Supporting Information

Electrochemical sulfonylation of N-allylbenzamides-triggered Truce-Smiles rearrangement: toward sulfone-containing β-arylethylamines and Saclofen analogues

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1. General informations

Unless otherwise stated, all reagents were obtained from commercial suppliers and used without further purification.

Analytical thin layer chromatography was performed on silica gel aluminum plates with F-254 indicator; spots were visualized by UV light (254 nm) and/or by staining with a $KMnO_4$ solution. Flash column chromatographies were performed on kieselgel 35-70 μm particle sized silica gel (200-400 mesh).

^{1}H, ^{13}C and ^{19}F NMR analyses were recorded on Bruker Avance 300, 400 or 500 spectrometers in $CDCl_3$, CD_3CN or CD_3OD. The chemical shifts (δ) are reported in parts per million (ppm) and were referenced to the residual isotopomer solvent signals ($CHCl_3$: δ = 7.26 ppm; CH_3CN: δ = 1.94 ppm; CH_3OH: δ = 3.31 ppm) for ^{1}H NMR spectra, to the solvent signal ($CDCl_3$: δ = 77.16 ppm; CD_3CN: δ = 1.32 ppm; CD_3OD: δ = 49.00 ppm) for ^{13}C NMR spectra. Coupling constants (*J*) are reported in Hertz (Hz). The following abbreviations are used: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad.

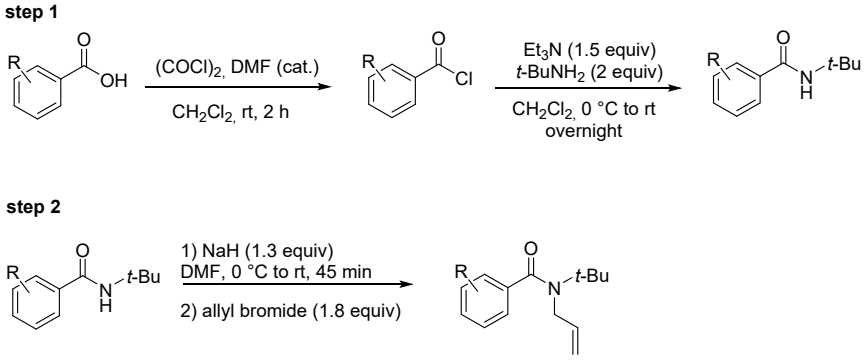
HRMS were determined on a Waters XevoQTof spectrometer using an electrospray ionization coupled with a time of flight analyzer (ESI-TOF) after dissolving the analyte in CH_3CN.

Infrared spectra were recorded on an IR spectrometer (Perkin Elmer BX FT-IR), and absorption frequencies were reported in reciprocal centimeters (cm^{-1}).

Electrochemical reactions were carried out with an IKA ElectraSyn 2.0 Pro apparatus using IKA electrodes.

2. Preparation of the substrates

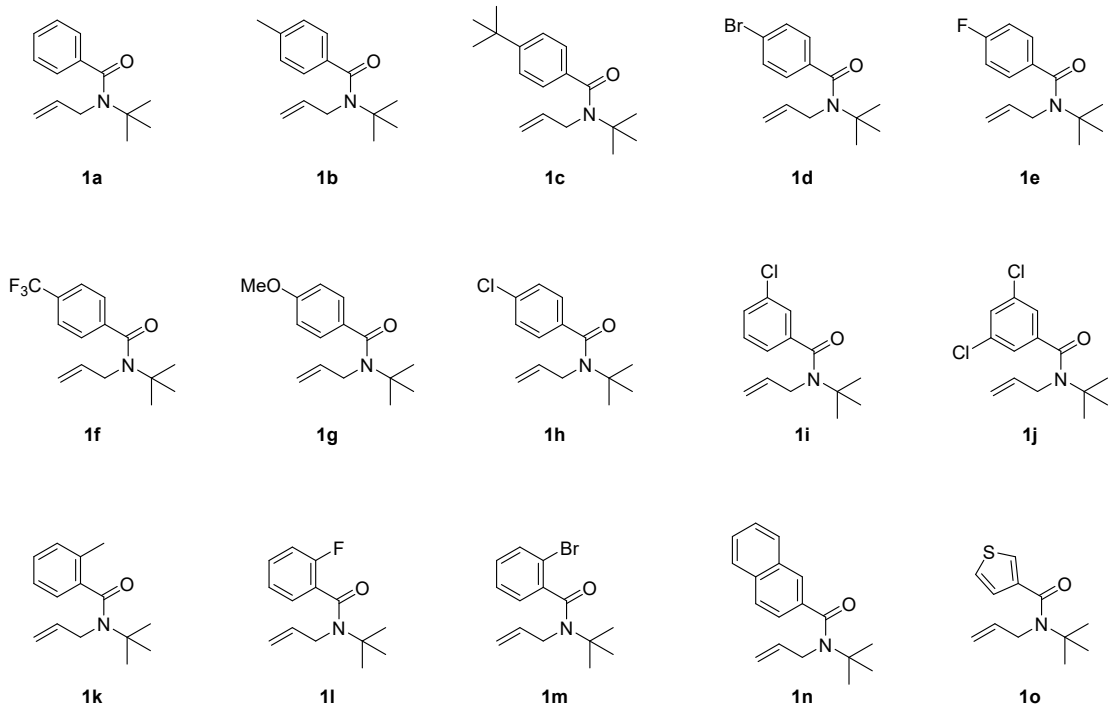
2.1. Preparation of N-allylbenzamides **1**



Scheme S1: Preparation of substrates **1**

Step 1: To a stirred suspension of the required benzoic acid derivative (1 equiv) in CH_2Cl_2 (0.5 M) containing 2 drops of DMF was added dropwise oxalyl chloride (2 equiv) at room temperature. The reaction mixture was stirred for 2 hours. After concentrating under reduced pressure, the resulting residue was dissolved in CH_2Cl_2 (0.4 M) and cooled down at 0 °C in an ice bath. Triethylamine (2 equiv) and *tert*-butylamine (1.5 equiv) were subsequently added. The ice bath was removed and the resulting reaction mixture was allowed to stir at room temperature overnight. The reaction was quenched with aq. $NaHCO_3$. The phases were separated and the aqueous phase was extracted twice with CH_2Cl_2. The combined organic phases were washed once with aq. NH_4Cl and twice with aq. $NaHCO_3$ (x2), dried over $MgSO_4$, filtered and concentrated under reduced pressure to afford the crude *N*-(*tert*-butyl)benzamide derivative as a solid which was used directly in the next step without further purification.

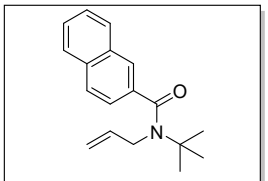
Step 2: To a solution of the crude *N*-(*tert*-butyl)benzamide derivative in DMF (0.5 M) was added portionwise NaH at 0 °C. The resulting reaction mixture was stirred for 45 min at room temperature. After cooling down to 0 °C (ice bath), allyl bromide (1.8 equiv) was added dropwise. The ice bath was removed and the resulting reaction mixture was allowed to stir at room temperature overnight. Water was carefully added and the reaction mixture was extracted with EtOAc (x3). The combined organic phases were washed with brine (x3), dried over $MgSO_4$, filtered and concentrated under reduced pressure. Purification by flash column chromatography (EP/EtOAc) afforded the desired substrates **1**.



Scheme S2: Structures of substrates **1**

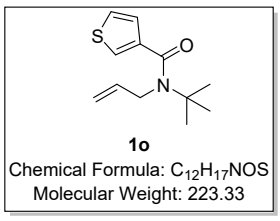
1a–**1m** have been characterized in our previous work.[1]

N-allyl-*N*-(*tert*-butyl)-2-naphthamide (**1n**)



IR: 2965, 2925, 1629, 1471, 1389, 1377, 1360, 1262, 1232, 1196, 1128, 943, 862, 821, 779, 757 cm^{-1}**; ^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.89 – 7.78 (m, 4H), 7.53 – 7.42 (m, 3H), 5.87 – 5.69 (m, 1H), 5.16 – 5.03 (m, 2H), 3.97 (dt, *J* = 5.2, 1.8 Hz, 2H), 1.60 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 173.62, 137.35, 136.92, 133.40, 132.87, 128.41, 128.19, 127.84, 126.68, 126.56, 125.43, 124.06, 116.28, 57.76, 50.17, 28.86; **HRMS (ESI$^+$)**: *m/z* calcd for $[C_{18}H_{22}NO]^+$ ($[M+H]^+$): 268.1701, found: 268.1703.

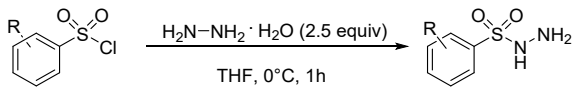
N-allyl-*N*-(*tert*-butyl)thiophene-3-carboxamide (**1o**)



IR: 2964, 2926, 1625, 1521, 1410, 1393, 1355, 1250, 1196, 977, 920, 849, 802, 747, 729 cm^{-1}**; ^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.50 (dd, *J* = 2.9, 1.3 Hz, 1H), 7.26 (dd, *J* = 5.0, 2.9 Hz, 1H), 7.19 (dd, *J* = 5.0, 1.3 Hz, 1H), 5.95 – 5.78 (m, 1H), 5.24 (t, *J* = 1.9 Hz, 1H), 5.22 – 5.16 (m, 1H), 4.01 (dt, *J* = 4.6, 1.9 Hz, 2H), 1.54 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 168.94, 139.85, 137.88, 127.10, 125.29, 124.52, 116.16, 57.88, 49.84, 28.70; **HRMS (ESI$^+$)**: *m/z* calcd for $[C_{12}H_{17}NOS]^+$ ($[M+H]^+$): 224.1109, found: 224.1110.

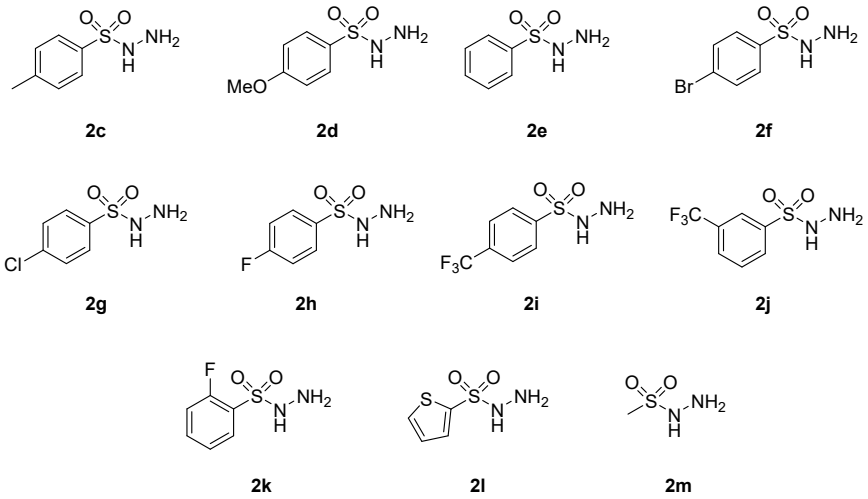
2.2. Preparation of hydrazides **2**

[1] Derat, E.; Masson, G.; Claraz, A. *Angew. Chem. Int. Ed.* **2024**, e202406017.



Scheme S3: Preparation of hydrazides **2**

To a stirred solution of sulfonyl chloride (1.0 equiv) in THF (0.5 M) at 0 °C under argon was added dropwise hydrazine monohydrate (2.5 equiv). The reaction mixture was stirred at 0 °C for 1 hour. The reaction mixture was evaporated under reduced pressure. The resulting residue was transferred to a separatory funnel with ethyl acetate containing cold brine and was extracted with EtOAc (x3). The combined organic phases were dried over $MgSO_4$, filtered and concentrated under reduced pressure to afford the hydrazide **2**, which was used without further purification.



Scheme S4: Structures of hydrazides **2**

Hydrazides **2** are known compounds and the NMR matched those previously reported in the literature.[2]

3. Electrochemical access to sulfone-containing β-arylethylamines 3

3.1. General procedure A:

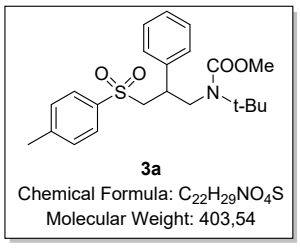
An Electrasyn undivided cell (5 mL) was charged with a magnetic stir bar, *N*-allylbenzamide **1** (0.25 mmol, 1.0 equiv.), sulfonyl hydrazide **2** (0.75 mmol, 3.0 equiv) and tetrabutylammonium hexafluorophosphate (116.2 mg, 0.3 mmol, 0.1 M). The vial was closed with the ElectraSyn cap holding the electrodes (anode: Graphite SK-50, cathode: Nickel plate, dimension of both electrodes: 0.8 cm wide and approximately 2.4 cm immersion depth in solution) and flushed under argon for 2 min. Acetonitrile (1.8 mL) and methanol (1.2 mL) were added and the mixture was stirred for 1 min until complete solubilization of the reagents. Argon gas (balloon) was bubbled through the solution during 5 minutes. The electrolysis was carried out at room temperature under constant current (10 mA, 6.9 mA.cm^{-2}) and

[2] a) Chen, G.; Zhang, X.; Zeng, Z.; Peng, W.; Liang, Q.; Liu, J. *ChemistrySelect* **2017**, *2*, 1979–1982; b) Yu, X.; Li, X.; Wan, B. *Org. Biomol. Chem.* **2012**, *10*, 7479-7482; c) Ma, Q.; Li, M.; Chen, Z.; Ni, S.-F.; Wright, J. S.; Wen, L.-R.; Zhang, L.-B. *Green Chem.* **2022**, *24*, 4425–4431; d) Li, G.; Kong, X.; Liang, Q.; Lin, L.; Yu, K.; Xu, B.; Chen, Q. *Eur. J. Org. Chem.* **2020**, 6135–6145.

was stopped after 6 h 03 min (electricity = 9 F.mol^{-1}). The two electrodes were rinsed with EtOAc (5 mL for each electrode) for 2 min in a ultrasonic bath. The rinsing solutions and water (10 mL) were added to the crude reaction mixture. The phases were separated, and the aqueous layer was extracted with EtOAc (2×5 mL). The combined organic phases were dried over $MgSO_4$, filtered and concentrated under vacuum. The residue was purified by flash column chromatography on silica gel (PE/EtOAc) to yield the desired product **3**.

3.2. Characterization of products **3**

methyl *tert*-butyl(2-phenyl-3-tosylpropyl)carbamate (**3a**)

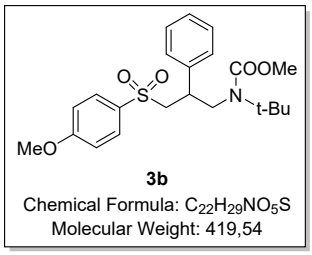


3a
Chemical Formula: $C_{22}H_{29}NO_4S$
Molecular Weight: 403,54

Following the general procedure A using *N*-allylbenzamide **1a** (54.3 mg), hydrazide **2c** (139.7 mg), **3a** was obtained as a yellow oil (56.1 mg, 0.139 mmol, 56%).

IR: ν 2957, 2926, 1699, 1598, 1495, 1455, 1440, 1381, 1364, 1312, 1301, 1290, 1250, 1226, 1187, 1137, 1088, 815, 802, 777, 752, 702, 667 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.50 (d, *J* = 8.1 Hz, 2H), 7.21 – 7.09 (m, 5H), 7.08 – 6.96 (m, 2H), 3.75 (dd, *J* = 15.0, 8.7 Hz, 1H), 3.57 (s, 3H),3.58 – 3.50 (m, 1H), 3.49 – 3.38 (m, 2H), 3.30 (dd, *J* = 15.0, 6.5 Hz, 1H), 2.35 (s, 3H), 1.32 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 157.1, 144.2, 139.6, 136.8, 129.6, 128.6, 128.1, 128.0, 127.2, 58.9, 56.5, 52.1, 50.5, 42.7, 29.4, 21.6; **HRMS (ESI$^+$)** *m/z* calcd for $[C_{22}H_{29}NO_4SNa]^+$ ([M+Na]$^+$): 426.1715, found: 426.1714.

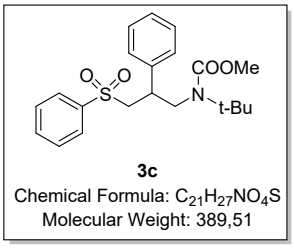
methyl *tert*-butyl(3-((4-methoxyphenyl)sulfonyl)-2-phenylpropyl)carbamate (**3b**)



3b
Chemical Formula: $C_{22}H_{29}NO_5S$
Molecular Weight: 419,54

Following the general procedure A using *N*-allylbenzamide **1a** (54.3 mg), hydrazide **2d** (151.7 mg), **3b** was obtained as a white solid (43 mg, 0.102 mmol, 41%).
IR: 2959, 1698, 1595, 1258, 1134, 1089, 835, 806 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.58 – 7.47 (m, 2H), 7.22 – 7.06 (m, 3H), 7.10 – 6.94 (m, 2H), 6.83 – 6.72 (m, 2H), 3.81 (s, 3H), 3.74 (dd, *J* = 14.7, 8.1 Hz, 1H), 3.57 (s, 3H), 3.62 – 3.36 (m, 3H), 3.30 (dd, *J* = 14.7, 6.1 Hz, 1H), 1.33 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 163.3, 157.0, 139.6, 131.3, 130.0, 128.5, 127.9, 127.2, 114.1, 58.9, 56.4, 55.6, 52.0, 50.4, 42.7, 29.3; **HRMS (ESI$^+$)** *m/z* calcd for $[C_{22}H_{29}NO_5SNa]^+$ ([M+Na]$^+$): 442.1664, found: 442.1664.

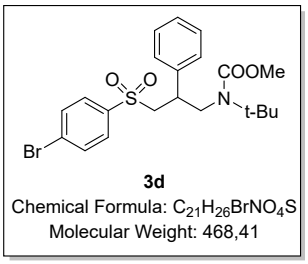
methyl *tert*-butyl(2-phenyl-3-(phenylsulfonyl)propyl)carbamate (**3c**)



3c
Chemical Formula: $C_{21}H_{27}NO_4S$
Molecular Weight: 389,51

Following the general procedure A using *N*-allylbenzamide **1a** (54.3 mg), hydrazide **2e** (129.1 mg), **3c** was obtained as a white solid (75.4 mg, 0.194 mmol, 77%).
IR: 2957, 1698, 1447, 1305, 1188, 1138. 1086, 748 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.67 – 7.57 (m, 2H), 7.53 – 7.41 (m, 1H), 7.39 – 7.27 (m, 2H), 7.13 (tdd, *J* = 4.6, 3.5, 1.5 Hz, 3H), 7.06 – 6.94 (m, 2H), 3.75 (dd, *J* = 14.8, 8.6 Hz, 1H), 3.58 (s, 3H), 3.66 – 3.37 (m, 3H), 3.29 (dd, *J* = 14.8, 6.3 Hz, 1H), 1.33 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 157.1, 139.8, 139.4, 133.3, 129.0, 128.7, 128.0, 128.0, 127.4, 58.8, 56.5, 52.2, 50.5, 42.8, 29.4; **HRMS (ESI$^+$):** *m/z* calcd for $[C_{21}H_{27}NO_4SNa]^+$ ([M+Na]$^+$) : 412.1555, found: 412.1559.

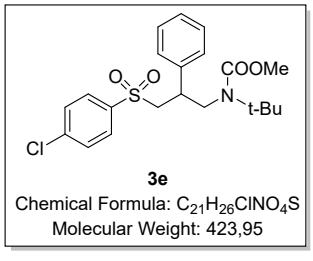
methyl (3-((4-bromophenyl)sulfonyl)-2-phenylpropyl)(*tert*-butyl)carbamate (**3d**)



3d
Chemical Formula: $C_{21}H_{26}BrNO_4S$
Molecular Weight: 468,41

Following the general procedure A using *N*-allylbenzamide **1a** (54.3 mg), hydrazide **2f** (188.3 mg), **3d** was obtained as a white solid (80.1 mg, 0.171 mmol, 68%).
IR: 2923, 1704, 1575, 1311, 1142, 1086 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.33 (s, 4H), 7.14 – 6.99 (m, 3H), 6.95 – 6.84 (m, 2H), 3.66 (dd, *J* = 14.8, 9.1 Hz, 1H), 3.55 (s, 3H), 3.53 (dd, *J* = 14.7, 10.4 Hz, 1H), 3.47 – 3.30 (m, 2H), 3.21 (dd, *J* = 14.8, 6.0 Hz, 1H), 1.28 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 157.1, 139.0, 138.6, 132.0, 129.4, 128.6, 128.4, 127.9, 127.3, 58.8, 56.4, 52.1, 50.4, 42.8, 29.3; **HRMS (ESI$^+$)** *m/z* calcd for $[C_{21}H_{26}BrNO_4SNa]^+$ ([M+Na]$^+$): 490.0664, found: 490.0662.

methyl *tert*-butyl(3-((4-chlorophenyl)sulfonyl)-2-phenylpropyl)carbamate (**3e**)

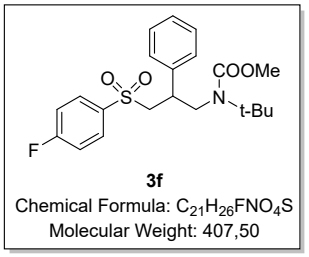


3e
Chemical Formula: $C_{21}H_{26}ClNO_4S$
Molecular Weight: 423,95

Following the general procedure A using *N*-allylbenzamide **1a** (54.3 mg), hydrazide **2g** (155 mg), **3e** was obtained as a white solid (63.1 mg, 0.149 mmol, 60%).
IR: 2957, 1695, 1584, 1310, 1139, 1087, 1014, 911, 830 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.54 – 7.45 (m, 2H), 7.31 – 7.22 (m, 2H), 7.14 (dd, *J* = 5.0, 1.9 Hz, 3H), 7.02 – 6.93 (m, 2H), 3.75 (dd, *J* = 14.8, 9.0 Hz, 1H), 3.67 – 3.55 (m, 1H), 3.62 (s, 3H), 3.55 – 3.41 (m, 2H), 3.29 (dd, *J* = 14.8, 6.0 Hz, 1H), 1.36 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 157.1, 139.8, 139.0, 138.1, 129.3, 129.0, 128.6, 127.9, 127.3, 58.8,

56.4, 52.1, 50.4, 42.8, 29.3; **HRMS (ESI$^+$)** *m/z* calcd for $[C_{21}H_{26}ClNO_4SNa]^+$ ($[M+Na]^+$): 446.1169, found: 446.1159.

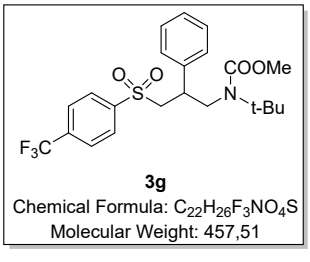
methyl *tert*-butyl(3-((4-fluorophenyl)sulfonyl)-2-phenylpropyl)carbamate (**3f**)



3f
Chemical Formula: $C_{21}H_{26}FNO_4S$
Molecular Weight: 407,50

Following the general procedure A using *N*-allylbenzamide **1a** (54.3 mg), hydrazide **2h** (142.5 mg), **3f** was obtained as a white solid (79.2 mg, 0.194 mmol, 78%).
IR: 2960, 1701, 1591, 1494, 1290, 1139, 1087, 838 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.73 – 7.46 (m, 2H), 7.19 – 7.07 (m, 3H), 7.01 – 6.87 (m, 4H), 3.73 (dd, *J* = 14.8, 8.9 Hz, 1H), 3.60 (s, 3H), 3.59 – 3.38 (m, 3H), 3.27 (dd, *J* = 14.8, 6.1 Hz, 1H), 1.34 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 165.4 (d, *J* = 254.3 Hz), 157.1, 139.1, 135.8 (d, *J* = 3.8 Hz), 130.7 (d, *J* = 9.75 Hz), 128.6, 127.9, 127.3, 116.0 (d, *J* = 22.5 Hz), 58.8, 56.4, 52.1, 50.4, 42.9, 29.3; **^{19}F {^{13}C} NMR** (282 MHz, $CDCl_3$) δ -104.3; **HRMS (ESI$^+$)** *m/z* calcd for $[C_{21}H_{26}FNO_4NaS]^+$ ($[M+Na]^+$): 430.1462, found: 430.1464.

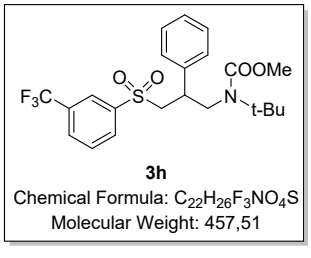
methyl *tert*-butyl(2-phenyl-3-((4-(trifluoromethyl)phenyl)sulfonyl)propyl)carbamate (**3g**)



3g
Chemical Formula: $C_{22}H_{26}F_3NO_4S$
Molecular Weight: 457,51

Following the general procedure A using *N*-allylbenzamide **1a** (54.3 mg), hydrazide **2i** (180.1 mg), **3g** was obtained as a white solid (92.8 mg, 0.203 mmol, 81%).
IR: 2980, 1700, 1320, 1131, 1061, 1016, 844 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.61 – 7.53 (m, 2H), 7.43 (d, *J* = 8.3 Hz, 2H), 7.08 – 6.93 (m, 3H), 6.92 – 6.81 (m, 2H), 3.58 (s, 3H), 3.74 – 3.31 (m, 4H), 3.19 (dd, *J* = 14.9, 5.9 Hz, 1H), 1.29 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 157.2, 143.1, 138.6, 134.7 (q, *J* = 33 Hz), 128.6, 128.4, 127.9, 127.5, 125.8 (q, *J* = 3.8 Hz), 123.1 (q, *J* = 272.3 Hz), 58.6, 56.4, 52.2, 50.4, 43.0, 29.4; **^{19}F {^{13}C} NMR** (282 MHz, $CDCl_3$) δ -63.4; **HRMS (ESI$^+$)** *m/z* calcd for $[C_{22}H_{26}F_3NO_4SNa]^+$ ($[M+Na]^+$): 480.1424, found: 480.1422.

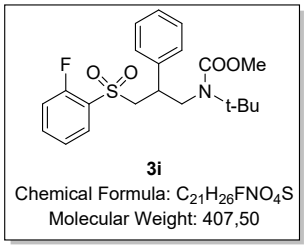
methyl *tert*-butyl(2-phenyl-3-((3-(trifluoromethyl)phenyl)sulfonyl)propyl)carbamate (**3h**)



3h
Chemical Formula: $C_{22}H_{26}F_3NO_4S$
Molecular Weight: 457,51

Following the general procedure A using *N*-allylbenzamide **1a** (54.4 mg), hydrazide **2j** (180.2 mg), **3h** was obtained as a white solid (78.0 mg, 0.170 mmol, 68%).
IR: 2960, 1698, 1325, 1304, 1132, 1097, 1072, 913, 734, 694 cm^{-1}; **mp:** 86°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.79 – 7.60 (m, 3H), 7.48 – 7.38 (m, 1H), 7.13 – 7.01 (m, 3H), 7.01 – 6.88 (m, 2H), 3.80 – 3.40 (m, 7H), 3.32 – 3.19 (m, 1H), 1.36 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 157.3, 140.9, 138.5, 131.4 (q, J = 32.9 Hz), 131.1, 129.8 (q, J = 3.5 Hz), 129.6, 128.7, 127.8, 127.6, 125.0 (q, J = 3.9 Hz), 121.2 (q, J = 272.9 Hz), 58.6, 56.4, 52.2, 50.4, 43.1, 29.4; **^{19}F {^{13}C} NMR** (282 MHz, $CDCl_3$) δ -62.82; **HRMS (ESI$^+$)** *m/z* calcd for $[C_{22}H_{26}F_3NO_4SNa]^+$ ($[M+Na]^+$): 480.1432, found: 480.1435.

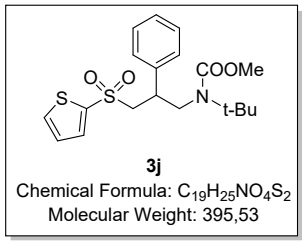
methyl *tert*-butyl(3-((2-fluorophenyl)sulfonyl)-2-phenylpropyl)carbamate (**3i**)



3i
Chemical Formula: $C_{21}H_{26}FNO_4S$
Molecular Weight: 407,50

Following the general procedure A using *N*-allylbenzamide **1a** (54.4 mg), hydrazide **2k** (142.6 mg), **3i** was obtained as a white solid (76.2 mg, 0.187 mmol, 75%).
IR: 2958, 1689, 1600, 1474, 1313, 1139, 1070, 915, 825, 762, 701 cm^{-1}; **mp:** 121°C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.64 – 7.50 (m, 1H), 7.48 – 7.35 (m, 1H), 7.20 – 7.02 (m, 4H), 7.02 – 6.85 (m, 3H), 3.94 – 3.70 (m, 2H), 3.70 – 3.36 (m, 5H), 3.29 (dd, 1H), 1.36 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 159.1 (d, J = 254.9 Hz), 157.0, 138.9, 135.7 (d, J = 8.6 Hz), 130.3, 128.5, 127.8, 127.4, 124.3 (d, J = 3.7 Hz), 116.6 (d, J = 21.3 Hz), 57.9, 57.9, 56.4, 52.1, 50.2, 42.9, 29.3; **^{19}F {^{13}C} NMR** (282 MHz, $CDCl_3$) δ -108.69; **HRMS (ESI$^+$)** *m/z* calcd for $[C_{21}H_{26}FNO_4SNa]^+$ ($[M+Na]^+$): 430.1464, found: 430.1468.

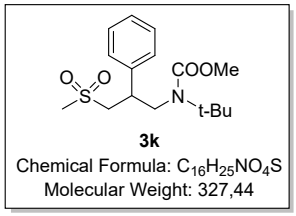
methyl *tert*-butyl(2-phenyl-3-(thiophen-2-ylsulfonyl)propyl)carbamate (**3j**)



3j
Chemical Formula: $C_{19}H_{25}NO_4S_2$
Molecular Weight: 395,53

Following the general procedure A using *N*-allylbenzamide **1a** (54.4 mg), hydrazide **2l** (133.6 mg), **3j** was obtained as a yellowish solid (71.0 mg, 0.180 mmol, 72%).
IR: 2958, 1694, 1310, 1187, 1133, 1090, 1013, 914, 776, 728, 701 cm^{-1}; **mp:** 96°C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.53 (dd, J = 4.9, 1.4 Hz, 1H), 7.31 (dd, J = 3.7, 1.4 Hz, 1H), 7.19 (m, 3H), 7.10 – 7.02 (m, 2H), 6.90 (dd, J = 4.9, 3.7 Hz, 1H), 3.86 – 3.43 (m, 5H), 3.59 (s, 3H), 3.37 – 3.25 (m, 1H), 1.33 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 157.0, 140.7, 139.4, 134.1, 133.7, 128.6, 127.8, 127.6, 127.4, 60.1, 56.4, 52.1, 50.4, 42.9, 29.3; **HRMS (ESI$^+$)** *m/z* calcd for $[C_{19}H_{25}NO_4S_2Na]^+$ ($[M+Na]^+$): 418.1123, found: 418.1124.

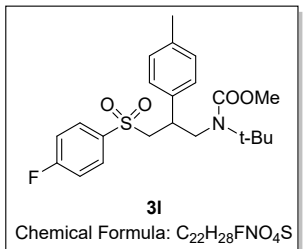
methyl *tert*-butyl(3-(methylsulfonyl)-2-phenylpropyl)carbamate (**3k**)



3k
Chemical Formula: $C_{16}H_{25}NO_4S$
Molecular Weight: 327,44

Following the general procedure A using *N*-allylbenzamide **1a** (54.3 mg), hydrazide **2m** (82.6 mg), **3k** was obtained as a white solid (54 mg, 0.165 mmol, 66%).
IR: 2919, 1698, 1299, 1129 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.40 – 7.13 (m, 5H), 3.79 (dd, *J* = 14.7, 8.8 Hz, 1H), 3.59 (s, 3H), 3.50 – 3.19 (m, 4H), 2.22 (s, 3H), 1.32 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 157.2, 139.7, 129.2, 128.1, 128.0, 58.2, 56.4, 52.2, 50.1, 43.1, 41.9, 29.4; **HRMS (ESI$^+$)** *m/z* calcd for $[C_{16}H_{25}NO_4SNa]^+$ ($[M+Na]^+$): 350.1402, found: 350.1391.

methyl *tert*-butyl(3-((4-fluorophenyl)sulfonyl)-2-(*p*-tolyl)propyl)carbamate (**3l**)

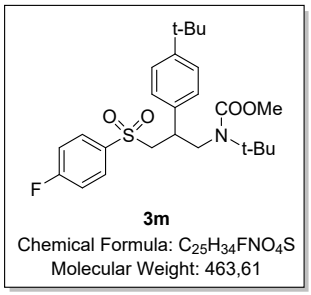


3l
Chemical Formula: $C_{22}H_{28}FNO_4S$
Molecular Weight: 421,53

Following the general procedure A using *N*-allylbenzamide **1b** (57.8 mg), hydrazide **2h** (142.5 mg), **3l** was obtained as a white solid (83 mg, 0.197 mmol, 79%).

IR: 2959,1701, 1591, 1494, 1364, 1311, 1290, 1188, 1139, 1087, 837, 817, 777 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.57 (dd, *J* = 8.8, 5.1 Hz, 2H), 7.05 – 6.91 (m, 4H), 6.87 (d, *J* = 8.2 Hz, 2H), 3.73 (dd, *J* = 14.8, 9.2 Hz, 1H), 3.63 (s, 3H), 3.60 – 3.36 (m, 3H), 3.26 (dd, *J* = 14.8, 6.0 Hz, 1H), 2.26 (s, 3H), 1.36 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 165.5 (d, *J* = 255.6 Hz), 157.2, 137.2, 130.8 (d, *J* = 9.5 Hz), 136.0, 135.9 (d, *J* = 3.3 Hz), 129.3, 127.8, 116.0 (d, *J* = 22.6 Hz), 59.0, 56.4, 52.2, 50.5, 42.6, 29.4, 21.0; **^{19}F {^{13}C} NMR** (282 MHz, $CDCl_3$) δ -104.6; **HRMS (ESI$^+$)** *m/z* calcd for $[C_{22}H_{28}FNO_4SNa]^+$ ($[M+Na]^+$): 444.1621, found: 444.1616.

methyl *tert*-butyl(2-(4-(tert-butyl)phenyl)-3-((4-fluorophenyl)sulfonyl)propyl)carbamate (**3m**)

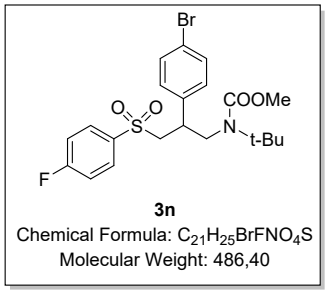


3m
Chemical Formula: $C_{25}H_{34}FNO_4S$
Molecular Weight: 463,61

Following the general procedure A using *N*-allylbenzamide **1c** (68.4 mg), hydrazide **2h** (142.5 mg), **3m** was obtained as a white solid (79.8 mg, 0.172 mmol, 69%).

IR: 2963, 1698, 1591, 1493, 1364, 1311, 1289, 1227, 1187, 1139, 1086, 914, 834, 730 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.59 – 7.43 (m, 2H), 7.12 – 7.01 (m, 2H), 6.97 – 6.77 (m, 4H), 3.75 – 3.64 (m, 1H), 3.60 (s, 3H), 3.59 – 3.33 (m, 3H), 3.23 (dd, *J* = 14.8, 6.0 Hz, 1H), 1.36 (s, 9H), 1.23 (s, 9H). **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 165.2 (d, *J* = 255.5 Hz), 157.2, 150.4, 135.84 (d, *J* = 3.5 Hz), 135.77, 130.8 (d, *J* = 9.5 Hz), 127.5, 125.5, 115.9 (d, *J* = 22.6 Hz), 58.7, 56.5, 52.2, 50.6, 42.7, 34.4, 31.4, 29.4; **^{19}F {^{13}C} NMR** (282 MHz, $CDCl_3$) δ -104.6; **HRMS (ESI$^+$)** *m/z* calcd for $[C_{25}H_{34}FNO_4SNa]^+$ ($[M+Na]^+$) : 486.2090, found: 486.2090.

methyl (2-(4-bromophenyl)-3-((4-fluorophenyl)sulfonyl)propyl)(*tert*-butyl)carbamate (**3n**)

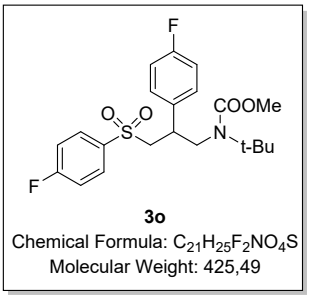


3n
Chemical Formula: $C_{21}H_{25}BrFNO_4S$
Molecular Weight: 486,40

Following the general procedure A using *N*-allylbenzamide **1d** (74.1 mg), hydrazide **2h** (142.5 mg), **3n** was obtained as a white solid (101.4 mg, 0.208 mmol, 84%).

IR: 2959, 1698, 1591, 1492, 1365, 1310, 1290, 1230, 1187, 1138, 1086, 1011, 917, 837 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.68 – 7.55 (m, 2H), 7.35 – 7.24 (m, 2H), 7.04 (t, *J* = 8.5 Hz, 2H), 6.89 (d, *J* = 8.4 Hz, 2H), 3.72 (dd, *J* = 14.7, 8.6 Hz, 1H), 3.61 (s, 3H), 3.58 – 3.37 (m, 3H), 3.29 (dd, *J* = 14.7, 6.0 Hz, 1H), 1.35 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 165.7 (d, *J* = 256.6 Hz), 157.3, 138.3, 135.8 (d, *J* = 3.3 Hz), 131.8, 130.8 (d, *J* = 9.6 Hz), 129.8, 121.5, 116.3 (d, *J* = 22.7 Hz), 58.9, 56.5, 52.3, 50.2, 42.5, 29.5; **^{19}F {^{13}C} NMR** (282 MHz, $CDCl_3$) δ -103.6; **HRMS (ESI$^+$)** *m/z* calcd for $[C_{21}H_{25}BrFNO_4SNa]^+$ ($[M+Na]^+$) : 508.0569, found: 508.0562.

methyl *tert*-butyl(2-(4-fluorophenyl)-3-((4-fluorophenyl)sulfonyl)propyl)carbamate (**3o**)

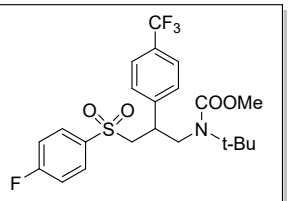


3o
Chemical Formula: $C_{21}H_{25}F_2NO_4S$
Molecular Weight: 425,49

Following the general procedure A using *N*-allylbenzamides **1e** (58.8 mg), hydrazide **2h** (142.5 mg), **3o** was obtained as a white solid (76.3 mg, 0.179 mmol, 72%).

IR: 2959,1698, 1581, 1510, 1494, 1365, 1310, 1290, 1224, 1187, 1137, 1086, 834 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.59 (dd, J = 8.9, 5.1 Hz, 2H), 7.14 – 6.93 (m, 4H), 6.93 – 6.77 (m, 2H), 3.71 (dd, J = 14.8, 8.2 Hz, 1H), 3.60 (s, 3H), 3.56 – 3.38 (m, 3H), 3.27 (dd, J = 14.8, 5.7 Hz, 1H), 1.34 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 165.6 (d, J = 256.3 Hz), 162.1 (d, J = 246.6 Hz), 157.2, 135.9 (d, J = 3.4 Hz), 135.1 (d, J = 3.4 Hz), 130.8 (d, J = 9.6 Hz), 129.6 (d, J = 8.0 Hz), 116.3 (d, J = 22.7 Hz), 115.6 (d, J = 21.4 Hz), 59.1, 56.5, 52.3, 50.5, 42.2, 29.5; **^{19}F {^{13}C} NMR** (282 MHz, $CDCl_3$) δ -103.8, -114.7; **HRMS (ESI$^+$)** *m/z* calcd for $[C_{21}H_{25}F_2NO_4SNa]^+$ ($[M+Na]^+$): 448.1370, found: 448.1370.

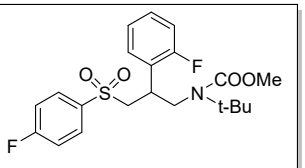
methyl *tert*-butyl(3-((4-fluorophenyl)sulfonyl)-2-(4-(trifluoromethyl)phenyl)propyl)carbamate (**3p**)



3p
Chemical Formula: $C_{22}H_{25}F_4NO_4S$
Molecular Weight: 475,50

Following the general procedure A using *N*-allylbenzamide **1f** (71.4 mg), hydrazide **2h** (142.6 mg), **3p** was obtained as a pale yellow solid (87.5 mg, 0.184 mmol, 74%).
IR: 2927, 1702, 1592, 1494, 1325, 1265, 1126, 1069, 1017, 733, 730 cm^{-1}; **mp:** 97°C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.61 – 7.50 (m, 2H), 7.45 – 7.35 (m, 2H), 7.18 – 7.08 (m, 2H), 7.05 – 6.91 (m, 2H), 3.81 – 3.69 (m, 1H), 3.60 (s, 3H), 3.58 – 3.45 (m, 3H), 3.38 – 3.24 (m, 1H), 1.36 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 165.7 (d, J = 257.0 Hz), 157.3, 143.4, 135.7 (d, J = 3.4 Hz), 130.8 (d, J = 9.6 Hz), 129.7 (q, J = 32.5 Hz), 128.5, 125.6 (q, J = 3.7 Hz), 123.3 (q, J = 271.6 Hz), 116.4 (d, J = 22.6 Hz), 58.7, 56.6, 52.3, 50.2, 43.0, 29.5; **^{19}F {^{13}C} NMR** (282 MHz, $CDCl_3$) δ -62.7, -103.5; **HRMS (ESI$^+$)** *m/z* calcd for $[C_{22}H_{25}F_4NO_4SNa]^+$ ($[M+Na]^+$): 498.1338, found: 498.1340.

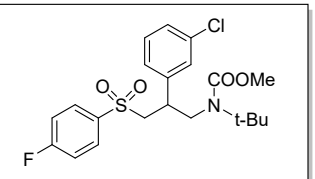
methyl *tert*-butyl(2-(2-fluorophenyl)-3-((4-fluorophenyl)sulfonyl)propyl)carbamate (**3q**)



3q
Chemical Formula: $C_{21}H_{25}F_2NO_4S$
Molecular Weight: 425,49

Following the general procedure A using *N*-allylbenzamide **1l** (63.4 mg), hydrazide **2h** (153.6 mg), **3q** was obtained as a white solid (77.9 mg, 0.183 mmol, 68%).
IR: 2959, 1700, 1591, 1492, 1290, 1227, 1138, 1085, 1014, 837, 818, 757, 733 cm^{-1}; **mp:** 84°C; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.71 – 7.55 (m, 2H), 7.18 – 7.08 (m, 1H), 7.08 – 6.91 (m, 4H), 6.86 – 6.75 (m, 1H), 3.79 – 3.60 (m, 3H), 3.58 (s, 3H), 3.51 – 3.32 (m, 2H), 1.31 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 165.5 (d, J = 256.0 Hz), 160.9 (d, J = 245.7 Hz), 156.8, 135.3 (d, J = 3.3 Hz), 130.7 (d, J = 9.7 Hz), 130.3 (d, J = 4.9 Hz), 129.2 (d, J = 8.6 Hz), 125.9 (d, J = 13.5 Hz), 124.3 (d, J = 3.4 Hz), 116.1 (d, J = 22.7 Hz), 115.7 (d, J = 22.3 Hz), 57.4 (d, J = 3.4 Hz), 56.4, 52.1, 48.7, 38.0, 29.2; **^{19}F {^{13}C} NMR** (282 MHz, $CDCl_3$) δ -104.08, -116.05; **HRMS (ESI$^+$)** *m/z* calcd for $[C_{21}H_{25}F_2NO_4SNa]^+$ ($[M+Na]^+$): 448.1370, found: 448.1367.

methyl *tert*-butyl(2-(3-chlorophenyl)-3-((4-fluorophenyl)sulfonyl)propyl)carbamate (**3r**)



3r
Chemical Formula: $C_{21}H_{25}ClFNO_4S$
Molecular Weight: 441,94

Following the general procedure A using *N*-allylbenzamide **1i** (62.8 mg), hydrazide **2h** (142.6 mg), **3r** was obtained as a colorless oil (68.0 mg, 0.153 mmol, 61%).
IR: 2960, 1695, 1591, 1493, 1289, 1138, 1084, 837, 730 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.65 – 7.49 (m, 2H), 7.15 – 6.83 (m, 6H), 3.77 – 3.64 (m, 1H), 3.61 (s, 3H), 3.57 – 3.34 (m, 3H), 3.34 – 3.18 (m, 1H), 1.35 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 165.5 (d, J = 256.4 Hz), 157.1, 141.1, 135.5 (d, J = 3.4 Hz), 134.4, 130.7 (d, J = 9.6 Hz), 129.9, 128.0, 127.5, 126.3, 116.1 (d, J = 22.7 Hz), 58.5, 56.5, 52.2, 50.2, 42.7, 29.4; **^{19}F {^{13}C} NMR** (282 MHz, $CDCl_3$) δ -103.75; **HRMS (ESI^{+})** *m/z* calcd for $[C_{21}H_{25}ClFNO_4SNa]^+$ ($[M+Na]^+$): 464.1075, found: 464.1080.

4. Electrochemical access to sulfonate ester-containing β-arylethylamines 4

4.1. General procedure B:

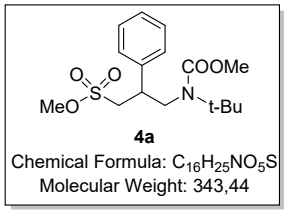
An Electrasyn undivided cell (5 mL) was charged with a magnetic stir bar, *N*-allylbenzamide **1** (0.25 mmol, 1.0 equiv.), $NaHSO_3$ (104.1 mg, 1 mmol, 4 equiv) and tetrabutylammonium perchlorate (102.6 mg, 0.3 mmol, 0.1 M). The vial was closed with the ElectraSyn cap holding the electrodes (anode: Graphite SK-50, cathode: Nickel plate, dimension of both electrodes: 0.8 cm wide and approximately 1.8 cm immersion depth in solution) and flushed under argon for 2 min. Acetonitrile (1.8 mL) and methanol (1.2 mL) were added. Argon gas (balloon) was bubbled during 5 min through the reaction mixture under stirring. The electrolysis was carried out at room temperature under constant current (20 mA, 13.9 $mA.cm^{-2}$) and was stopped after 1 h 10 min (electricity = 3.5 $F.mol^{-1}$). At the end of the electrolysis, the two electrodes were rinsed with EtOAc. The rinsing solutions and water (10 mL) were added to the crude reaction mixture. The phases were separated, and the aqueous layer was extracted with EtOAc (2×5 mL). The combined organic phases were washed with brine, dried over $MgSO_4$, filtered and concentrated under vacuum. The residue was purified by flash column chromatography on silica gel (PE/EtOAc) to yield the desired product **4**.

At 1 mmol scale:

An Electrasyn undivided cell (20 mL) was charged with a magnetic stir bar, *N*-allylbenzamide **1a** (217.3 mg, 1 mmol, 1.0 equiv.), $NaHSO_3$ (416.4 mg, 1 mmol, 4 equiv) and tetrabutylammonium perchlorate (410.3 mg, 1.2 mmol, 0.1 M). The vial was closed with the ElectraSyn cap holding the electrodes (anode: Graphite SK-50, cathode: Nickel plate) and flushed under argon for 2 min. Acetonitrile (7.2 mL) and methanol (4.8 mL) were added. Argon gas (balloon) was bubbled during 5 min through the reaction mixture under stirring. The electrolysis was carried out at room temperature under constant current (60 mA) and was stopped after 1 h 30 min (electricity = 3.5 $F.mol^{-1}$). At the end of the electrolysis, the two electrodes were rinsed with EtOAc. The rinsing solutions and water (20 mL) were added to the crude reaction mixture. The phases were separated, and the aqueous layer was extracted with EtOAc (2×15 mL). The combined organic phases were washed with brine, dried over $MgSO_4$, filtered and concentrated under vacuum. The residue was purified by flash column chromatography on silica gel (PE/EtOAc) to yield **4a** (208 mg, 0.606 mmol, 61%).

4.1. Characterization of products **4**

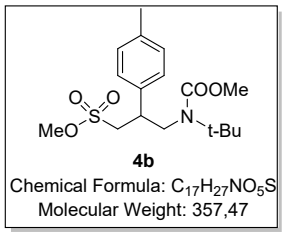
methyl 3-(*tert*-butyl(methoxycarbonyl)amino)-2-phenylpropane-1-sulfonate (**4a**)



4a
Chemical Formula: $C_{16}H_{25}NO_5S$
Molecular Weight: 343,44

Following the general procedure B using *N*-allylbenzamide **1a** (54.3 mg), **4a** was obtained as a colorless foam (60.3 mg, 0.176 mmol, 70%).
IR: 2959, 1699, 1455, 1436, 1381, 1360, 1346, 1252, 1229, 1177, 1154, 1094, 1077, 986, 812, 776, 759, 701 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.40 – 7.32 (m, 2H), 7.32 – 7.22 (m, 3H), 3.93 – 3.81 (m, 1H), 3.65 (s, 3H), 3.56 – 3.42 (m, 3H), 3.50 (s, 3H), 3.41 – 3.31 (m, 1H), 1.40 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 157.2, 139.9, 128.9, 127.9, 127.8, 56.5, 55.2, 52.7, 52.2, 50.0, 43.3, 29.4; **HRMS (ESI$^+$)**: *m/z* calcd for $[C_{16}H_{25}NO_5SNa]^+$ ($[M+Na]^+$): 366.1351, found: 366.1347.

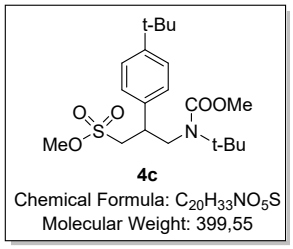
methyl 3-(*tert*-butyl(methoxycarbonyl)amino)-2-(p-tolyl)propane-1-sulfonate (**4b**)



4b
Chemical Formula: $C_{17}H_{27}NO_5S$
Molecular Weight: 357,47

Following the general procedure B using *N*-allylbenzamide **1b** (57.8 mg), **4b** was obtained as a colorless foam (66.5 mg, 0.186 mmol, 74%).
IR: 2957, 1699, 1516, 1436, 1360, 1341, 1290, 1251, 1228, 1177, 1155, 1091, 987, 816, 776, 754 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.21 – 7.05 (m, 4H), 3.96 – 3.73 (m, 1H), 3.64 (s, 3H), 3.56 – 3.25 (m, 4H), 3.51 (s, 3H), 2.32 (s, 3H), 1.38 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 157.2, 137.4, 136.8, 129.5, 127.8, 56.5, 55.2, 52.7, 52.2, 50.1, 42.9, 29.4, 21.1; **HRMS (ESI$^+$)**: *m/z* calcd for $[C_{17}H_{27}NO_5SNa]^+$ ($[M+Na]^+$): 380.1508, found: 380.1500.

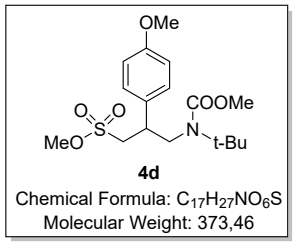
methyl 3-(*tert*-butyl(methoxycarbonyl)amino)-2-(4-(tert-butyl)phenyl)propane-1-sulfonate (**4c**)



4c
Chemical Formula: $C_{20}H_{33}NO_5S$
Molecular Weight: 399,55

Following the general procedure B using *N*-allylbenzamide **1c** (68.3 mg), **4c** was obtained as a colorless foam (73.1 mg, 0.183 mmol, 73%).
IR: 2960, 1701, 1512, 1463, 1436, 1380, 1361, 1341, 1252, 1228, 1178, 1156, 1091, 990 827, 813, 775 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.35 (d, J = 8.4 Hz, 2H), 7.15 (d, J = 8.3 Hz, 2H), 3.93 – 3.76 (m, 1H), 3.61 (s, 3H), 3.52 – 3.28 (m, 4H), 3.47 (s, 3H), 1.38 (s, 9H), 1.29 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 157.2, 150.8, 150.7, 136.8, 127.6, 125.7, 56.6, 55.2, 52.7, 52.2, 50.0, 42.8, 34.6, 31.4, 29.4; **HRMS (ESI$^+$)**: *m/z* calcd for $[C_{20}H_{33}NO_5SNa]^+$ ($[M+Na]^+$): 422.1977, found: 422.1972.

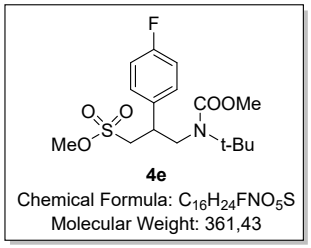
methyl 3-(*tert*-butyl(methoxycarbonyl)amino)-2-(4-methoxyphenyl)propane-1-sulfonate (**4d**)



4d
Chemical Formula: $C_{17}H_{27}NO_6S$
Molecular Weight: 373,46

Following the general procedure B using *N*-allylbenzamide **1g** (57.8 mg), **4d** was obtained as a colorless foam (73.1 mg, 0.198 mmol, 79%).
IR: 2959, 1698, 1612, 1514, 1461, 1436, 1380, 1359, 1341, 1304, 1248, 1177, 1155, 1091, 1031, 987, 828, 806 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.15 (d, J = 8.7 Hz, 2H), 6.86 (d, J = 8.7 Hz, 2H), 3.85 – 3.78 (m, 1H), 3.78 (s, 3H), 3.64 (s, 3H), 3.51 (s, 3H), 3.48 – 3.26 (m, 4H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 159.1, 157.2, 131.8, 128.9, 114.2, 56.5, 55.3, 55.2, 52.8, 52.2, 50.1, 42.5, 29.4; **HRMS (ESI$^+$)**: *m/z* calcd for $[C_{17}H_{27}NO_6SNa]^+$ ($[M+Na]^+$): 396.1457, found: 396.1453.

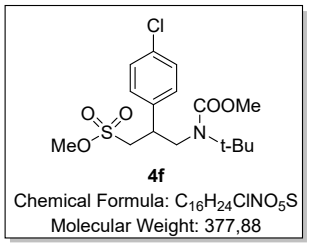
methyl 3-(*tert*-butyl(methoxycarbonyl)amino)-2-(4-fluorophenyl)propane-1-sulfonate (**4e**)



4e
Chemical Formula: $C_{16}H_{24}FNO_5S$
Molecular Weight: 361,43

Following the general procedure B using *N*-allylbenzamide **1e** (58.8 mg), **4e** was obtained as a colorless foam (65.5 mg, 0.181 mmol, 72%).
IR: 2959, 1699, 1605, 1511, 1488, 1436, 1360, 1339, 1291, 1251, 1224, 1177, 1154, 1090, 988, 841, 832, 813, 776, 755 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.25 – 7.15 (m, 2H), 7.08 – 6.97 (m, 2H), 3.87 – 3.75 (m, 1H), 3.63 (s, 3H), 3.55 (s, 3H), 3.52 – 3.25 (m, 4H), 1.37 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 162.2 (d, J = 246.4 Hz), 157.3, 135.6 (d, J = 3.6 Hz), 129.5 (d, J = 8.0 Hz), 115.8 (d, J = 21.5 Hz), 56.6, 55.2, 52.7, 52.3, 50.0, 42.6, 29.4; **^{19}F {^{13}C} NMR (282 MHz, $CDCl_3$)** δ -114.5; **HRMS (ESI$^+$)**: *m/z* calcd for $[C_{16}H_{24}NNO_5SNa]^+$ ($[M+Na]^+$): 384.1257, found: 384.1250.

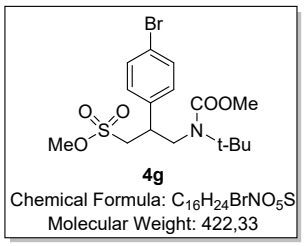
methyl 3-(*tert*-butyl(methoxycarbonyl)amino)-2-(4-chlorophenyl)propane-1-sulfonate (**4f**)



4f
Chemical Formula: $C_{16}H_{24}ClNO_5S$
Molecular Weight: 377,88

Following the general procedure B using *N*-allylbenzamide **1h** (62.9 mg), **4f** was obtained as a colorless foam (68 mg, 0.180 mmol, 72%).
IR: 2957, 1699, 1516, 1436, 1360, 1341, 1290, 1251, 1228, 1177, 1155, 1091, 987, 816, 776, 754 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.38 – 7.28 (m, 2H), 7.21 – 7.09 (m, 2H), 3.88 – 3.74 (m, 1H), 3.63 (s, 3H), 3.56 (s, 3H), 3.52 – 3.26 (m, 4H), 1.37 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 157.2, 138.4, 133.6, 129.3, 129.0, 56.6, 55.2, 52.5, 52.3, 49.9, 42.8, 29.4; **HRMS (ESI$^+$)**: *m/z* calcd for $[C_{16}H_{24}ClNO_5SNa]^+$ ($[M+Na]^+$): 400.0961, found: 400.0950.

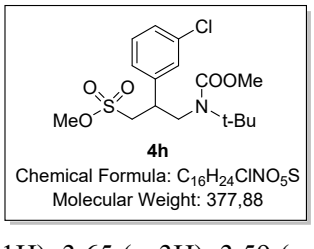
methyl 2-(4-bromophenyl)-3-(*tert*-butyl(methoxycarbonyl)amino)propane-1-sulfonate (**4g**)



4g
Chemical Formula: $C_{16}H_{24}BrNO_5S$
Molecular Weight: 422,33

Following the general procedure B using *N*-allylbenzamide **1d** (74.1 mg), **4g** was obtained as a colorless foam (63.9 mg, 0.151 mmol, 61%).
IR: 2958, 1699, 1489, 1436, 1360, 1339, 1251, 1228, 1177, 1155, 1091, 1074, 1009, 988, 924, 821, 775 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.47 (d, J = 8.4 Hz, 2H), 7.12 (d, J = 8.4 Hz, 2H), 3.80 (ddd, J = 14.3, 5.1, 2.4 Hz, 1H), 3.63 (s, 3H), 3.57 (s, 3H), 3.51 – 3.27 (m, 4H), 1.38 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 157.3, 138.9, 132.0, 129.7, 121.7, 56.6, 55.2, 52.5, 52.3, 49.8, 42.9, 29.5; **HRMS (ESI$^+$)**: *m/z* calcd for $[C_{16}H_{24}BrNO_5SNa]^+$ ($[M+Na]^+$): 444.0456, found: 444.0456.

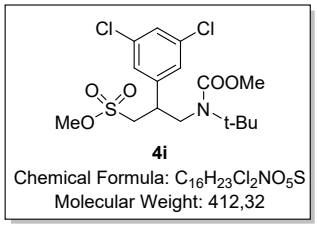
methyl 3-(*tert*-butyl(methoxycarbonyl)amino)-2-(3-chlorophenyl)propane-1-sulfonate (**4h**)



4h
Chemical Formula: $C_{16}H_{24}ClNO_5S$
Molecular Weight: 377,88

Following the general procedure B using *N*-allylbenzamide **1i** (62.9 mg), **4h** was obtained as a colorless foam (59.7 mg, 0.156 mmol, 63%).
IR: 2958, 1701, 1463, 1436, 1379, 1361, 1341, 1291, 1253, 1229, 1171, 1153, 1111, 1087, 989, 915, 815, 728 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.33 – 7.20 (m, 3H), 7.14 (ddd, J = 6.4, 2.5, 1.7 Hz, 1H), 3.91 – 3.75 (m, 1H), 3.65 (s, 3H), 3.59 (s, 3H), 3.52 – 3.30 (m, 4H), 1.39 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 157.2, 141.9, 134.7, 130.1, 128.1, 128.0, 126.3, 56.6, 55.2, 52.5, 52.3, 49.8, 43.1, 29.4; **HRMS (ESI$^+$)**: *m/z* calcd for $[C_{16}H_{24}ClNO_5SNa]^+$ ($[M+Na]^+$): 400.0961, found: 400.0961.

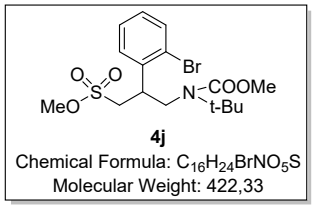
methyl 3-(*tert*-butyl(methoxycarbonyl)amino)-2-(3,5-dichlorophenyl)propane-1-sulfonate (**4i**)



4i
Chemical Formula: $C_{16}H_{23}Cl_2NO_5S$
Molecular Weight: 412,32

Following the general procedure B using *N*-allylbenzamide **1j** (71.6 mg), **4i** was obtained as a colorless foam (63.9 mg, 0.155 mmol, 62%).
IR: 2958, 1697, 1589, 1569, 1435, 1361, 1338, 1251, 1227, 1179, 1157, 1093, 988, 858, 853, 798, 776 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.28 (t, *J* = 1.8 Hz, 1H), 7.13 (d, *J* = 1.9 Hz, 2H), 3.85 – 3.70 (m, 1H), 3.65 (s, 6H), 3.49 – 3.31 (m, 4H), 1.38 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 157.2, 143.3, 135.3, 128.0, 126.6, 56.7, 55.2, 52.4, 52.2, 49.6, 43.0, 29.5; **HRMS (ESI$^+$)**: *m/z* calcd for $[C_{16}H_{24}Cl_2NO_5SNa]^+$ ([M+Na]$^+$): 435.0572, found: 435.0570.

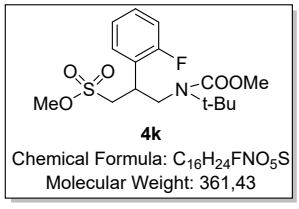
methyl 2-(2-bromophenyl)-3-(*tert*-butyl(methoxycarbonyl)amino)propane-1-sulfonate (**4j**)



4j
Chemical Formula: $C_{16}H_{24}BrNO_5S$
Molecular Weight: 422,33

Following the general procedure B using *N*-allylbenzamide **1m** (74.1 mg), **4j** was obtained as a colorless foam (69.6 mg, 0.165 mmol, 66%).
IR: 2958, 1699, 1474, 1435, 1361, 1342, 1228, 1178, 1155, 1092, 1022, 987, 812, 778, 756 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$, mixture of rotamers) δ 7.59 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.44 – 7.23 (m, 2H), 7.14 (ddd, *J* = 8.0, 7.0, 2.0 Hz, 1H), 4.22 (br s, 1H), 3.90 – 3.66 (m, 1H), 3.71 (s, 3H), 3.60 (s, 3H), 3.57 – 3.21 (m, 3H), 1.42 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$, mixture of rotamers) δ 157.3, 146.7, 139.2, 133.4, 129.0, 127.8, 125.1, 56.6, 55.1, 52.3, 52.0, 49.1, 40.9, 29.4; **HRMS (ESI$^+$)**: *m/z* calcd for $[C_{16}H_{24}BrNO_5SNa]^+$ ([M+Na]$^+$): 444.0456, found: 444.0453.

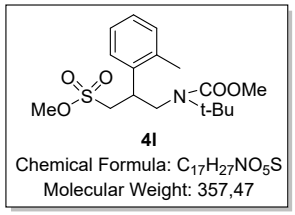
methyl 3-(*tert*-butyl(methoxycarbonyl)amino)-2-(2-fluorophenyl)propane-1-sulfonate (**4k**)



4k
Chemical Formula: $C_{16}H_{24}FNO_5S$
Molecular Weight: 361,43

Following the general procedure B using *N*-allylbenzamide **1l** (58.8 mg), **4k** was obtained as a colorless foam (68.2 mg, 0.189 mmol, 76%).
IR: 2959, 1699, 1493, 1456, 1436, 1397, 1361, 1347, 1251, 1229, 1175, 1156, 1087, 987, 758 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.34 – 7.20 (m, 2H), 7.19 – 7.01 (m, 2H), 3.89 (dd, *J* = 14.6, 7.8 Hz, 1H), 3.78 – 3.57 (m, 2H), 3.64 (s, 3H), 3.63 (s, 3H), 3.57 – 3.43 (m, 2H), 1.39 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 161.3 (d, *J* = 245.6 Hz), 157.0, 130.6 (d, *J* = 5.0 Hz), 129.5 (d, *J* = 8.6 Hz), 126.5 (d, *J* = 13.6 Hz), 124.5 (d, *J* = 3.5 Hz), 115.9 (d, *J* = 22.2 Hz), 56.6, 55.2, 52.2, 51.4 (d, *J* = 3.6 Hz), 48.3, 38.9, 29.3; **^{19}F {^{13}C} NMR (282 MHz, $CDCl_3$)** δ -116.3; **HRMS (ESI$^+$)**: *m/z* calcd for $[C_{16}H_{24}NNO_5SNa]^+$ ([M+Na]$^+$): 384.1257, found: 384.1248.

methyl 3-(*tert*-butyl(methoxycarbonyl)amino)-2-(o-tolyl)propane-1-sulfonate (**4l**)

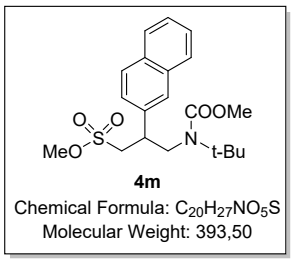


4l
Chemical Formula: $C_{17}H_{27}NO_5S$
Molecular Weight: 357,47

Following the general procedure B using *N*-allylbenzamide **1k** (57.8 mg), **4l** was obtained as a colorless foam (54.5 mg, 0.152 mmol, 61%).

IR: 2958, 1701, 1463, 1436, 1379, 1361, 1341, 1291, 1253, 1229, 1171, 1153, 1111, 1087, 989, 915, 815, 728 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.26 – 7.05 (m, 4H), 3.95 – 3.62 (m, 2H), 3.73 (s, 3H), 3.48 (d, *J* = 6.4 Hz, 2H), 3.40 (s, 3H), 3.27 (dd, *J* = 14.3, 5.4 Hz, 1H), 2.40 (s, 3H), 1.37 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 157.5, 138.7, 136.9, 130.9, 127.3, 126.5, 126.4, 56.6, 55.2, 52.7, 52.4, 49.8, 37.5, 29.4, 19.8; **HRMS (ESI$^+$)**: *m/z* calcd for $[C_{17}H_{27}NO_5SNa]^+$ ([M+Na]$^+$): 380.1508, found: 380.1504.

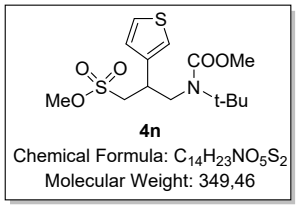
methyl 3-(*tert*-butyl(methoxycarbonyl)amino)-2-(naphthalen-2-yl)propane-1-sulfonate (**4m**)



4m
Chemical Formula: $C_{20}H_{27}NO_5S$
Molecular Weight: 393,50

Following the general procedure B using *N*-allylbenzamide **1n** (66.9 mg), **4m** was obtained as a colorless foam (62.4 mg, 0.159 mmol, 63%).
IR: 2958, 1697, 1436, 1358, 1332, 1251, 1229, 1170, 1161, 1092, 987, 818, 749, 731 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.92 – 7.73 (m, 3H), 7.71 (d, *J* = 1.8 Hz, 1H), 7.54 – 7.40 (m, 2H), 7.37 (dd, *J* = 8.5, 1.8 Hz, 1H), 4.06 – 3.82 (m, 1H), 3.72 – 3.51 (m, 3H), 3.64 (s, 3H), 3.51 – 3.37 (m, 1H), 3.45 (s, 3H), 1.41 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 157.3, 137.2, 133.5, 132.9, 128.7, 127.8, 127.8, 127.2, 126.5, 126.2, 125.3, 56.6, 55.2, 52.6, 52.3, 50.0, 43.5, 29.5; **HRMS (ESI$^+$)**: *m/z* calcd for $[C_{20}H_{27}NO_5SNa]^+$ ([M+Na]$^+$): 416.1508, found: 416.1507.

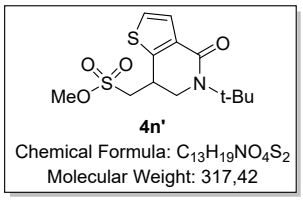
methyl 3-(*tert*-butyl(methoxycarbonyl)amino)-2-(thiophen-3-yl)propane-1-sulfonate (**4n**)



4n
Chemical Formula: $C_{14}H_{23}NO_5S_2$
Molecular Weight: 349,46

Following the general procedure B using *N*-allylthiophene-3-carboxamide **1o** (55.8 mg), **4n** was obtained as a yellow foam (20.7 mg, 0.059 mmol, 24%).
IR: 2958, 1698, 1436, 1397, 1360, 1332, 1254, 1227, 1180, 1168, 1093, 990, 815, 777 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.32 (dd, *J* = 5.0, 2.9 Hz, 1H), 7.13 (dd, *J* = 2.9, 1.4 Hz, 1H), 7.00 (dd, *J* = 5.0, 1.4 Hz, 1H), 3.84 (dd, *J* = 14.8, 9.0 Hz, 1H), 3.69 – 3.60 (m, 1H), 3.64 (s, 3H), 3.58 (s, 3H), 3.48 – 3.30 (m, 3H), 1.38 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 157.3, 140.4, 126.4, 126.4, 122.6, 56.6, 55.3, 53.0, 52.3, 49.6, 38.6, 29.5; **HRMS (ESI$^+$)**: *m/z* calcd for $[C_{14}H_{23}NO_5SNa]^+$ ([M+Na]$^+$): 372.0915, found: 372.0914.

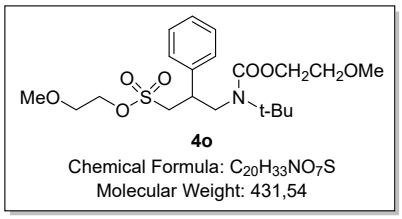
methyl (5-(*tert*-butyl)-4-oxo-4,5,6,7-tetrahydrothieno[3,2-c]pyridin-7-yl)methanesulfonate (**4n'**)



4n'
Chemical Formula: $C_{13}H_{19}NO_4S_2$
Molecular Weight: 317,42

Following the general procedure B using *N*-allylthiophene-3-carboxamide **1o** (55.8 mg), **4n'** was obtained as a yellow foam (36.5 mg, 0.115 mmol, 46%).
IR: 2963, 2926, 1637, 1459, 1435, 1413, 1358, 1331, 1305, 1203, 1178, 1162, 983, 775 cm^{-1}; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.46 (d, *J* = 5.0 Hz, 1H), 6.94 (d, *J* = 5.0 Hz, 1H), 4.04 (dd, *J* = 13.1, 3.5 Hz, 1H), 3.91 (s, 3H), 3.74 (dd, *J* = 13.1, 3.9 Hz, 1H), 3.59 – 3.51 (m, 1H), 3.42 (dd, *J* = 14.4, 9.7 Hz, 1H), 3.20 (dd, *J* = 14.5, 3.3 Hz, 1H), 1.53 (s, 9H); **^{13}C {^{1}H} NMR** (126 MHz, $CDCl_3$) δ 157.3, 140.4, 126.4, 126.4, 122.6, 56.6, 55.3, 53.0, 52.3, 49.6, 38.6, 29.5; **HRMS (ESI$^+$)**: *m/z* calcd for $[C_{13}H_{20}NO_4S]^+$ ([M+H]$^+$): 318.0834, found: 318.0829.

2-methoxyethyl 3-(*tert*-butyl((2-methoxyethoxy)carbonyl)amino)-2-phenylpropane-1-sulfonate (**4o**)



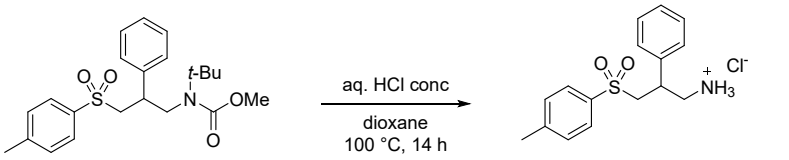
4o
Chemical Formula: $C_{20}H_{33}NO_7S$
Molecular Weight: 431,54

Following the general procedure B using *N*-allylbenzamide **1a** (54.3 mg) and 2-methoxyethanol (1.2 mL) instead of methanol as co-solvent, **4o** was obtained as a pale yellow oil (34.2 mg, 0.0793 mmol, 32%).

IR: 2930, 1699, 1456, 1389, 1344, 1195, 1174, 1156, 1129, 1090, 1077, 1024, 920, 703 cm^{-1}; **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.38 – 7.23 (m, 5H), 4.30 (ddd, *J* = 11.9, 5.4, 4.1 Hz, 1H), 4.10 (ddd, *J* = 11.9, 5.2, 3.9 Hz, 1H), 3.98 (t, *J* = 4.7 Hz, 2H), 3.87 (dd, *J* = 14.9, 9.2 Hz, 1H), 3.67 – 3.58 (m, 3H), 3.58 – 3.47 (m, 2H), 3.41 (s, 3H), 3.40 – 3.29 (m, 3H), 3.27 (s, 3H), 1.39 (s, 9H); **^{13}C {^{1}H} NMR** (75 MHz, $CDCl_3$) δ 156.3, 140.1, 128.9, 128.1, 127.7, 70.9, 70.1, 68.3, 64.0, 59.0, 58.9, 56.7, 53.3, 50.1, 43.3, 29.3; **HRMS (ESI$^+$)**: *m/z* calcd for $[C_{20}H_{33}NO_7SNa]^+$ ($[M+Na]^+$): 454.1875, found: 454.1877.

5. Synthesis of protecting-group free β-arylethylamines 5-6

2-phenyl-3-tosylpropan-1-ammonium chloride (**5**)



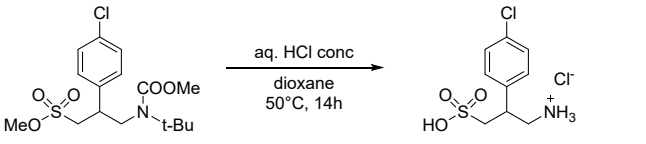
3a

5, 87%
Chemical Formula: $C_{16}H_{20}NO_2S^+Cl^-$
Molecular Weight: 325,85

To a stirred solution of methyl *tert*-butyl(2-phenyl-3-tosylpropyl)carbamate **3a** (161,4 mg, 0.4 mmol) in dioxane (2.4 mL) was added aq. conc HCl (37%, 1.9 mL) and the reaction mixture was stirred overnight at 100 °C. The volatiles were evaporated. The residue was recrystallized from propan-2-ol to afford the ammonium chloride **5** as a beige solid (114.3 mg, 0.35 mmol, 87%).

IR: 2863, 1597, 1496, 1288, 1142, 1083, 941, 769 cm^{-1}; **^{1}H NMR** (300 MHz, MeOD) δ 7.72 – 7.63 (m, 2H), 7.39 – 7.25 (m, 5H), 7.30 – 7.15 (m, 2H), 3.78 (dd, *J* = 14.5, 6.3 Hz, 1H), 3.66 (dd, *J* = 14.5, 6.0 Hz, 1H), 3.64 – 3.48 (m, 2H), 3.34 – 3.19 (m, 1H), 2.43 (s, 3H). **^{13}C {^{1}H} NMR** (75 MHz, MeOD) δ 146.4, 138.7, 137.7, 131.0, 130.5, 129.4, 129.1 (2 overlapping peaks), 59.9, 44.9, 40.6, 21.6; **HRMS (ESI$^+$):** *m/z* calcd for $[C_{16}H_{20}NO_2S]^+$ ($[M]^+$) : 290.1215, found: 290.1215.

2-(4-chlorophenyl)-3-sulfopropan-1-amonium chloride (**6**)



4f

6, 95%
Chemical Formula: $C_9H_{13}ClNO_3S^+Cl^-$
Molecular Weight: 286,17

To a stirred solution of methyl 3-(*tert*-butyl(methoxycarbonyl)amino)-2-(4-chlorophenyl)propane-1-sulfonate **4f** (160 mg; 0.42 mmol) in dioxane (2.6 mL) at 0°C was added aq. conc HCl (37%, 2.9 mL) and the reaction mixture was stirred overnight at 50°C. The residue was triturated with Et_2O to afford the ammonium chloride **6** as a beige solid (114.2 mg, 0.40 mmol, 95%).

IR: 3069, 2949, 1619, 1496, 1419, 1251, 1202, 1175, 1093, 1037, 1018, 911, 842, 781, 722, 666 cm^{-1}; **^{1}H NMR** (300 MHz, D_2O) δ 8.10 – 6.87 (m, 4H), 3.97 – 3.43 (m, 2H), 3.43 – 2.87 (m, 3H); **^{13}C {^{1}H} NMR** (126 MHz, D_2O) δ 136.74, 133.38, 129.43, 129.24, 54.07, 43.43, 39.75; **HRMS (ESI$^+$):** *m/z* calcd for $[C_9H_{13}ClNO_3S]^+$ ($[M]^+$) : 250.0305, found: 250.0303.

6. Cyclic voltammetry analyses

Cyclic voltammetry analyses were carried out in a three-electrode cell with a potentiostat (Origalys 500). Working electrode: Glassy Carbon (3 mm diameter); Counter electrode: Pt wire; Reference electrode: AgCl/Ag (aqueous NaCl 3 M).

External reference: The redox potential of ferrocene (Fc^+/Fc) was measured in the solvent system (Figure S1).
E (initial) = 0 V; E (high) = 0.8 V; E (low) = 0 V; 2 segments; sweep rate = 200 $mV.s^{-1}$.

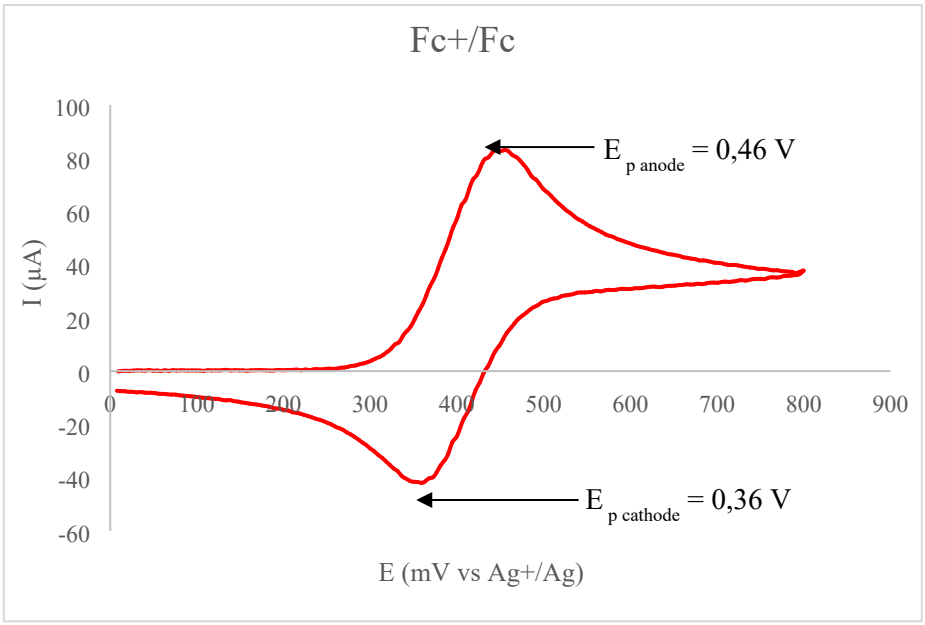


Figure S1: Cyclic voltammogram of Fc^{+}/Fc (0.002 M) + *n*-Bu_4NPF_6 (0.1 M) in MeCN/MeOH (6:4)

$Fc^{+/0}$: $E_{1/2}$ = +0.41 V *vs* $Ag^{+/0}$ in MeCN/MeOH (6:4)

0.0025 M analyte and 0.1 M *n*-Bu_4NPF_6 in MeCN/MeOH (6:4, 11 mL).
E (initial) = 0 V; E (high) = 2.5 V; E (low) = 0 V; 2 segments; Sweep rate: 100 $mV.s^{-1}$.

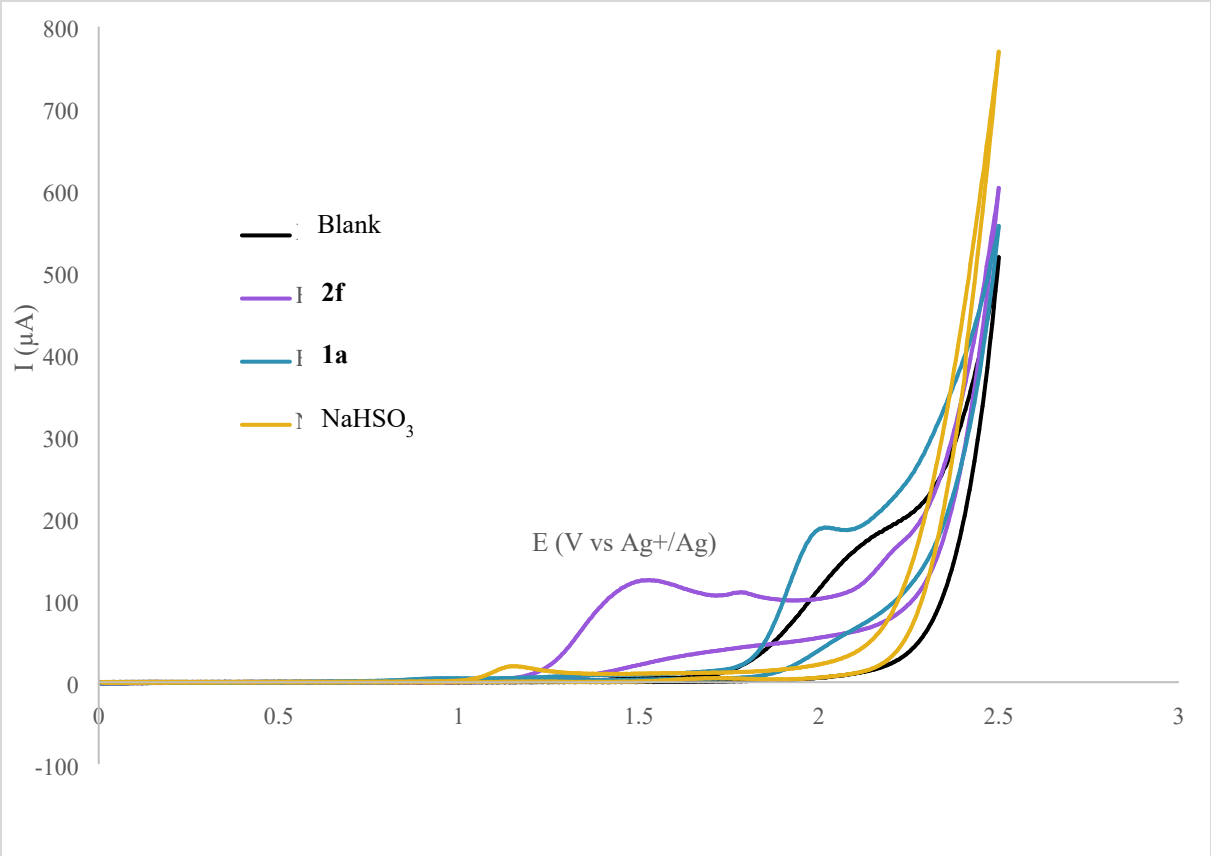


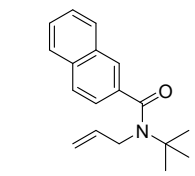
Figure S2: Cyclic voltammograms of 0.1 M *n*-Bu_4NPF_6 solutions in MeCN/MeOH (6:4): none (black curve); **2f** (purple curve); $NaHSO_3$ (yellow curve); **1a** (blue curve).

1a: $E_{p/2}$ = 1.90 V *vs* $Ag^{+/0}$ = 1.49 V *vs* $Fc^{+/0}$

2f: $E_{p/2}$ = 1.34 V *vs* $Ag^{+/0}$ = 0.93 V *vs* $Fc^{+/0}$

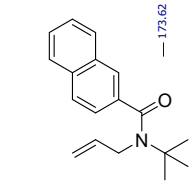
$NaHSO_3$: $E_{p/2}$ = 1.08 V *vs* $Ag^{+/0}$ = 0.67 V *vs* $Fc^{+/0}$ (the current response is very low because $NaHSO_3$ is barely soluble in the solvent system)

7. NMR spectra



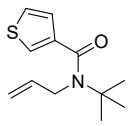
1n

^{1}H NMR (300 MHz, $CDCl_3$)



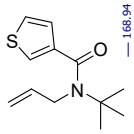
1n

^{13}C NMR (75 MHz, $CDCl_3$)



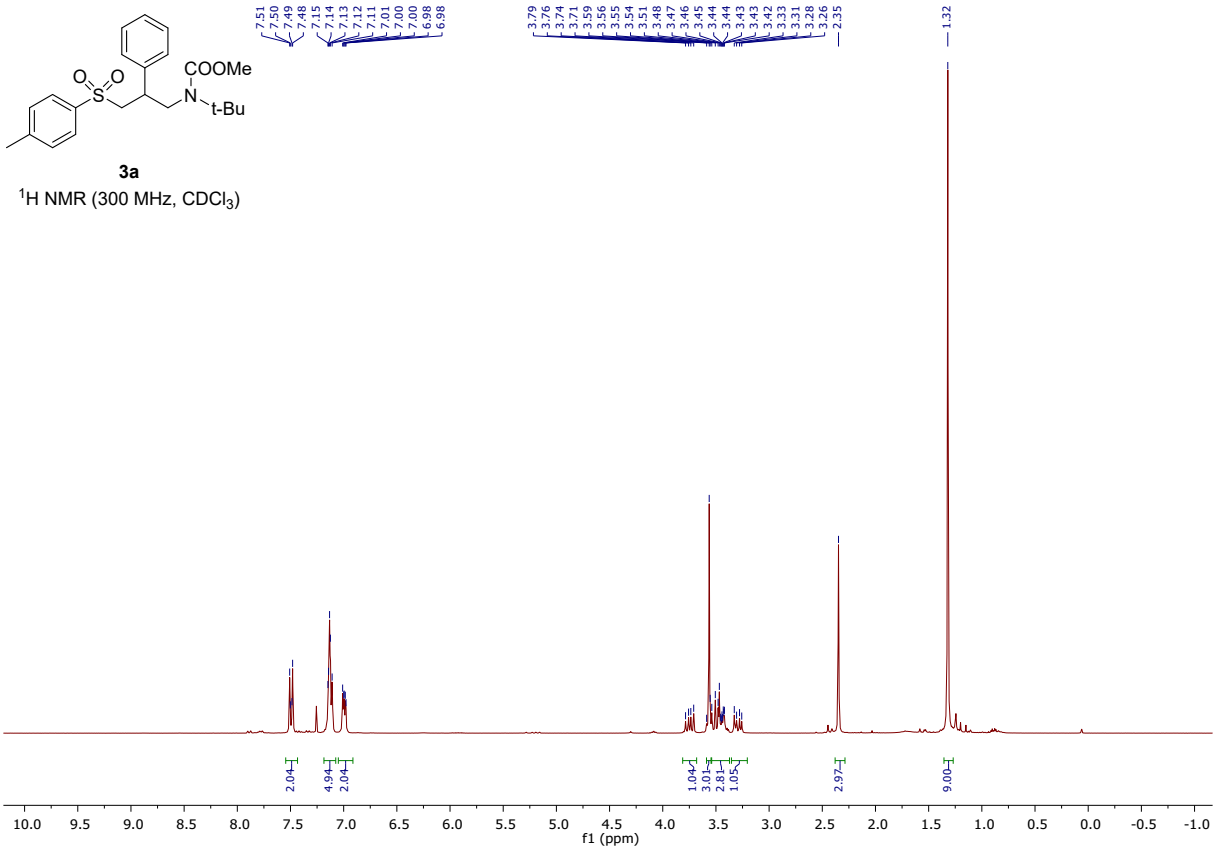
1o

^{1}H NMR (300 MHz, $CDCl_3$)



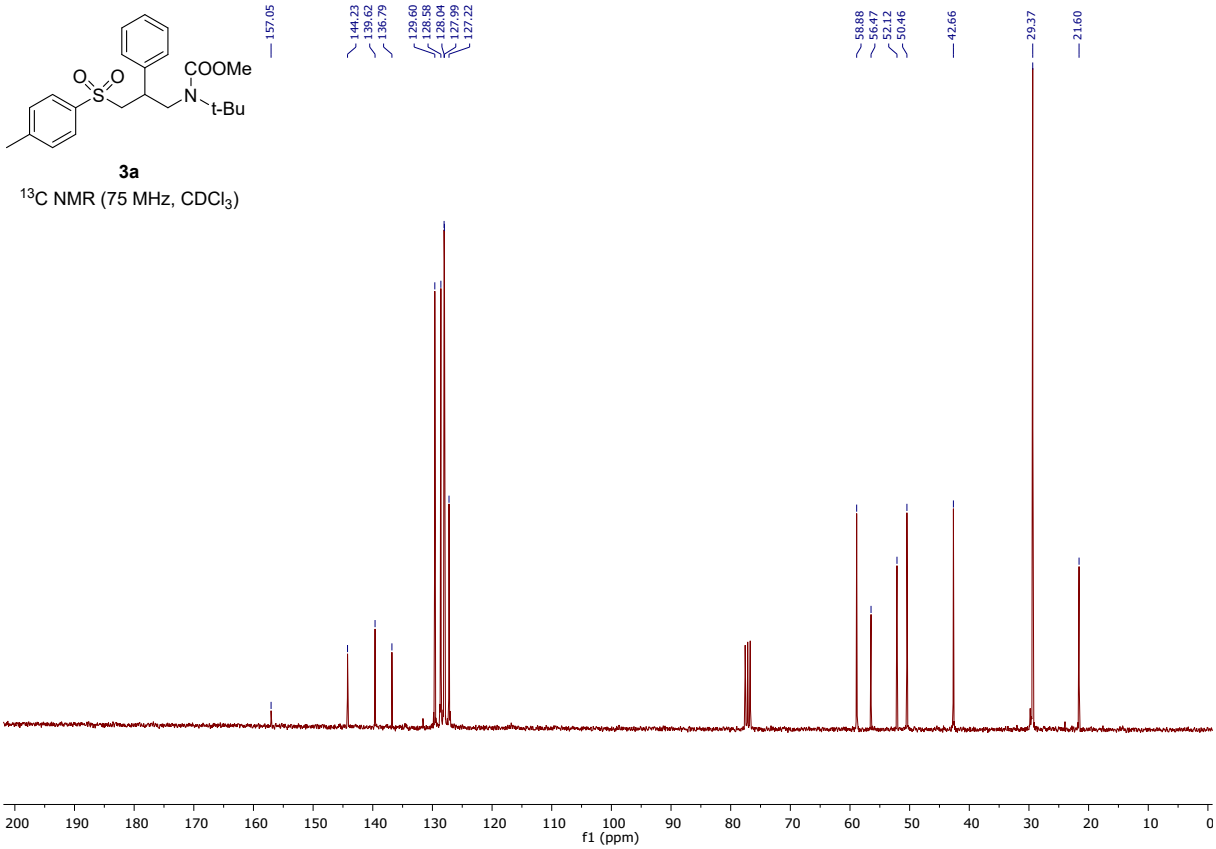
1o

^{13}C NMR (75 MHz, $CDCl_3$)



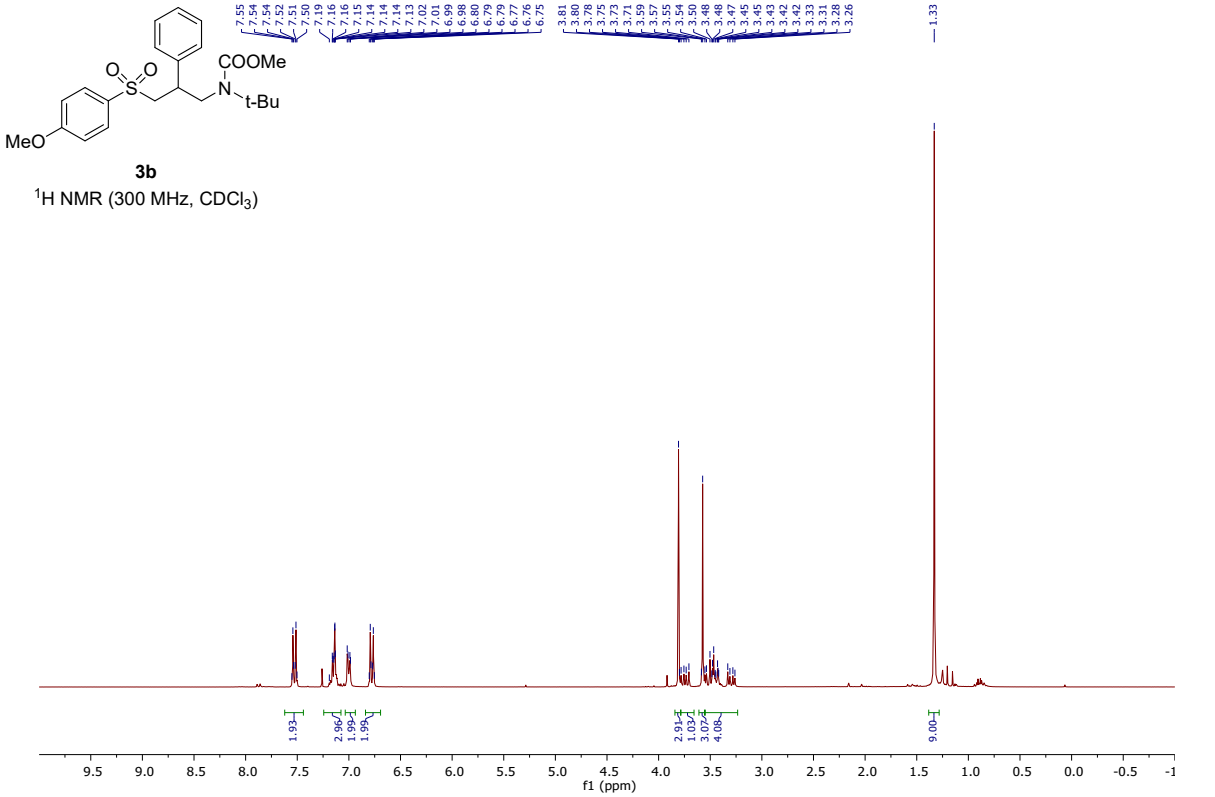
3a

^{1}H NMR (300 MHz, $CDCl_3$)



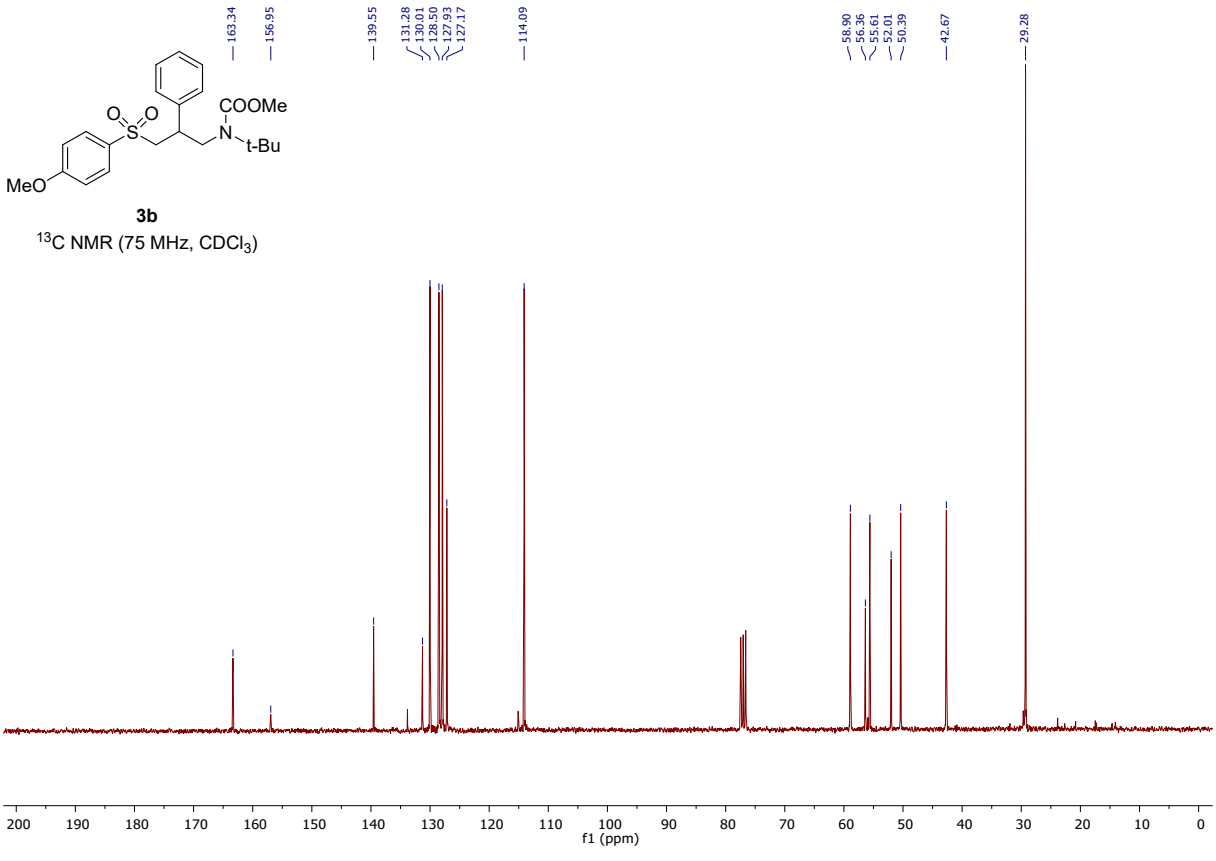
3a

^{13}C NMR (75 MHz, $CDCl_3$)



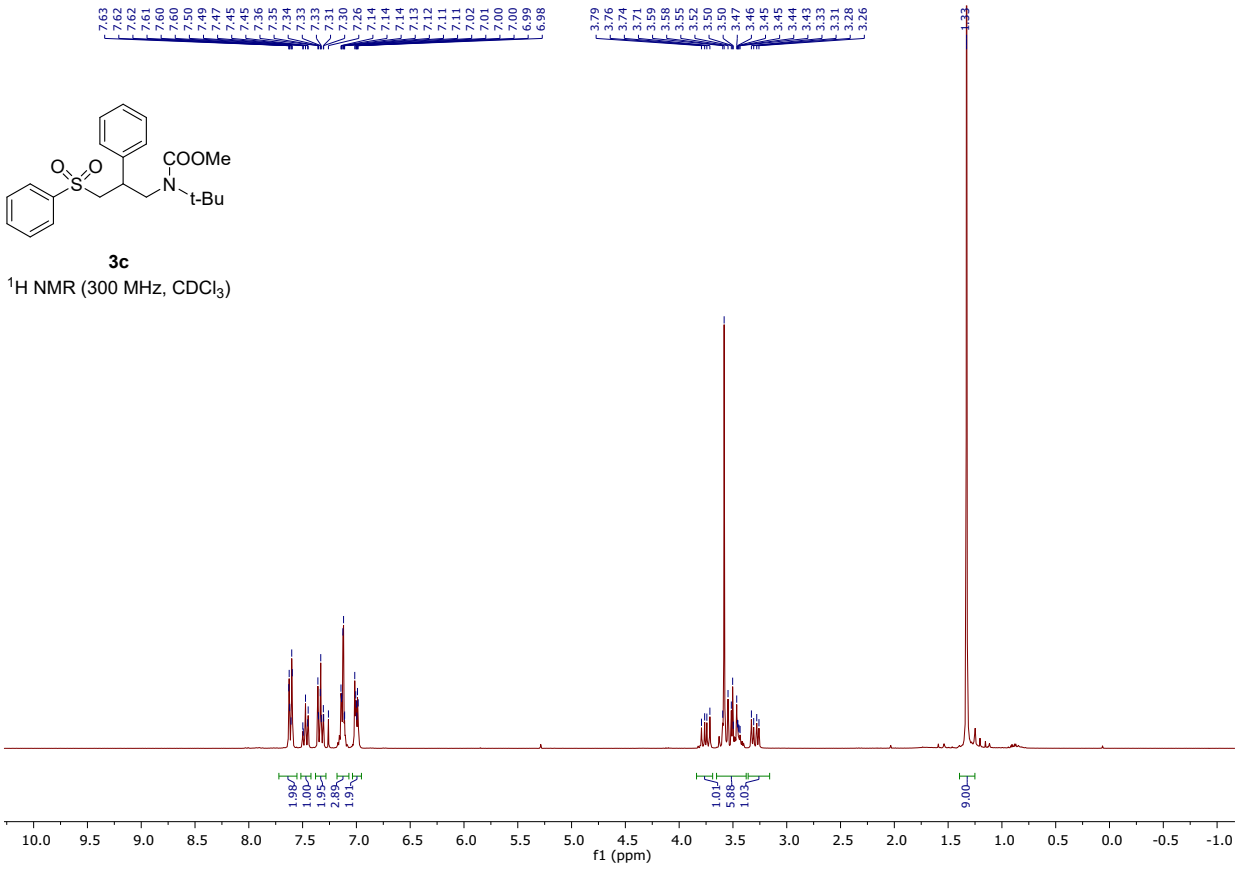
3b

^{1}H NMR (300 MHz, $CDCl_3$)



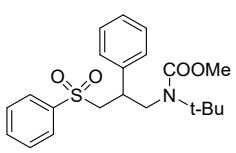
3b

^{13}C NMR (75 MHz, $CDCl_3$)



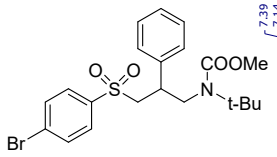
3c

^{1}H NMR (300 MHz, $CDCl_3$)



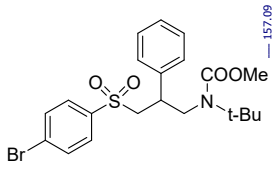
3c

^{13}C NMR (75 MHz, $CDCl_3$)



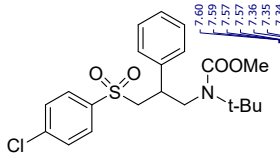
3d

^{1}H NMR (300 MHz, $CDCl_3$)



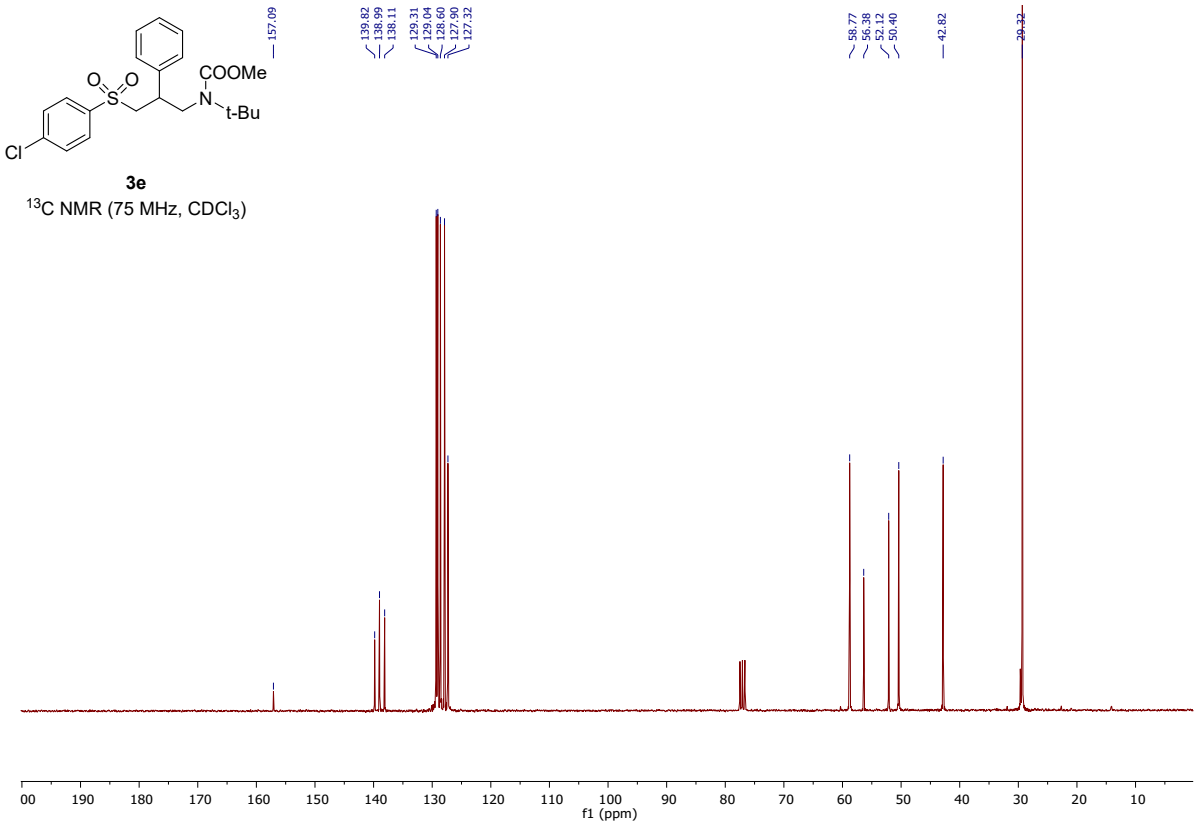
3d

^{13}C NMR (75 MHz, $CDCl_3$)



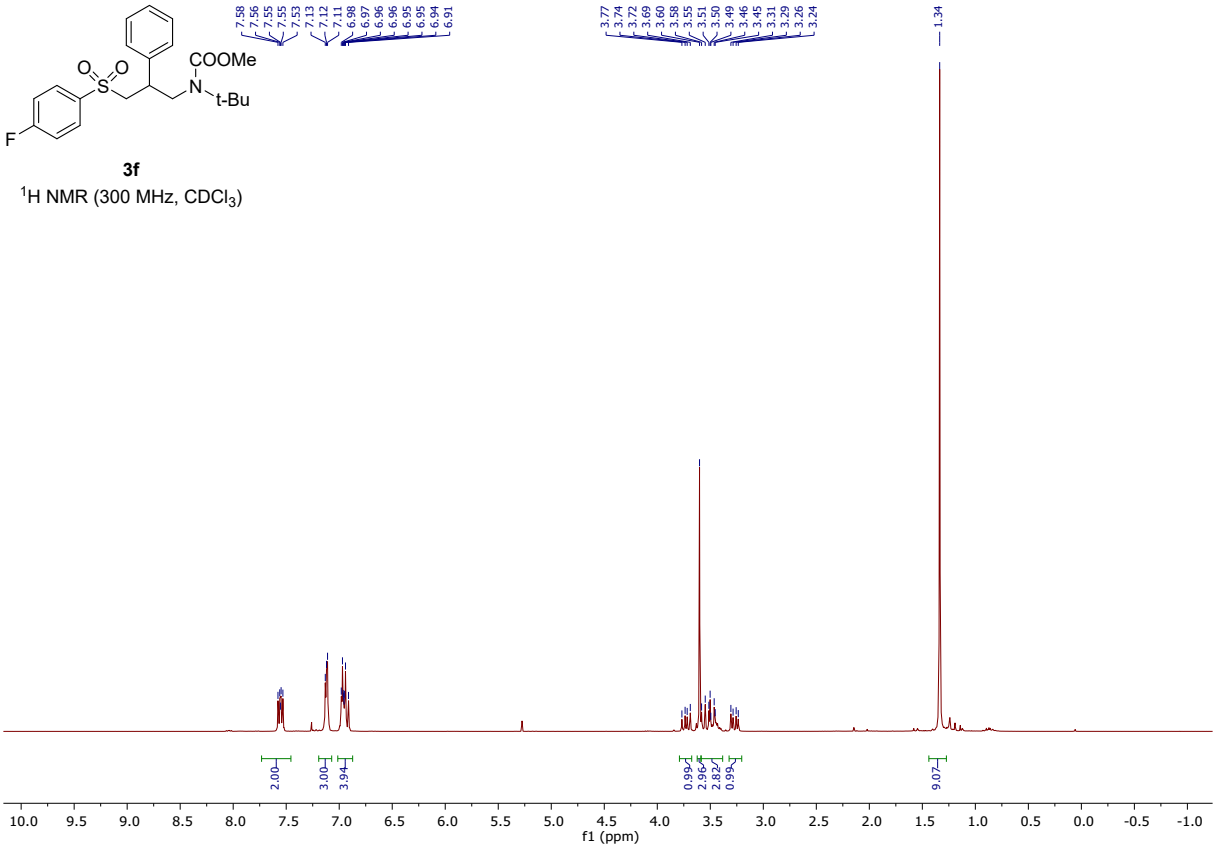
3e

^{1}H NMR (300 MHz, $CDCl_3$)



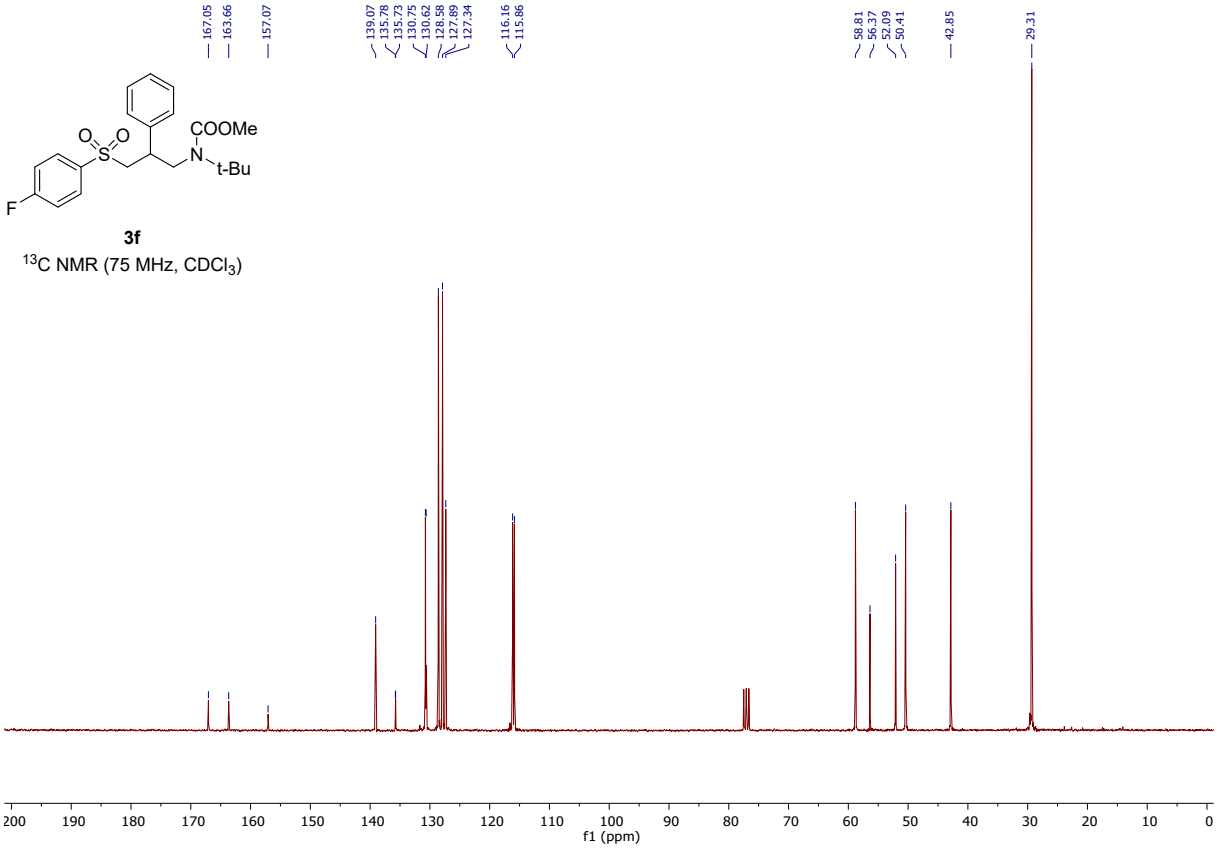
3e

^{13}C NMR (75 MHz, $CDCl_3$)



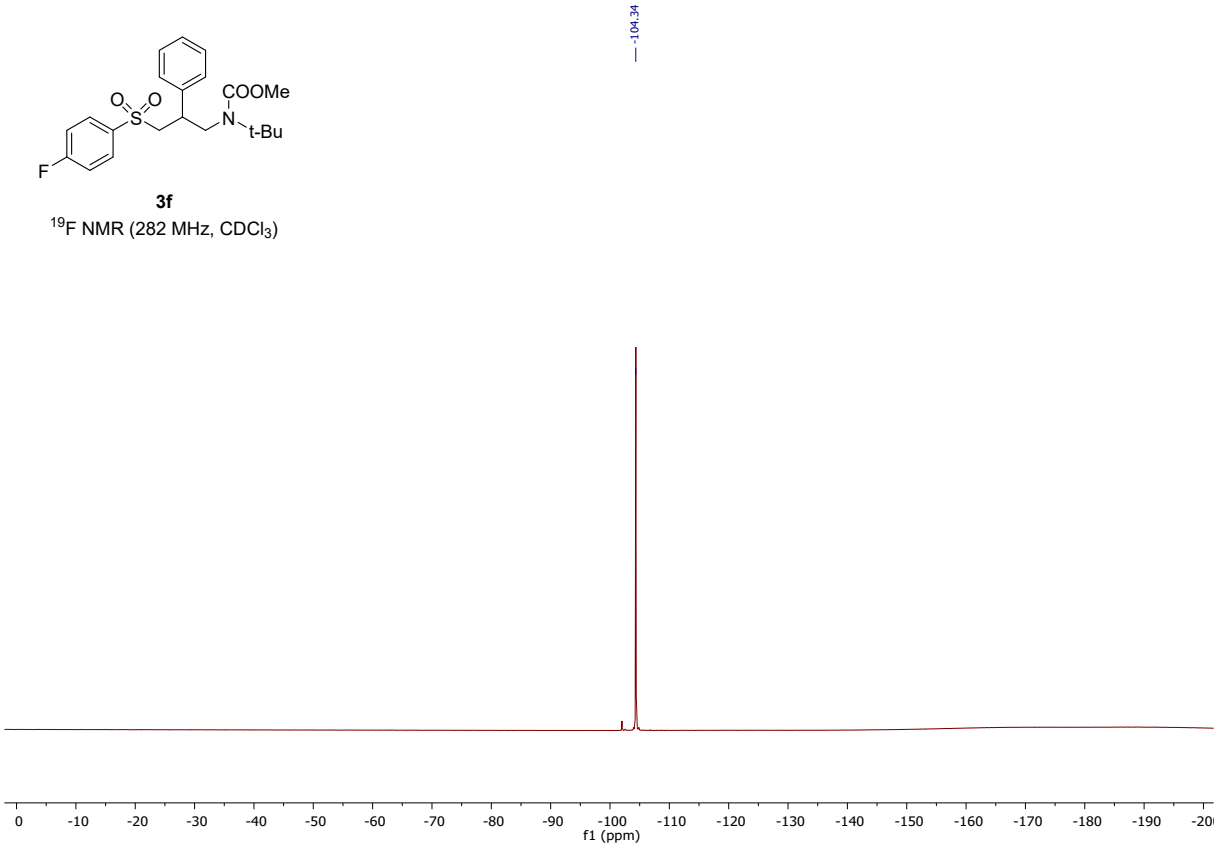
3f

^{1}H NMR (300 MHz, $CDCl_3$)



3f

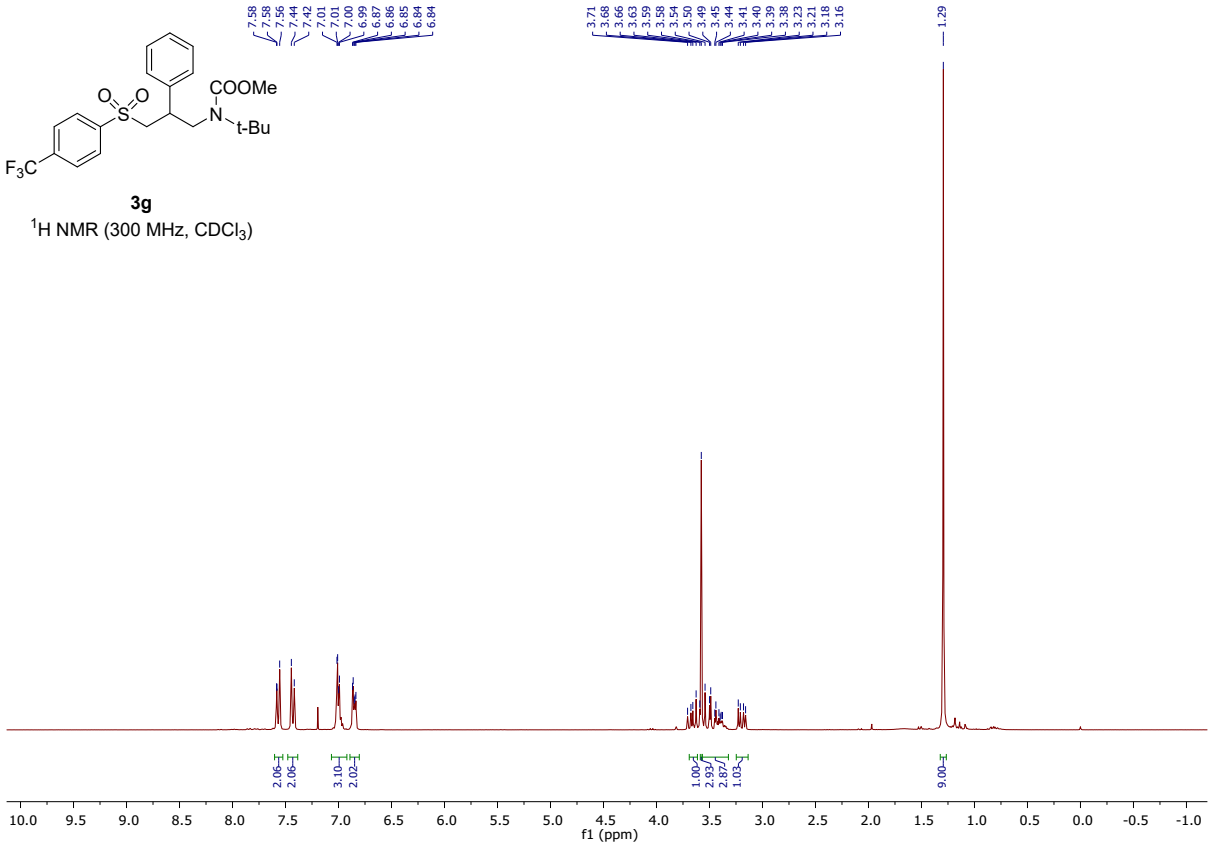
^{13}C NMR (75 MHz, $CDCl_3$)

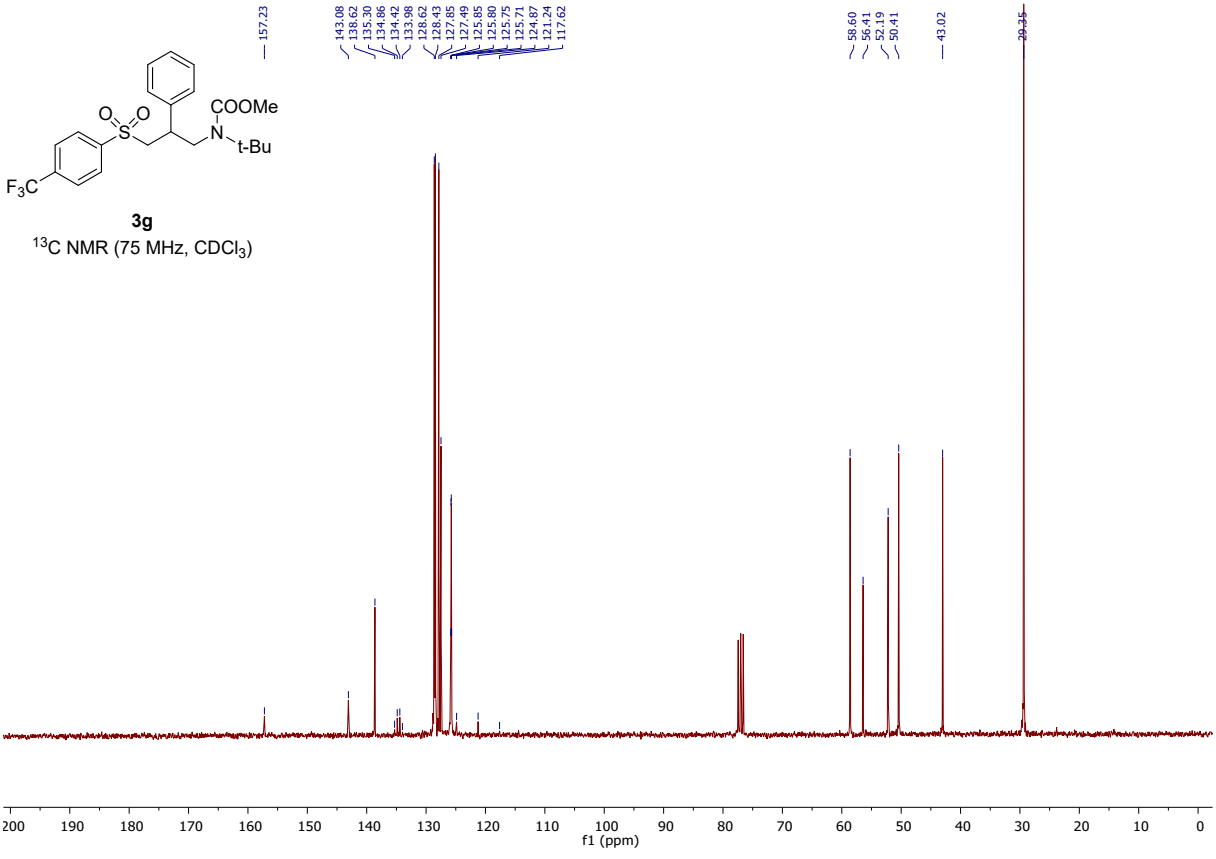


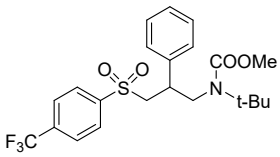
3f

^{19}F NMR (282 MHz, $CDCl_3$)

3g

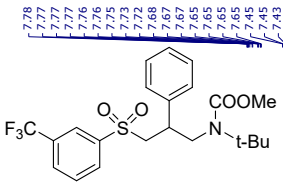

COOMe
t-Bu
F3C
3g
1H NMR (300 MHz, CDCl3)
f1 (ppm)


COOMe
t-Bu
F3C
3g
13C NMR (75 MHz, CDCl3)
f1 (ppm)



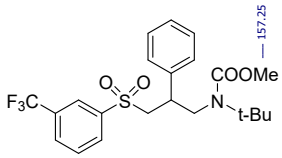
3g

^{19}F NMR (282 MHz, $CDCl_3$)



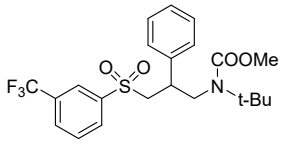
3h

^{1}H NMR (300 MHz, $CDCl_3$)



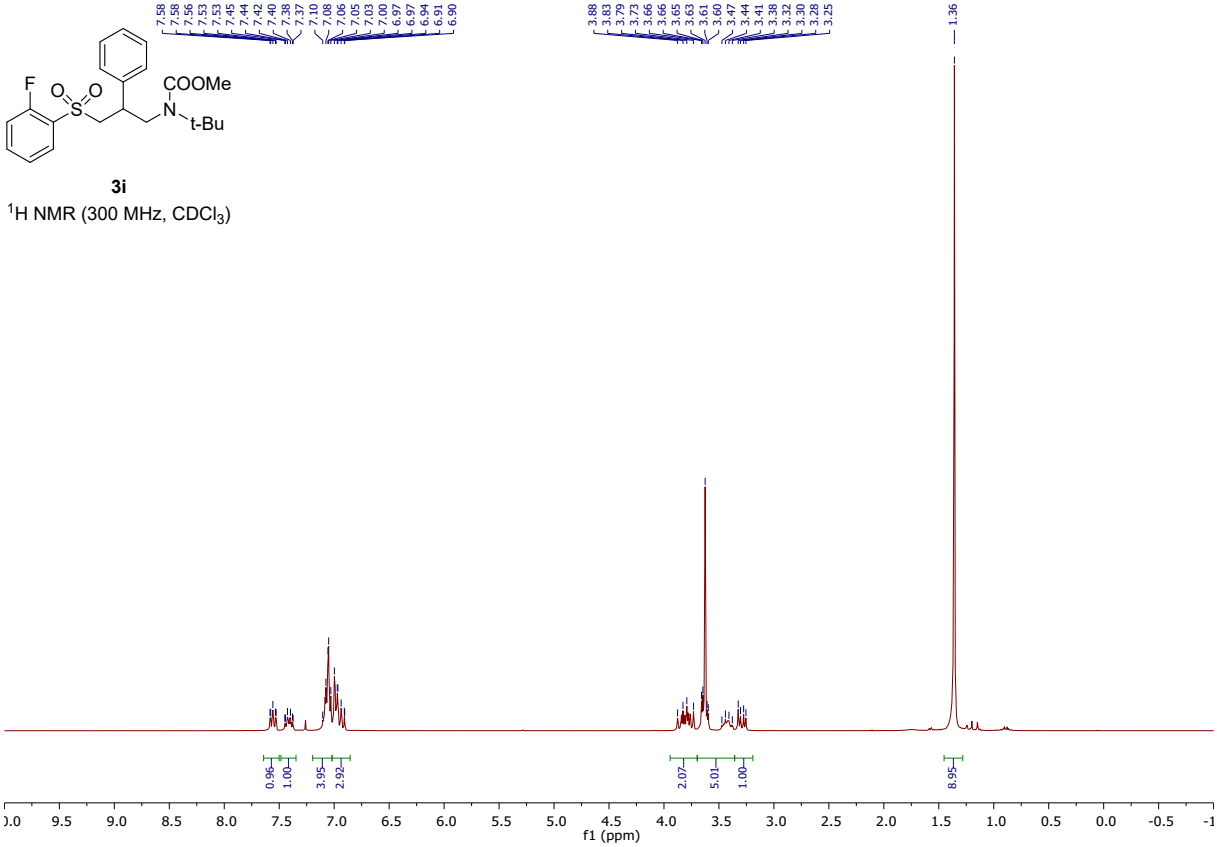
3h

^{13}C NMR (75 MHz, $CDCl_3$)



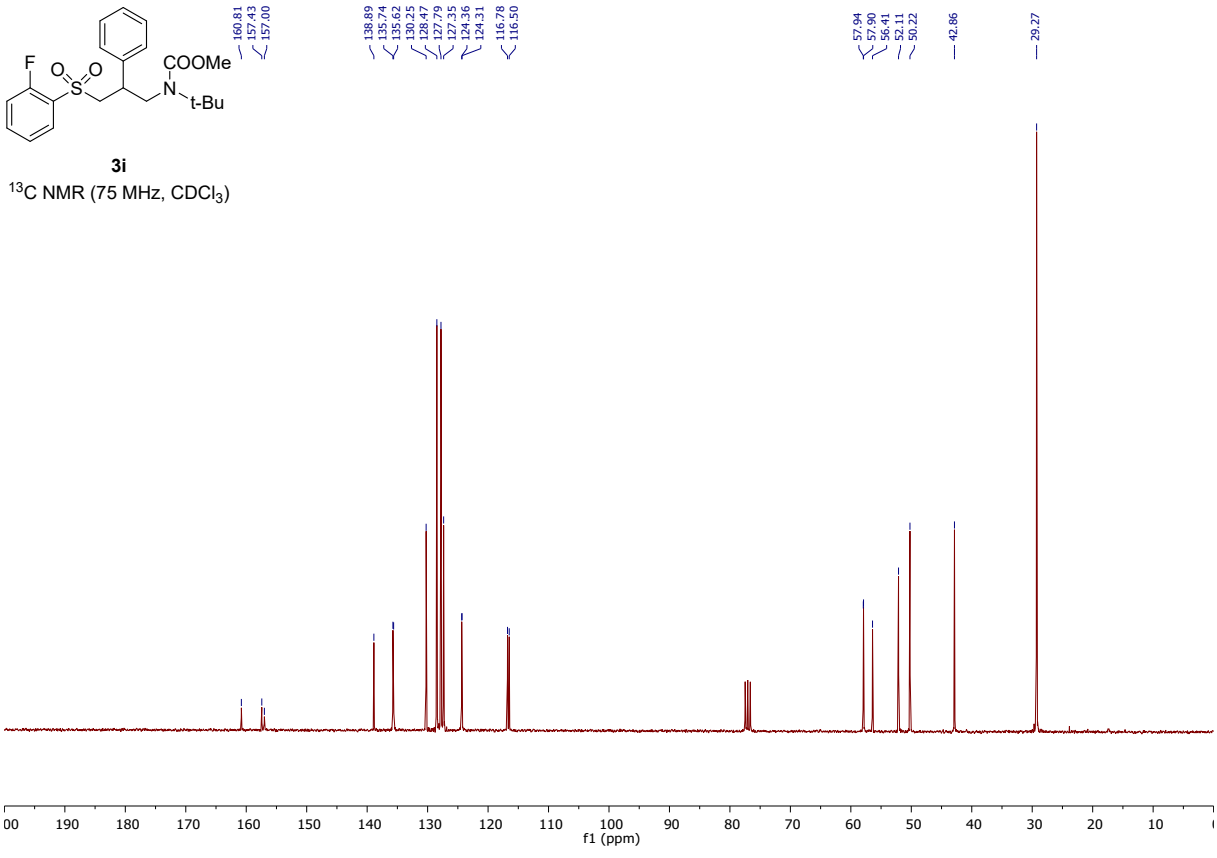
3h

^{19}F NMR (282 MHz, $CDCl_3$)



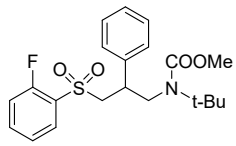
3i

^{1}H NMR (300 MHz, $CDCl_3$)



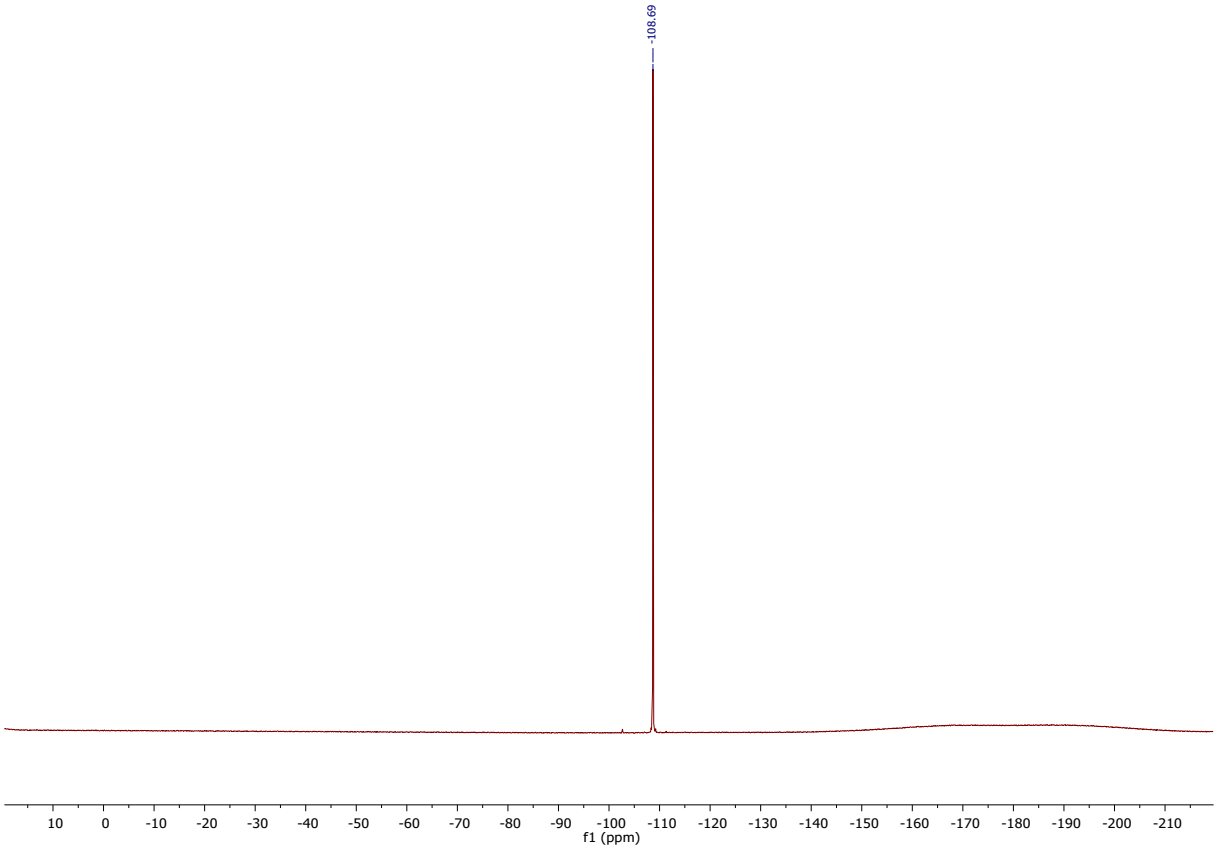
3i

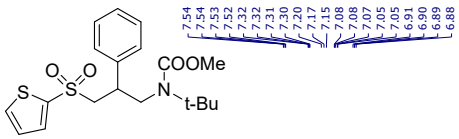
^{13}C NMR (75 MHz, $CDCl_3$)



3i

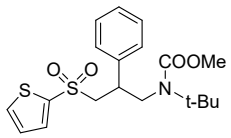
^{19}F NMR (282 MHz, $CDCl_3$)





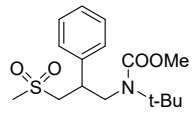
3j

^{1}H NMR (300 MHz, $CDCl_3$)



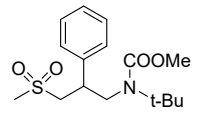
3j

^{13}C NMR (75 MHz, $CDCl_3$)



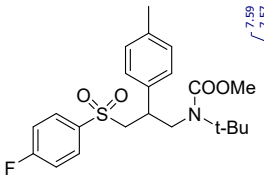
3k

^{1}H NMR (300 MHz, $CDCl_3$)



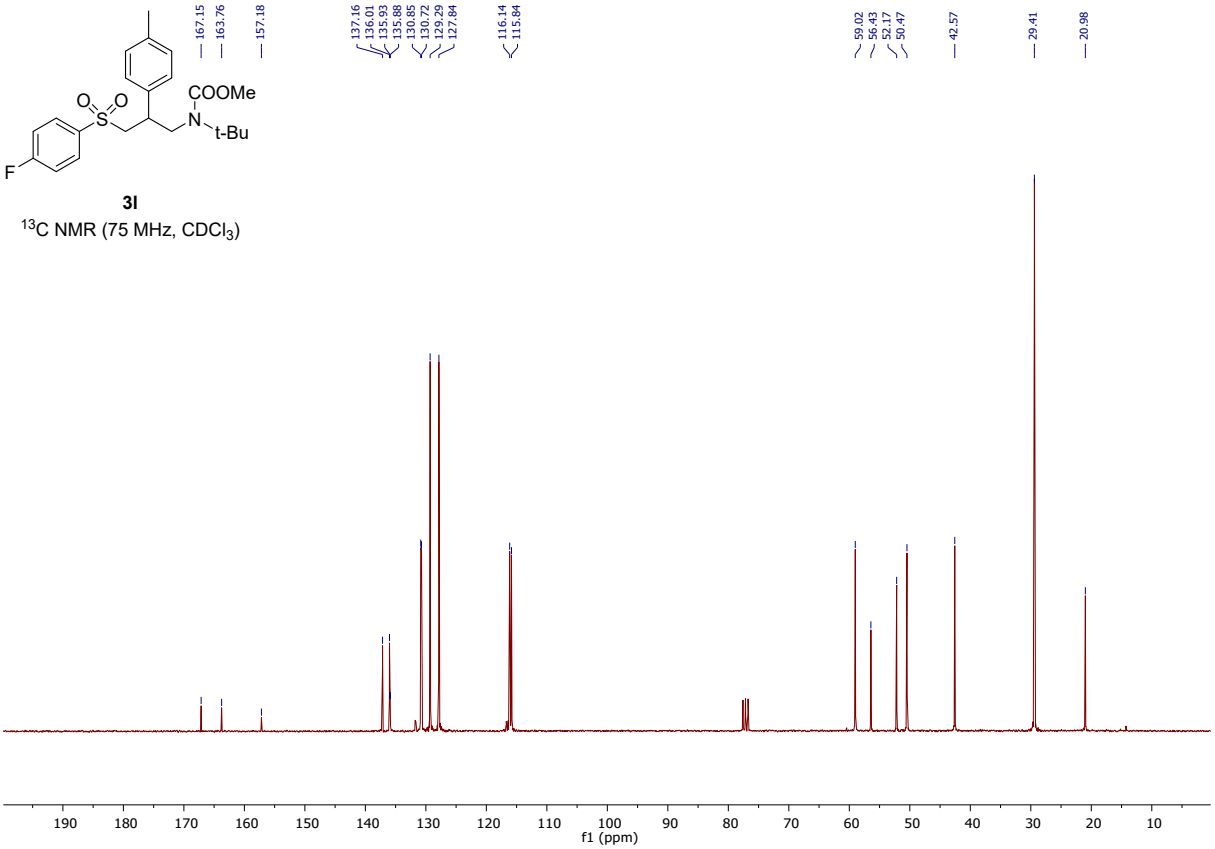
3k

^{13}C NMR (75 MHz, $CDCl_3$)



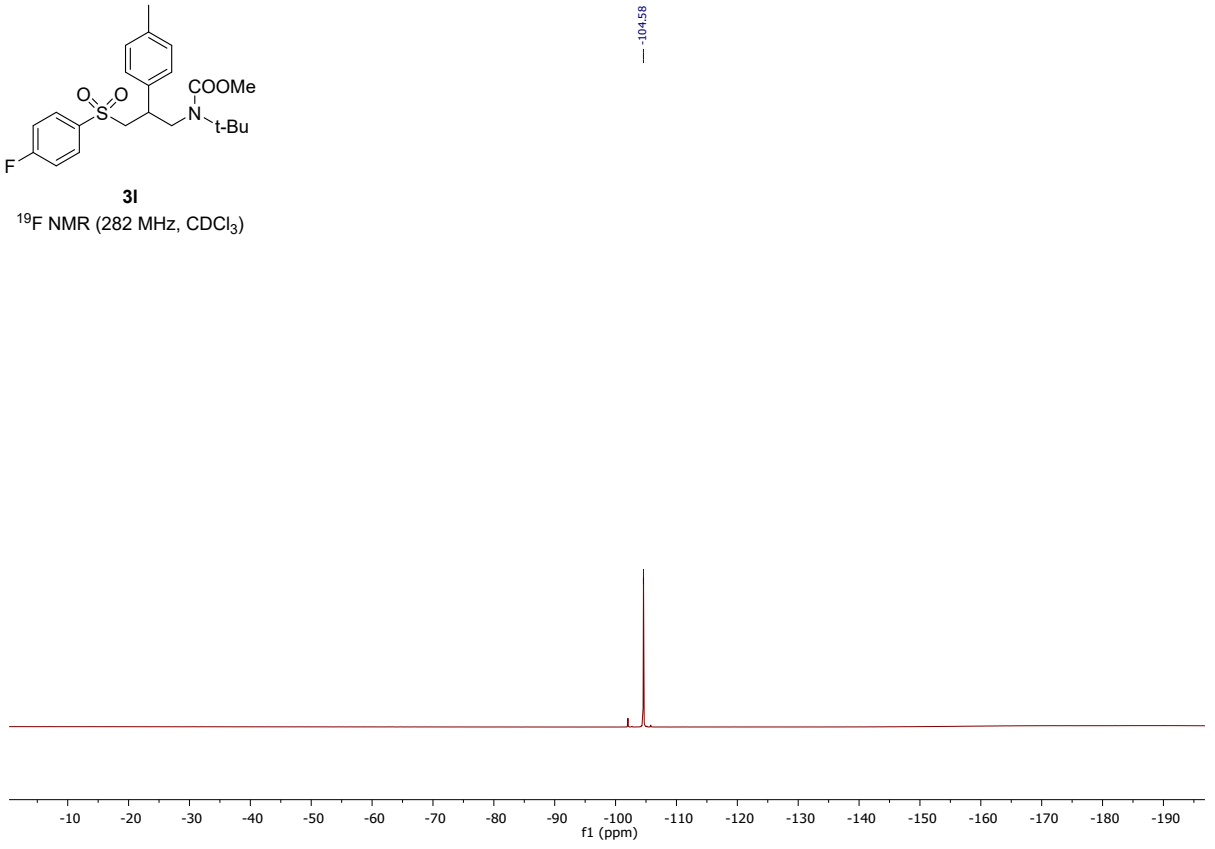
3l

^{1}H NMR (300 MHz, $CDCl_3$)



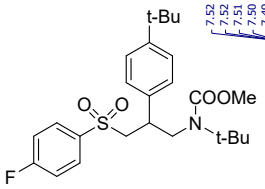
3l

^{13}C NMR (75 MHz, $CDCl_3$)



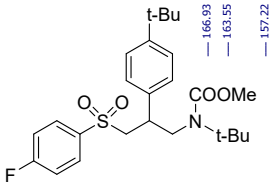
3l

^{19}F NMR (282 MHz, $CDCl_3$)



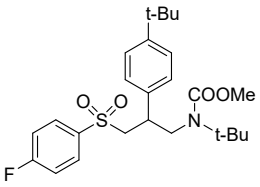
3m

^{1}H NMR (300 MHz, $CDCl_3$)



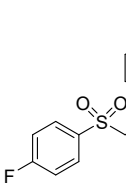
3m

^{13}C NMR (75 MHz, $CDCl_3$)



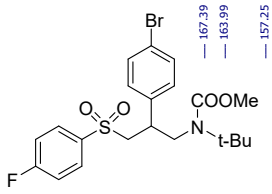
3m

^{19}F NMR (282 MHz, $CDCl_3$)



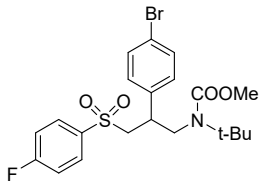
3n

^{1}H NMR (300 MHz, $CDCl_3$)



3n

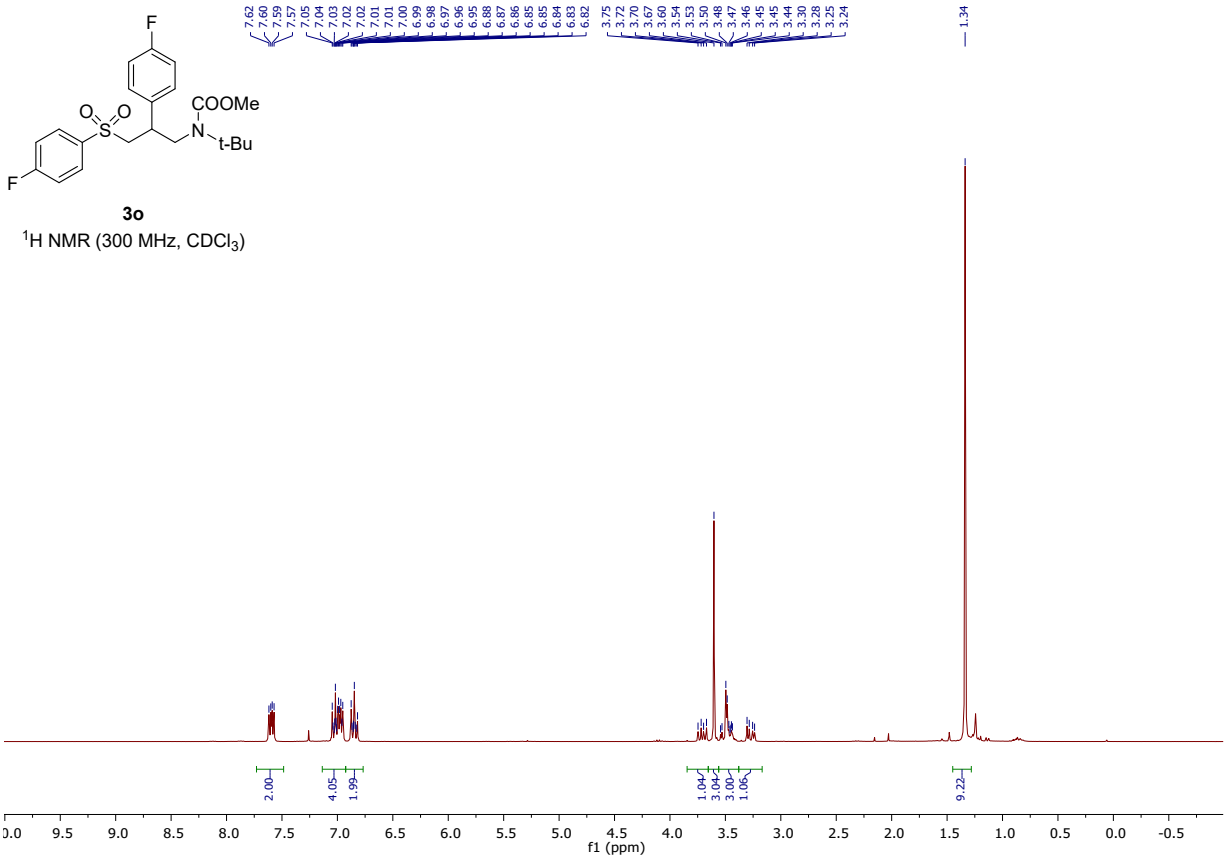
^{13}C NMR (75 MHz, $CDCl_3$)



3n

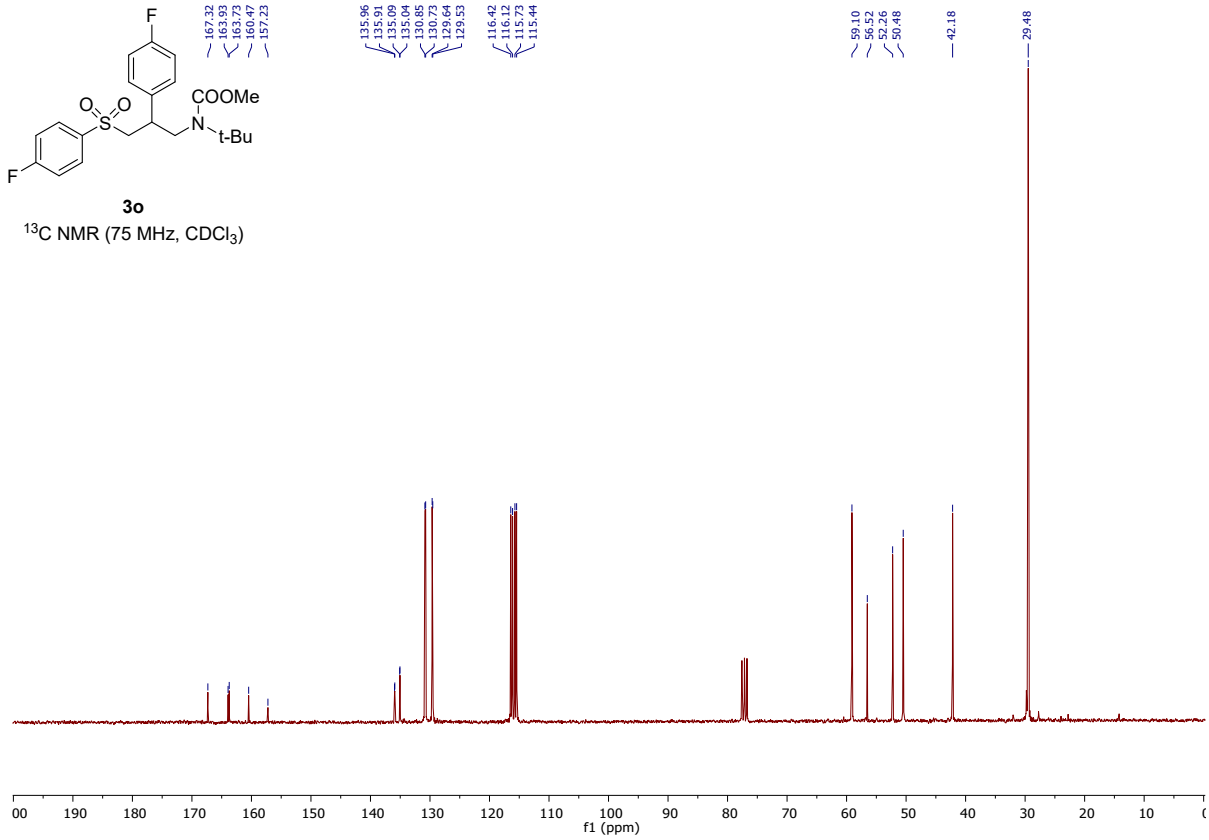
^{19}F NMR (282 MHz, $CDCl_3$)

3o



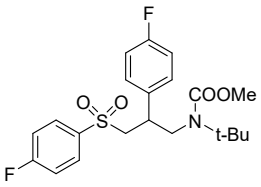
3o

^{1}H NMR (300 MHz, $CDCl_3$)



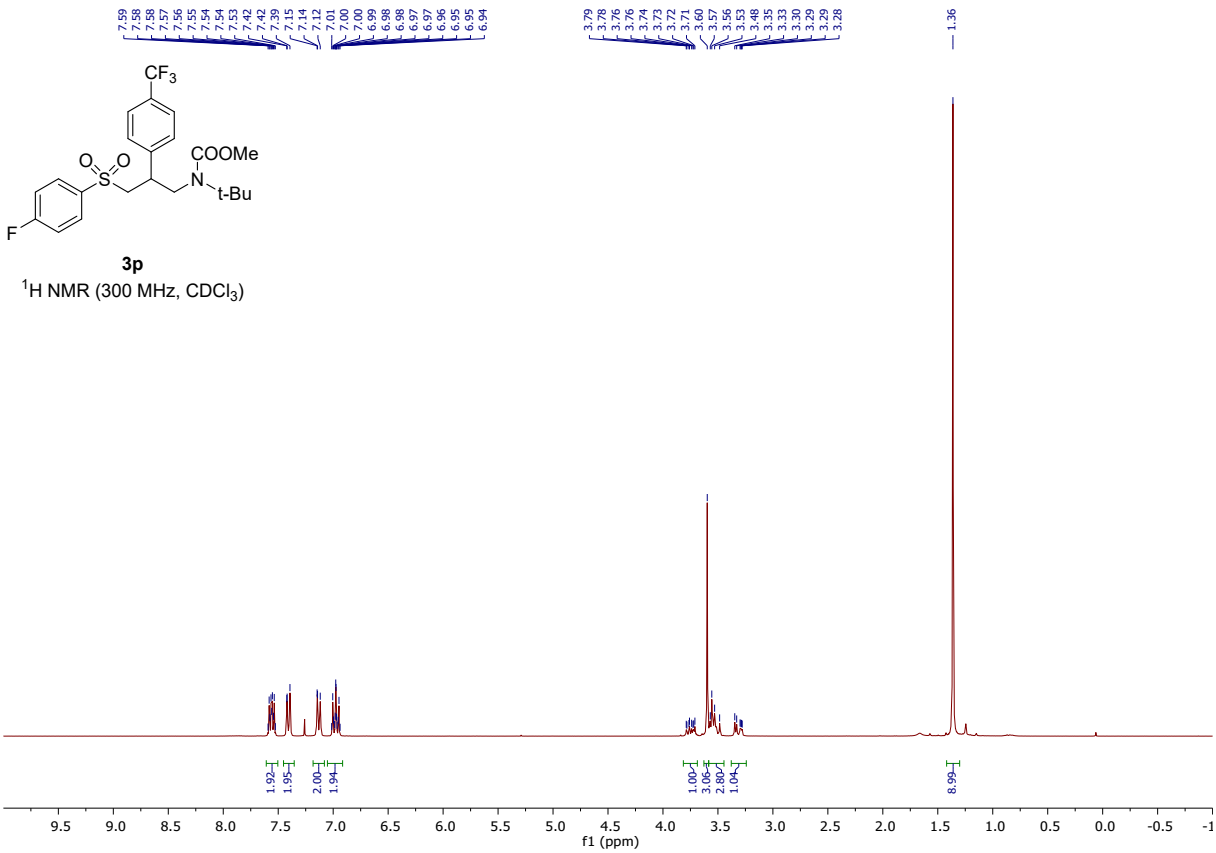
3o

^{13}C NMR (75 MHz, $CDCl_3$)



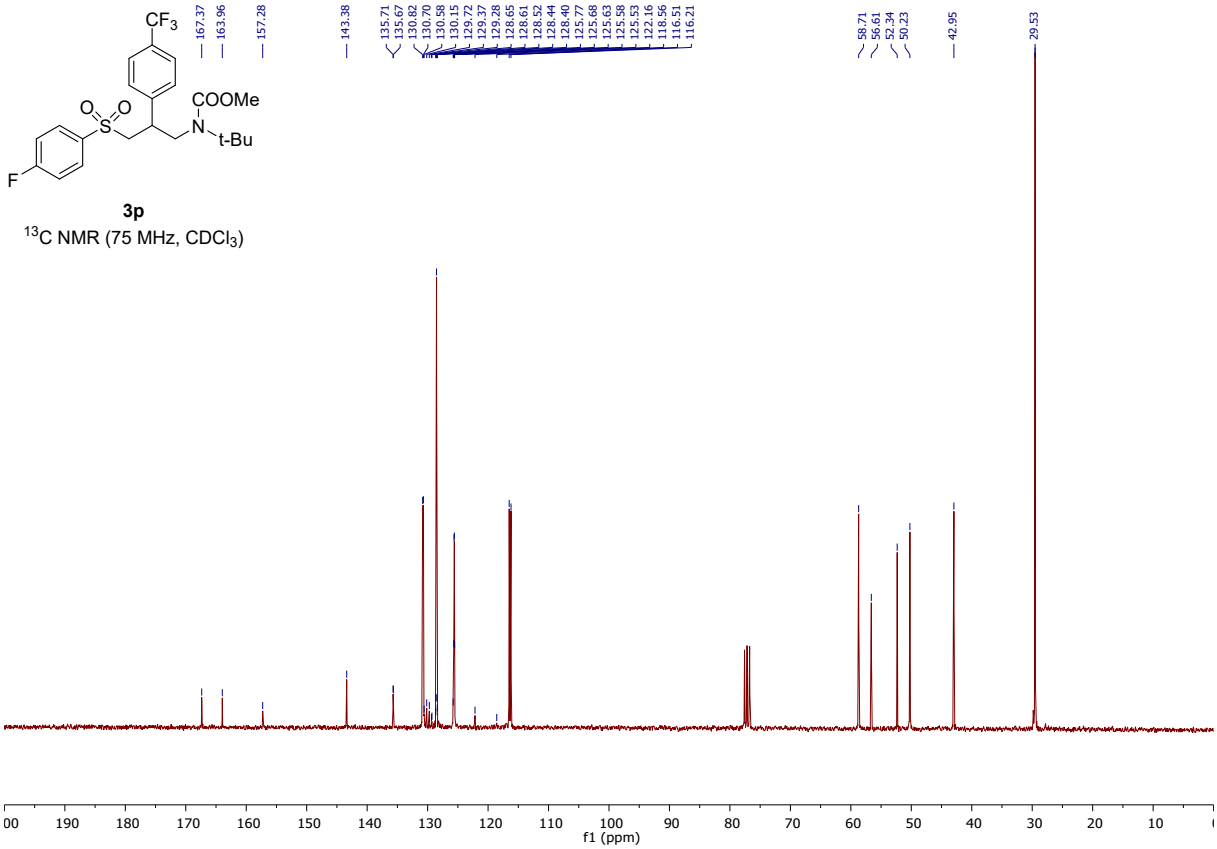
3o

^{19}F NMR (282 MHz, $CDCl_3$)



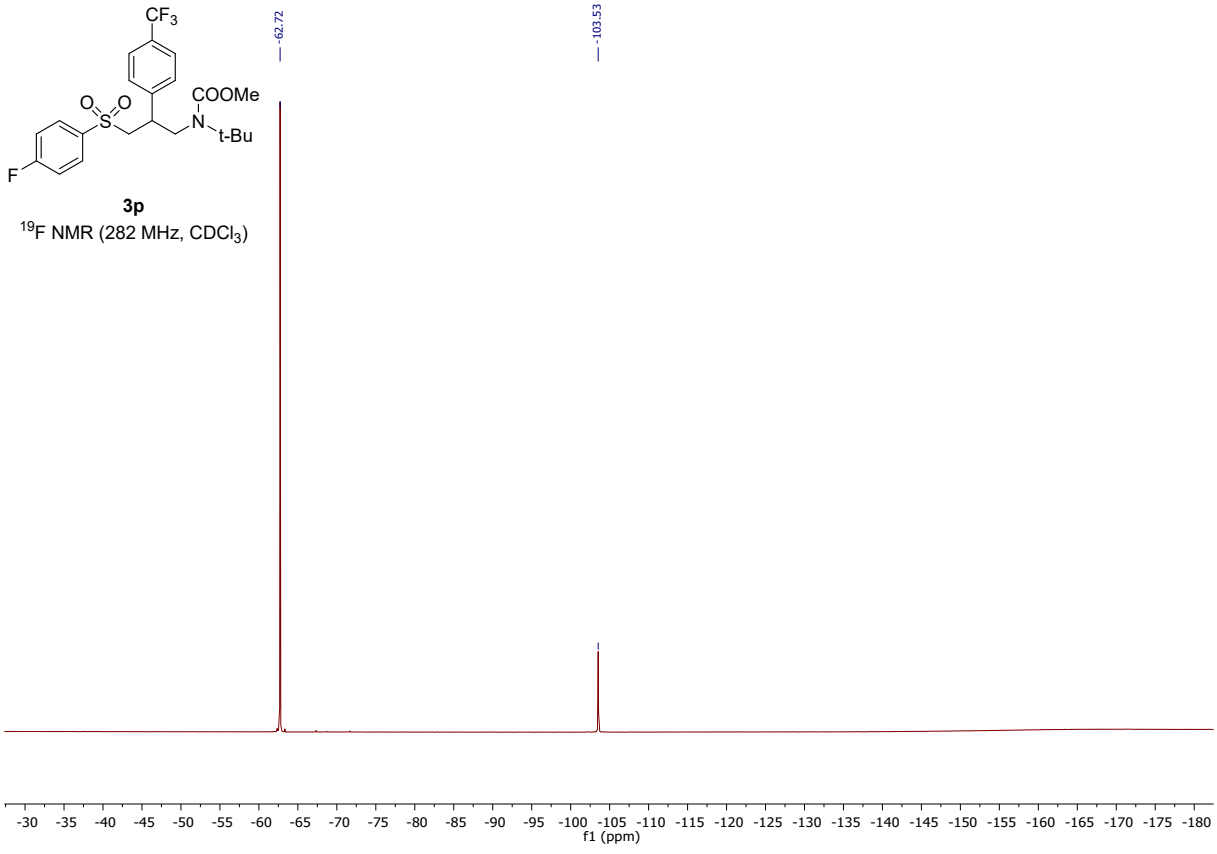
3p

^{1}H NMR (300 MHz, $CDCl_3$)



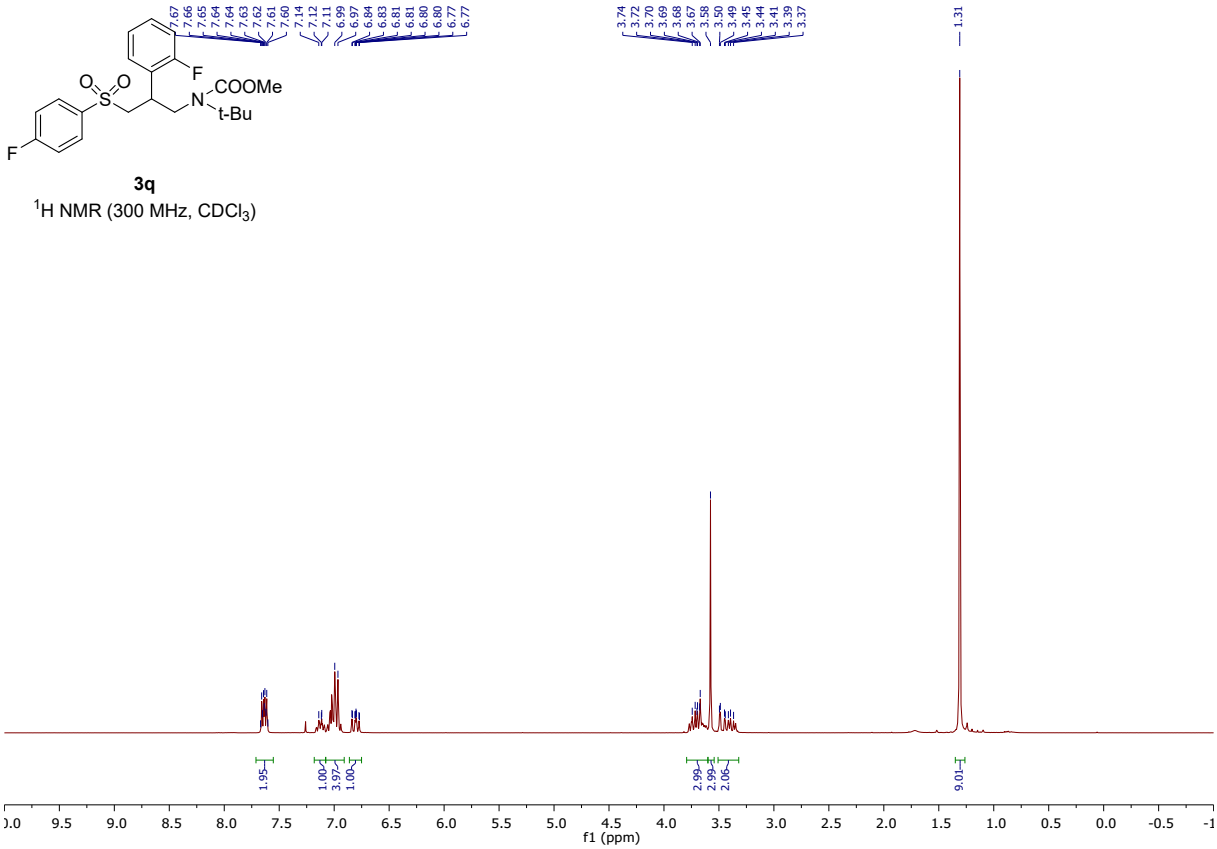
3p

^{13}C NMR (75 MHz, $CDCl_3$)



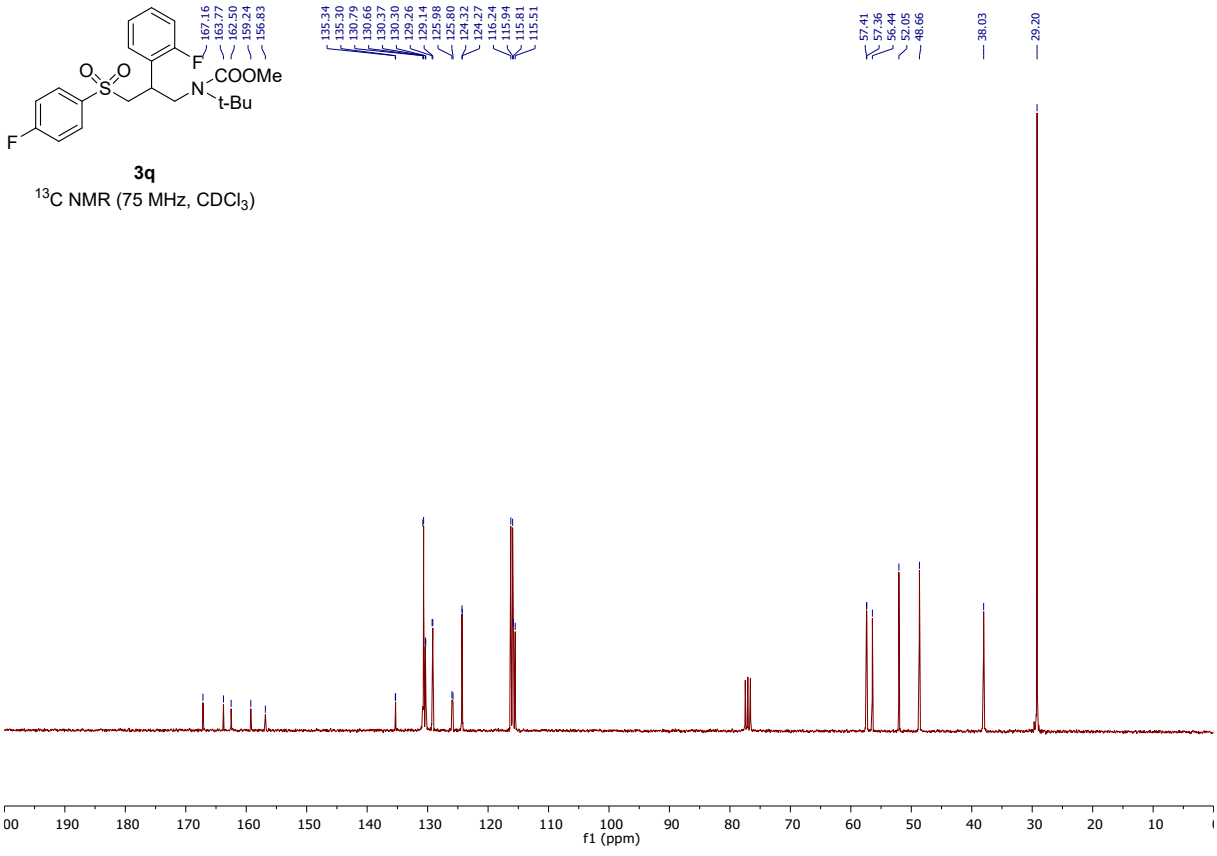
3p

^{19}F NMR (282 MHz, $CDCl_3$)



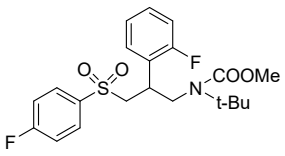
3q

^{1}H NMR (300 MHz, $CDCl_3$)



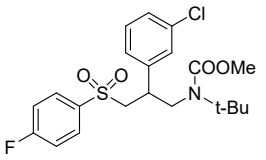
3q

^{13}C NMR (75 MHz, $CDCl_3$)



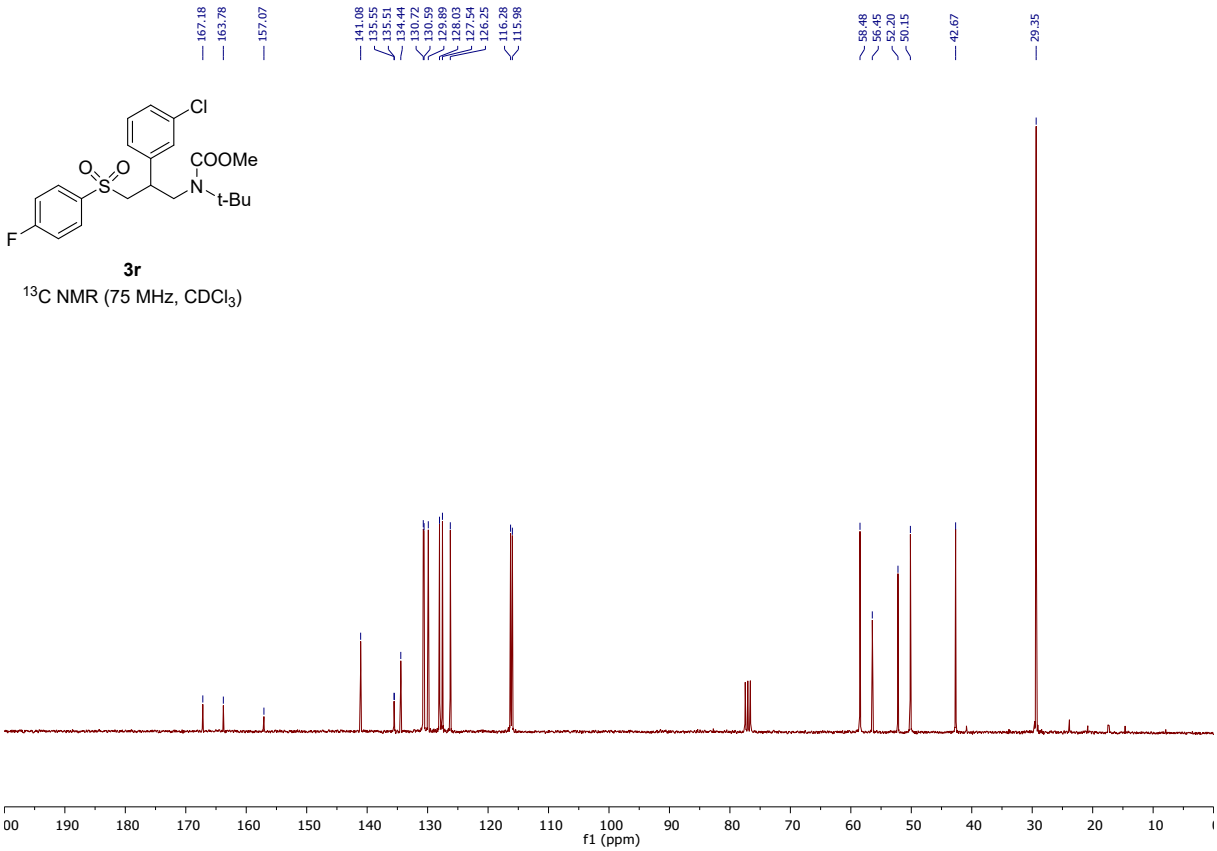
3q

^{19}F NMR (282 MHz, $CDCl_3$)



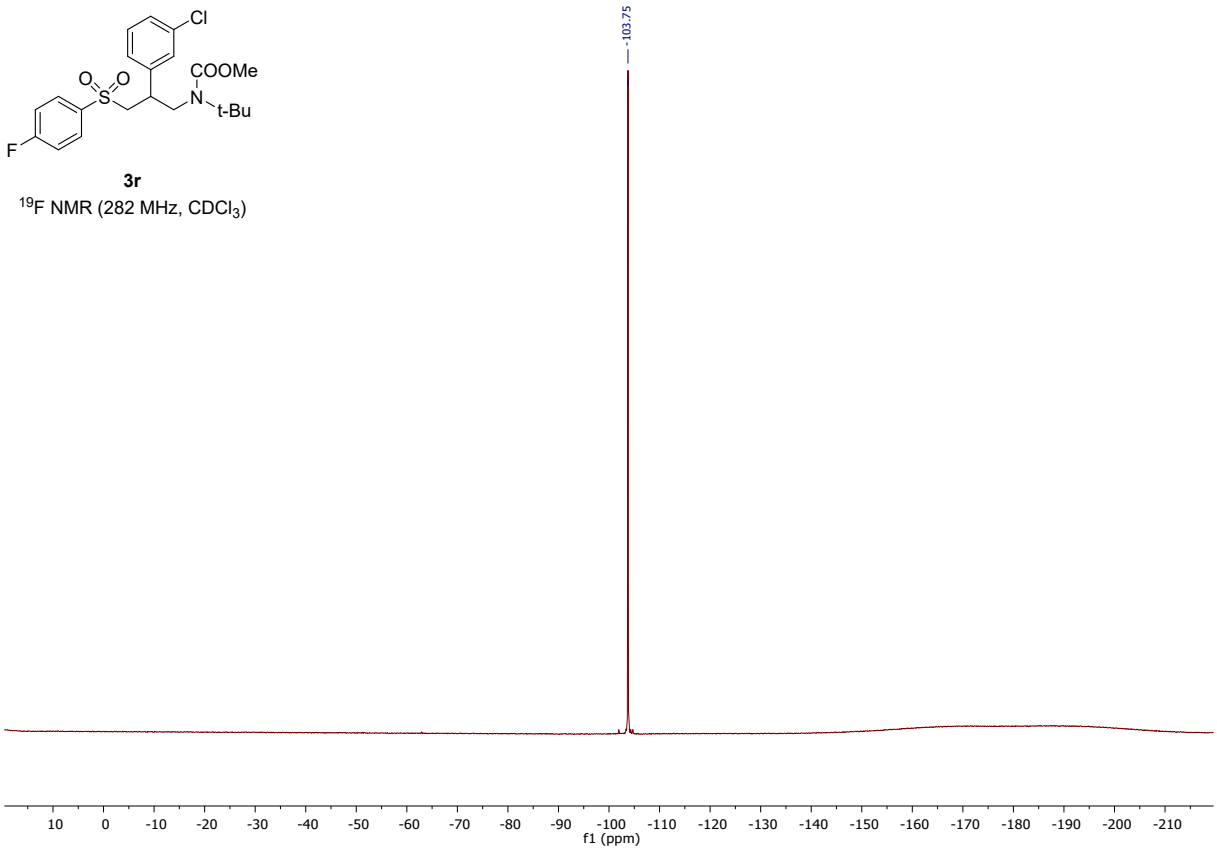
3r

^{1}H NMR (300 MHz, $CDCl_3$)



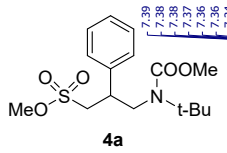
3r

^{13}C NMR (75 MHz, $CDCl_3$)



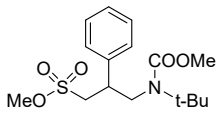
3r

^{19}F NMR (282 MHz, $CDCl_3$)



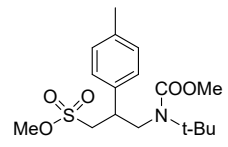
4a

^{1}H NMR (300 MHz, $CDCl_3$)



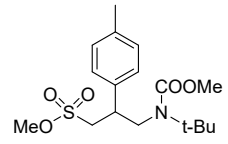
4a

^{13}C NMR (75 MHz, $CDCl_3$)



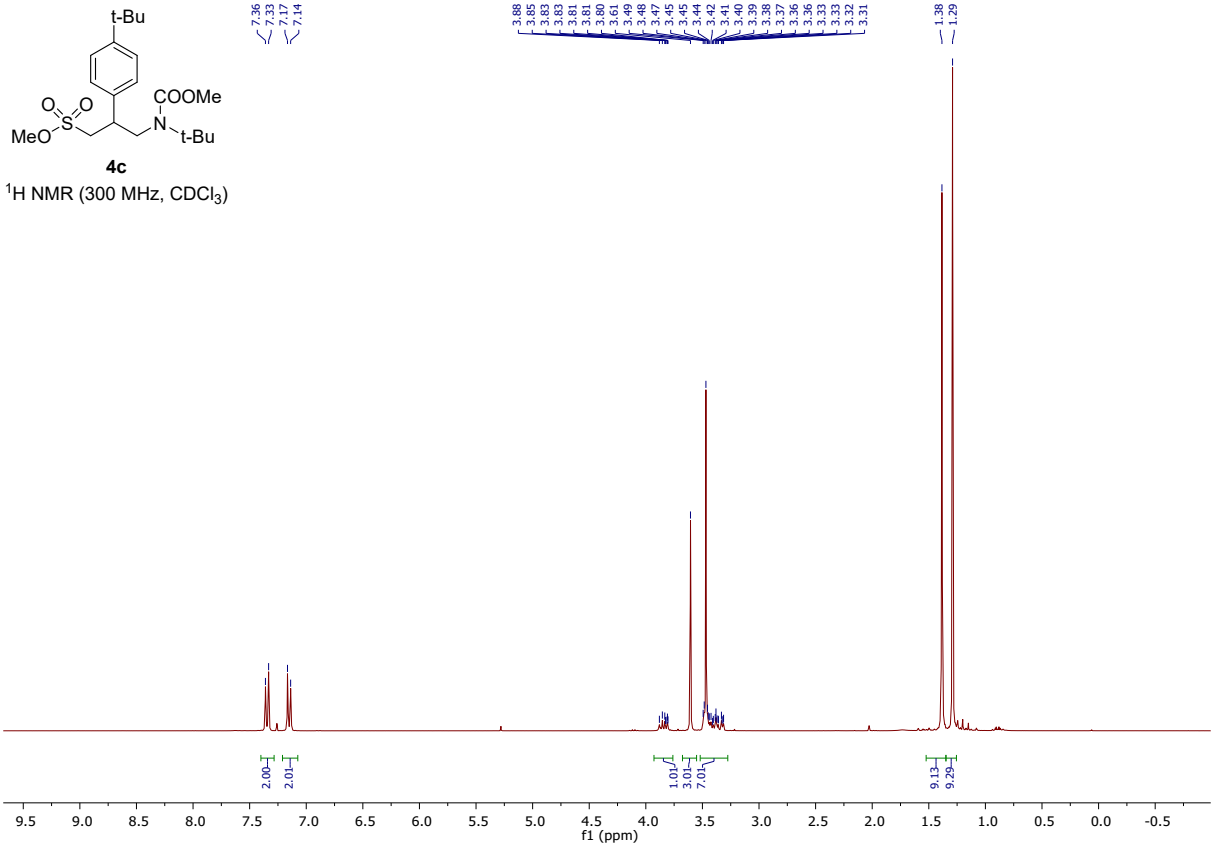
4b

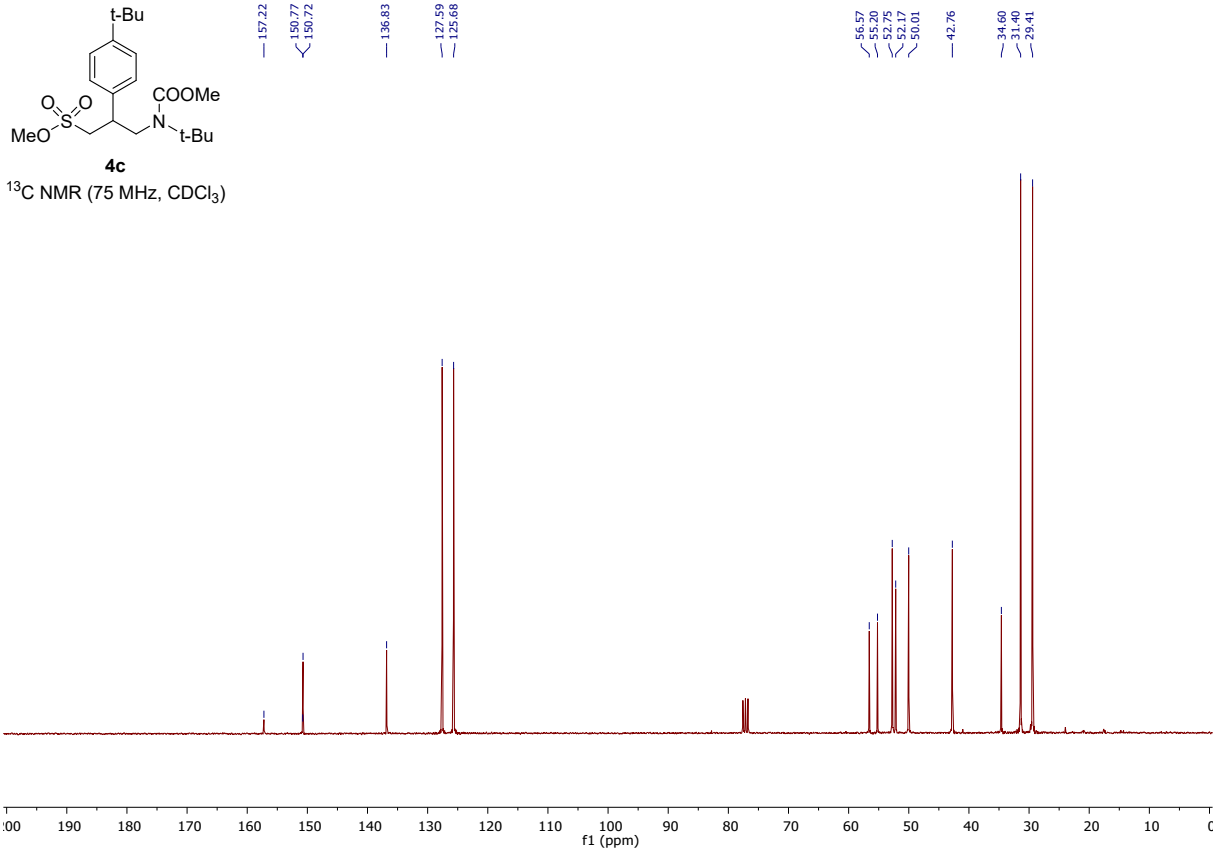
^{1}H NMR (300 MHz, $CDCl_3$)

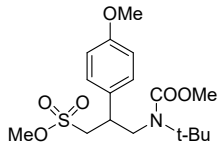


4b

^{1}H NMR (300 MHz, $CDCl_3$)

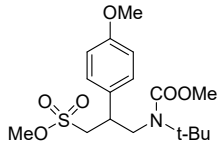

t-Bu
COOMe
MeO
t-Bu
4c
1H NMR (300 MHz, CDCl3)
7.36
7.33
7.17
7.14
3.88
3.85
3.83
3.83
3.81
3.81
3.80
3.61
3.49
3.48
3.47
3.45
3.45
3.44
3.42
3.41
3.40
3.39
3.38
3.37
3.36
3.36
3.33
3.33
3.32
3.31
1.38
1.29
2.00
2.01
1.01
3.01
7.01
9.13
9.29
f1 (ppm)


t-Bu
COOMe
MeO
t-Bu
4c
13C NMR (75 MHz, CDCl3)
157.22
150.77
150.72
136.83
127.59
125.68
56.57
55.20
52.75
52.17
50.01
42.76
34.60
31.40
29.41
f1 (ppm)



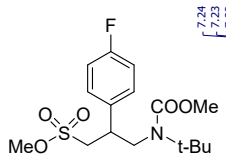
4d

^{1}H NMR (300 MHz, $CDCl_3$)



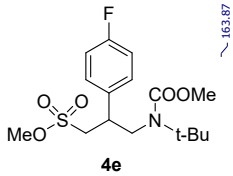
4d

^{1}H NMR (300 MHz, $CDCl_3$)



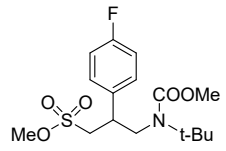
4e

^{1}H NMR (300 MHz, $CDCl_3$)



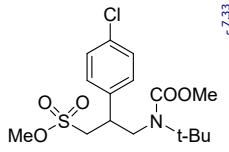
4e

^{13}C NMR (75 MHz, $CDCl_3$)



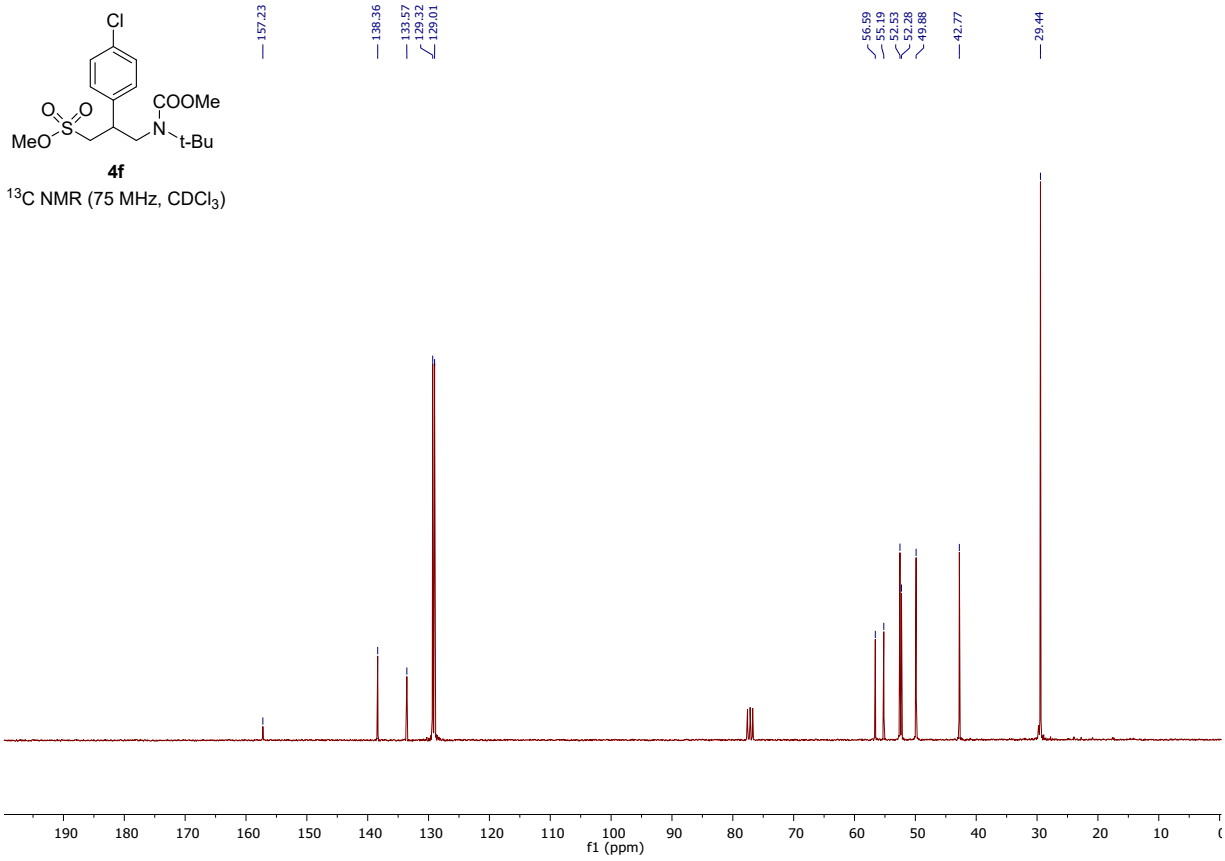
4e

^{19}F NMR (282 MHz, $CDCl_3$)



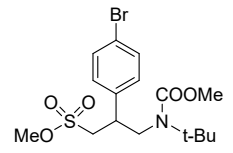
4f

^{1}H NMR (300 MHz, $CDCl_3$)



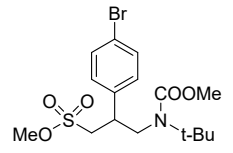
4f

^{13}C NMR (75 MHz, $CDCl_3$)



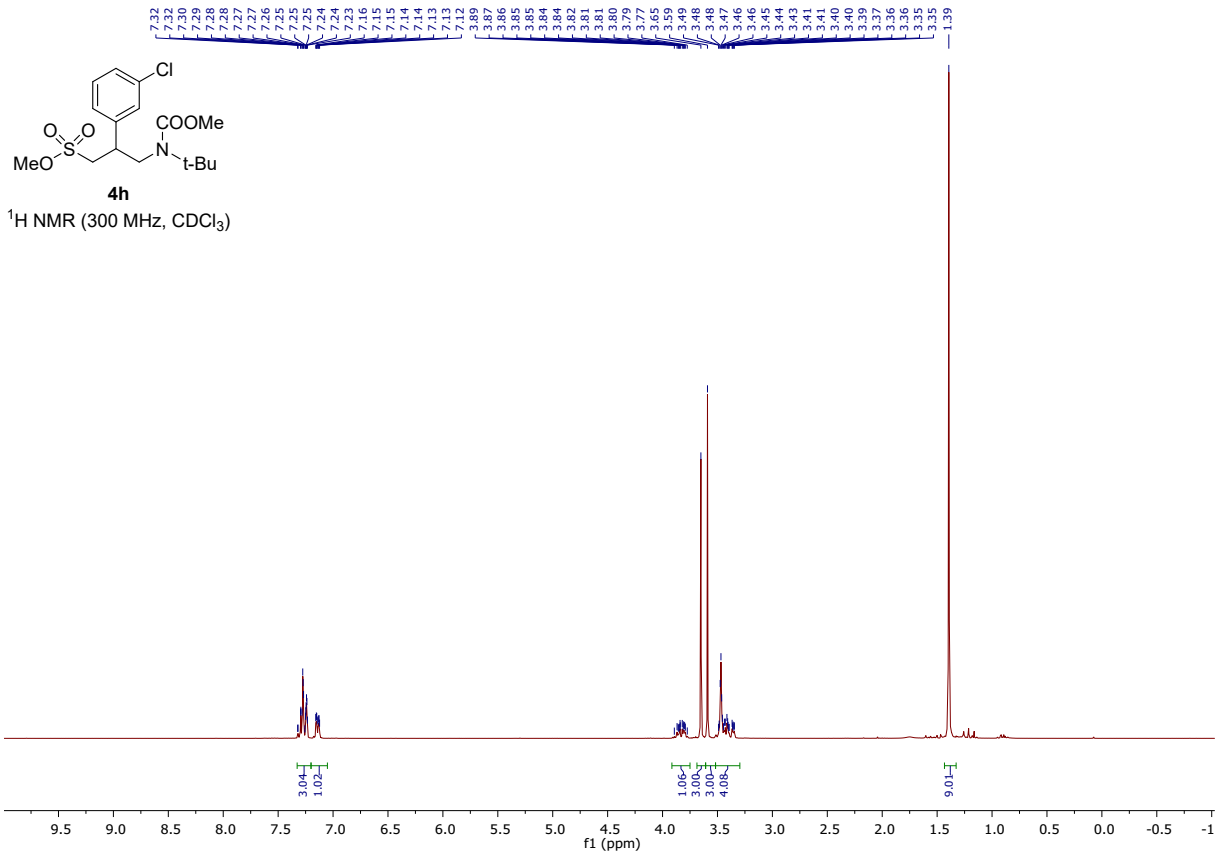
4g

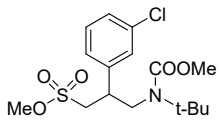
^{1}H NMR (300 MHz, $CDCl_3$)



4g

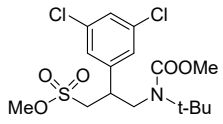
^{13}C NMR (75 MHz, $CDCl_3$)


Cl
COOMe
O
O
S
MeO
N
t-Bu
4h
1H NMR (300 MHz, CDCl3)
f1 (ppm)



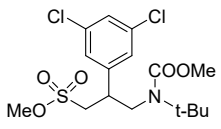
4h

^{13}C NMR (75 MHz, $CDCl_3$)



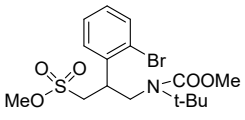
4i

^{1}H NMR (300 MHz, $CDCl_3$)



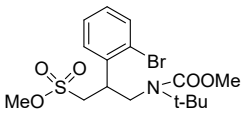
4i

^{13}C NMR (75 MHz, $CDCl_3$)



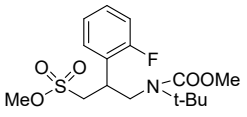
4j

^{1}H NMR (300 MHz, $CDCl_3$)



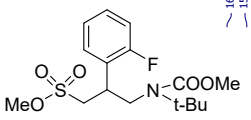
4j

^{13}C NMR (75 MHz, $CDCl_3$)



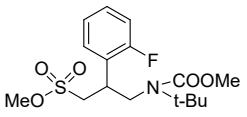
4k

^{1}H NMR (300 MHz, $CDCl_3$)



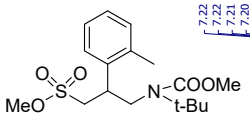
4k

^{13}C NMR (75 MHz, $CDCl_3$)



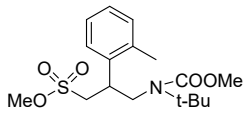
4k

^{19}F NMR (282 MHz, $CDCl_3$)



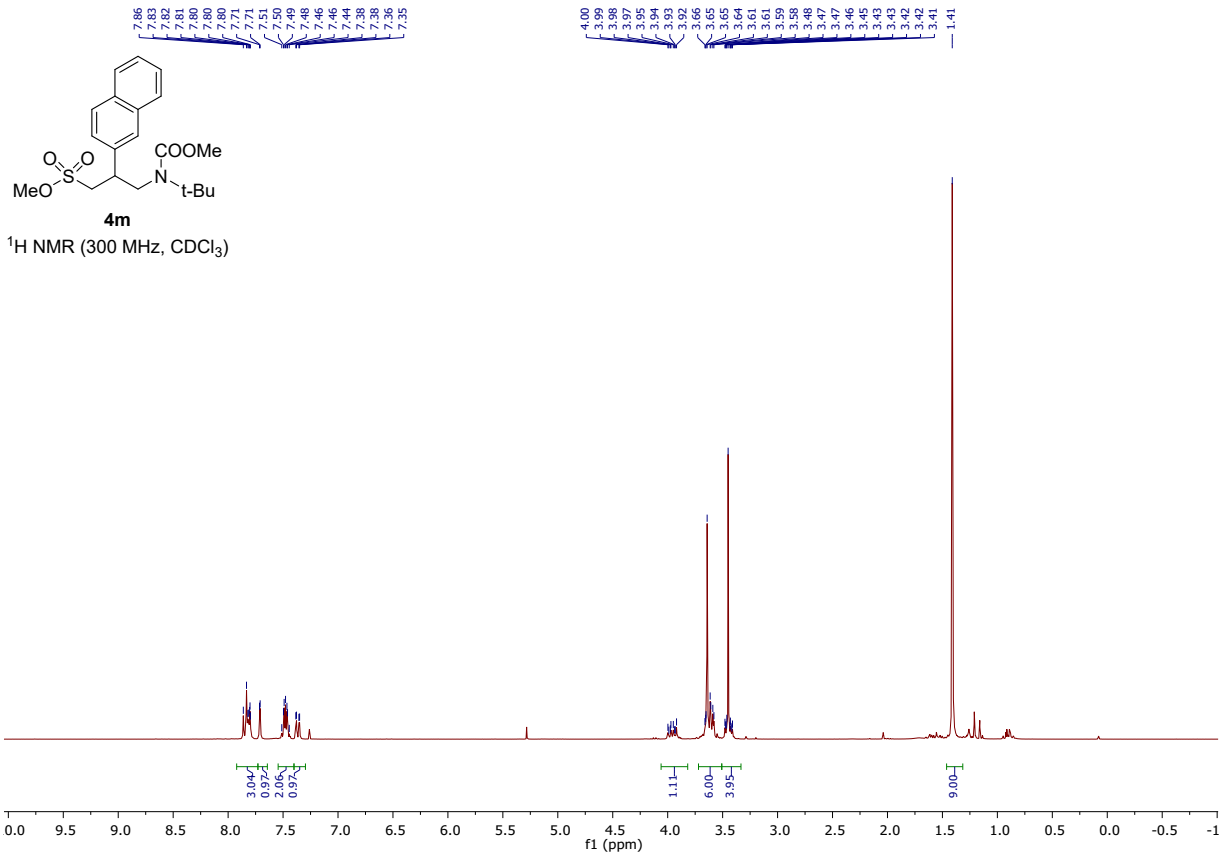
4l

^{1}H NMR (300 MHz, $CDCl_3$)



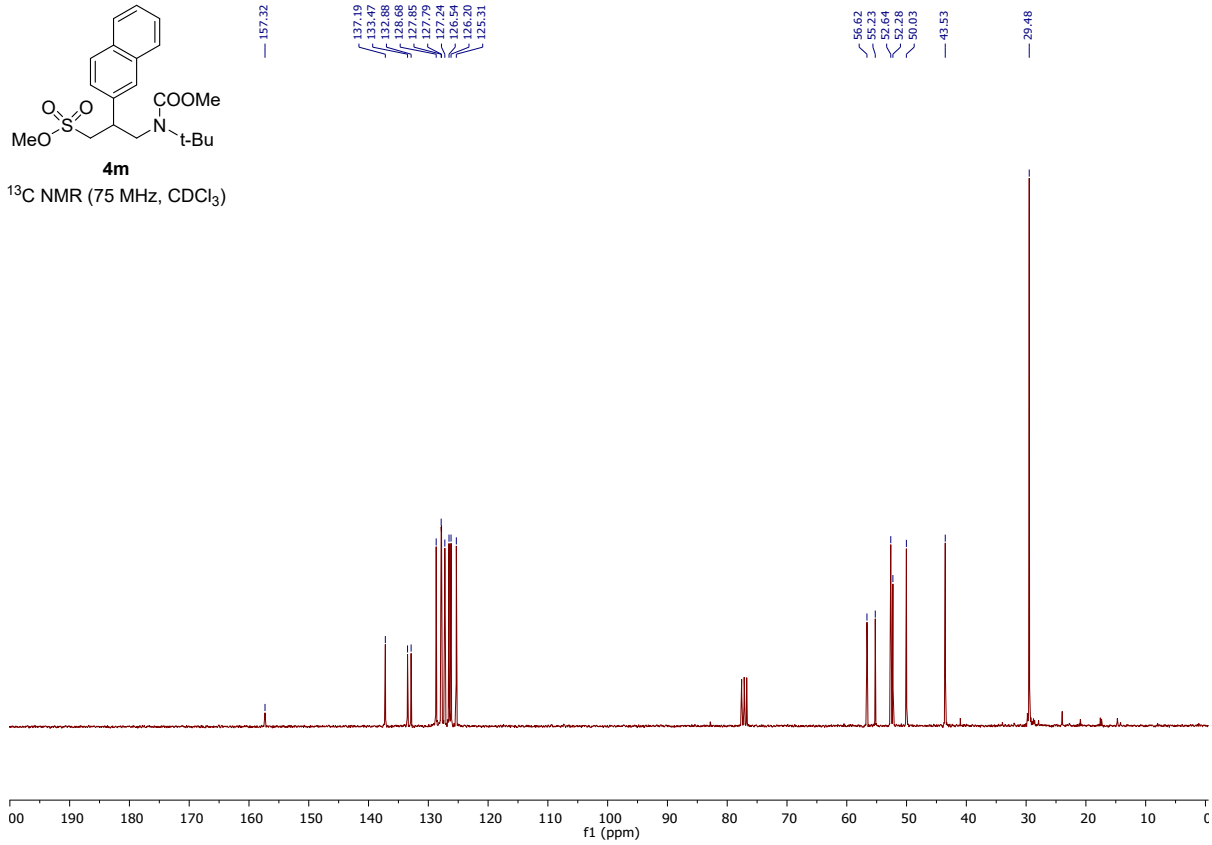
4l

^{13}C NMR (75 MHz, $CDCl_3$)



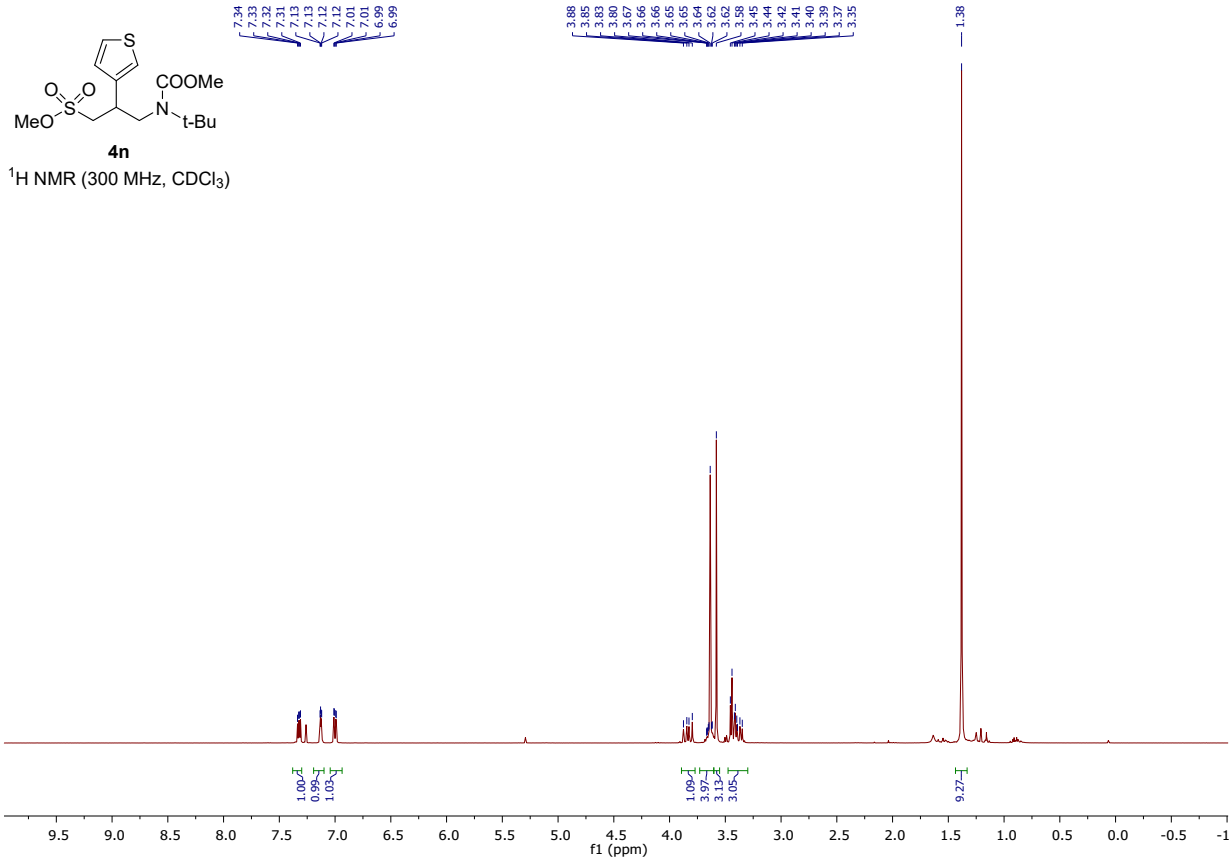
4m

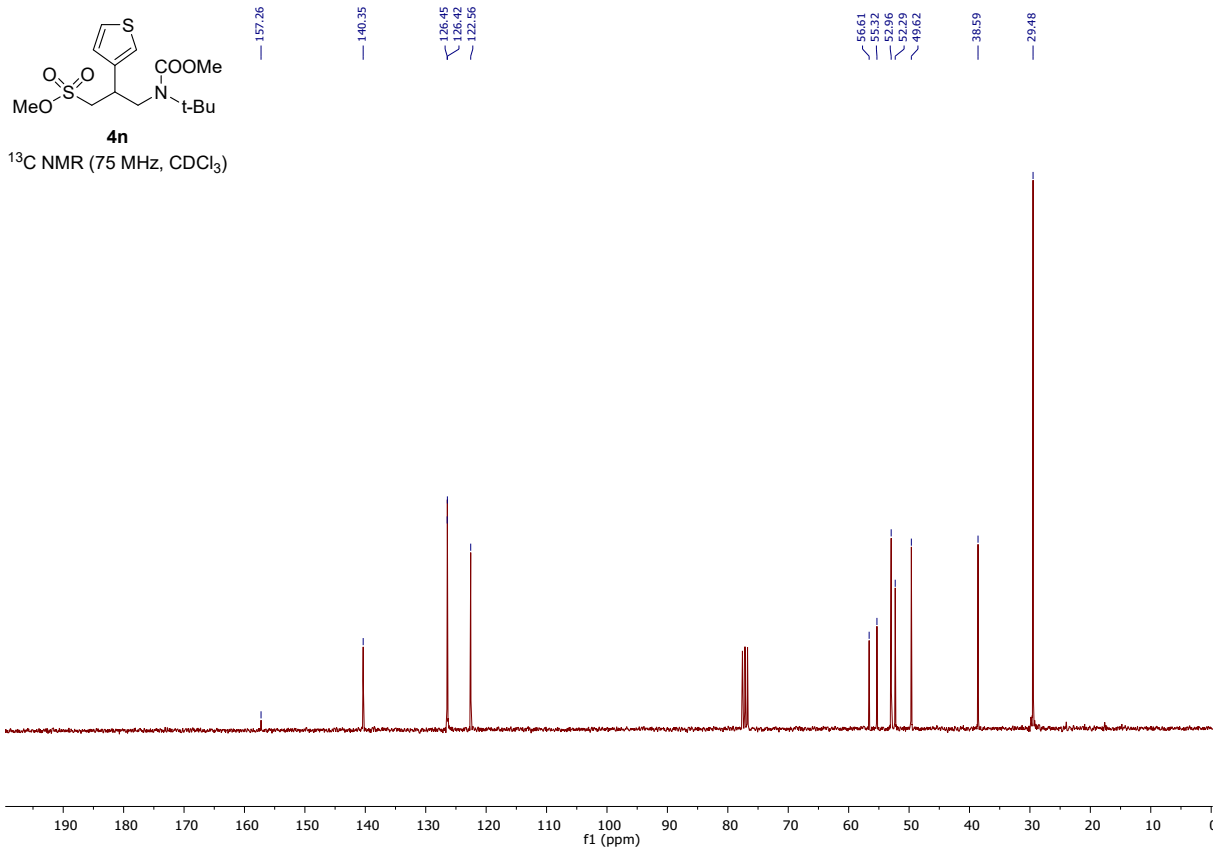
^{1}H NMR (300 MHz, $CDCl_3$)

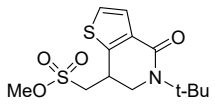


4m

^{13}C NMR (75 MHz, $CDCl_3$)

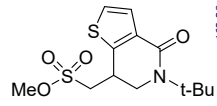

7.34
7.33
7.32
7.31
7.13
7.13
7.12
7.12
7.01
7.01
6.99
6.99
3.88
3.85
3.83
3.80
3.67
3.66
3.66
3.65
3.65
3.64
3.62
3.62
3.58
3.45
3.44
3.42
3.41
3.40
3.39
3.37
3.35
1.38
1.00
0.99
1.03
1.09
3.97
3.13
3.05
9.27
f1 (ppm)


157.26
140.35
126.45
126.42
122.56
56.61
55.32
52.96
52.29
49.62
38.59
29.48
f1 (ppm)



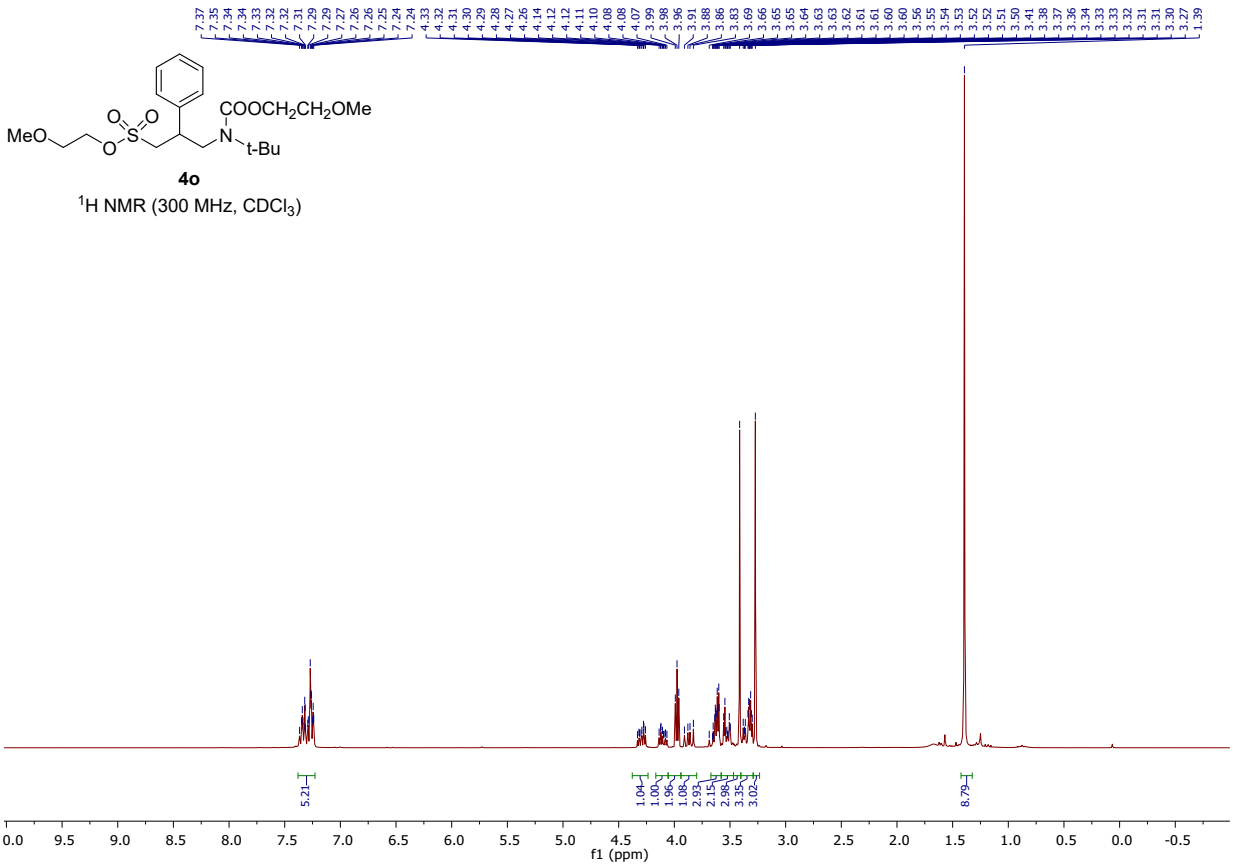
4n'

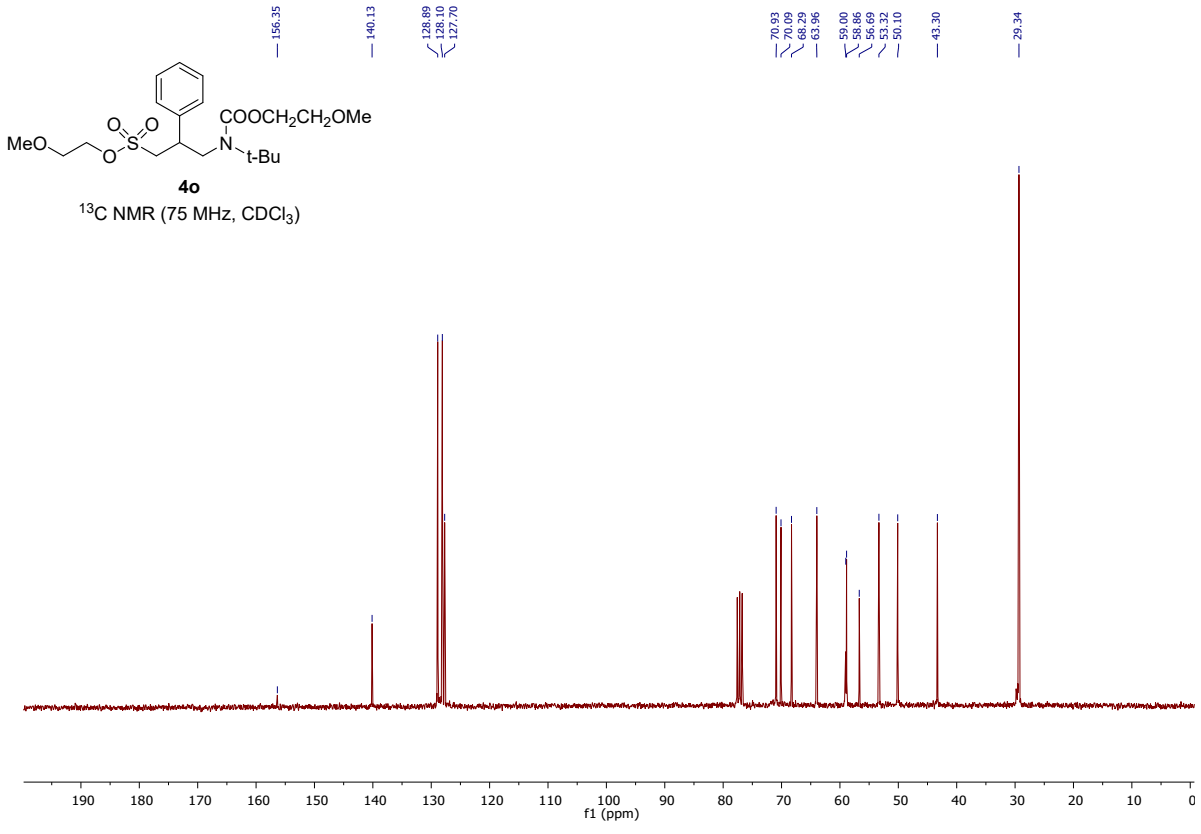
^{1}H NMR (500 MHz, $CDCl_3$)

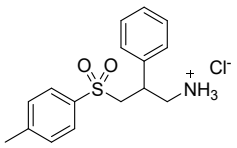


4n'

^{13}C NMR (126 MHz, $CDCl_3$)

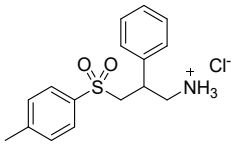

MeO
O
O
S
O
N
COOCH2CH2OMe
t-Bu
4o
1H NMR (300 MHz, CDCl3)
f1 (ppm)


MeO
O
O
S
O
N
COOCH2CH2OMe
t-Bu
4o
13C NMR (75 MHz, CDCl3)
f1 (ppm)



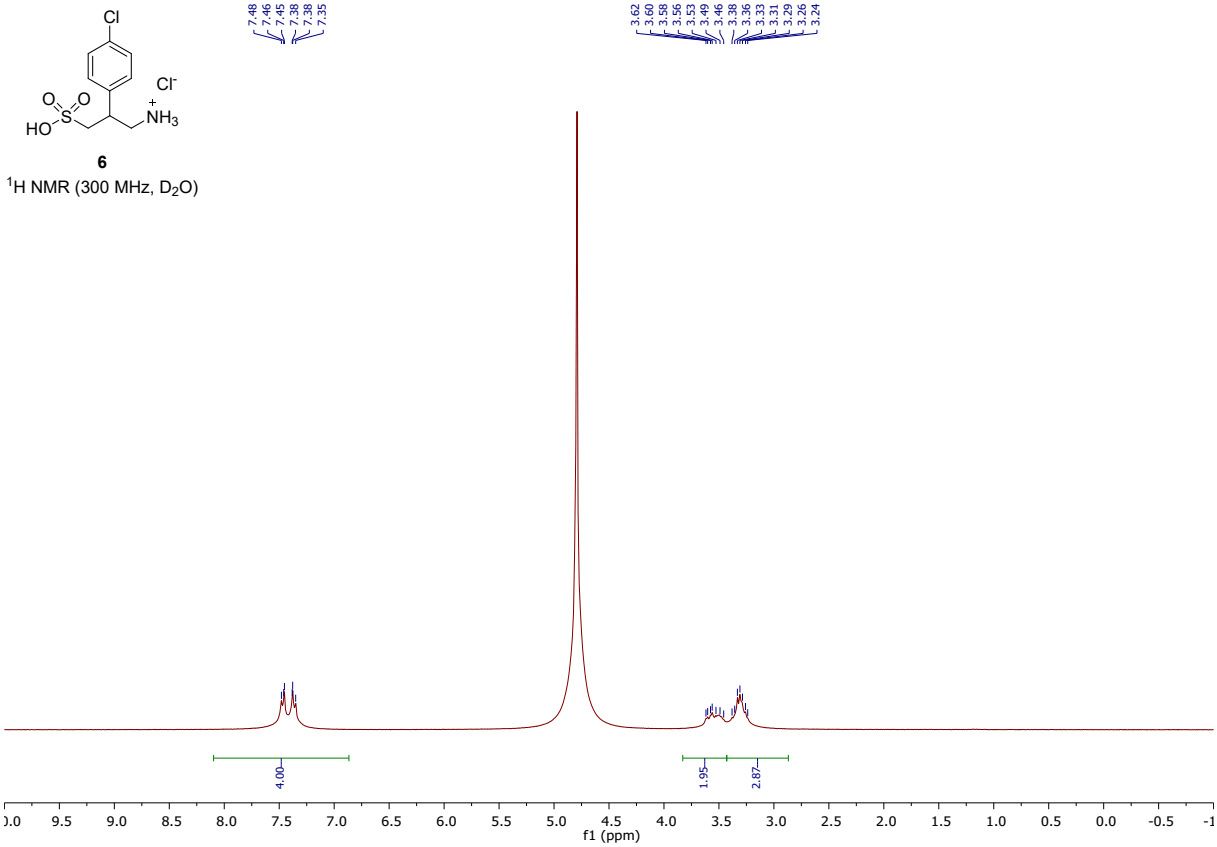
5

^{1}H NMR (300 MHz, MeOD)



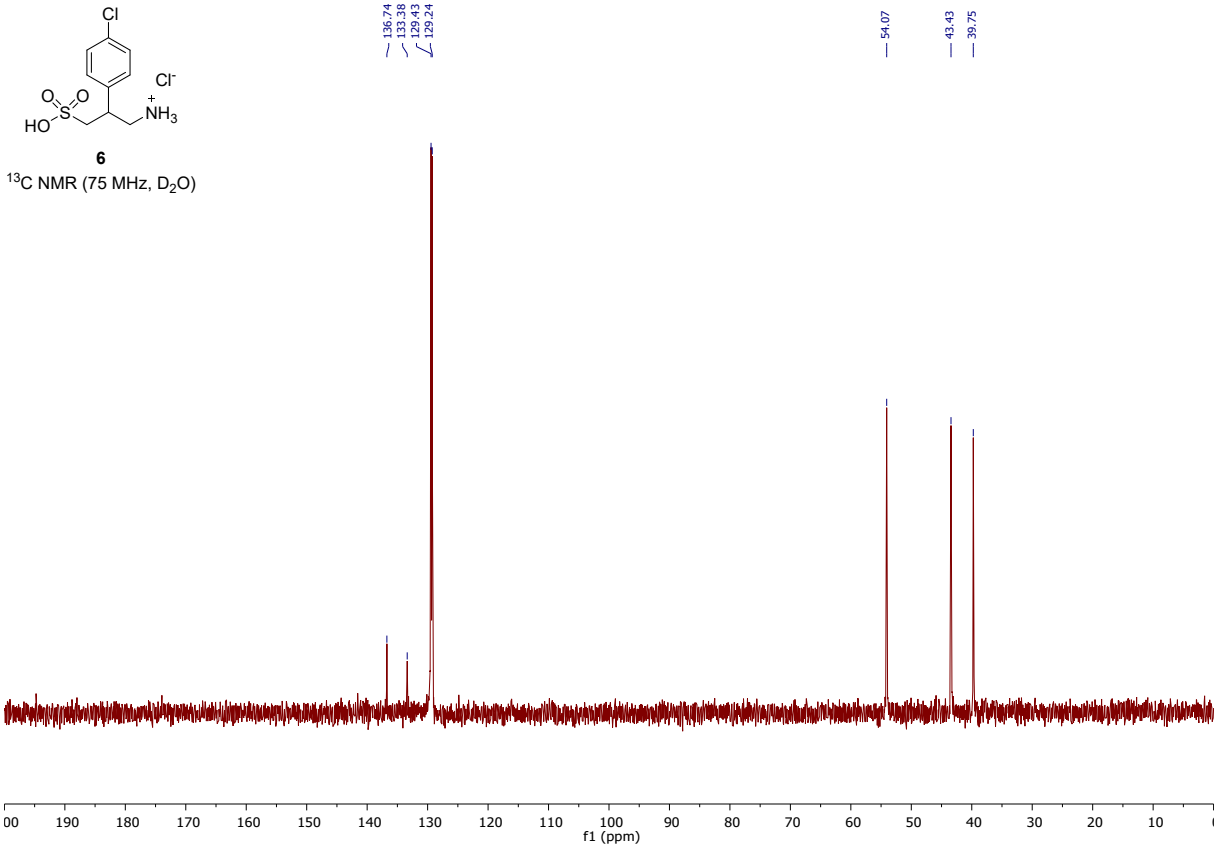
5

^{13}C NMR (75 MHz, MeOD)



6

^{1}H NMR (300 MHz, D_2O)



6

^{13}C NMR (75 MHz, D_2O)