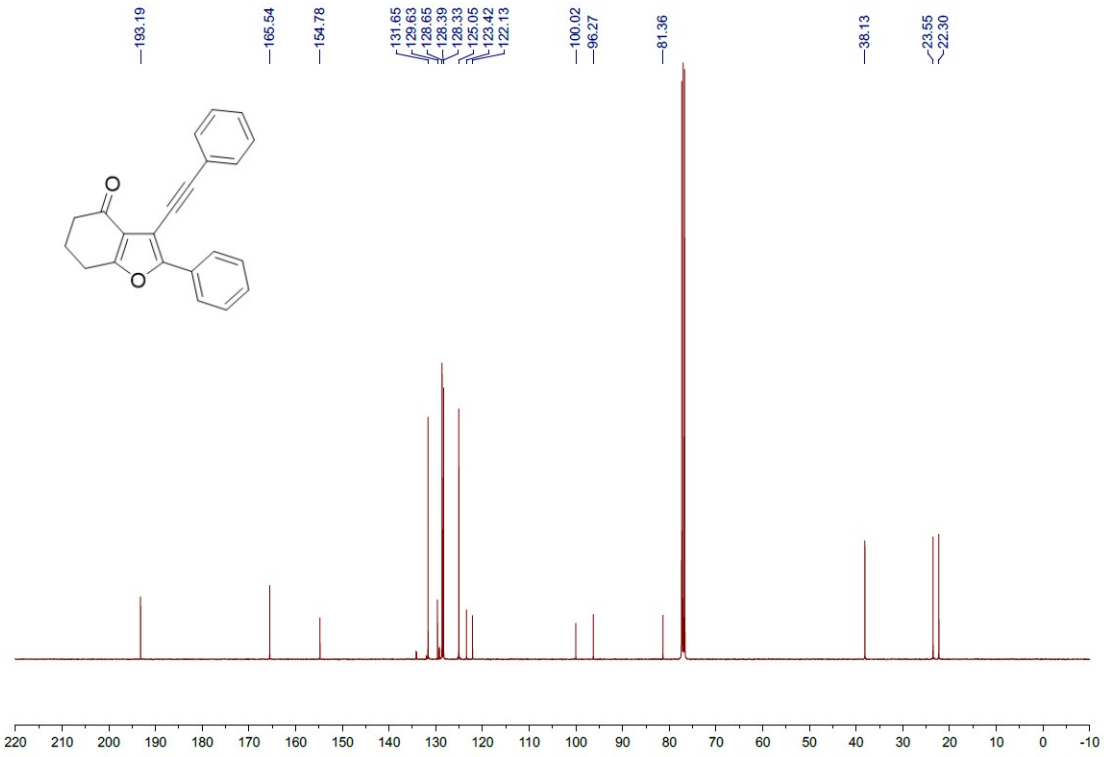
^{13}C NMR (75 MHz, $CDCl_3$) Spectrum of Compound **3wa**



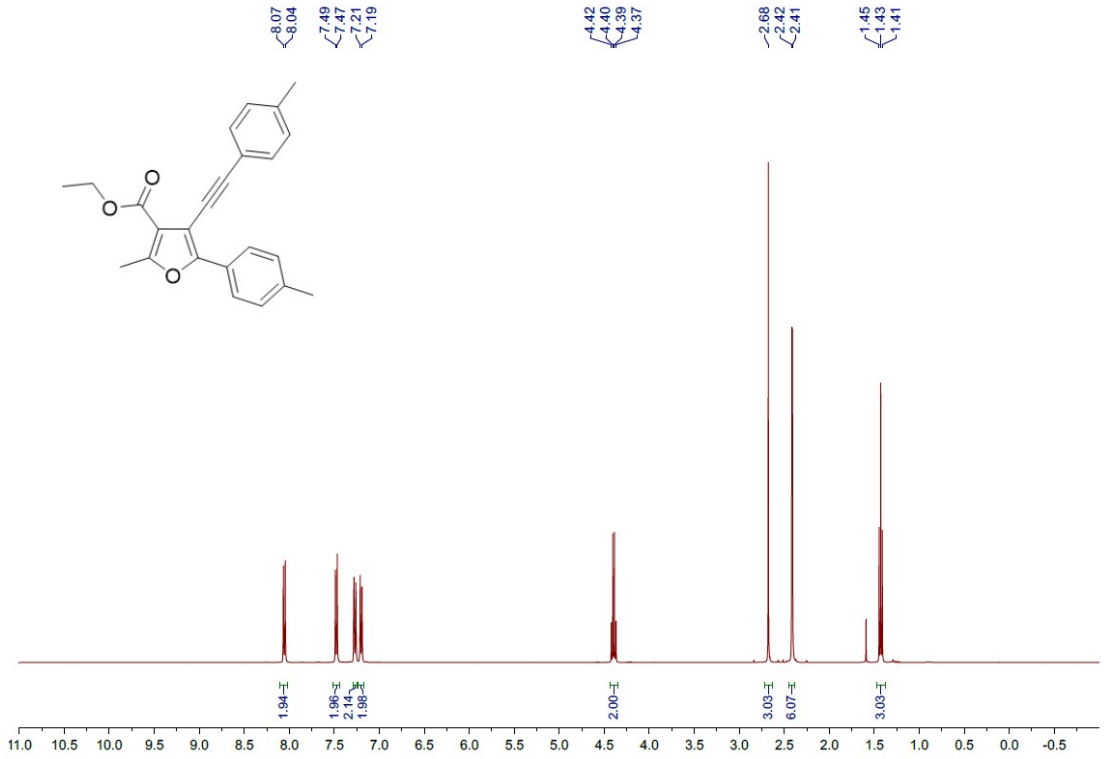
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3xa**



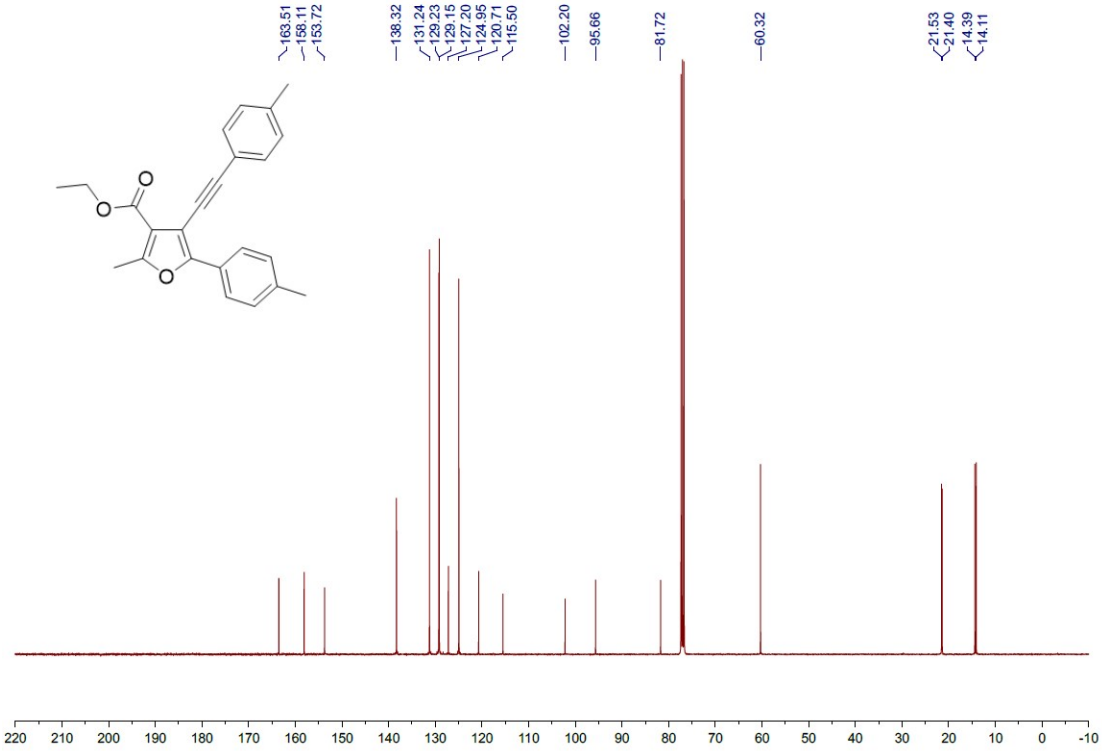
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3xa**



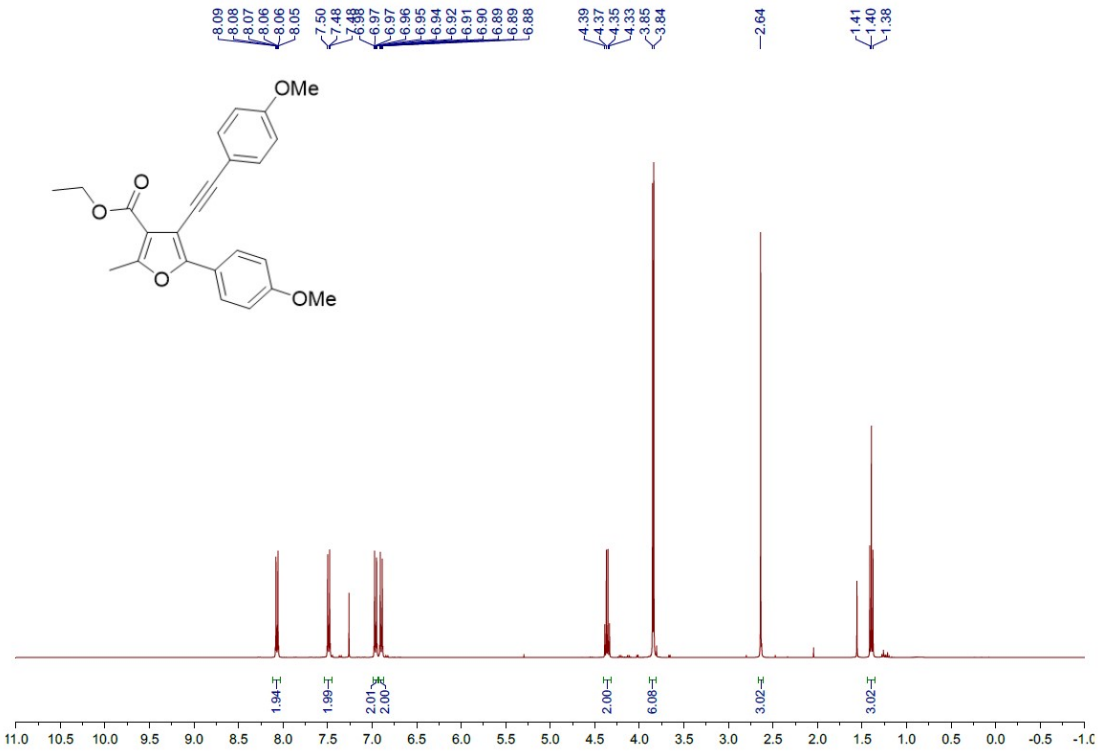
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3ab**



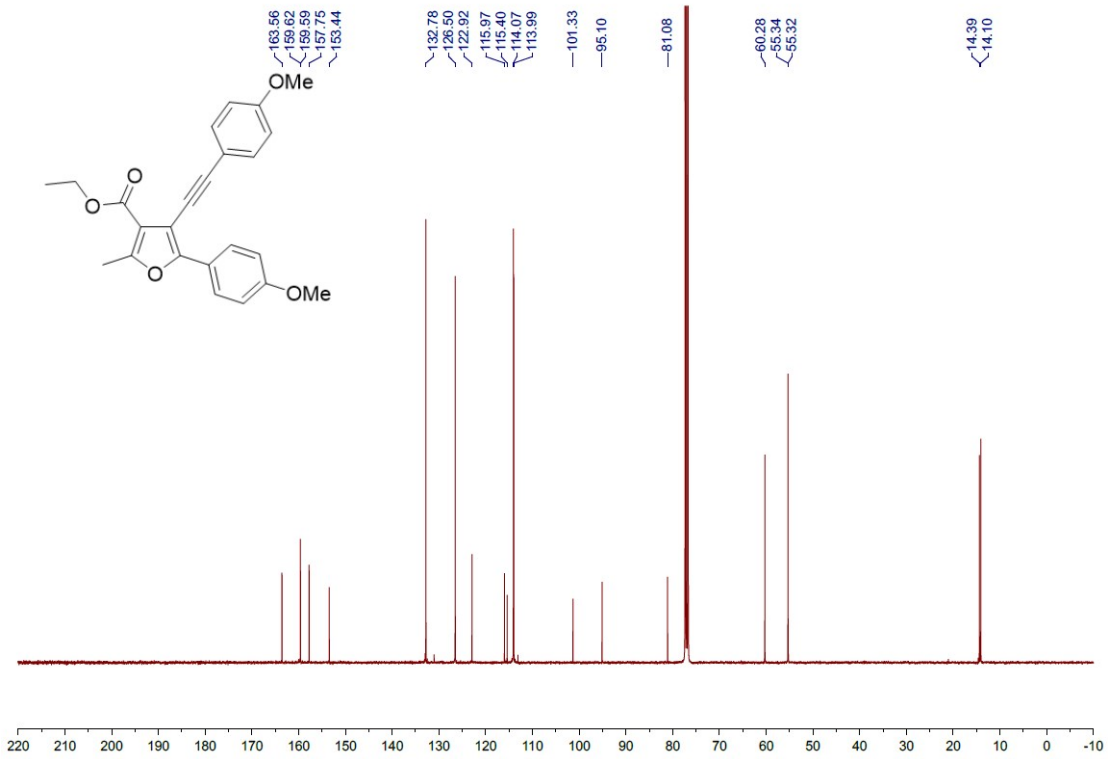
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3ab**



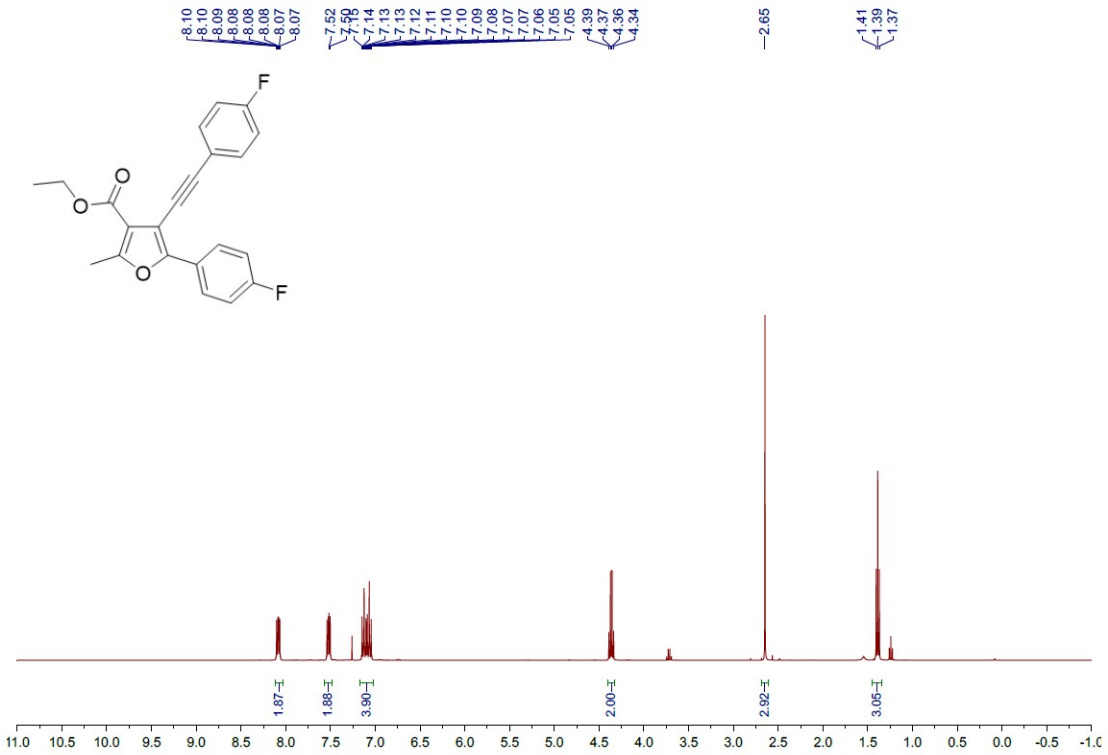
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3ac**



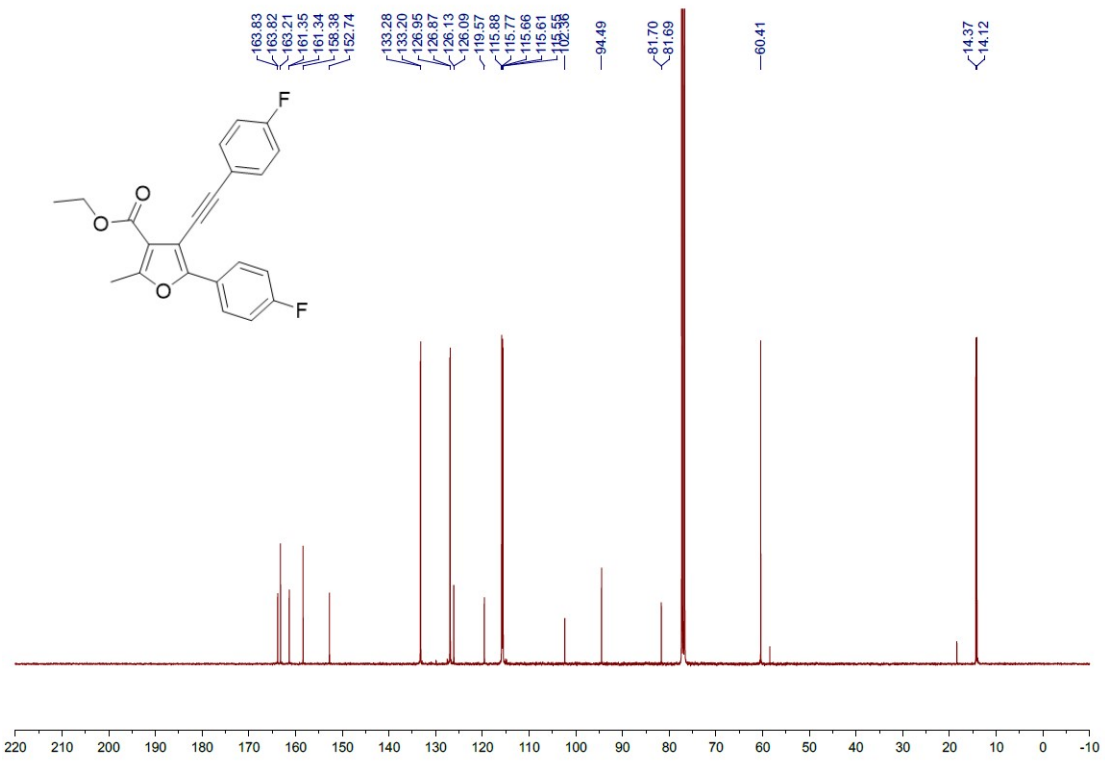
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3ac**



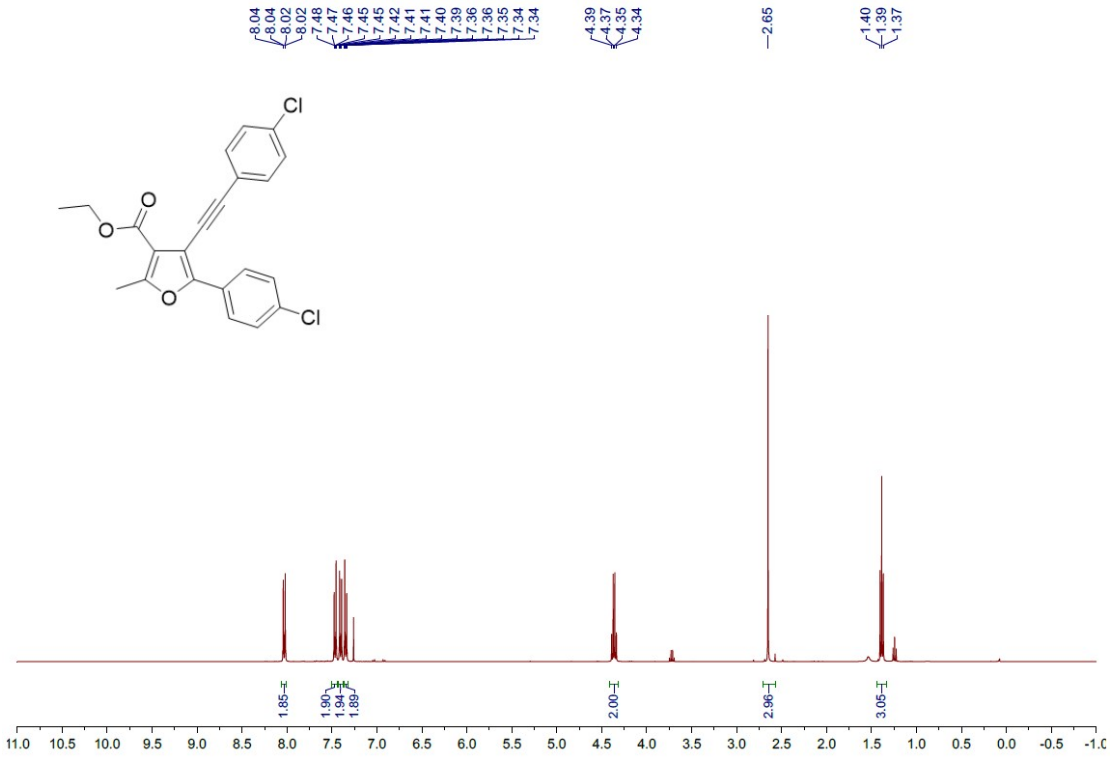
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3ad**



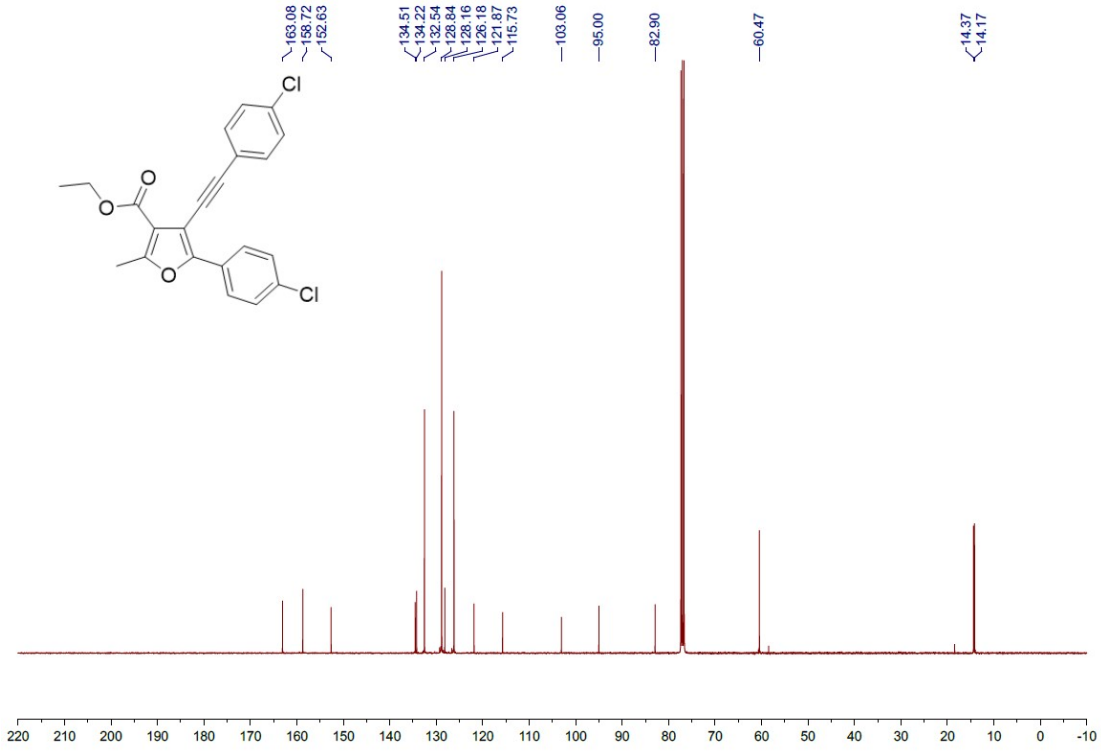
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3ad**



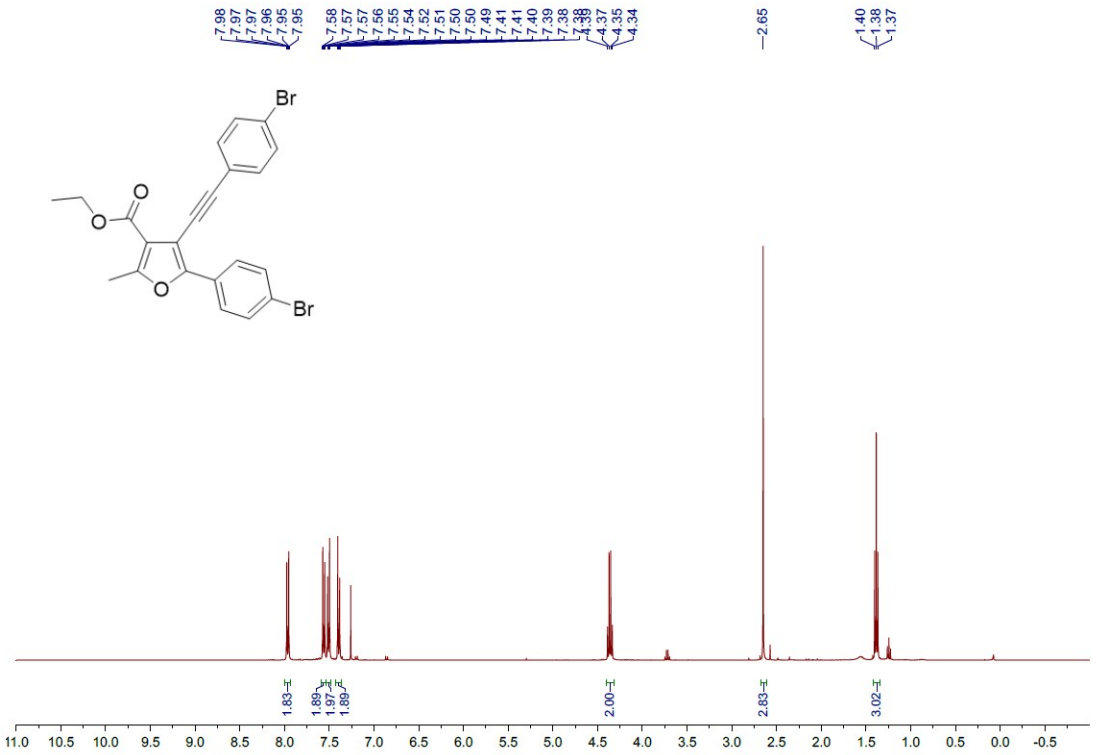
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3ae**



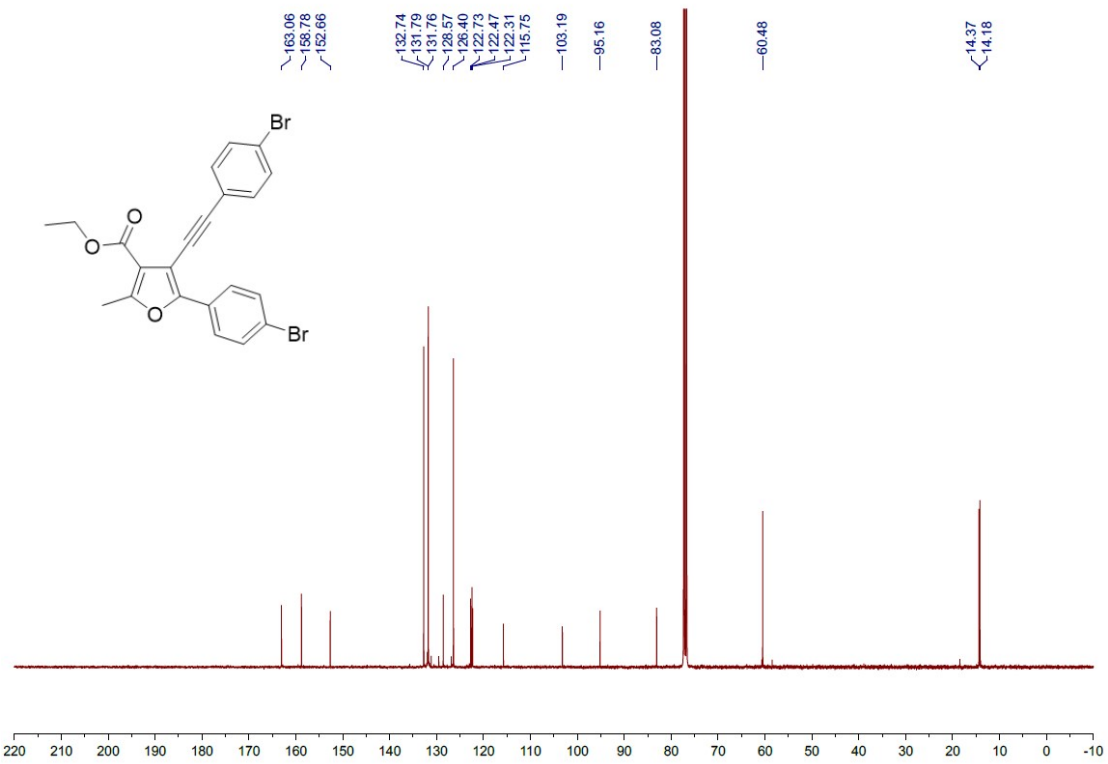
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3ae**



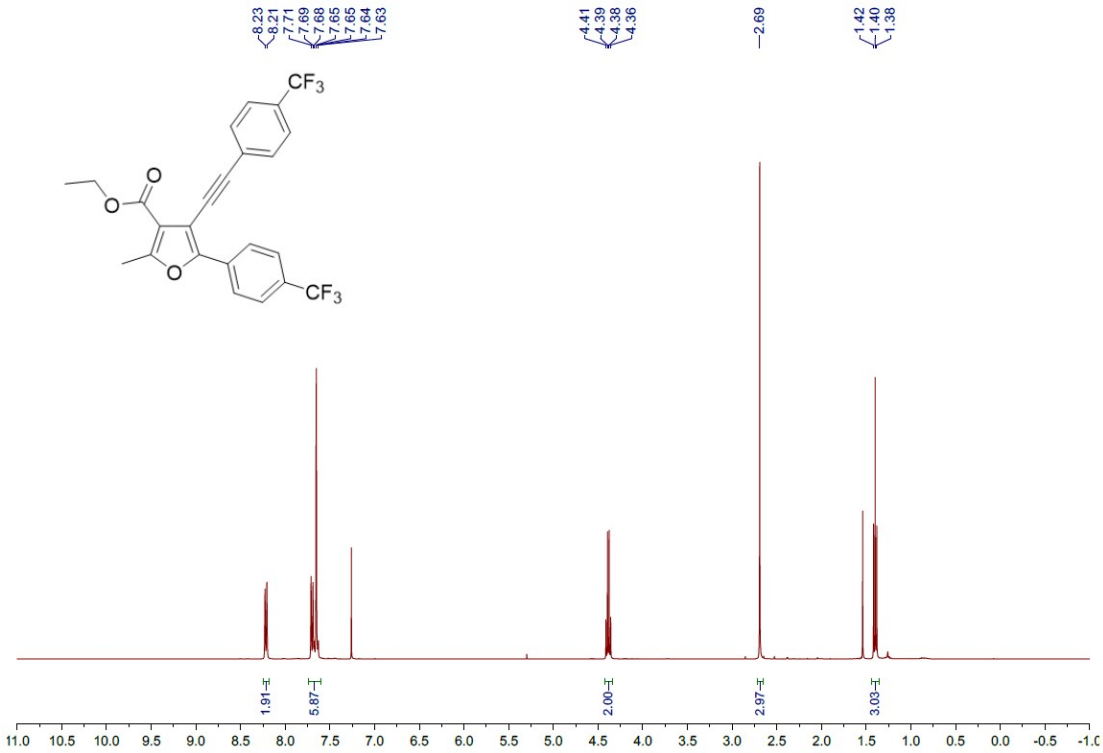
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3af**



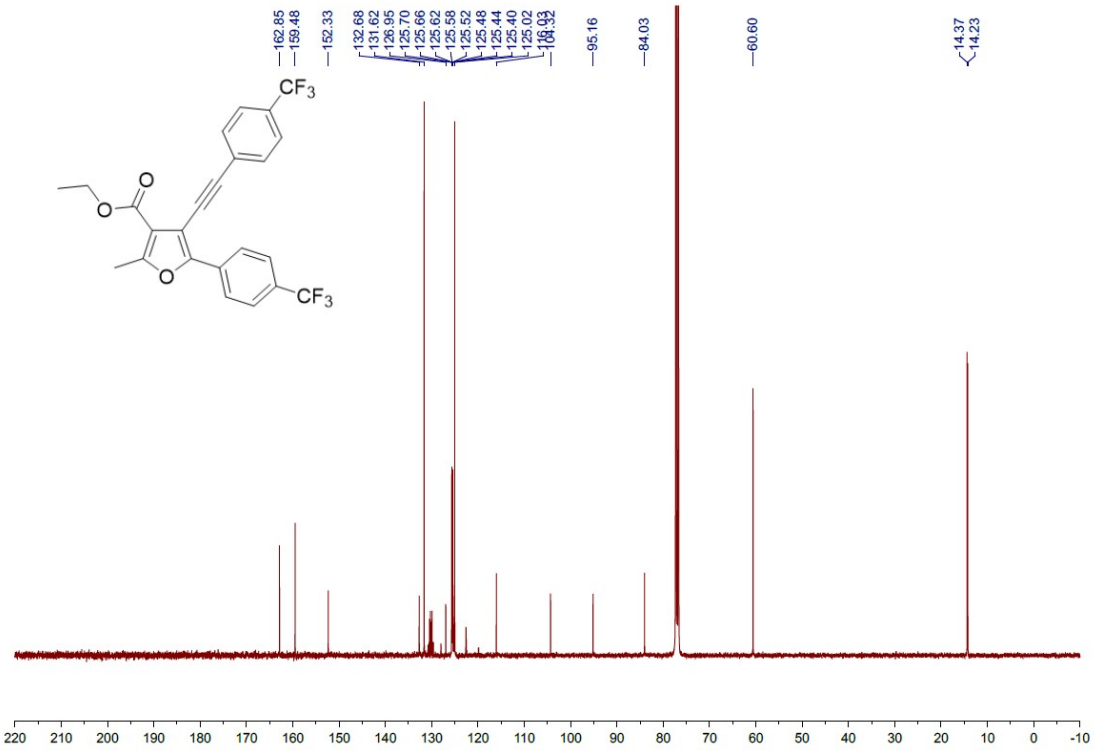
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3af**



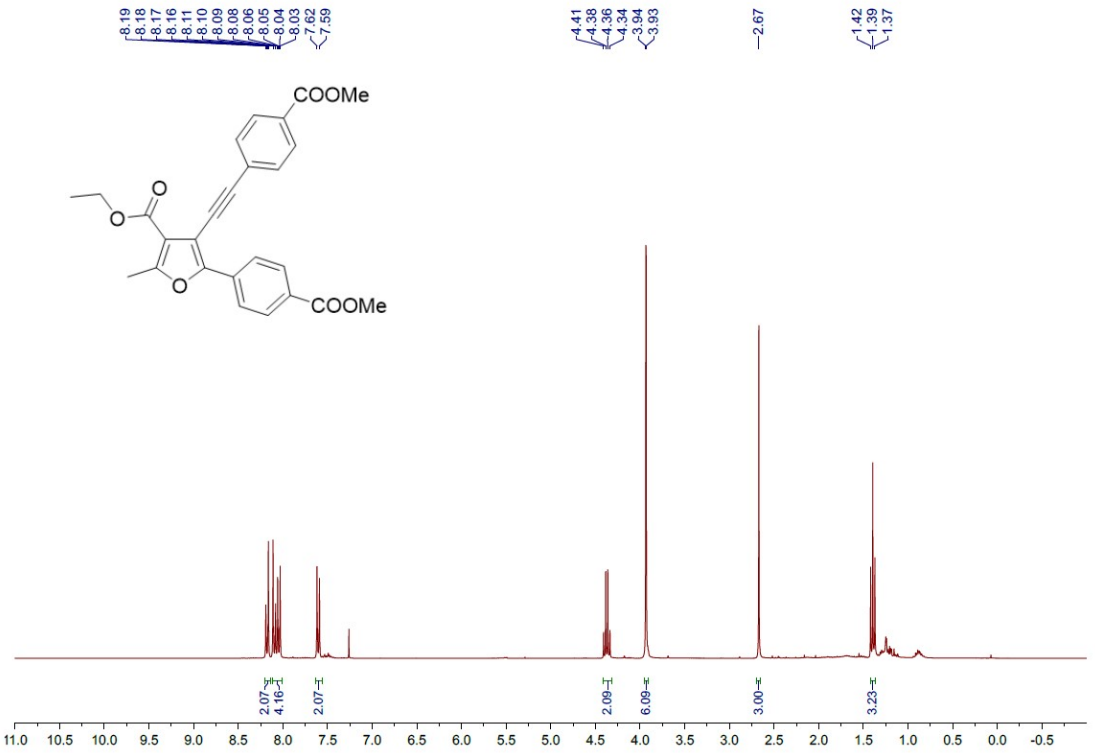
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3ag**



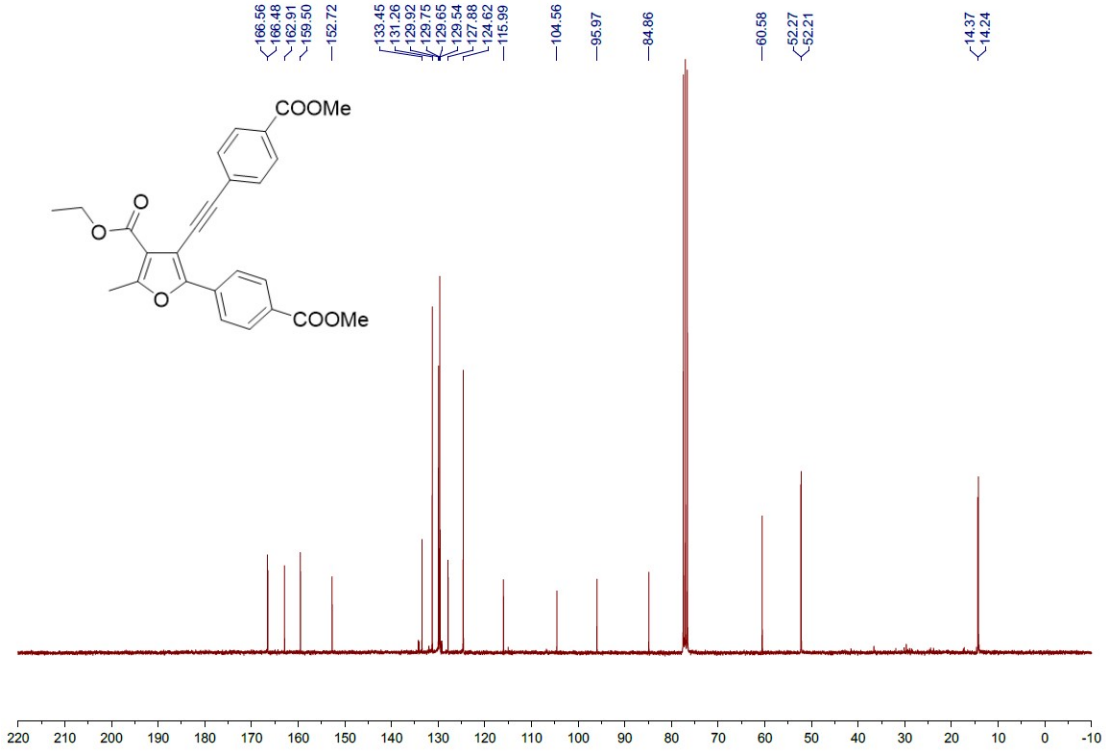
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3ag**



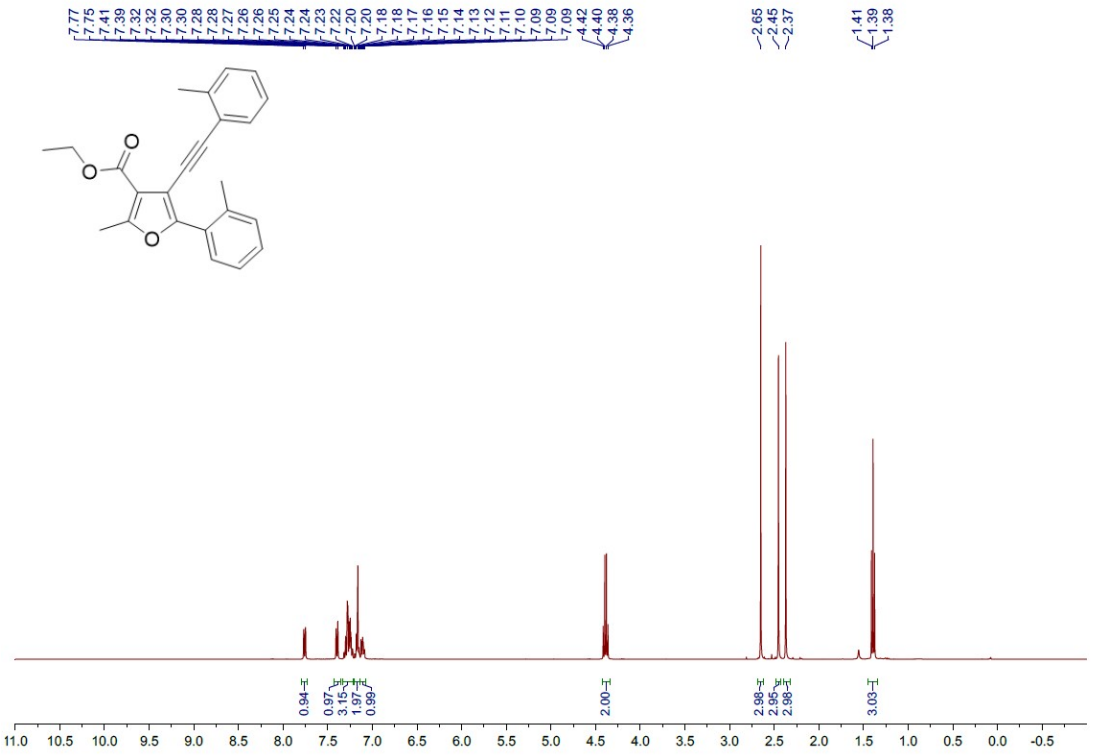
^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of Compound **3ah**



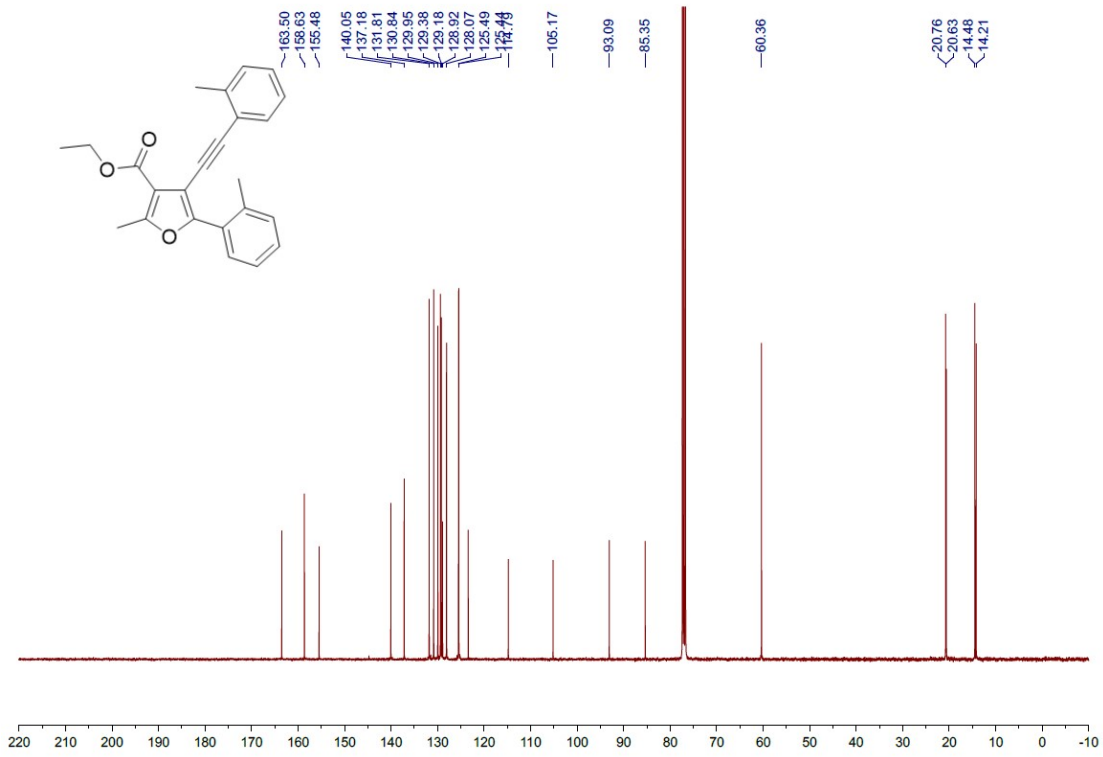
^{13}C NMR (75 MHz, $CDCl_3$) Spectrum of Compound **3ah**



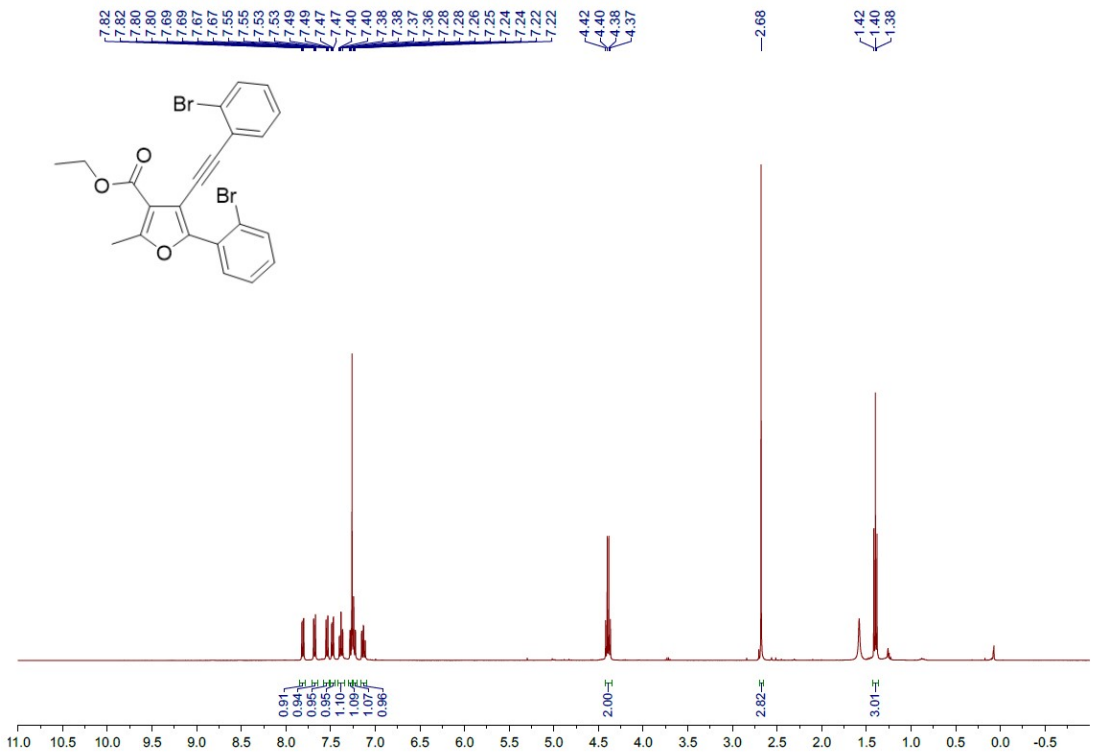
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3ai**



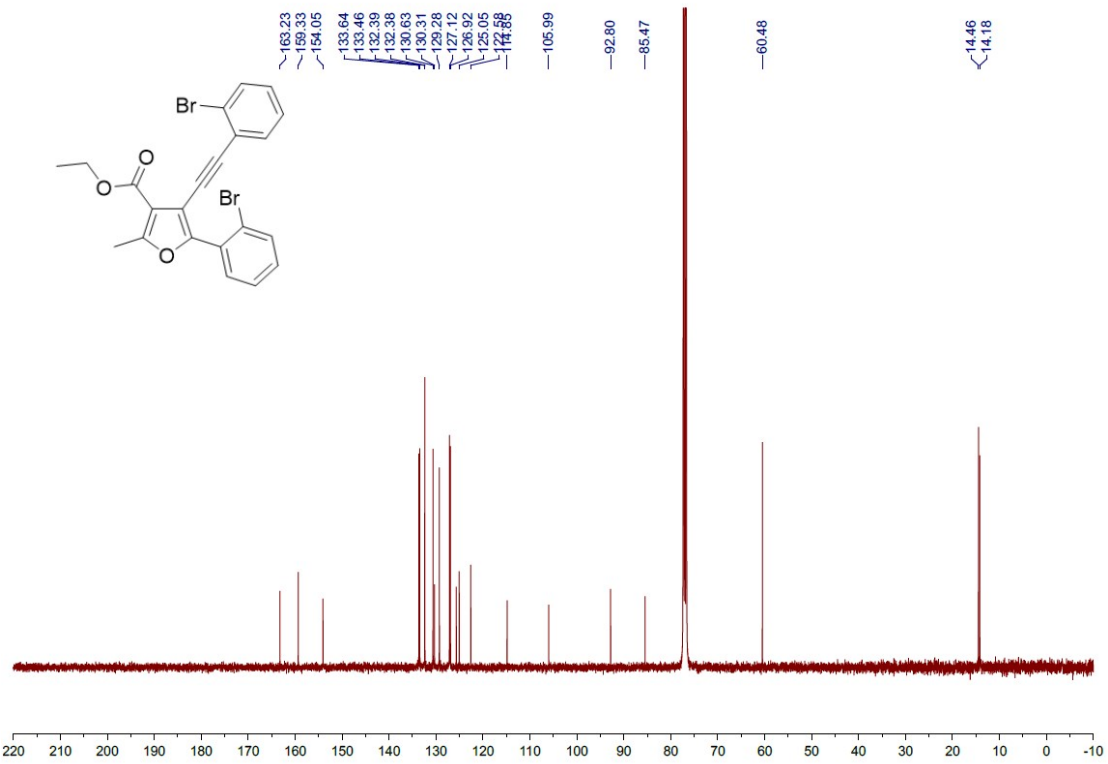
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3ai**



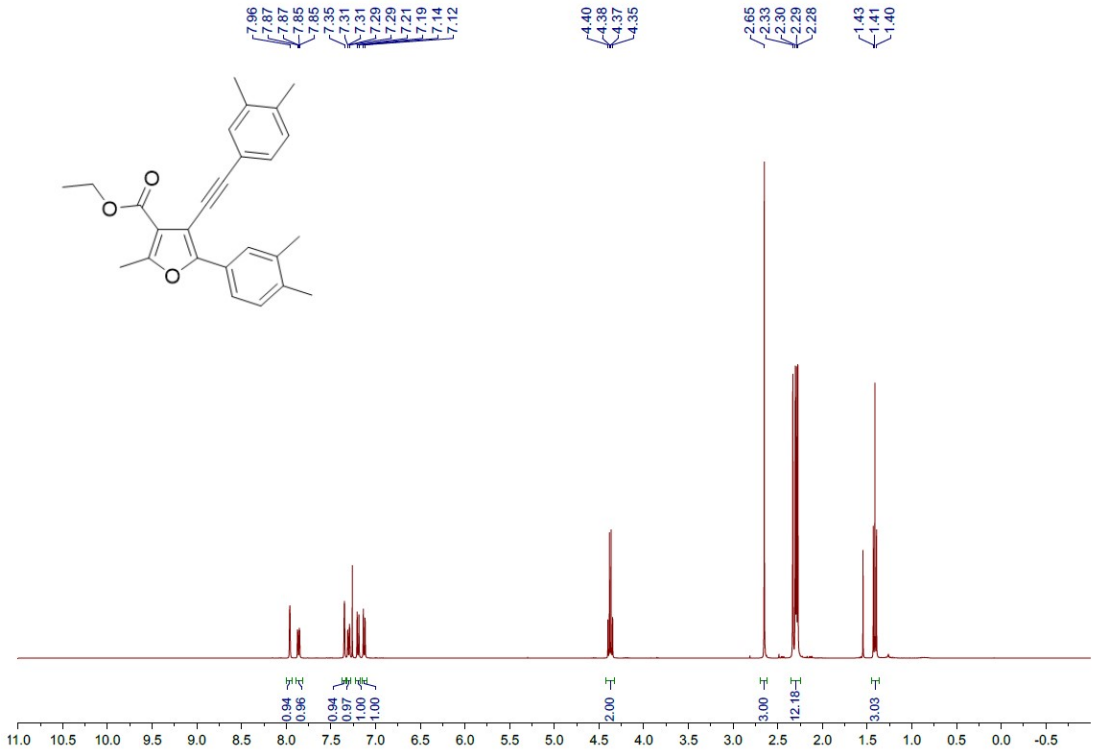
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3aj**



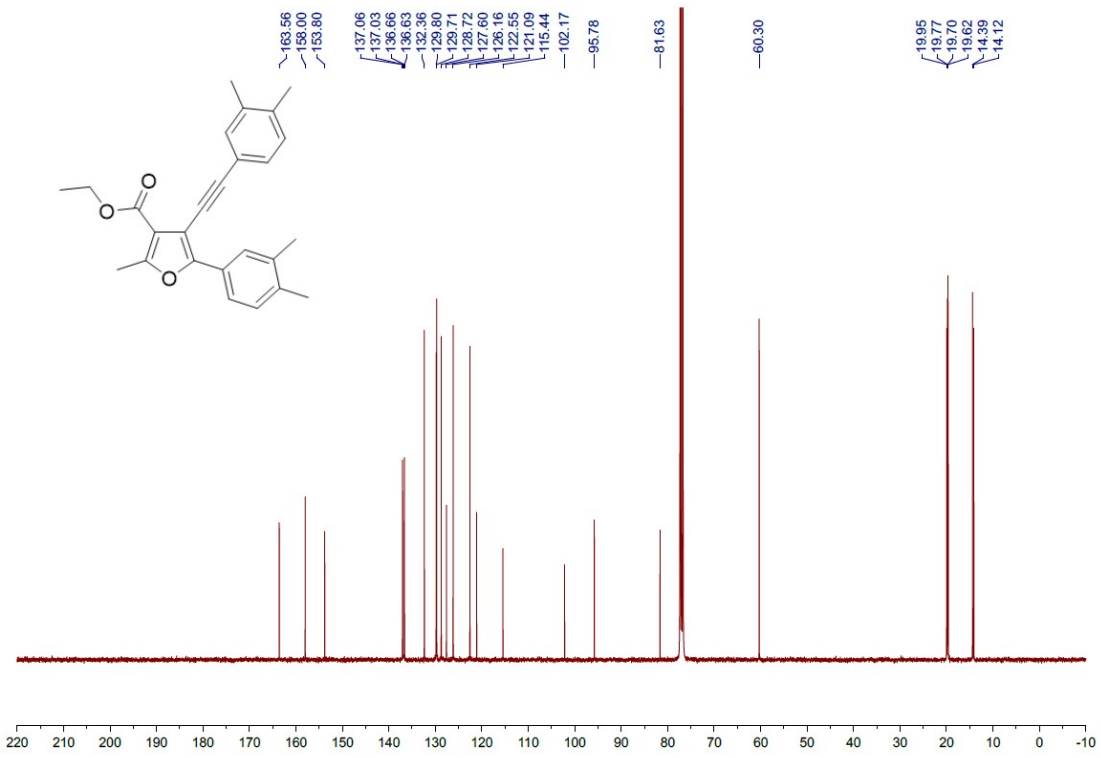
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3aj**



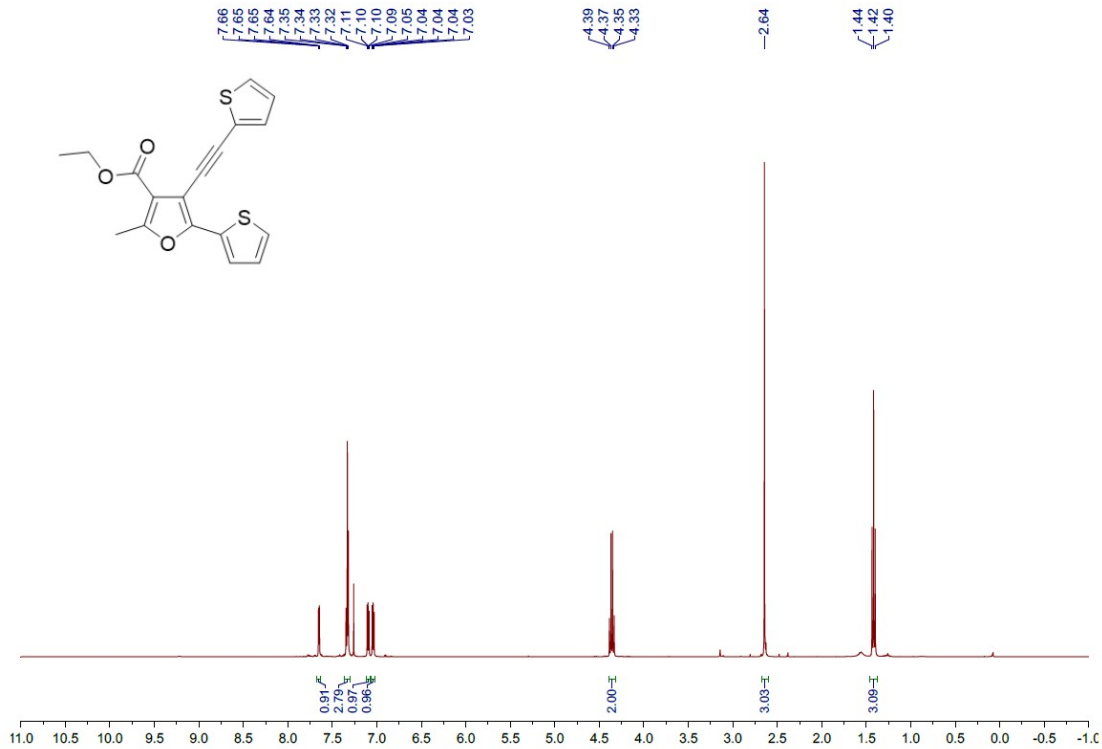
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3ak**



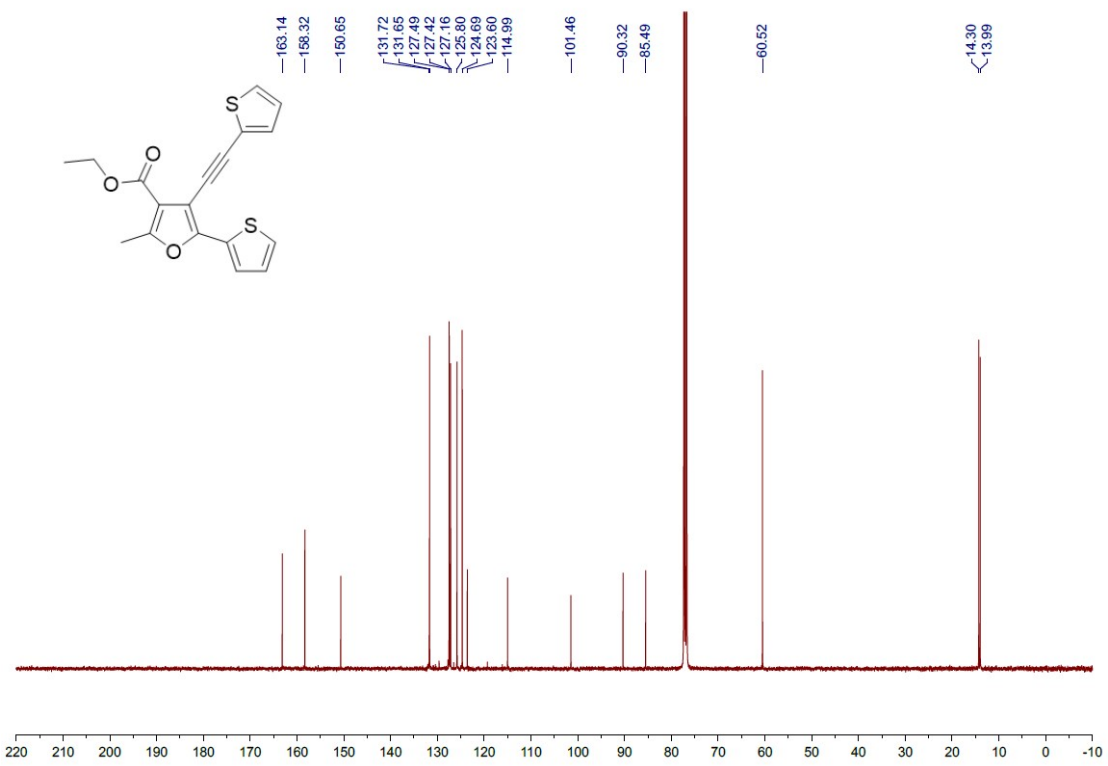
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3ak**



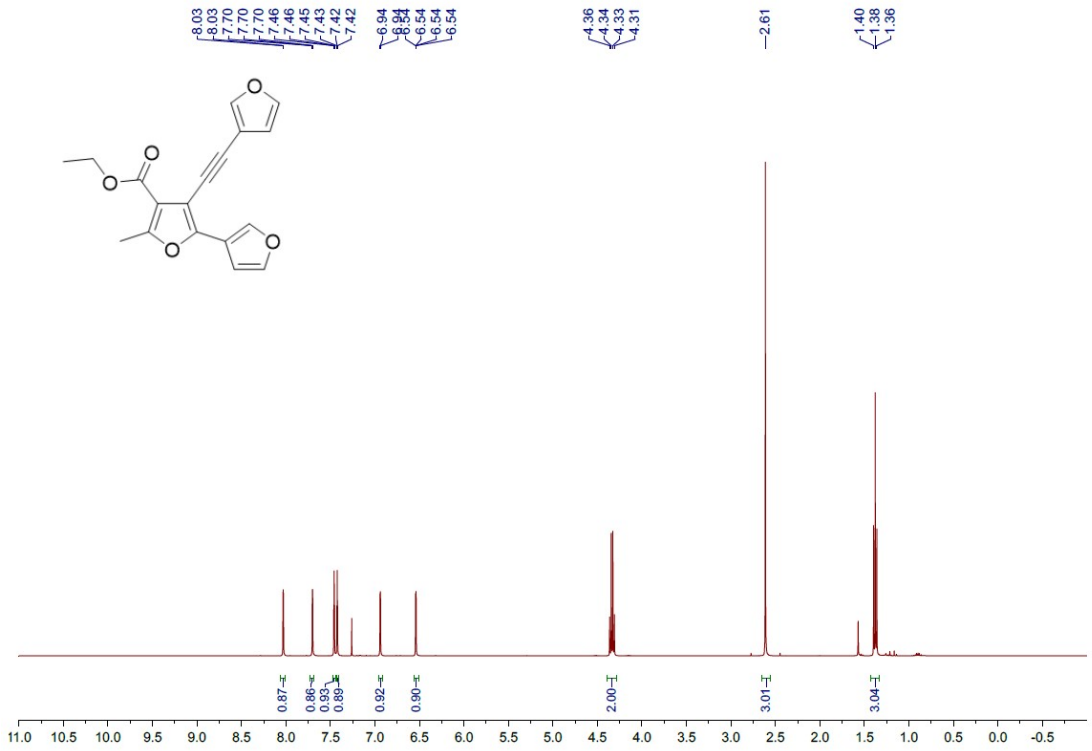
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3al**



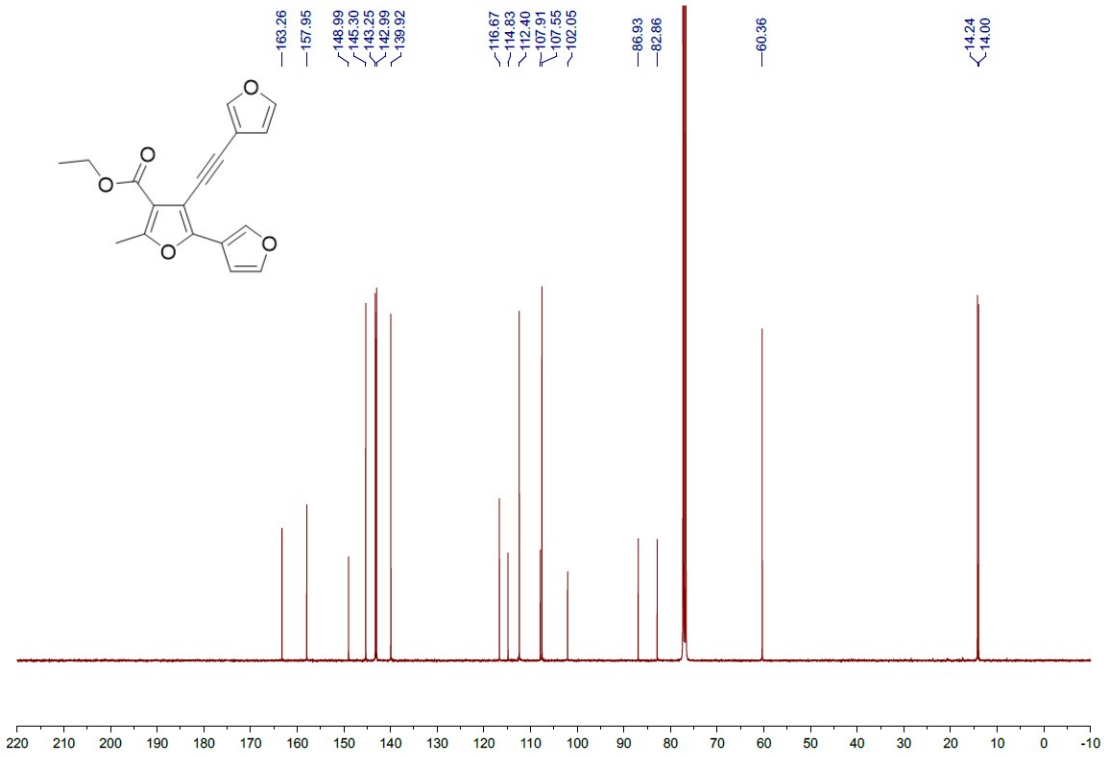
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3al**



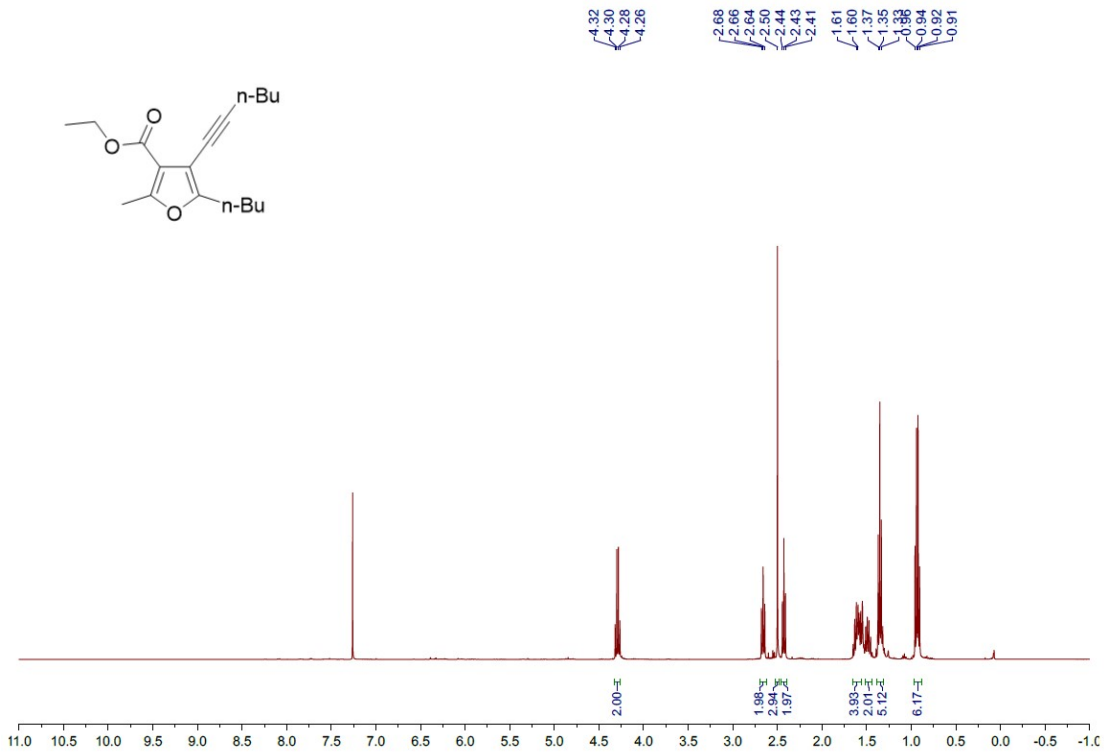
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3am**



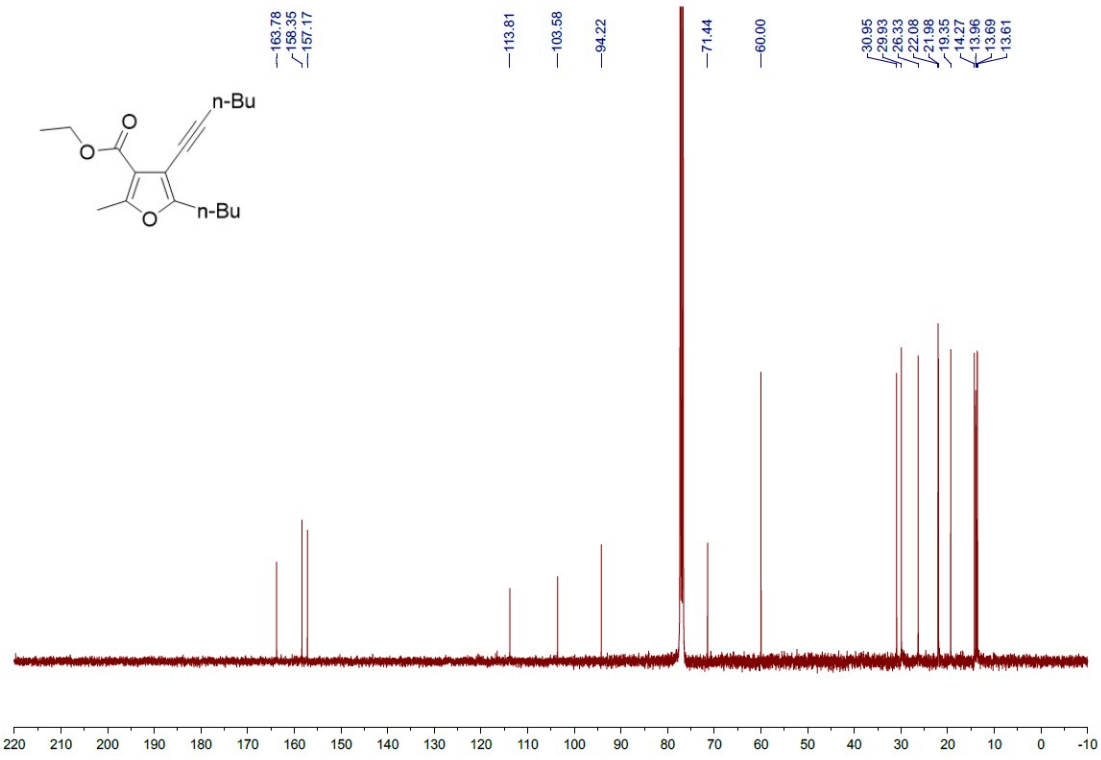
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3am**



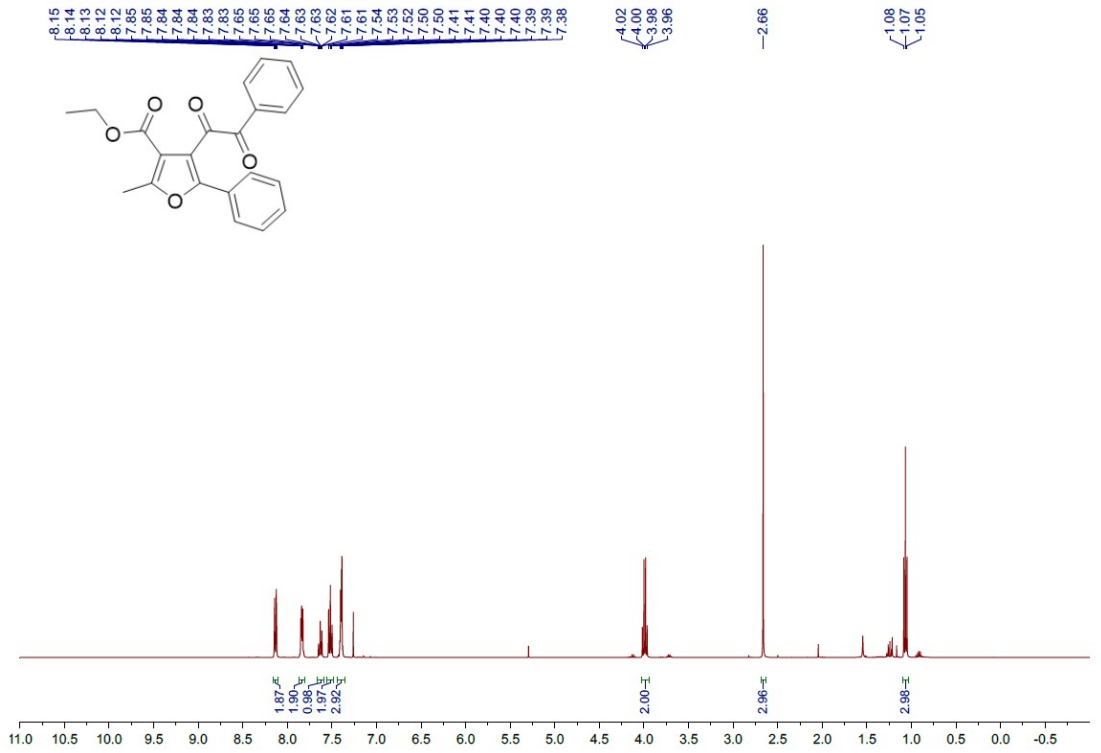
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **3an**



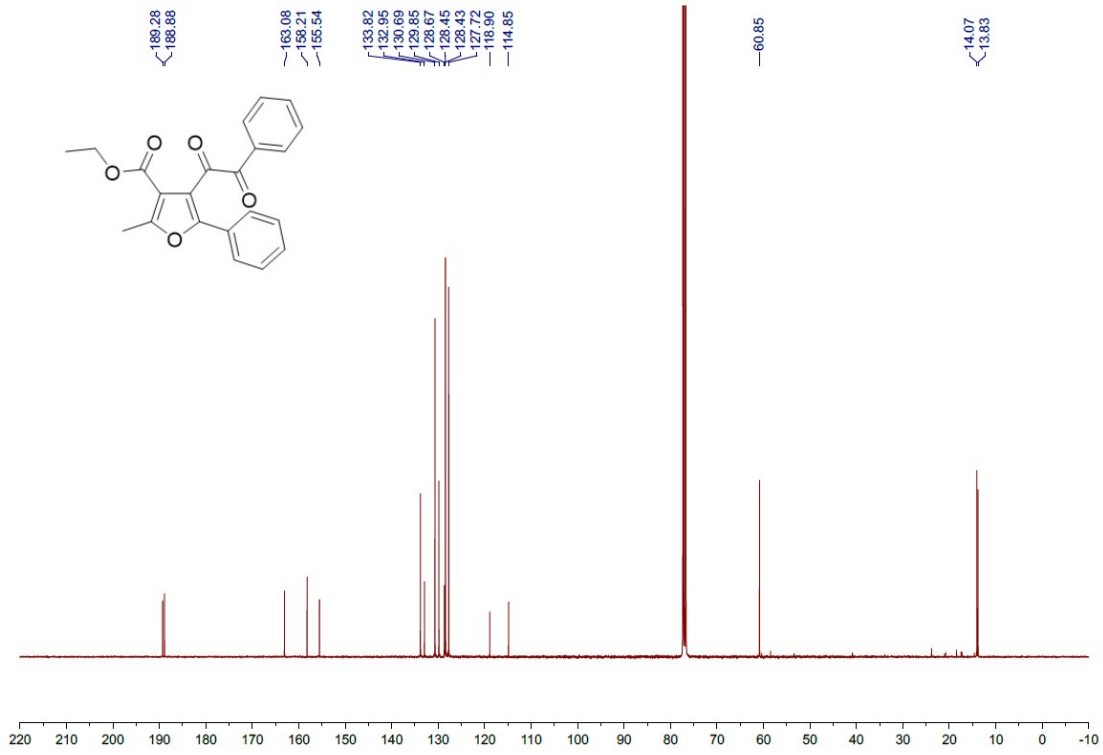
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **3an**



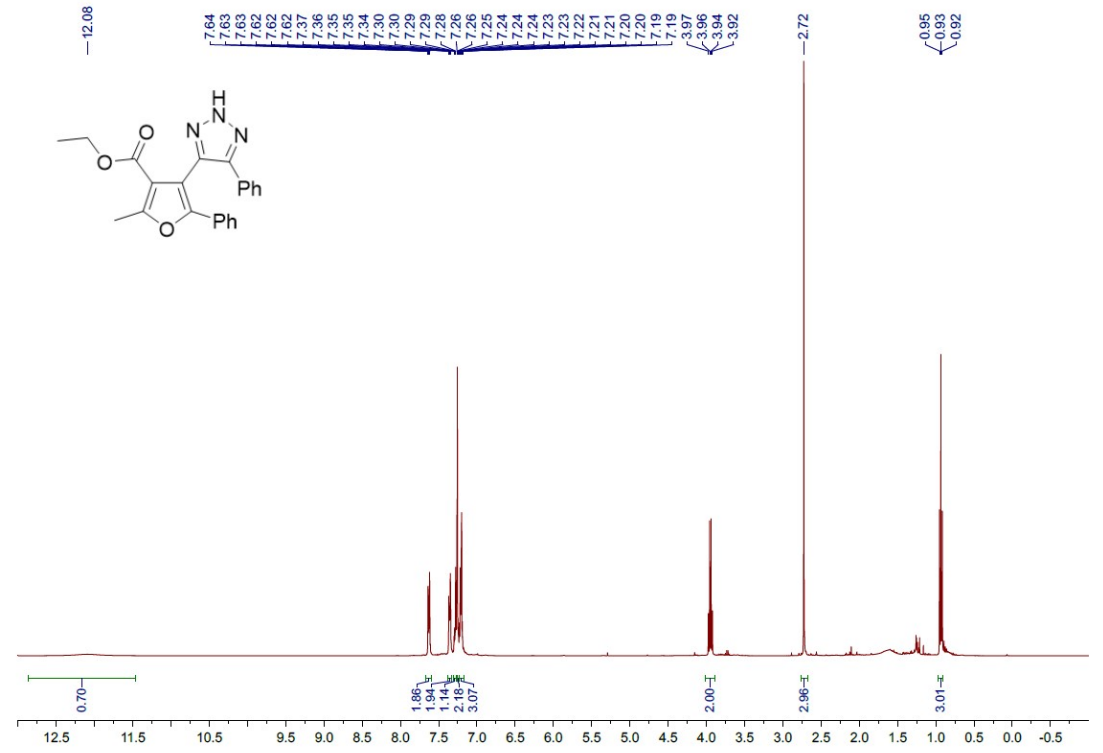
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **4**



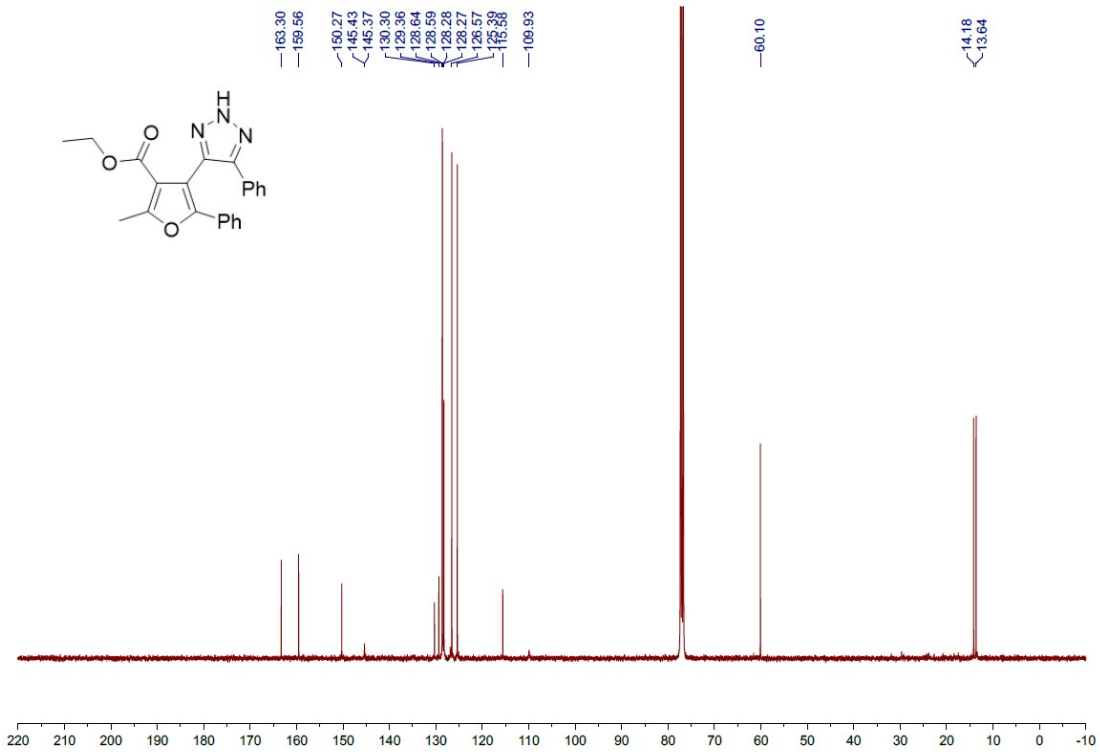
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **4**



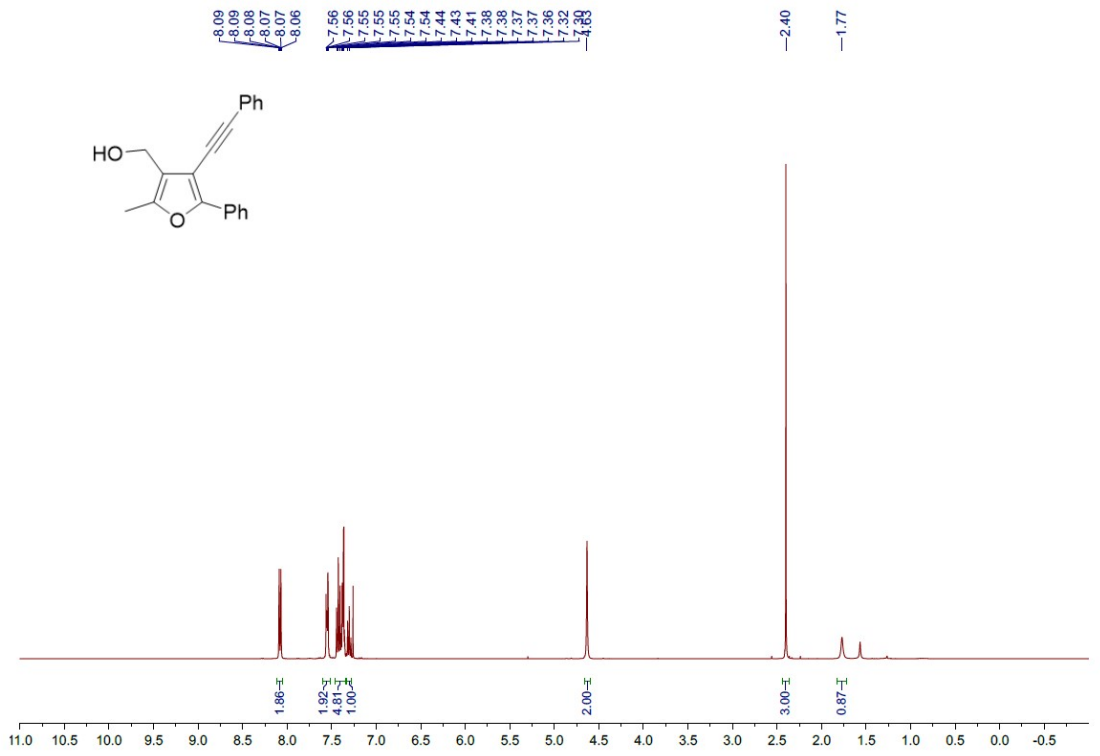
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **5**



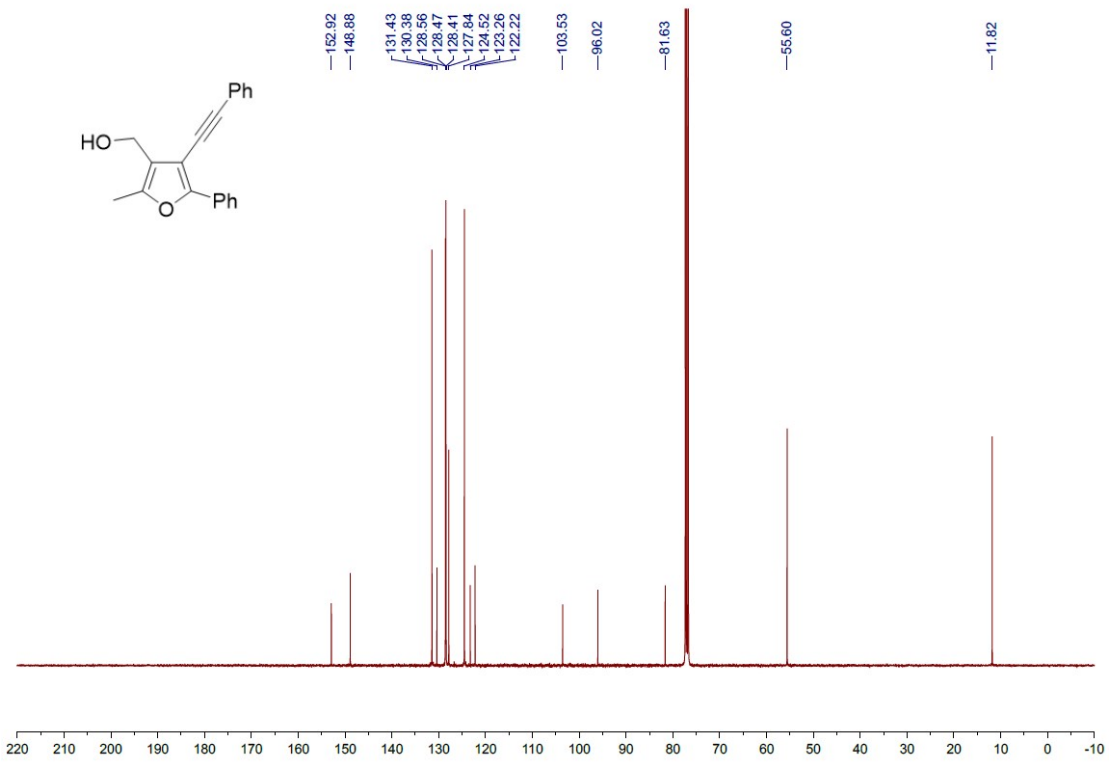
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **5**



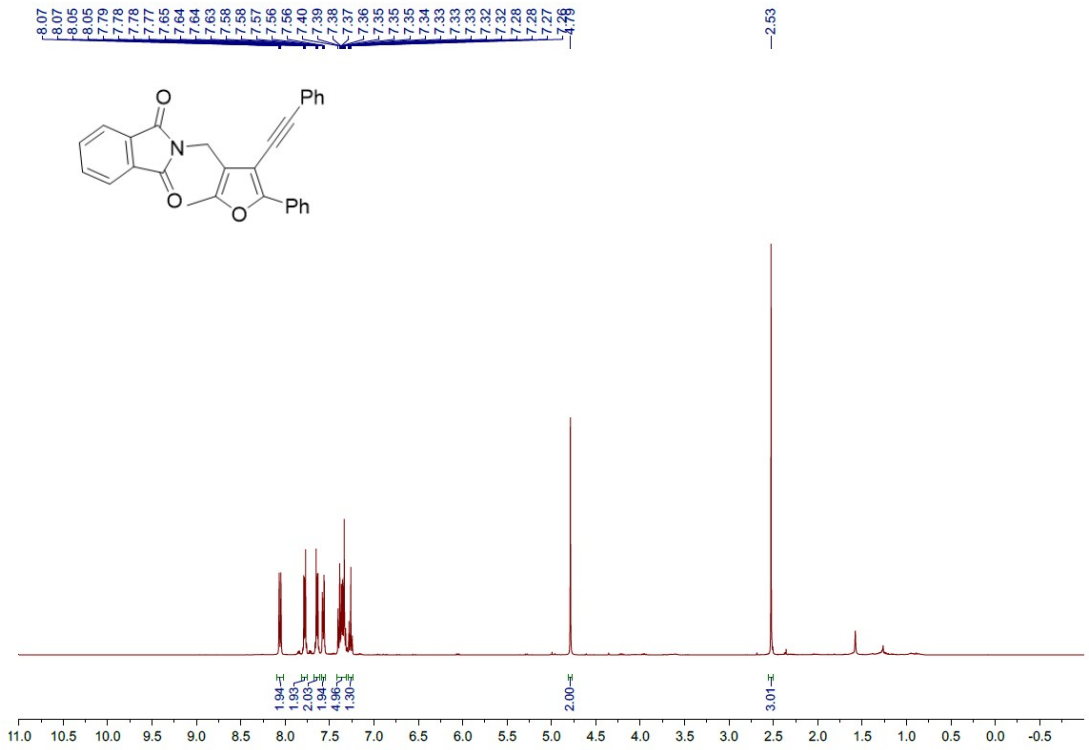
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **6**



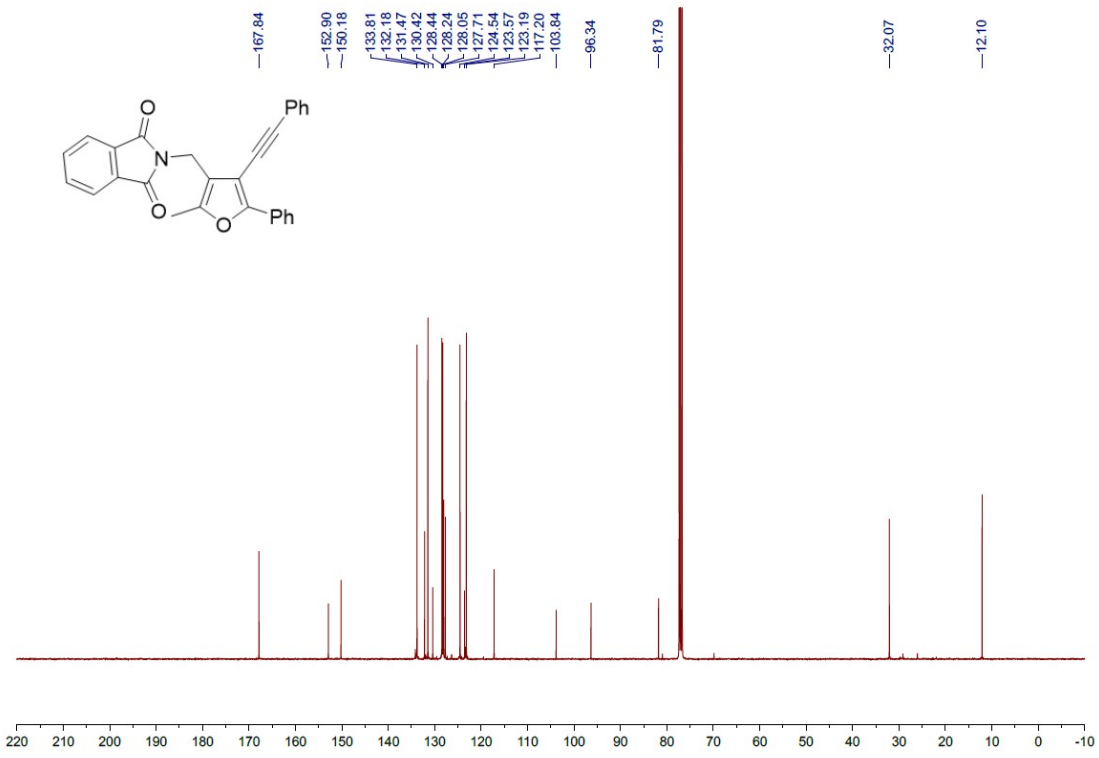
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **6**



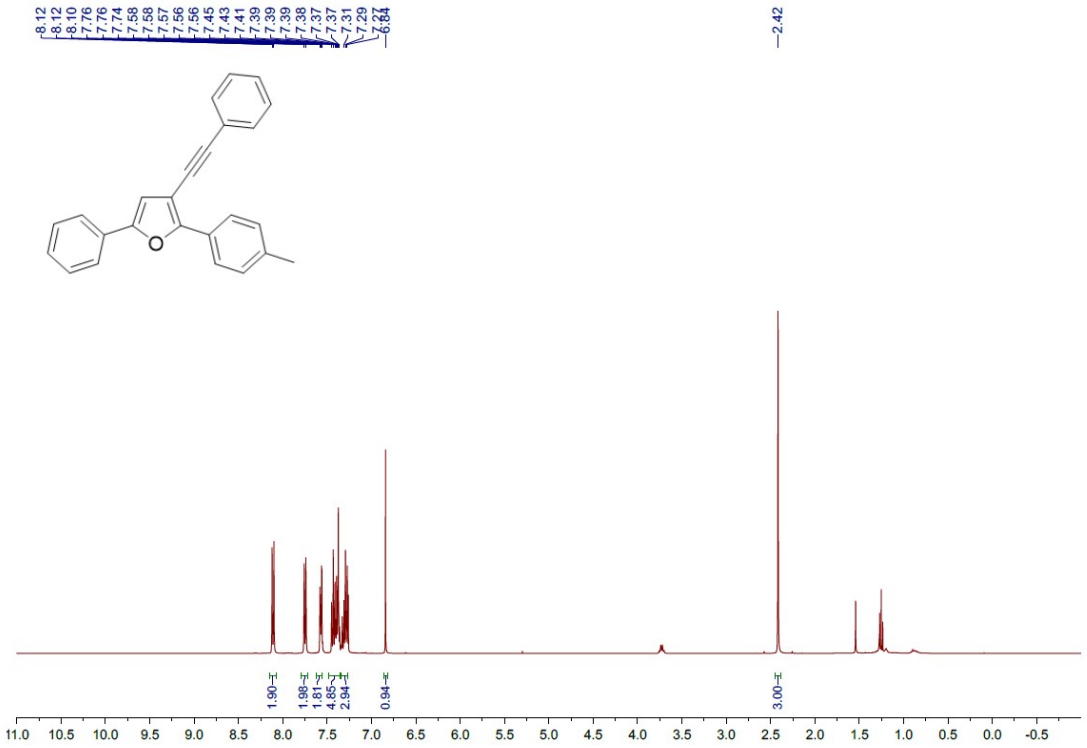
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **7**



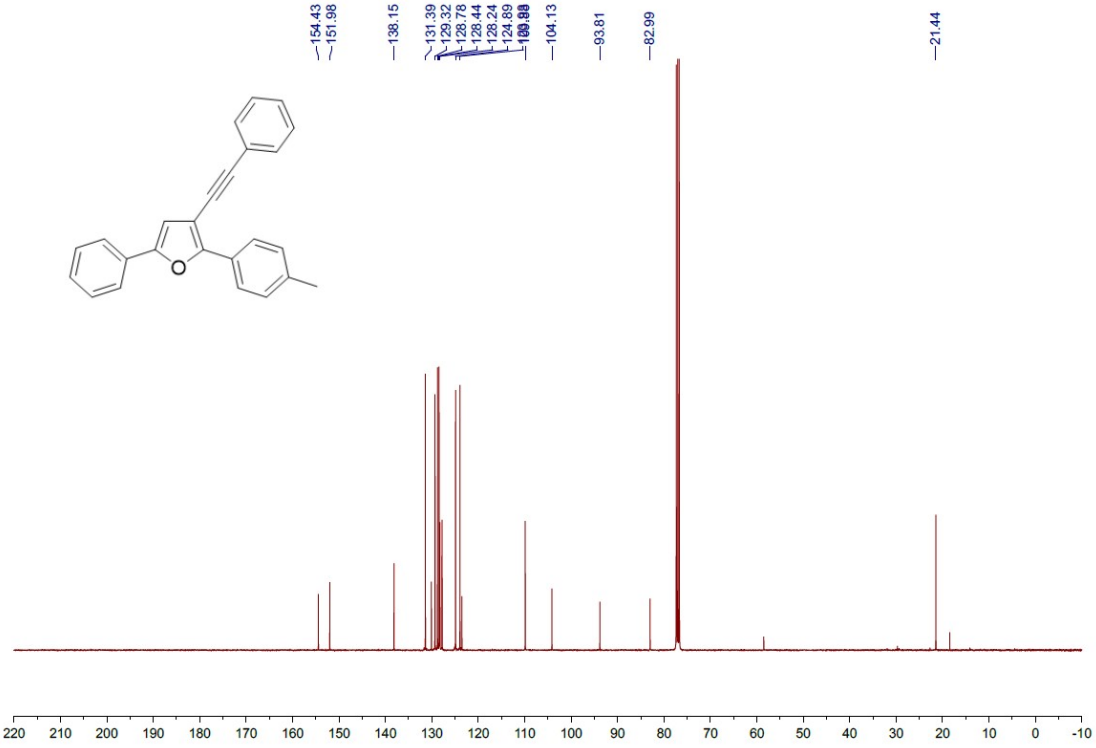
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **7**



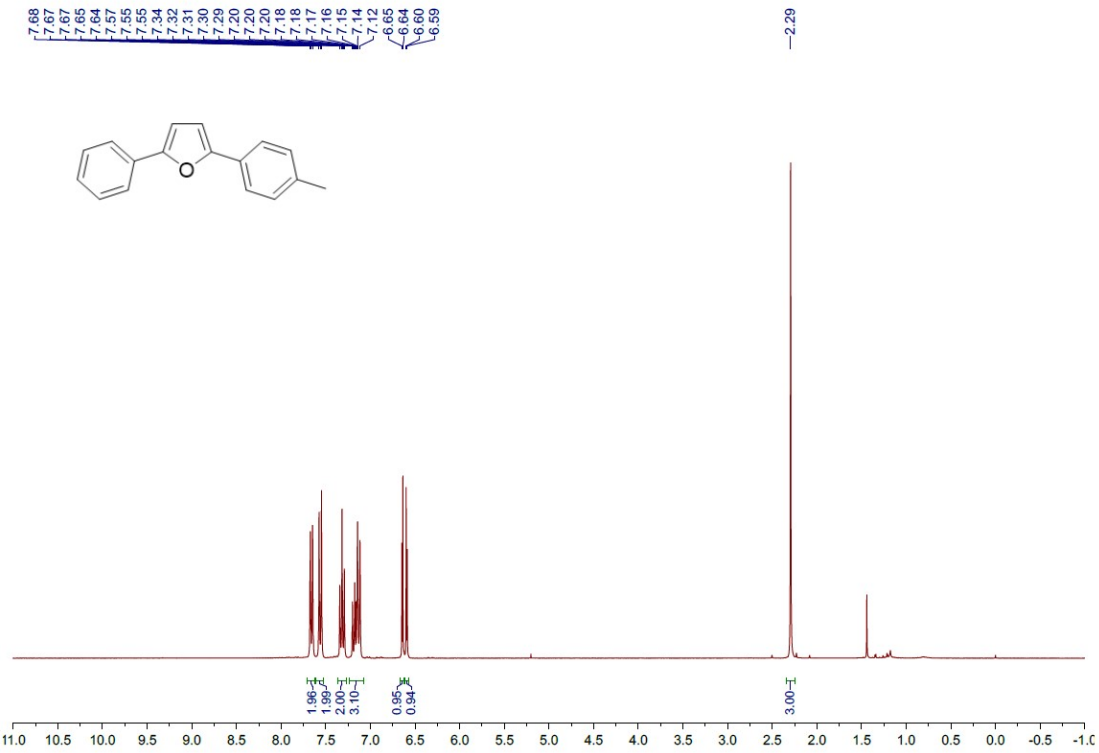
^{1}H NMR (400 MHz, $CDCl_3$) Spectrum of Compound **9**



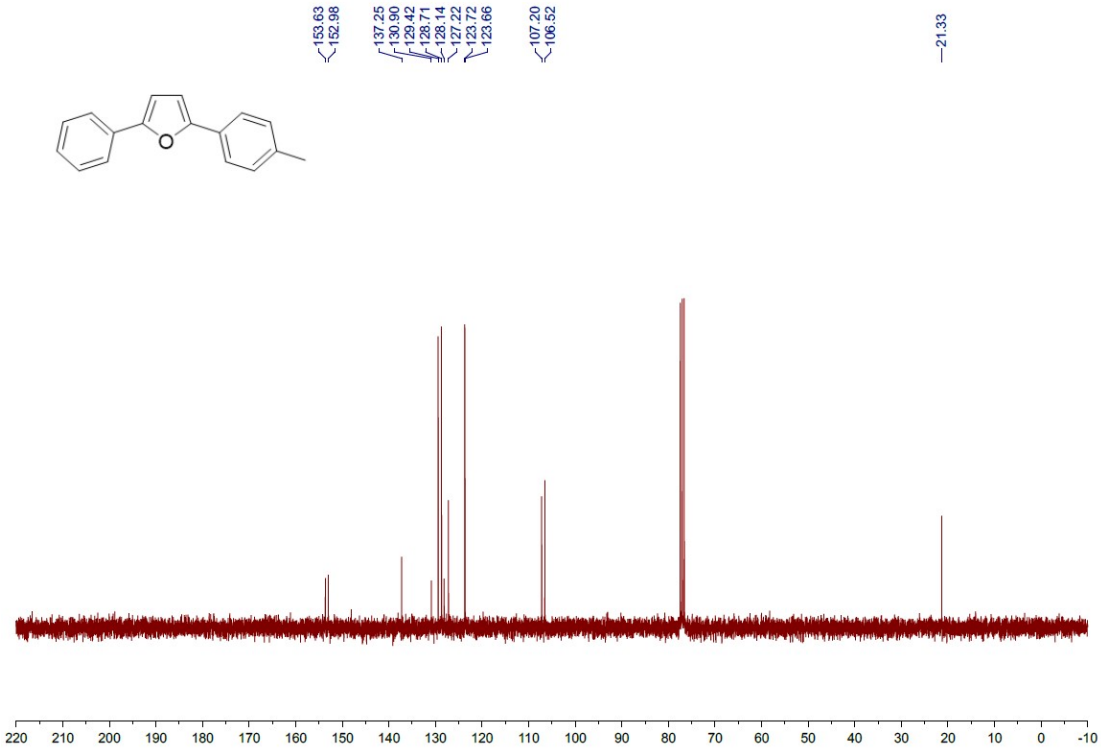
^{13}C NMR (100 MHz, $CDCl_3$) Spectrum of Compound **9**



^{1}H NMR (300 MHz, $CDCl_3$) Spectrum of Compound **10**



^{13}C NMR (75 MHz, $CDCl_3$) Spectrum of Compound **10**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130.90
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128.71
128.14
127.22
123.72
123.66
107.20
106.52
21.33
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10