Supplementary Information for

Organocatalytic SuFEx click reactions of SO_2F_2

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1. General experimental methods

^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded in $CDCl_3$ on a Bruker Avance III 400 MHz spectrometer with TMS as internal standard at room temperature, the chemical shifts (δ) were expressed in ppm and *J* values were given in Hz. High resolution mass spectra (HRMS) were obtained on a Thermo Scientific Q Exactive mass spectrometry equipped with an ESI or EI source. All commercially available chemicals were purchased from Adamas, Alfa-Aesar and Bide Pharmatech and used as received without further purification unless otherwise stated. Anhydrous THF, toluene, 1,4-dioxane, DCM, DCE and CH_3CN were distilled from sodium/benzophenone or calcium hydride prior to use. molecular sieves 4Å were calcined in a muffle furnace under air at 350°C for 5h prior to use. Column chromatography was performed on silica gel (200-300 mesh). Petroleum ether (PE), where used, has a boiling point range of 60-90°C.

2. General Procedure

2.1 General Procedure A: DBU-catalyzed synthesis of aryl fluorosulfates

To a suspension of phenol **1** (0.8 mmol) and MS 4Å (800 mg) in anhydrous CH_3CN (2.0 mL) was added DBU (0.08 mmol, 12.2 mg, 10 mol%). The mixture was stirred at room temperature for 10 min. Then the reaction tube was sealed with a septum. The atmosphere above the mixture was removed with gentle vacuum, and SO_2F_2 gas from a balloon was introduced. The reaction mixture was stirred vigorously at room temperature for 24 hours, and the progress was monitored by TLC. After completion, the reaction mixture was then diluted with ethyl acetate, filtered through a short pad of silica gel and concentrated. The crude product was purified by flash column chromatography on silica gel to afford the desired product.

2.2 General Procedure B: DBU-catalyzed synthesis of 3am

To a suspension of pyridin-4(1H)-one **4** (0.8 mmol, 76.1 mg) and MS 4Å (800 mg) in anhydrous CH_3CN (2.0 mL) was added DBU (0.08 mmol, 12.2 mg, 10 mol%). The mixture was stirred at room temperature for 10 min. Then the reaction tube was sealed

with a septum. The atmosphere above the mixture was removed with gentle vacuum, and SO_2F_2 gas from a balloon was introduced. The reaction mixture was stirred vigorously at room temperature for 24 hours, and the progress was monitored by TLC. After completion, the reaction mixture was then diluted with ethyl acetate, filtered through a short pad of silica gel and concentrated. The crude product was purified by flash column chromatography on silica gel to afford the desired product **3am** as a white solid (46.6 mg, 33% yield).

2.3 General Procedure C: DBU-catalyzed synthesis of 3an

To a suspension of 5-methyl-2-(p-tolyl)-2,4-dihydro-3H-pyrazol-3-one **5** (0.8 mmol, 150.6 mg) and MS 4Å (800 mg) in anhydrous CH_3CN (2.0 mL) was added DBU (0.08 mmol, 12.2 mg, 10 mol%). The mixture was stirred at room temperature for 10 min. Then the reaction tube was sealed with a septum. The atmosphere above the mixture was removed with gentle vacuum, and SO_2F_2 gas from a balloon was introduced. The reaction mixture was stirred vigorously at room temperature for 24 hours, and the progress was monitored by TLC. After completion, the reaction mixture was then diluted with ethyl acetate, filtered through a short pad of silica gel and concentrated. The crude product was purified by flash column chromatography on silica gel to afford the desired product **3an** as a yellow oil (155.5 mg, 72% yield).

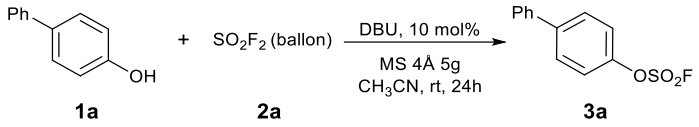
2.4 General Procedure D: DBU-catalyzed synthesis of sulfamoyl fluorides

To a suspension of amine (0.8 mmol) and MS 4Å (800 mg) in anhydrous CH_3CN (2.0 mL) was added DBU (0.08 mmol, 12.2 mg, 10 mol%). The mixture was stirred at room temperature for 10 min. Then the reaction tube was sealed with a septum. The atmosphere above the mixture was removed with gentle vacuum, and SO_2F_2 gas from a balloon was introduced. The reaction mixture was stirred vigorously at room temperature for 24 hours, and the progress was monitored by TLC. After completion, the reaction mixture was then diluted with ethyl acetate, filtered through a short pad of silica gel and concentrated. The crude product was purified by flash column chromatography on silica gel to afford the desired product.

3. Gram-Scale Experiments

3.1 Gram-scale synthesis of 3a

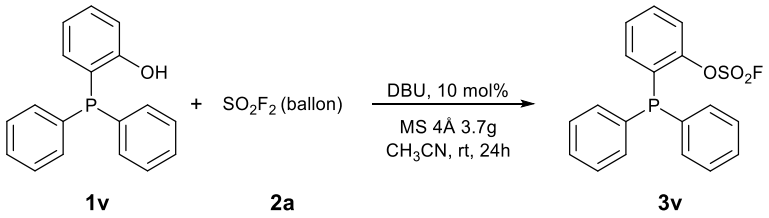
Scheme S1. Gram-scale synthesis of **3a**



To a suspension of Phenol **1a** (5 mmol) and MS 4Å (5 g) in anhydrous CH_3CN (12.5 mL) was added DBU (0.5 mmol, 76.1 mg, 10 mol%). The mixture was stirred at room temperature for 10 min. Then the reaction tube was sealed with a septum. The atmosphere above the mixture was removed with gentle vacuum, and SO_2F_2 gas from a balloon was introduced. For large scale reactions, depletion of the sulfuryl fluoride from the balloon was easily observed, and more SO_2F_2 gas was introduced with a fresh balloon when required. The reaction mixture was stirred vigorously at room temperature for 24 hours, and the progress was monitored by TLC. After completion, the reaction mixture was then diluted with ethyl acetate, filtered through a sand core funnel and concentrated. The crude product was purified by flash column chromatography on silica gel to afford **3a** as a white solid (1.235 g, 98% yield).

3.2 Gram-scale synthesis of 3v

Scheme S2. Gram-scale synthesis of **3v**

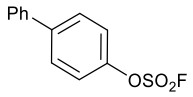


To a suspension of Phenol **1v** (3.7 mmol) and MS 4Å (3.7 g) in anhydrous CH_3CN (9.3 mL) was added DBU (0.37 mmol, 56.3 mg, 10 mol%). The mixture was stirred at room temperature for 10 min. Then the reaction tube was sealed with a septum. The atmosphere above the mixture was removed with gentle vacuum, and SO_2F_2 gas from a balloon was introduced. For large scale reactions, depletion of the sulfuryl fluoride from the balloon was easily observed, and more SO_2F_2 gas was introduced with a fresh balloon when required. The reaction mixture was stirred vigorously at room temperature for 24 hours, and the progress was monitored by TLC. After completion,

the reaction mixture was then diluted with ethyl acetate, filtered through a sand core funnel and concentrated. The crude product was purified by flash column chromatography on silica gel to afford **3v** as a white solid (0.857 g, 65% yield).

4. Spectroscopic data for all products

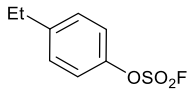
*[1,1'-biphenyl]-4-yl sulfurofluoridate (**3a**)*[1]



3a

Compound **3a** was prepared according to the general **A**. White solid (196.5 mg, 97% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68-7.63 (m, 2H), 7.57-7.52 (m, 2H), 7.49-7.43 (m, 2H), 7.42-7.37 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.4, 142.0, 139.3, 129.03, 129.01, 128.1, 127.2, 121.2.

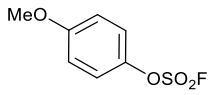
*4-ethylphenyl sulfurofluoridate (**3b**)*[2]



3b

Compound **3b** was prepared according to the general **A**. Yellow oil (138.8 mg, 85% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 (q, J = 8.7 Hz, 4H), 2.68 (q, J = 7.6 Hz, 2H), 1.25 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.2, 145.0, 129.7, 120.6, 28.3, 15.4.

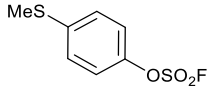
*4-methoxyphenyl sulfurofluoridate (**3c**)*[3]



3c

Compound **3c** was prepared according to the general **A**. Yellow oil (119.6 mg, 73% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.21 (m, 2H), 6.95-6.90 (m, 2H), 3.80 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.3, 143.6, 121.9, 115.1, 55.7.

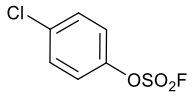
*4-(methylthio)phenyl sulfurofluoridate (**3d**)*[3]



3d

Compound **3d** was prepared according to the general **A**. Yellow oil (168.9 mg, 95% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30-7.26 (m, 2H), 7.26-7.22 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.4, 140.2, 127.6, 121.3, 15.7.

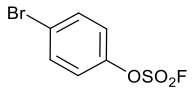
*4-chlorophenyl sulfurofluoridate (**3e**)*[4]



3e

Compound **3e** was prepared according to the general **A**. Colorless oil (134.8 mg, 80% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46-7.41 (m, 2H), 7.31-7.26 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.3, 134.6, 130.5, 122.4.

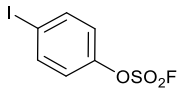
*4-bromophenyl sulfurofluoridate (**3f**)*[4]



3f

Compound **3f** was prepared according to the general **A**. Colorless oil (157.1 mg, 77% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63-7.58 (m, 2H), 7.26-7.21 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.9, 133.6, 122.7, 122.4…

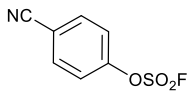
*4-iodophenyl sulfurofluoridate (**3g**)*[5]



3g

Compound **3g** was prepared according to the general **A**. Yellow oil (223.0 mg, 92% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81-7.76 (m, 2H), 7.12-7.07 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.8, 139.6, 122.9, 93.6.

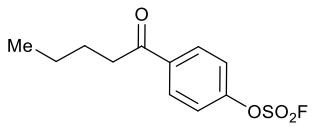
*4-cyanophenyl sulfurofluoridate (**3h**)*[6]



3h

Compound **3h** was prepared according to the general **A**. White solid (146.5 mg, 91% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86-7.81 (m, 2H), 7.53-7.48 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.3, 134.7, 122.2, 117.0, 113.2.

*4-pentanoylphenyl sulfurofluoridate (**3i**)*

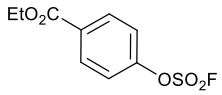


3i

Compound **3i** was prepared according to the general **A**. Red oil (206.1 mg, 99% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11-8.06 (m, 2H), 7.47-7.42 (m, 2H), 2.98 (t, J = 7.44

Hz, 2H), 1.77-1.69 (m, 2H), 1.47-1.37 (m, 2H), 0.96 (t, J = 7.28 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.5, 152.6, 137.1, 130.4, 121.1, 38.5, 26.2, 22.4, 13.9; IR (KBr, thin film): 2960, 1690, 1594, 1497, 1456, 1410, 1236, 1145, 1014, 915, 851, 818 cm^{-1}; HRMS (ESI) *m/z*: $[M+H]^+$ Calcd. for $C_{11}H_{14}FO_4S$, 261.0591, Found 261.0592.

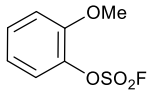
*ethyl 4-((fluorosulfonyl)oxy)benzoate (**3j**)*[6]



3j

Compound **3j** was prepared according to the general **A**. Colorless oil (192.0 mg, 97% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20-8.15 (m, 2H), 7.44-7.39 (m, 2H), 4.41 (q, J = 7.12 Hz, 2H), 1.41 (t, J = 7.12 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.8, 152.7, 132.0, 131.0, 120.9, 61.6, 14.2.

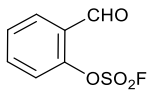
*2-methoxyphenyl sulfurofluoridate (**3k**)*[6]



3k

Compound **3k** was prepared according to the general **A**. Yellow oil (121.0 mg, 73% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.28 (m, 2H), 7.06-7.03 (m, 1H), 7.01-6.95 (m, 1H), 3.90 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.2, 139.0, 129.6, 122.4, 120.9, 113.5, 56.2.

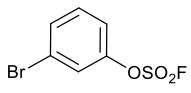
*2-formylphenyl sulfurofluoridate (**3l**)*[6]



3l

Compound **3l** was prepared according to the general **A**. Yellow oil (119.7 mg, 73% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.31 (s, 1H), 8.05-8.01 (m, 1H), 7.79-7.74 (m, 1H), 7.63-7.58 (m, 1H), 7.50 (d, J = 8.32 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.3, 150.5, 136.0, 130.9, 129.3, 128.1, 122.1.

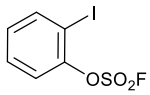
*3-bromophenyl sulfurofluoridate (**3m**)*[7]



3m

Compound **3m** was prepared according to the general **A**. Colorless oil (161.2 mg, 79% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59-7.52 (m, 2H), 7.39-7.29 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.0, 132.1, 131.4, 124.5, 123.2, 119.7.

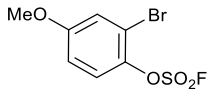
*2-iodophenyl sulfurofluoridate (**3n**)*[5]



3n

Compound **3n** was prepared according to the general **A**. Yellow oil (226.6 mg, 94% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (dd, *J* = 7.96, 1.36 Hz, 1H), 7.47-7.38 (m, 2H), 7.15-7.10 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.4, 140.8, 130.3, 130.1, 121.7, 88.4.

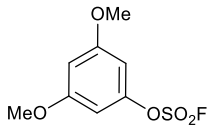
*2-bromo-4-methoxyphenyl sulfurofluoridate (**3o**)*[8]



3o

Compound **3o** was prepared according to the general **A**. Yellow oil (218.3 mg, 96% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (dd, *J* = 9.12, 1.16 Hz, 1H), 7.17 (d, *J* = 2.96, Hz, 1H), 6.89 (dd, *J* = 9.12, 3.0 Hz, 1H), 3.81 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.6, 140.9, 123.0, 119.2, 115.9, 114.5, 56.0.

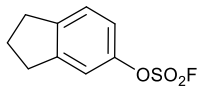
*3,5-dimethoxyphenyl sulfurofluoridate (**3p**)*



3p

Compound **3p** was prepared according to the general **A**. White solid (177.4 mg, 94% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.49-6.46 (m, 3H), 3.81 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.5, 151.2, 100.5, 99.4, 55.8; IR (KBr, thin film): 2986, 1739, 1626, 1592, 1451, 1374, 1233, 1159, 1110, 1047, 972, 738 cm^{-1}; mp: 38.6-39.1°C; HRMS (ESI) *m/z*: $[M+H]^+$ Calcd. for $C_8H_{10}FO_5S$, 237.0228, Found 237.0230.

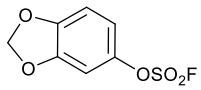
*2,3-dihydro-1H-inden-5-yl sulfurofluoridate (**3q**)*



3q

Compound **3q** was prepared according to the general **A**. Colorless oil (165.4 mg, 96% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 (d, *J* = 8.2 Hz, 1H), 7.16 (s, 1H), 7.07 (d, *J* = 8.2 Hz, 1H), 2.96-2.88 (m, 4H), 2.17-2.08 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.9, 147.0, 144.9, 125.6, 118.4, 116.8, 33.0, 32.3, 25.8; IR (KBr, thin film): 2983, 2360, 2340, 1558, 1501, 1447, 1234, 1111, 943, 891, 800, 675 cm^{-1}; HRMS (ESI) *m/z*: $[M+H]^+$ Calcd. for $C_9H_{10}FO_3S$, 217.0329, Found 217.0329.

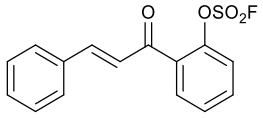
*benzo[d][1,3]dioxol-5-yl sulfurofluoridate (**3r**)*[1]



3r

Compound **3r** was prepared according to the general **A**. Yellow oil (137.0 mg, 78% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.83-6.80 (m, 3H), 6.05 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.6, 147.7, 144.1, 114.0, 108.3, 103.0, 102.5.

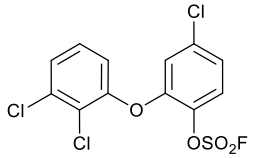
*2-cinnamoylphenyl sulfurofluoridate (**3s**)*



3s

Compound **3s** was prepared according to the general **A**. Yellow oil (150.7 mg, 61% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (dd, J = 7.6, 1.72 Hz, 1H), 7.68-7.58 (m, 4H), 7.56-7.51 (m, 1H), 7.49-7.46 (m, 1H), 7.46-7.38 (m, 3H), 7.23-7.18 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 189.5, 147.3, 147.0, 134.2, 133.1, 132.7, 131.2, 130.9, 129.1, 128.9, 128.7, 124.7, 122.1; IR (KBr, thin film): 2922, 1652, 1608, 1575, 1448, 1332, 1232, 1156, 1113, 1060, 918, 816, 773 cm^{-1}; HRMS (ESI) *m/z*: $[M+H]^+$ Calcd. for $C_{15}H_{12}FO_4S$, 307.0435, Found 307.0432.

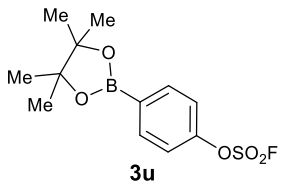
*4-chloro-2-(2,3-dichlorophenoxy)phenyl sulfurofluoridate (**3t**)*



3t

Compound **3t** was prepared according to the general **A**. Colorless oil (280.4 mg, 94% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (d, J = 2.48 Hz, 1H), 7.47-7.45 (m, 1H), 7.30-7.25 (m, 2H), 7.02 (d, J = 8.72 Hz, 1H), 6.73 (d, J = 8.88 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.2, 147.4, 139.2, 131.4, 131.0, 129.8, 128.64, 128.62, 127.4, 123.6, 122.4, 118.7; IR (KBr, thin film): 3096, 1735, 1581, 1472, 1458, 1273, 1234, 1168, 1119, 1099, 938, 878 cm^{-1}; HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd. for $C_{12}H_6Cl_3FO_4SNa$, 392.8929, Found 392.8927.

*4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl sulfurofluoridate (**3u**)*

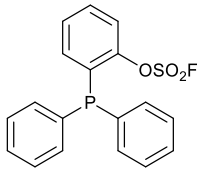


3u

Compound **3u** was prepared according to the general **A**. Colorless oil (172.3 mg, 71%

yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94-7.89 (m, 2H), 7.35-7.31 (m, 2H), 1.35 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.2, 137.0, 120.1, 84.4, 24.9; IR (KBr, thin film): 2982, 1601, 1489, 1455, 1401, 1363, 1235, 1173, 1141, 918, 857, 809 cm^{-1}; HRMS (ESI) *m/z*: $[M+H]^+$ Calcd. For $C_{12}H_{17}BFO_5S$, 303.0868, Found 303.0868.

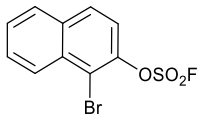
*2-(diphenylphosphaneyl)phenyl sulfurofluoridate (**3v**)*[9]



3v

Compound **3v** was prepared according to the general **A**. White solid (184.5 mg, 64% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48-7.43 (m, 1H), 7.42-7.27 (m, 12H) 6.99-6.94 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.8 (d, *J* = 19.9 HZ), 135.2, 134.5 (d, *J* = 10.4 HZ), 134.0 (d, *J* = 20.7 HZ), 131.8 (d, *J* = 21.3 HZ), 130.9, 129.4 128.8 (d, *J* = 7.25 HZ), 128.6, 120.4.

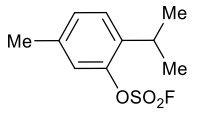
*1-bromonaphthalen-2-yl sulfurofluoridate (**3w**)*[8]



3w

Compound **3w** was prepared according to the general **A**. White solid (196.5 mg, 81% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.35-8.31 (m, 1H), 7.95-7.88 (m, 2H), 7.73-7.68 (m, 1H), 7.66-7.60 (m, 1H), 7.54-7.50 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.2, 133.2, 132.7, 129.9, 128.9, 128.4, 127.9, 127.7, 119.4, 115.7.

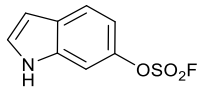
*2-isopropyl-5-methylphenyl sulfurofluoridate (**3x**)*[1]



3x

Compound **3x** was prepared according to the general **A**. Colorless oil (158.7 mg, 85% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 (d, *J* = 7.96 Hz, 1H), 7.16 (d, *J* = 8.08 Hz, 1H), 7.11 (s, 1H), 3.31-3.19 (m, 1H), 2.35 (s, 3H), 1.24 (d, *J* = 6.92 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.8, 137.8, 137.5, 129.7, 127.6, 121.0, 26.7, 23.1, 20.8.

*1H-indol-6-yl sulfurofluoridate (**3y**)*

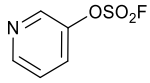


3y

Compound **3y** was prepared according to the general **A**. Yellow oil (121.1 mg, 70%

yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.40 (s, 1H), 7.40 (d, *J* = 8.08 Hz, 1H), 7.27-7.25 (m, 1H), 7.19 (t, *J* = 7.96 Hz, 1H), 7.13-7.10 (m, 1H), 6.66-6.64 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.9, 138.2, 125.9, 122.2, 120.6, 111.8, 111.7, 99.1; IR (KBr, thin film): 3448, 1444, 1343, 1233, 1188, 1153, 1092, 1000, 896, 831, 742 cm^{-1}; HRMS (ESI) *m/z*: $[M+H]^+$ Calcd. for $C_8H_7FNO_3S$, 216.0125, Found 216.0126.

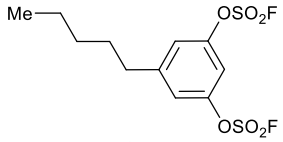
*pyridin-4-yl sulfurofluoridate (**3z**)*[3]



3z

Compound **3z** was prepared according to the general **A**. Yellow oil (91.3 mg, 64% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.72-8.68 (m,, 2H), 7.75-7.71 (m, 1H), 7.50-7.45 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.8, 147.1, 142.7, 128.7, 124.8.

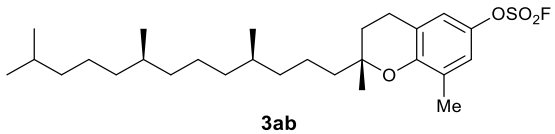
*5-pentyl-1,3-phenylene bis(sulfurofluoridate) (**3aa**)*



3aa

Compound **3aa** was prepared according to the general **A**. Yellow oil (234.2 mg, 85% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.25 (m, 2H), 7.23-7.20 (m, 1H), 2.75-2.7 (m, 2H), 1.70-1.62 (m, 2H), 1.42--1.29 (m, 4H), 0.93-0.89 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.8, 148.7,121.2, 111.9, 35.6, 31.2, 30.3, 22.3, 13.9; IR (KBr, thin film): 1868, 1749, 1720, 1701, 1688, 1653, 1558, 1540, 1522, 1511, 1457, 1396 cm^{-1}; HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd. for $C_{11}H_{14}F_2O_6S_2Na$, 367.0092, Found 367.0092.

*(R)-2,8-dimethyl-2-((4R,8R)-4,8,12-trimethyltridecyl)chroman-6-yl sulfurofluoridate (**3ab**)*

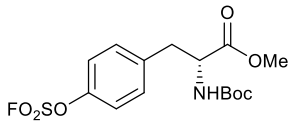


3ab

Compound **3ab** was prepared according to the general **A**. Yellow oil (160.0 mg, 83% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.89 (dd, *J* = 19.08, 2.48 Hz, 2H), 2.79-2.73 (m, 2H), 2.17 (s, 3H), 1.86-1.71 (m, 2H), 1.60-1.50 (m, 3H), 1.46-1.35 (m, 4H), 1.31-1.19 (m, 10H), 1.18-1.00 (m, 7H), 0.88-0.83 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.9, 142.1, 128.5, 121.8, 120.3, 118.6, 76.9, 40.1, 39.4, 37.5, 37.41, 37.38, 37.3, 32.8, 32.7, 30.7, 28.0, 24.8, 24.5, 24.2, 22.7, 22.6, 22.5, 20.9, 19.8, 19.7, 16.2; IR (KBr, thin film): 2926, 1761, 1448, 1378, 1238, 1220, 1048, 985, 923, 885, 802 cm^{-1}; HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd. for $C_{27}H_{46}FO_4SNa$, 507.2915, Found 507.2917.

Methyl (R)-2-((tert-butoxycarbonyl)amino)-3-(4-((fluorosulfonyl)oxy)phe-

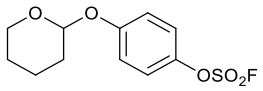
*nyl)propanoate (**3ac**)*[10]



3ac

Compound **3ac** was prepared according to the general **A**. White solid (276.0 mg, 91% yield); ^{1}H NMR (400 MHz, Chloroform-d) δ 7.31 – 7.22 (m, 4H), 5.04 (d, *J* = 7.4 Hz, 1H), 4.61 (q, *J* = 6.3 Hz, 1H), 3.73 (s, 3H), 3.12 (ddd, *J* = 57.9, 13.8, 6.0 Hz, 2H), 1.41 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.9, 154.9, 149.1, 137.2, 131.3, 120.9, 80.2, 54.2, 52.4, 37.9, 28.2.

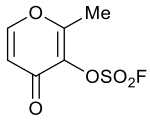
*4-((tetrahydro-2H-pyran-2-yl)oxy)phenyl sulfurofluoridate (**3ad**)*



3ad

Compound **3ad** was prepared according to the general **A**. White solid (205.9 mg, 93% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.21 (m, 2H), 7.13-7.08 (m, 2H), 5.41 (t, *J* = 3.08 Hz, 1H), 3.89-3.82 (m, 1H), 3.64-3.58 (m, 1H), 2.05-1.94 (m, 1H), 1.90-1.84 (m, 2H), 1.76-1.56 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 156.9, 144.1, 121.8, 117.7, 96.6, 62.0, 30.2, 25.1, 18.5; IR (KBr, thin film): 2947, 1596, 1499, 1448, 1233, 1169, 1143, 1037, 960, 916, 838, 799 cm^{-1}; mp: 48.6-48.9 °C; HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd. for $C_{11}H_{13}FO_5SNa$, 299.0360, Found 299.0360.

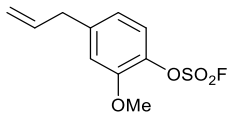
*2-methyl-4-oxo-4H-pyran-3-yl sulfurofluoridate (**3ae**)*



3ae

Compound **3ae** was prepared according to the general **A**. Yellow oil (141.9 mg, 85% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 5.8 Hz, 1H), 6.50 (d, *J* = 5.8 Hz, 1H), 2.47 (d, *J* = 0.56 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.0, 162.0, 154.7, 139.3, 117.9, 15.5; IR (KBr, thin film): 3086, 1669, 1456, 1417, 1363, 1226, 1139, 929, 893, 817, 792, 693 cm^{-1}; HRMS (ESI) *m/z*: $[M+H]^+$ Calcd. for $C_6H_6FO_5S$, 208.9915, Found 208.9917.

*4-allyl-2-methoxyphenyl sulfurofluoridate (**3af**)*[1]

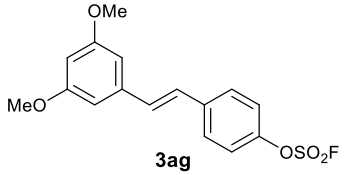


3af

Compound **3af** was prepared according to the general **A**. Yellow oil (159.5 mg, 81% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 (dd, *J* = 8.28, 1.12 Hz, 1H), 6.86 (d, *J* = 1.8 Hz, 1H), 6.80 (dd, *J* = 8.28, 1.96 Hz, 1H), 5.99-5.88 (m, 1H), 5.14 (t, J=1.32 Hz, 1H),

5.12-5.08 (m, 1H), 3.88 (s, 3H), 3.39 (d, J = 6.72 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.0, 142.2, 137.4, 136.2, 122.1, 120.8, 116.8, 113.6, 56.1, 40.0.

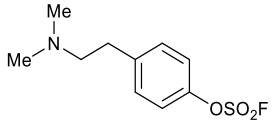
*(E)-4-(3,5-dimethoxystyryl)phenyl sulfurofluoridate (**3ag**)*[3]



3ag

Compound **3ag** was prepared according to the general **A**. White solid (238.7 mg, 88% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60-7.55 (m, 2H), 7.34-7.30 (m, 2H), 7.05 (d, J = 1.0 Hz, 2H), 6.67 (d, J = 2.24 Hz, 2H), 6.43 (t, J = 2.24 Hz, 1H), 3.83 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.1, 149.1, 138.6, 137.9, 131.0, 128.2, 127.0, 121.2, 104.8, 100.5, 55.4.

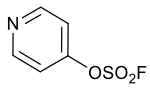
*4-(2-(dimethylamino)ethyl)phenyl sulfurofluoridate (**3ah**)*



3ah

Compound **3ah** was prepared according to the general **A**. Yellow oil (105.0 mg, 53% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32-7.28 (m, 2H), 7.27-7.23 (m, 2H), 2.84-2.78 (m, 2H), 2.56-2.51 (m, 2H), 2.29 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.4, 141.4, 130.5, 120.7, 61.0, 45.4, 33.6; IR (KBr, thin film): 2953, 1505, 1447, 1282, 1233, 1136, 1077, 919, 738 cm^{-1}; HRMS (ESI) *m/z*: $[M+H]^+$ Calcd. for $C_{10}H_{15}FNO_3S$, 248.0751, Found 248.0753.

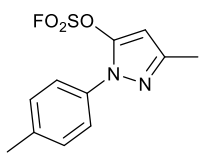
*pyridin-4-yl sulfurofluoridate (**3am**)*



3am

Compound **3am** was prepared according to the general **B**. White solid (46.6 mg, 33% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78-7.73 (m, 2H), 6.40-6.36 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.2, 134.0, 120.4; IR (KBr, thin film): 3057, 1667, 1572, 1475, 1337, 1232, 1202, 1076, 987, 869, 783 cm^{-1}; mp: 116.2-116.5 °C; HRMS (ESI) *m/z*: $[M+H]^+$ Calcd. for $C_5H_5FNO_3S$, 177.9969, Found 177.9969.

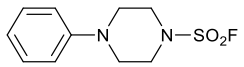
*3-methyl-1-phenyl-1H-pyrazol-5-yl sulfurofluoridate (**3an**)*



3an

Compound **3an** was prepared according to the general **C**. Yellow oil (155.5 mg, 72% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41-7.37 (m, 2H), 7.29-7.25 (m, 2H), 6.19 (s, 1H), 2.40 (s, 3H), 2.33 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 149.0, 141.1, 138.5, 134.2, 130.0, 123.4, 95.8, 21.1, 14.5; IR (KBr, thin film): 2929, 1558, 1518, 1463, 1235, 1137, 1109, 1036, 1017, 885, 820, 749 cm^{-1}; HRMS (ESI) *m/z*: $[M+H]^+$ Calcd. for $C_{11}H_{12}FN_2O_3S$, 271.0547, Found 271.0548.

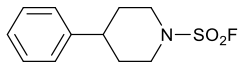
*4-phenylpiperazine-1-sulfonyl fluoride (7**a**)*[11]



7a

Compound **7a** was prepared according to the general **D**. White solid (148.0 mg, 76% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.30 (m, 2H), 7.05-6.98 (m, 3H), 3.70-3.65 (m, 4H), 3.35-3.29 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 150.4, 129.4, 121.4, 117.2, 48.9, 47.0.

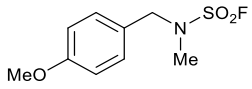
*4-phenylpiperidine-1-sulfonyl fluoride (7**b**)*[11]



7b

Compound **7b** was prepared according to the general **D**. White solid (157.8 mg, 81% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.16 (m, 5H), 4.06-3.97 (m, 2H), 3.14-3.03 (m, 2H), 2.70-2.60 (m, 1H), 1.96-1.75 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.3, 128.8, 126.9, 126.7, 47.9, 41.4, 32.0.

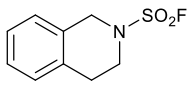
*(4-methoxybenzyl)(methyl)sulfamoyl fluoride (7**c**)*



7c

Compound **7c** was prepared according to the general **D**. Colorless oil (122.5 mg, 58% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.23 (m, 2H), 6.93-6.89 (m, 2H), 4.41 (s, 2H), 3.82 (s, 3H), 2.87 (d, *J* = 2.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.9, 130.1, 125.6, 114.3, 55.3, 54.6, 34.9; IR (KBr, thin film): 2939, 1612, 1514, 1457, 1408, 1033, 1295, 988, 923, 823, 781, 729 cm^{-1}; HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd. for $C_9H_{12}FNO_3SNa$, 256.0414, Found 256.0414.

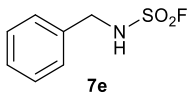
*3,4-dihydroisoquinoline-2(1H)-sulfonyl fluoride (7**d**)*[11]



7d

Compound **7d** was prepared according to the general **D**. Colorless oil (133.9 mg, 68% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28-7.04 (m, 4H), 4.60 (s, 2H), 3.71 (td, *J* = 6.0, 1.8 Hz, 2H), 2.98 (t, *J* = 6.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 132.5, 130.3, 129.1, 127.4, 126.9, 126.2, 48.2, 45.0, 28.0.

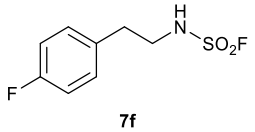
*benzylsulfamoyl fluoride (**7e**)*[12]



7e

Compound **7e** was prepared according to the general **D**. Colorless oil (68.1 mg, 45% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39-7.29 (m, 5H), 5.34 (s, 1H), 4.39 (dd, *J* = 5.76, 1.32 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.8, 129.1, 128.8, 128.1, 48.4.

*(4-fluorophenethyl)sulfamoyl fluoride (**7f**)*



7f

Compound **7f** was prepared according to the general **D**. Colorless oil (31.9 mg, 18% yield);^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 – 7.14 (m, 2H), 7.06 – 6.99 (m, 2H), 5.05 (s, 1H), 3.56-3.50 (m, 2H), 2.90 (t, *J* = 6.96 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.0 (d, *J* = 244.2 Hz), 132.4 (d, *J* = 3.2 Hz), 130.3 (d, *J* = 7.9 Hz), 115.9 (d, *J* = 21.2 Hz), 45.6, 34.7. IR (KBr, thin film): 2923, 1490, 1409, 1250, 1209, 1138, 1064, 1009, 950, 832, 754, 709 cm^{-1}; HRMS (ESI) *m/z*: $[M+Na]^+$ Calcd. for $C_8H_9F_2NO_2SNa$, 244.0214, Found 244.0214.

5. References

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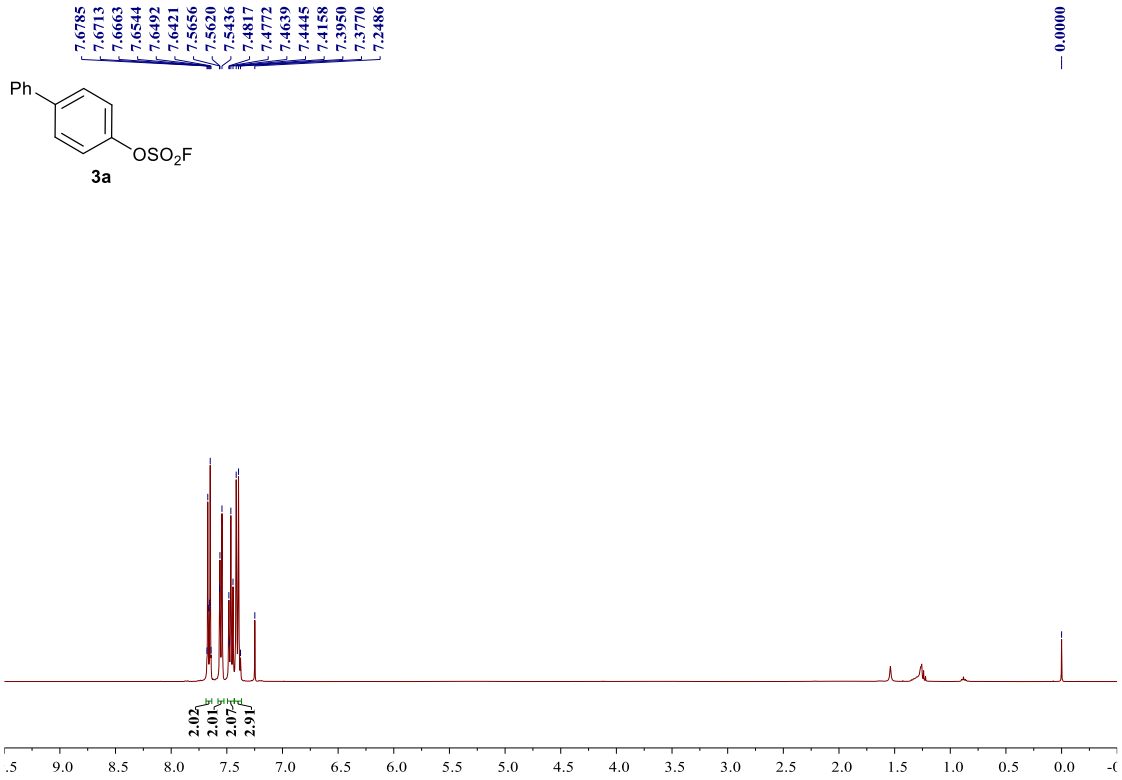
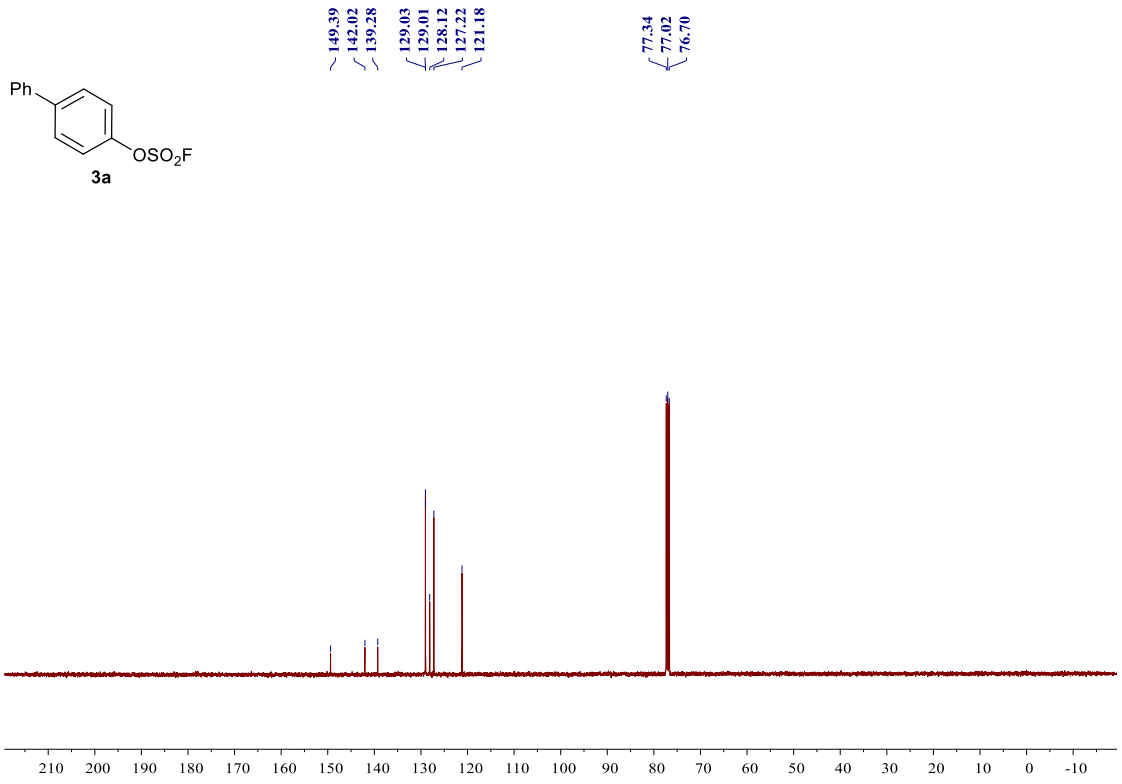
6. ^{1}H and ^{13}C NMR Spectra for all products

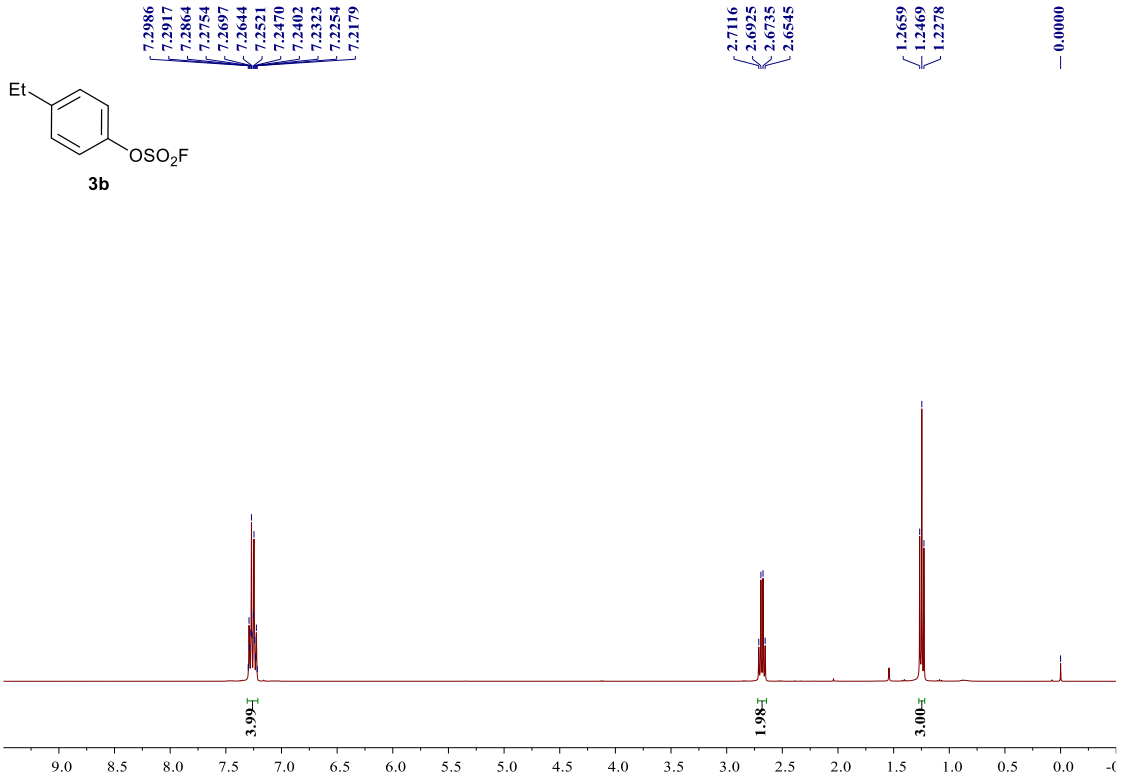
^{1}H NMR Spectrum for Compound **3a** (400 MHz, $CDCl_3$)



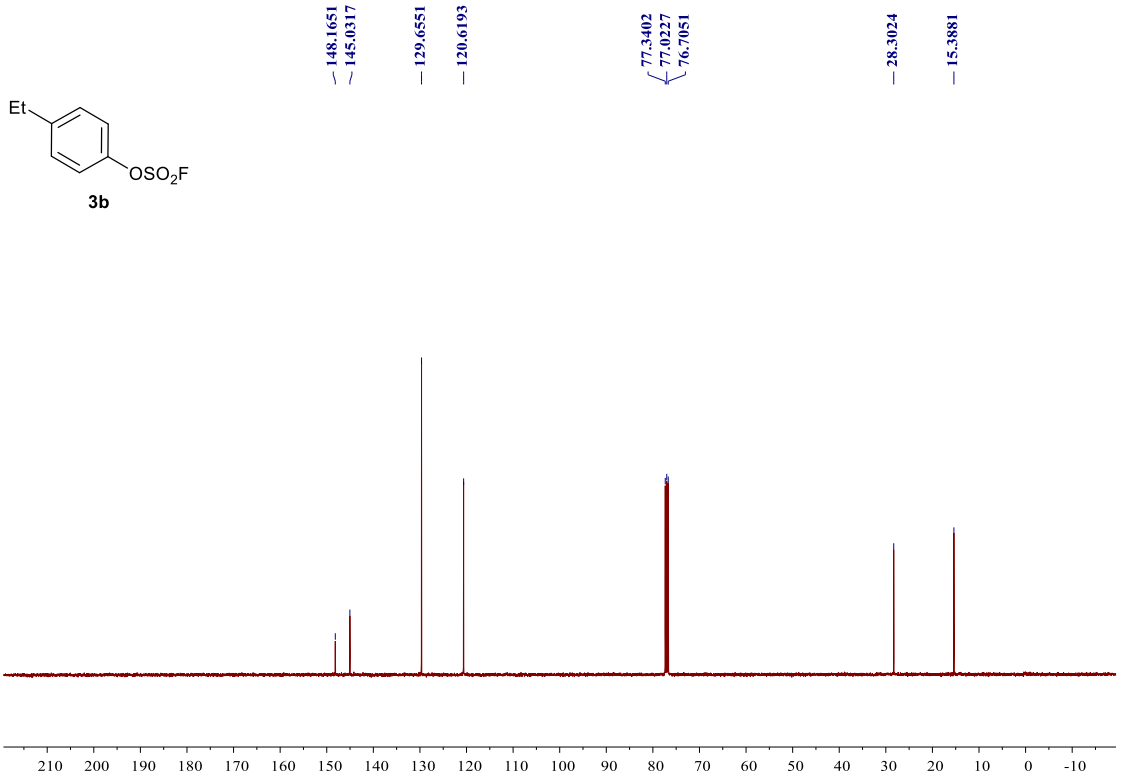
^{13}C NMR Spectrum for Compound **3a** (100 MHz, $CDCl_3$)



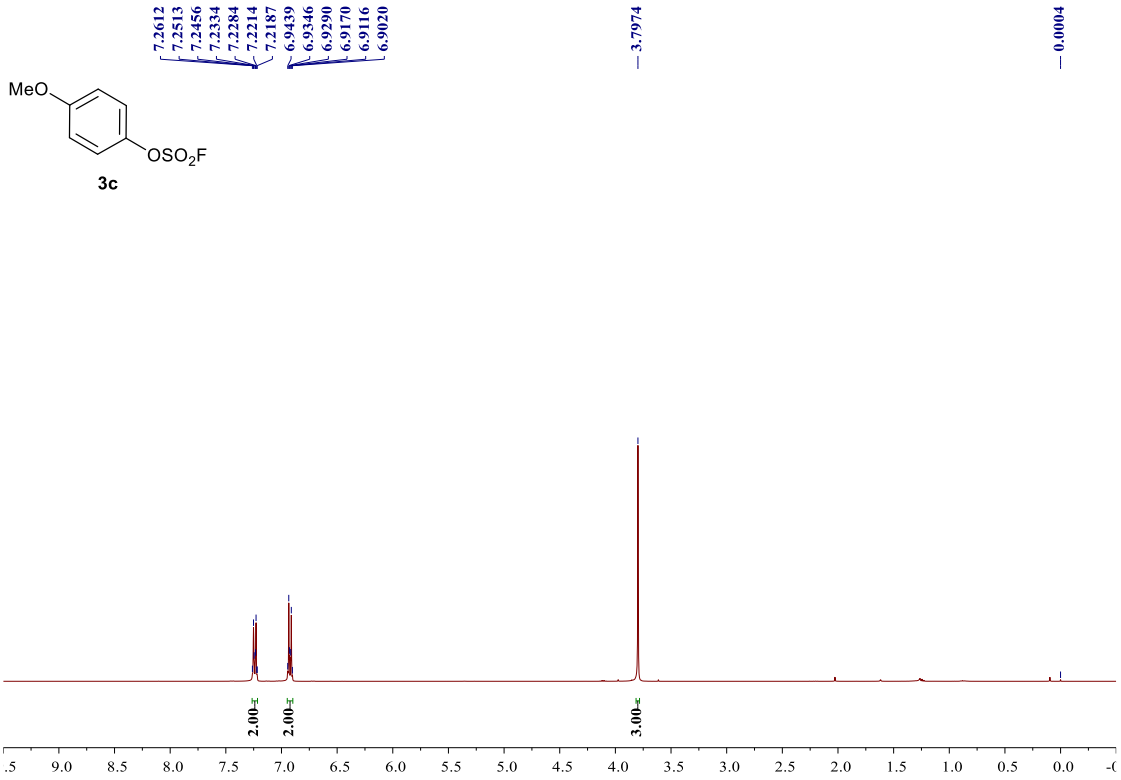
^{1}H NMR Spectrum for Compound **3b** (400 MHz, $CDCl_3$)



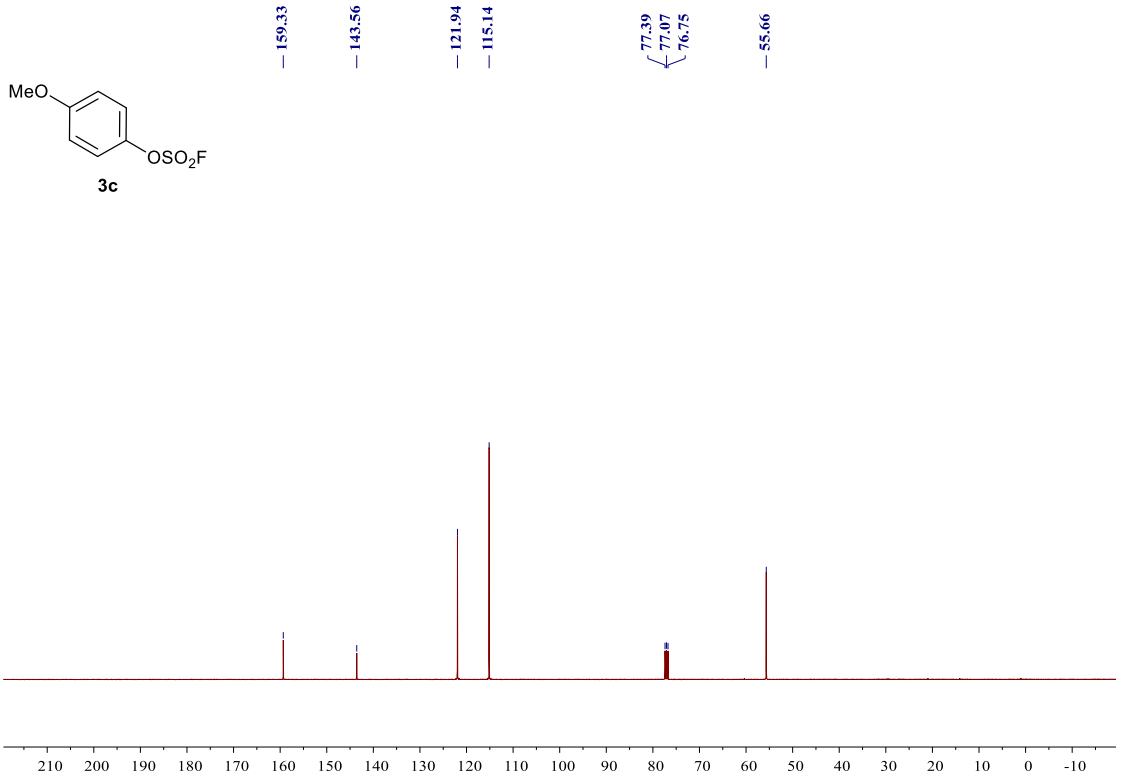
^{13}C NMR Spectrum for Compound **3b** (100 MHz, $CDCl_3$)



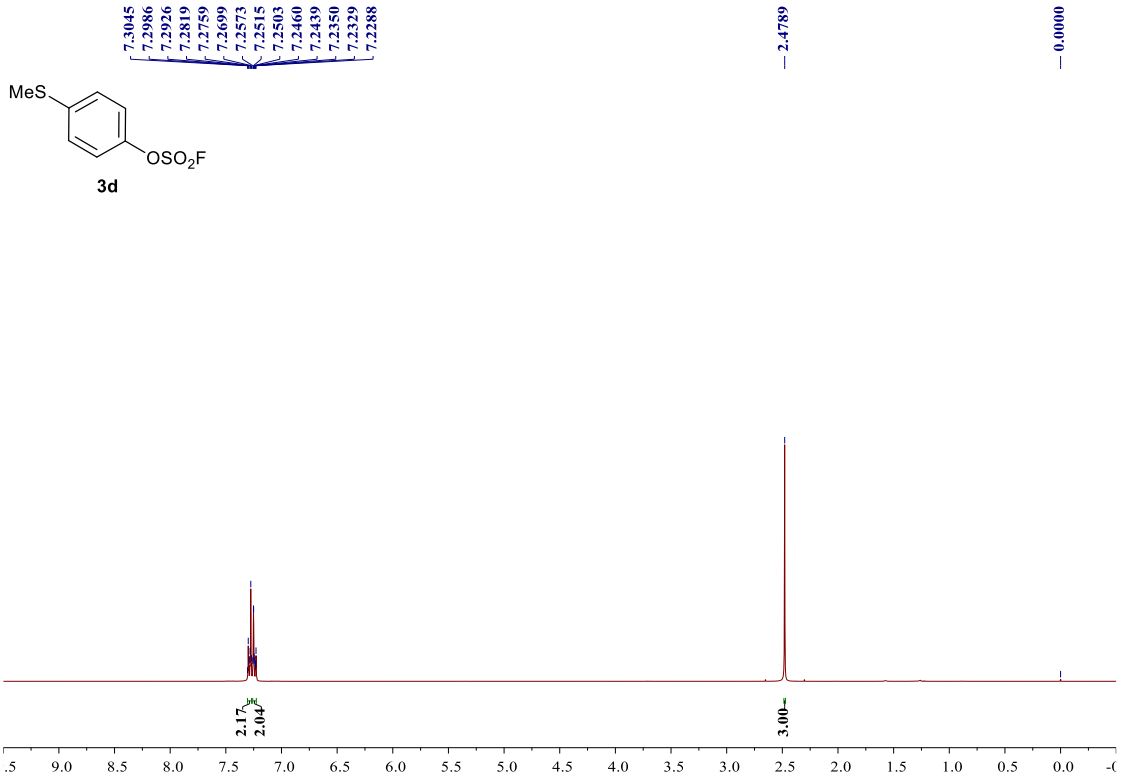
^{1}H NMR Spectrum for Compound **3c** (400 MHz, $CDCl_3$)



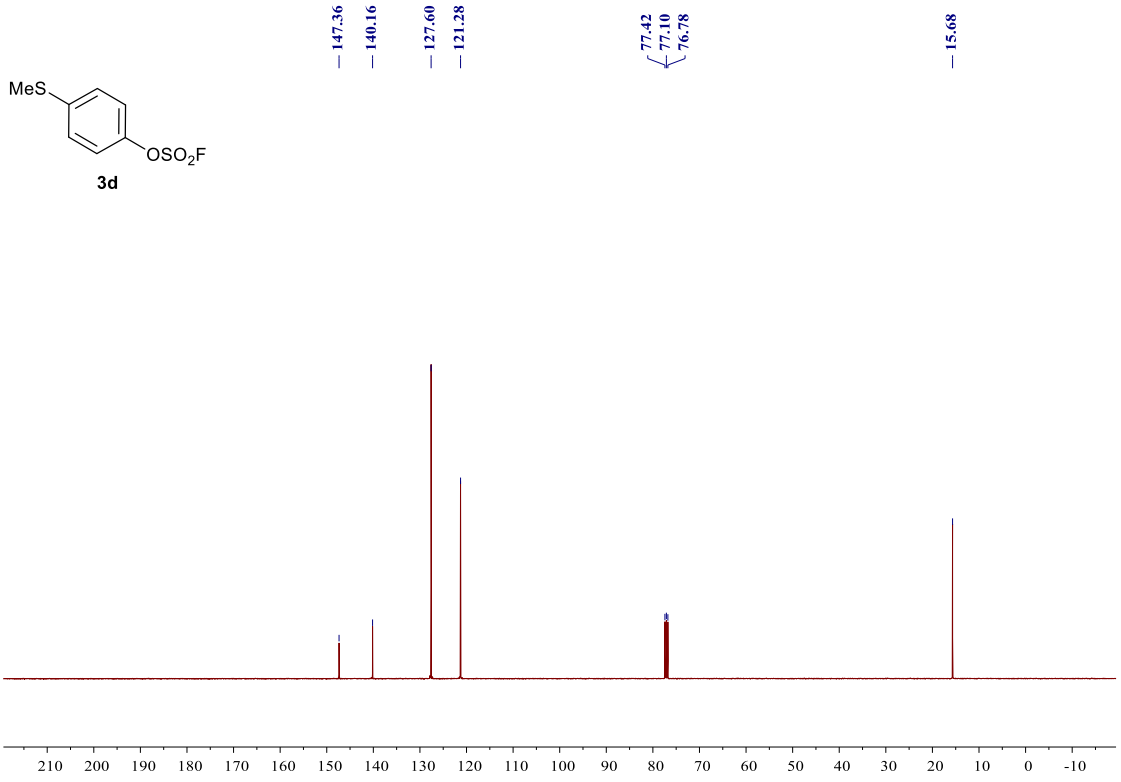
^{13}C NMR Spectrum for Compound **3c** (100 MHz, $CDCl_3$)



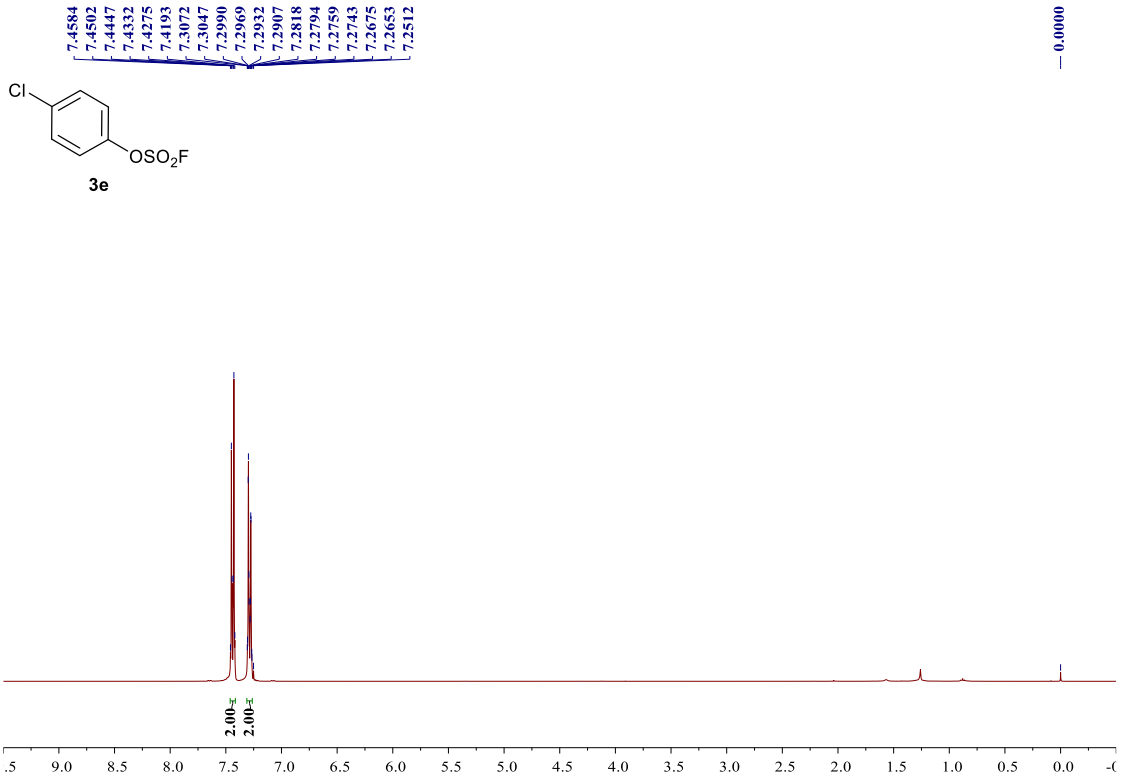
^{1}H NMR Spectrum for Compound **3d** (400 MHz, $CDCl_3$)



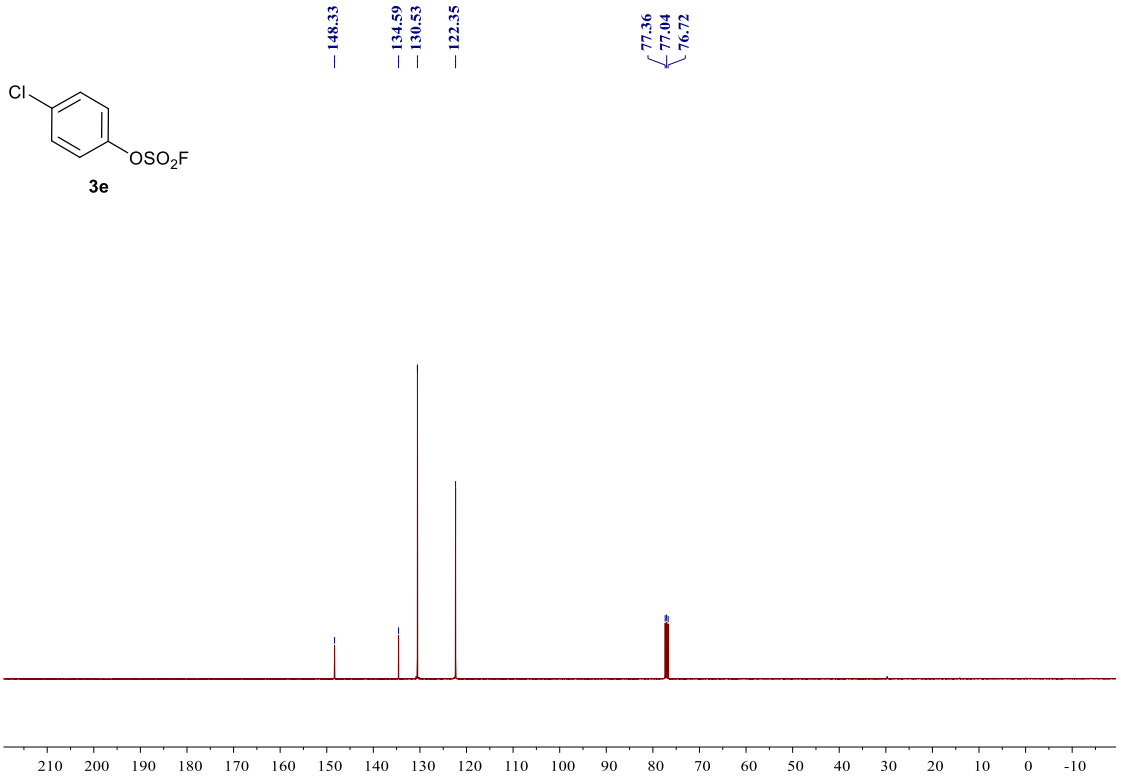
^{13}C NMR Spectrum for Compound **3d** (100 MHz, $CDCl_3$



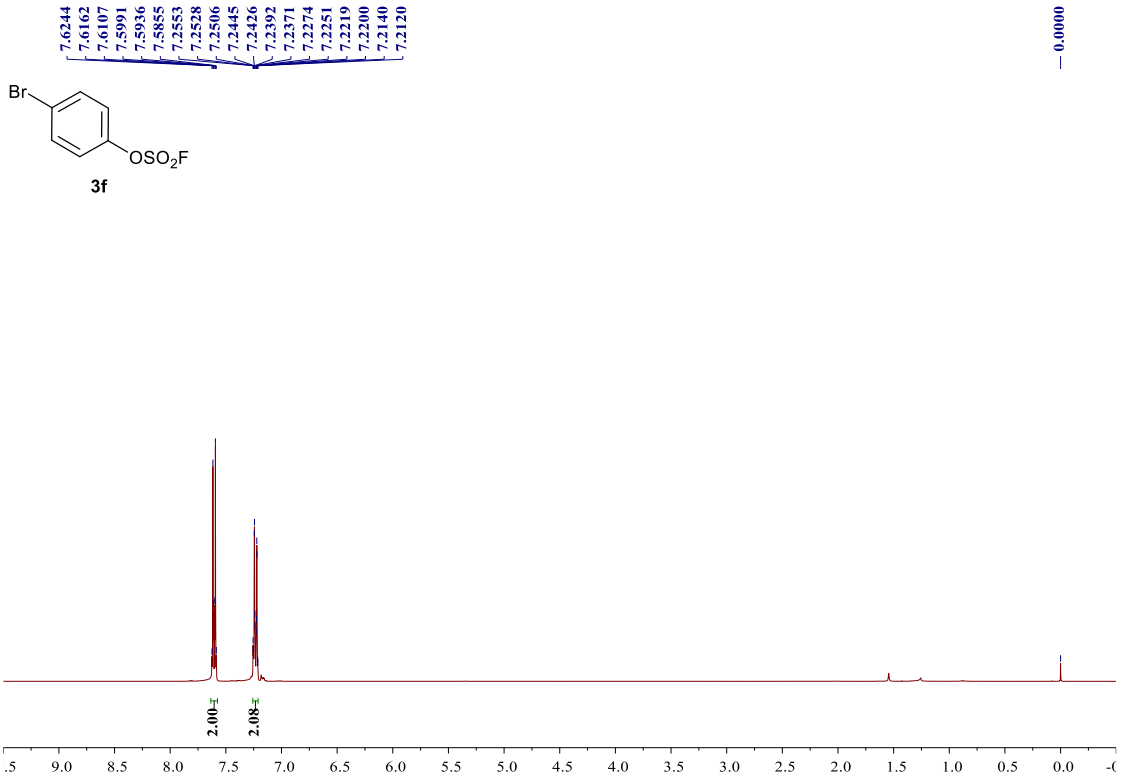
^{1}H NMR Spectrum for Compound **3e** (400 MHz, $CDCl_3$)



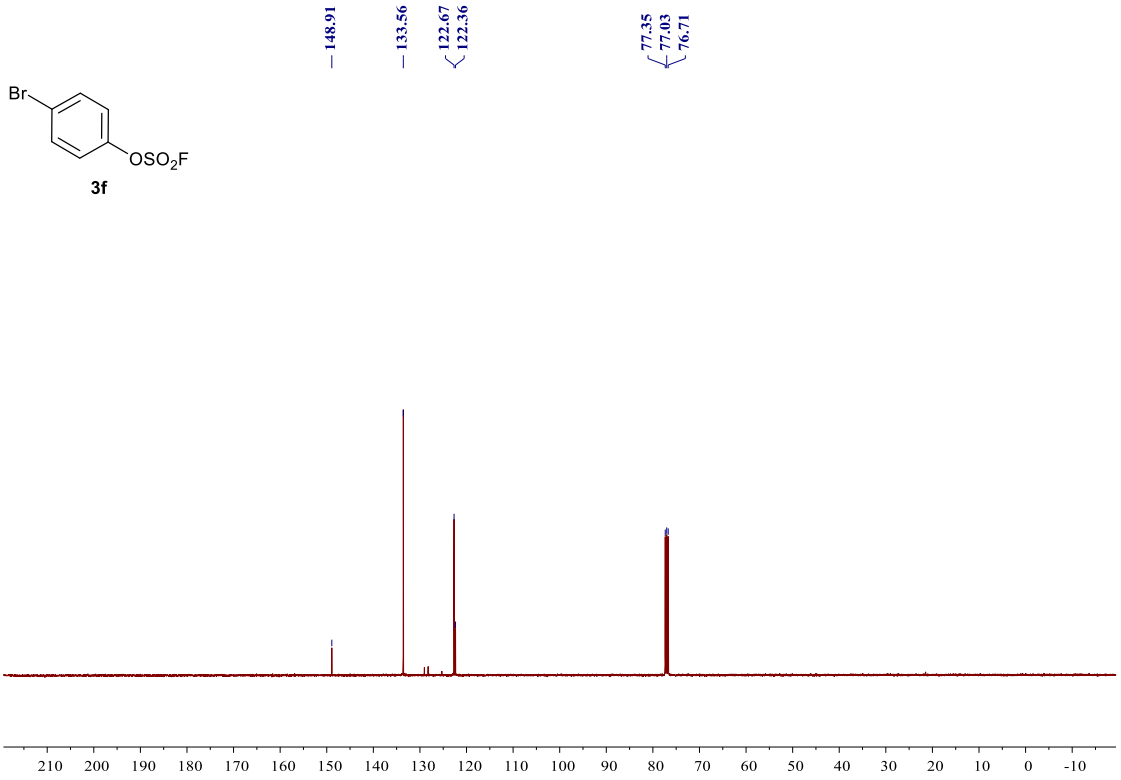
^{13}C NMR Spectrum for Compound **3e** (100 MHz, $CDCl_3$)



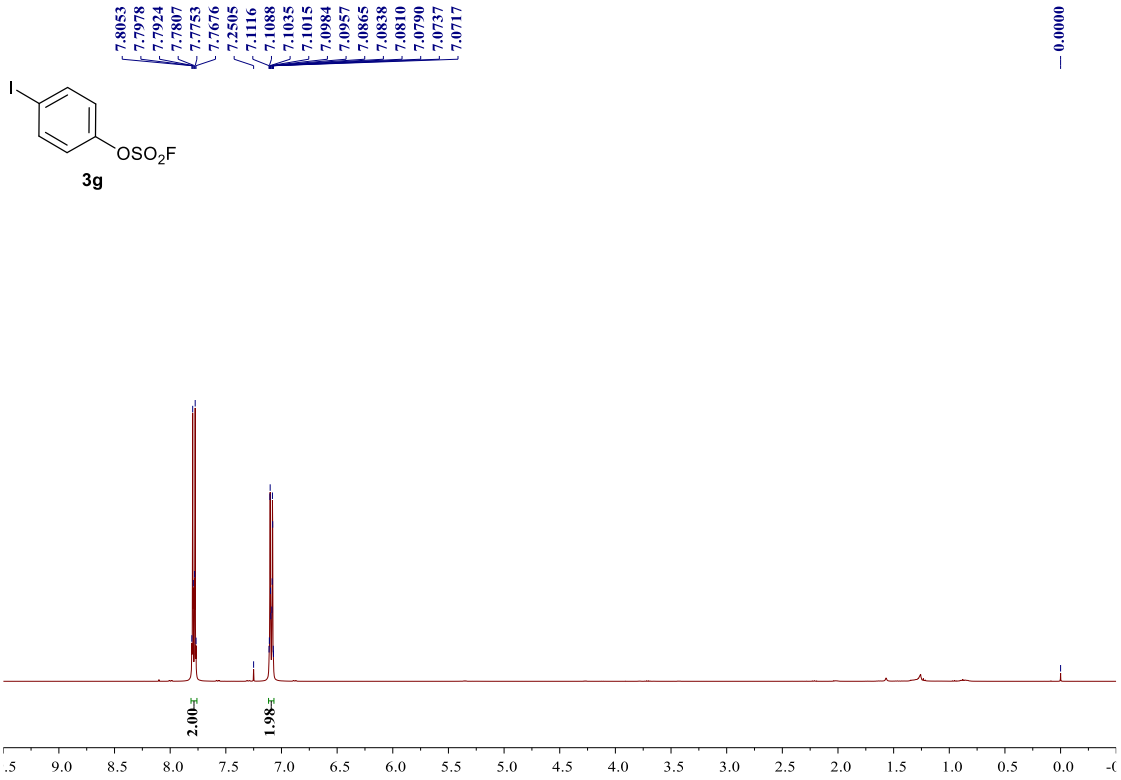
^{1}H NMR Spectrum for Compound **3f** (400 MHz, $CDCl_3$)



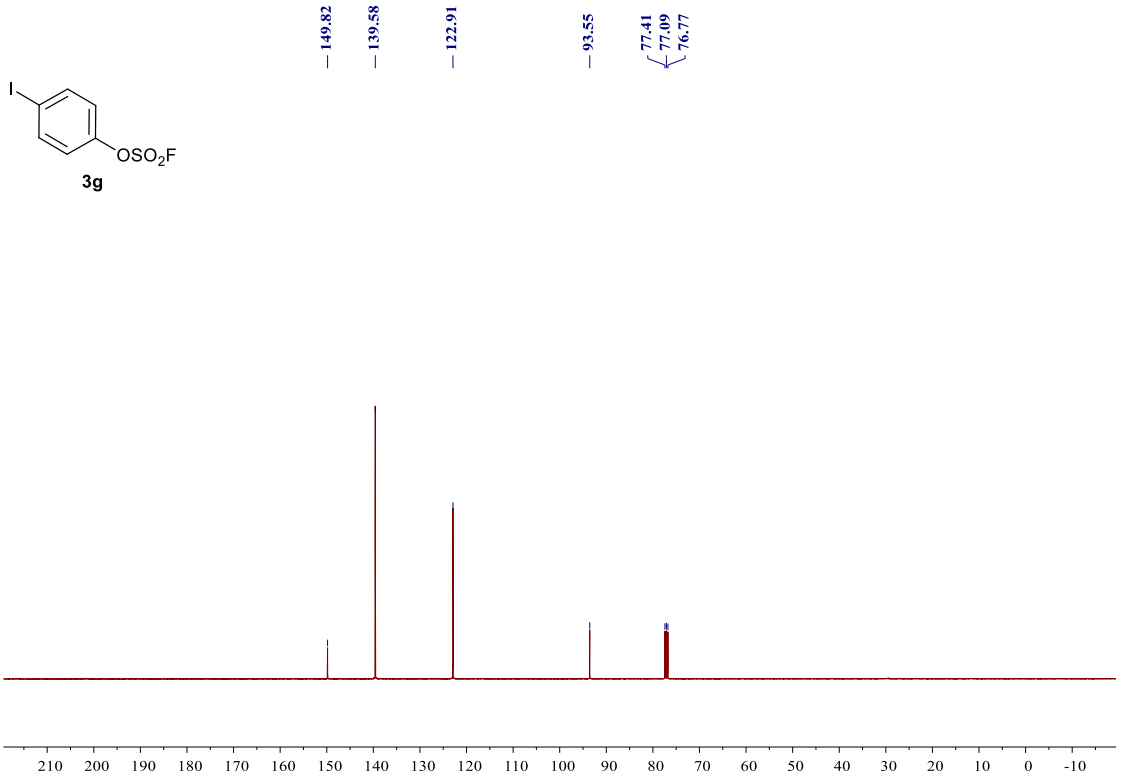
^{13}C NMR Spectrum for Compound **3f** (100 MHz, $CDCl_3$)



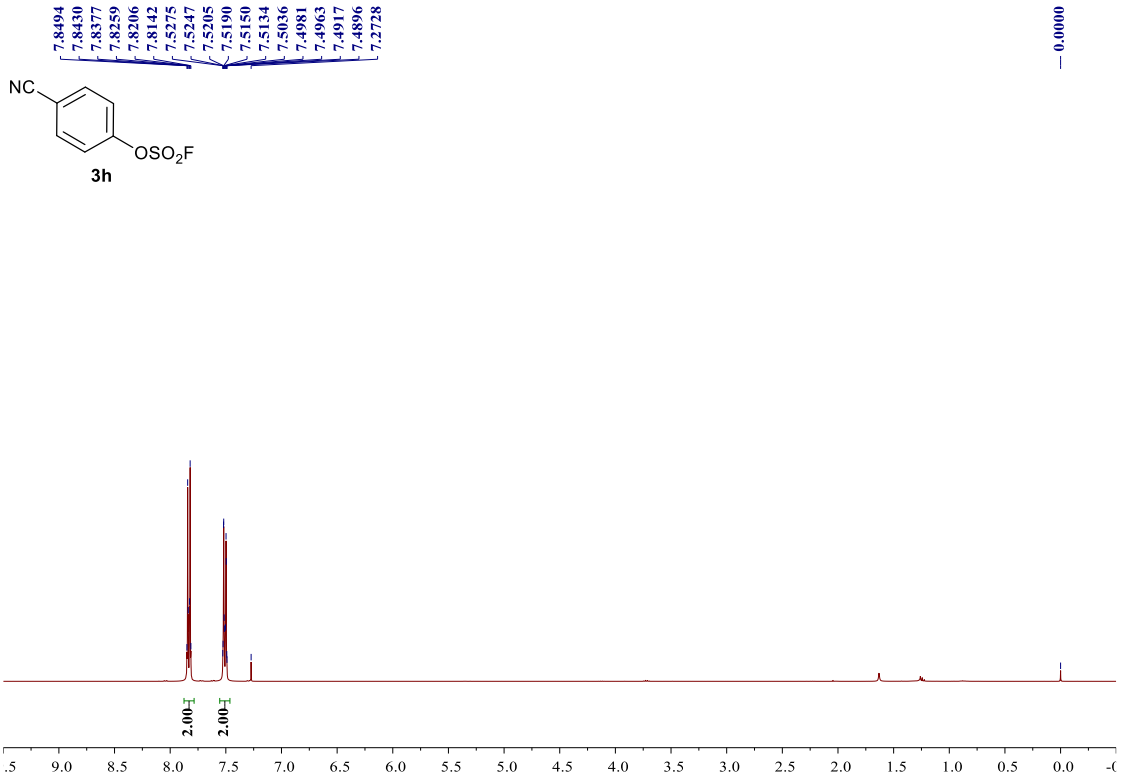
^{1}H NMR Spectrum for Compound **3g** (400 MHz, $CDCl_3$)



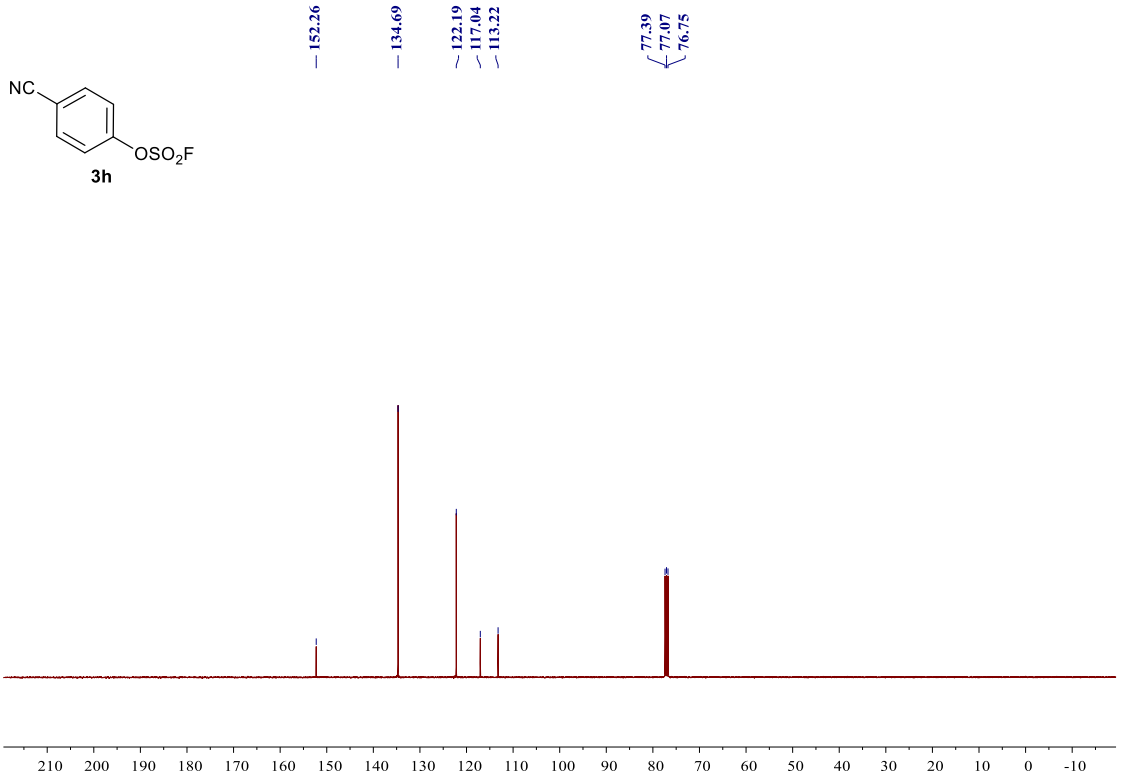
^{13}C NMR Spectrum for Compound **3g** (100 MHz, $CDCl_3$)



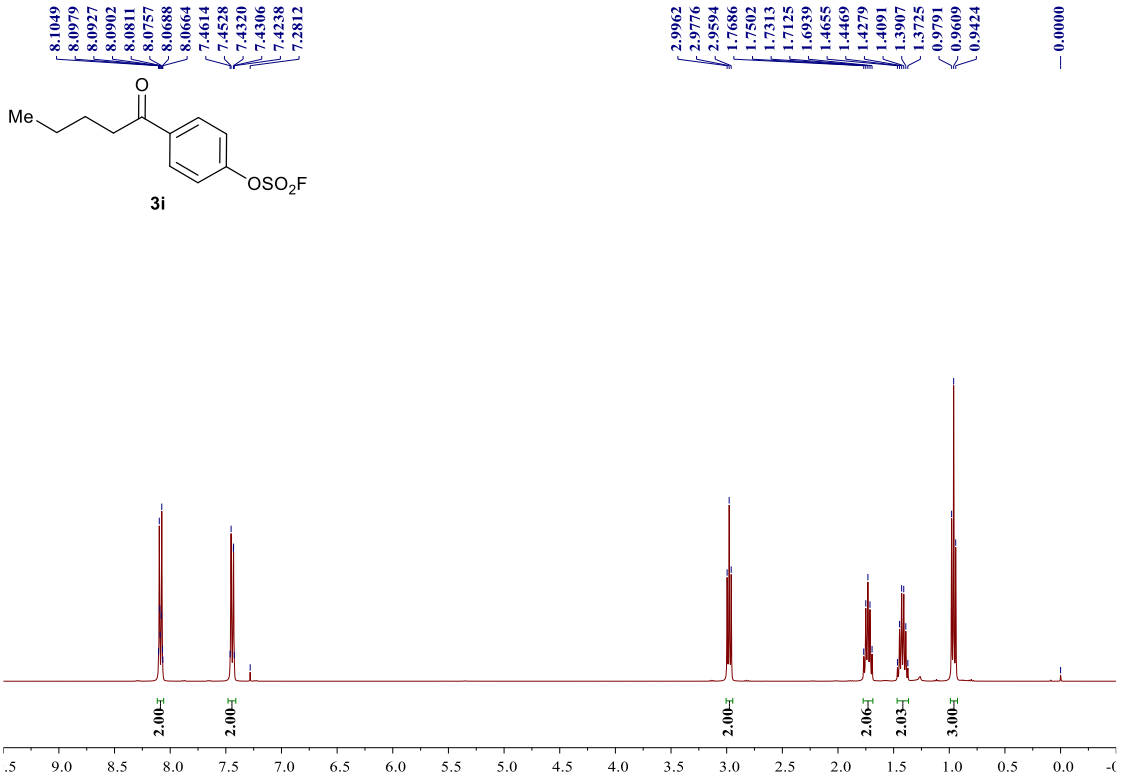
^{1}H NMR Spectrum for Compound **3h** (400 MHz, $CDCl_3$)



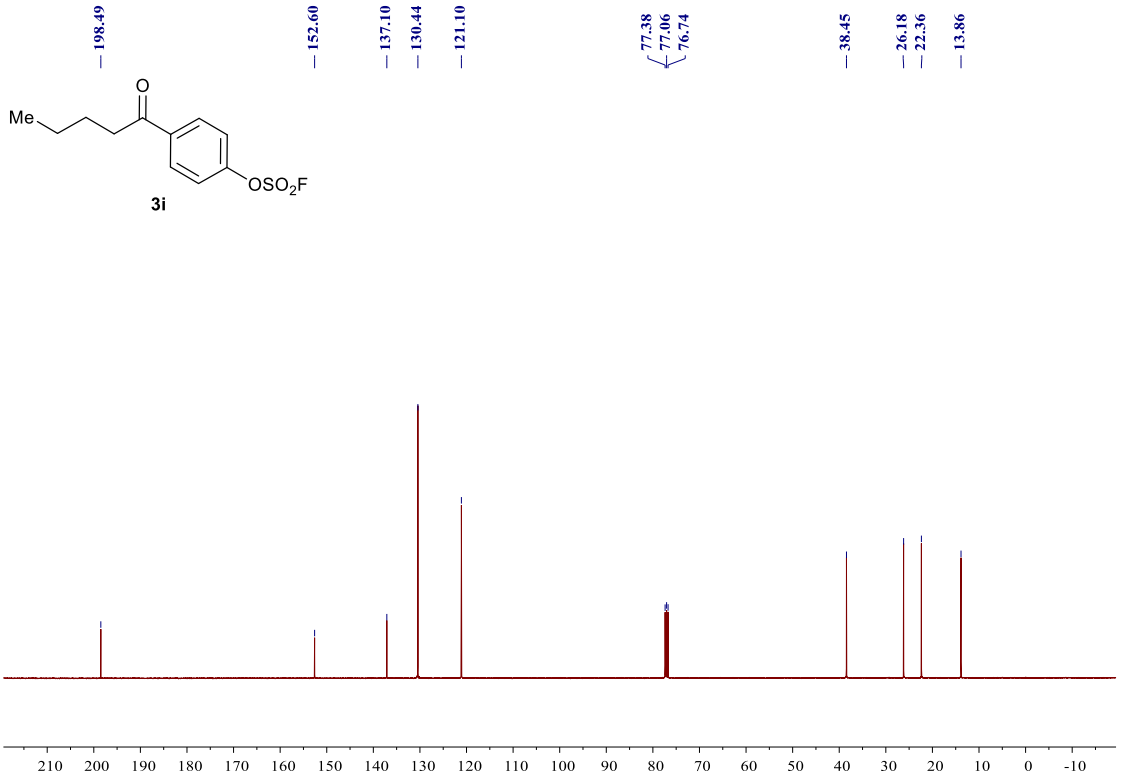
^{13}C NMR Spectrum for Compound **3h** (100 MHz, $CDCl_3$)



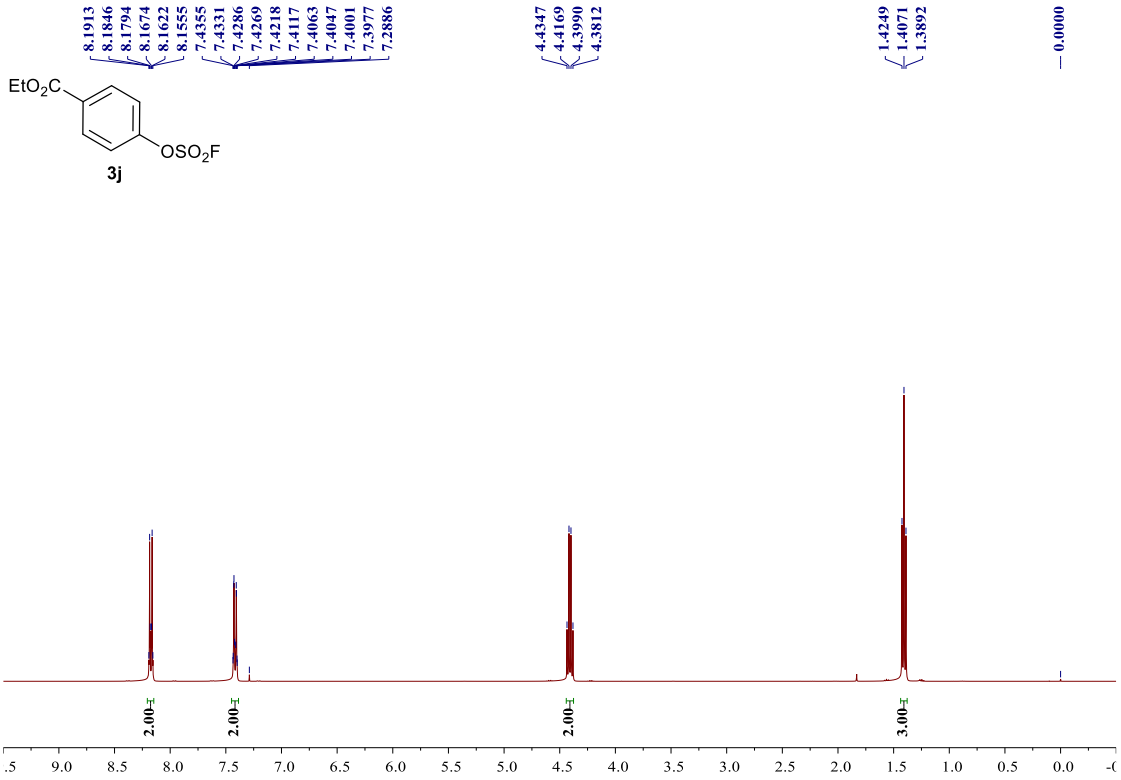
^{1}H NMR Spectrum for Compound **3i** (400 MHz, $CDCl_3$)



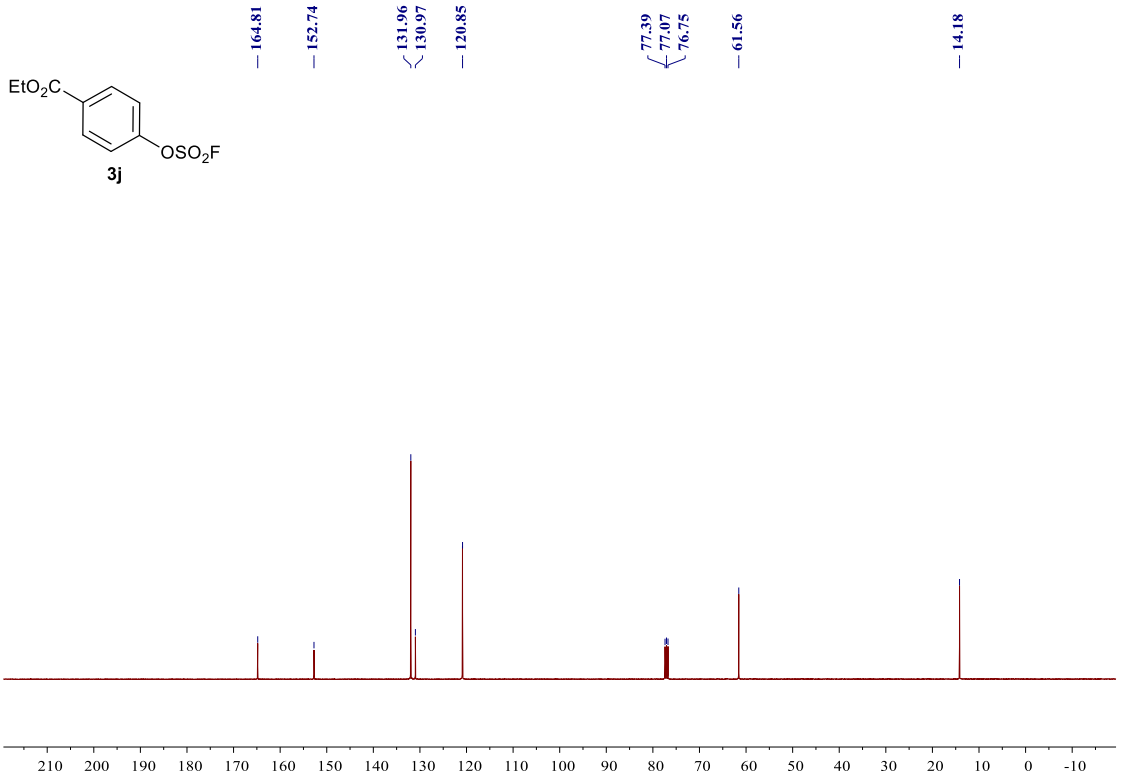
^{13}C NMR Spectrum for Compound **3i** (100 MHz, $CDCl_3$)



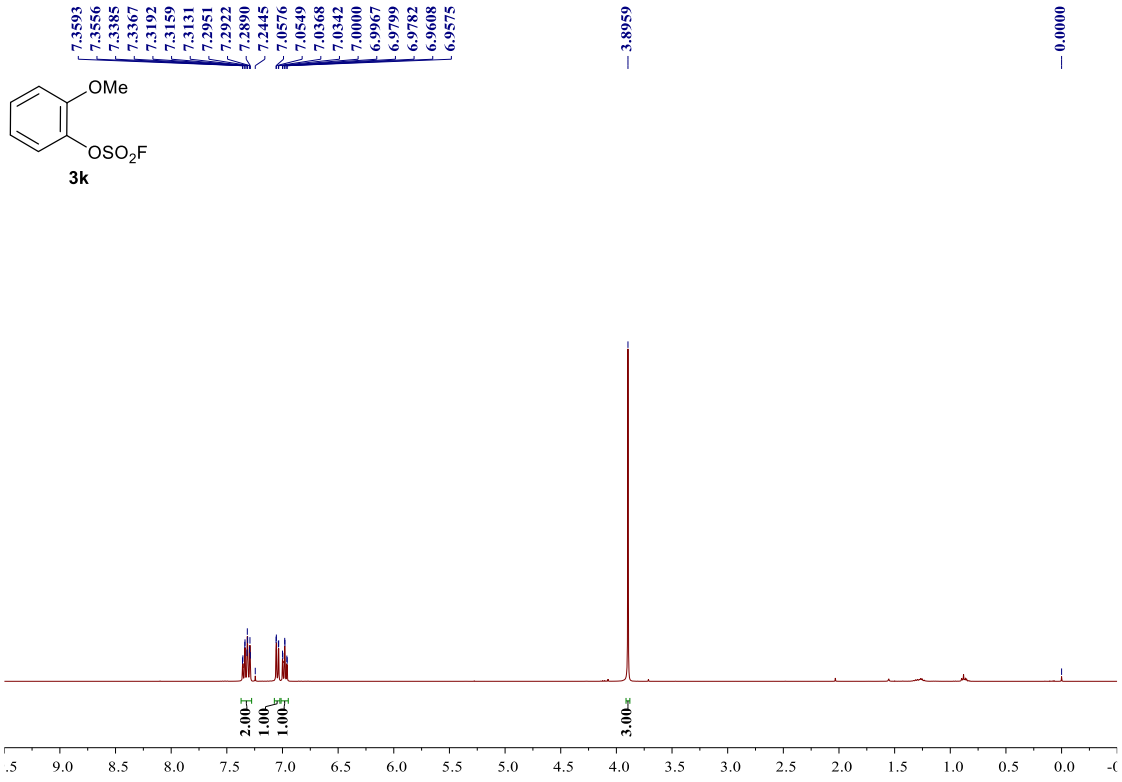
^{1}H NMR Spectrum for Compound **3j** (400 MHz, $CDCl_3$)



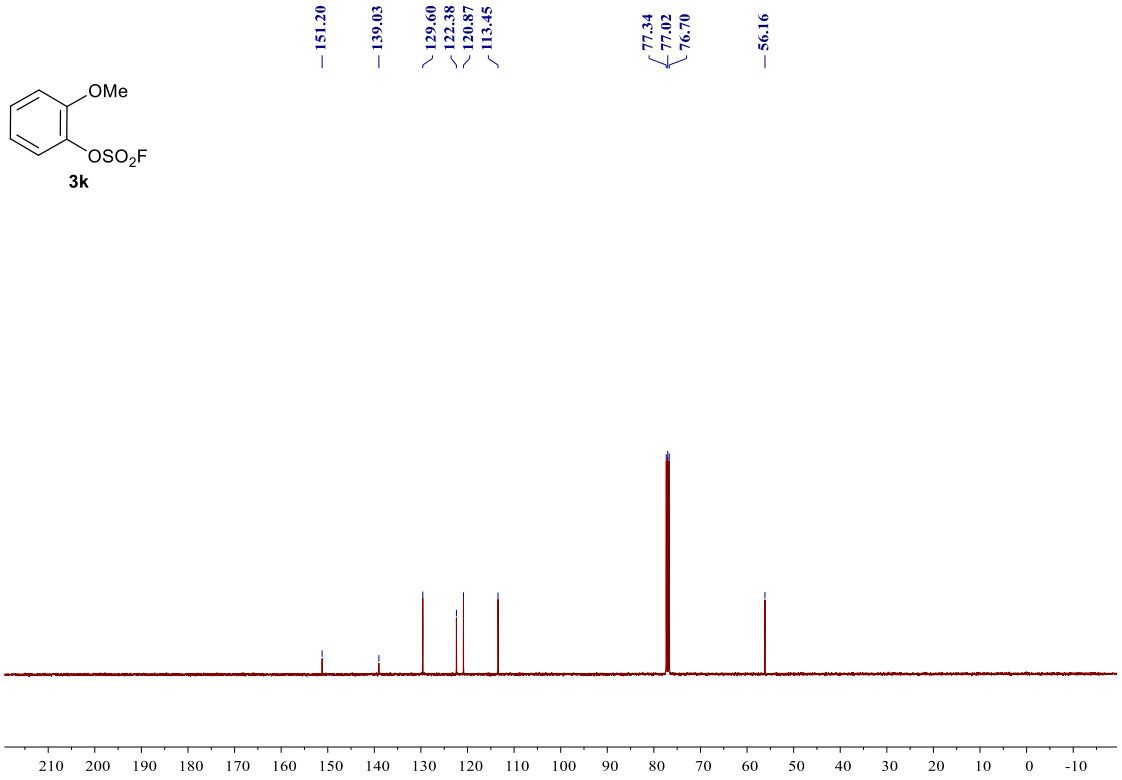
^{13}C NMR Spectrum for Compound **3j** (100 MHz, $CDCl_3$)



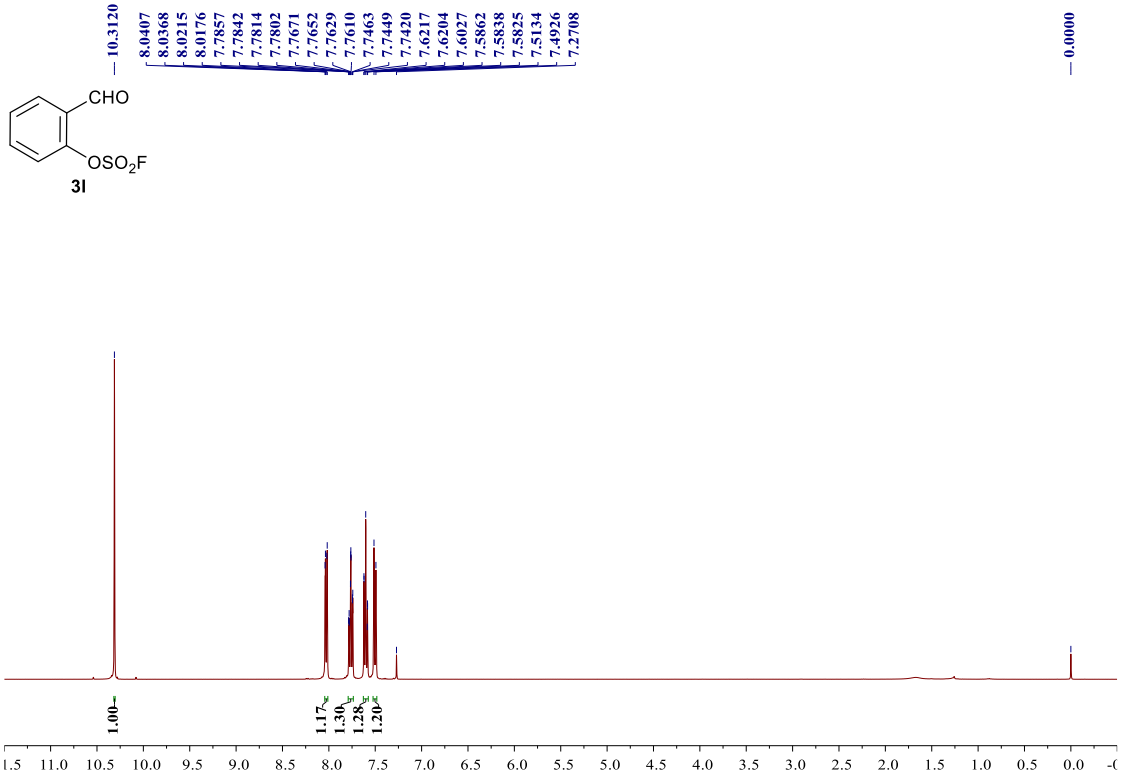
^{1}H NMR Spectrum for Compound **3k** (400 MHz, $CDCl_3$)



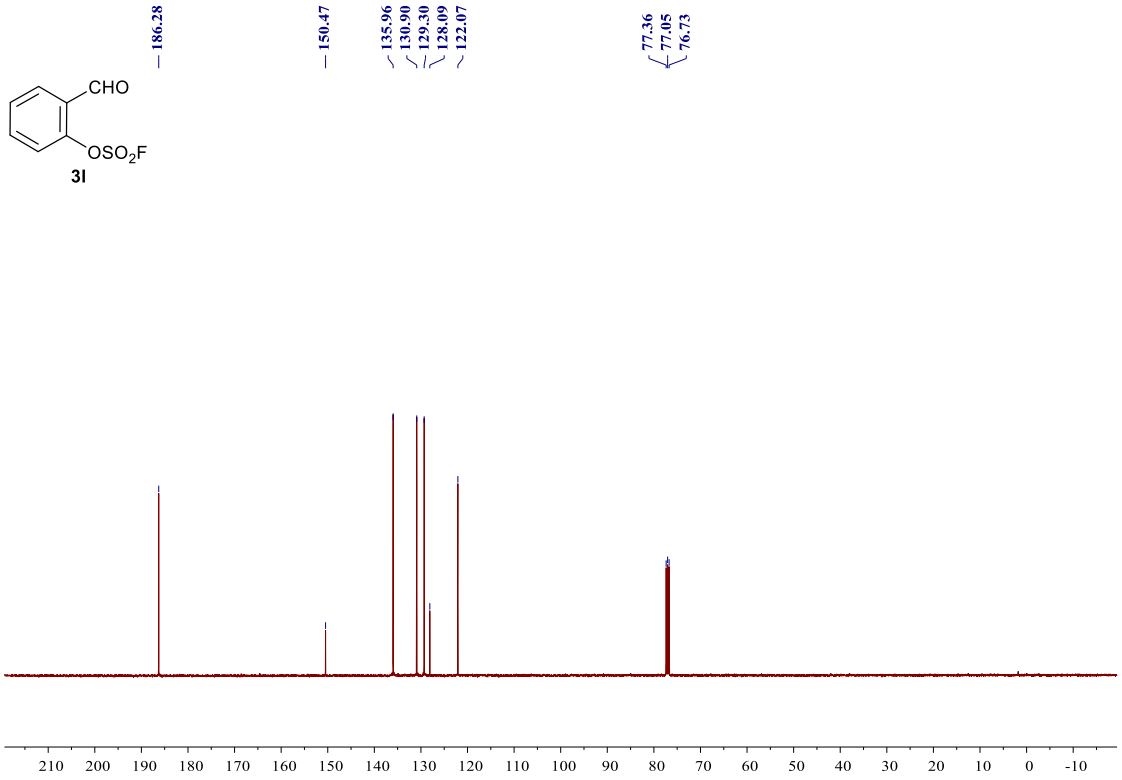
^{13}C NMR Spectrum for Compound **3k** (100 MHz, $CDCl_3$)



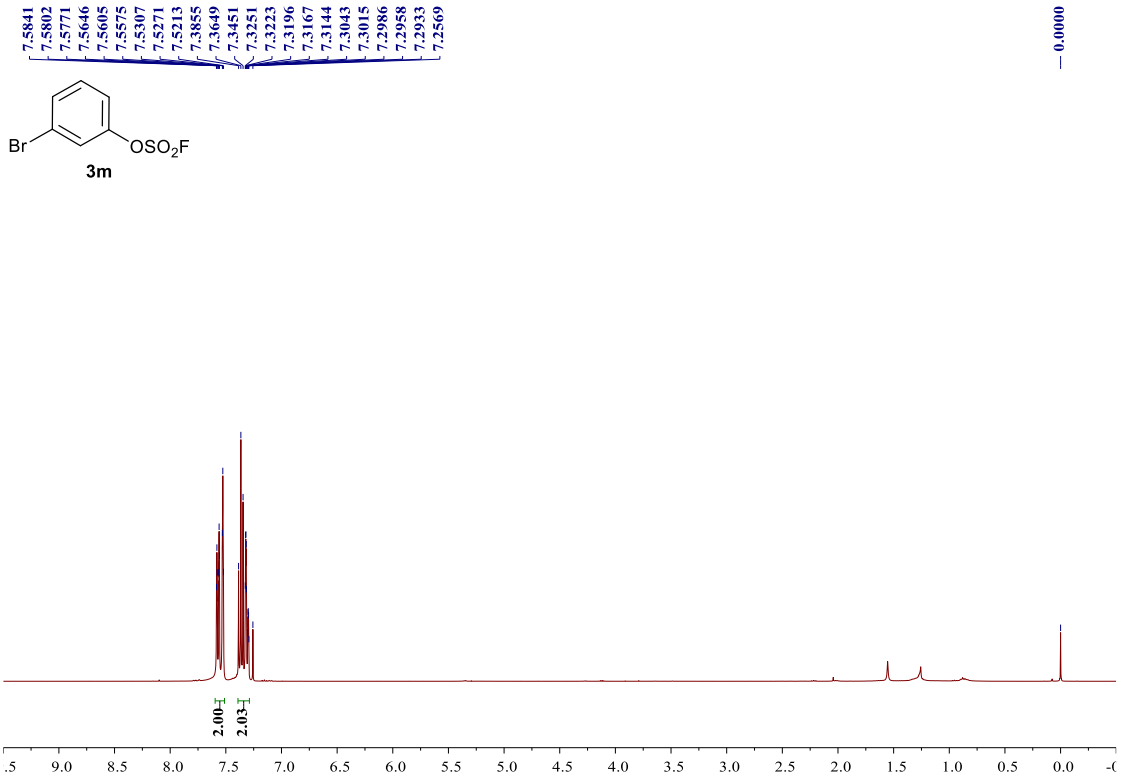
^{1}H NMR Spectrum for Compound **3l** (400 MHz, $CDCl_3$)



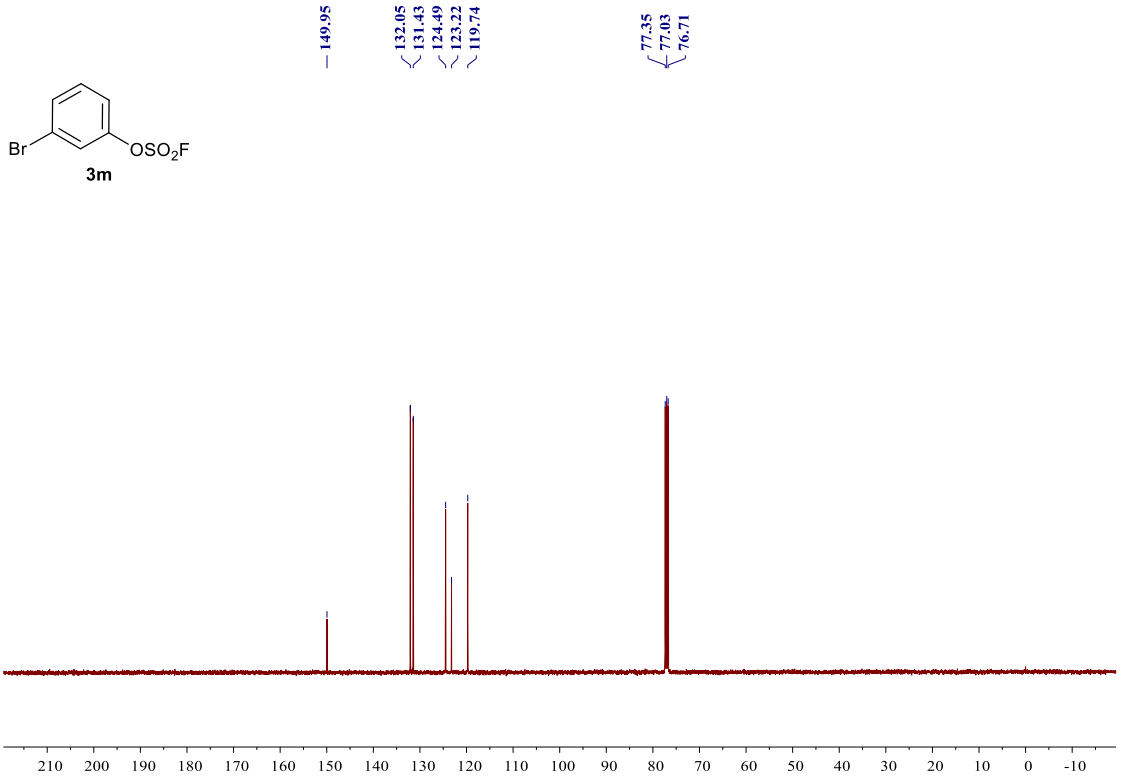
^{13}C NMR Spectrum for Compound **3l** (100 MHz, $CDCl_3$)



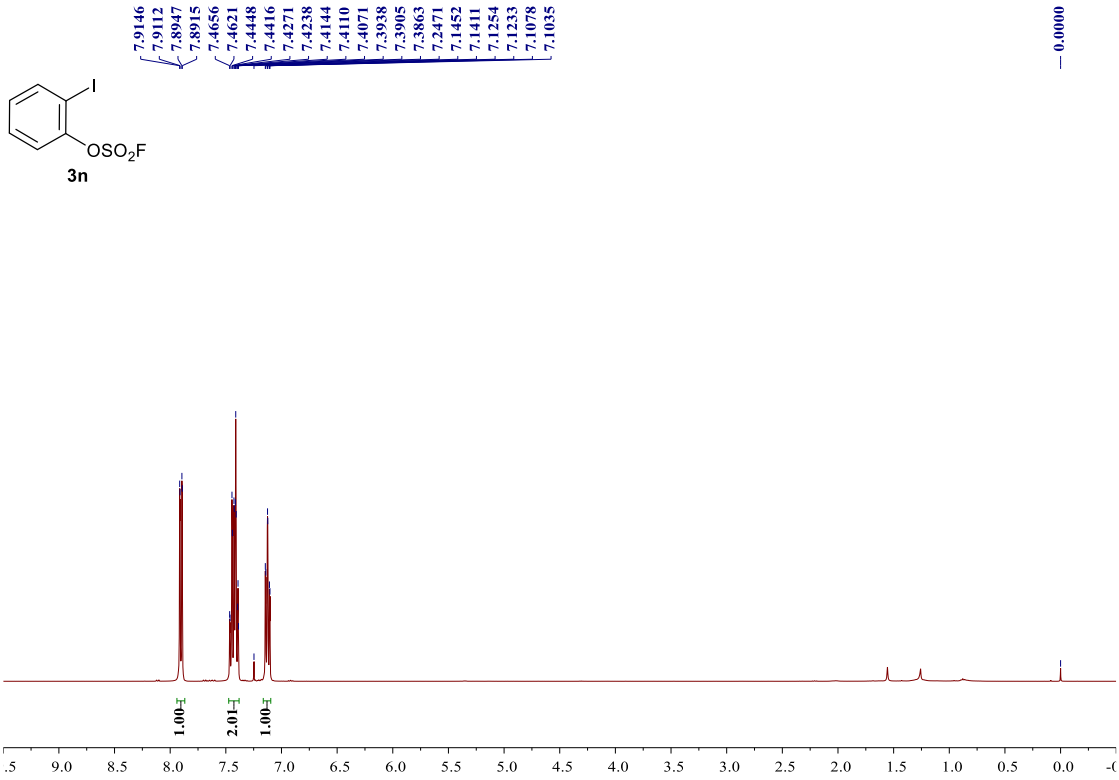
^{1}H NMR Spectrum for Compound **3m** (400 MHz, $CDCl_3$)



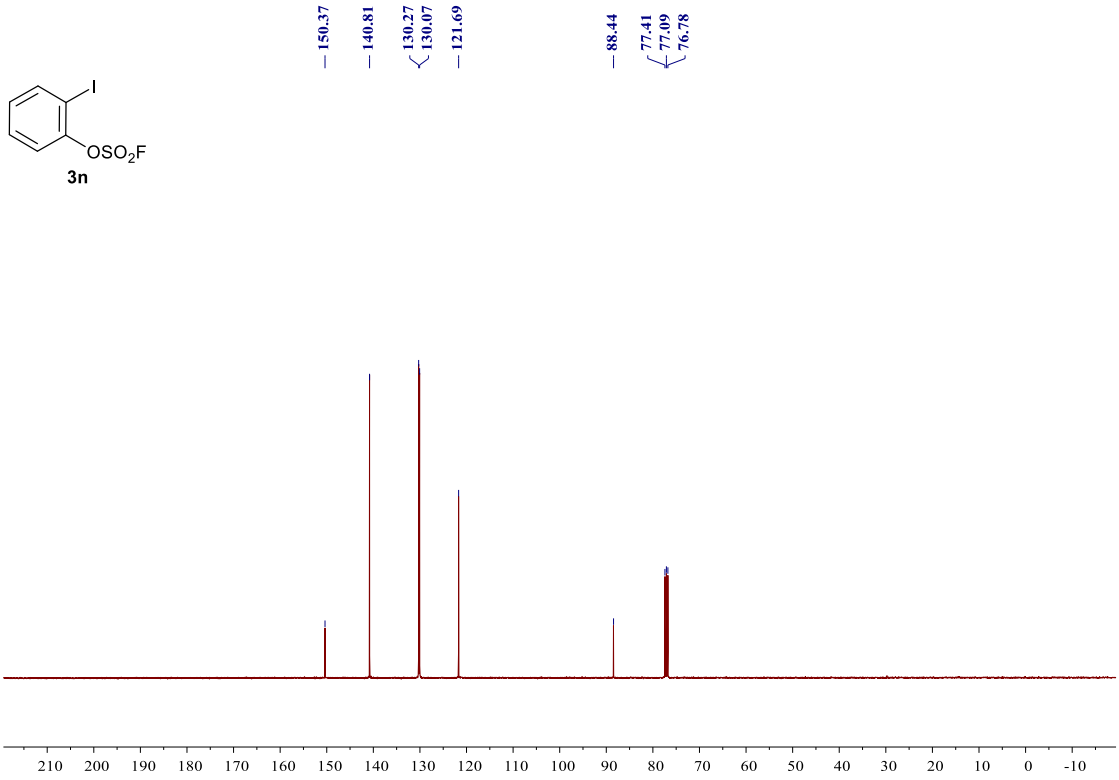
^{13}C NMR Spectrum for Compound **3m** (100 MHz, $CDCl_3$)



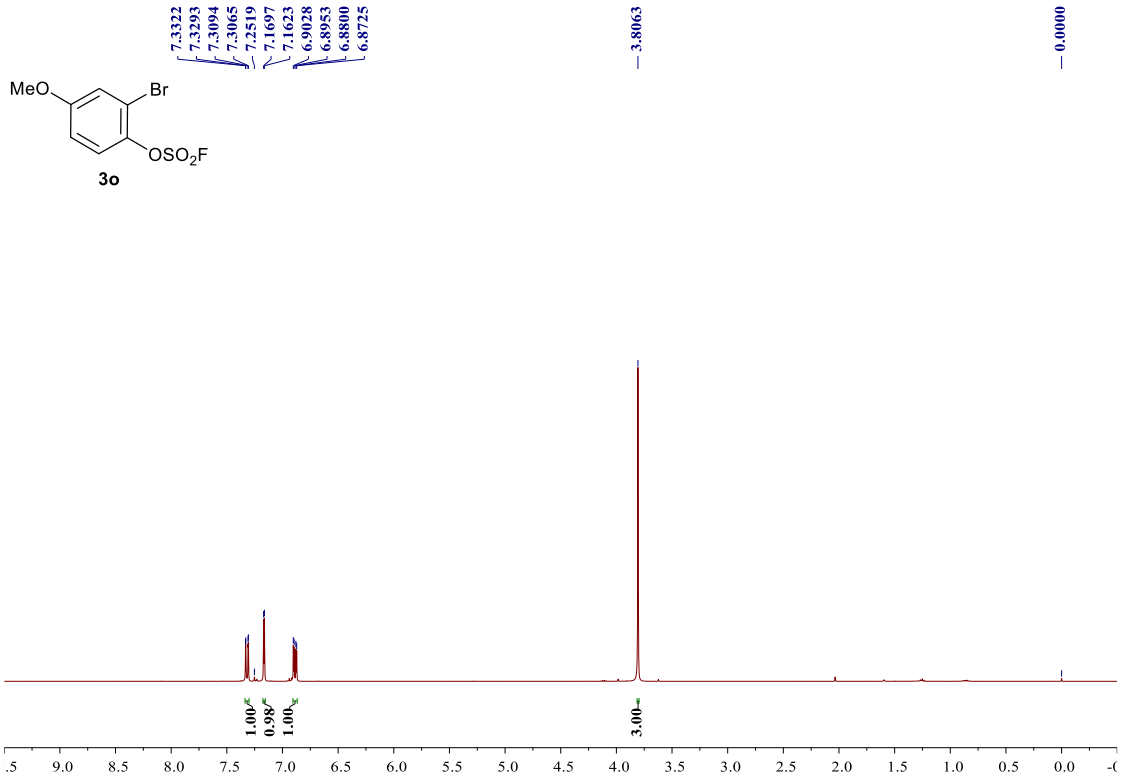
^{1}H NMR Spectrum for Compound **3n** (400 MHz, $CDCl_3$)



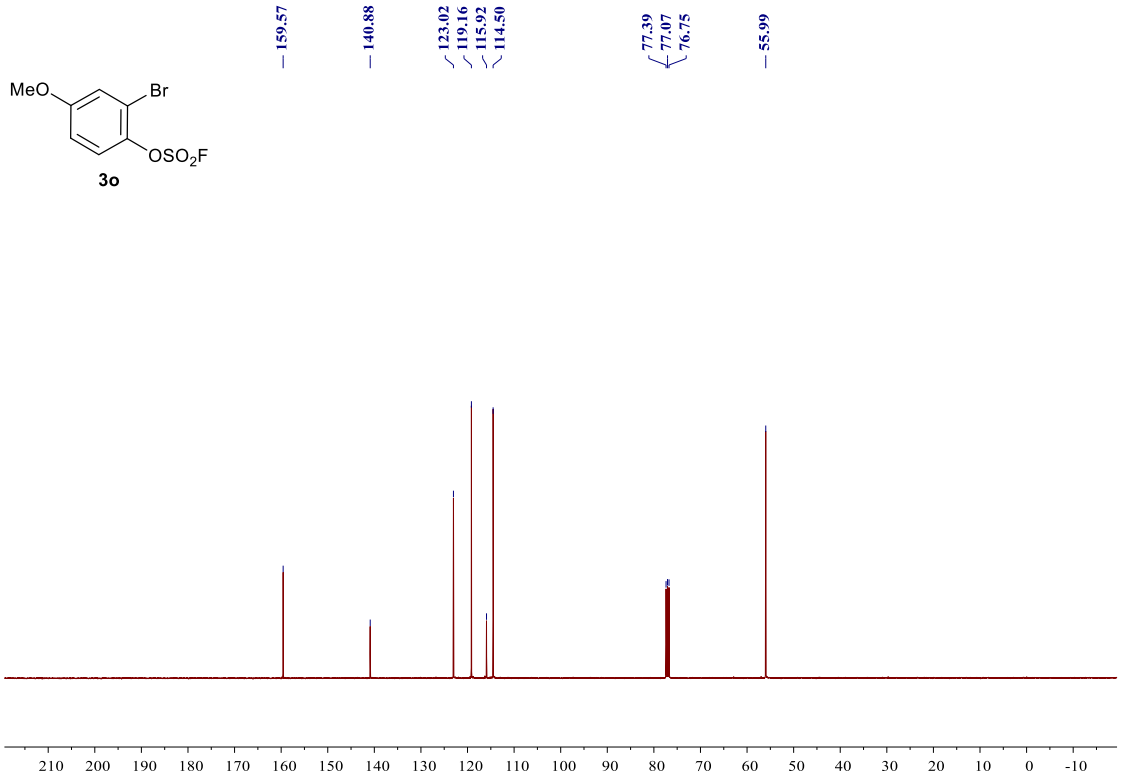
^{13}C NMR Spectrum for Compound **3n** (100 MHz, $CDCl_3$)



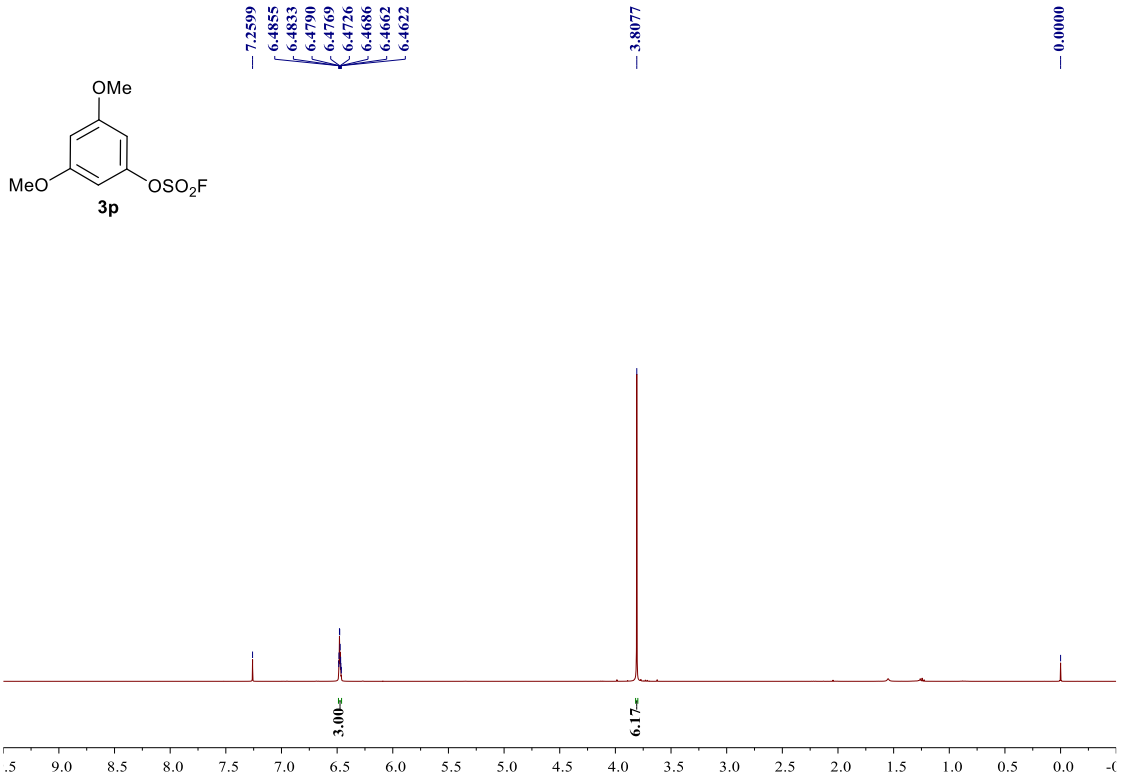
^{1}H NMR Spectrum for Compound **3o** (400 MHz, $CDCl_3$)



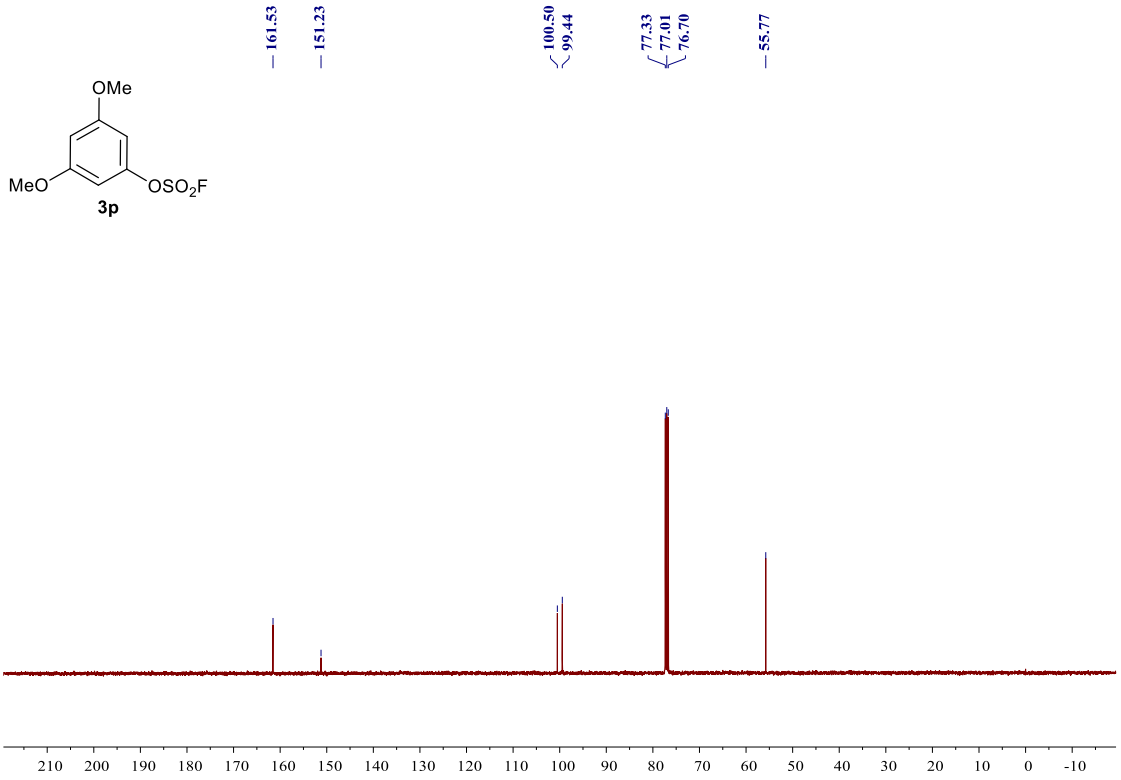
^{13}C NMR Spectrum for Compound **3o** (100 MHz, $CDCl_3$)



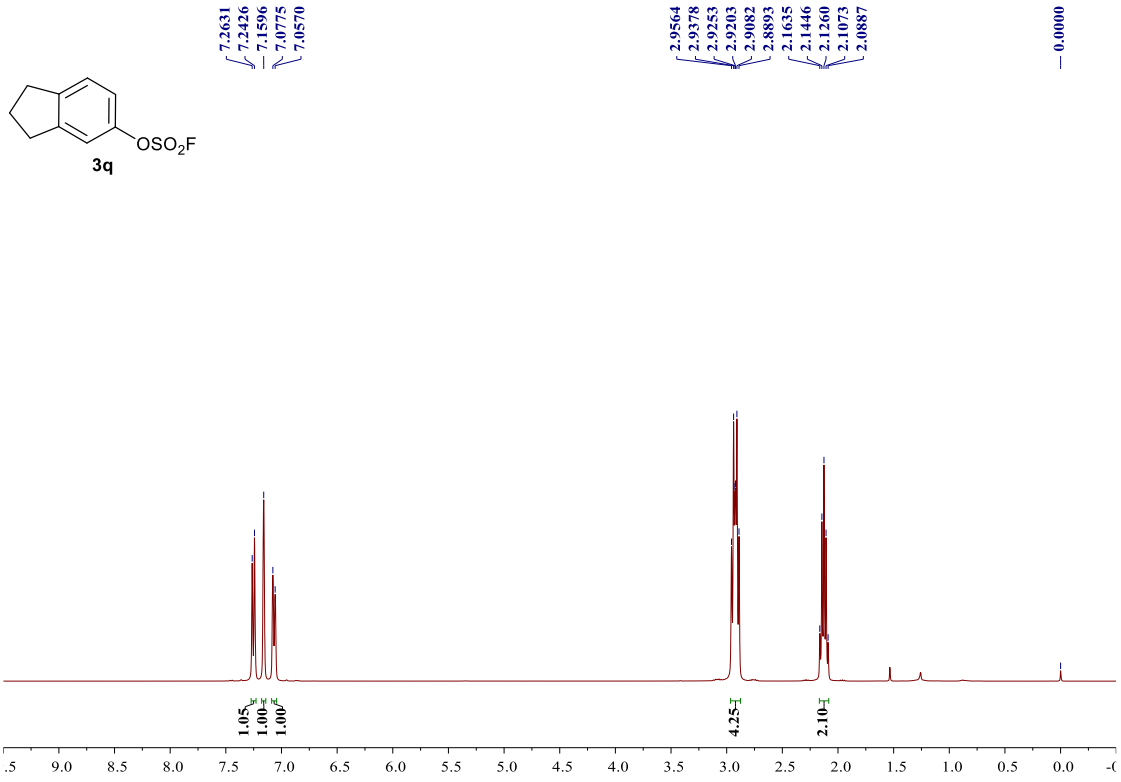
^{1}H NMR Spectrum for Compound **3p** (400 MHz, $CDCl_3$)



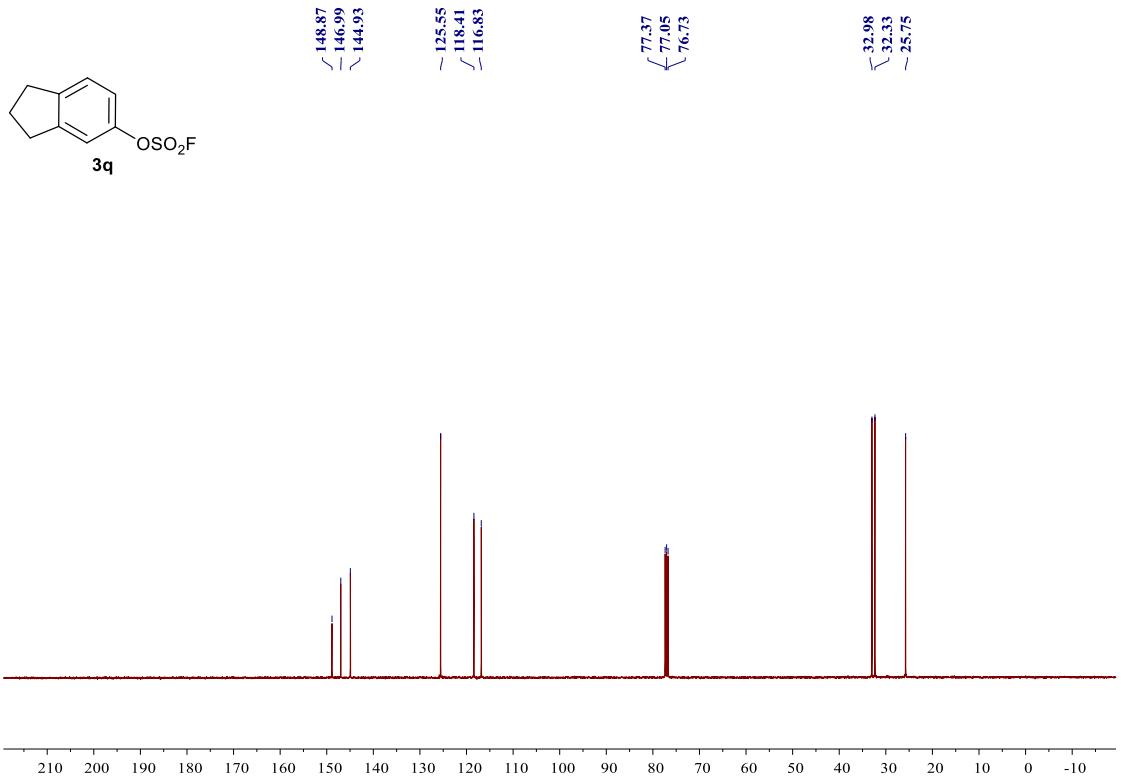
^{13}C NMR Spectrum for Compound **3p** (100 MHz, $CDCl_3$)



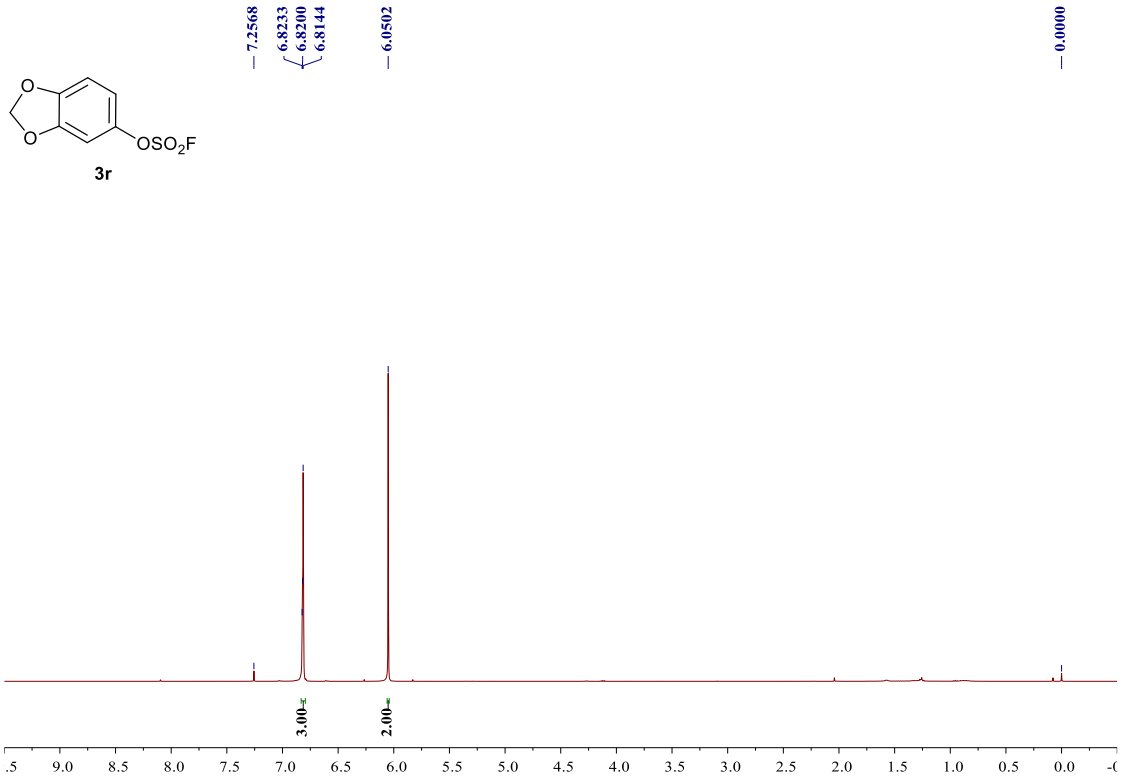
^{1}H NMR Spectrum for Compound **3q** (400 MHz, $CDCl_3$)



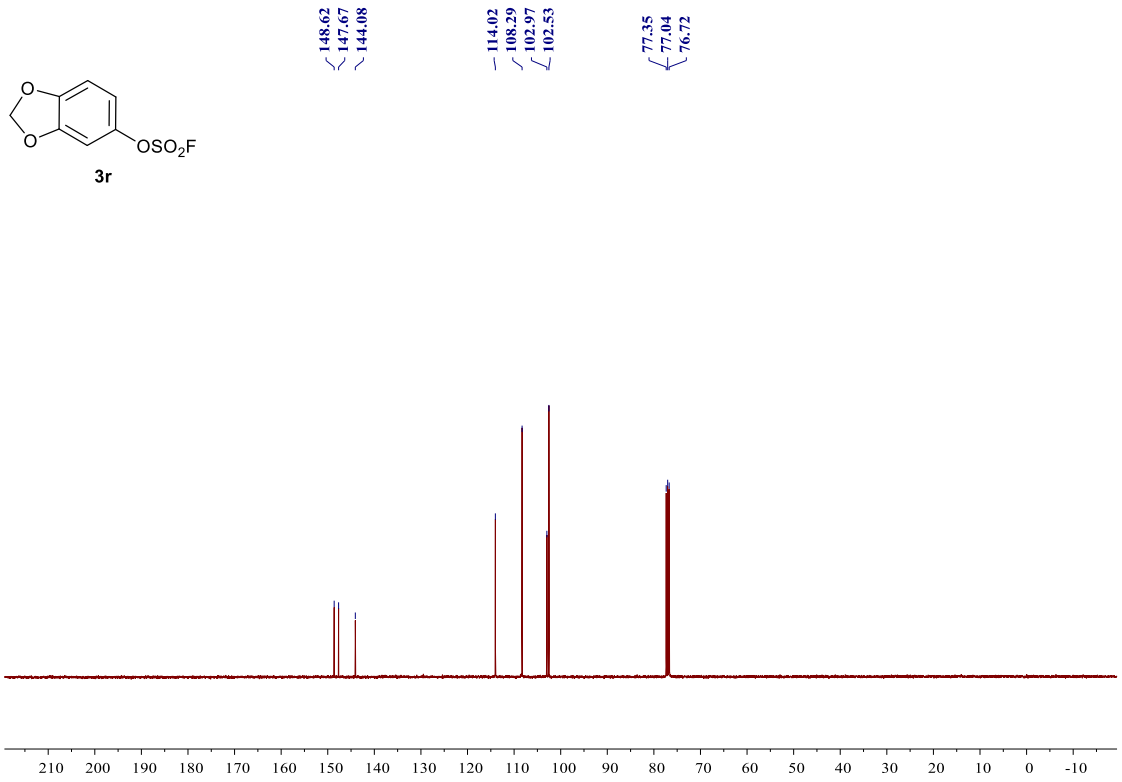
^{13}C NMR Spectrum for Compound **3q** (100 MHz, $CDCl_3$)



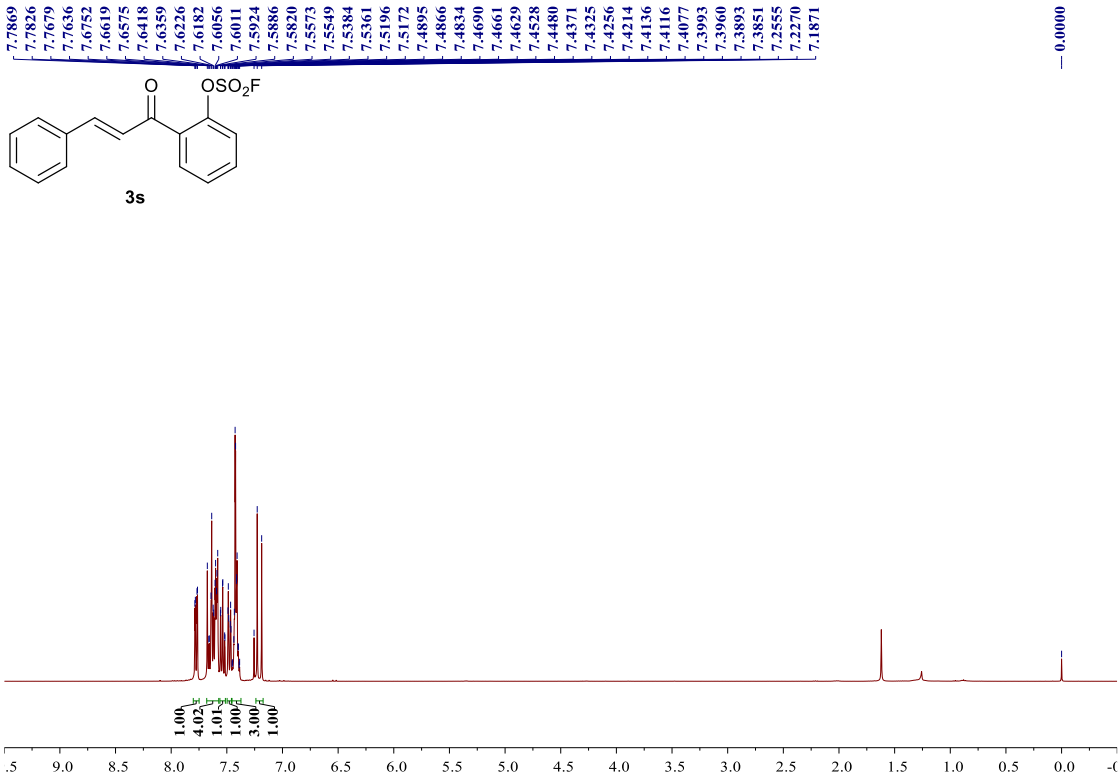
^{1}H NMR Spectrum for Compound **3r** (400 MHz, $CDCl_3$)



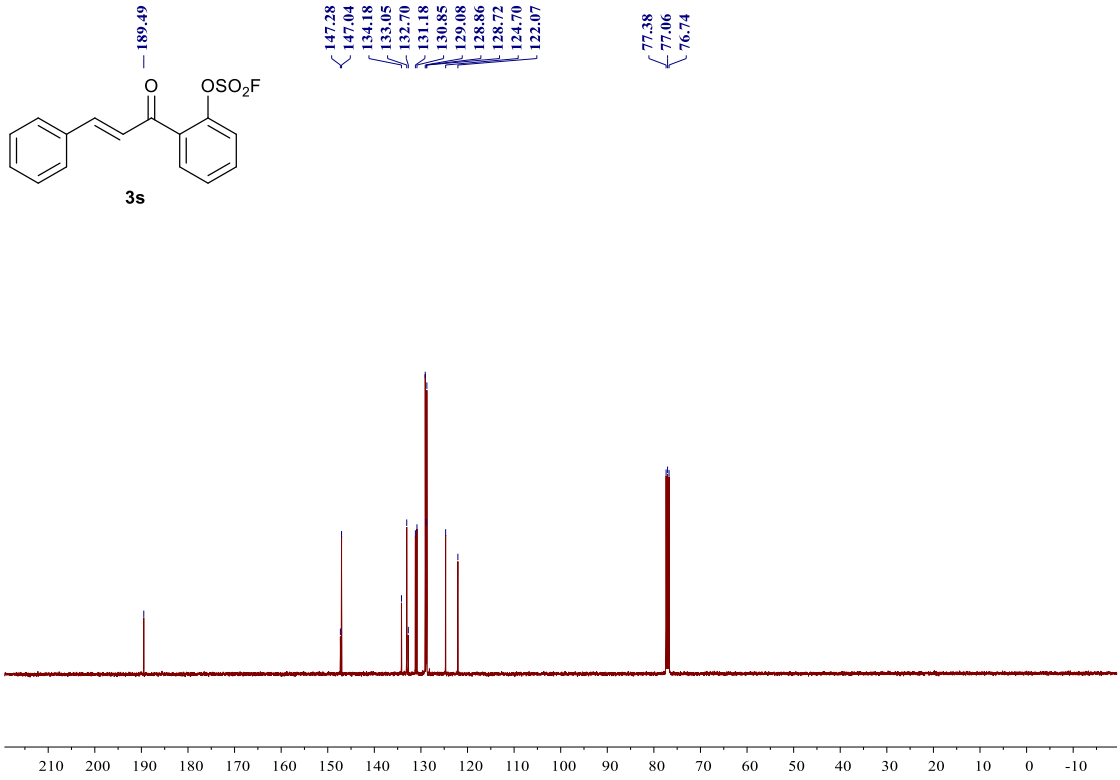
^{13}C NMR Spectrum for Compound **3r** (100 MHz, $CDCl_3$)



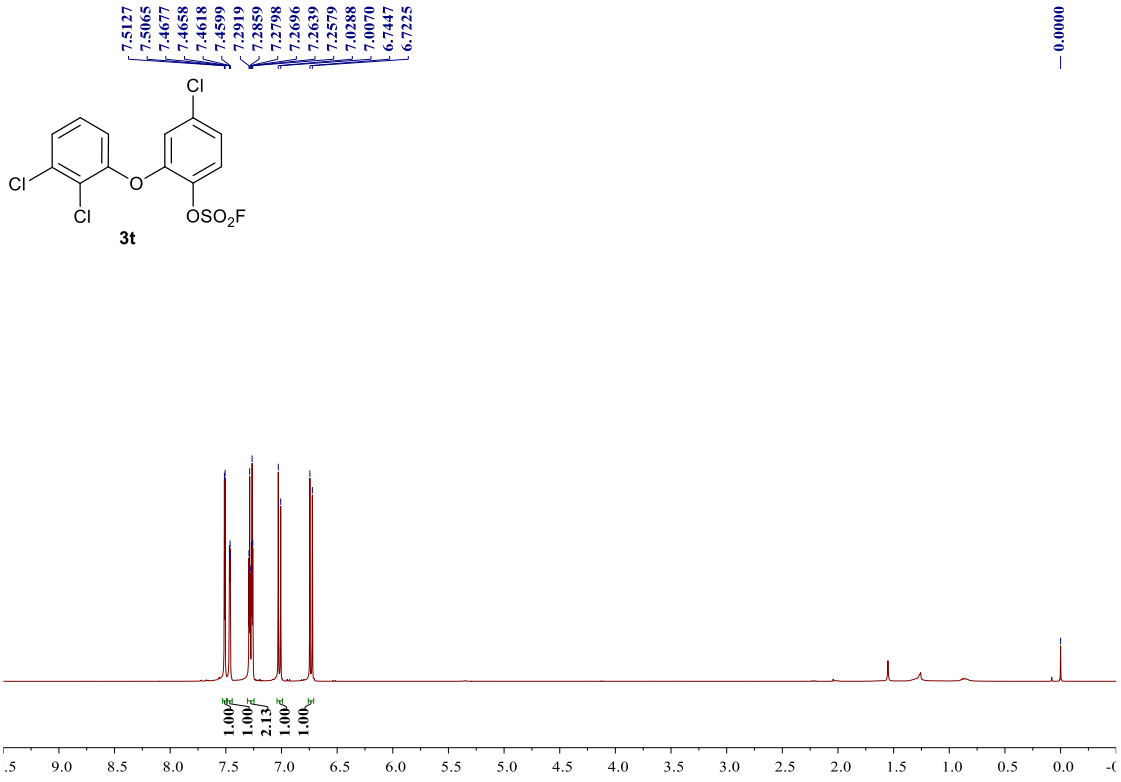
^{1}H NMR Spectrum for Compound **3s** (400 MHz, $CDCl_3$)



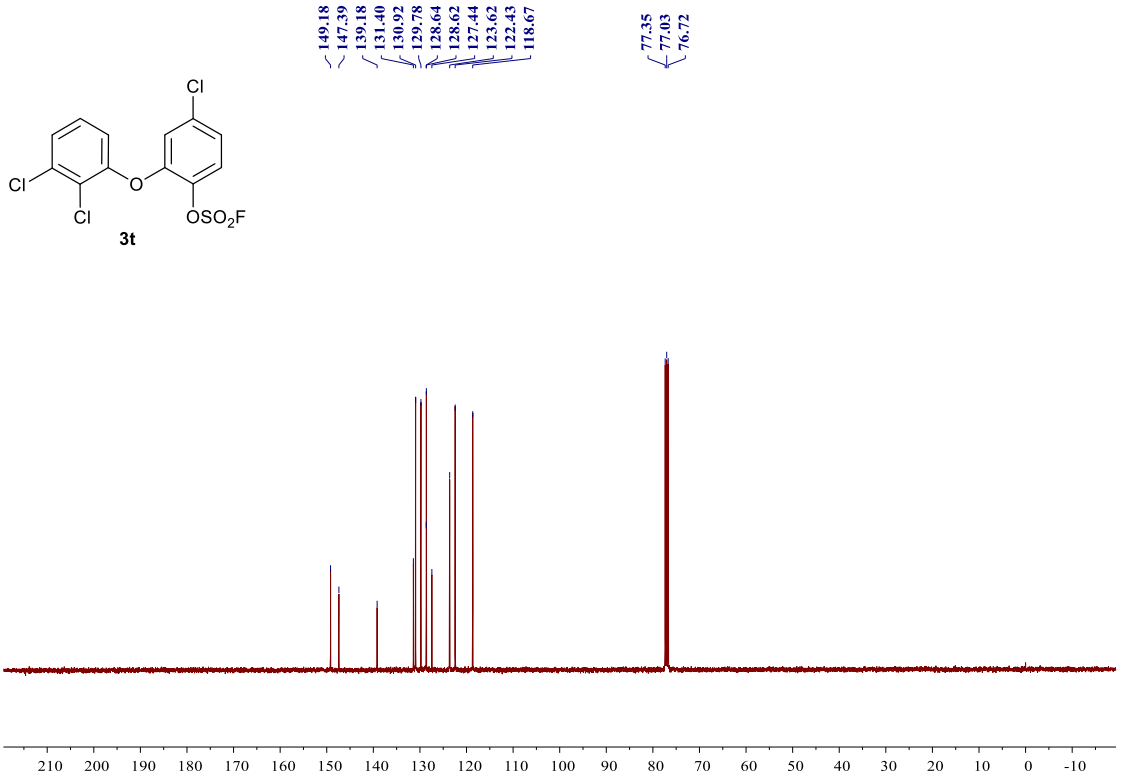
^{13}C NMR Spectrum for Compound **3s** (100 MHz, $CDCl_3$)



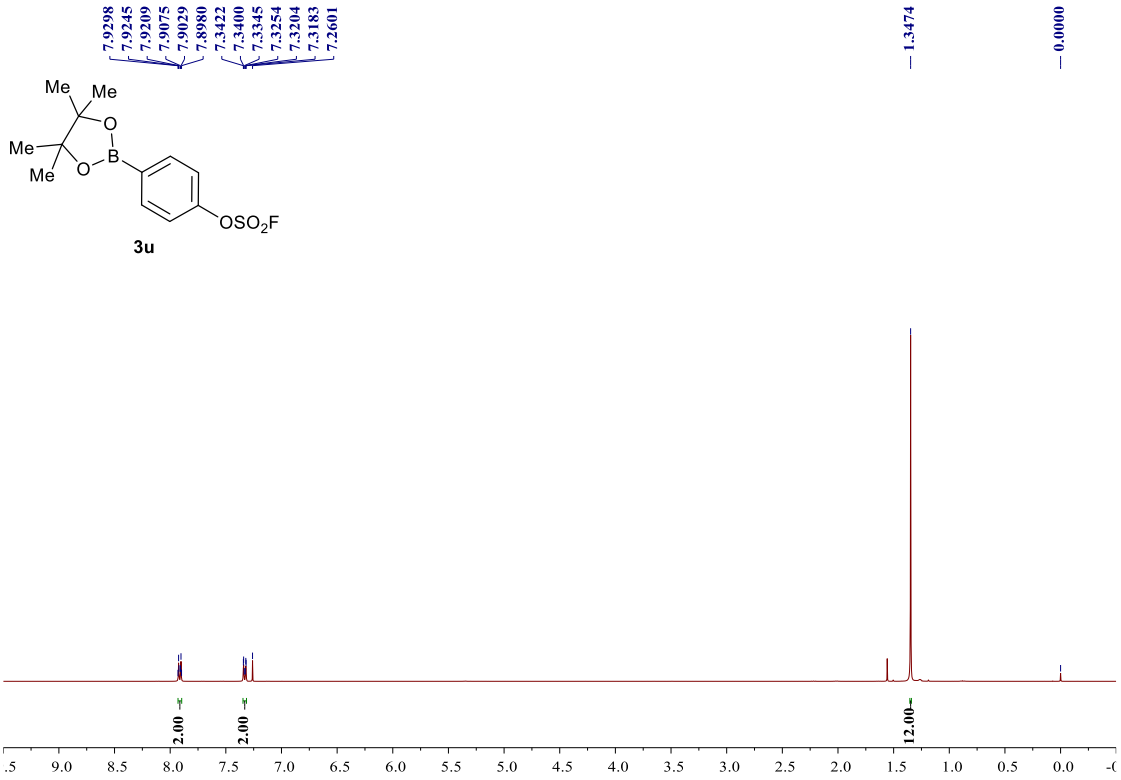
^{1}H NMR Spectrum for Compound **3t** (400 MHz, $CDCl_3$)



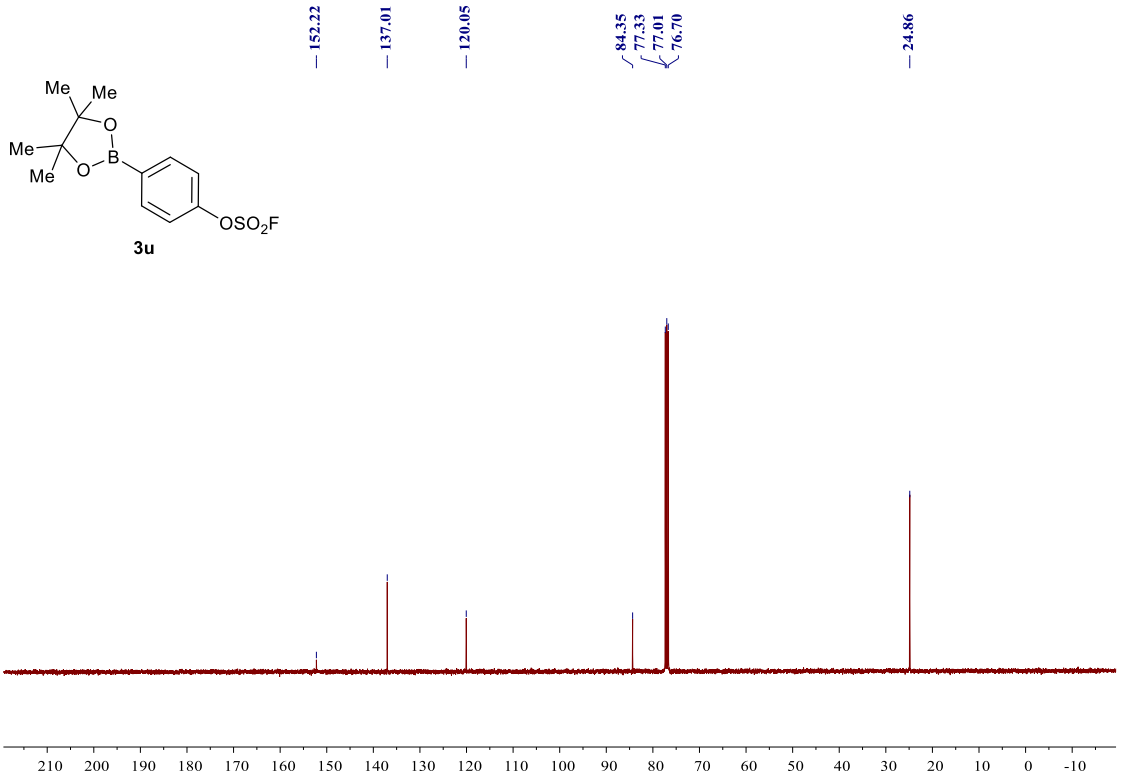
^{13}C NMR Spectrum for Compound **3t** (100 MHz, $CDCl_3$)



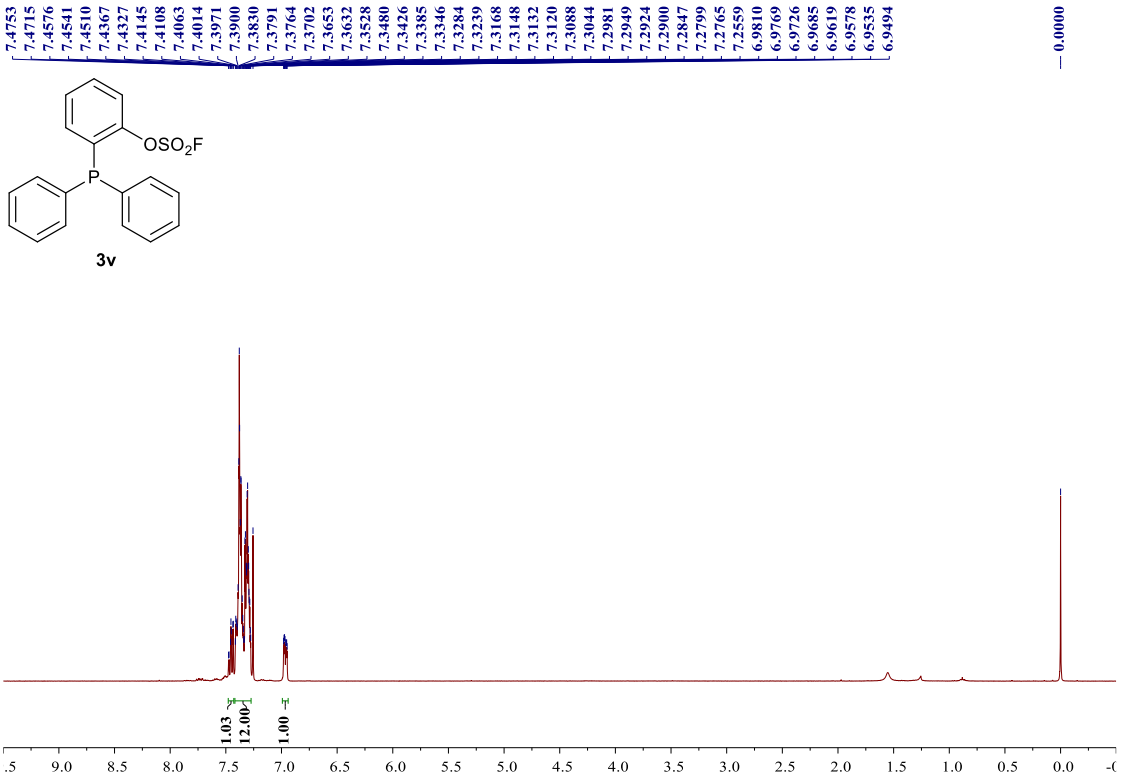
^{1}H NMR Spectrum for Compound **3u** (400 MHz, $CDCl_3$)



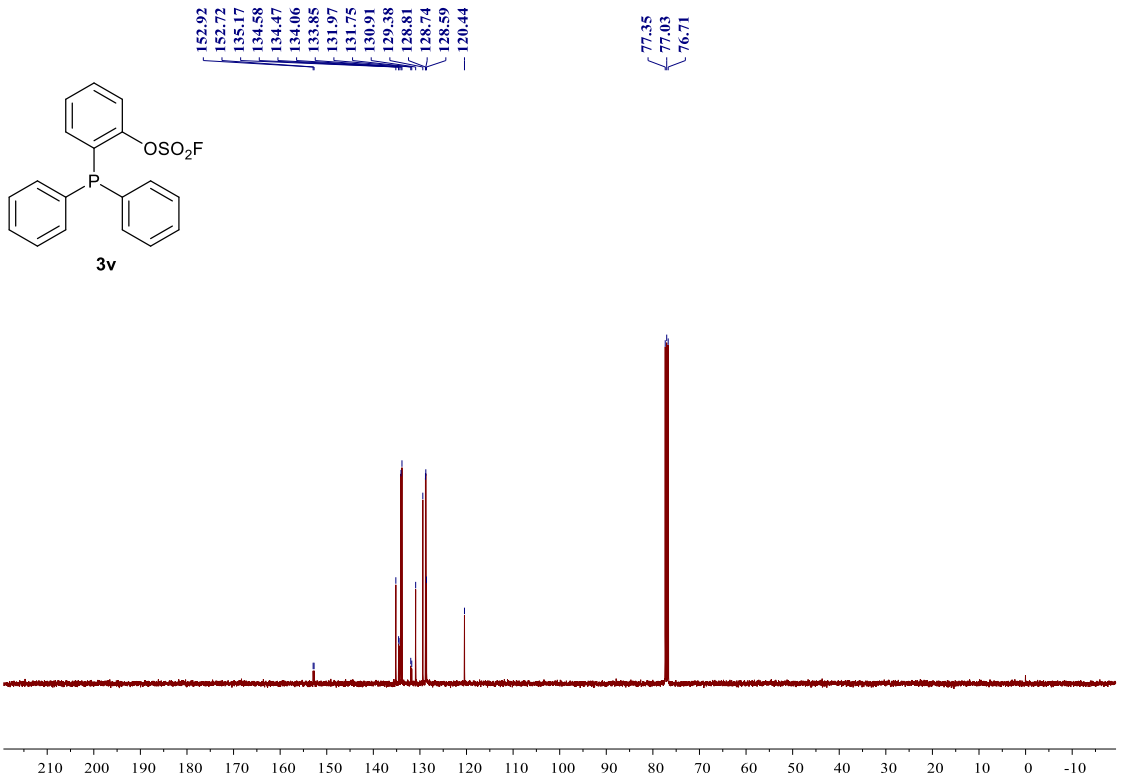
^{13}C NMR Spectrum for Compound **3u** (100 MHz, $CDCl_3$)



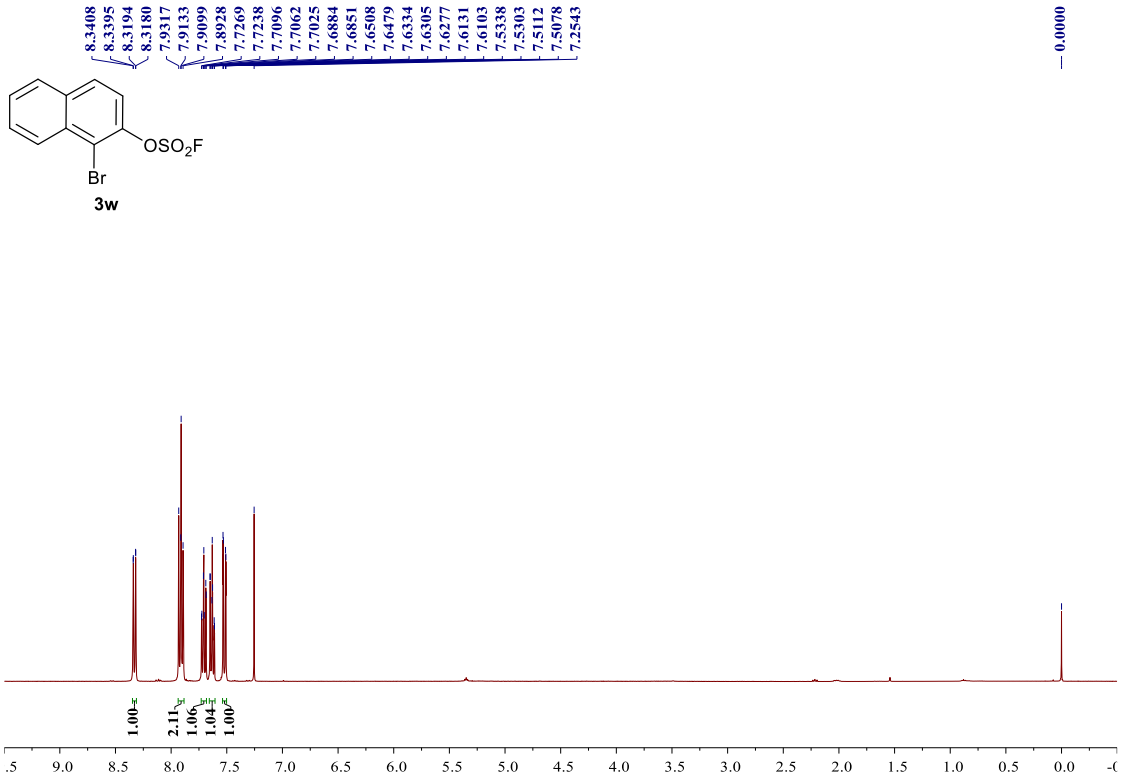
^{1}H NMR Spectrum for Compound **3v** (400 MHz, $CDCl_3$)



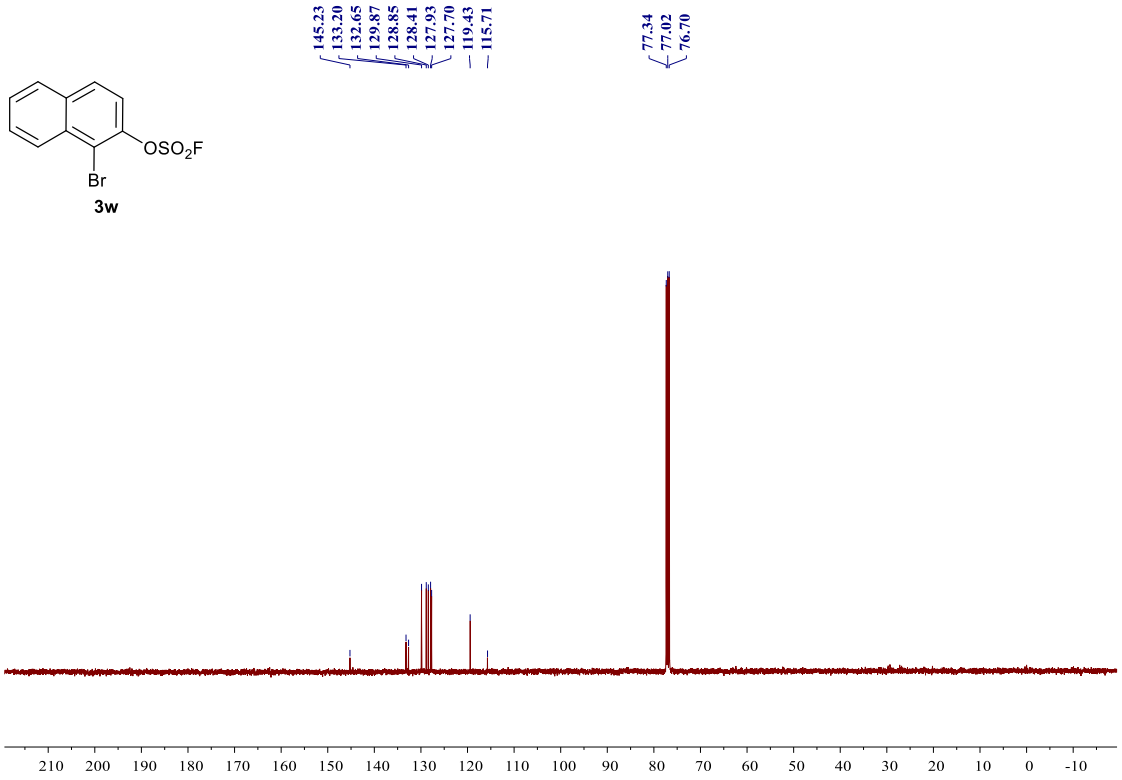
^{13}C NMR Spectrum for Compound **3v** (100 MHz, $CDCl_3$)



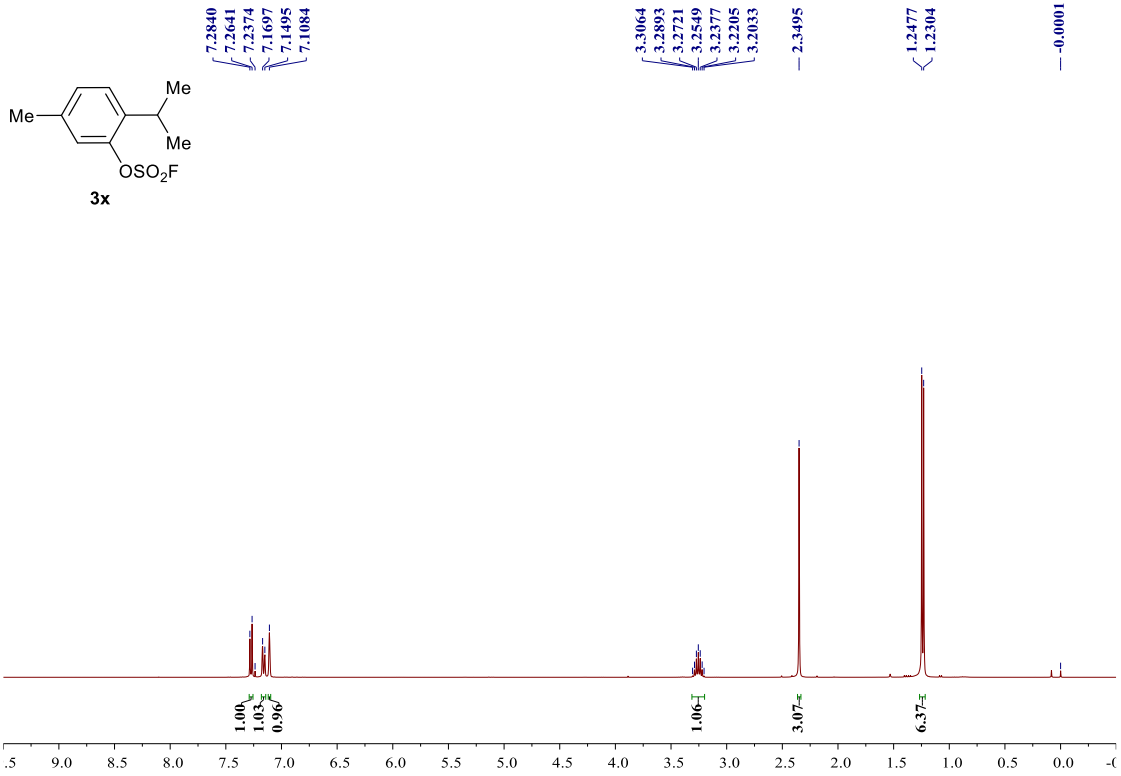
^{1}H NMR Spectrum for Compound **3w** (400 MHz, $CDCl_3$)



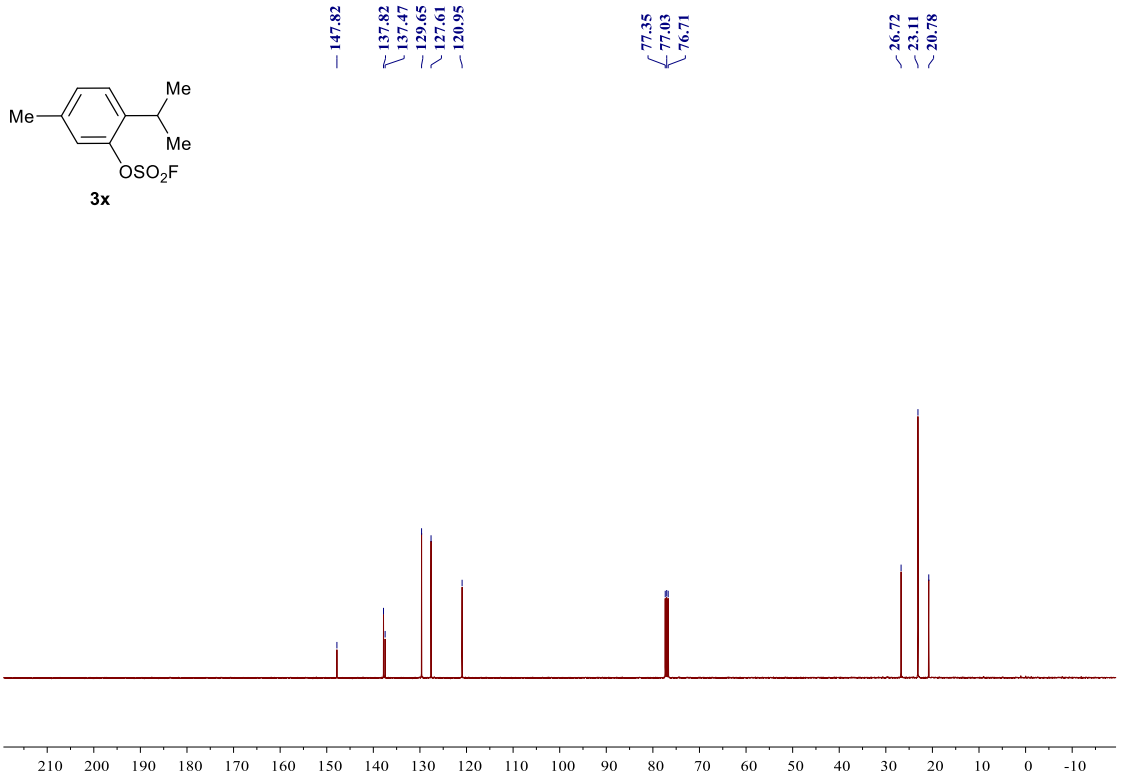
^{13}C NMR Spectrum for Compound **3w** (100 MHz, $CDCl_3$)



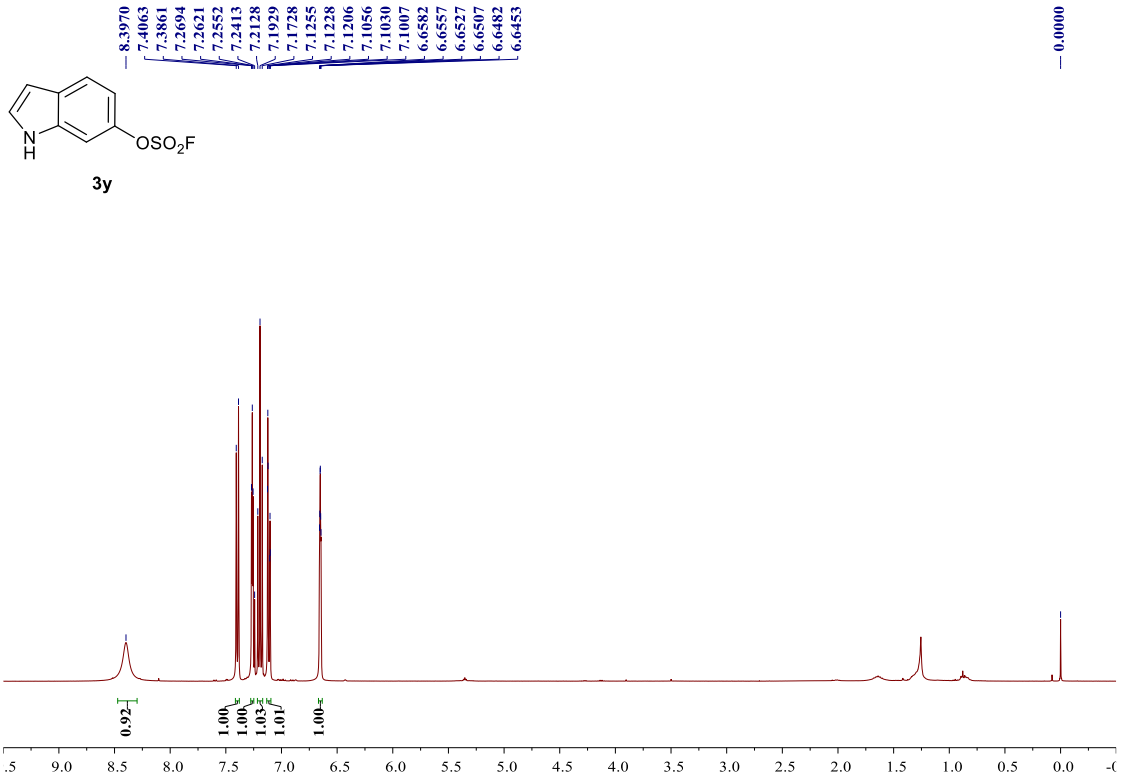
^{1}H NMR Spectrum for Compound **3x** (400 MHz, $CDCl_3$)



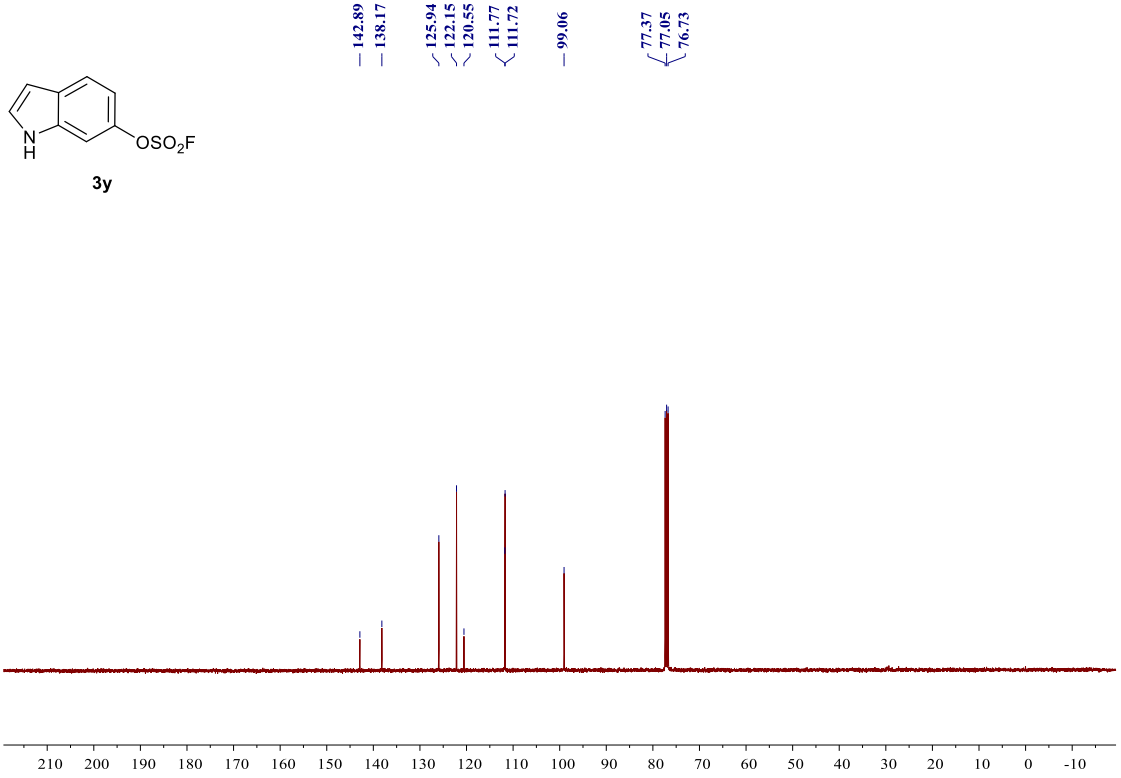
^{13}C NMR Spectrum for Compound **3x** (100 MHz, $CDCl_3$)



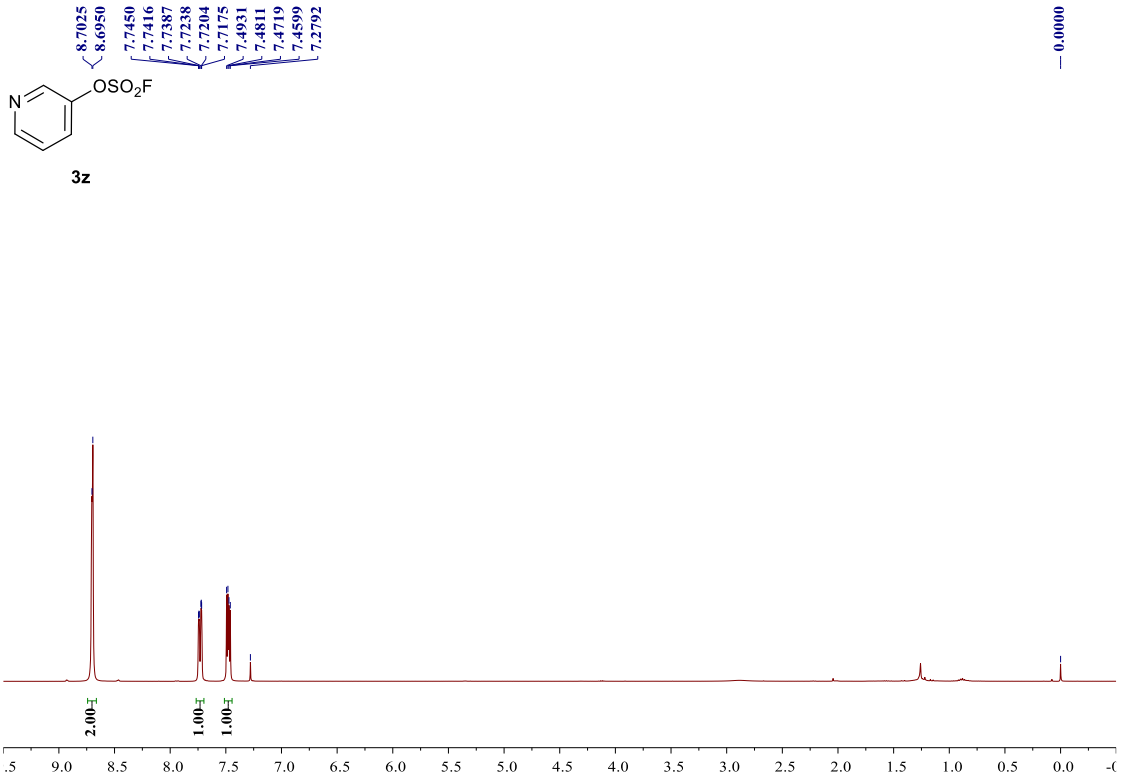
^{1}H NMR Spectrum for Compound **3y** (400 MHz, $CDCl_3$)



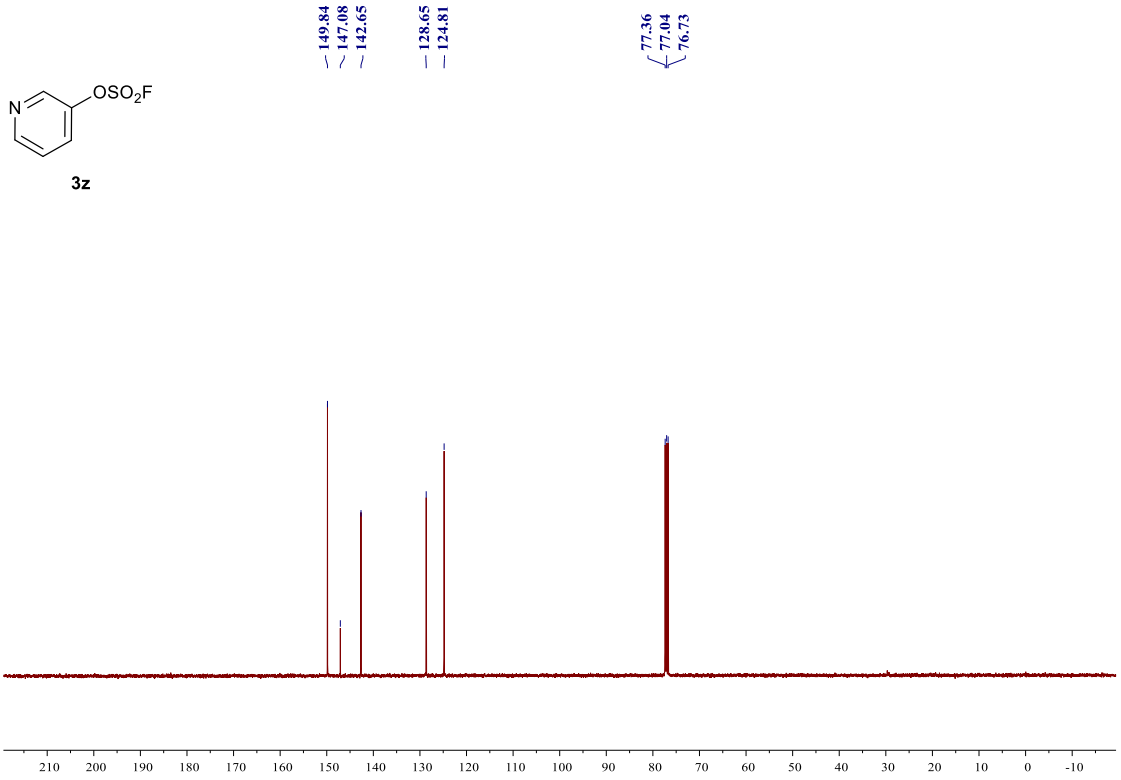
^{13}C NMR Spectrum for Compound **3y** (100 MHz, $CDCl_3$)



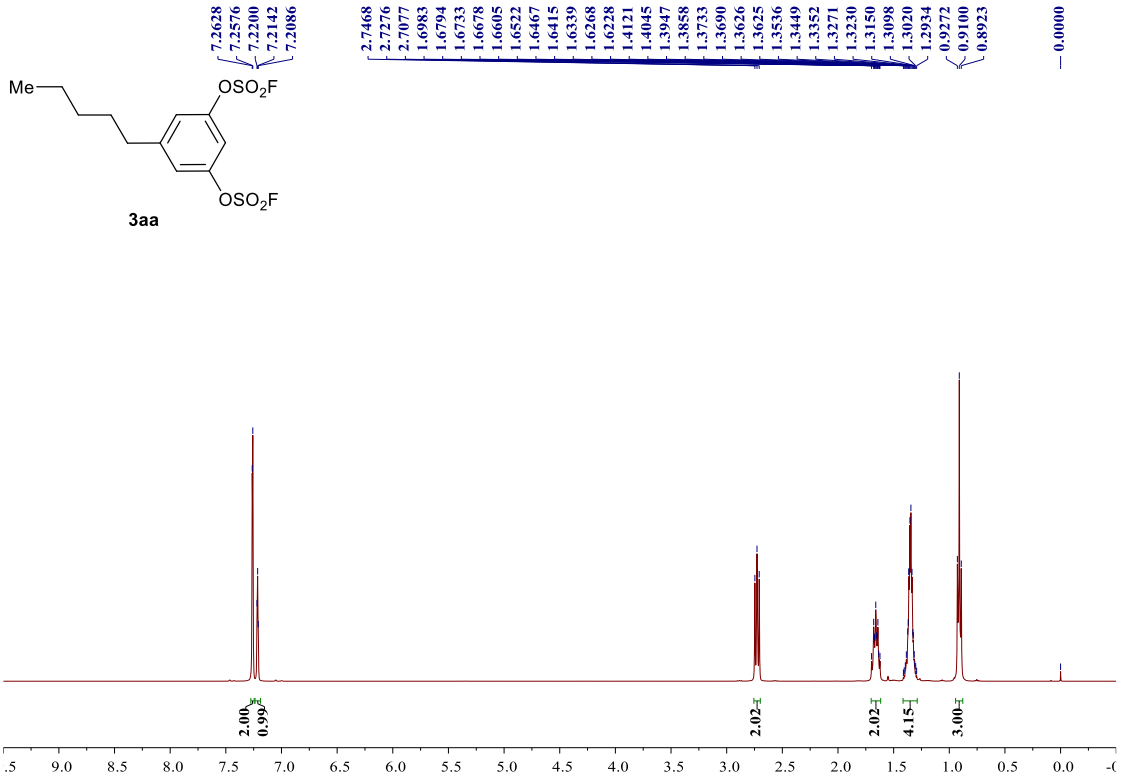
^{1}H NMR Spectrum for Compound **3z** (400 MHz, $CDCl_3$)



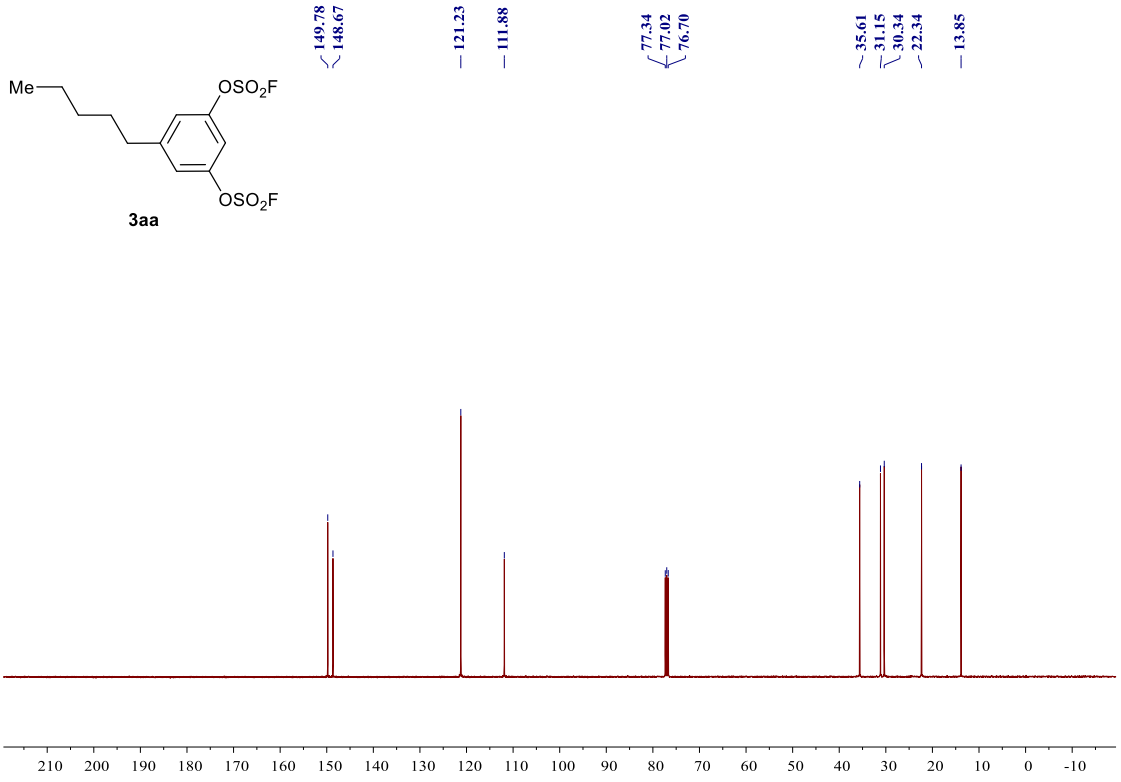
^{13}C NMR Spectrum for Compound **3z** (100 MHz, $CDCl_3$)



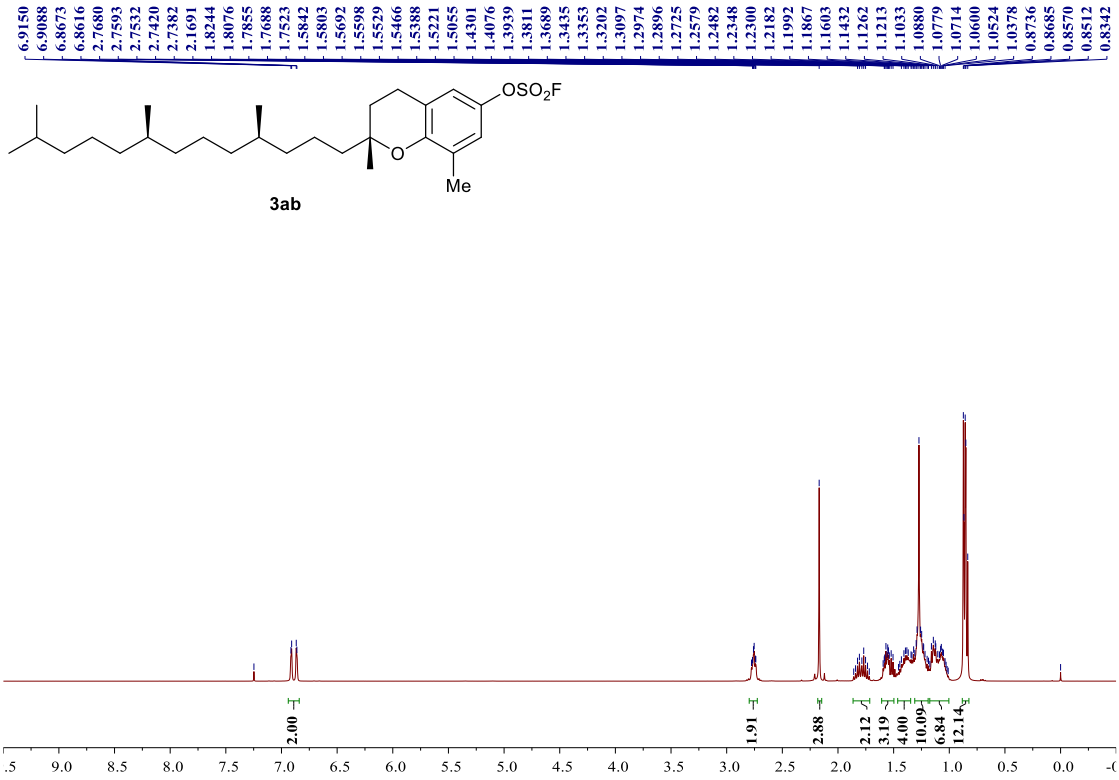
^{1}H NMR Spectrum for Compound **3aa** (400 MHz, $CDCl_3$)



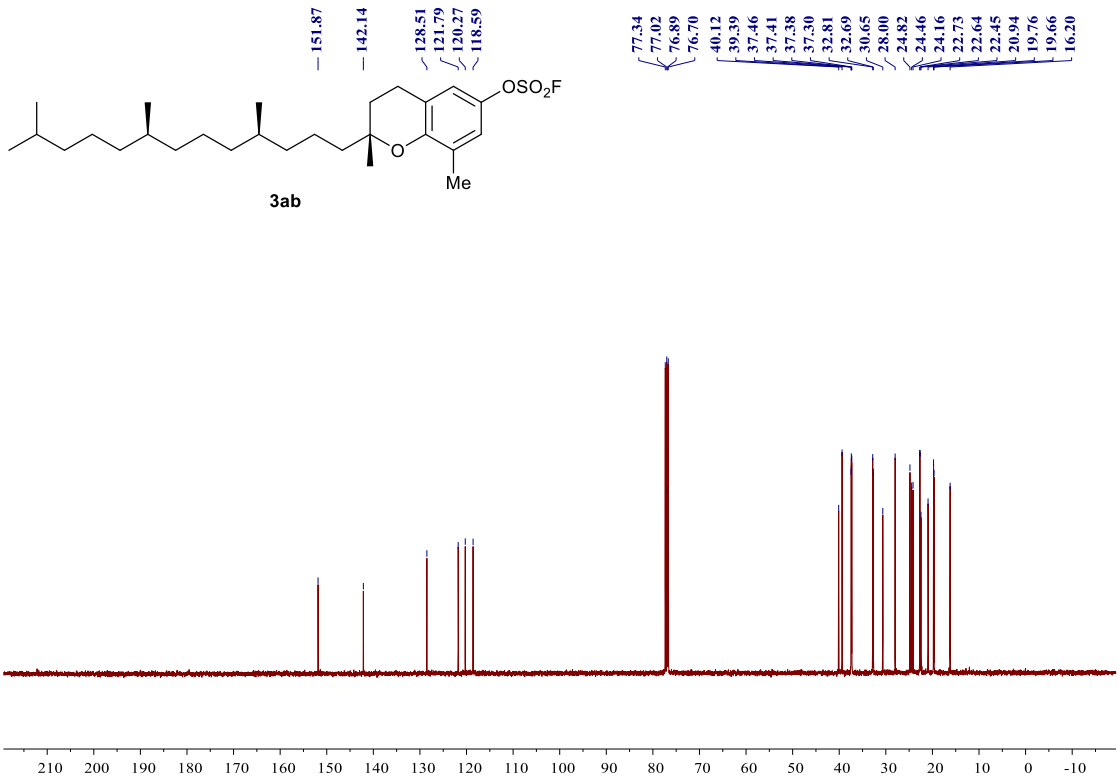
^{13}C NMR Spectrum for Compound **3aa** (100 MHz, $CDCl_3$)



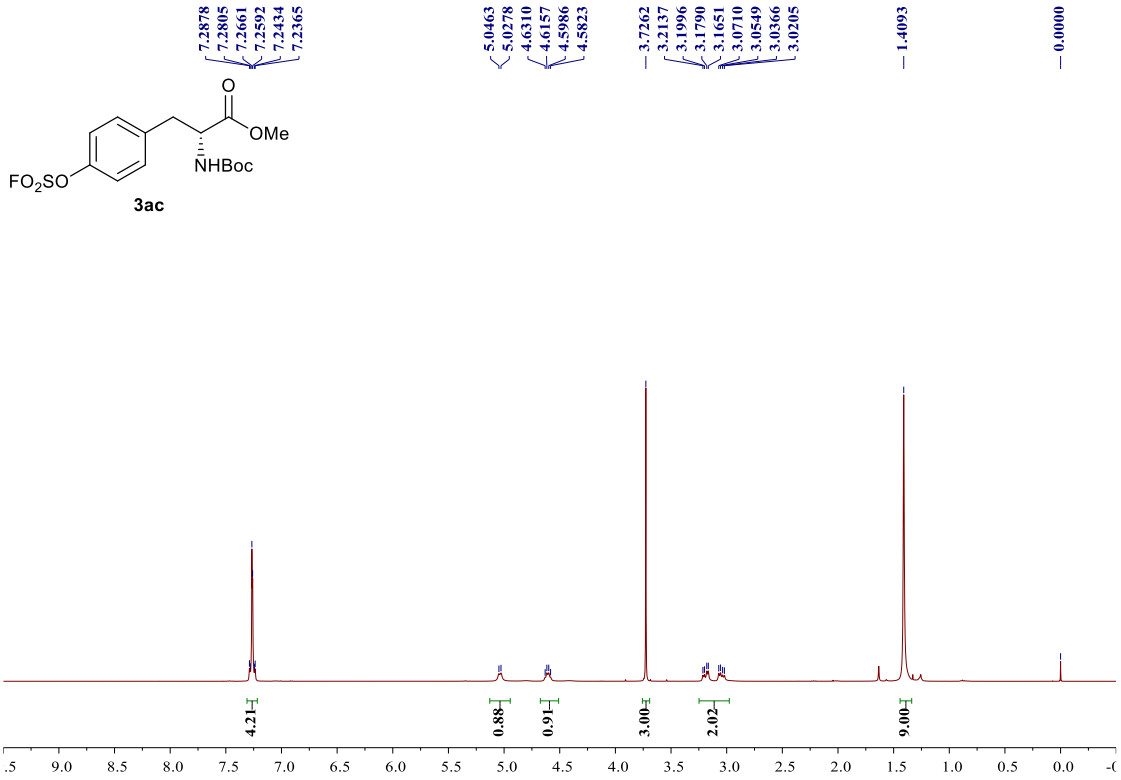
^{1}H NMR Spectrum for Compound **3ab** (400 MHz, $CDCl_3$)



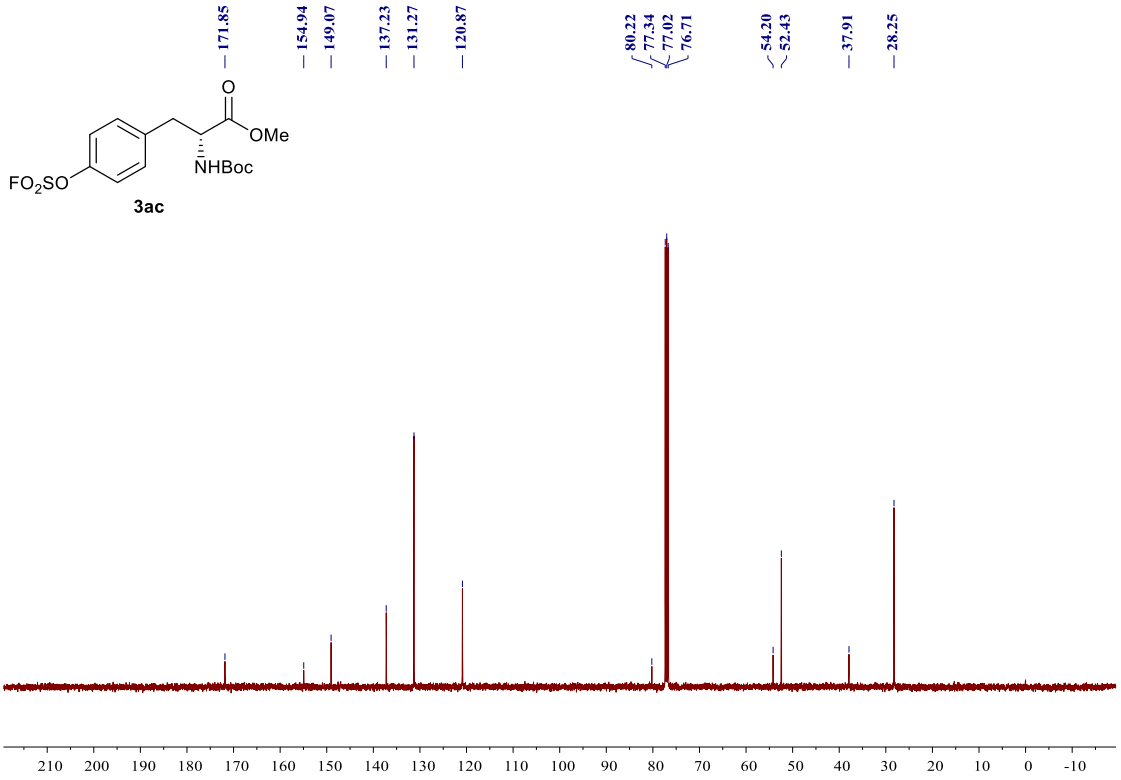
^{13}C NMR Spectrum for Compound **3ab** (100 MHz, $CDCl_3$)



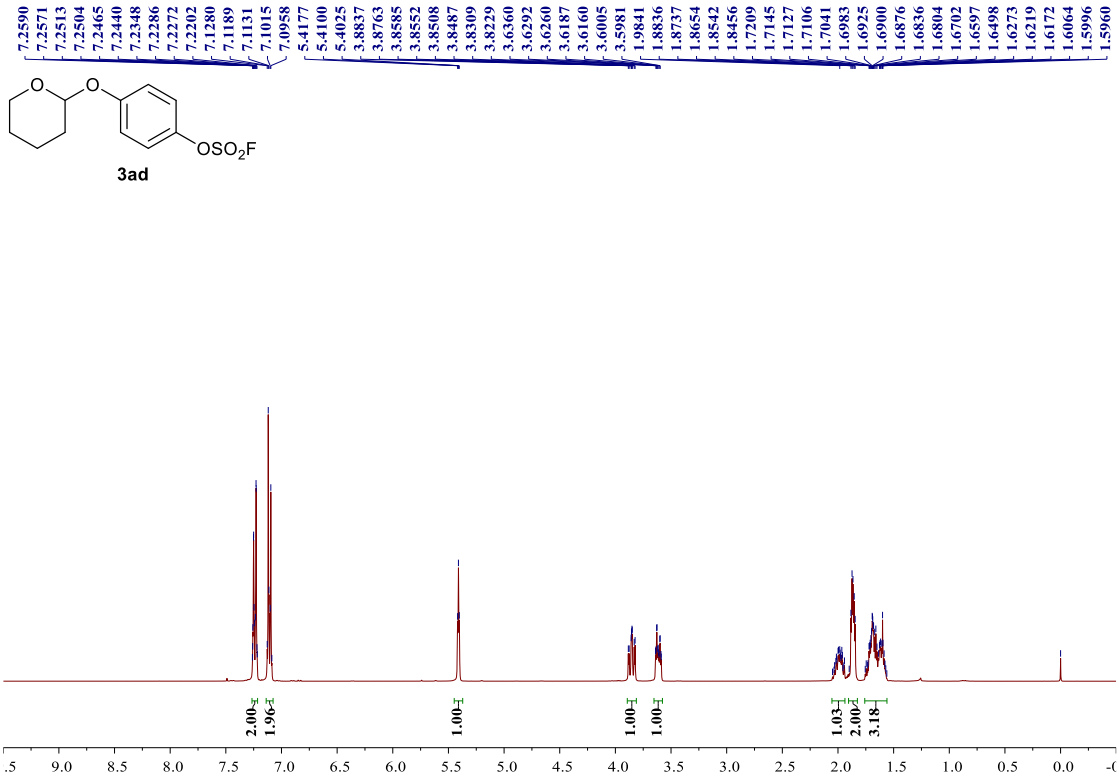
^{1}H NMR Spectrum for Compound **3ac** (400 MHz, $CDCl_3$)



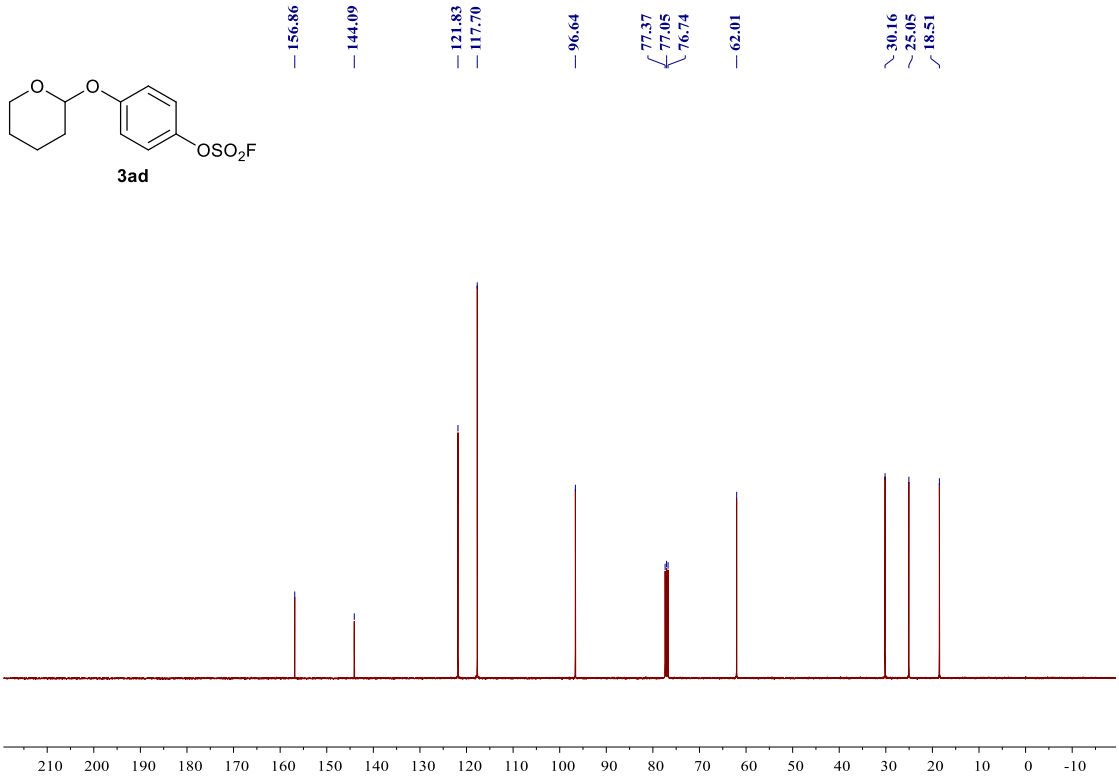
^{13}C NMR Spectrum for Compound **3ac** (100 MHz, $CDCl_3$)



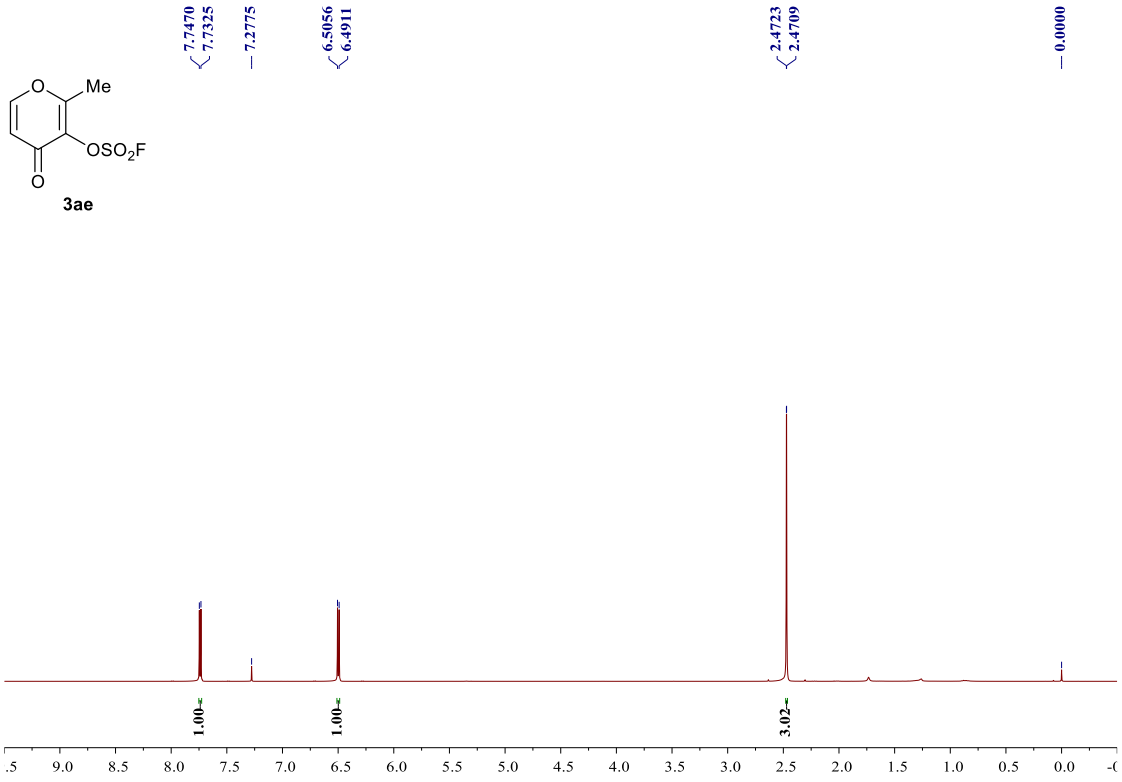
^{1}H NMR Spectrum for Compound **3ad** (400 MHz, $CDCl_3$)



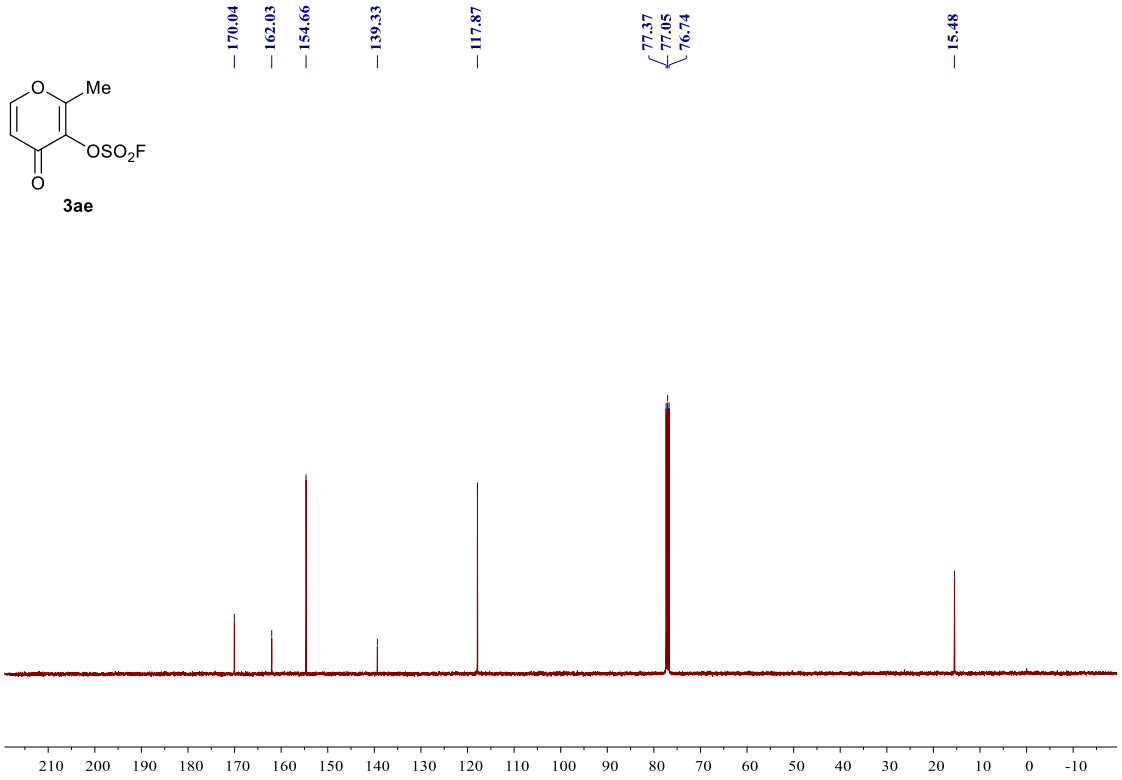
^{13}C NMR Spectrum for Compound **3ad** (100 MHz, $CDCl_3$)



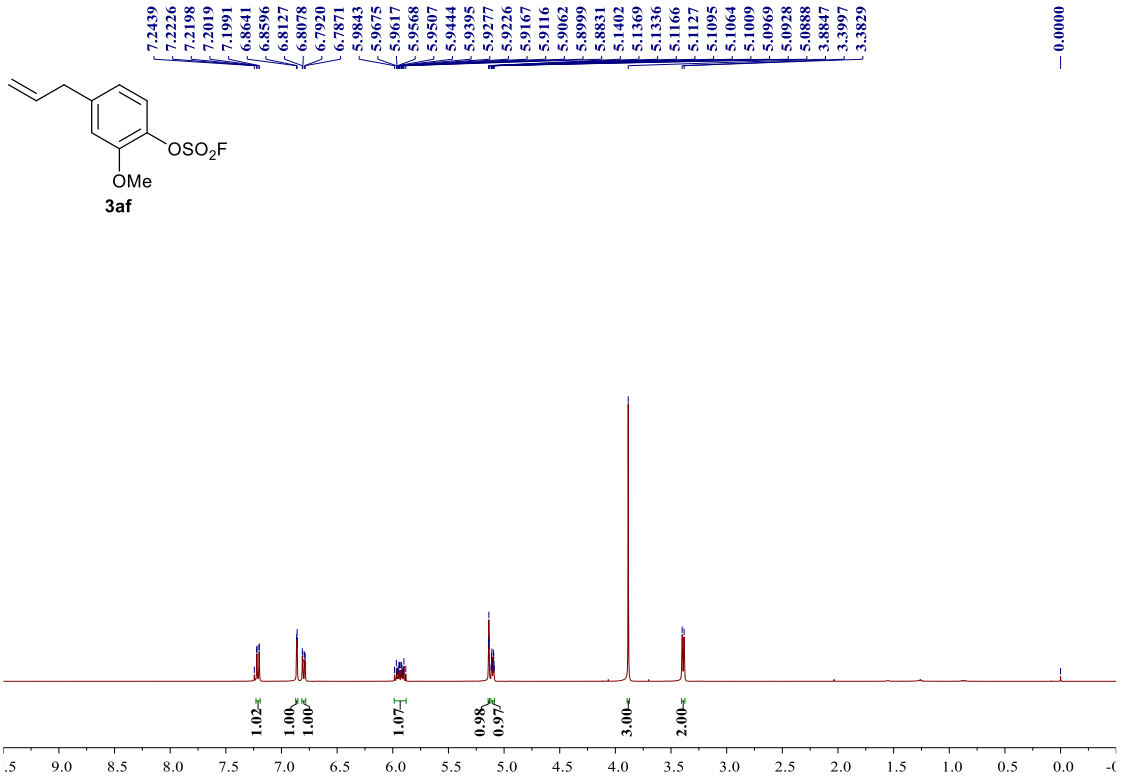
^{1}H NMR Spectrum for Compound **3ae** (400 MHz, $CDCl_3$)



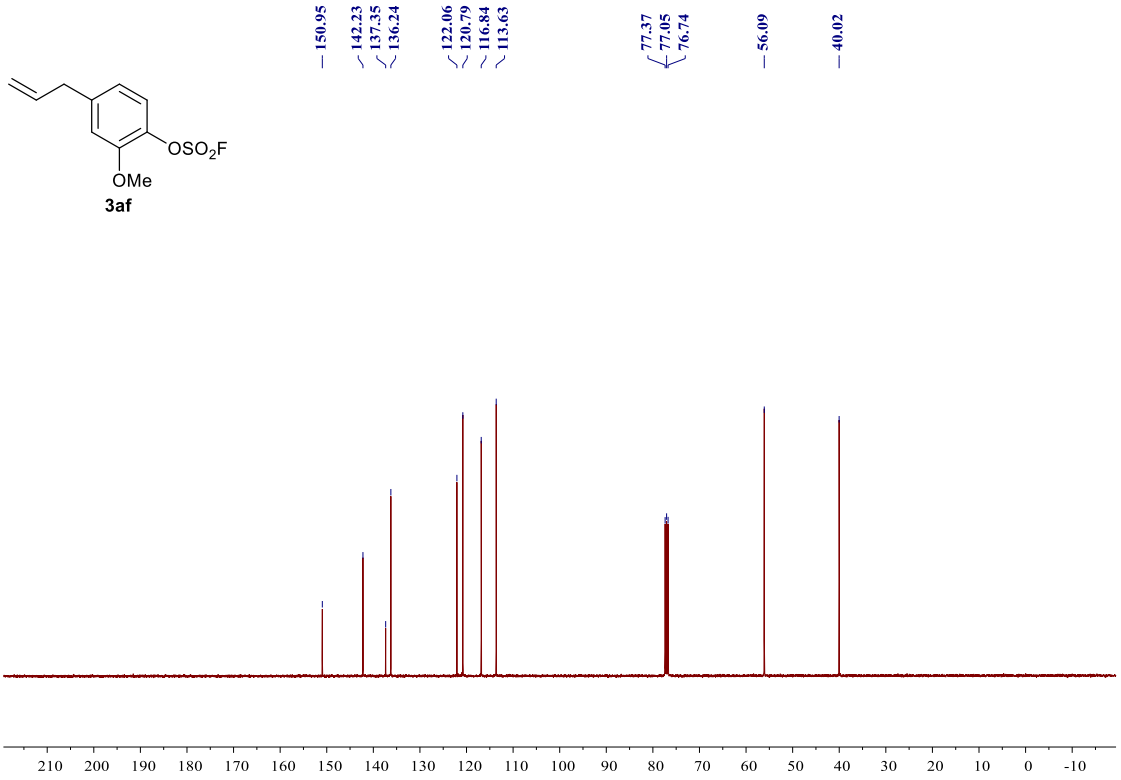
^{13}C NMR Spectrum for Compound **3ae** (100 MHz, $CDCl_3$)



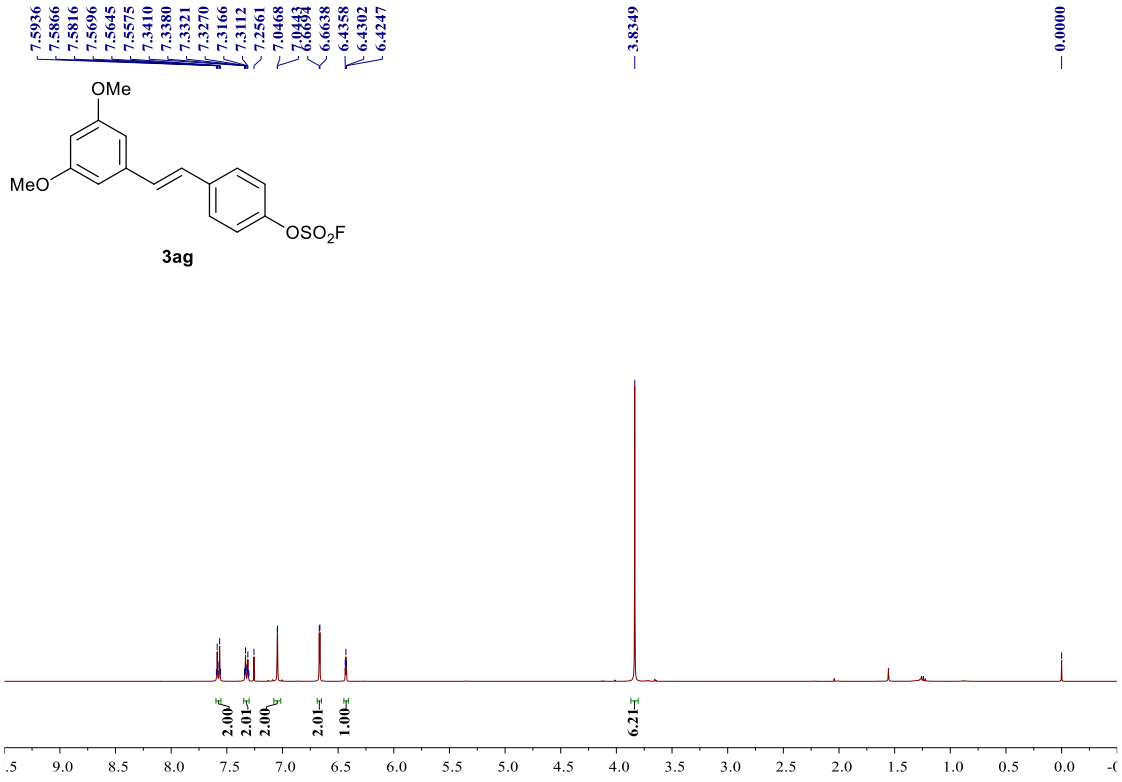
^{1}H NMR Spectrum for Compound **3af** (400 MHz, $CDCl_3$)



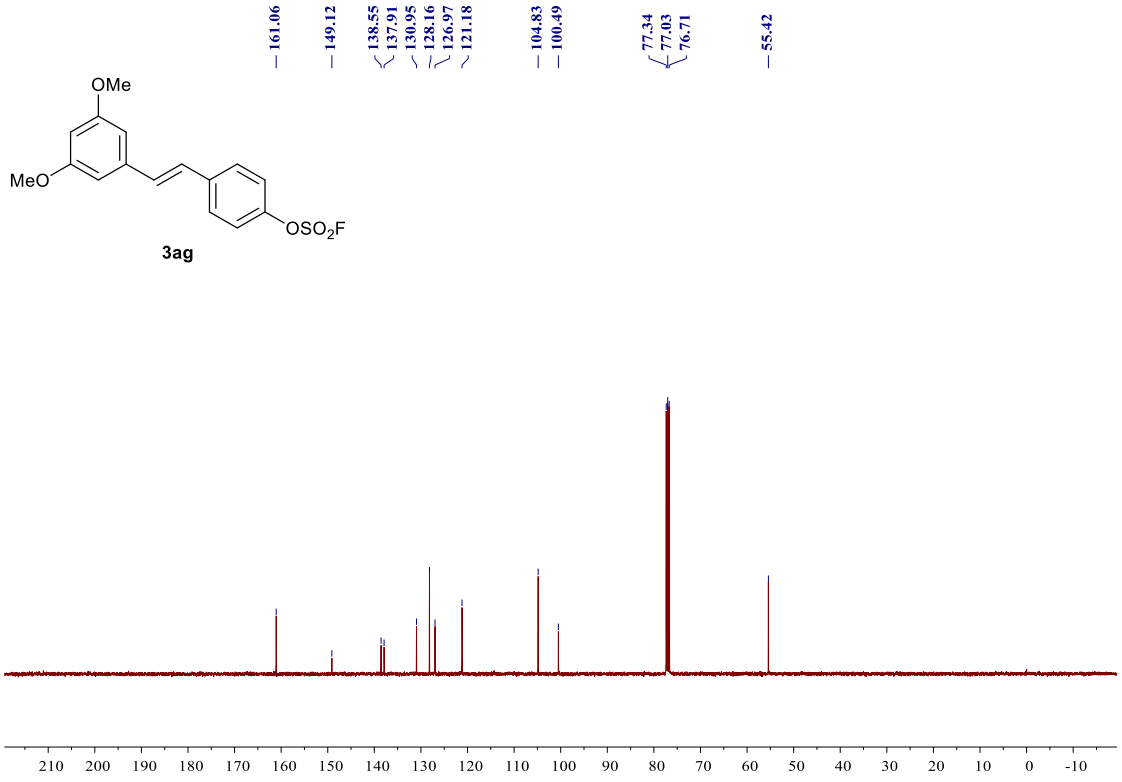
^{13}C NMR Spectrum for Compound **3af** (100 MHz, $CDCl_3$)



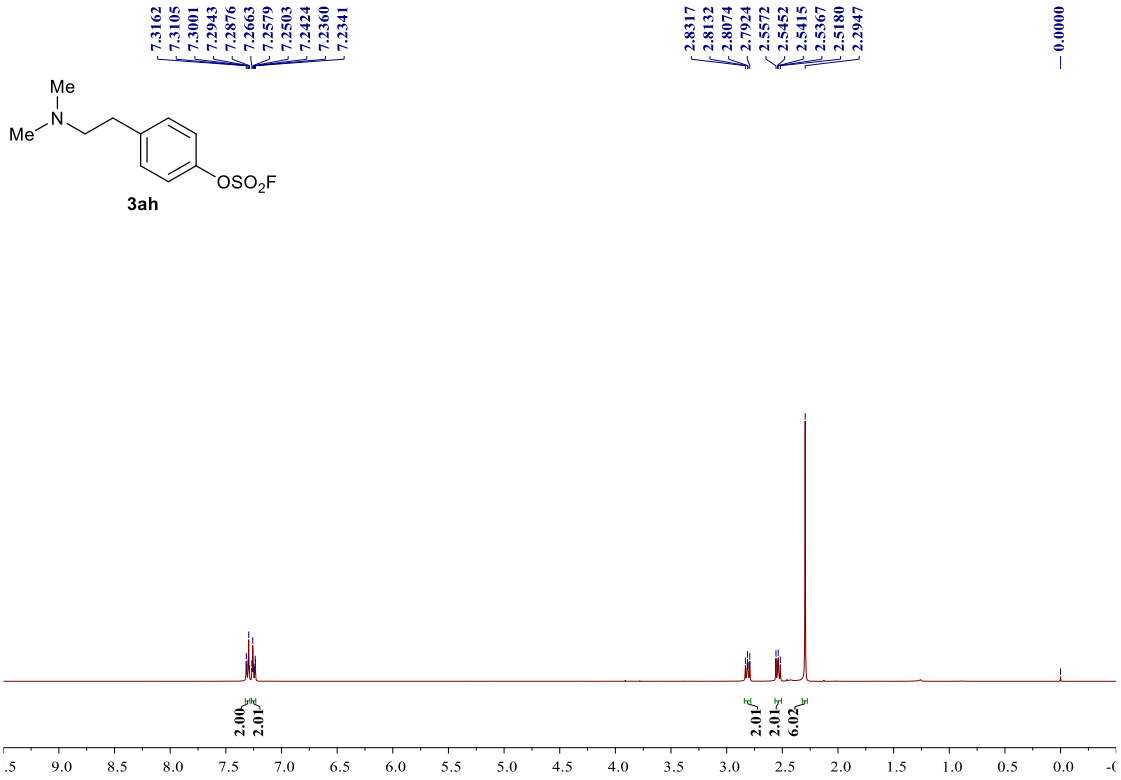
^{1}H NMR Spectrum for Compound **3ag** (400 MHz, $CDCl_3$)



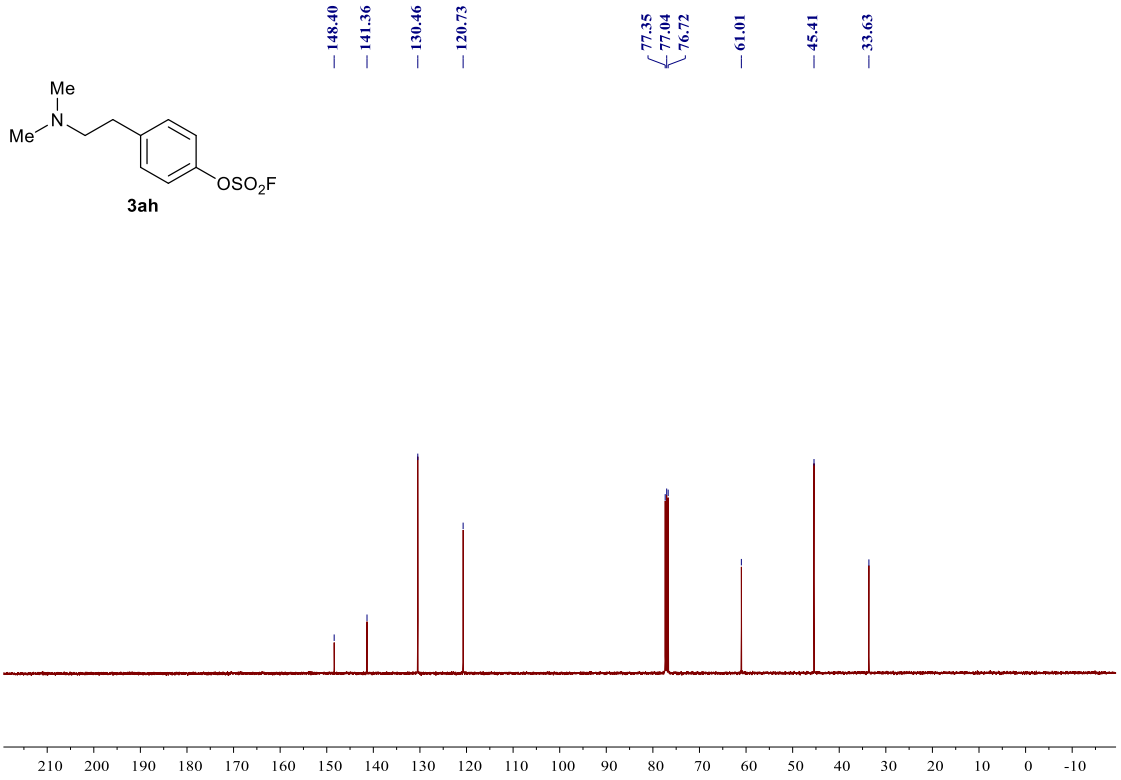
^{13}C NMR Spectrum for Compound **3ag** (100 MHz, $CDCl_3$)



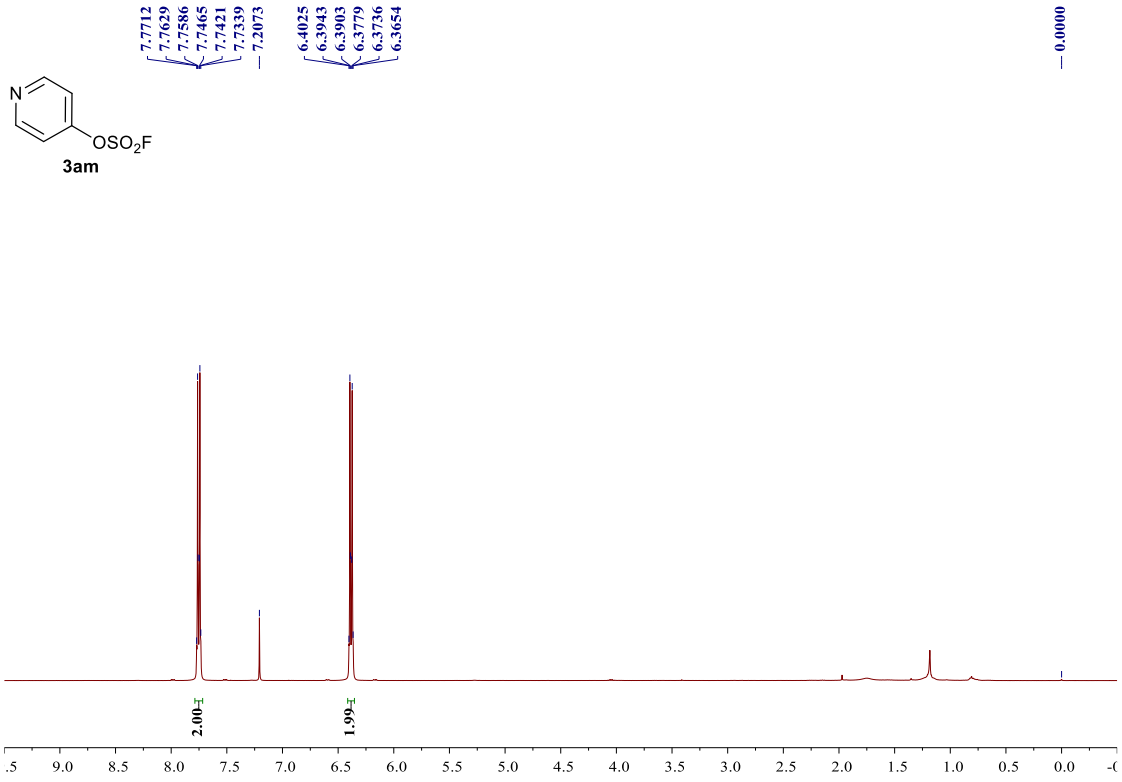
^{1}H NMR Spectrum for Compound **3ah** (400 MHz, $CDCl_3$)



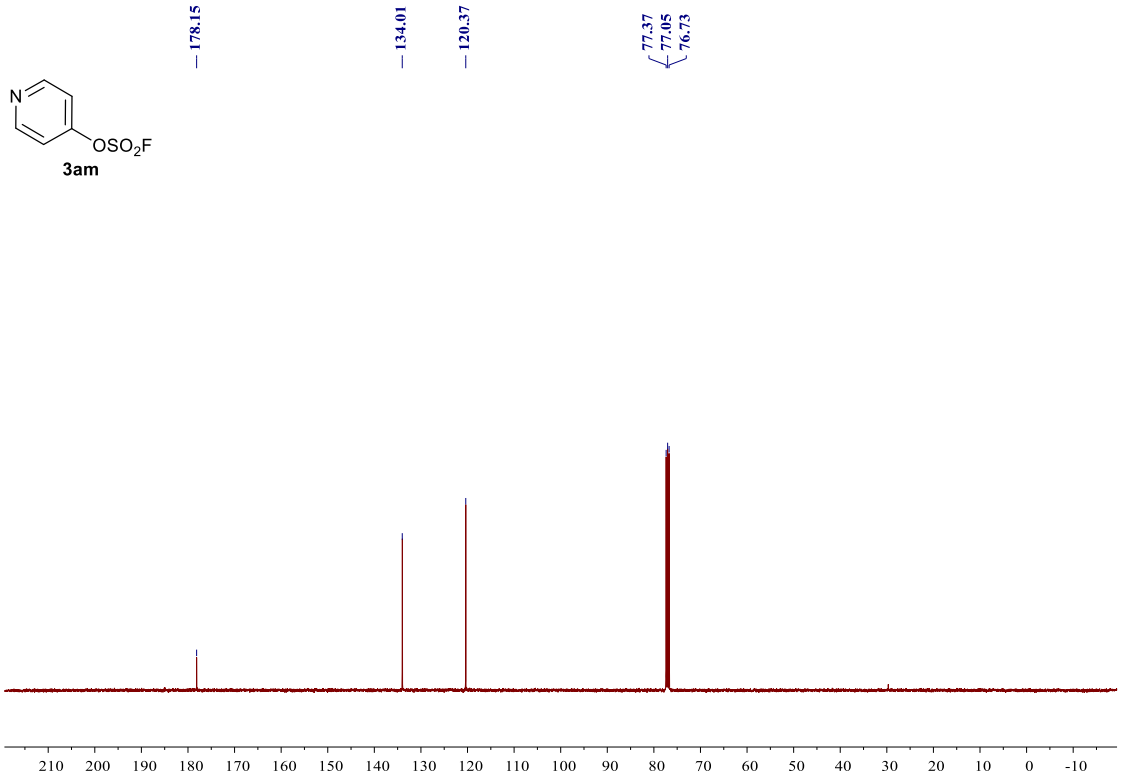
^{13}C NMR Spectrum for Compound **3ah** (100 MHz, $CDCl_3$)



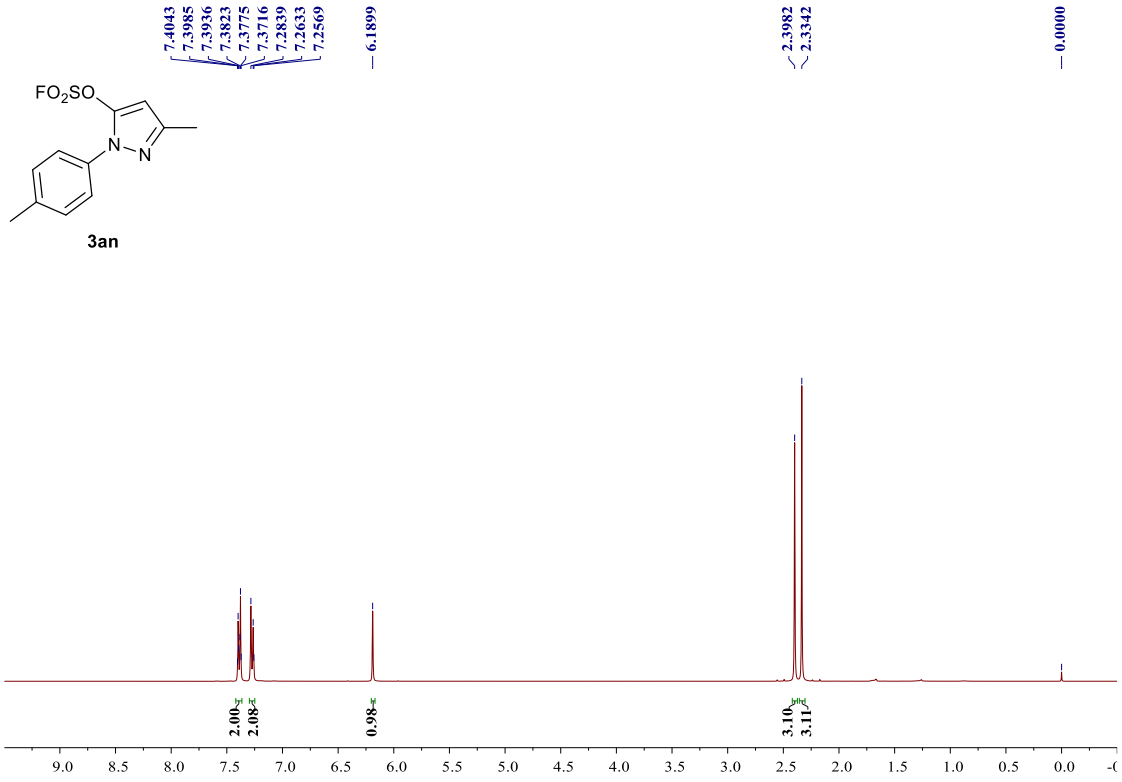
^{1}H NMR Spectrum for Compound **3am** (400 MHz, $CDCl_3$)



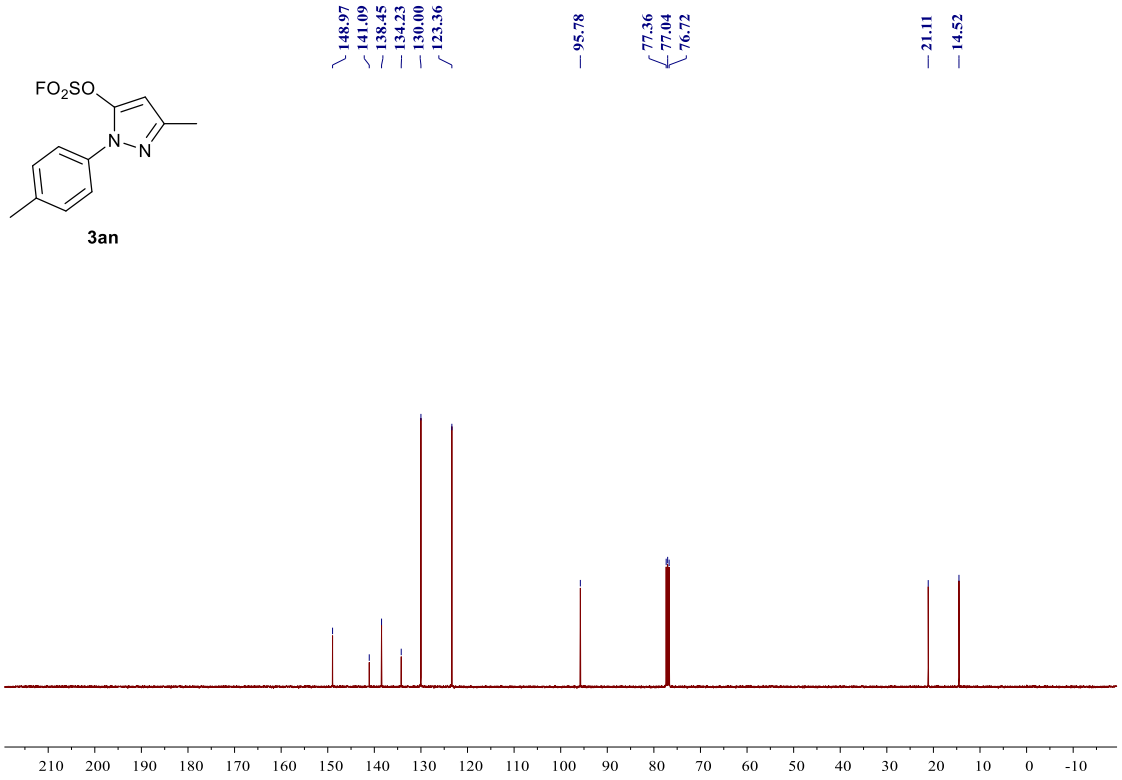
^{13}C NMR Spectrum for Compound **3am** (100 MHz, $CDCl_3$)



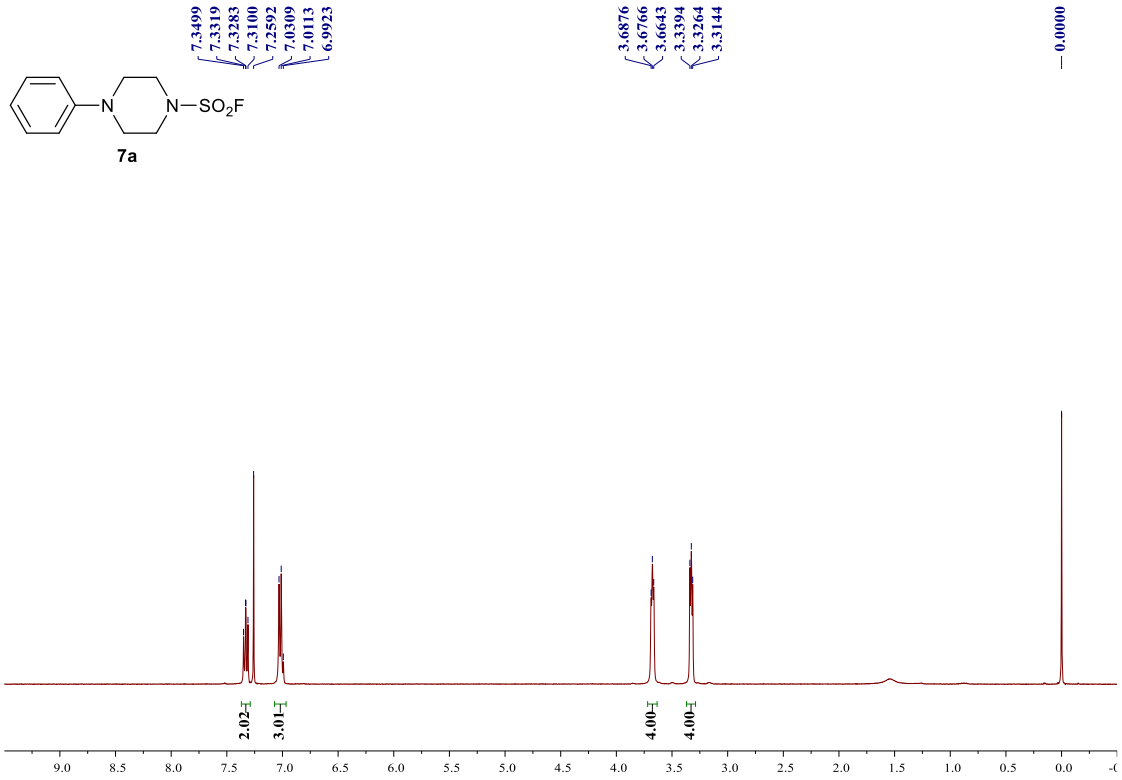
^{1}H NMR Spectrum for Compound **3an** (400 MHz, $CDCl_3$)



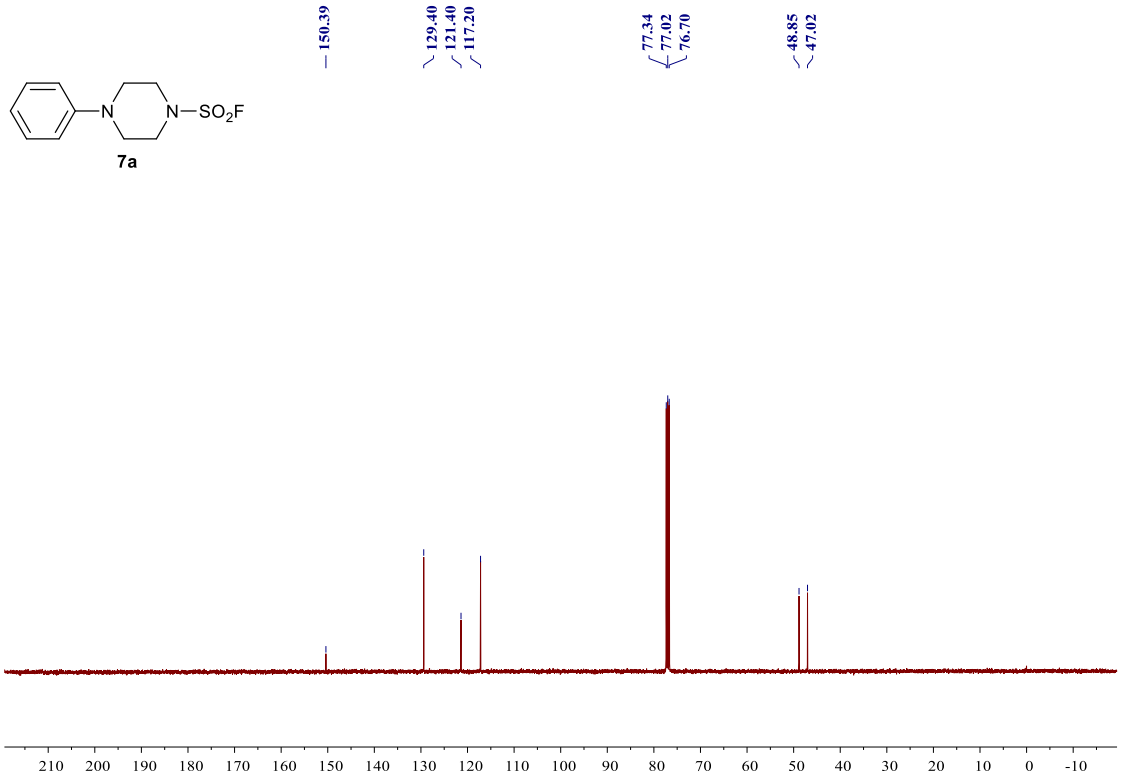
^{13}C NMR Spectrum for Compound **3an** (100 MHz, $CDCl_3$)



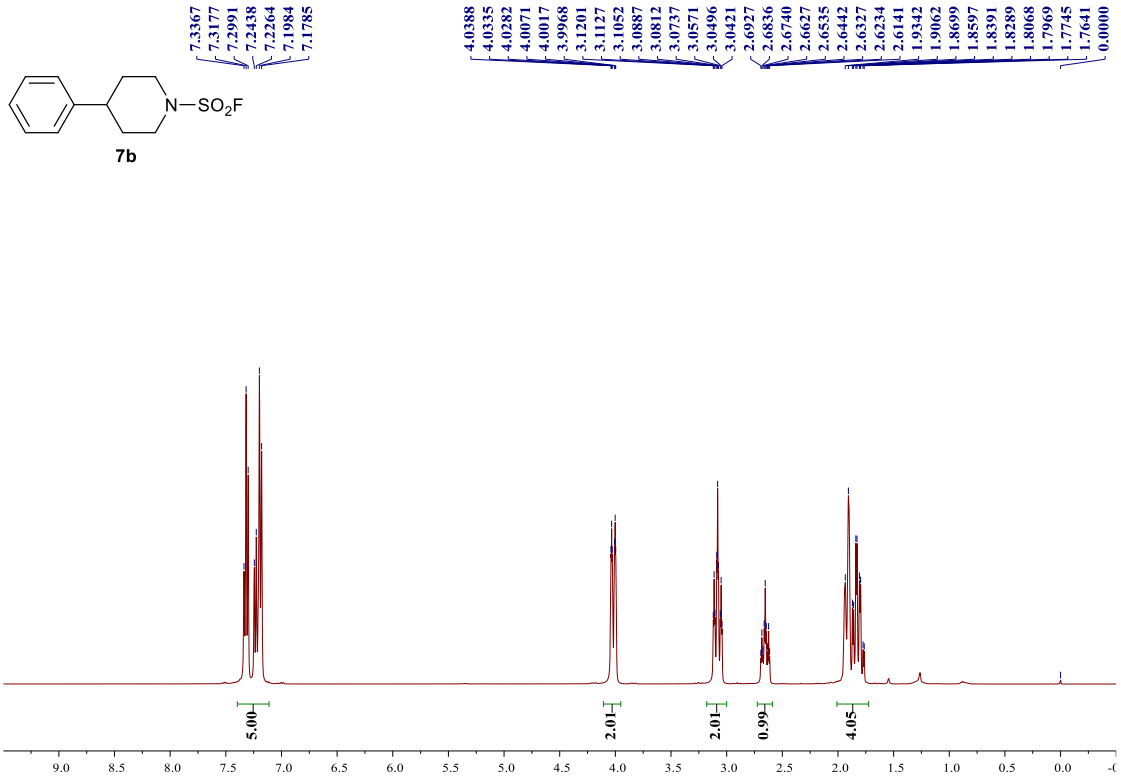
^{1}H NMR Spectrum for Compound **7a** (400 MHz, $CDCl_3$)



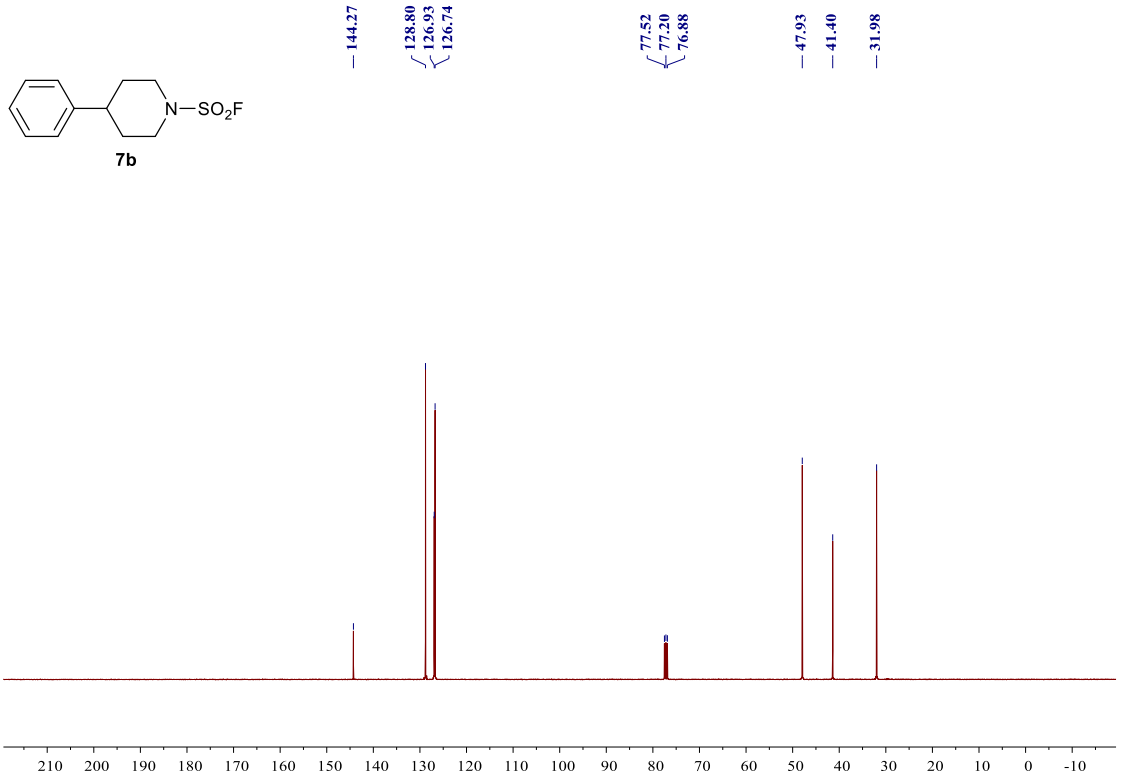
^{13}C NMR Spectrum for Compound **7a** (100 MHz, $CDCl_3$)



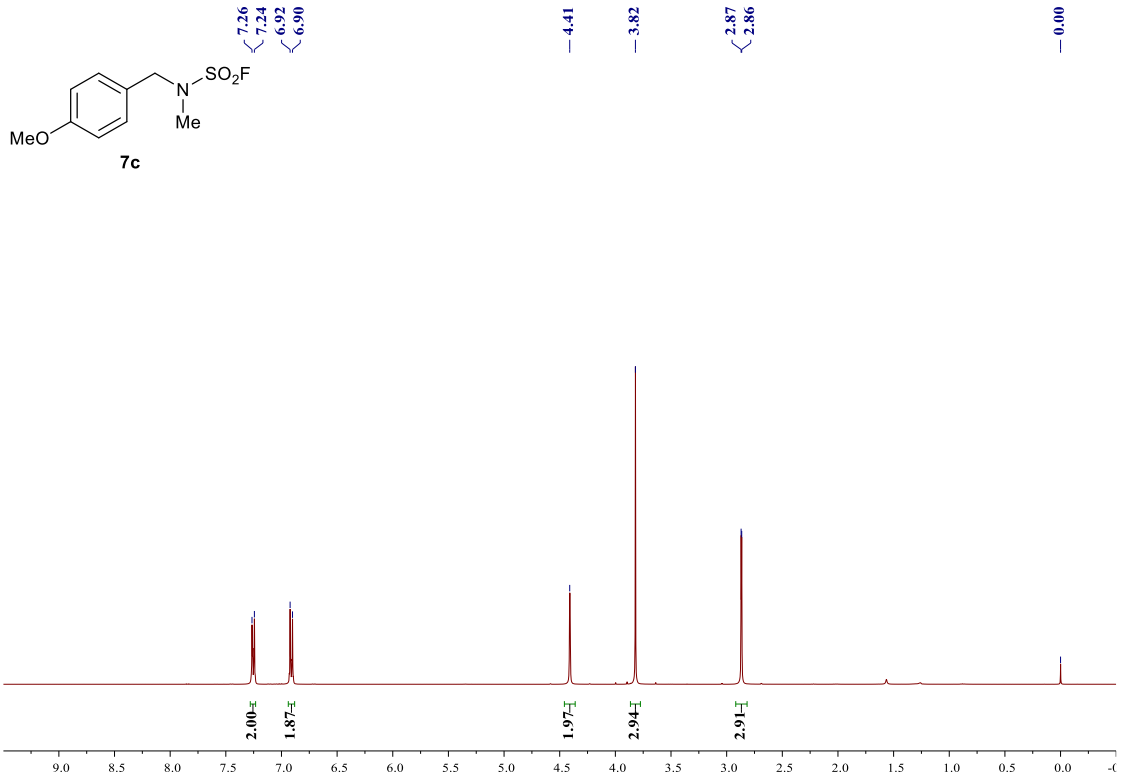
^{1}H NMR Spectrum for Compound **7b** (400 MHz, $CDCl_3$)



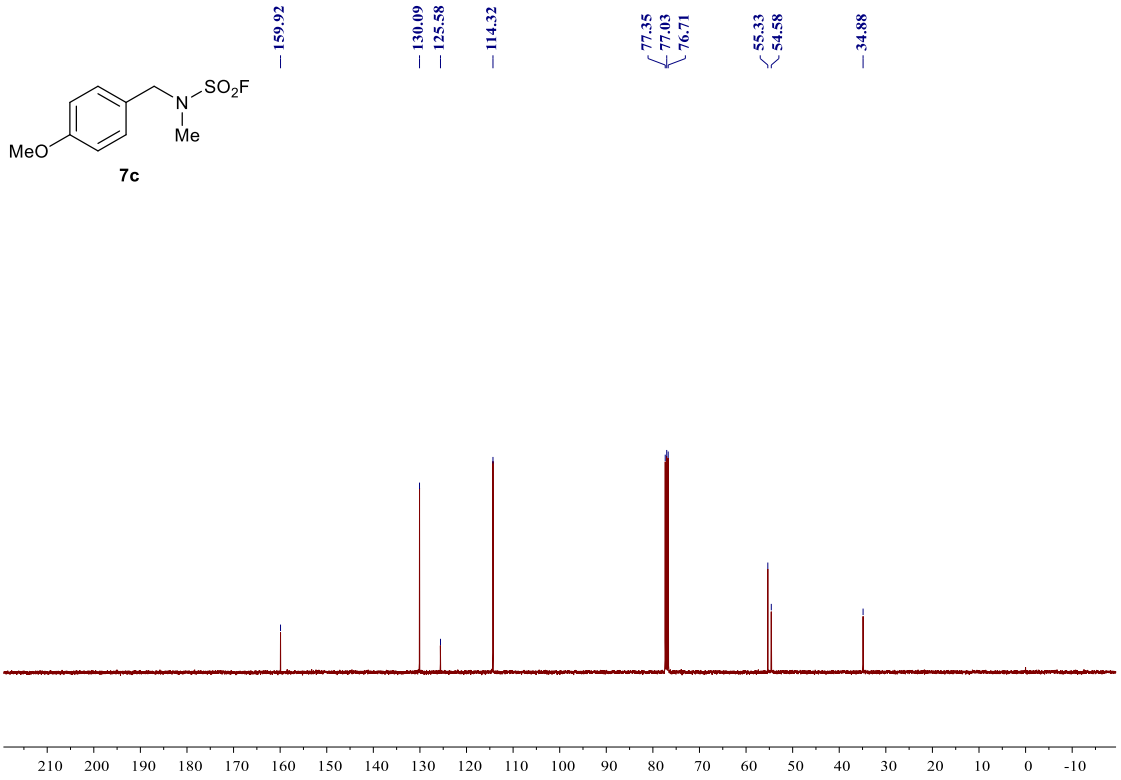
^{13}C NMR Spectrum for Compound **7b** (100 MHz, $CDCl_3$)



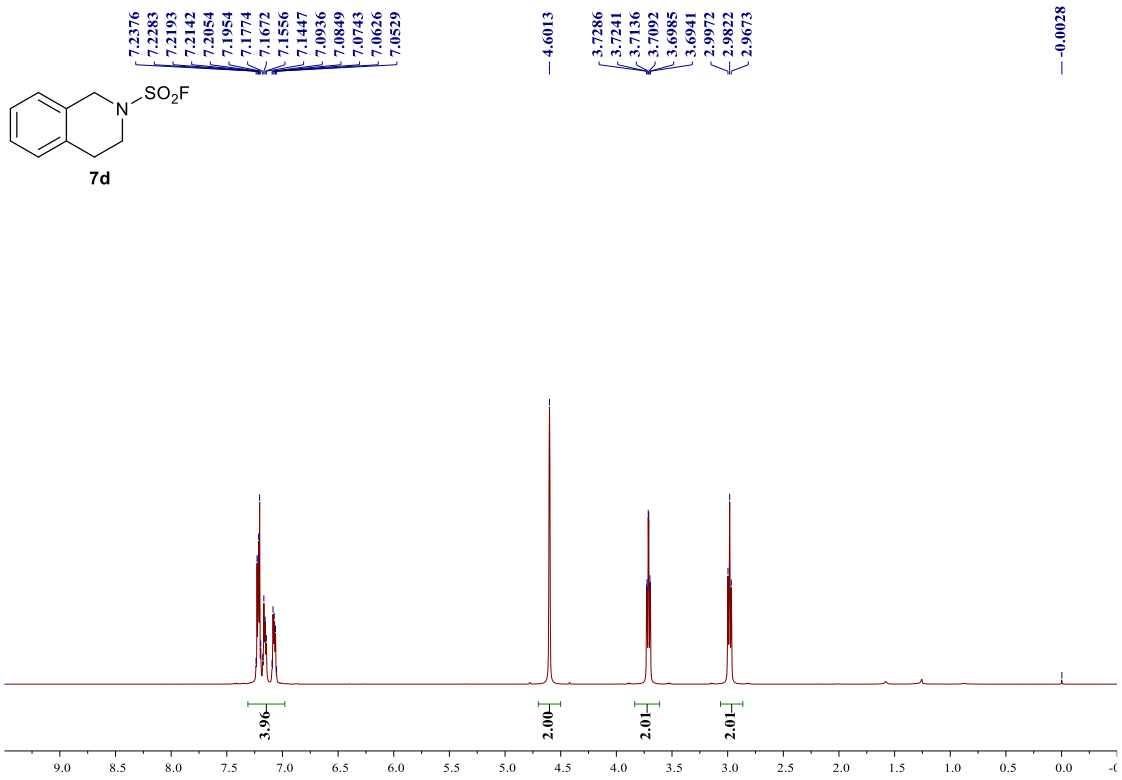
^{1}H NMR Spectrum for Compound **7c** (400 MHz, $CDCl_3$)



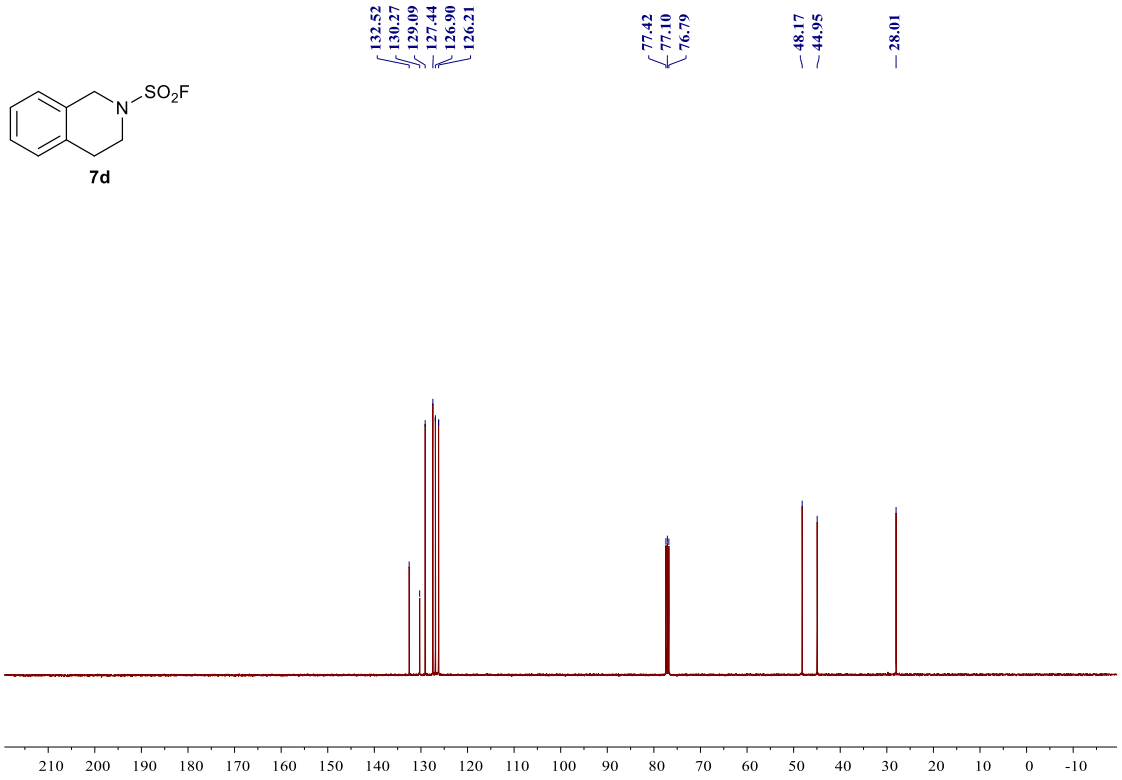
^{13}C NMR Spectrum for Compound **7c** (100 MHz, $CDCl_3$)



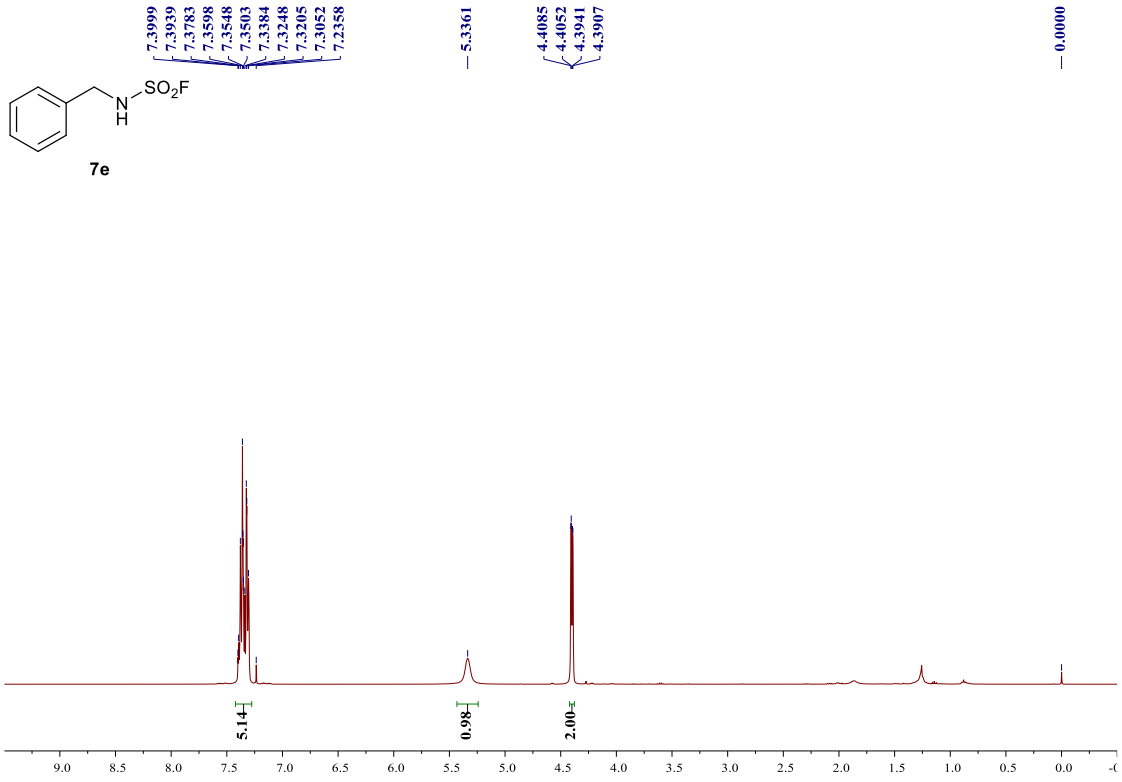
^{1}H NMR Spectrum for Compound **7d** (400 MHz, $CDCl_3$)



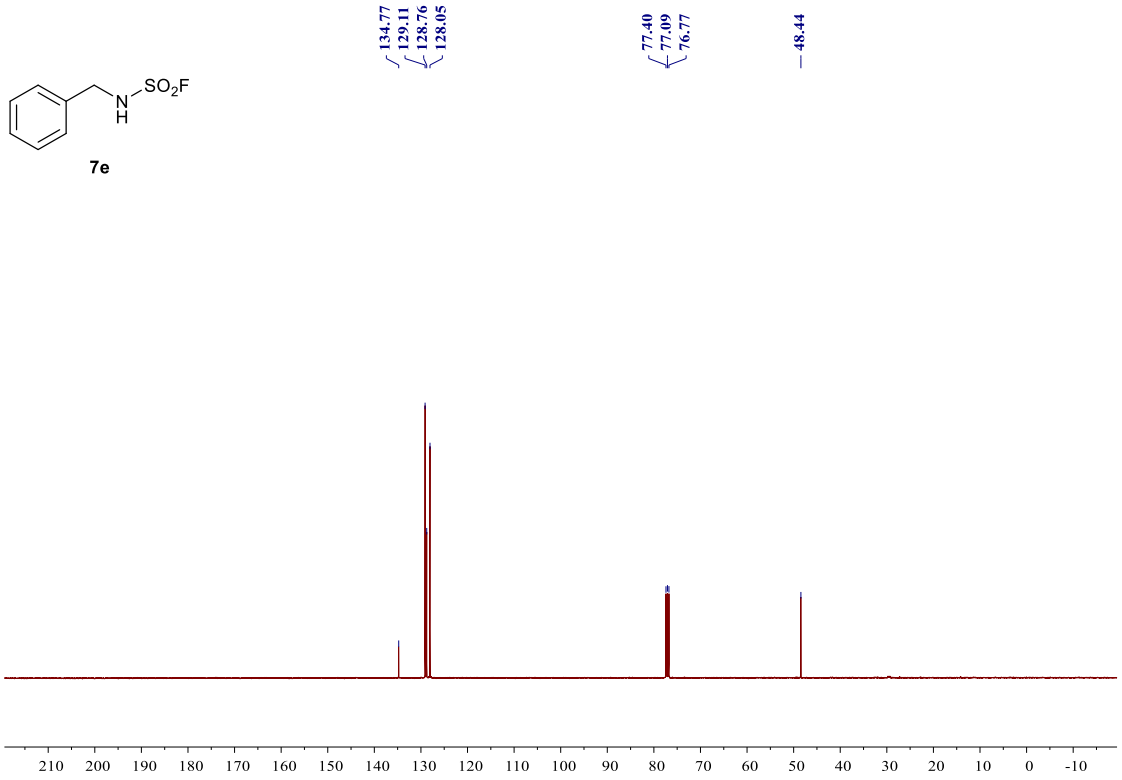
^{13}C NMR Spectrum for Compound **7d** (100 MHz, $CDCl_3$)



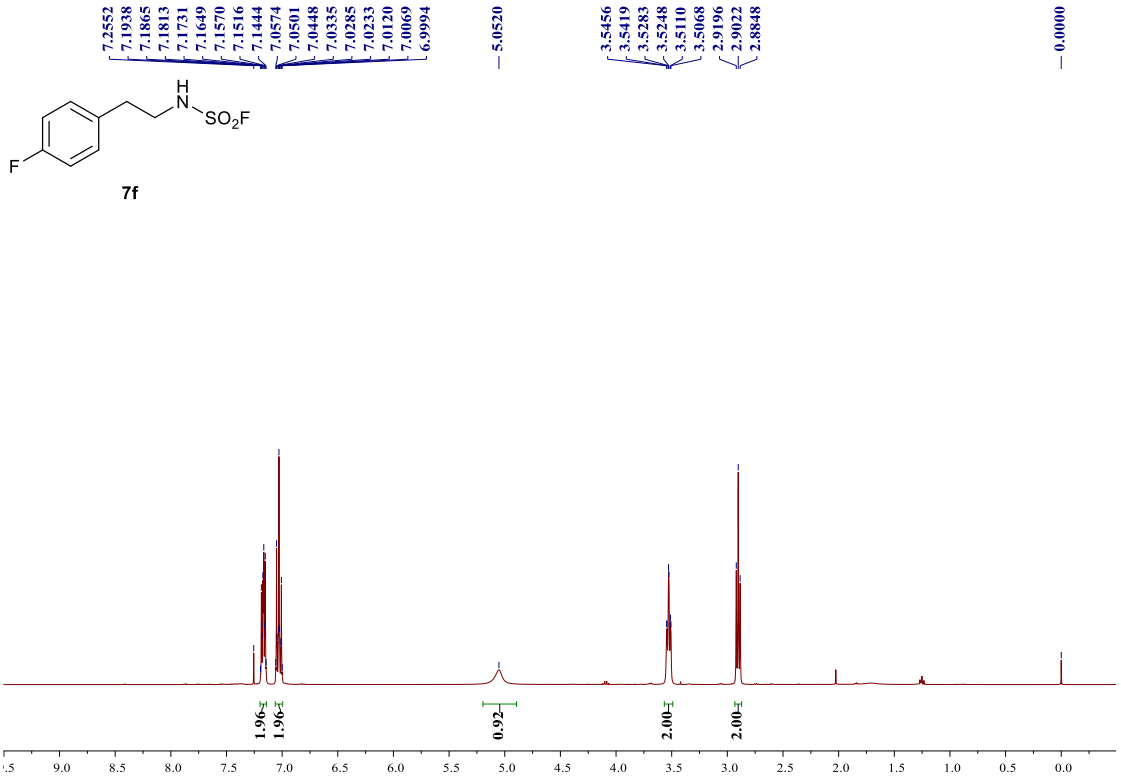
^{1}H NMR Spectrum for Compound **7e** (400 MHz, $CDCl_3$)



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



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



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