Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

SUPPORTING IMFORMATION

Sulfonylation of Aryl Boronic Acids by Sulfonyl Fluorides in water under Visible Light Irradiation

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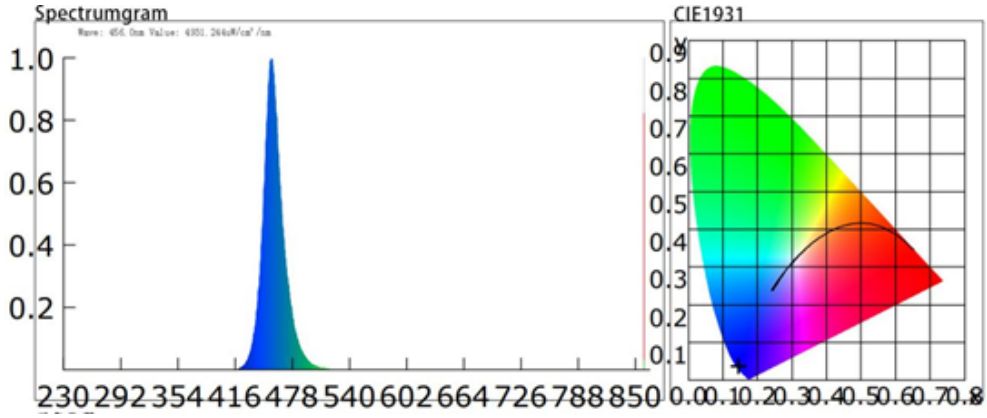
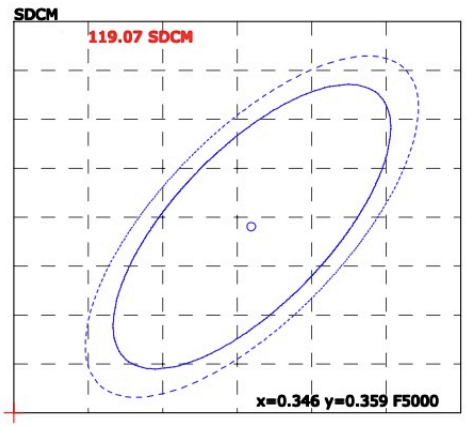
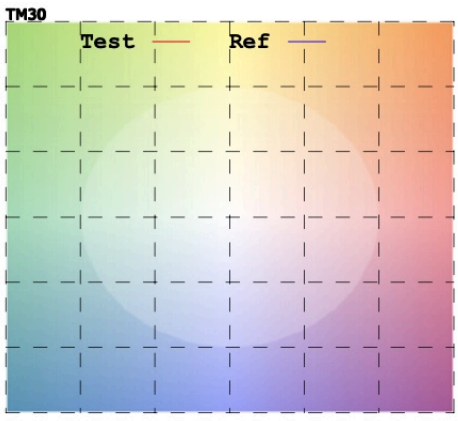
1. General Information

Sulfonyl chlorides, boronic acids and solvents were purchased from commercial sources (Adamas-Beta Co., Bidepharm Co. or Energy Chemical Co.) and used as received. $[Ir[dF(CF_3)ppy]_2(dtbbpy)]PF_6$, $[Ir[dF(CF_3)ppy]_2(bpy)]PF_6$, $[Ir(ppy)_2(dtbbpy)]PF_6$, $[Ir(ppy)_2(bpy)]PF_6$, $[Ru(bpy)_3]PF_6$ and 4-CzIPN were prepared according to the literature procedures.[1-3] Thin-layer chromatography (TLC) was performed on glass plates coated with silica gel 60 F254, 0.2 mm thickness. The plates were visualized using a 254 nm ultraviolet lamp or iodine cylinder. Flash chromatography was perform using silica gel, 300-400 mesh. Products were identified using NMR analysis and comparison with authentic samples. NMR spectra were recorded in $CDCl_3$ on Bruker spectrometers at 400 MHz. All chemical shifts are reported in parts per million (ppm) relative to residual $CDCl_3$ peak (7.26 and 77.0 ppm, ^1H NMR and ^{13}C NMR, respectively). Data for NMR are reported as follows: chemical shift (δ ppm), integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constant (Hz), and assignment. High resolution mass spectra were obtained on a Waters Synapt G2-Si (ESI) or Thermofisher Q Exactive GC (EI). UV-Visible absorption spectra were recorded on a YOKE double beam UV-vis spectrophotometer-T3202S. UV-Visible emission spectra were recorded on a TECHCOMP fluorescence spectrometer-FL970. GC data were recorded on a FULI INSTRUMENT GC9790Plus.

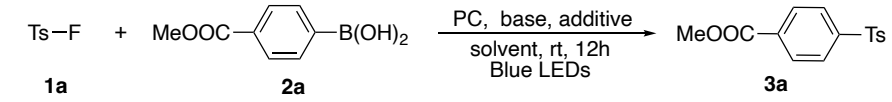
Blue LEDs (30 W, 455 nm) were purchased from Shenzhen FENGSHENG Electronic Technology Co., and the parameter information was shown below.







2. Optimization of Reaction Conditions



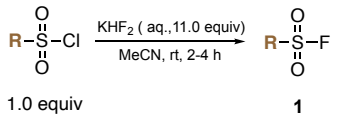
1a **2a** **3a**

Entry	PC	base	additive	solvent	[b] Yield (%)
1	$[Ir(ppy_2)(dtbbpy)]PF_6$	K_2CO_3	-	1,4-dioxane	trace
2	$[Ru(bpy)_3]PF_6$	K_2CO_3	-	1,4-dioxane	nd
3	Solvent Red	K_2CO_3	-	1,4-dioxane	trace
4	Rhodamine B	K_2CO_3	-	1,4-dioxane	11
5	Flurescin	K_2CO_3	-	1,4-dioxane	trace
6	Mes-Arc	K_2CO_3	-	1,4-dioxane	nd
7	4-CzIPN	K_2CO_3	-	1,4-dioxane	70
8	4-CzIPN	-	-	1,4-dioxane	nd
9	4-CzIPN	CsF	-	1,4-dioxane	38
10	4-CzIPN	K_2HPO_4	-	1,4-dioxane	16
11	4-CzIPN	K_3PO_4	-	1,4-dioxane	14
12	4-CzIPN	Cs_2CO_3	-	1,4-dioxane	68
13	4-CzIPN	NEt_3	-	1,4-dioxane	nd
14	4-CzIPN	DMAP	-	1,4-dioxane	nd
15	4-CzIPN	DABCO	-	1,4-dioxane	nd
16	4-CzIPN	Rb_2CO_3	-	1,4-dioxane	74
17	4-CzIPN	Rb_2CO_3	-	THF	46
18	4-CzIPN	Rb_2CO_3	-	DME	24
19	4-CzIPN	Rb_2CO_3	-	DMSO	13
20	4-CzIPN	Rb_2CO_3	-	DMA	15
21	4-CzIPN	Rb_2CO_3	-	MeCN	trace
22	4-CzIPN	Rb_2CO_3	-	H_2O	nd
23	4-CzIPN	Rb_2CO_3	Tween20	H_2O	80
24	4-CzIPN	Rb_2CO_3	Tween60	H_2O	74
25	4-CzIPN	Rb_2CO_3	Span40	H_2O	nd
26	4-CzIPN	Rb_2CO_3	TBAB	H_2O	23
27	4-CzIPN	Rb_2CO_3	BrijL23	H_2O	43
28	4-CzIPN	Rb_2CO_3	DDBS	H_2O	13
29	4-CzIPN	Rb_2CO_3	Betaine	H_2O	nd
30	-	Rb_2CO_3	Tween20	H_2O	nd
31	4-CzIPN	Rb_2CO_3	Tween20	H_2O	[c]nd
32	4-CzIPN	Rb_2CO_3	Tween20	H_2O	[d]nd

[a] Reaction conditions:: **1a** (0.3 mmol, 1.5 equiv), **2a** (0.2 mmol, 1.0 equiv), PC (1 mol%), base (0.4 mmol, 2.0 equiv), additive (3.2 equiv), solvent (1 mL), 30 W blue LED, N_2, 12 h, room temperature. nd = not detected. TBAB = Tetrabutylammonium bromide. BrijL23 = Polyoxyethylene lauryl ether. DDBS = Sodium dodecylbenzenesulphonate. [b] GC yield was calculated with Dodecane as internal standard. [c] Under air. [d] In dark.

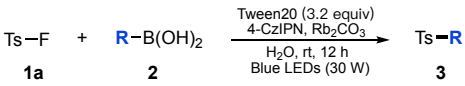
3. General Procedures

3.1 Preparation of Sulfonyl Fluorides (Cl-F exchanging)



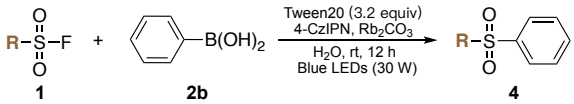
Sulfonyl chloride (5 mmol, 1.0 equiv) was dissolved in MeCN (10 mL), and aq. KHF_2 (10 mL, 11.0 equiv) was added. The suspension was stirred for 2-4 hours at room temperature. Then the reaction mixture was diluted with H_2O (5 mL) and extracted with EtOAc (3 x 5 mL). The combined organic layer was dried over Na_2SO_4, and filtrate was concentrated in vacuo to directly give the desired sulfonyl fluoride.

3.2. General Procedure A: Substrate Scope of Boronic Acids



An oven-dried Schlenk tube equipped with a stir bar was charged with sulfonyl fluoride **1a** (0.3 mmol, 1.5 equiv), boronic acid **2** (0.2 mmol, 1.0 equiv), Rb_2CO_3 (0.4 mmol, 2.0 equiv) and 4-CzIPN (1 mol%). Then the tube was sealed with a cap, evacuated and backfilled with nitrogen for 3 times. Subsequently, Tween20 (0.2 mL, 3.2 equiv) and H_2O (1 mL) was added. The tube was placed in a water bath with irradiation by 30 W blue LEDs (455 nm, distance: 1 cm, the reaction temperature reached room temperature) for 12 hours. Then the mixture was washed with sat. aq. NH_4Cl (1 x 2 mL), diluted with EtOAc (1 x 4 mL), and concentrated. The crude product was purified by flash chromatography (EtOAc/PE: 1/4, unless otherwise noted) to give analytically pure product **3**.

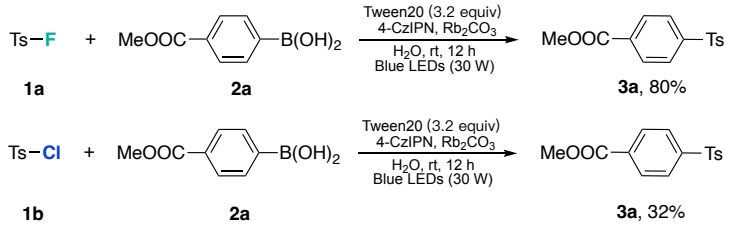
3.3. General Procedure B: Substrate Scope of Sulfonyl Fluorides



An oven-dried Schlenk tube equipped with a stir bar was charged with sulfonyl fluoride **1** (0.3 mmol, 1.5 equiv), boronic acid **2b** (0.2 mmol, 1.0 equiv), Rb_2CO_3 (0.4 mmol, 2.0 equiv) and 4-CzIPN (1 mol%). Then the tube was sealed with a cap, evacuated and backfilled with nitrogen for 3 times. Subsequently, Tween20 (0.2 mL, 3.2 equiv) and H_2O (1 mL) was added. The tube was placed in a water bath with irradiation by 30 W blue LEDs (455 nm, distance: 1 cm, the reaction temperature reached room temperature) for 12 hours. Then the mixture was washed with sat. aq. NH_4Cl (1 x 2 mL), diluted with EtOAc (1 x 4 mL), and concentrated. The crude product was purified by flash chromatography (EtOAc/PE: 1/4, unless otherwise noted) to give analytically pure product **4**.

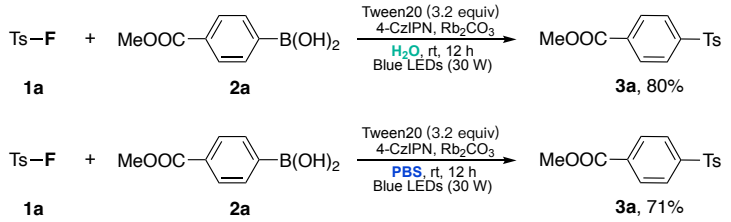
4. Derivative Experiments

4.1. Control Experiments: Sulfonyl Fluoride vs. Sulfonyl Chloride



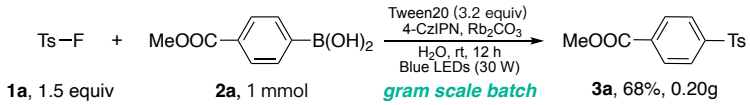
An oven-dried Schlenk tube equipped with a stir bar was charged with sulfonyl fluoride **1a** or **1b** (0.3 mmol, 1.5 equiv), boronic acid **2a** (0.2 mmol, 1.0 equiv), Rb_2CO_3 (0.4 mmol, 2.0 equiv) and 4-CzIPN (1 mol%). Then the tube was sealed with a cap, evacuated and backfilled with nitrogen for 3 times. Subsequently, Tween20 (0.2 mL, 3.2 equiv) and H_2O (1 mL) was added. The tube was placed in a water bath with irradiation by 30 W blue LEDs (455 nm, distance: 1 cm, the reaction temperature reached room temperature) for 12 hours. Then the mixture was washed with sat. aq. NH_4Cl (1 x 2 mL), diluted with EtOAc (1 x 4 mL), and concentrated. The crude product was purified by flash chromatography (EtOAc/PE: 1/4) to give analytically pure product **3a**.

4.2. Control Experiments: Water vs. PBS



An oven-dried Schlenk tube equipped with a stir bar was charged with sulfonyl fluoride **1a** (0.3 mmol, 1.5 equiv), boronic acid **2a** (0.2 mmol, 1.0 equiv), Rb_2CO_3 (0.4 mmol, 2.0 equiv) and 4-CzIPN (1 mol%). Then the tube was sealed with a cap, evacuated and backfilled with nitrogen for 3 times. Subsequently, Tween20 (0.2 mL, 3.2 equiv) and H_2O or PBS (1 mL) was added. The tube was placed in a water bath with irradiation by 30 W blue LEDs (455 nm, distance: 1 cm, the reaction temperature reached room temperature) for 12 hours. Then the mixture was washed with sat. aq. NH_4Cl (1 x 2 mL), diluted with EtOAc (1 x 4 mL), and concentrated. The crude product was purified by flash chromatography (EtOAc/PE: 1/4) to give analytically pure product **3a**.

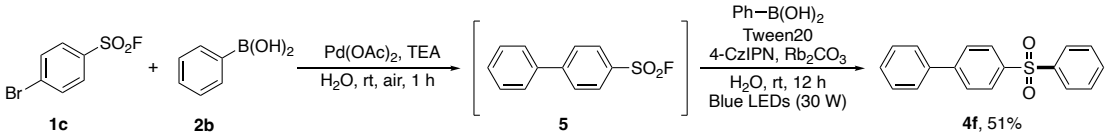
4.3. Scale-up Experiments



A scale-up experiment for synthesis of **3a** was carried out by following the **General Procedure A**. The whole process was carried out in an oven-dried 50 mL round bottom flask. Finally, **3a** was purified to afford as colorless solid, 0.20 g, 68% yield.

4.4. Tandem One-pot Reactions of Sulfonyl Fluorides

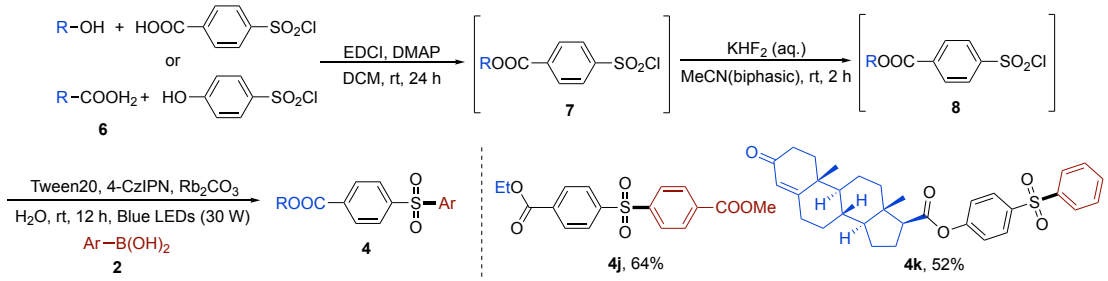
4.4.1. Cross-coupling



1c **2b** **5** **4f**, 51%

An oven-dried Schlenk tube equipped with a stir bar was charged with 4-bromobenzenesulfonyl fluoride **1c** (0.3 mmol, 1.5 equiv), phenylboronic acid **2b** (0.45 mmol, 2.25 equiv), TEA (0.9 mmol, 4.5 equiv), palladium (II) acetate (1 mol%) and H_2O (1 mL). The suspension was stirred under air at room temperature for 1 hour. After that, the suspension was washed with aq. HBr (1 mL, 1 M), and extracted with EtOAc (2 x 1 mL). The organic layer was placed in a new oven-dried Schlenk tube, and concentrated. Then phenylboronic acid (0.2 mmol, 1.0 equiv), Rb_2CO_3 (0.4 mmol, 2.0 equiv) and 4-CzIPN (1 mol%) was added in H_2O (1 mL). Then the tube was sealed with a cap, evacuated and backfilled with nitrogen for 3 times. Subsequently, Tween20 (0.2 mL, 3.2 equiv) was added. The tube was placed in a water bath with irradiation by 30 W blue LEDs (455 nm, distance: 1 cm, the reaction temperature reached room temperature) for 12 hours. Then the mixture was washed with sat. aq. NH_4Cl (1 x 2 mL) and diluted with EtOAc (1 x 4 mL). The crude product was purified by flash chromatography (EtOAc/PE: 1/4) to give analytically pure product **4f**.

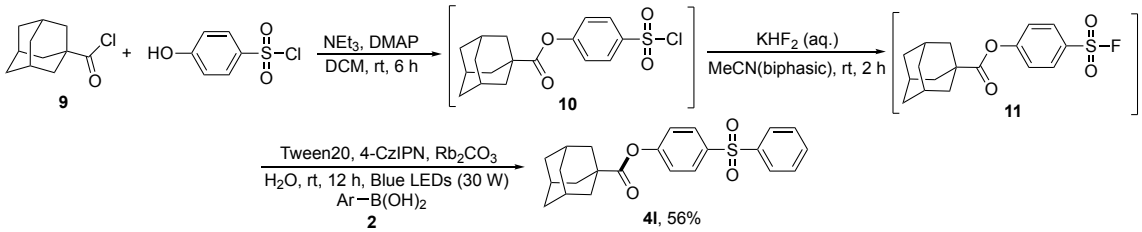
4.4.2. Esterification



6 **7** **8** **2** **4** **4j**, 64% **4k**, 52%

Carboxylic acid (1 mmol, 5.0 equiv.), hydroxyl substrate (1 mmol) and EDCI (1-ethyl-3-(3-(dimethylamino)propyl) carbodiimide hydrochloride) (1.1 mmol) was suspended in DCM (10 mL) and stirred at room temperature for 24 hours. The reaction mixture was diluted with DCM and quenched with sat. aq. $NaHCO_3$ (10 mL). The aqueous layer was extracted with DCM (3 x 5 mL). The combined organic layer was dried over Na_2SO_4. The filtrate was concentrated in vacuo and the residue was then suspended in sat. aq. KHF_2 (1.1 mmol, 2 mL) and MeCN (2 mL), and stirred at room temperature for 2 hours. The reaction mixture was diluted with EtOAc (1 mL) and water (2 mL). The aqueous layer was extracted with EtOAc (3 x 1 mL). The combine organic layer was dried over Na_2SO_4. The filtrate was concentrated in vacuo. Then follow the **General Procedure B** to give analytically pure product **4j** and **4k** (EtOAc/PE: 1/1).

4.4.3. Acyl Esterification

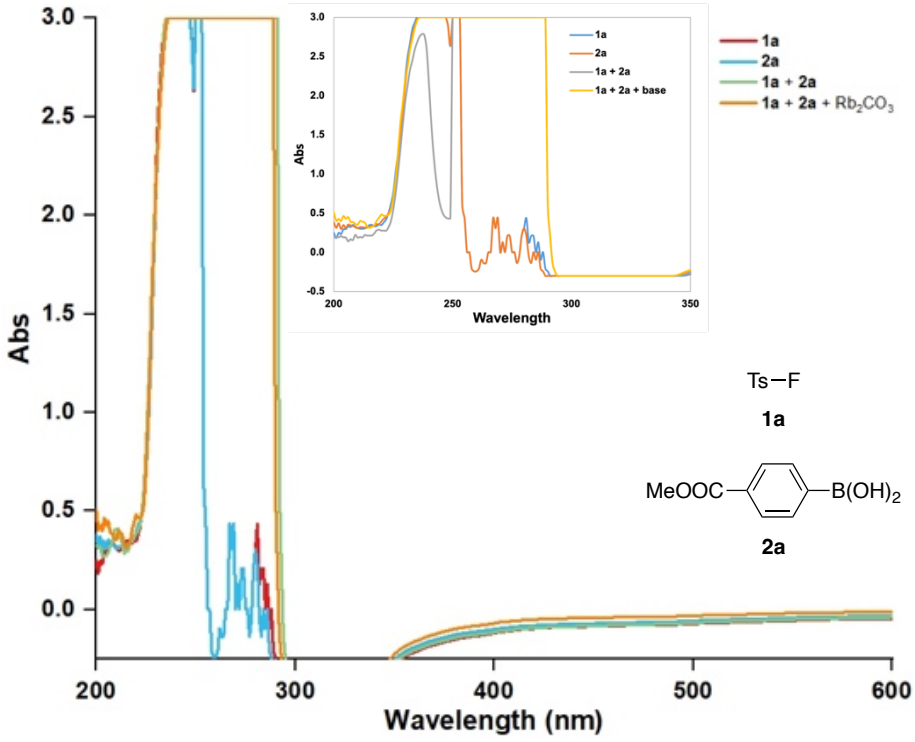


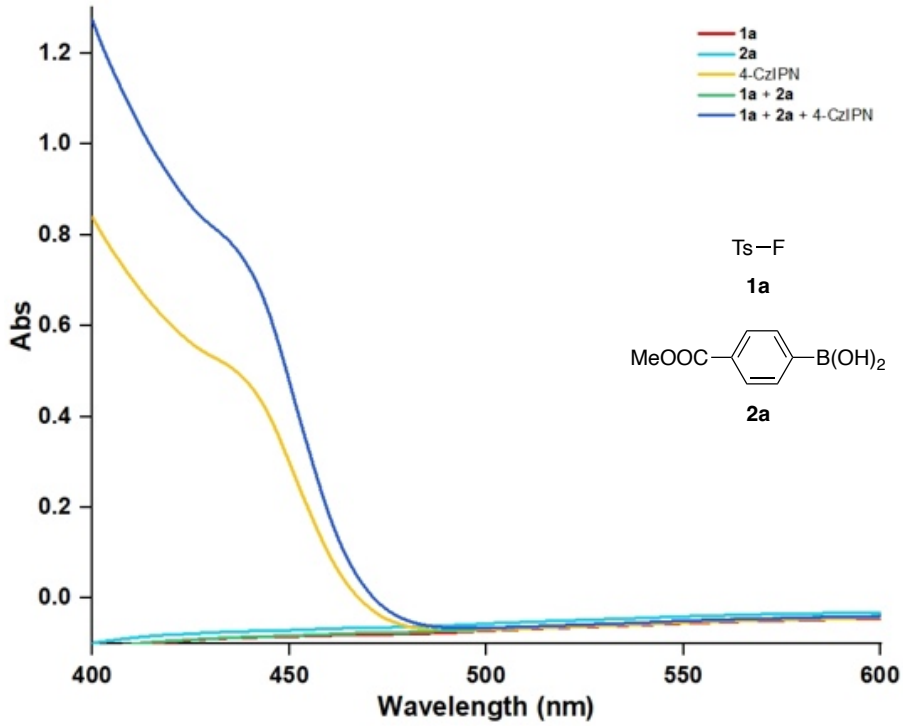
4-hydroxybenzenesulfonyl chloride (1.1 mmol, 5.5 equiv), DMAP (4-dimethylaminepyridine) (0.2 mmol) and Et_3N (2.2 mmol) in DCM (10 mL) was suspended and stirred for 10 minutes. Then 1-Adamantanecarbonyl chloride **9** (1 mmol, 5.0 equiv) was added dropwise at 0 ºC. The reaction mixture was warmed to room temperature and stirred for 6 hours before quenching with H_2O (5 mL) and extracting with DCM (3 x 5 mL). The combined organic layer was dried over Na_2SO_4. The filtrate was concentrated in vacuo and the residue was then suspended in sat. aq. KHF_2 (1.1 mmol, 2 mL) and MeCN (2 mL), and stirred at room temperature for 2 hours. The reaction mixture was diluted with EtOAc (1 mL) and water (2 mL). The aqueous layer was extracted with EtOAc (3 x 1 mL). The combine organic layer was dried over Na_2SO_4. The filtrate was concentrated in vacuo. Then follow the **General Procedure B** to give analytically pure product **4l.**

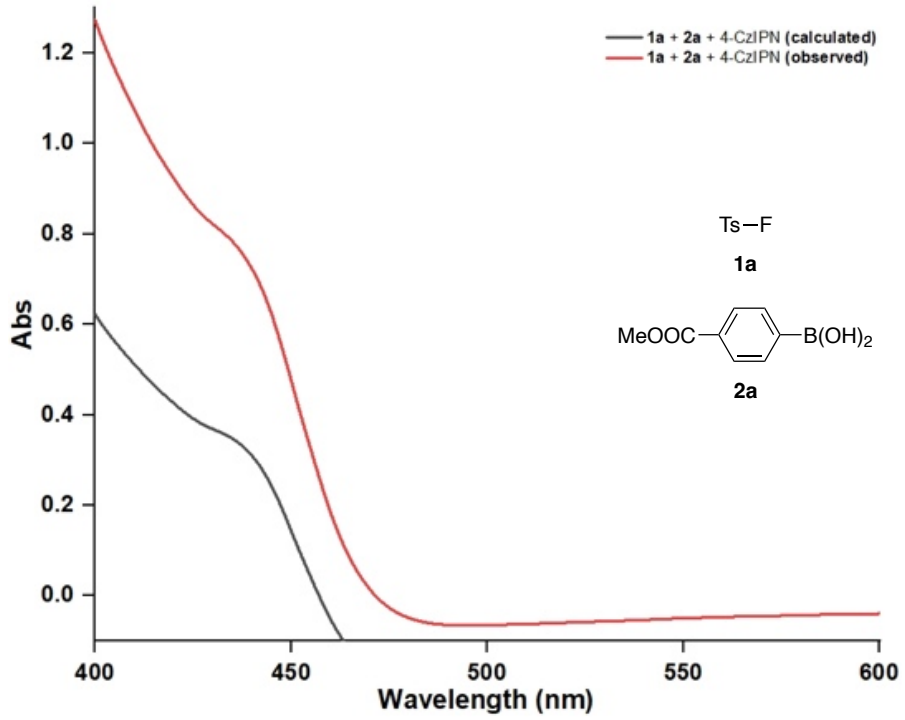
5. Mechanism Studies

5.1. UV-Vis Absorption Spectra Studies

UV-visible spectrum was conducted with **1a** (0.075 M), **2a** (0.05 M), base (Rb_2CO_3, 0.1 M) and 4-CzIPN (1 mol%) in degassed THF.

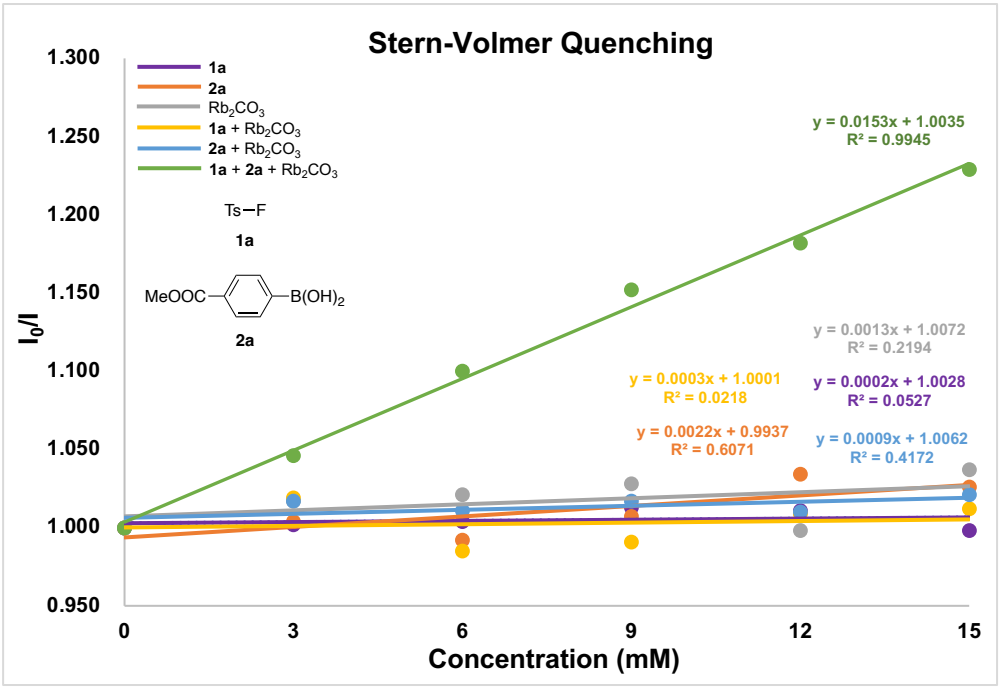






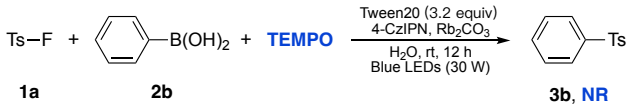
5.2. Stern-Volmer Quenching Experiments

Stern-Volmer experiments were conducted with 0.3 mM $[Ir(ppy)_2(dtbbpy)]PF_6$ and while probing the quenching ability of **1a**, **2a**, **1a** + base, **2a +** base and **1a** + **2a** + base (in degassed THF). The solutions were irradiated at 361 nm and luminescence was measured at 524 nm.

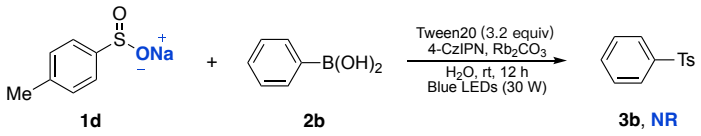


5.3. Control Experiments

a) Radical Trapping with TEMPO (2,2,6,6-tetramethylpiperidinooxy)



*b) Sodium 4-toluenesulfinate (**1d**) Instead*



c) Radical Trapping with DPE (1,1-diphenylethylene)

a) Radical Trapping Experiments with TEMPO

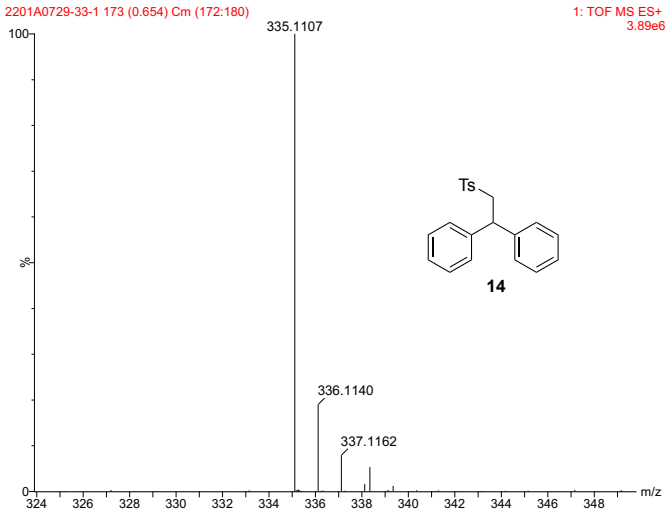
An oven-dried Schlenk tube equipped with a stir bar was charged with sulfonyl fluoride **1a** (0.3 mmol, 1.5 equiv), boronic acid **2b** (0.2 mmol, 1.0 equiv), Rb_2CO_3 (0.4 mmol, 2.0 equiv), 4-CzIPN (1 mol%) and TEMPO (2,2,6,6-Tetramethylpiperidinooxy) (0.2 mmol, 1.0 equiv). Then follow the **General Procedure B**. Finally, the desired product was not detected.

b) Sodium 4-toluenesulfinate Instead

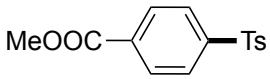
An oven-dried Schlenk tube equipped with a stir bar was charged with sodium 4-toluenesulfinate **1d** (0.3 mmol, 1.5 equiv), boronic acid **2b** (0.2 mmol, 1.0 equiv), Rb_2CO_3 (0.4 mmol, 2.0 equiv) and 4-CzIPN (1 mol%). Then follow the **General Procedure B**. Finally, the desired product was not detected.

c) Radical Trapping Experiments with DPE

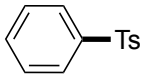
An oven-dried Schlenk tube equipped with a stir bar was charged with sulfonyl fluoride **1a** (0.3 mmol, 1.5 equiv), boronic acid **2b** (0.2 mmol, 1.0 equiv), Rb_2CO_3 (0.4 mmol, 2.0 equiv), 4-CzIPN (1 mol%) and DPE (1,1-diphenylethylene) (0.2 mmol, 1.0 equiv). Then follow the **General Procedure B**. Finally, the desired product was not detected yet DPE adduct **14** was detected by **HRMS** (ESI/$[M-H]^+$): Calculated for $C_{21}H_{20}O_2S$: 335.1106, Found: 335.1107.



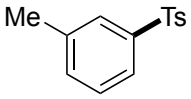
6. Characterization of Products



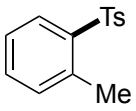
Following the **General Procedure A**, **3a** was isolated as white solid (46.5 mg, 80%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (d, *J* = 8.0 Hz, 2H), 7.98 (d, *J* = 8.0 Hz, 2H), 7.83 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 3.92 (s, 3H), 2.40 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 165.5, 145.9, 144.8, 137.8, 134.0, 130.4, 130.1,127.9, 127.5, 52.7, 21.6. **HRMS** (ESI/$[M+H]^+$) calculated for $C_{15}H_{15}O_4S$: 291.0686, found: 291.0682.



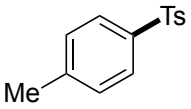
Following the **General Procedure A**, **3b** was isolated as white solid (23.7 mg, 51%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.0 Hz, 2H), 7.83 (d, *J* = 8.0 Hz, 2H), 7.56–7.47 (m, 3H), 7.29 (d, *J* = 8.0 Hz, 2H), 2.39 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.3, 142.1, 138.7, 133.1, 130.0, 129.3, 127.8, 127.6, 21.6. **HRMS** (ESI/$[M+H]^+$) calculated for $C_{14}H_{15}O_2S$: 247.0787, found: 247.0788.



Following the **General Procedure A**, **3c** was isolated as white solid (26.1 mg, 53%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, *J* = 8.0 Hz, 2H), 7.72 (d, *J* = 8.0 Hz, 2H), 7.36 (d, *J* = 8.0 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 2.39 (s, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.1, 141.8, 139.5, 138.8, 133.8, 129.9, 129.1, 127.8, 127.7, 124.7, 21.6, 21.4. **HRMS** (ESI/$[M+H]^+$) calculated for $C_{14}H_{15}O_2S$: 247.0787, found: 247.0788.

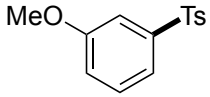


Following the **General Procedure A**, **3d** was isolated as white solid (27.1 mg, 55%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04-7.99 (m, 4H), 7.82 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 2.60 (s, 3H), 2.39 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.0, 139.2, 138.3, 137.9, 133.7, 132.6, 129.7, 129.3, 127.8, 126.4, 21.6, 20.2. **HRMS** (ESI/$[M+H]^+$) calculated for $C_{14}H_{15}O_2S$: 247.0787, found: 247.0787.

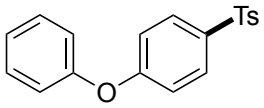


Following the **General Procedure A**, **3e** was isolated as white solid (32.5 mg, 66%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, *J* = 8.0 Hz, 4H), 7.30 (d, *J* = 8.0 Hz, 4H), 2.41 (s, 6H). **^{13}C**

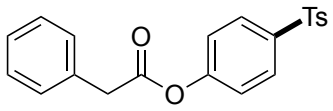
NMR (101 MHz, $CDCl_3$) δ 144.0, 139.1, 129.9, 127.6, 21.6. **HRMS** (ESI/$[M+H]^+$) calculated for $C_{14}H_{15}O_2S$: 247.0787, found: 247.0785.



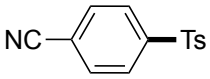
Following the **General Procedure A**, **3f** was isolated as white solid (31.5 mg, 60%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, *J* = 8.0 Hz, 2H), 7.50–7.36 (m, 3H), 7.29 (d, *J* = 8.0 Hz, 2H), 7.05 (d, *J* = 8.0 Hz, 1H), 3.83 (s, 3H), 2.39 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 160.1, 144.3, 143.2, 138.6, 130.4, 130.0, 127.8, 119.8, 119.4, 112.2, 55.8, 21.7. **HRMS** (ESI/$[M+H]^+$) calculated for $C_{14}H_{15}O_3S$: 263.0736, found: 263.0734.



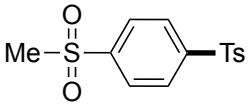
Following the **General Procedure A**, **3g** was isolated as light yellow solid (44.8 mg, 69%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.0 Hz, 2H), 7.81 (d, *J* = 8.0 Hz, 2H), 7.41–7.37 (m, 2H), 7. 29 (d, *J* = 8.0 Hz, 2H), 7.23–7.19 (m, 1H), 7.04–6.99 (m, 4H), 2.40 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 162.0, 155.0, 144.0, 139.1, 135.4, 130.2, 129.9, 129.8, 127.5, 125.0, 120.4, 117.7, 21.6. **HRMS** data are in accordance with the literature report.[10]



Following the **General Procedure A**, **3h** was isolated as white solid (52.0 mg, 71%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (d, *J* = 8.0 Hz, 2H), 7.98 (d, *J* = 8.0 Hz, 2H), 7. 82 (d, *J* = 8.0 Hz, 2H), 7.42–7.34 (m, 5H), 7.30 (d, *J* = 8.0 Hz, 2H), 5.36 (s, 2H), 2.40 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 165.0, 146.1, 144.8, 137.9, 135.5, 134.2, 130.6, 130.2, 128.8, 128.6, 128.4, 128.0, 127.6, 67.5, 21.7. **HRMS** (ESI/$[M+H]^+$) calculated for $C_{21}H_{18}O_4S$: 367.0999, found: 367.1027.

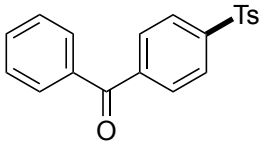


Following the **General Procedure A**, **3i** was isolated (EtOAc/PE: 1/2 to 1/1) as white solid (40.7 mg, 79%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.0 Hz, 2H), 7.83–7.76 (m, 4H), 7.33 (d, *J* = 8.0 Hz, 2H), 2.41 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 146.3, 145.3, 137.1, 133.1, 130.3, 128.2, 128.1, 117.3, 116.7, 21.7; **HRMS** (ESI/$[M+H]^+$) calculated for $C_{14}H_{12}O_2S$: 258.0583, found: 258.0582.

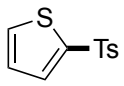


Following the **General Procedure A**, **3j** was isolated as white solid (45.9 mg, 74%).

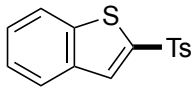
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.12–8.04 (m, 4H), 7.83 (d, *J* = 8.0 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 3.05 (s, 3H), 2.41 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 147.3, 145.4, 144.6, 137.2, 130.4, 128.64, 128.56,128.2, 44.3, 21.7. **HRMS** (ESI/$[M+H]^+$) calculated for $C_{14}H_{14}O_4S_2$: 311.0406, found: 311.0419.



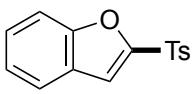
Following the **General Procedure A**, **3k** was isolated as white solid (37.7 mg, 56%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (d, *J* = 12.0 Hz, 2H), 7.86 (d, *J* = 8.0 Hz, 4H), 7.76 (d, *J* = 8.0 Hz, 2H), 7.64–7.60 (m, 1H), 7.51–7.47 (m, 2H), 7. 33 (d, *J* = 8.0 Hz, 2H), 2.42 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 195.3, 145.1, 144.8, 141.5, 137.9, 136.4, 133.3, 130.5, 130.1, 128.6, 128.0, 127.5, 21.7. **HRMS** data are in accordance with the literature report.[7]



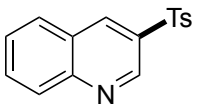
Following the **General Procedure A**, **3l** was isolated as white solid (37.2 mg, 78%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.0 Hz, 2H), 7.66 (d, *J* = 4.0 Hz, 1H), 7.62 (d, *J* = 4.0 Hz, 1H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.07–7.05 (m, 1H), 2.40 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.4, 143.5, 139.1, 133.6, 133.1, 130.0, 127.8, 127.4, 21.6. **HRMS** data are in accordance with the literature report.[5]



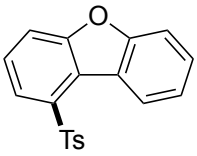
Following the **General Procedure A**, **3m** was isolated as white solid (46.1 mg, 80%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95–7.91 (m, 3H), 7.86 (d, *J* = 8.0 Hz, 1H), 7.81 (d, *J* = 8.0 Hz, 1H), 7.45–7.40 (m, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 2.40 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.7, 143.4, 142.7, 138.4, 137.8, 130.0, 127.7, 127.4, 125.9, 125.5, 122.8, 21.7. **HRMS** data are in accordance with the literature report.[5]



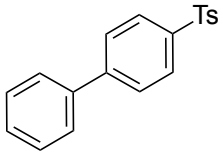
Following the **General Procedure A**, **3n** was isolated as white solid (28.9 mg, 53%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.0 Hz, 2H), 7.67 (d, *J* = 8.0 Hz,1H), 7.54–7.49 (m, 2H), 7.45–7.41 (m, 1H), 7.36–7.29 (m, 3H), 2.42 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 156.3, 151.9, 145.3, 136.4, 130.1, 128.3, 127.9, 126.0, 124.3, 123.1, 112.9, 112.5, 21.7. **HRMS** (ESI/$[M+H]^+$) calculated for $C_{15}H_{13}O_3S$: 273.0580, found: 273.0577.



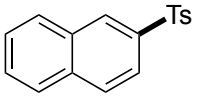
Following the **General Procedure A**, **3o** was isolated as yellow solid (23.2 mg, 41%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 9.28 (s, 1H), 8.83 (s, 1H), 8.18 (d, *J* = 8.0 Hz, 1H),7.99-7.87 (m, 4H), 7.72-7.68 (m, 1H), 7.35 (d, *J* = 8.0, 2H), 2.42 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 149.3, 147.1, 144.9, 138.0, 136.7, 135.1, 132.7, 130.2, 129.6, 129.2, 128.3, 127.9, 126.4, 21.6. **HRMS** (ESI/$[M+H]^+$) calculated for $C_{16}H_{14}NO_2S$: 284.0745, found: 284.0747.



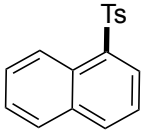
Following the **General Procedure A**, **3p** was isolated as white solid (32.9 mg, 51%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.81 (d, *J* = 8.0 Hz, 1H), 8.08 (d, *J* = 8.0 Hz,1H), 7.89–7.83 (m, 3H), 7.64–7.54 (m, 3H), 7.45–7.41 (m, 1H), 7.27 (d, *J* = 8.0 Hz, 2H), 2.37 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 156.8, 144.3, 138.1, 134.6, 129.8, 128.9, 127.3, 126.6, 125.9, 124.5, 123.5, 121.6, 121.1, 117.0, 111.6, 21.6. **HRMS** (ESI/$[M+H]^+$) calculated for $C_{19}H_{15}O_3S$: 323.0736, found: 323.0731.



Following the **General Procedure A**, **3q** was isolated as white solid (55.5 mg, 90%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, *J* = 8.0 Hz, 2H), 7.87 (d, *J* = 8.0 Hz, 2H), 7.69 (d, *J* = 8.0 Hz, 2H), 7.56 (d, *J* = 8.0 Hz, 2H), 7.47–7.40 (m, 3H), 7.31 (d, *J* = 8.0 Hz, 2H), 2.40 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ146.0, 144.2, 140.5, 139.2, 138.8, 130.0, 129.07, 128.6, 128.1, 127.9, 127.7, 127.4, 21.6. **HRMS** data are in accordance with the literature report.[7]

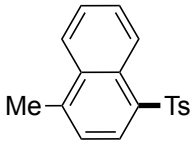


Following the **General Procedure A**, **3r** was isolated as white solid (33.9 mg, 60%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.56 (s, 1H), 7.97 (d, *J* = 8.0 Hz, 1H), 7.92–7.83 (m, 5H), 7.64–7.57 (m, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 2.38 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.2, 138.8, 138.7, 134.9, 132.2, 130.0, 129.6, 129.4, 129.1, 128.8, 127.9, 127.8, 127.6, 122.6, 21.6. **HRMS** data are in accordance with the literature report.[6]

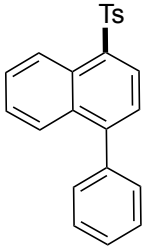


Following the **General Procedure A**, **3s** was isolated as white solid (32.2 mg, 57%)。

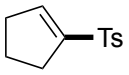
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.66 (d, *J* = 8.0 Hz, 1H), 8.51 (d, *J* = 8.0 Hz, 1H), 8.09 (d, *J* = 8.0 Hz, 1H), 7.91–7.86 (m, 3H), 7.64–7.54 (m, 4H), 7.27 (d, *J* = 8.0 Hz, 2H), 2.37 (s, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 144.0, 138.8, 136.2, 135.0, 134.2, 129.8, 129.0, 128.4, 128.3, 127.5, 126.9, 124.4, 124.4, 21.6. **HRMS** data are in accordance with the literature report.[6]



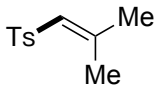
Following the **General Procedure A**, **3t** was isolated as white solid (43.4 mg, 73%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.69–8.66 (m, 1H), 8.42 (d, *J* = 8.0 Hz, 1H), 8.07–8.04 (m, 1H), 7.85 (d, *J* = 8.0 Hz, 2H), 7.58–7.56 (m, 2H), 7.47 (d, *J* = 8.0 Hz, 1H), 7.25 (d, *J* = 8.0 Hz, 2H), 2.76 (s, 3H), 2.35 (s, 3H). **^{13}C NMR** (126 MHz, $CDCl_3$) δ 143.8, 142.4, 139.1, 134.2, 133.3, 129.7, 128.5, 127.8, 127.4, 126.7, 125.3, 125.04, 125.0, 21.5, 20.2. **HRMS** data are in accordance with the literature report.[7]



Following the **General Procedure A**, **3u** was isolated as white solid (47.3 mg, 66%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.74 (d, *J* = 8.0 Hz, 1H), 8.55 (d, *J* = 8.0 Hz, 1H), 7.91 (d, *J* = 8.0 Hz, 3H), 7.58–7.43 (m, 8H), 7.29 (d, *J* = 8.0 Hz, 2H), 2.37 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 147.3, 144.0, 139.4, 138.9, 135.3, 132.6, 129.8, 129.3, 128.9, 128.5, 128.2, 128.0, 127.6, 127.4, 126.8, 125.5, 124.6, 21.6. **HRMS** data are in accordance with the literature report.[7]

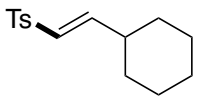


Following the **General Procedure A**, **3v** was isolated as white solid (17.78 mg, 40%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.0 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 6.71(s, 1H), 2.53 (t, *J* = 8.0 Hz, 4H), 2.44 (s, 3H), 2.05–1.98 (m, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.9, 144.3, 142.8, 136.7, 129.8, 128.0, 32.9,30.8, 23.7, 21.6. **HRMS** data are in accordance with the literature report.[4]

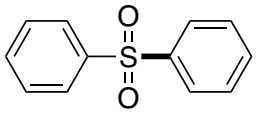


Following the **General Procedure A**, **3w** was isolated as light yellow solid (14.9 mg, 38%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 6.17(s, 1H), 2.43 (s, 3H), 2.14 (s, 3H), 1.87 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 153.7, 143.9, 139.5, 129.8, 127.2,

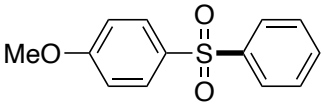
126.6, 27.1, 21.6, 19.2. **HRMS** data are in accordance with the literature report.[8]



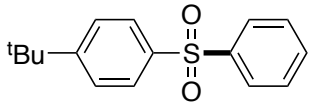
Following the **General Procedure A**, **3x** was isolated as white solid (30.1 mg, 57%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 6.93-6.88 (m 1H), 6.22 (d, *J* = 16.0 Hz, 1H), 2.42 (s, 3H), 1.76-1.63 (m 6H), 1.25-1.10 (m, 5H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 151.2, 144.1, 137.9, 129.9, 128.6, 127.6, 39.9, 31.3, 25.7, 25.6, 21.6. **HRMS** data are in accordance with the literature report.[9]



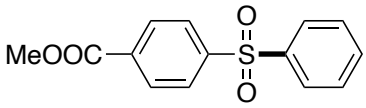
Following the **General Procedure B**, **4a** was isolated as white solid (26.2 mg, 60%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96–7.94 (m, 4H), 7.58–7.49 (m, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 141.6, 133.2, 129.3, 127.7. **HRMS** (ESI/$[M+H]^+$) calculated for $C_{12}H_{10}O_2S$: 219.0474, found: 219.0457.



Following the **General Procedure B**, **4b** was isolated as white solid (27.3 mg, 55%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92–7.86 (m, 4H), 7.54–7.46 (m, 3H), 6.97–6.94 (m, 2H), 3.83 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 163.4, 142.4, 133.1, 132.8, 129.9, 129.2, 127.3, 114.5, 55.7. **HRMS** (ESI/$[M+H]^+$) calculated for $C_{13}H_{12}O_3S$: 249.0580, found: 249.0590.

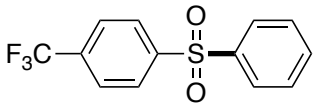


Following the **General Procedure B**, **4c** was isolated as white solid (28.1 mg, 51%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.0 Hz, 2H), 7.86 (d, *J* = 8.0 Hz, 2H), 7.57–7.53 (m, 1H), 7.51–7.47 (m, 4H), 1.30 (s, 9H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 157.1, 141.9, 138.5, 133.0, 129.2, 127.6, 127.5, 126.3, 35.2, 31.0. **HRMS** (ESI/$[M+H]^+$) calculated for $C_{16}H_{18}O_2S$: 275.1100, found: 275.1076.

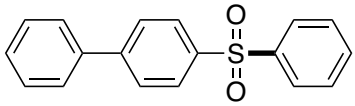


Following the **General Procedure B**, **4d** was isolated as white solid (35.4 mg, 64%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (d, J = 8.0 Hz, 2H), 8.03 (d, J = 8.0 Hz, 2H), 7.97 (d, J = 8.0 Hz, 2H), 7.63–7.60 (m, 1H), 7.56–7.52 (m, 2H), 3.95 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 165.5,

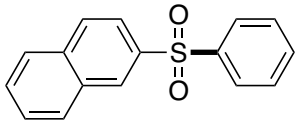
145.5, 140.8, 134.3, 133.7, 130.5, 129.5, 127.9, 127.7, 52.7. **HRMS** (ESI/$[M+H]^+$) calculated for $C_{14}H_{13}O_4S$: 277.0529, found: 277.0525.



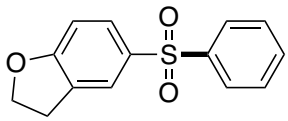
Following the **General Procedure B**, **4e** was isolated as white solid (40.8 mg, 71%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, J = 8.0 Hz, 2H), 7.96 (d, J = 8.0 Hz, 2H), 7.76 (d, J = 8.0 Hz, 2H), 7.63–7.52 (m, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 145.2, 140.6, 134.9 (q, J = 32.8 Hz), 133.8, 129.6, 128.2, 127.9, 126.5, 122.0 (q, J = 274.7 Hz). **^{19}F NMR** (377 MHz, $CDCl_3$) δ 63.20. **HRMS** (ESI/$[M+Na]^+$) calculated for $C_{13}H_9O_2F_3S$: 309.0168, found: 309.0153.



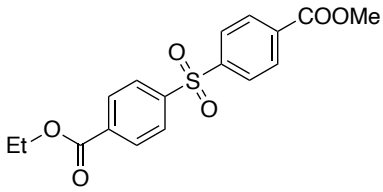
Following the **General Procedure B**, **4f** was isolated as white solid (40.0 mg, 68%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05–8.01 (m, 4H), 7.72 (d, J = 12.0 Hz, 2H), 7.59–7.53 (m, 5H), 7.50–7.41 (m, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 146.2, 141.7, 140.1, 139.2, 133.2, 129.4, 129.1, 128.6, 128.2, 128.0, 127.7, 127.4. **HRMS** data are in accordance with the literature report.[10]



Following the **General Procedure B**, **4g** was isolated as white solid (38.6 mg, 72%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.59 (s, 1H), 8.02–7.85 (m, 6H), 7.65–7.58 (m, 2H), 7.55–7.48 (m, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ141.6, 138.4, 135.0, 133.2, 132.2, 129.7, 129.4, 129.3, 129.2, 129.1, 127.9, 127.7, 127.7, 122.7. **HRMS** data are in accordance with the literature report.[11]

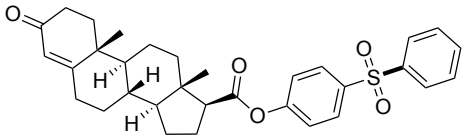


Following the **General Procedure B**, **4h** was isolated (EtOAc/PE: 1/2 to 1/1) as white solid (38.5 mg, 74%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, J = 8.0 Hz, 2H), 7.76 (d, J = 4.0 Hz, 2H), 7.57–7.48 (m, 3H), 6.84 (d, J = 8.0 Hz, 1H), 4.66 (t, J = 8.0 Hz, 2H), 3.25 (t, J = 8.0 Hz, 2H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 164.4, 142.5, 133.0, 132.8, 129.5, 129.2, 128.7, 127.2, 125.0, 109.8, 72.4, 29.0. **HRMS** data are in accordance with the literature report.[11]



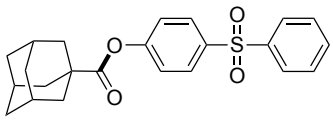
According to tandem one-pot reaction, **4j** was isolated as white solid (44.6 mg, 64%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19-8.16 (m, 4H), 8.03 (d, *J* = 8.0 Hz, 4H), 4.40 (q, *J* = 8.0 Hz, 2H), 3.95 (s, 3H), 1.40 (t, *J* = 8.0 Hz, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 165.3, 164.8, 144.8, 144,6, 135.1, 134.7, 130.6, 130.5, 127.9, 61.8, 52.7, 14.2. **HRMS** (ESI/$[M+H]^+$) calculated for $C_{17}H_{16}O_6S$: 349.0740, found: 349.0734.



According to tandem one-pot reaction, **4k** was isolated (EtOAc/PE: 1/1 to 2/1) as colorless liquid (55.3 mg, 52%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96–7.91 (m, 4H), 7.56–7.47 (m, 3H), 7.22 (d, *J* = 8.0 Hz, 2H), 5.73 (s, 1H), 3.86 (s, 1H), 2.63–2.58 (m, 1H), 2.42–2.36 (m, 2H), 2.30–2.26 (m, 1H), 2.21–2.12 (m, 2H), 2.04–2.01 (m, 1H), 1.95–1.85 (m, 2H), 1.79–1.71 (m, 2H), 1.64–1.54 (m, 2H), 1.47–1.39 (m, 2H), 1.27–1.23 (m, 2H), 1.18 (s, 3H), 1.08–0.96 (m, 2H), 0.82 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 199.7, 171.8, 171.1, 154.5, 141.4, 138.7, 133.9, 133.3, 129.4, 127.6, 124.0, 122.6, 55.4, 55.1, 53.6, 44.6, 38.6, 38.2, 35.7, 33.9, 32.8, 31.9, 29.7, 24.4, 23.6, 20.9, 17.4, 13.7. **HRMS** data are in accordance with the literature report.[6]



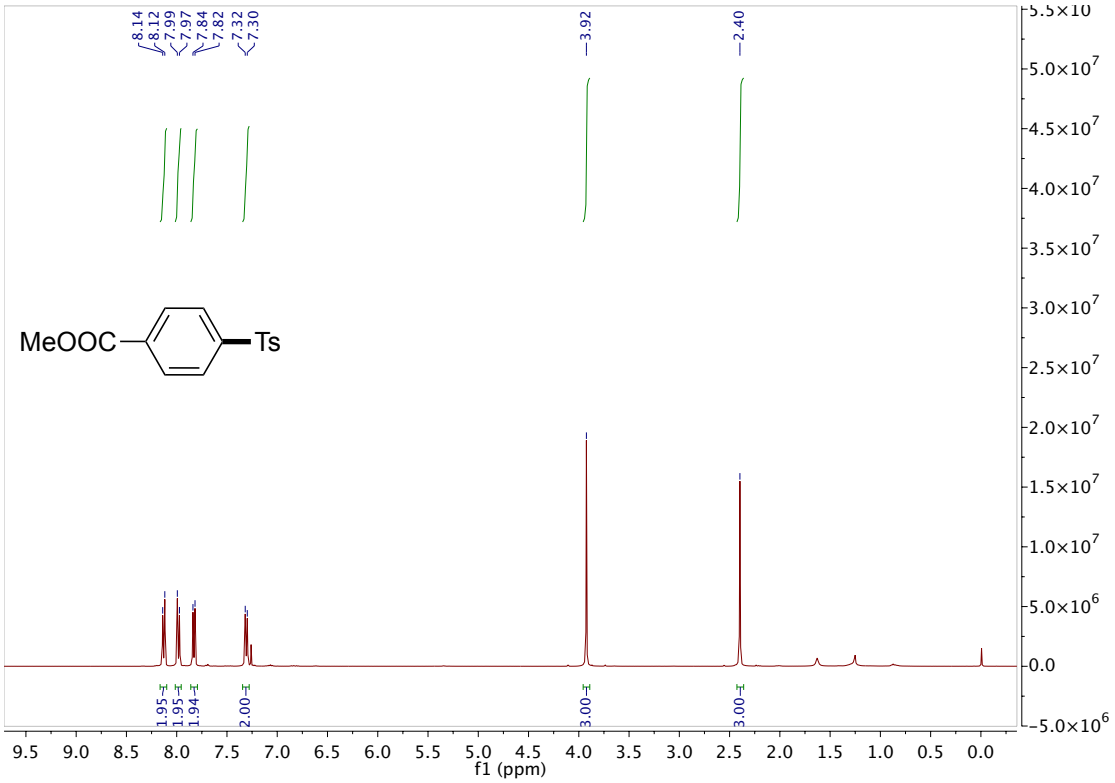
According to tandem one-pot reactions, **4l** was isolated as white solid (44.4 mg, 56%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96–7.92 (m, 4H), 7.58–7.48 (m, 3H), 7.19 (d, *J* = 8.0 Hz, 2H), 2.08–2.02 (m, 9H), 1.79–1.72 (m, 6H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 175.4, 154.9, 141.5, 138.5, 133.3, 129.4, 127.6, 122.6, 41.2, 38.6, 36.3, 27.8. **HRMS** data are in accordance with the literature report.[6]

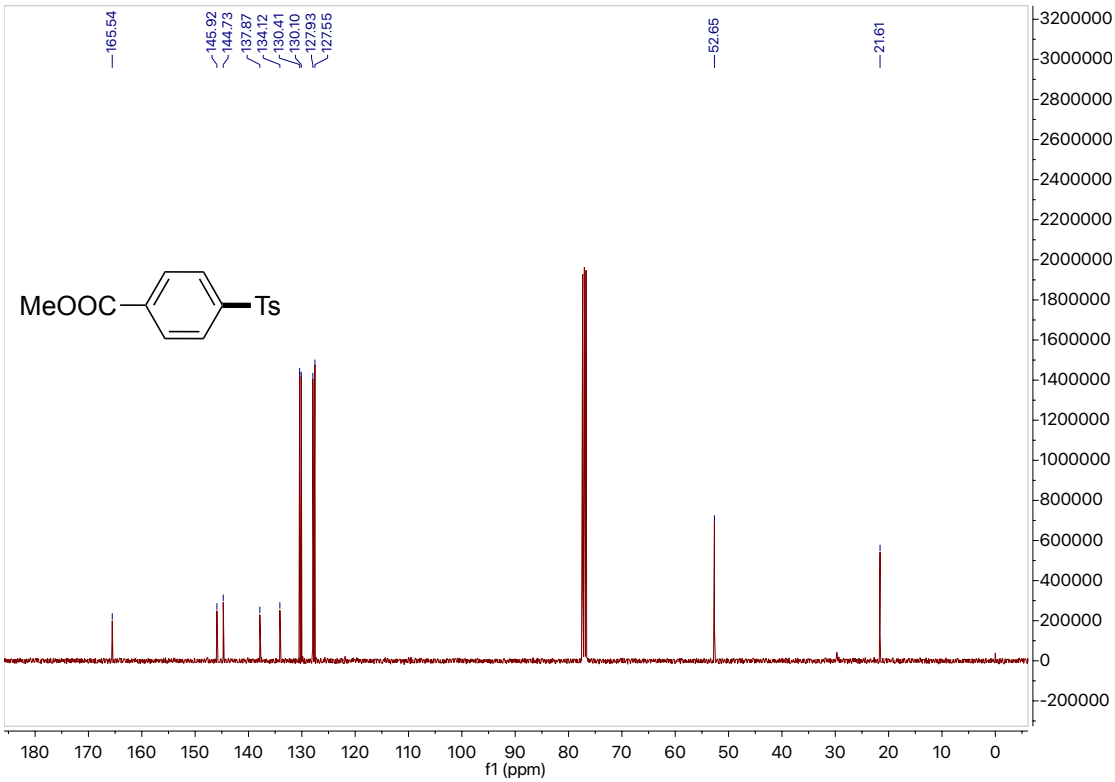
7. Spectra for Described Compounds

(3a)

^{1}H Spectra

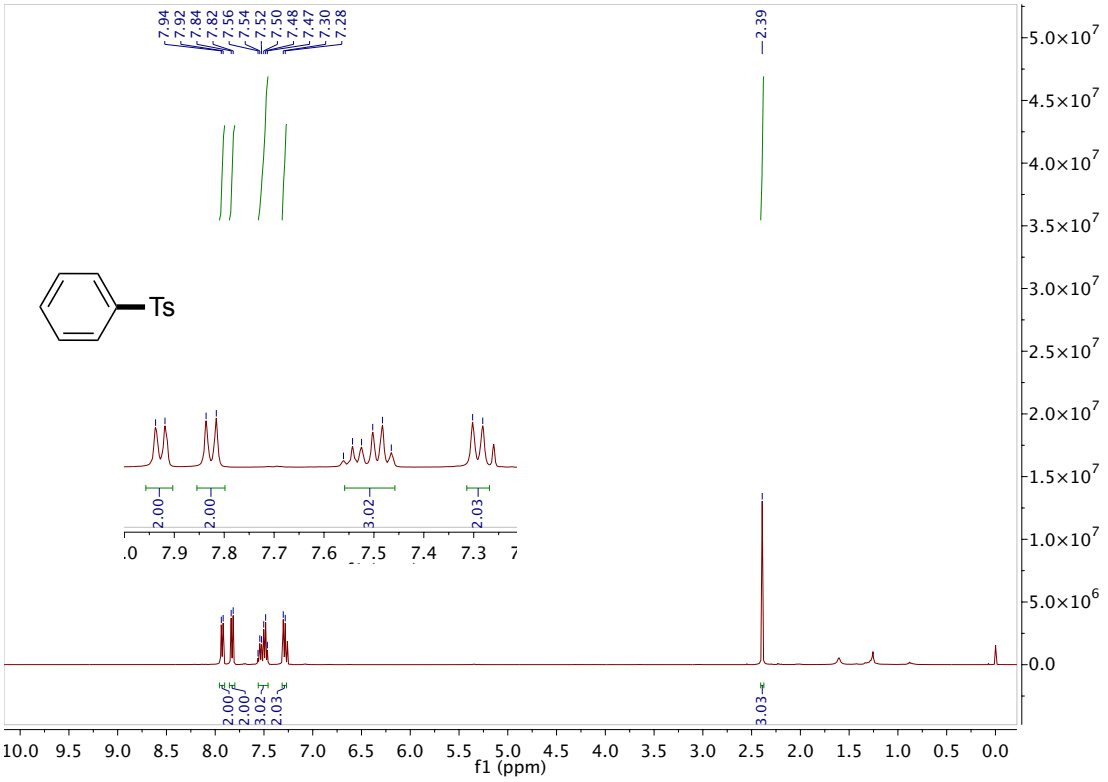


^{13}C Spectra



(3b)

^{1}H Spectra



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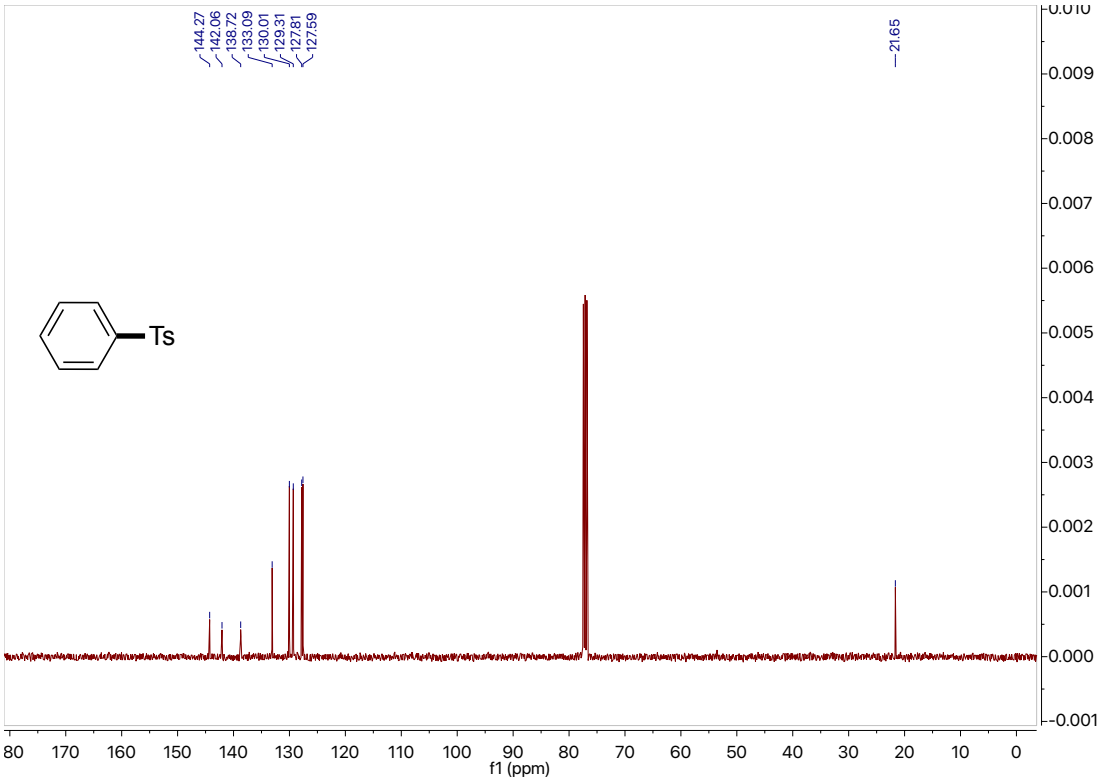
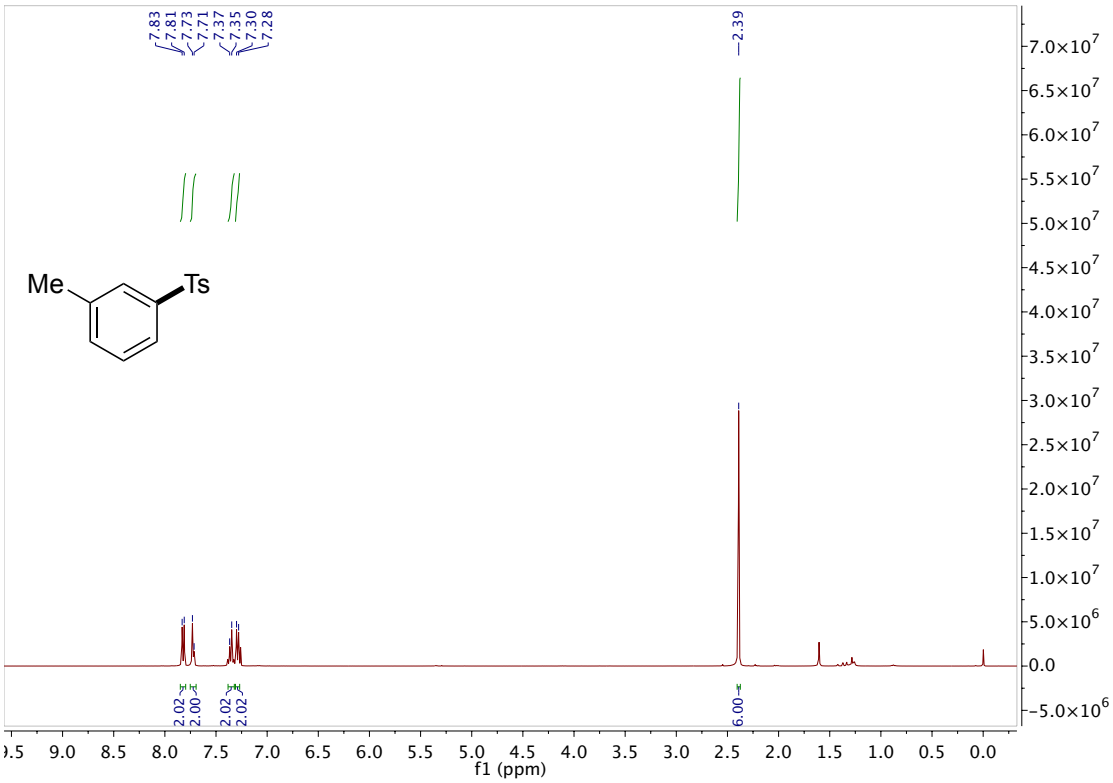


(3c)

^{1}H Spectra



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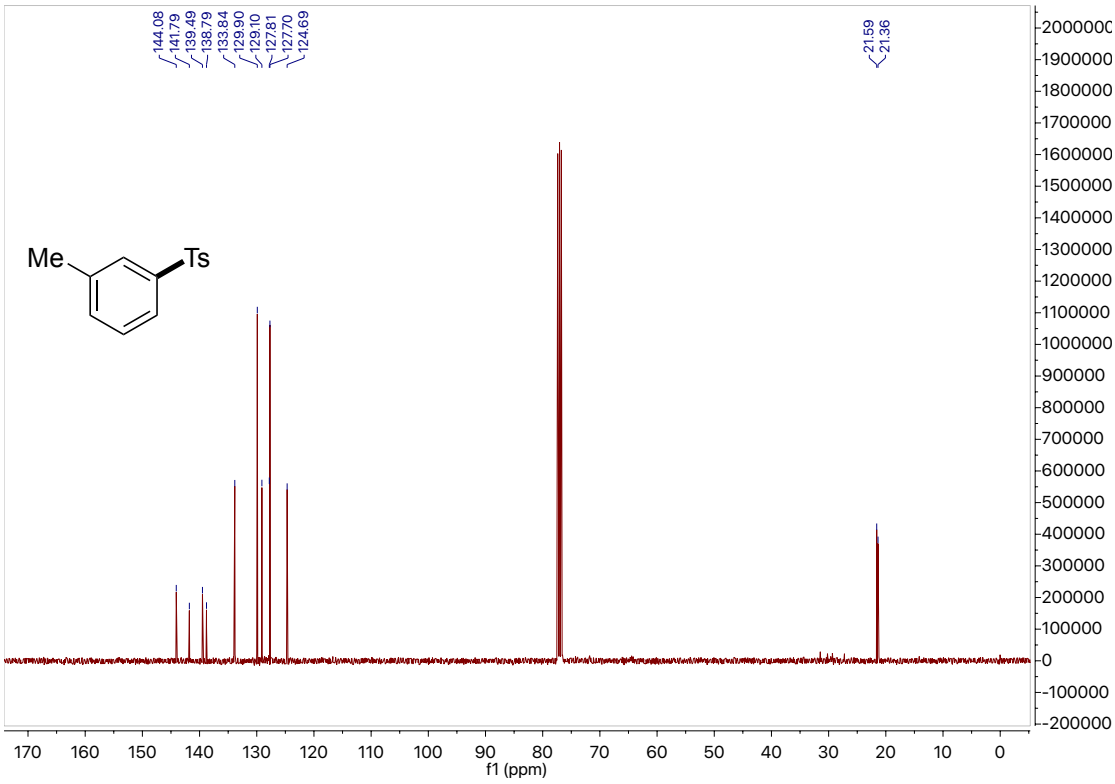
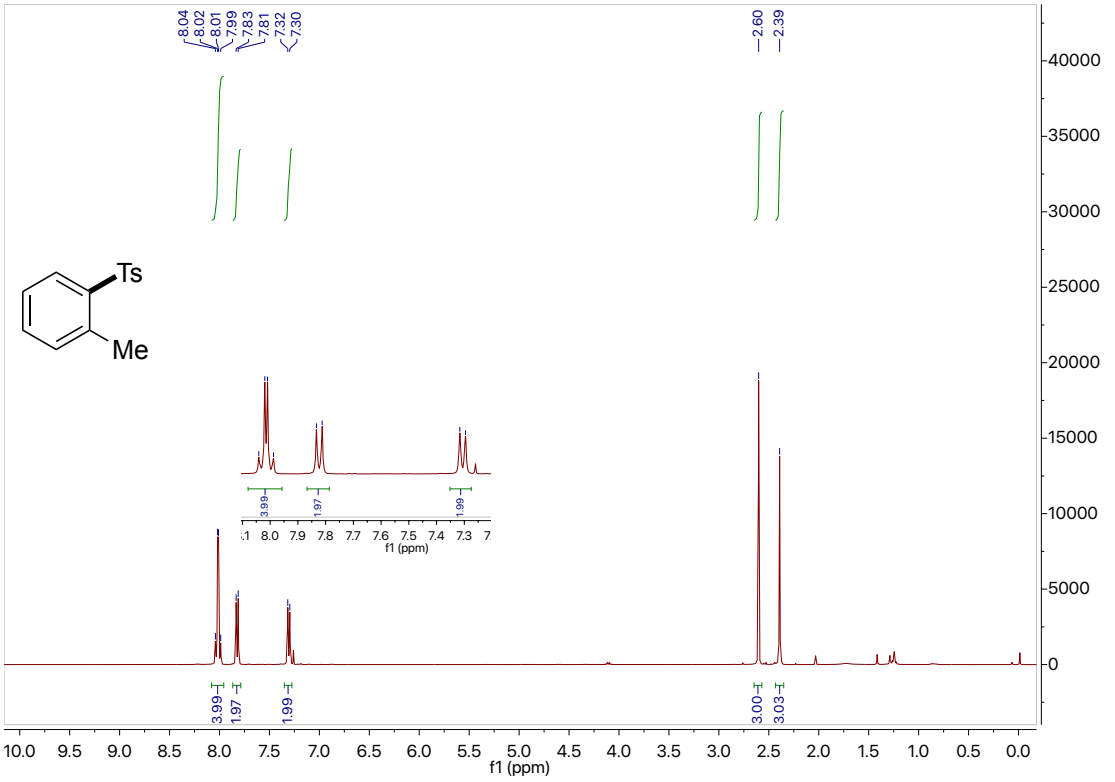
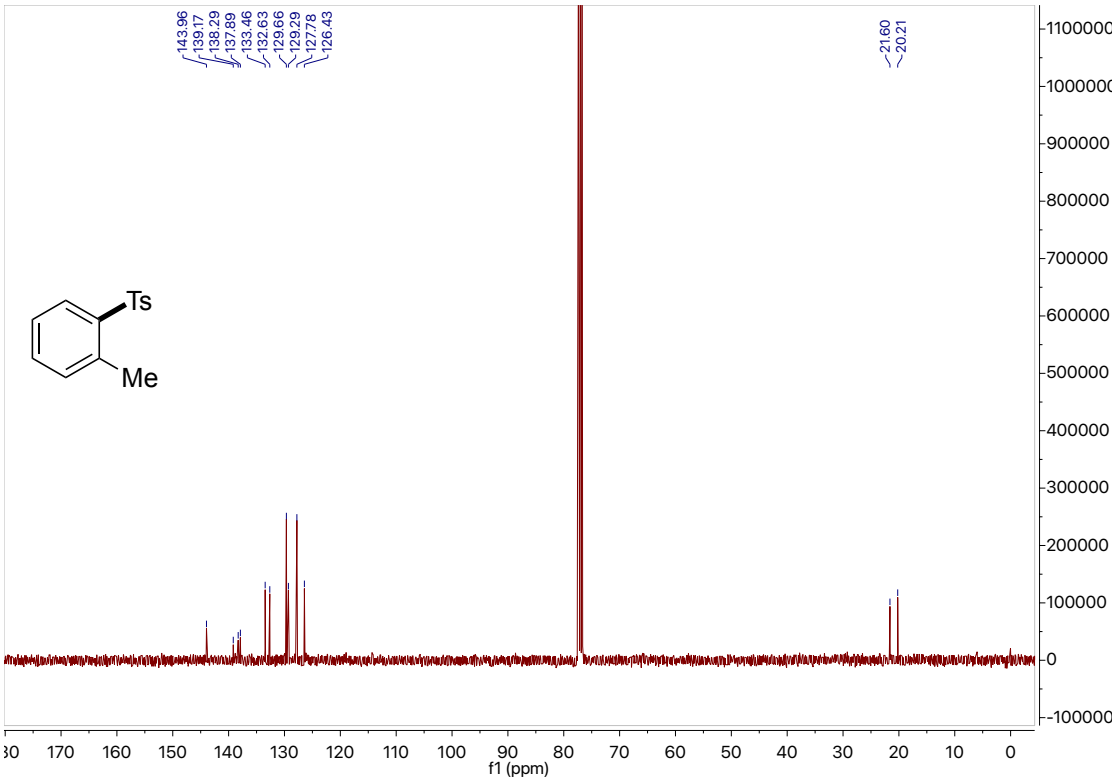


(3d)

^{1}H Spectra

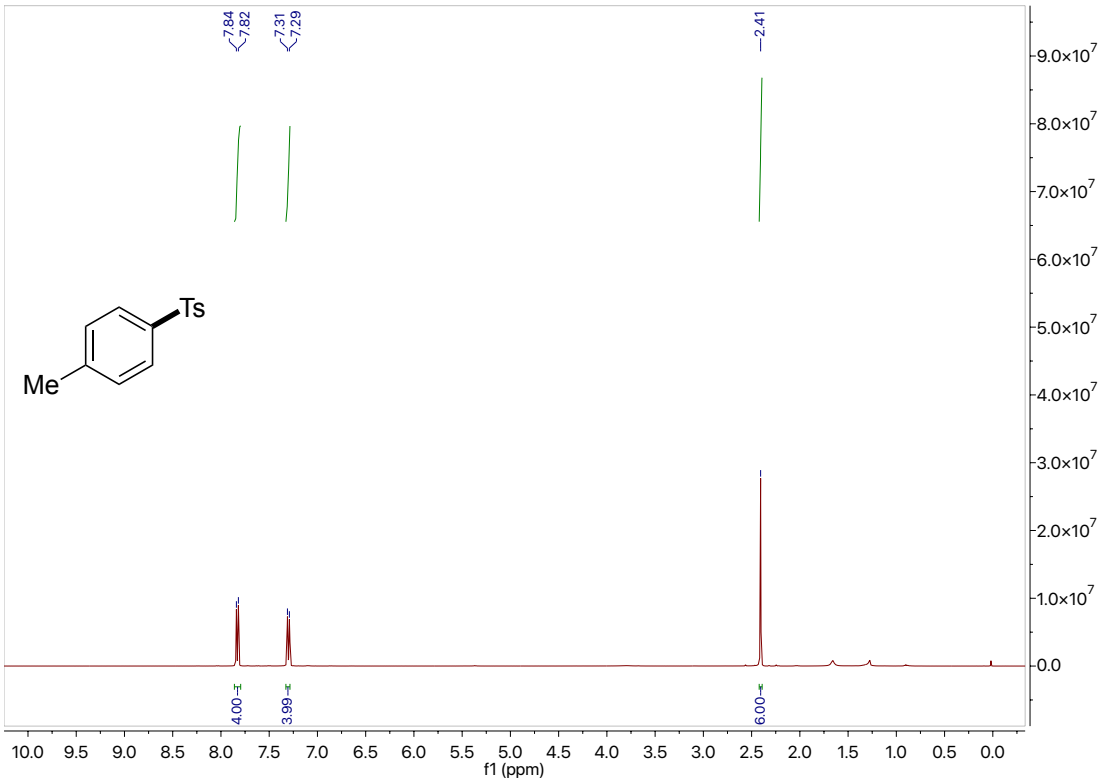


^{13}C Spectra



(3e)

^{1}H Spectra



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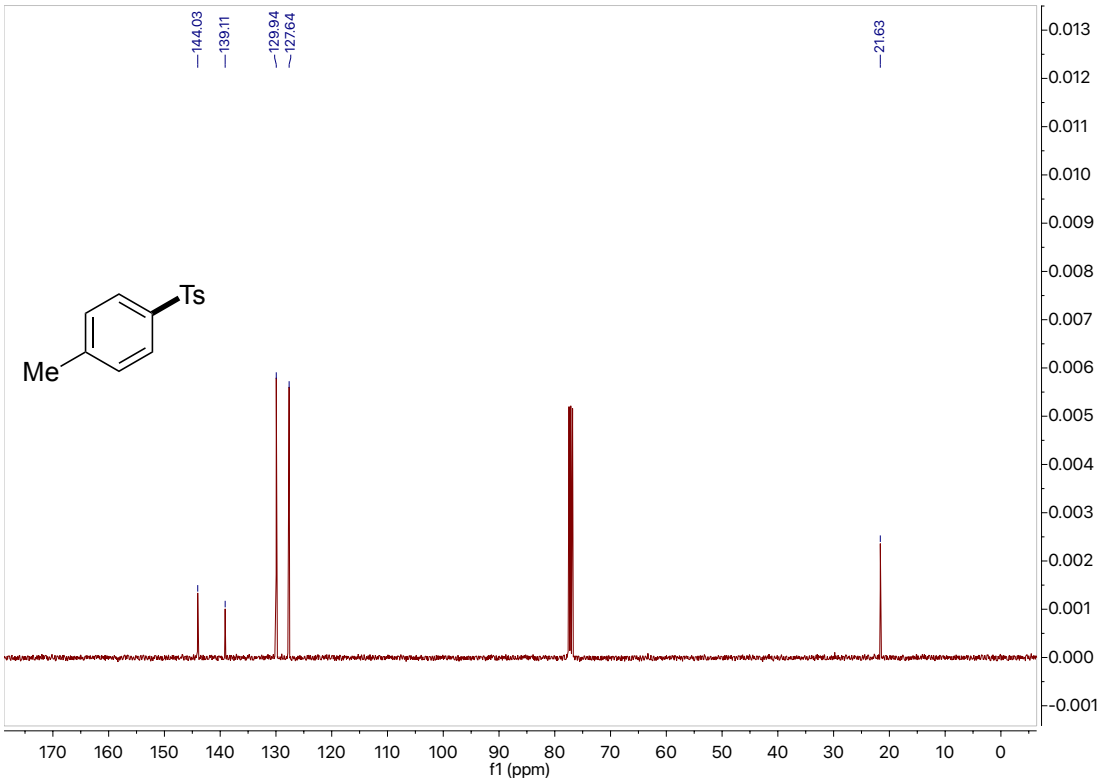

(3f)

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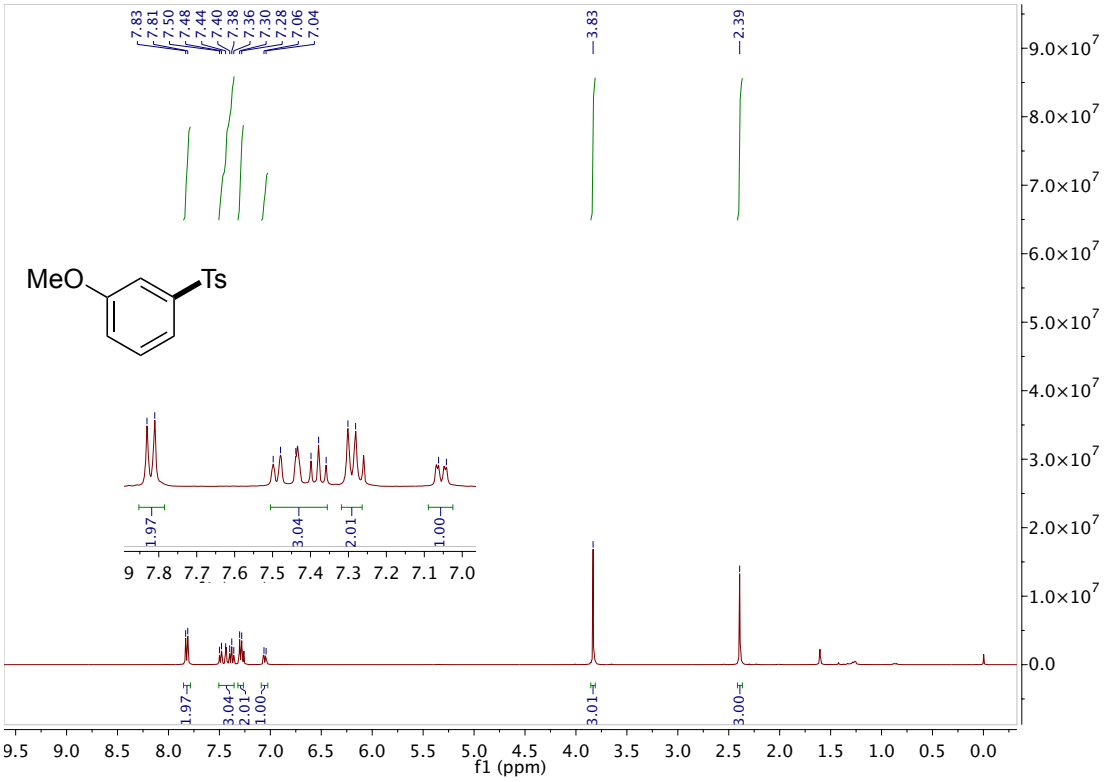
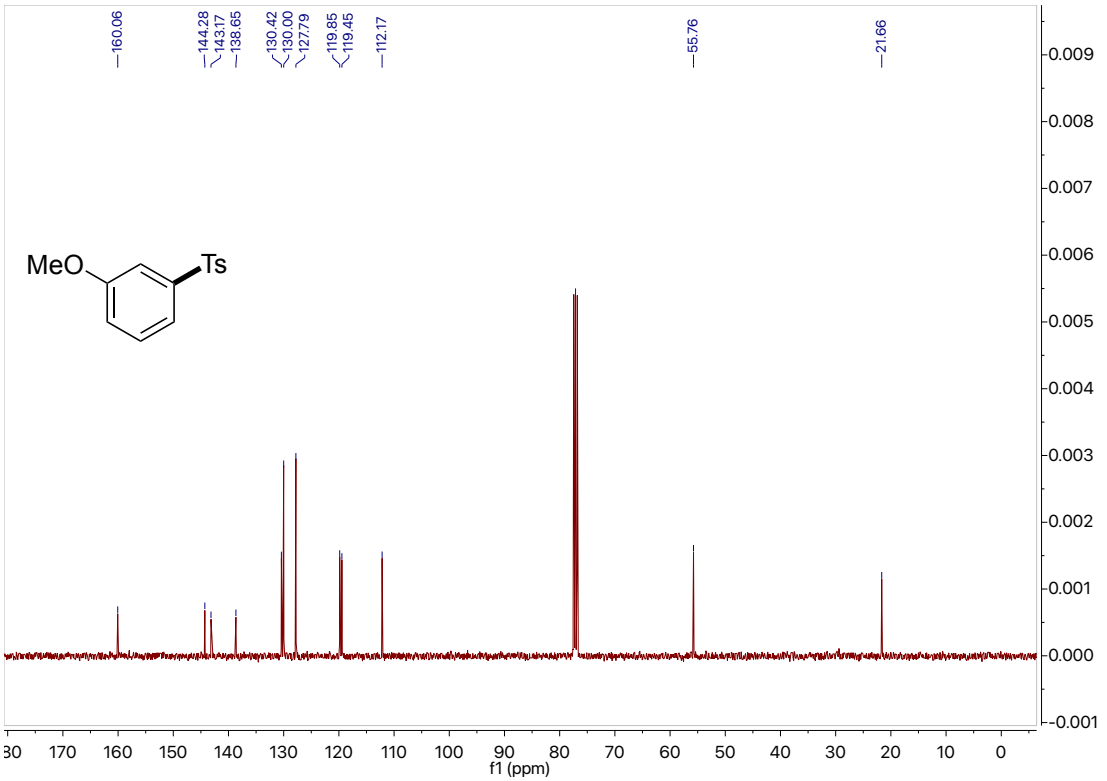
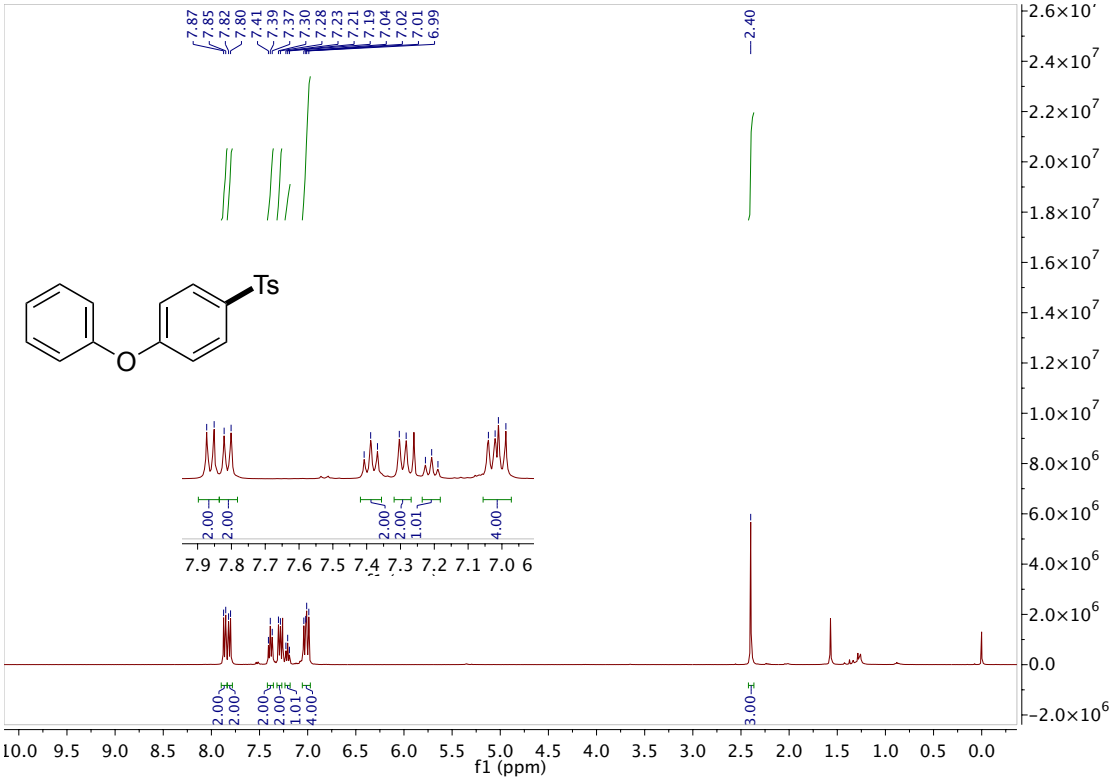


^{13}C Spectra

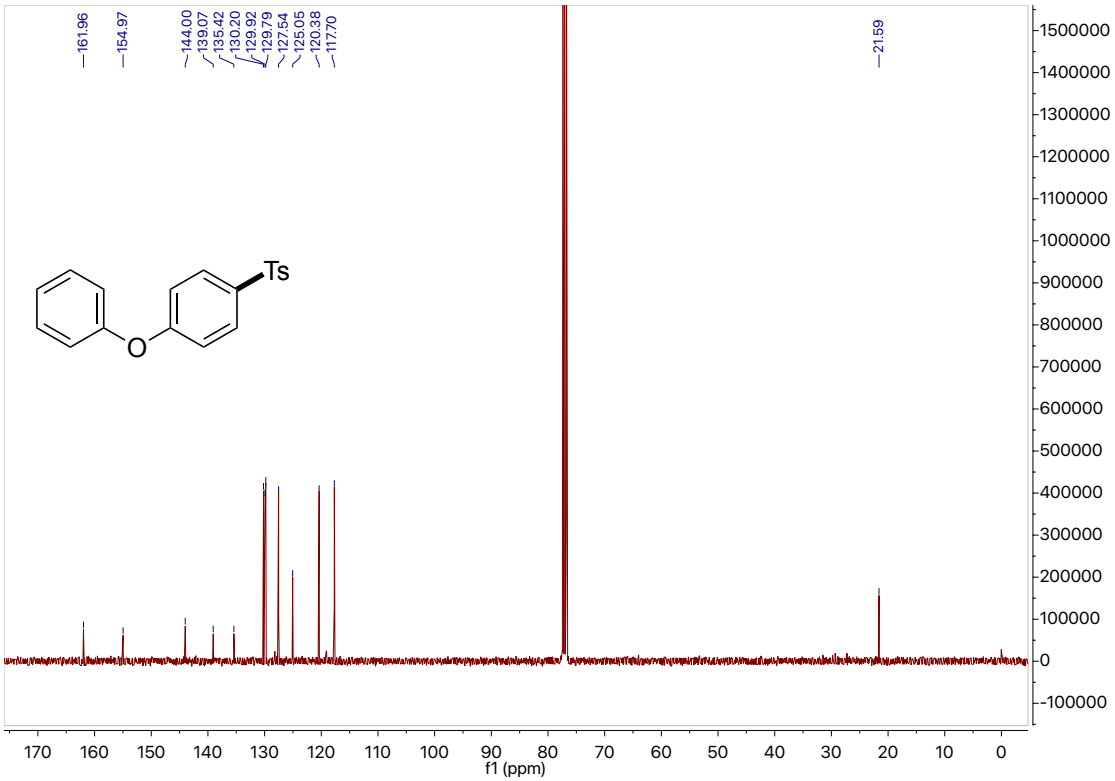


(3g)

^{1}H Spectra

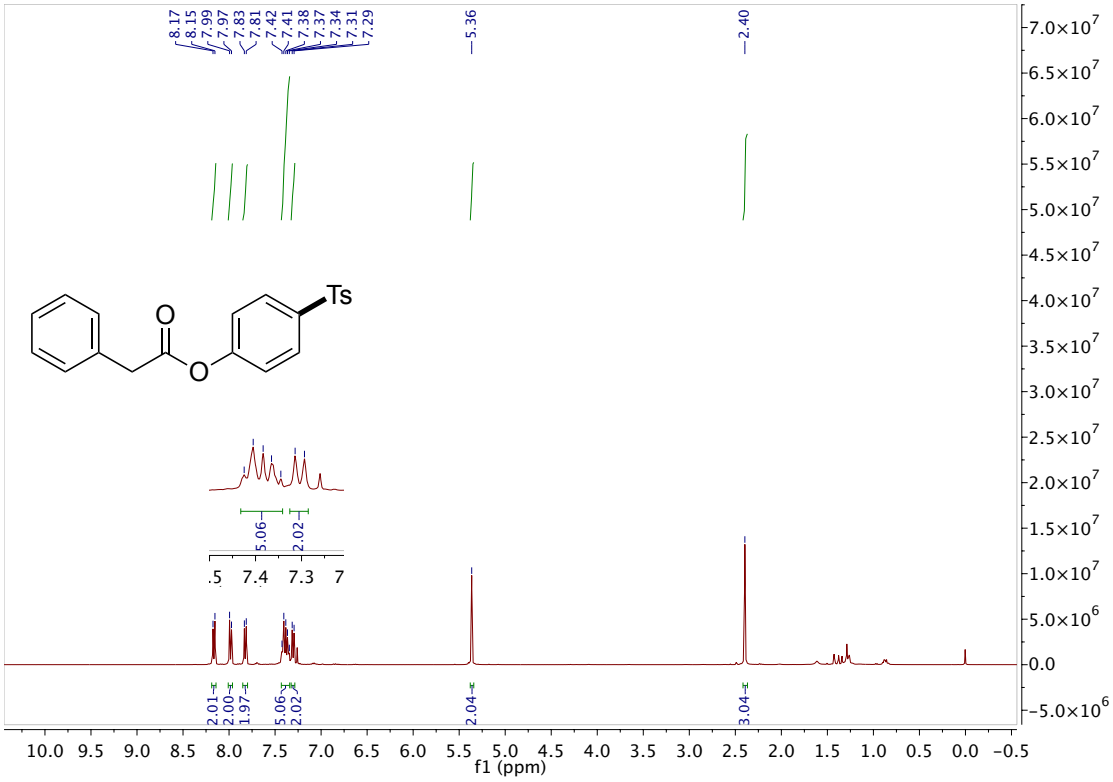


^{13}C Spectra

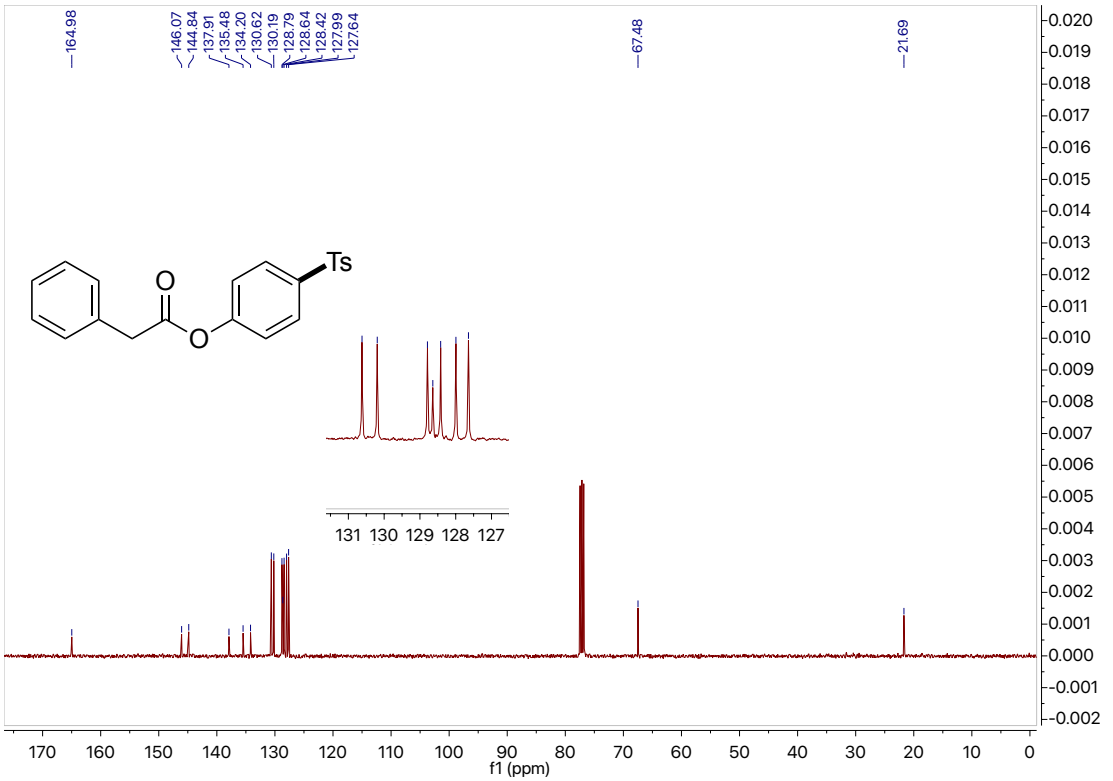


(3h)

^{1}H Spectra

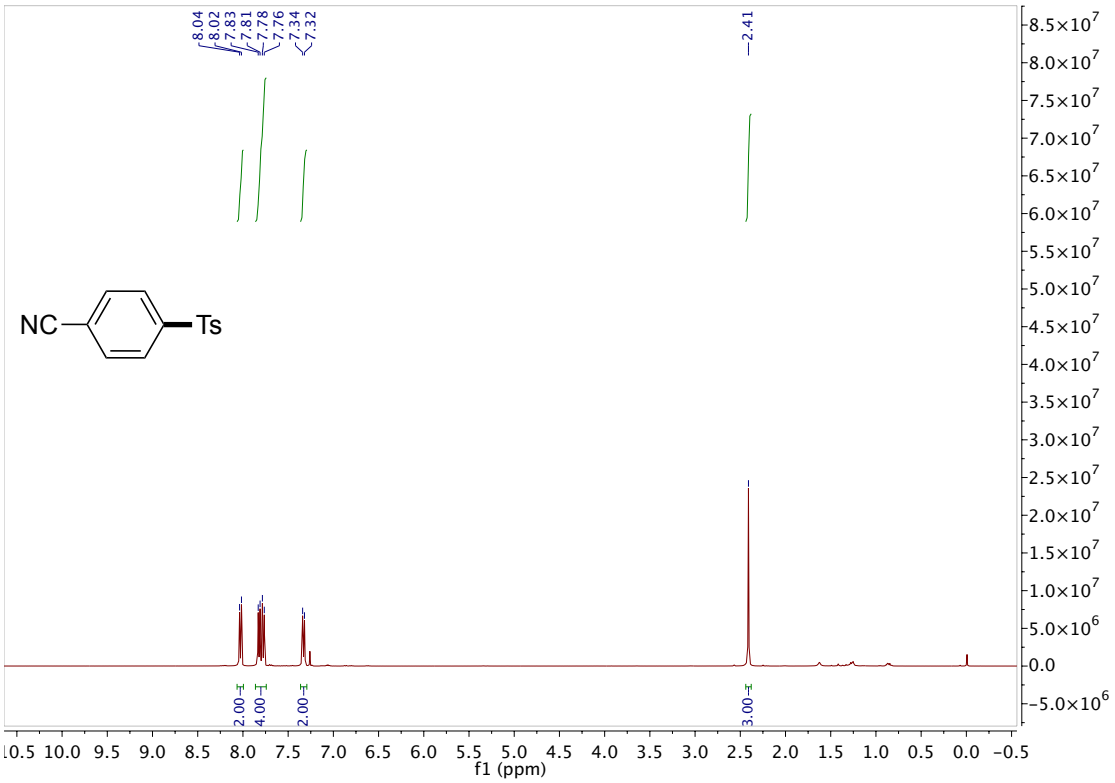


^{13}C Spectra

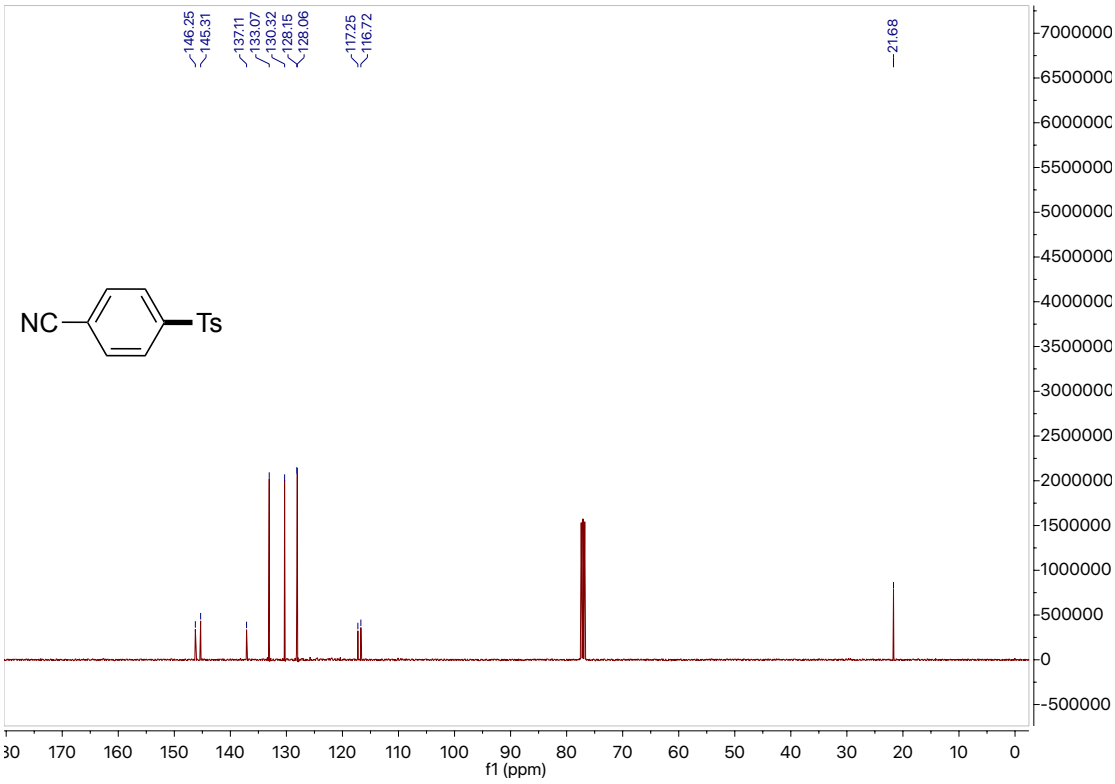


(3i)

^{1}H Spectra

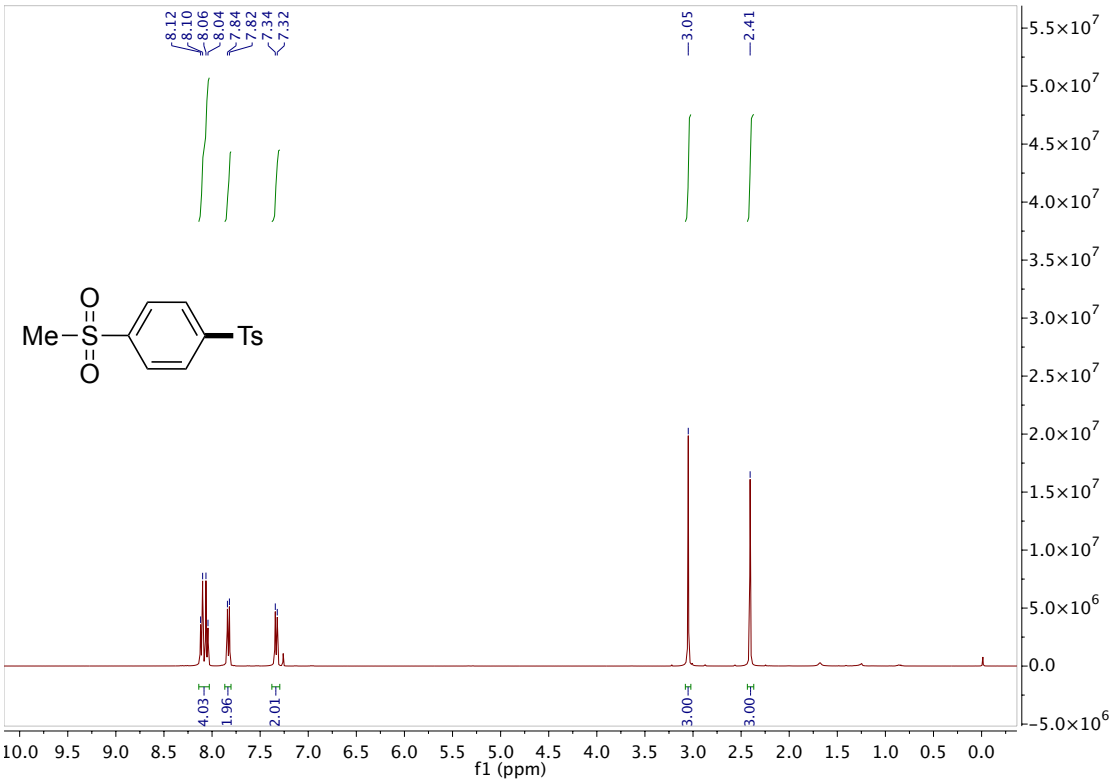


^{13}C Spectra

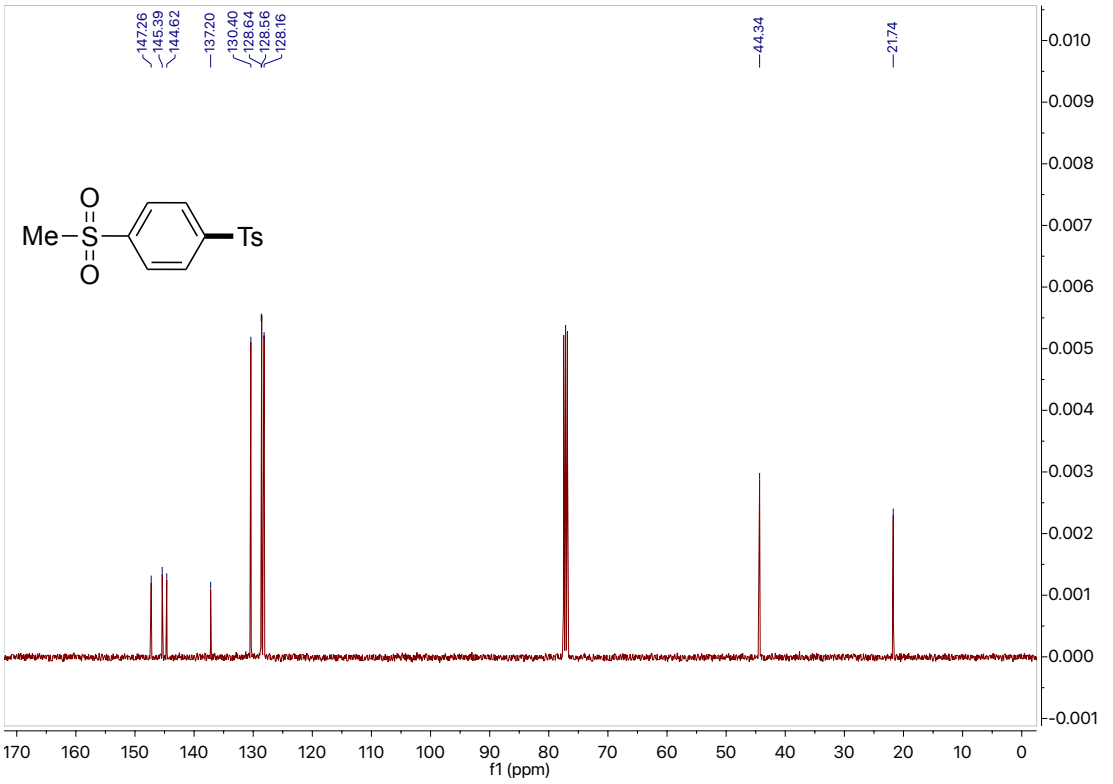


(3j)

^{1}H Spectra

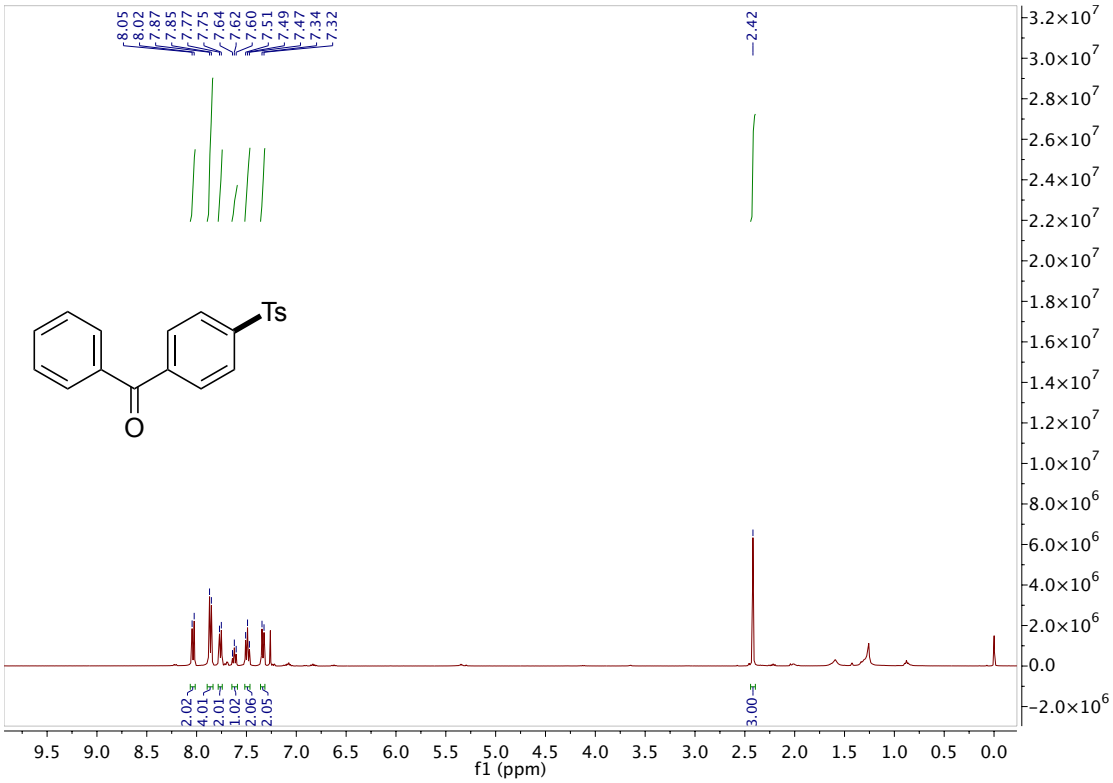


^{13}C Spectra



(3k)

^{1}H Spectra



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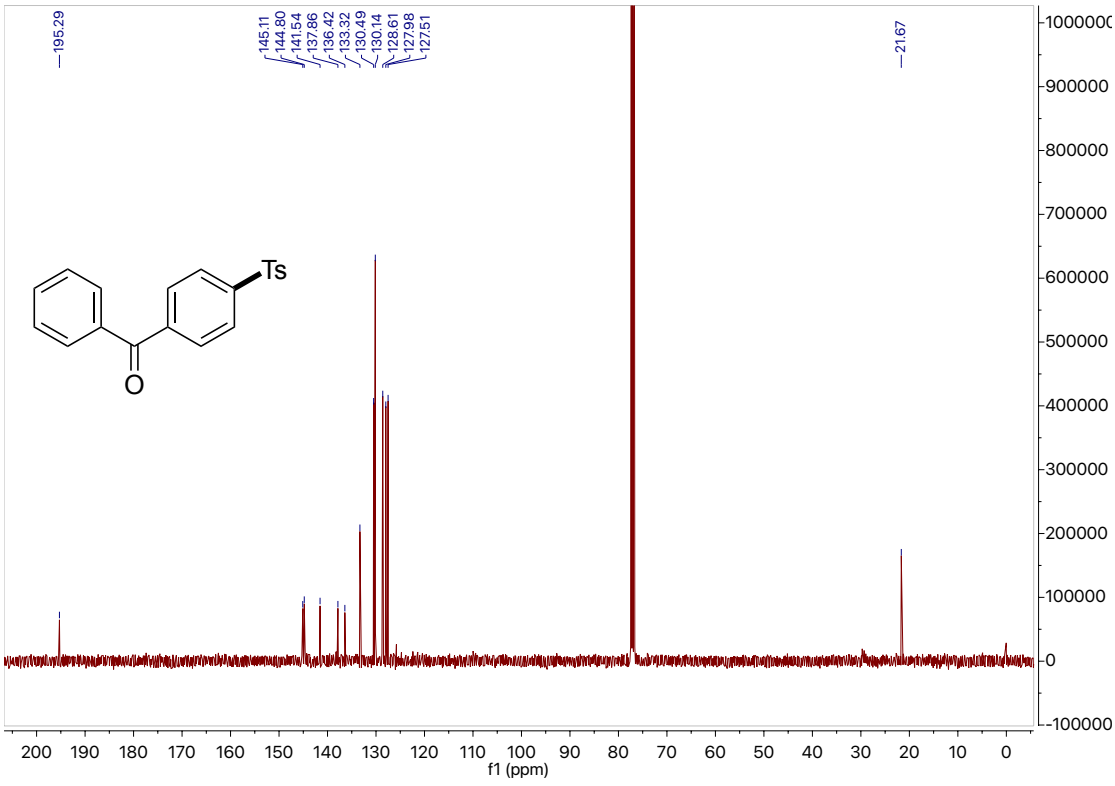
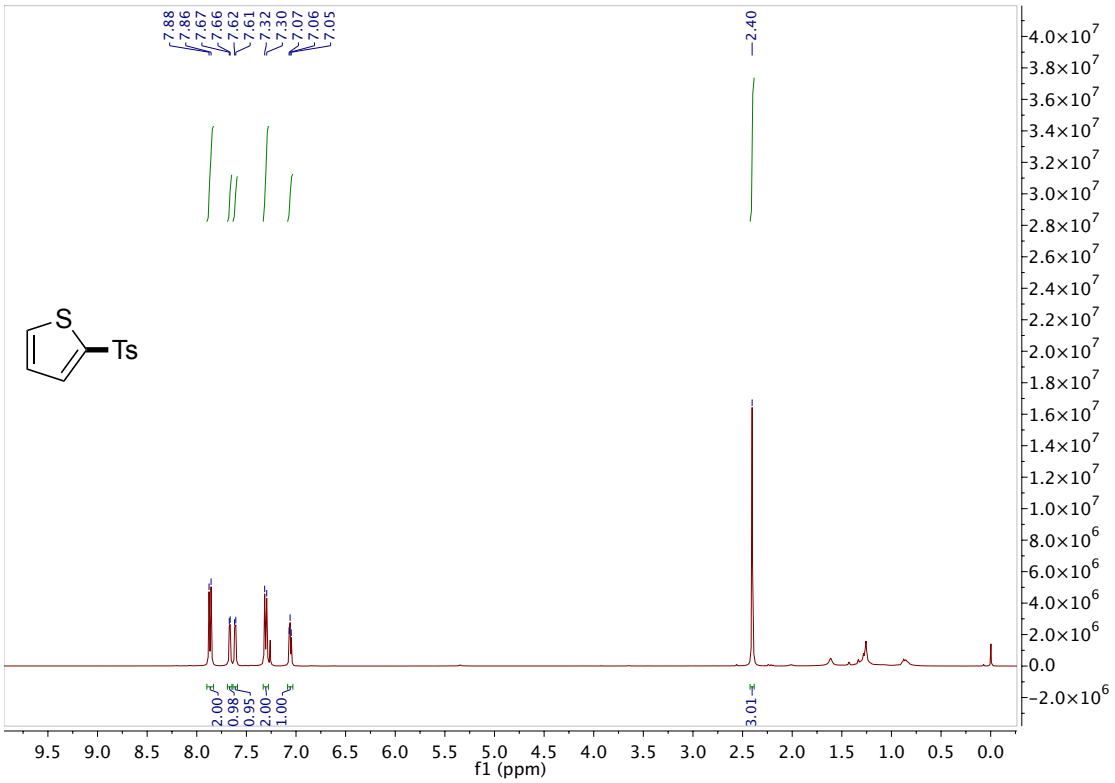
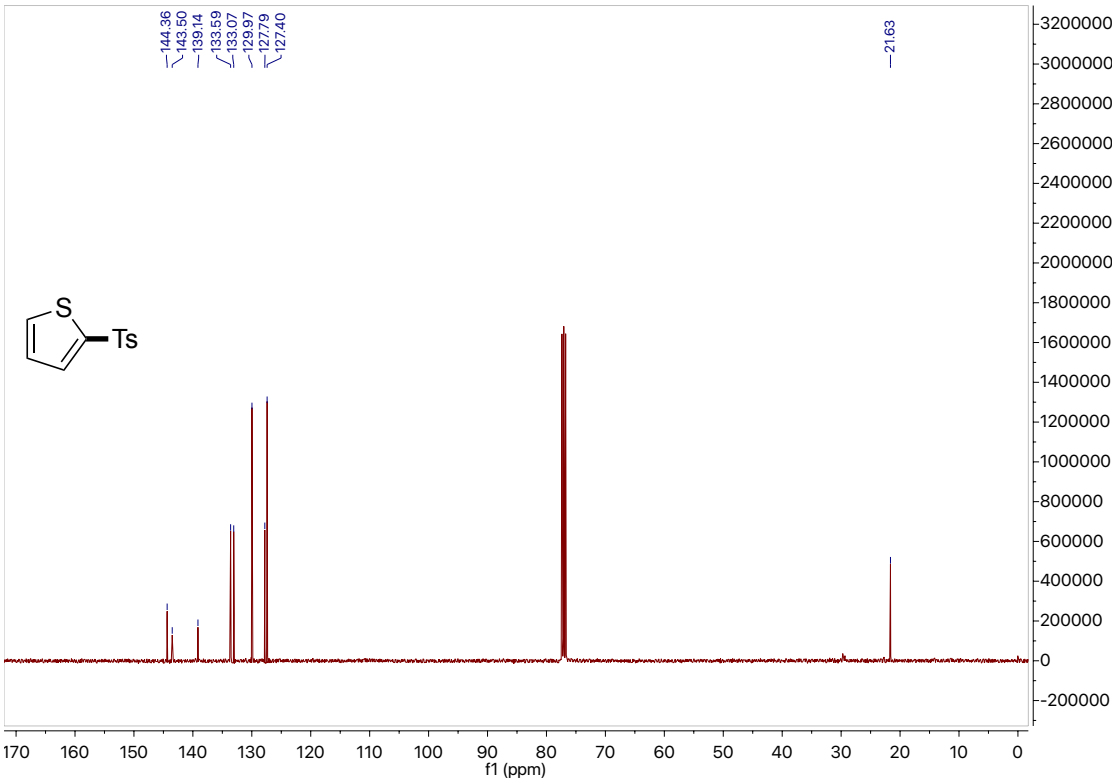


(3l)

^{1}H Spectra

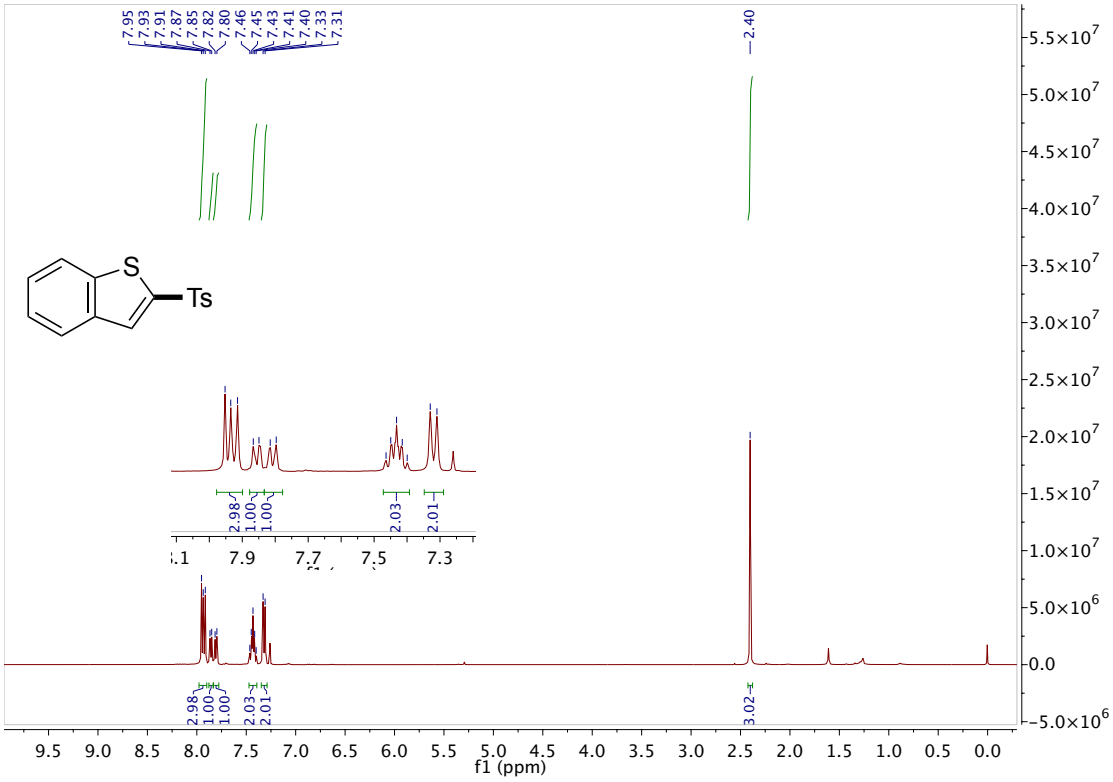


^{13}C Spectra

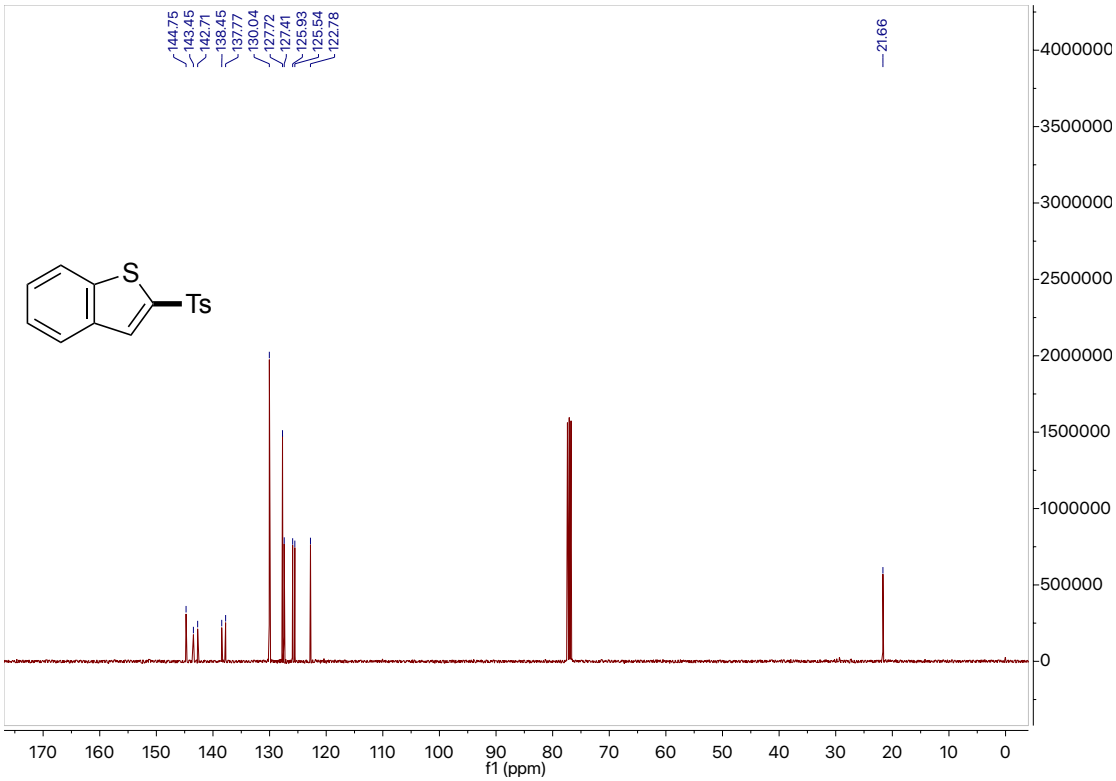


(3m)

^{1}H Spectra

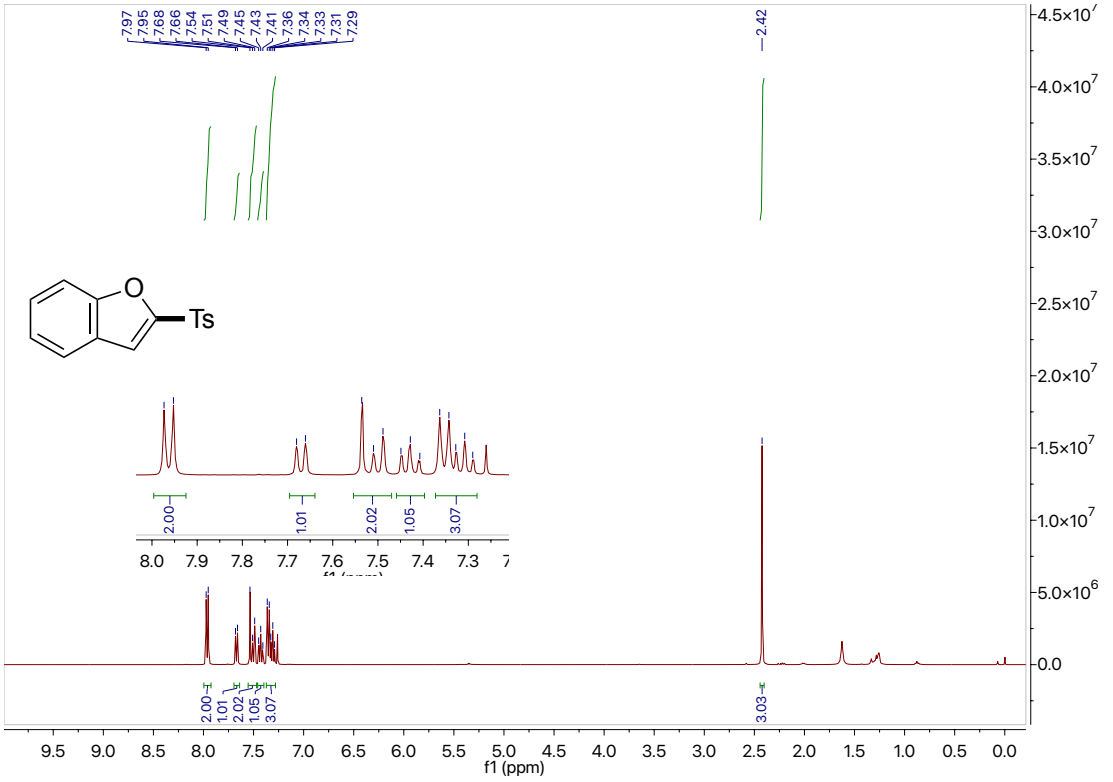


^{13}C Spectra



(3n)

^{1}H Spectra



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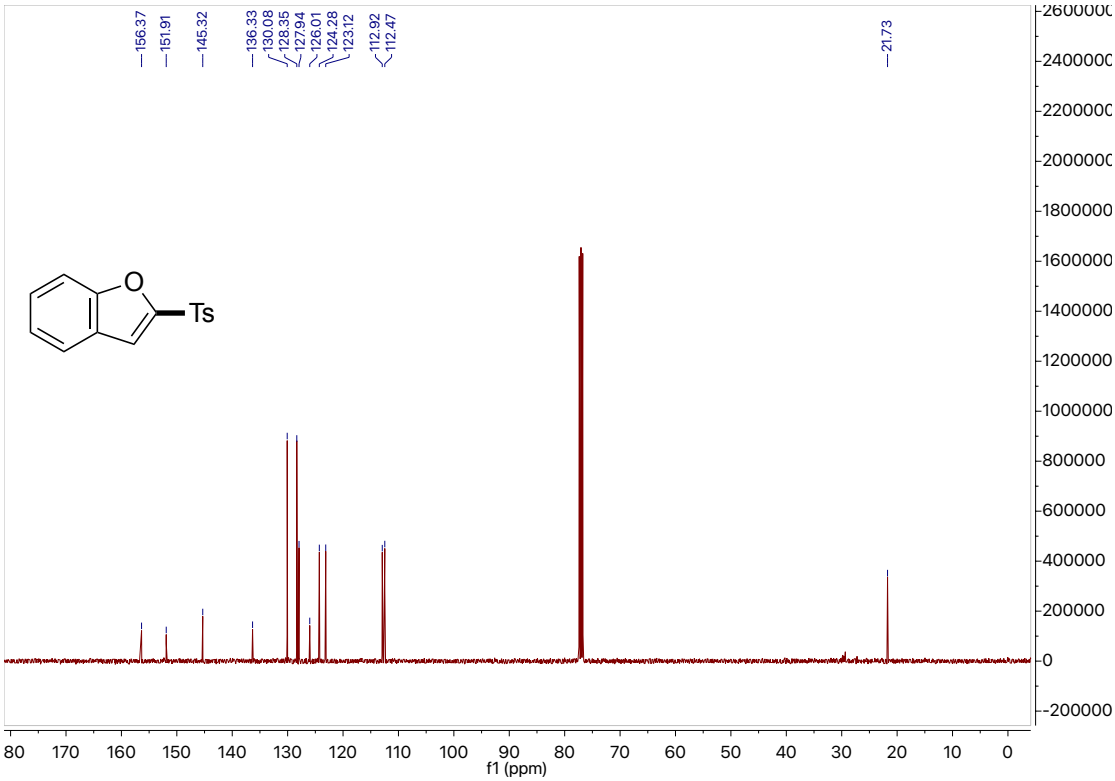
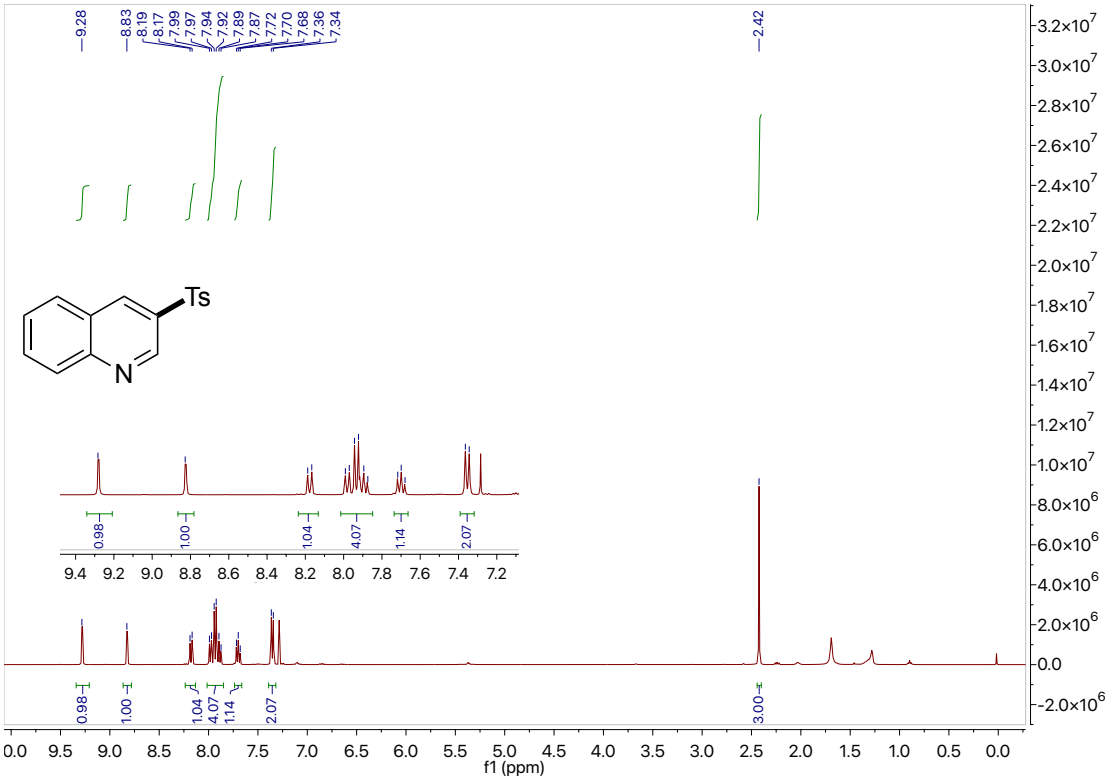
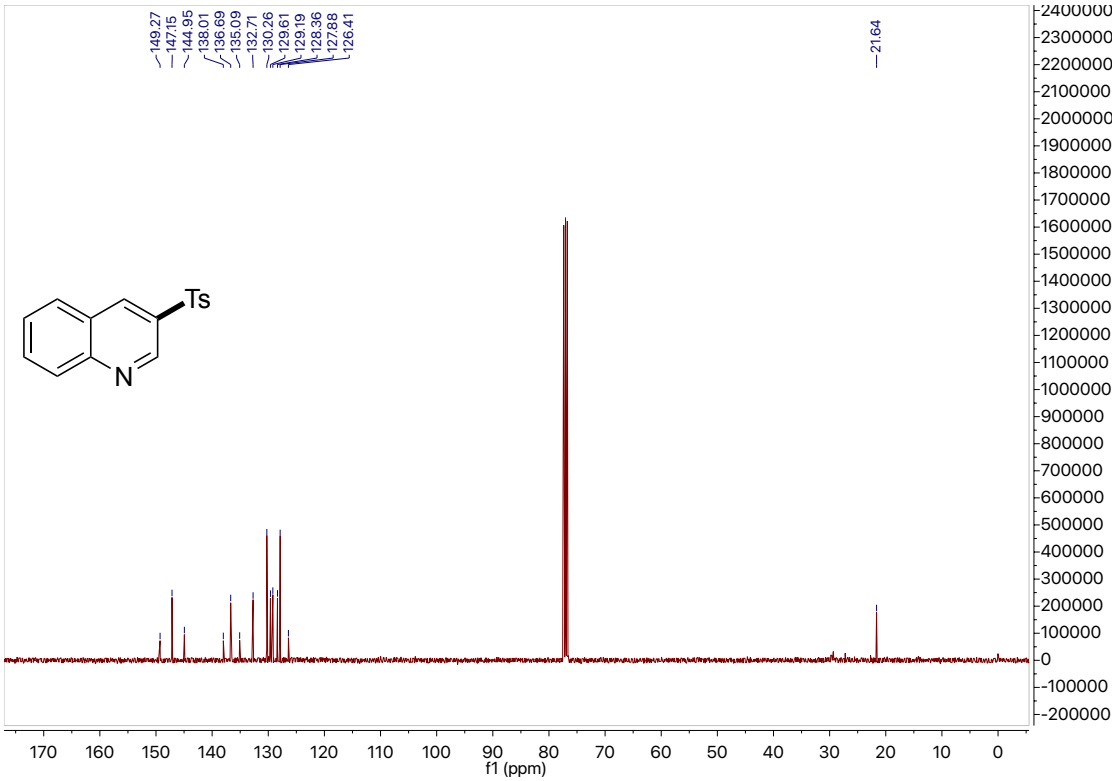


(3o)

^{1}H Spectra



^{13}C Spectra



(3p)

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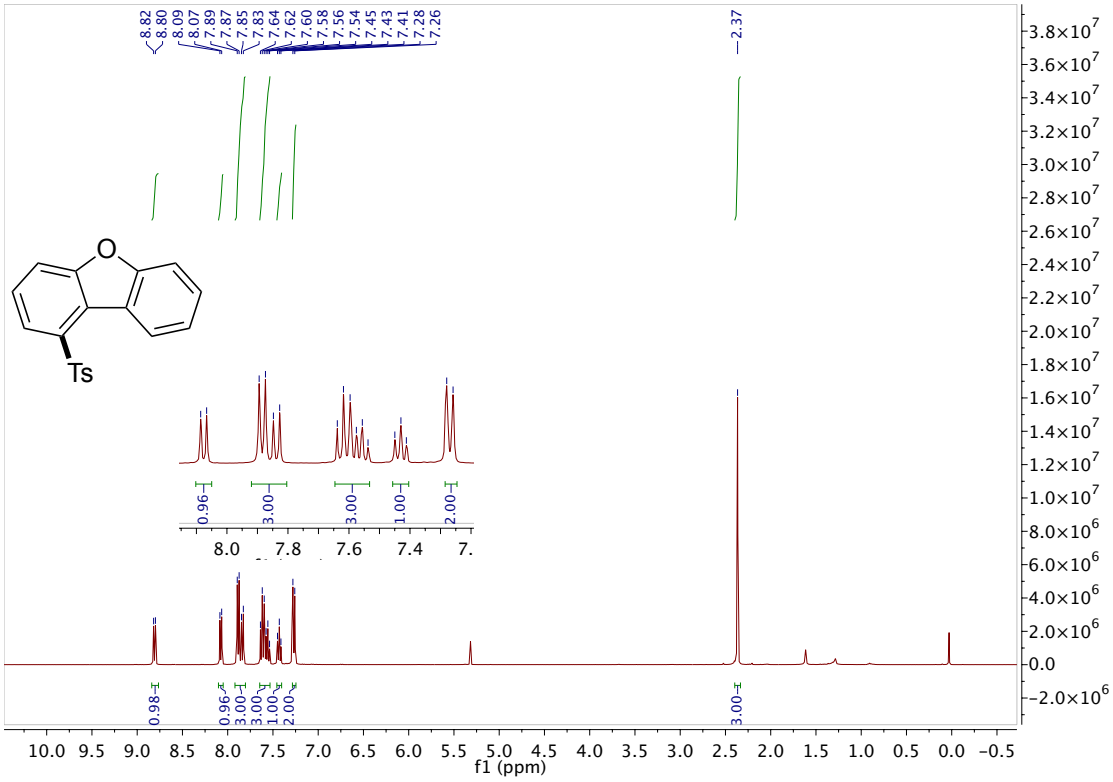

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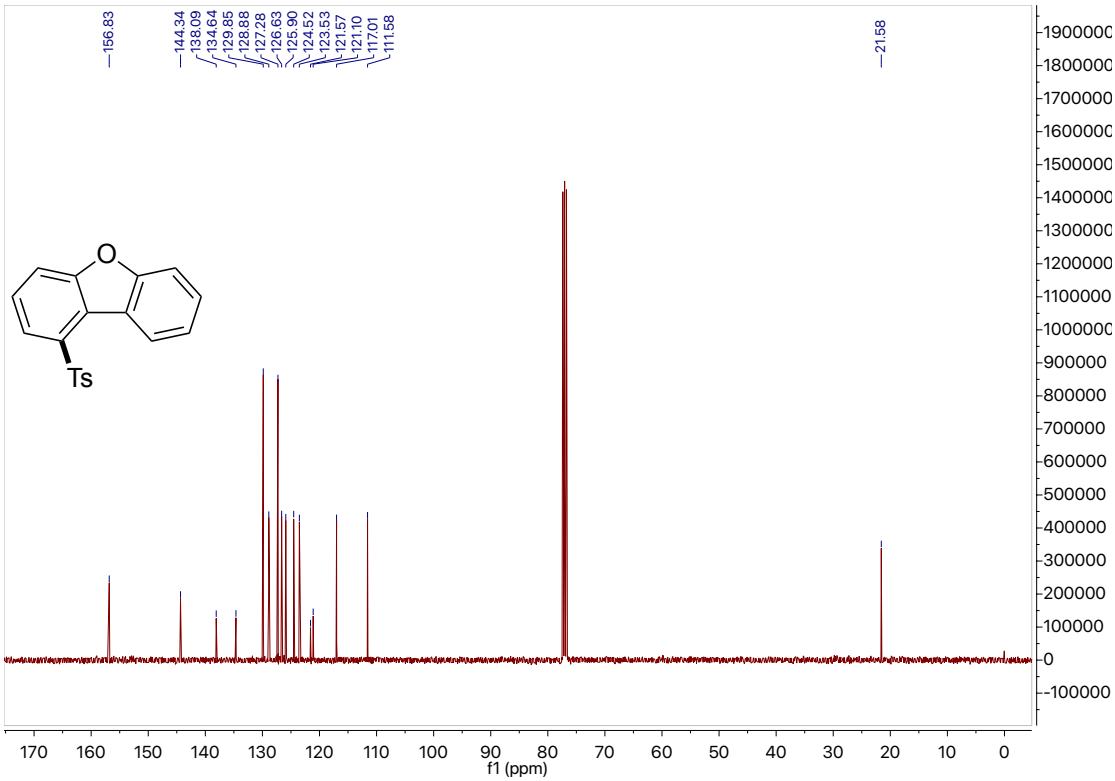

(3q)

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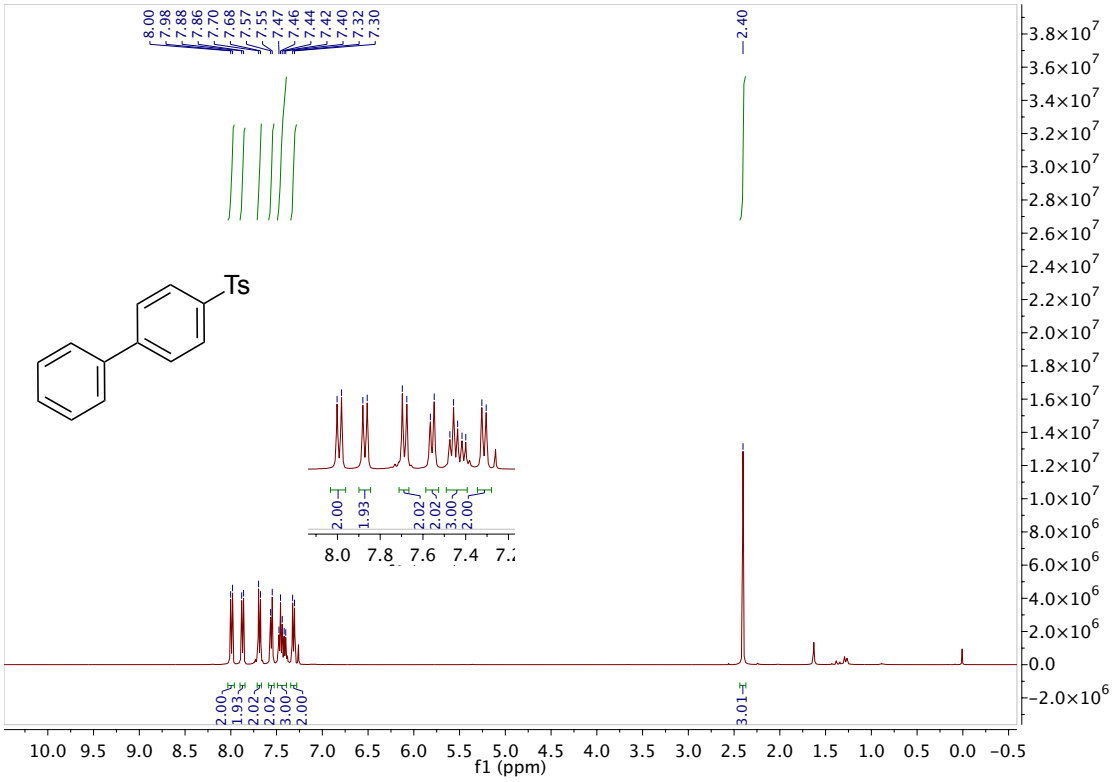

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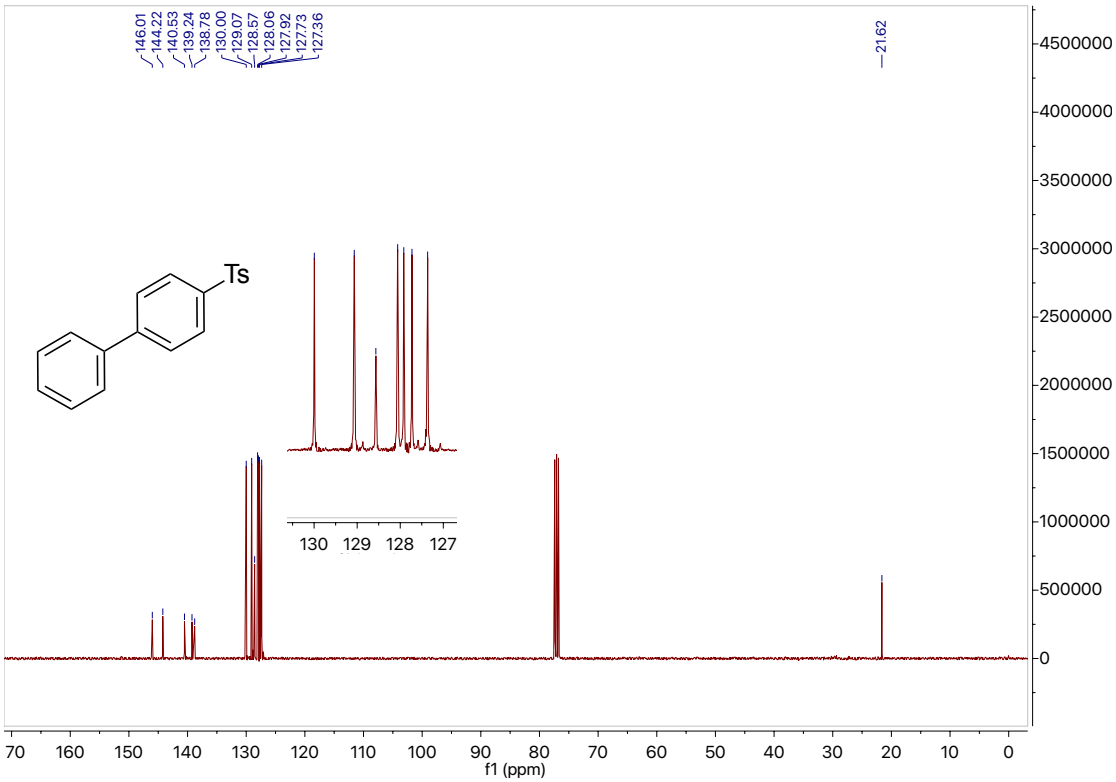
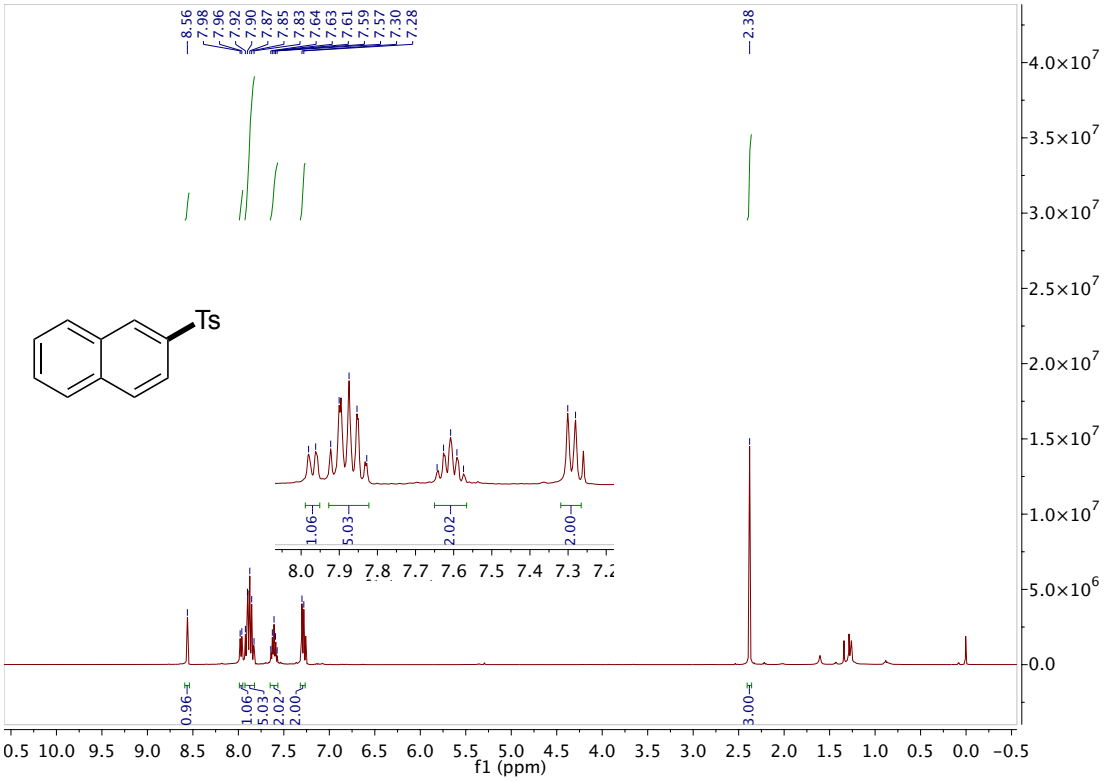


(3r)

^{1}H Spectra



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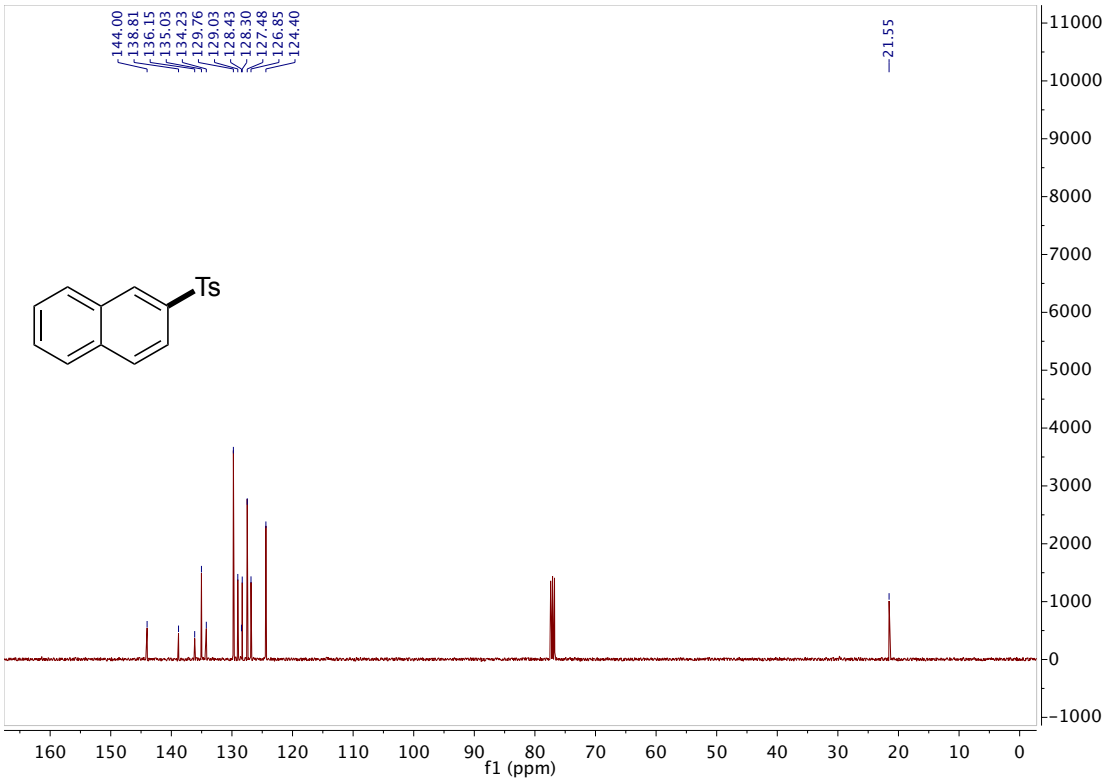

(3s)

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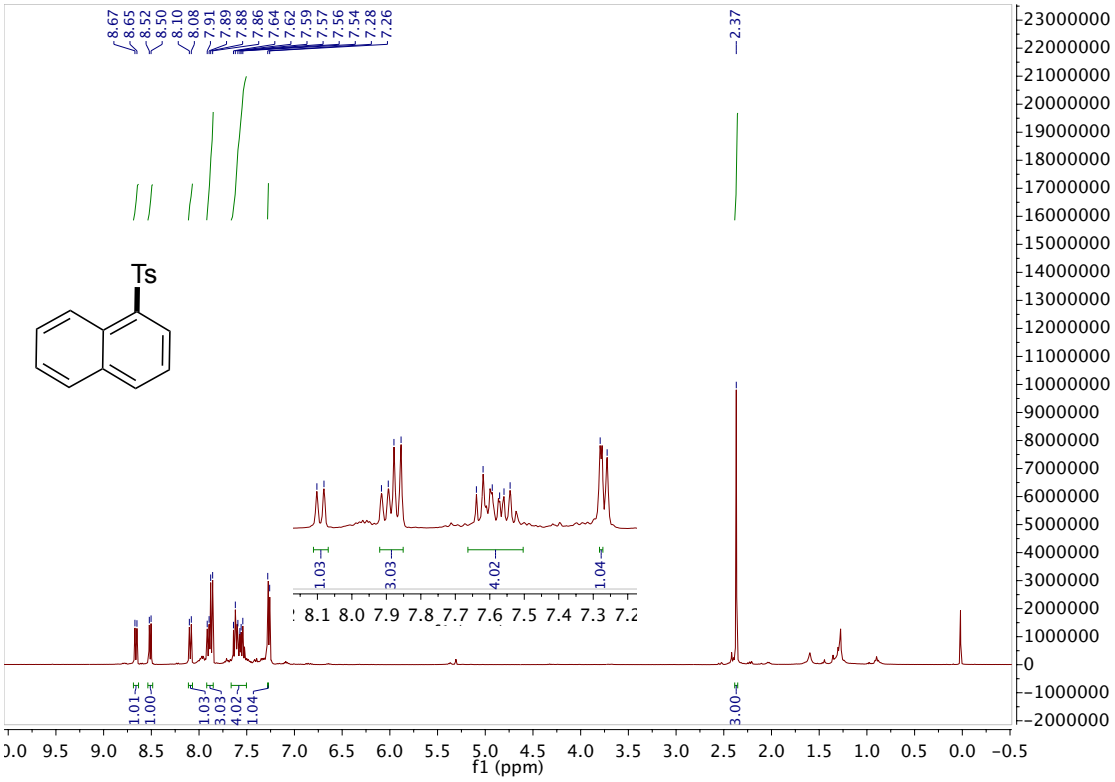

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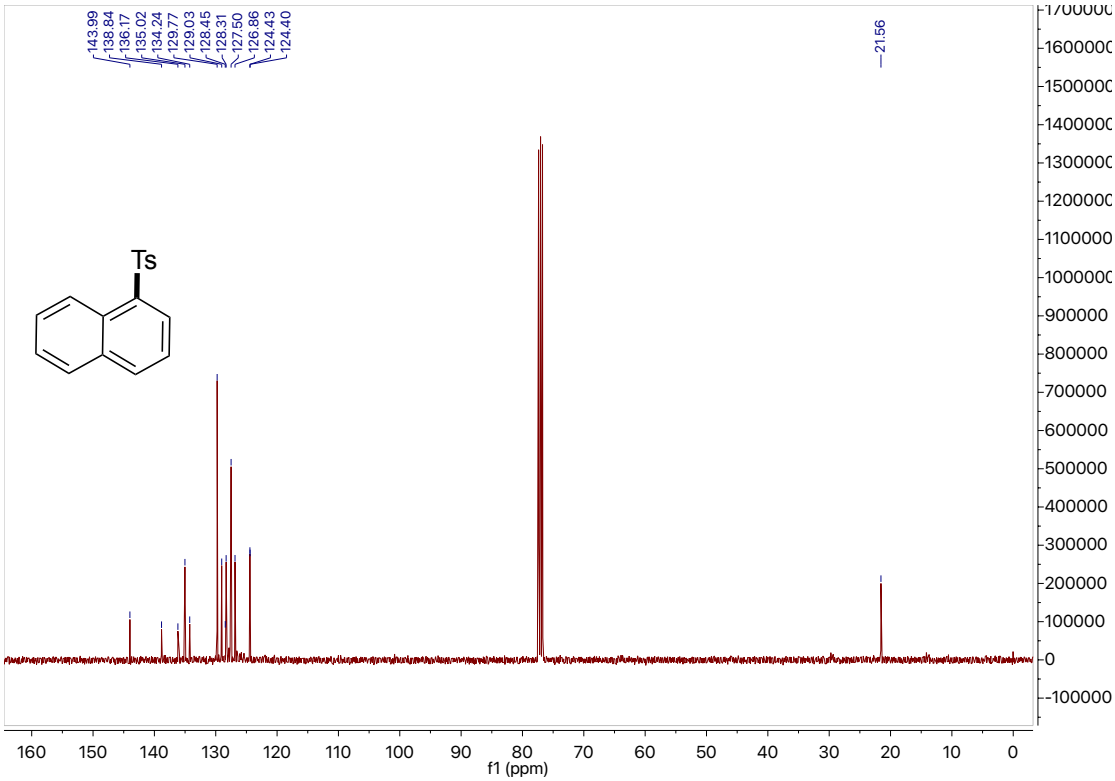
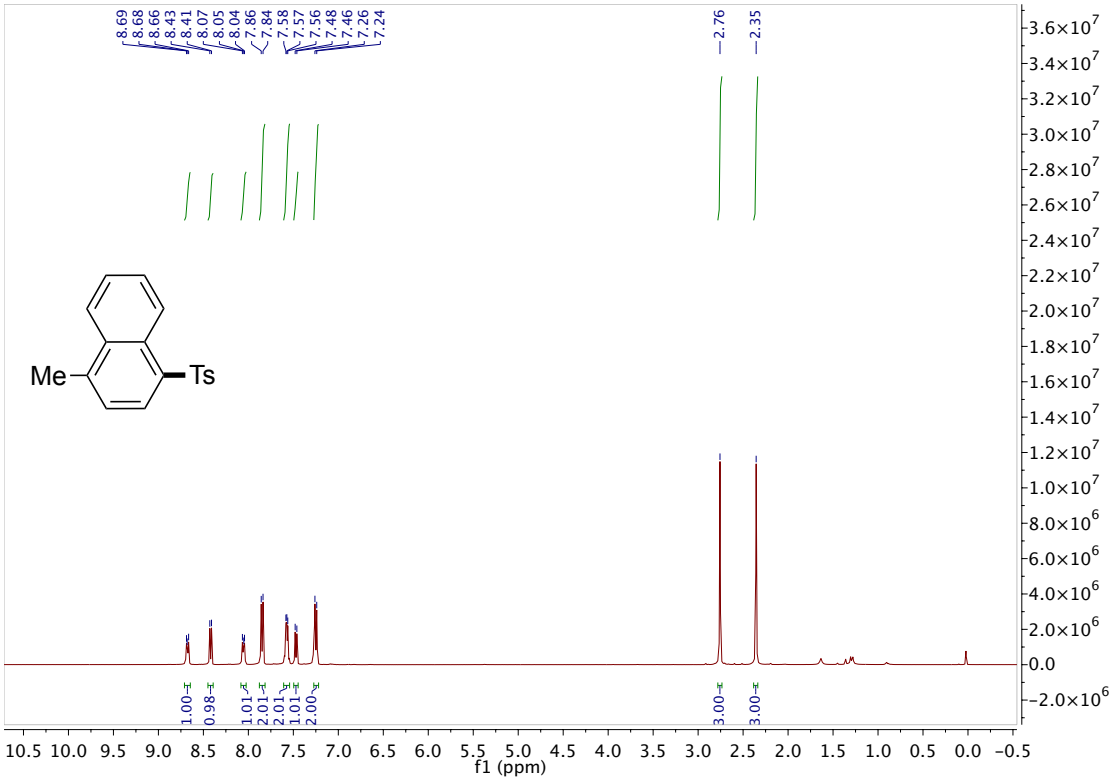


(3t)

^{1}H Spectra



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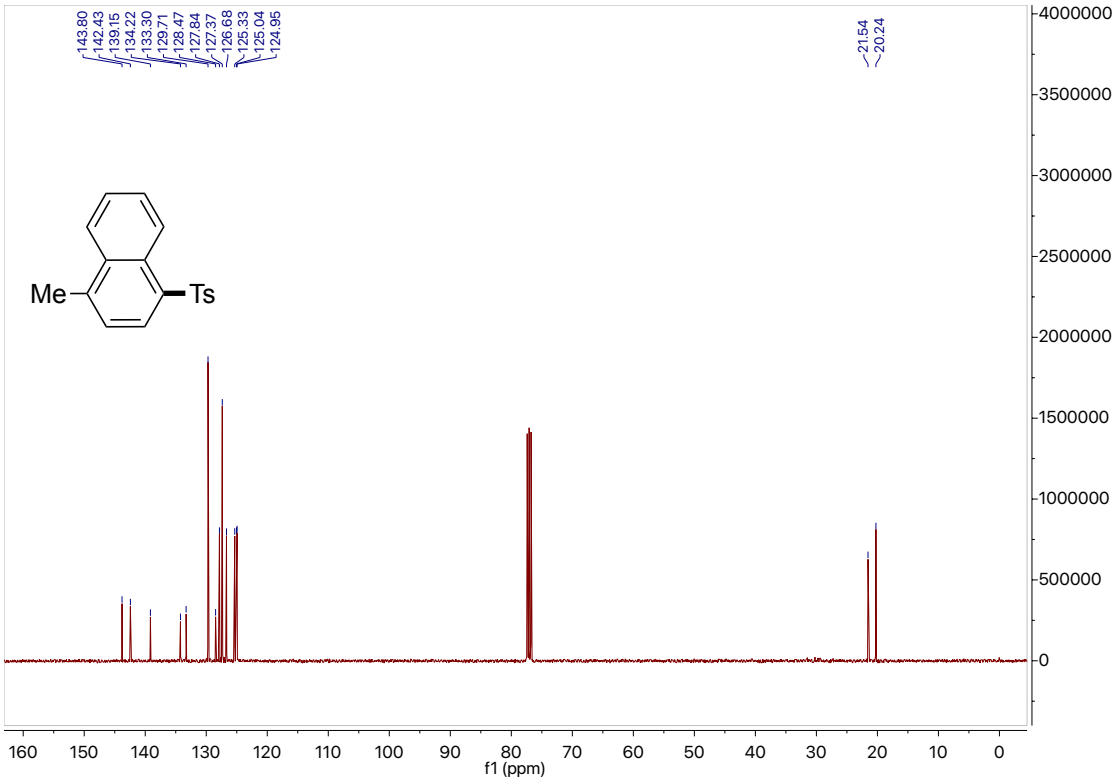
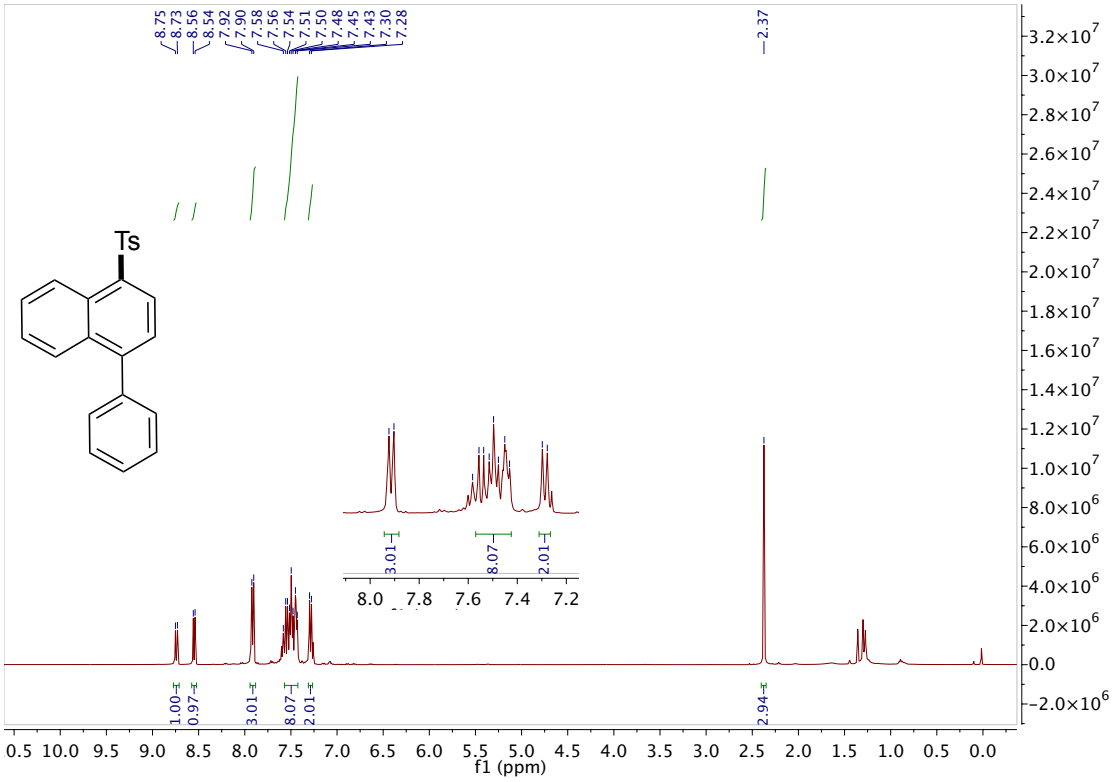


(3u)

^{1}H Spectra



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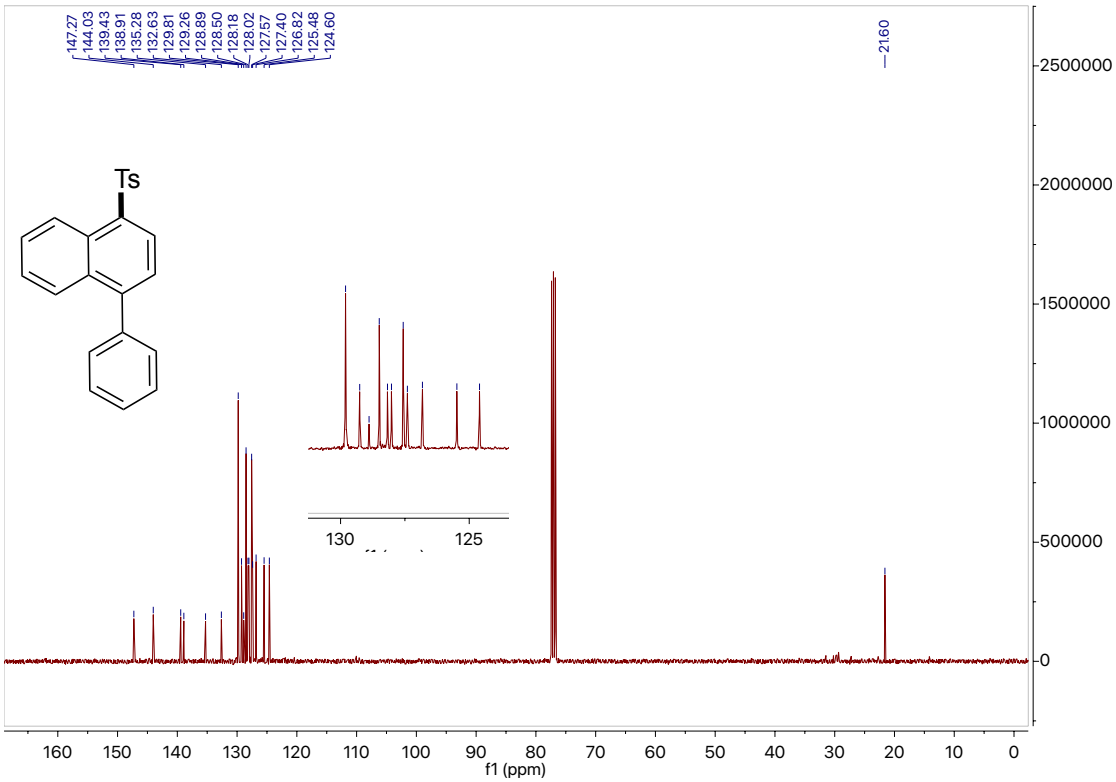

(3v)

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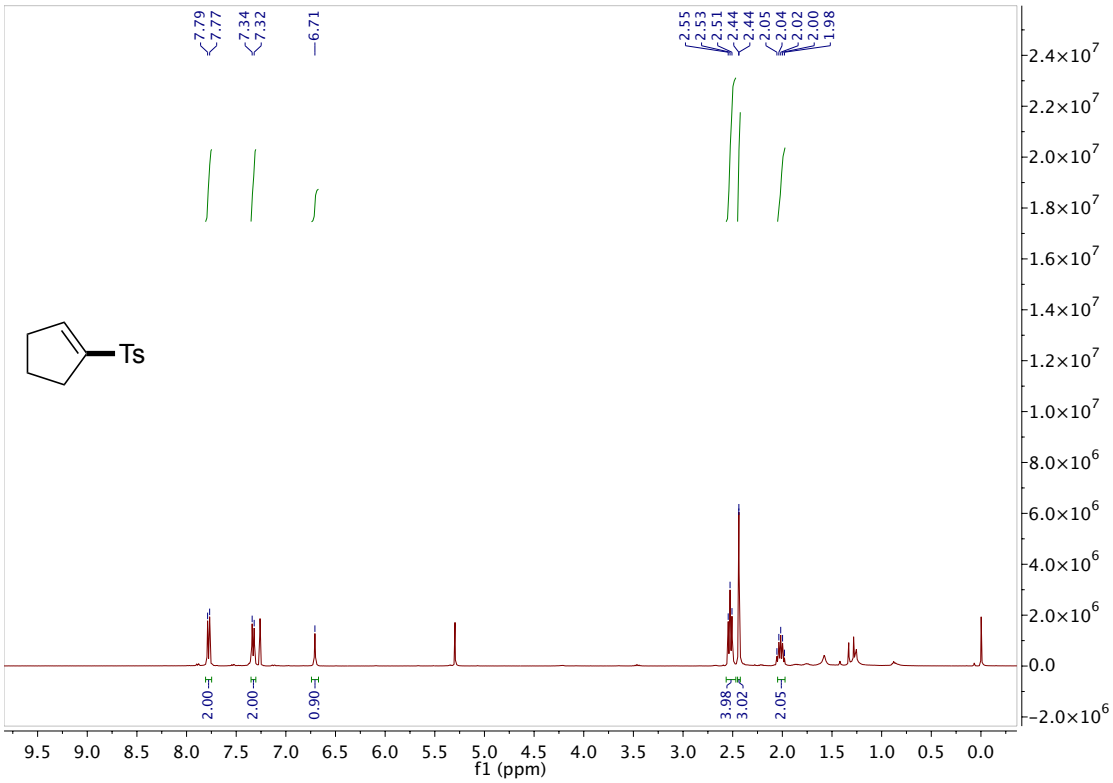

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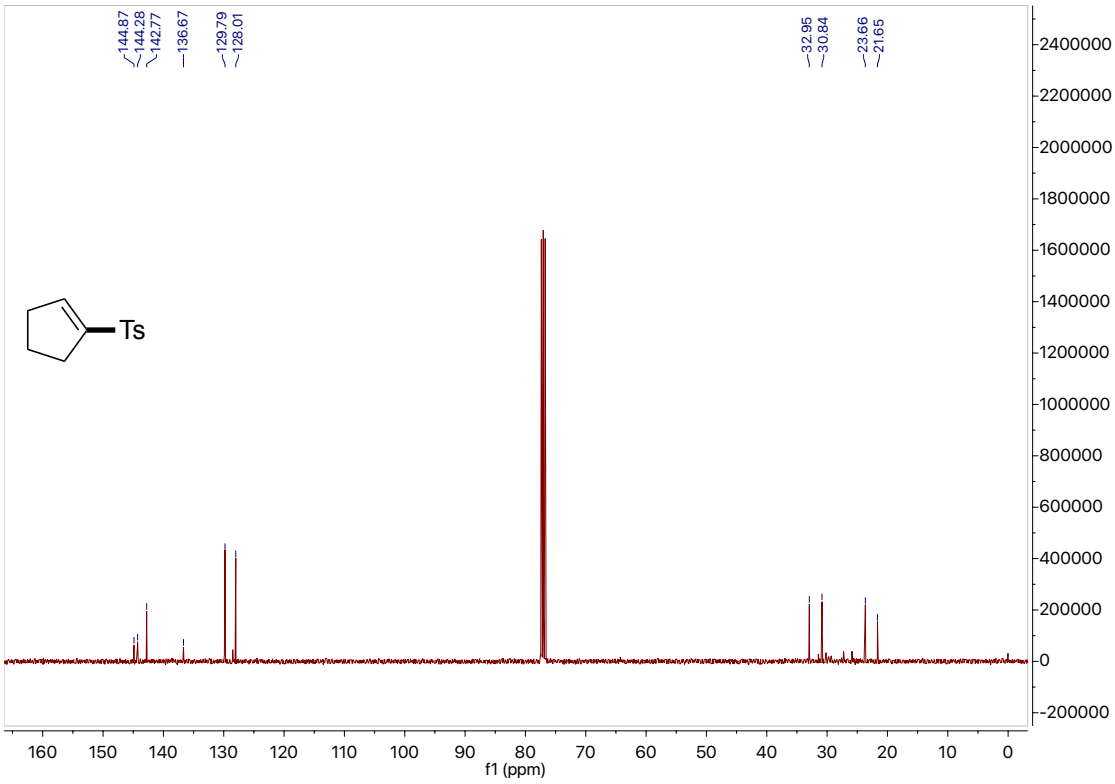

(3w)

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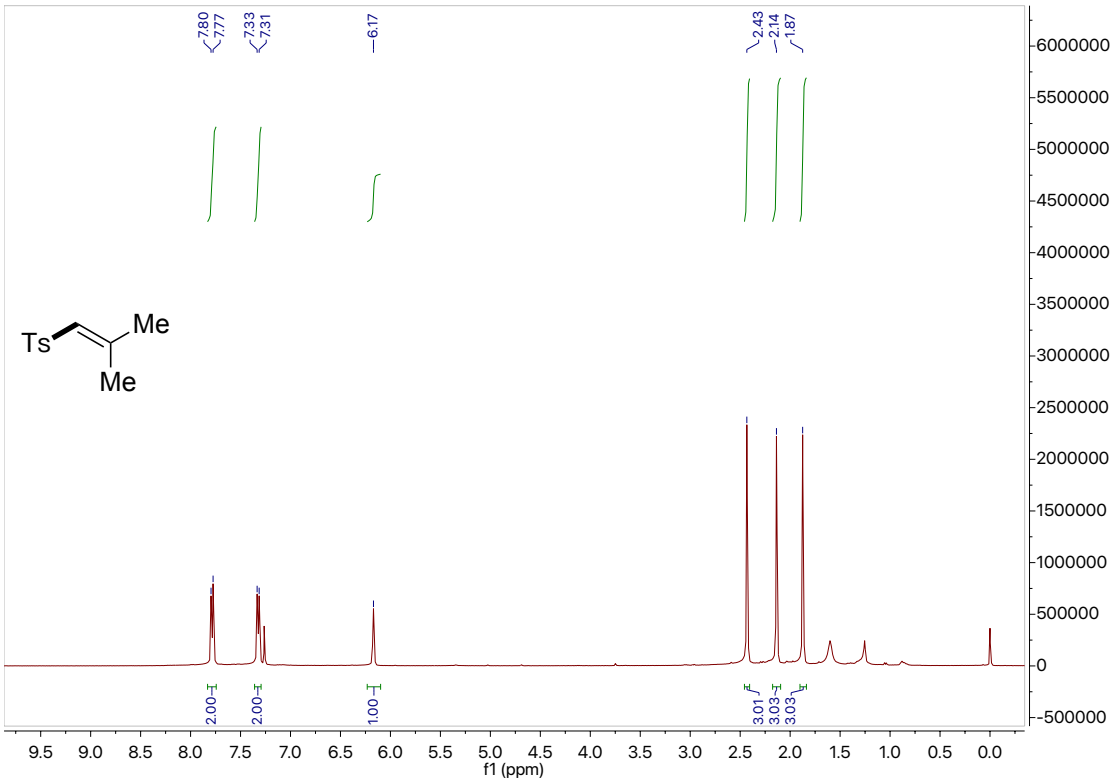

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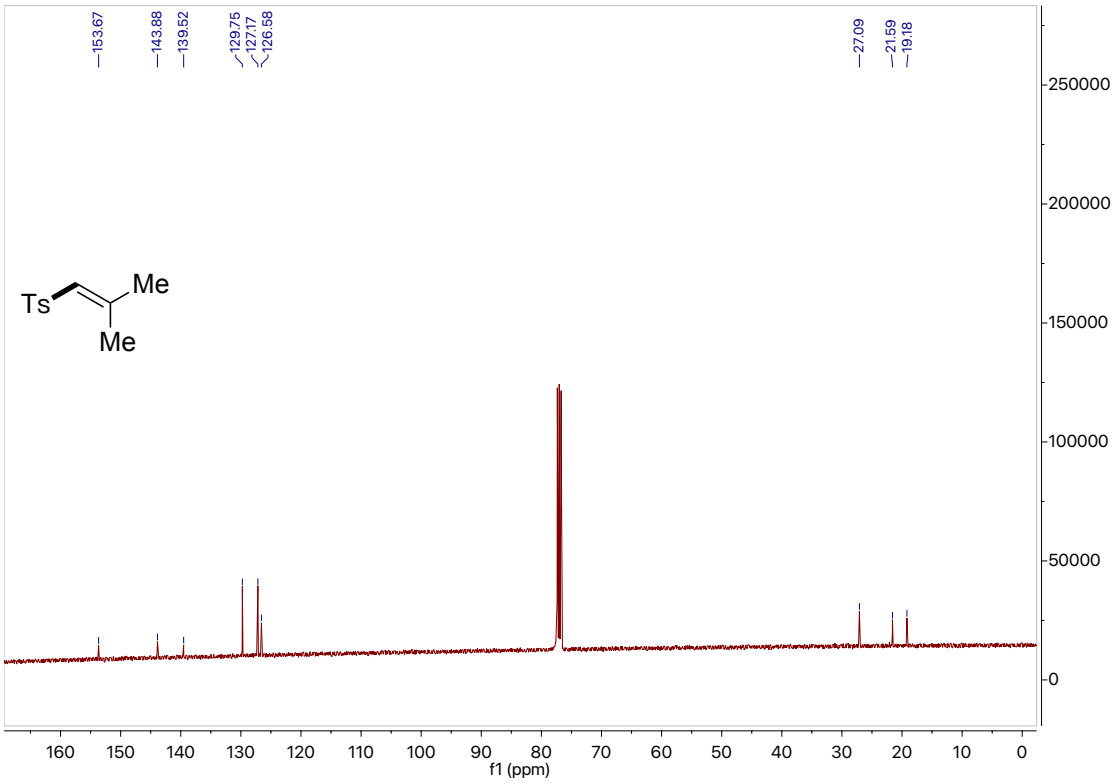

(3x)

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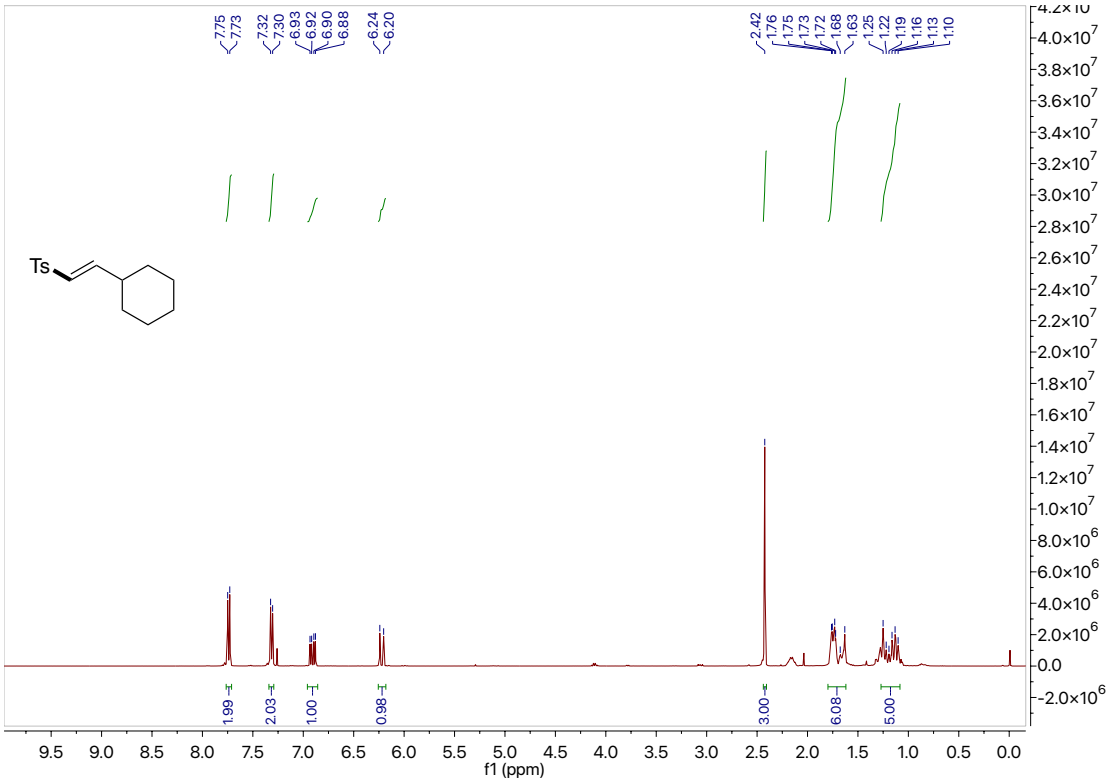

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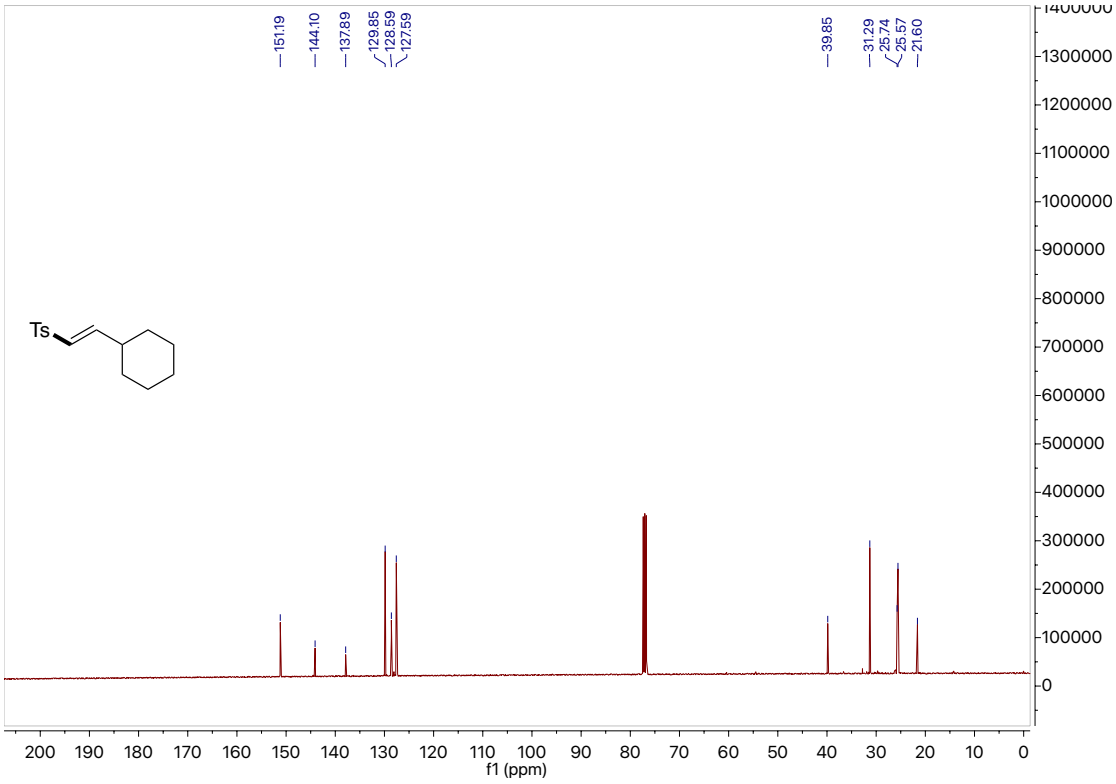

(4a)

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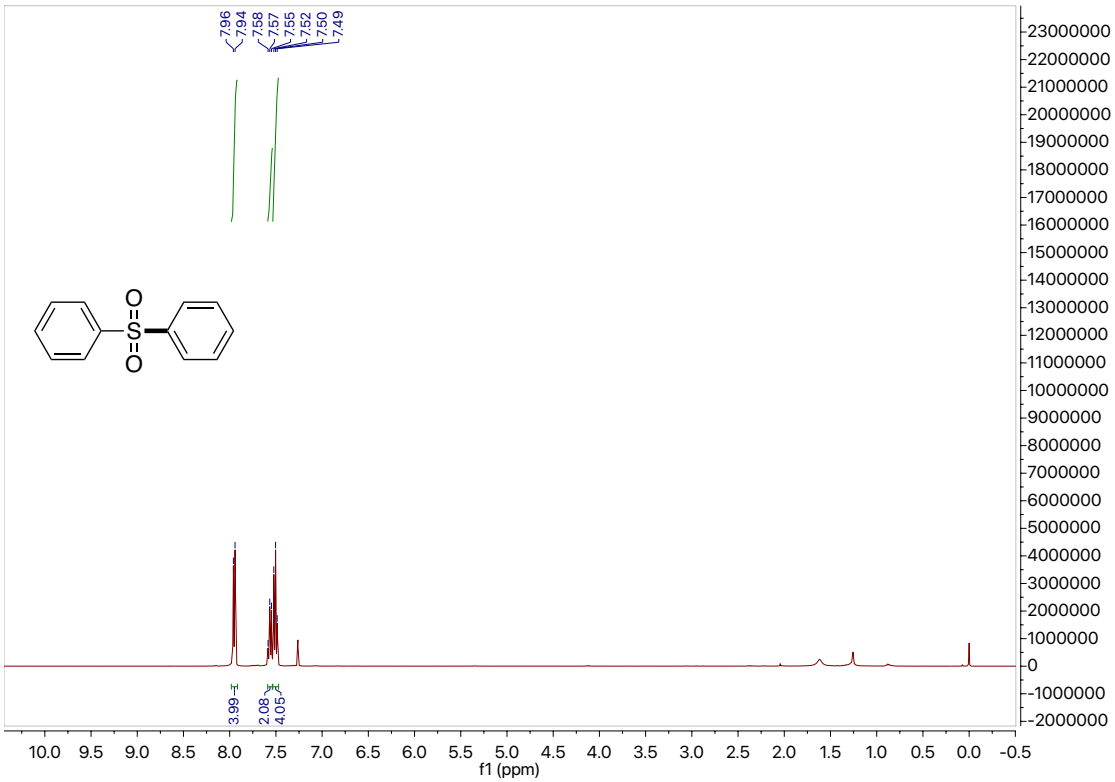

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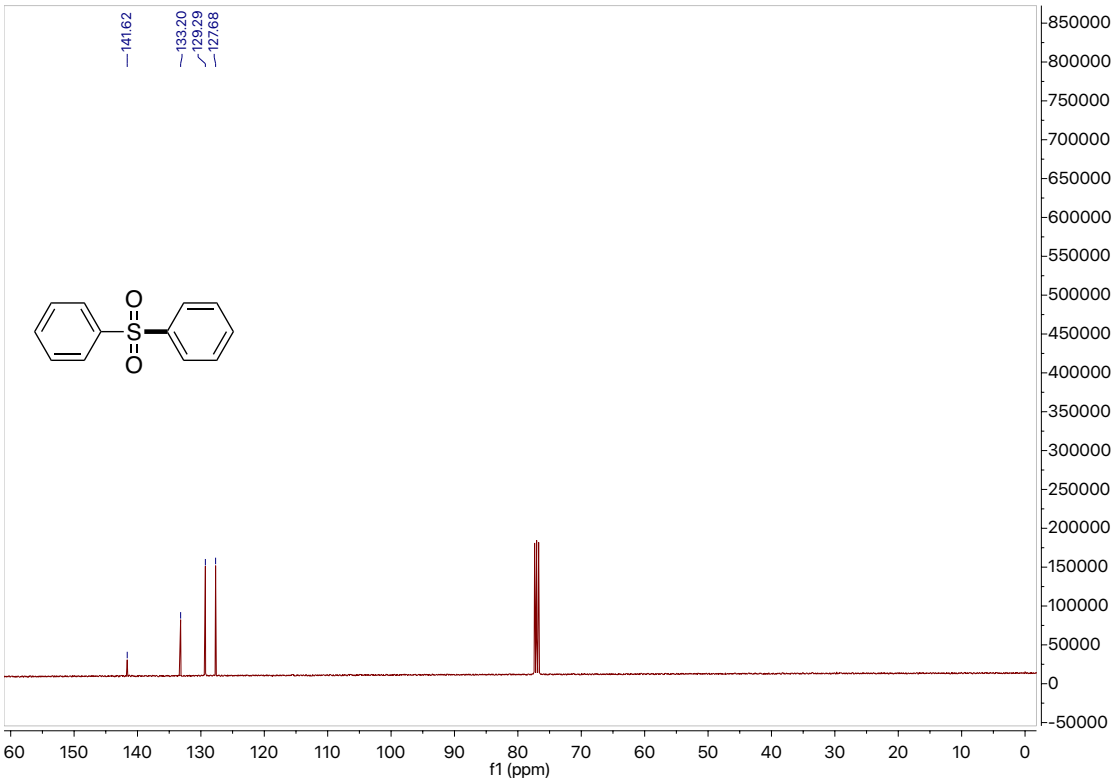
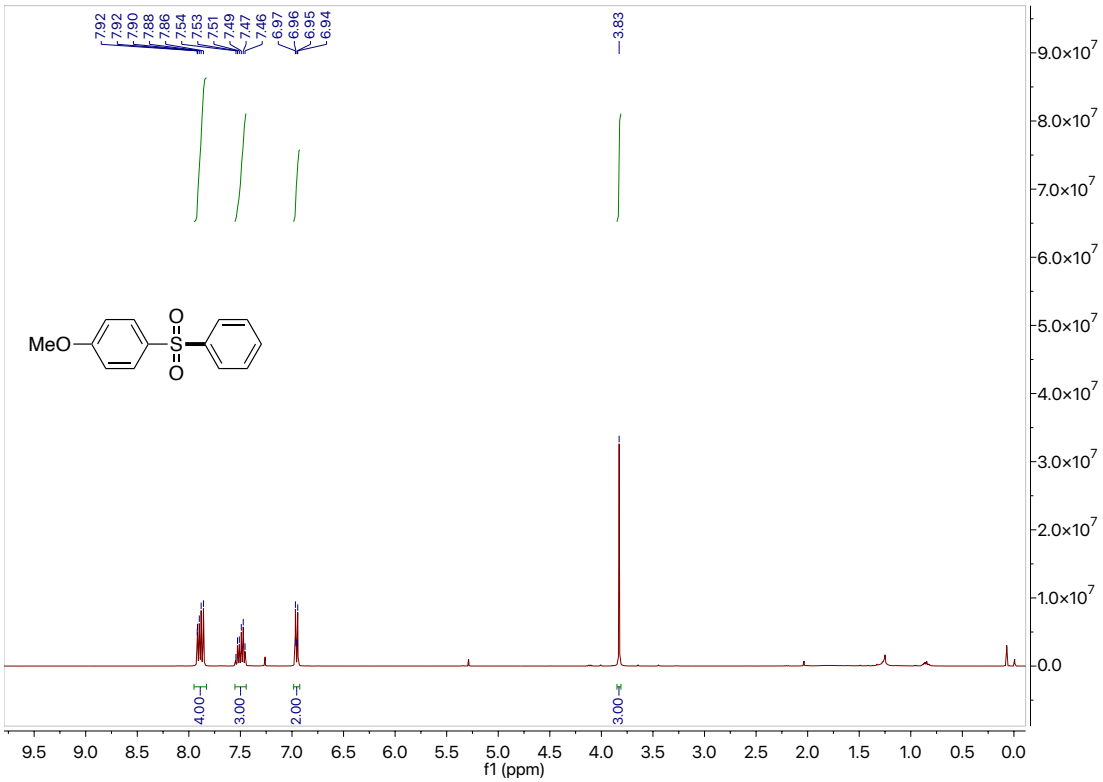


(4b)

^{1}H Spectra



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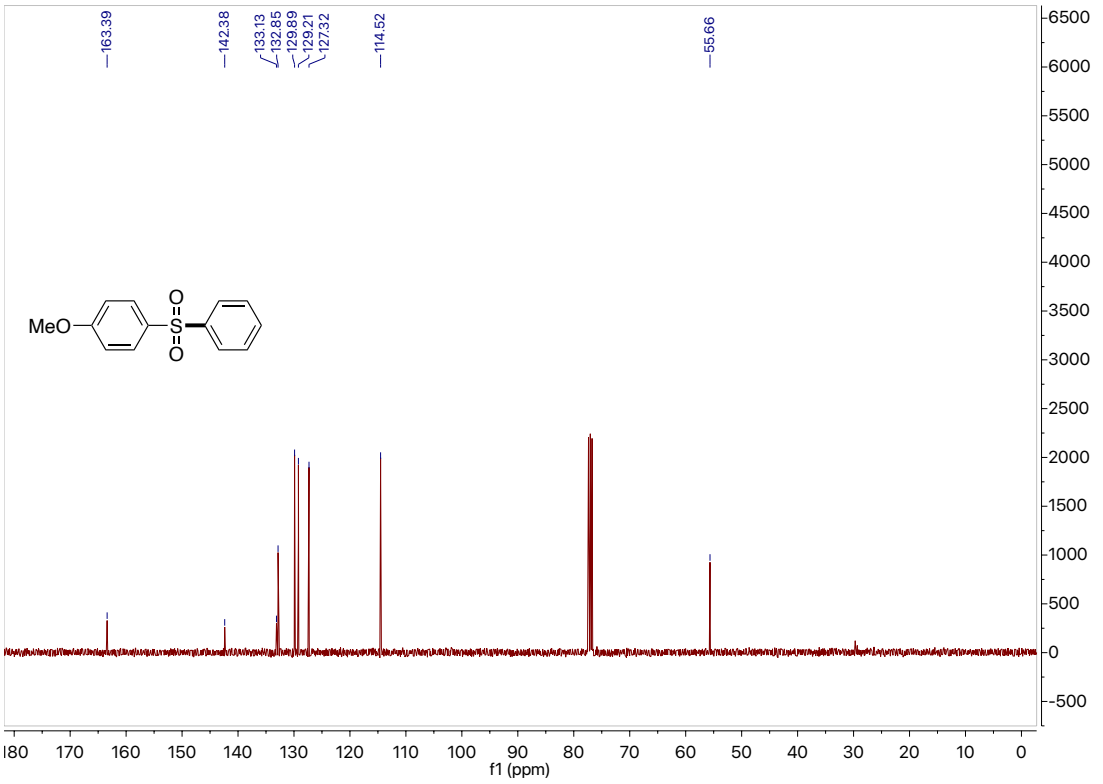
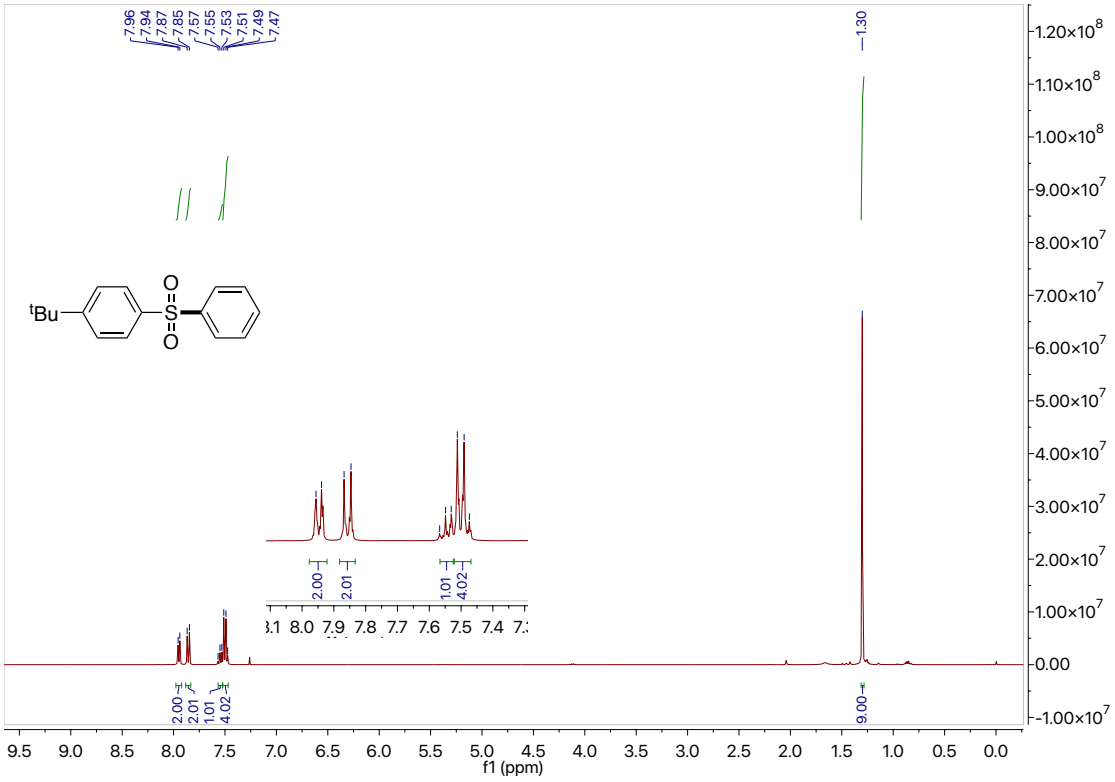
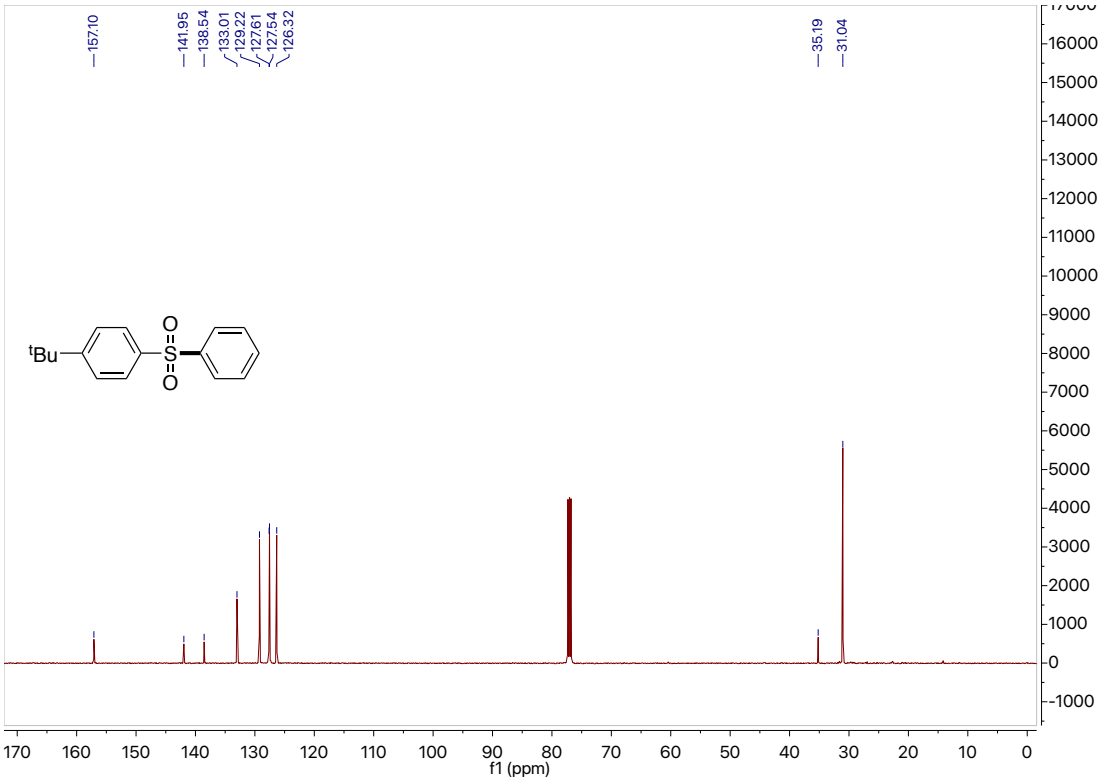


(4c)

^{1}H Spectra

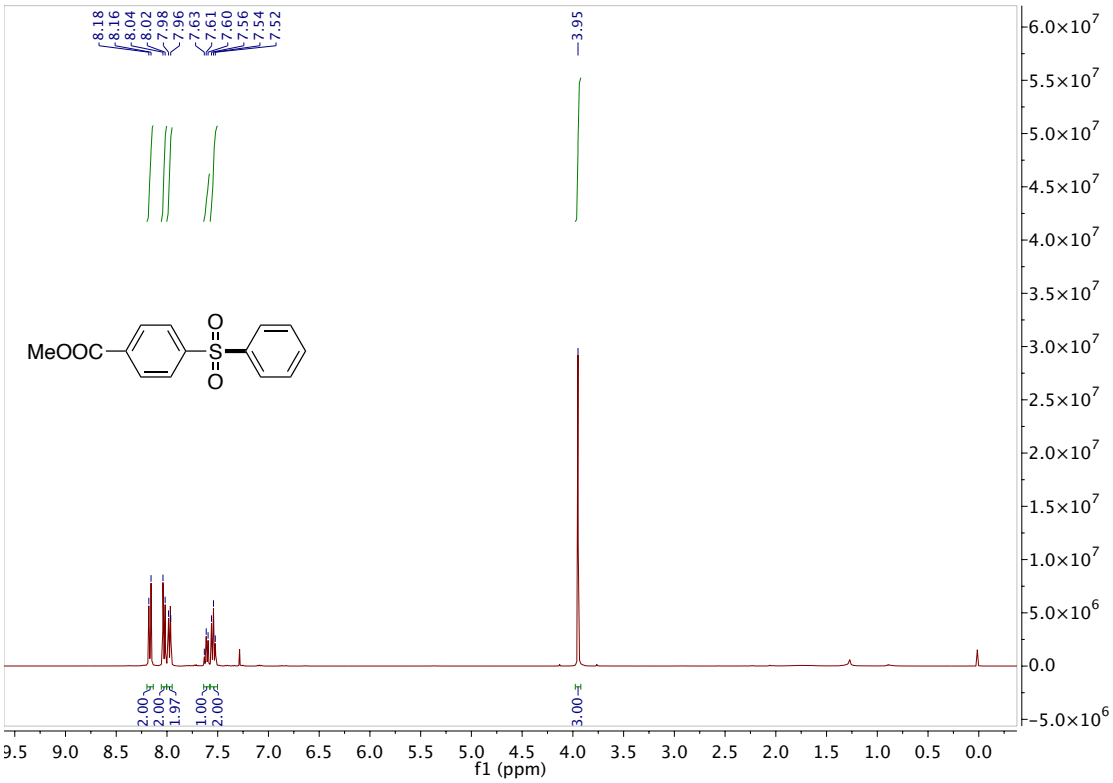


^{13}C Spectra

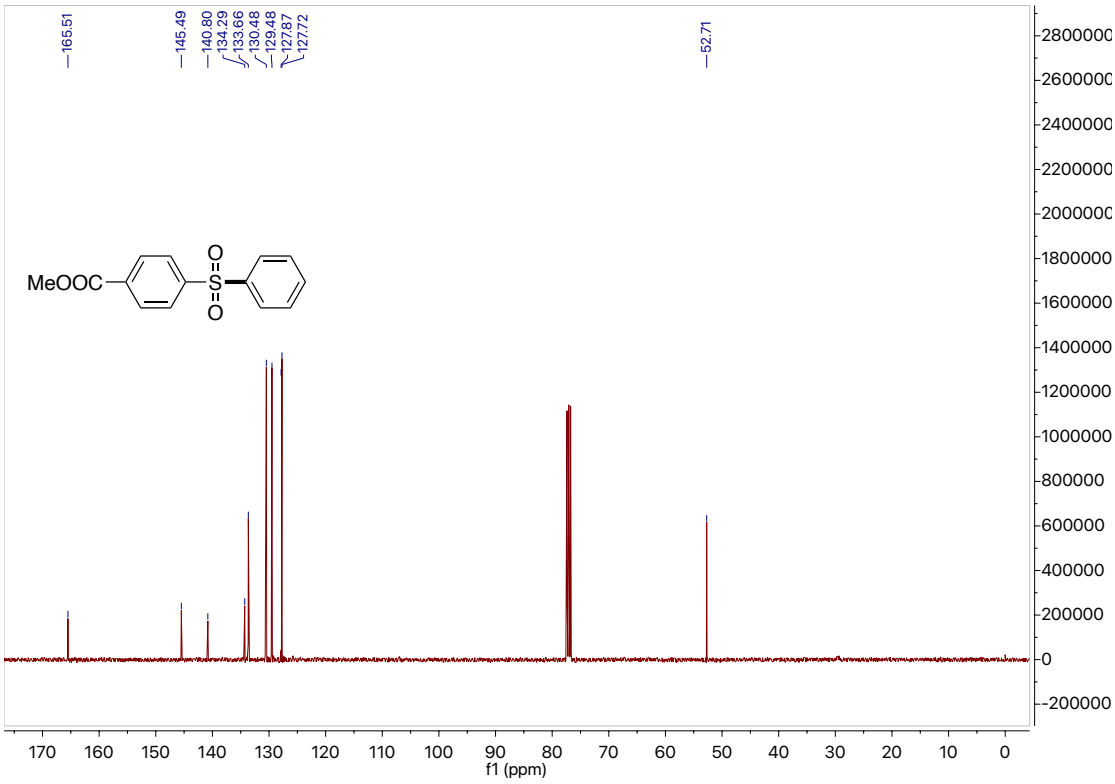


(4d)

^{1}H Spectra

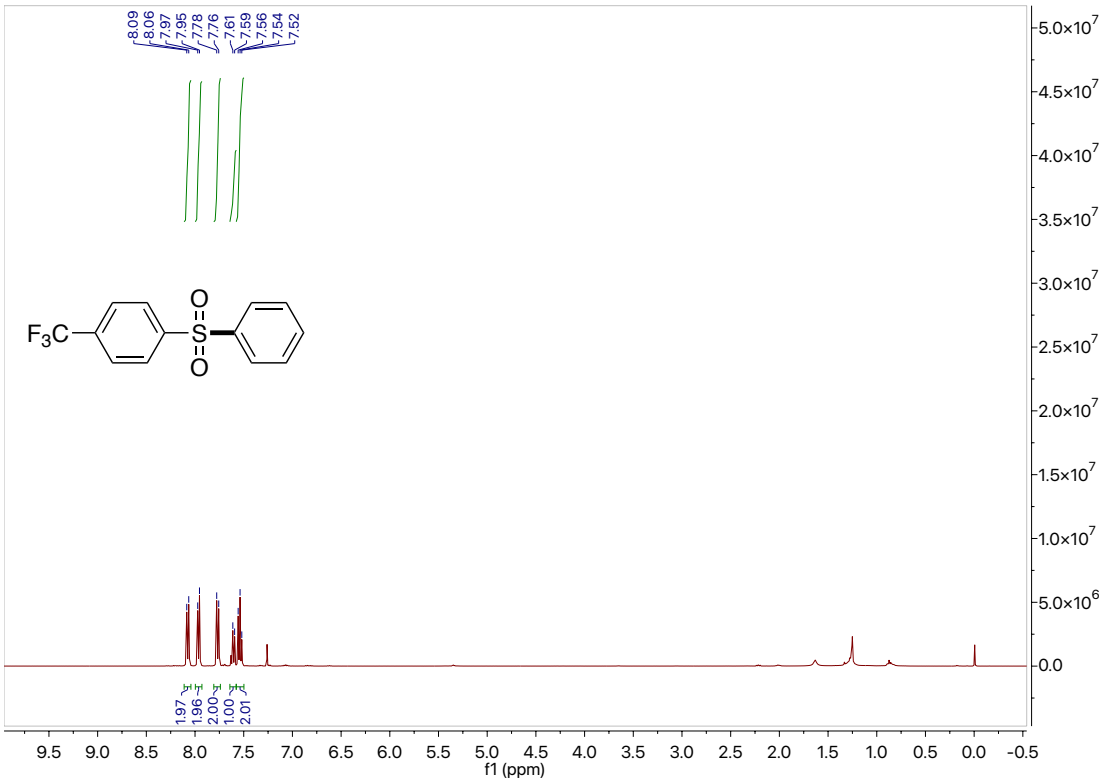


^{13}C Spectra



(4e)

^{1}H Spectra



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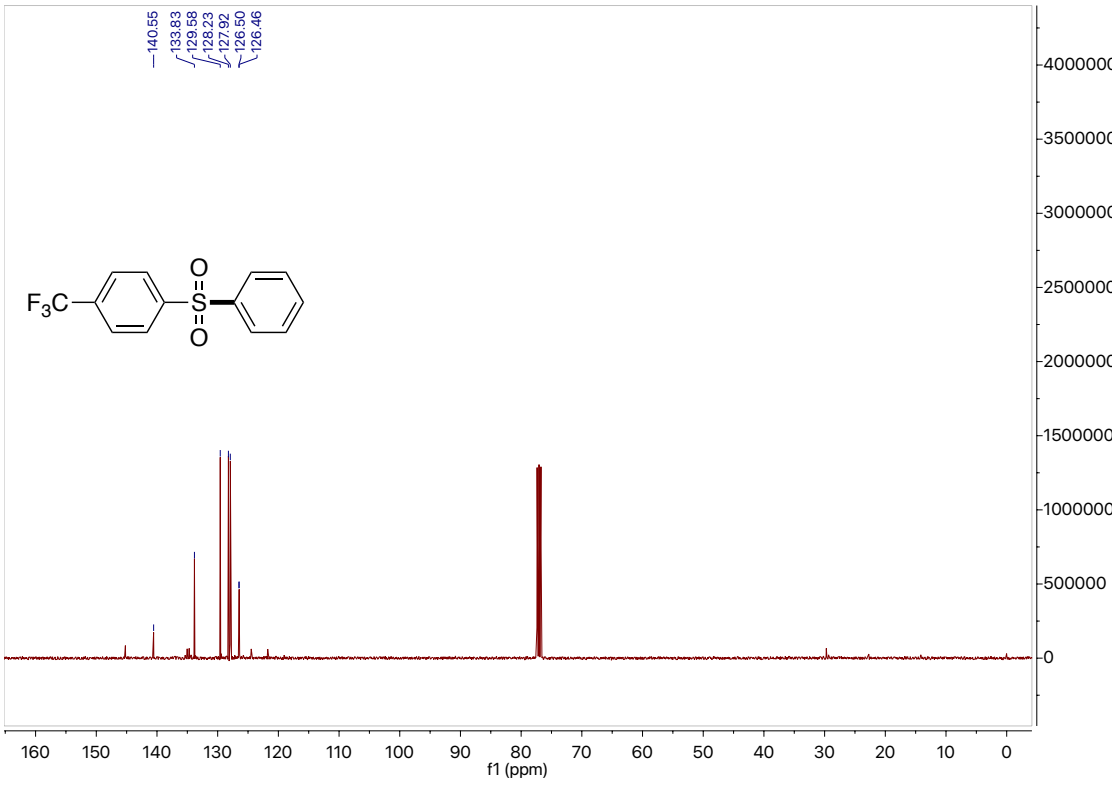
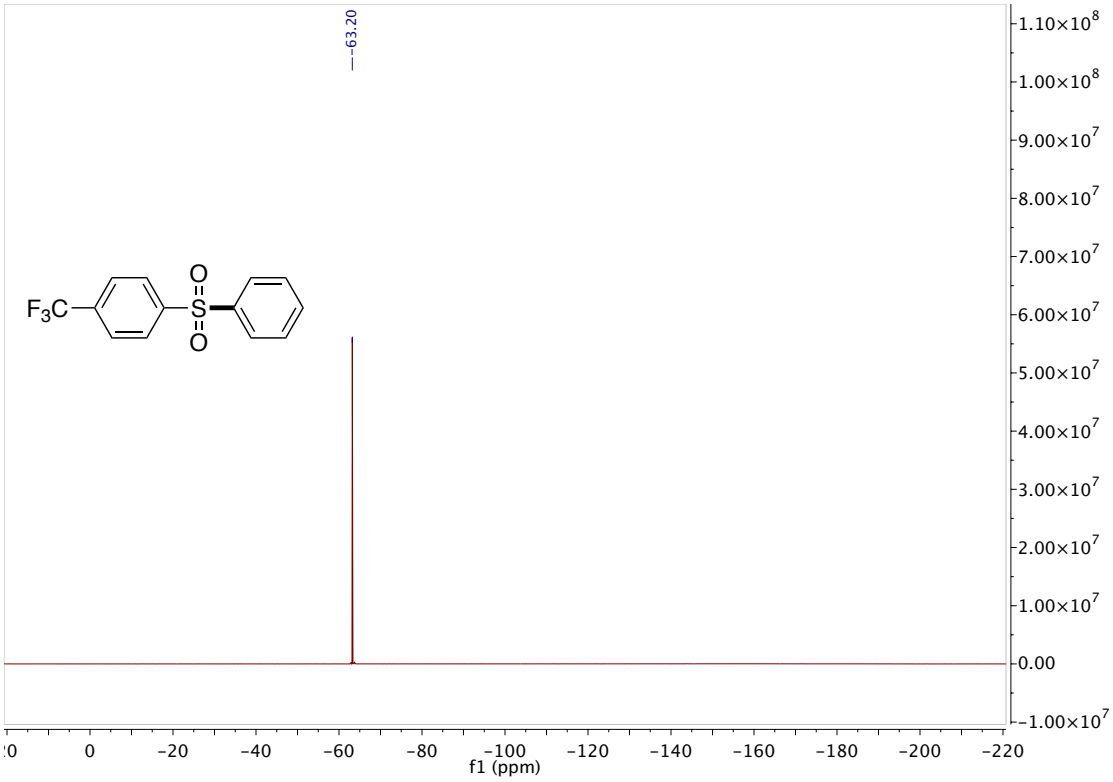
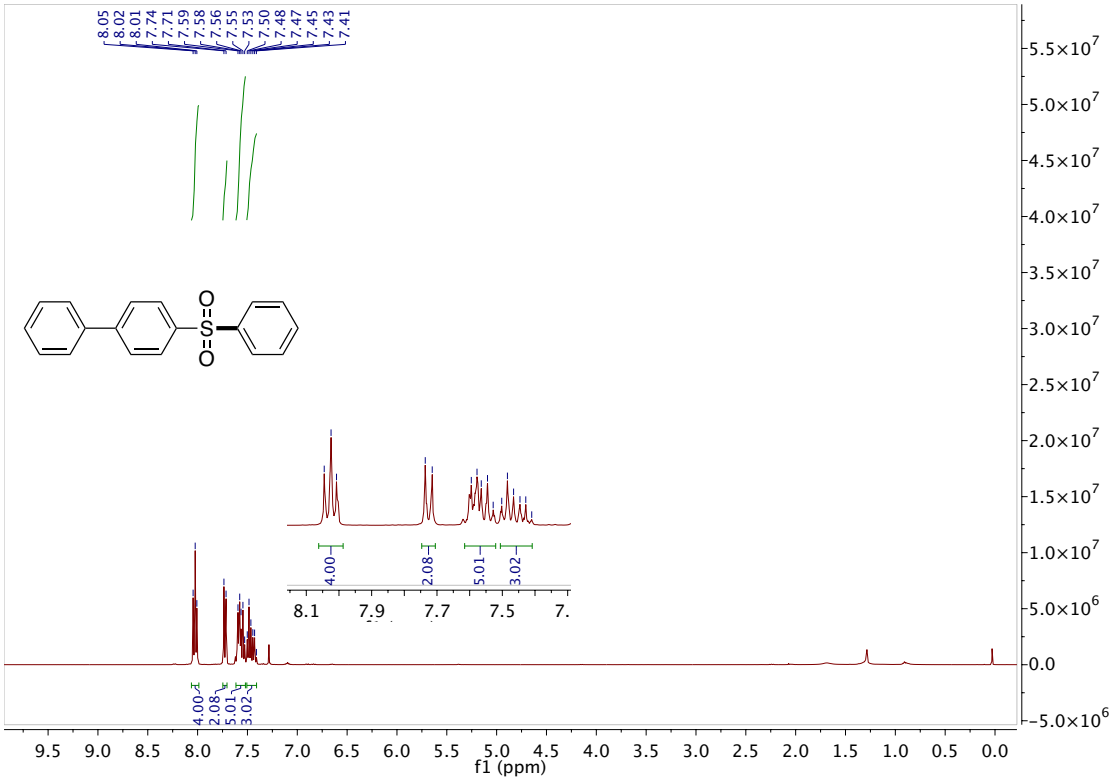


^{19}F Spectra

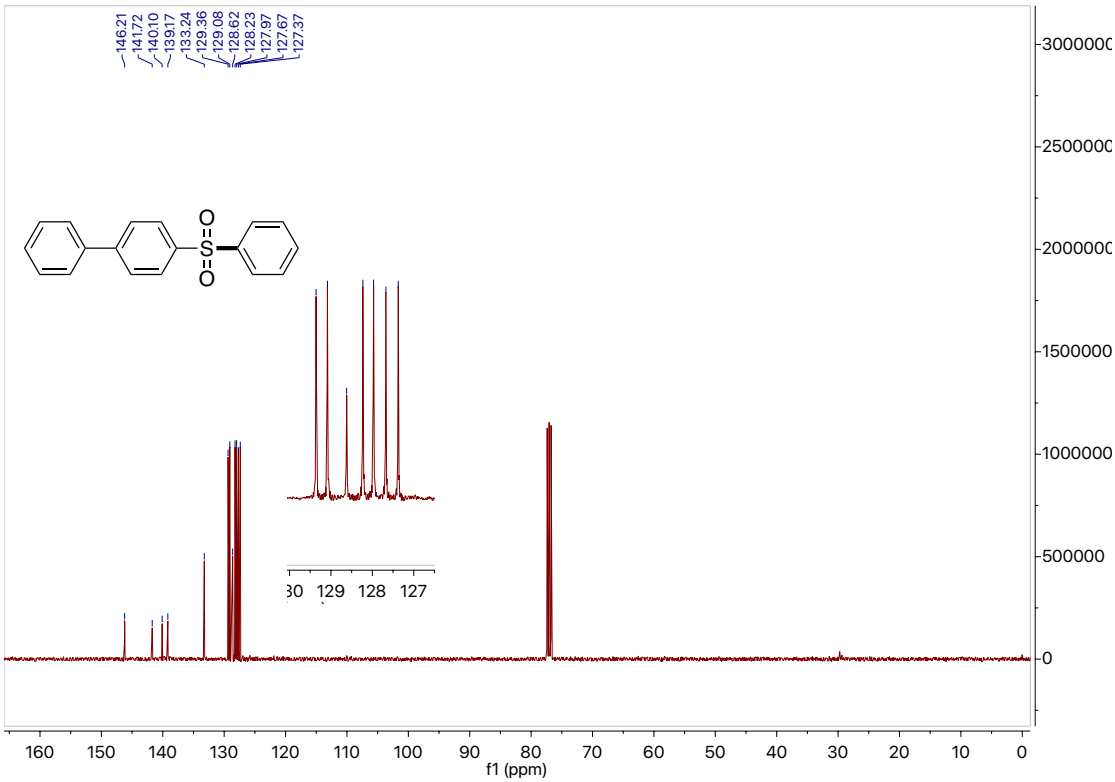

63.20
F3C
O
S
O
1.10×10^8
1.00×10^8
9.00×10^7
8.00×10^7
7.00×10^7
6.00×10^7
5.00×10^7
4.00×10^7
3.00×10^7
2.00×10^7
1.00×10^7
0.00
-1.00×10^7
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
f1 (ppm)

(4f)

^{1}H Spectra

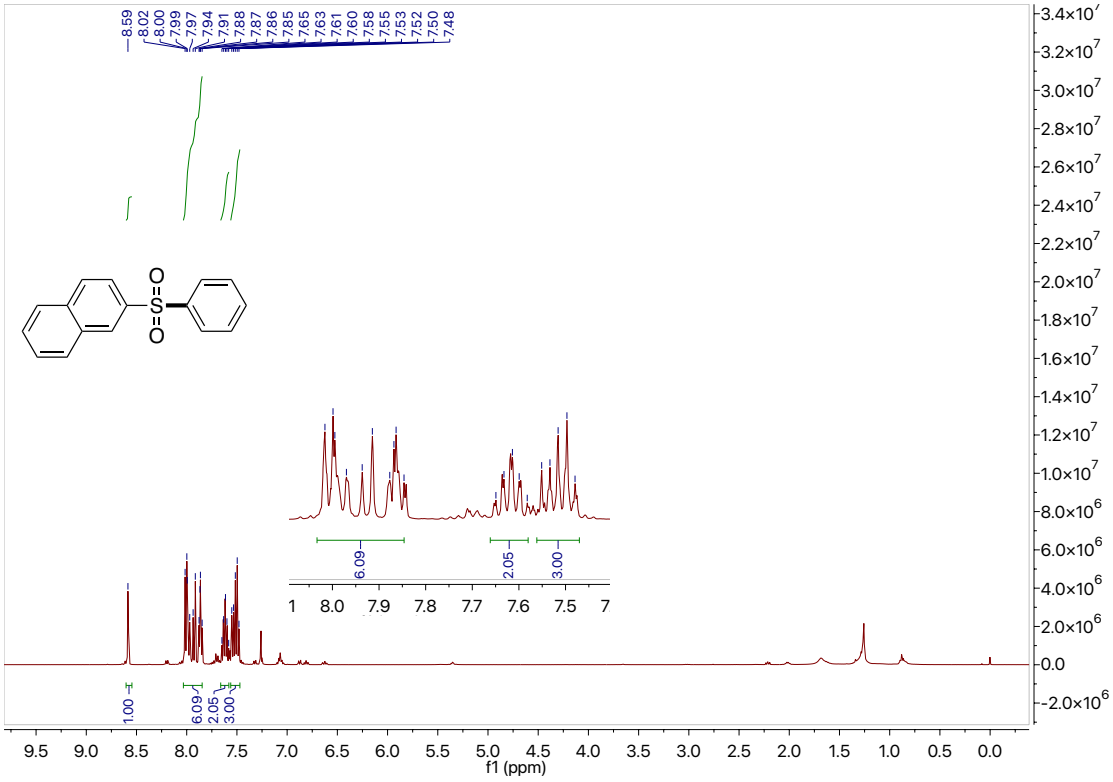


^{13}C Spectra

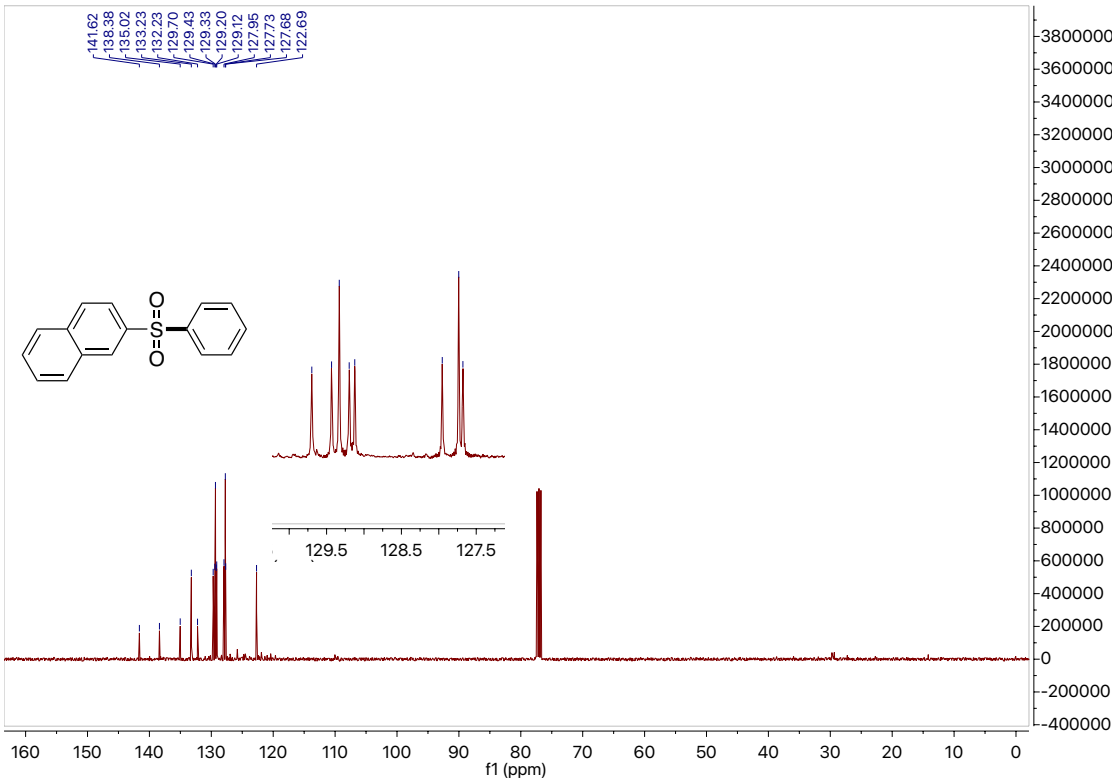


(4g)

^{1}H Spectra

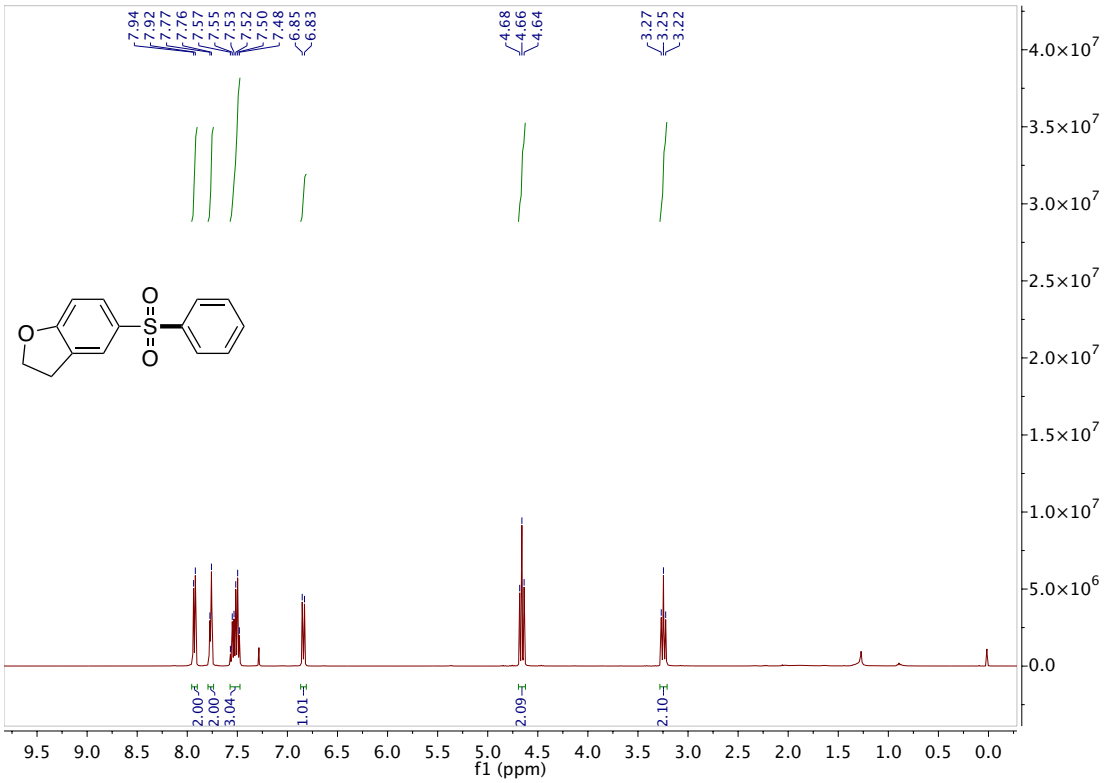


^{13}C Spectra

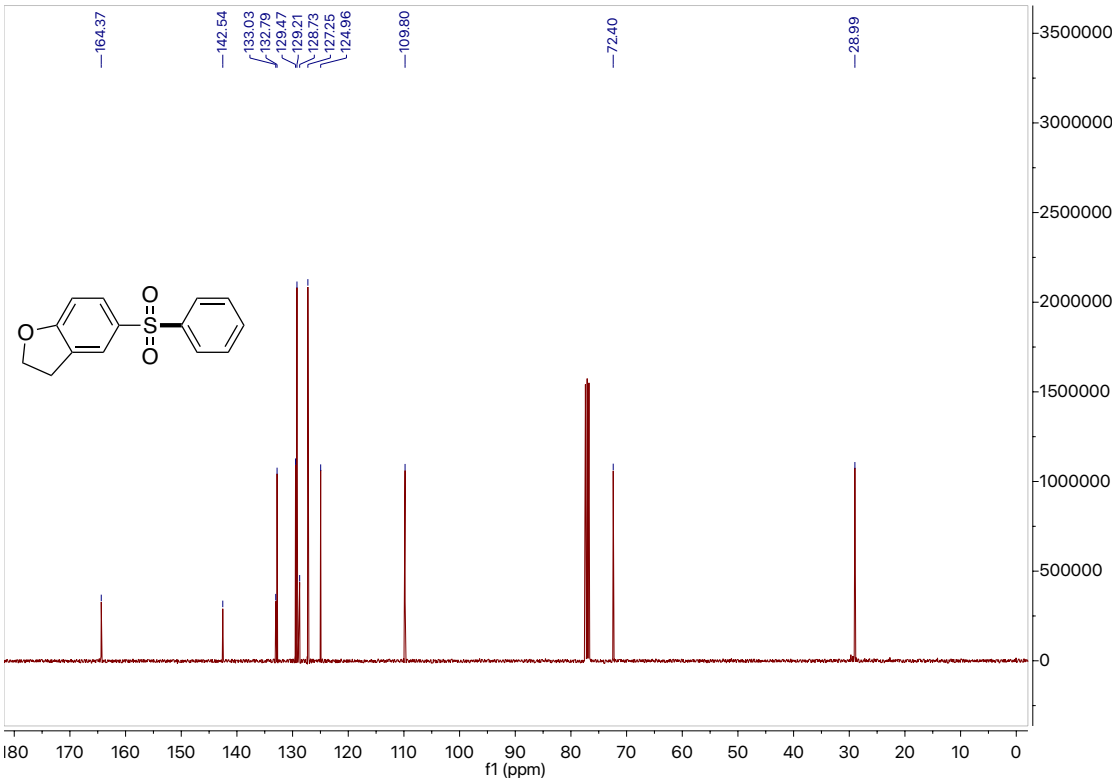


(4h)

^{1}H Spectra

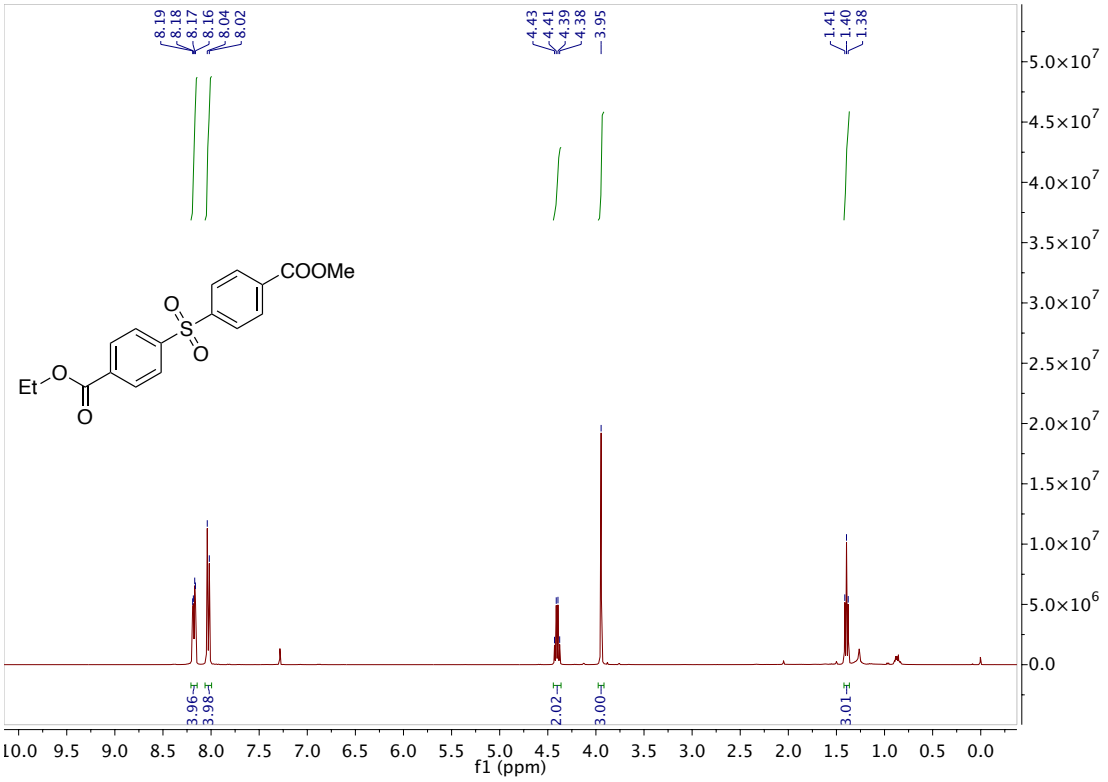


^{13}C Spectra



(4j)

^{1}H Spectra



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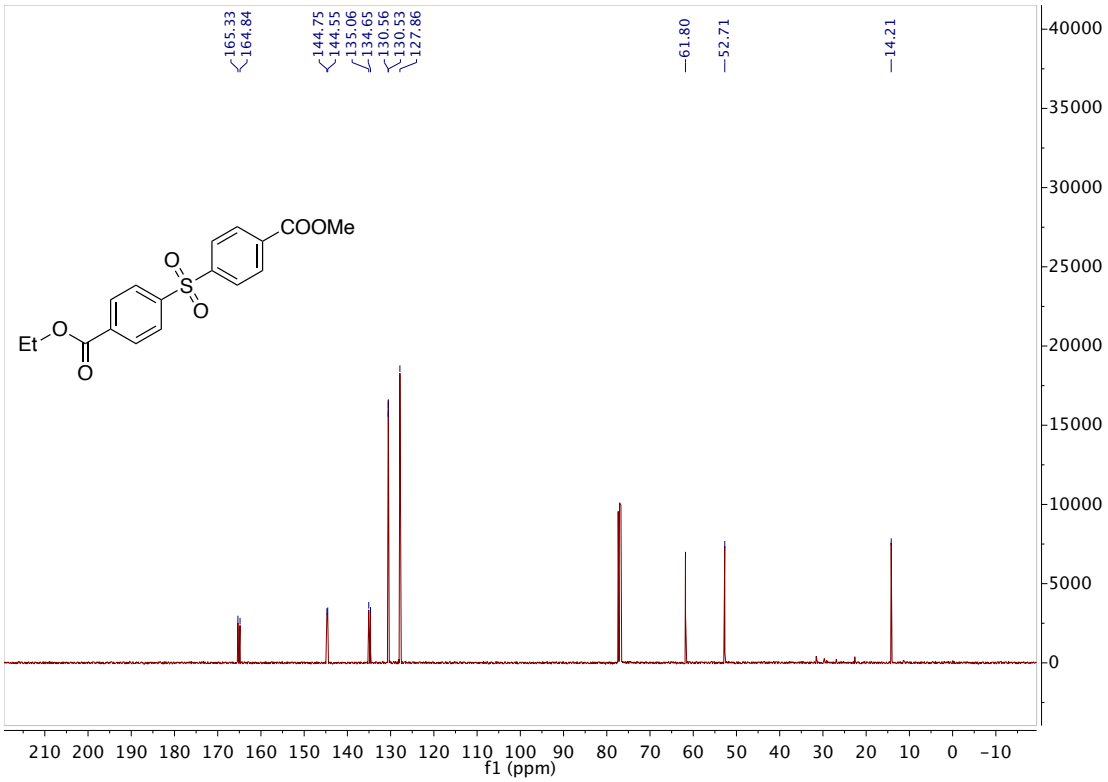
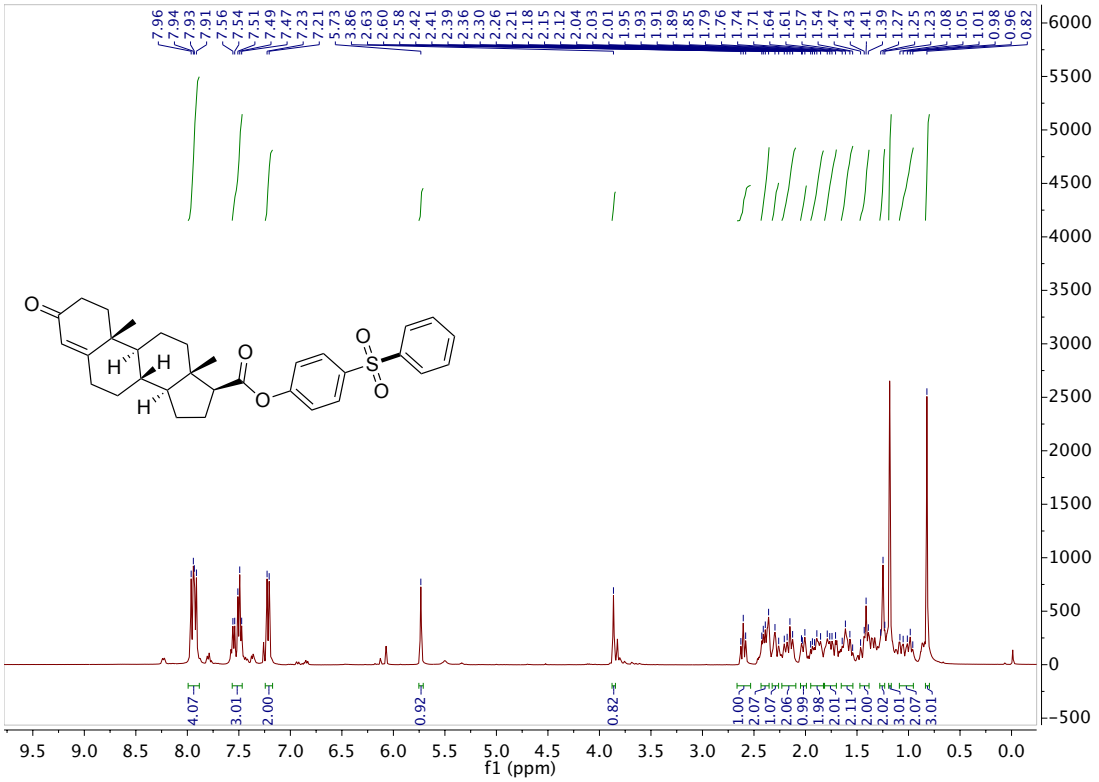
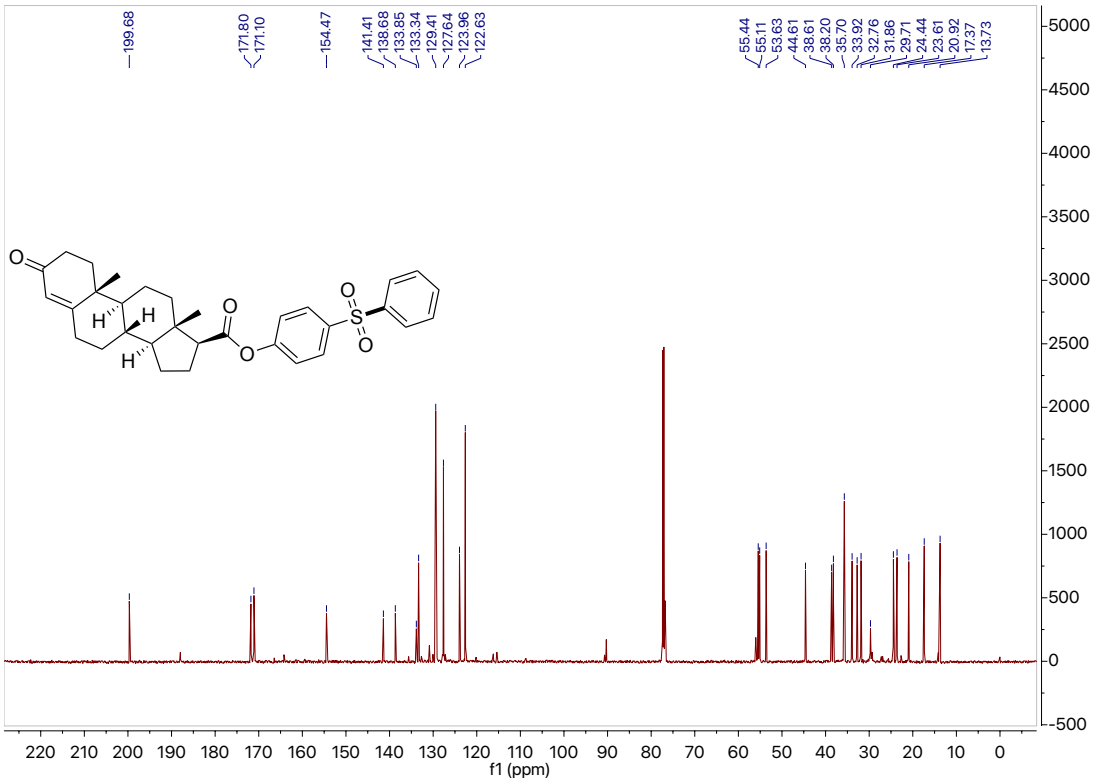


(4k)

^{1}H Spectra

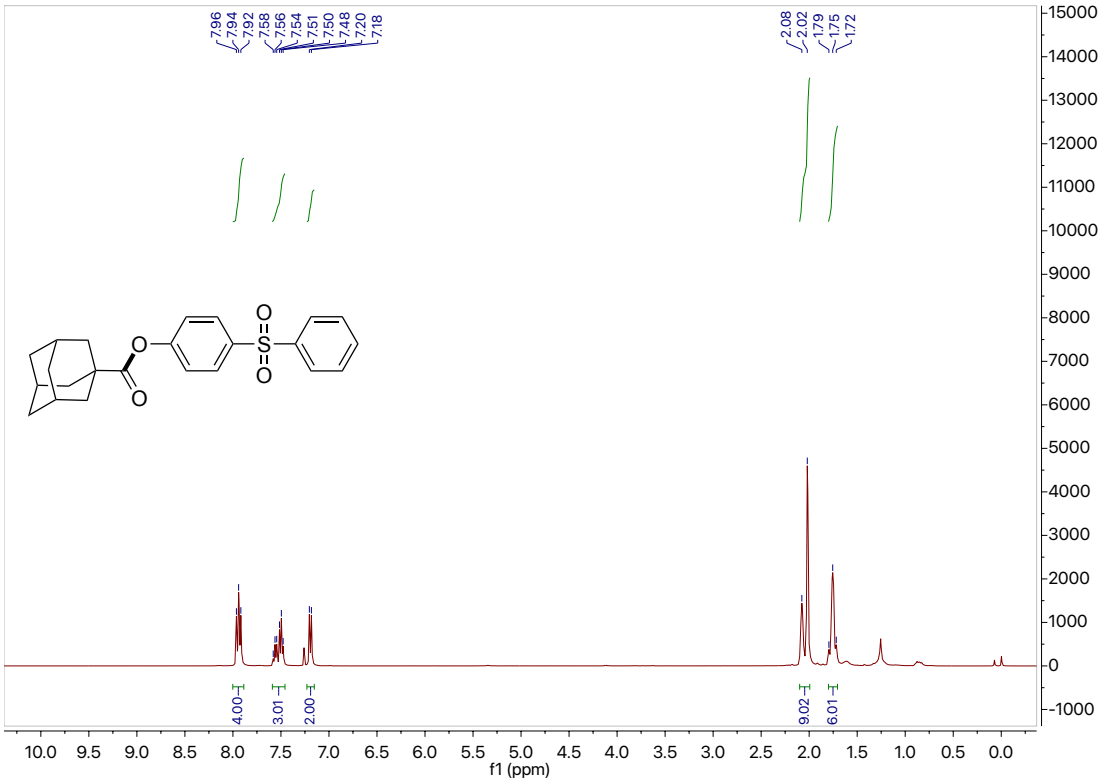


^{13}C Spectra

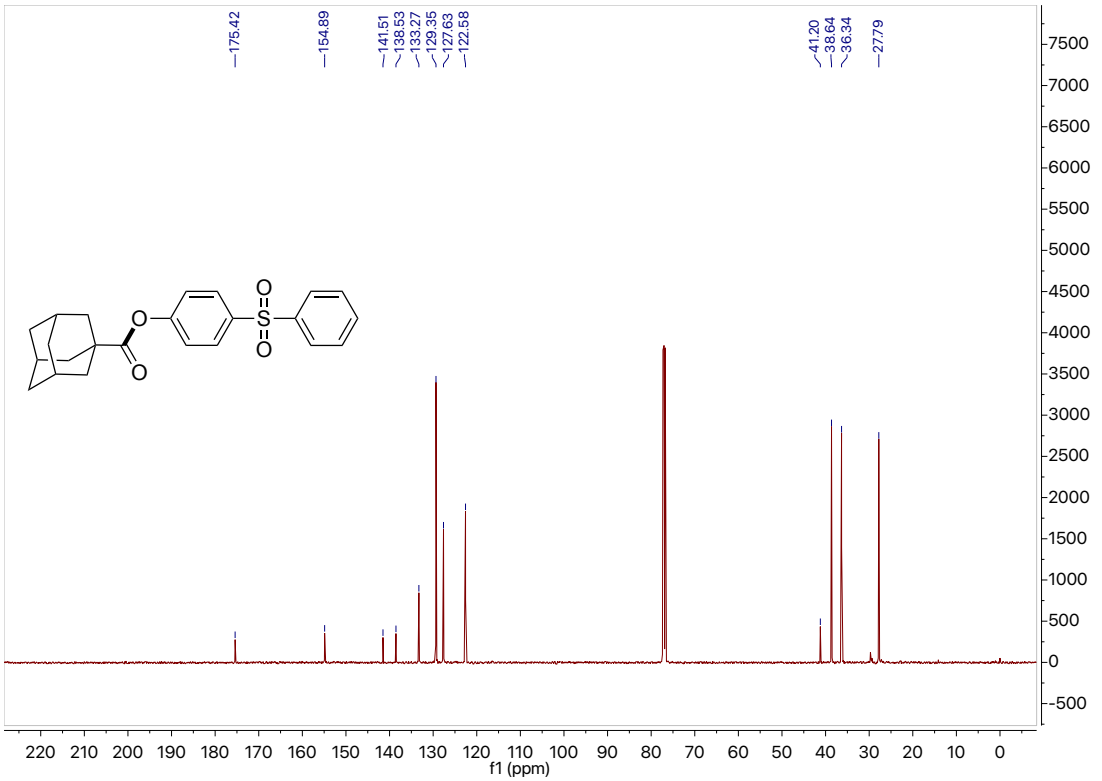


(4l)

^{1}H Spectra



^{13}C Spectra



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