Supporting Information

Phenylsulfonate as a Photolabile Group for Intramolecular Carbon-Carbon Cross-Coupling Reactions

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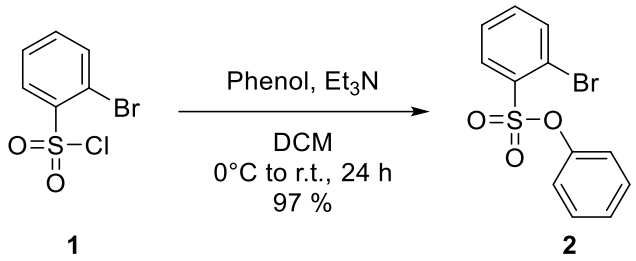
General Methods

Chemical reagents were purchased from Sigma-Aldrich Co. Canada, Alfa Aesar Co., TCI America Co., and Oakwood Products Inc., and used as supplied. Organic solvents were purchased from Fisher Scientific and purified using a Solvent Purifier System (SPS) from Vacuum Atmosphere Co., Hawthorne, USA. Tetrahydrofuran (THF) was freshly distilled over sodium/benzophenone for photochemical reactions. THF, toluene, ethanol and H_2O were degassed with nitrogen for 10 minutes before usage for Suzuki-Miyaura reactions. All anhydrous and air-sensitive reactions were conducted in oven-dried glassware under a positive nitrogen atmosphere. Thin-layer chromatography (TLC) was performed on silica gel 60 F254, 0.25 mm pre-coated plates (Silicycle, Québec, Canada), visualized under 254 nm and/or 365 nm UV light. Column chromatographies utilized 230-400 mesh silica gel R10030B (Silicycle, Québec, Canada).

A Luzchem photochemical reactor with 16 x 7.2 W low-pressure lamps (λ = 300 nm or 360 nm) was employed for photochemical reactions in quartz vessels or UV cells. NMR spectra were recorded on a Varian Inova AS400 (400 MHz) or an Agilent DD2 500 MHz spectrometer. Signals are reported with their respective patterns and coupling constants (*J*) in Hz. Chemical shifts are noted in ppm (δ) relative to the residual solvent peak. High-resolution mass spectra (HRMS) were obtained using an Agilent 6210 TOF LCMS with ESI and APPI ion sources (Agilent Technologies, Toronto, Canada). UV-visible spectra were recorded on a Varian Cary 7000 diode-array spectrophotometer using 10 mm quartz cells. Kinetic analyses were conducted using a Thermo Instrument Trace Ultra gas chromatograph and ITQ 900 Series GC/MS Ion Trap Mass Spectrometer (Thermo Fisher Scientific), injecting samples into an Agilent J&W DB-5-ms fused-silica capillary column (J&W Scientific, Folsom, CA).

Experimental section

Compound 2 (Phenyl 2-bromobenzenesulfonate)



1 **2**

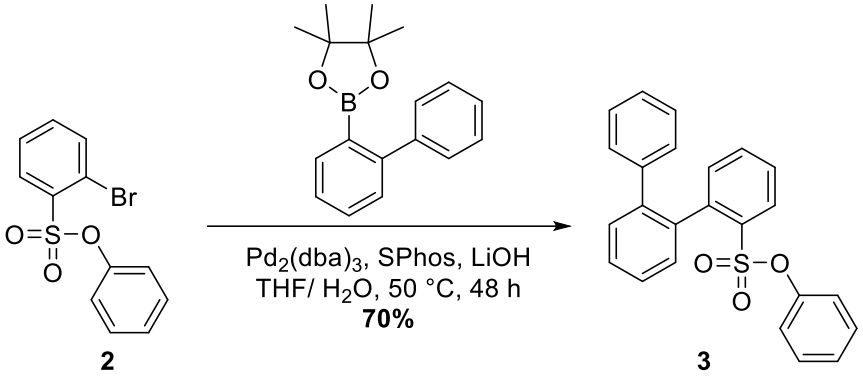
To a stirred solution of phenol (0.44 g, 4.70 mmol, 1.20 eq) in CH_2Cl_2 (7 mL) at 0 °C, 2-bromobenzenesulfonyl chloride **1** (1.00 g, 3.91 mmol, 1.00 eq.) and Et_3N (0.66 ml, 4.70 mmol, 1.20 eq.) were added. The reaction mixture was warmed at room temperature and stirred for 6 h. The mixture was quenched with 1 M HCl solution, extracted with CH_2Cl_2, dried over Na_2SO_4 and the solvent was removed under reduced pressure. The crude product was purified using column chromatography (silica gel, hexanes:EtOAc 9:1 to 3:1 as eluent) to give a colorless, viscous liquid **2** (1.43 g, 4.60 mmol, 97%).

^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95 (dd, *J* = 8.0, 1.8 Hz, 1H), 7.84 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.48 (td, *J* = 7.7, 1.9 Hz, 1H), 7.40 (td, *J* = 7.6, 1.3 Hz, 1H), 7.35 – 7.20 (m, 3H), 7.18 – 7.07 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$): δ 121.24, 122.09, 127.34, 127.62, 129.77, 132.75, 135.04, 135.33, 135.73, 149.42.

HRMS (ESI-TOF) calc for $C_{12}H_9BrO_3S$ [M+H$^+$] 313.9607 m/z, found : 313.9609 m/z (M+H$^+$).

Compound 3 (Phenyl [1,1':2',1''-terphenyl]-2-sulfonate)



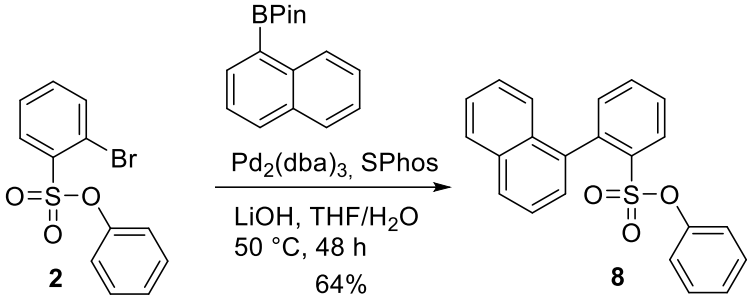
2 **3**

Phenyl[1,1':2',1"-terphenyl] -2-sulfonate **2** (1.00 g, 3.20 mmol, 1.00 eq.), 2-(biphenyl-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (897 mg, 3.20 mmol, 1.00 eq.) and $LiOH \cdot H_2O$ (245 mg, 5.83 mmol, 7.30 eq) tube were introduced in a Schlenk under argon atmosphere. The powders were purged and refilled with nitrogen 3 times, then dry THF (7.0 mL) and water (1.8 ml) were added before argon was bubbled into the mixture for 20 minutes. Finally, $Pd_2(dba)_3$ (147 mg, 0.16 mmol, 5% eq.) and SPhos (263 mg, 0.64 mmol, 0.20 eq.) were added before the tube was sealed and the mixture heated to 50 °C during 48 h. Upon cooling at room temperature, the mixture was extracted with CH_2Cl_2 (3 x 20 mL). The combined organic layers were washed with water and NH_4Cl, dried over Na_2SO_4, filtered, and the solvent was removed under reduced pressure. The crude product was purified by column chromatography (silica gel, hexanes:AcOEt, 9:1 to 3:1 as the eluent) to give the desired compound **3** as a white powder (877 mg, 2.27 mmol, 70%).

^{1}H NMR (500 MHz, $CDCl_3$): δ 7.98 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.54 – 7.47 (m, 2H), 7.47 – 7.41 (m, 2H), 7.41 – 7.33 (m, 2H), 7.34 – 7.22 (m, 3H), 7.21 – 7.17 (m, 2H), 7.15 – 7.10 (m, 4H), 6.91 – 6.83 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$): δ, 122.52, 126.37, 126.61, 127.07, 127.28, 127.79, 128.51, 129.63, 129.69, 130.18, 130.19, 130.48, 132.99, 134.05, 135.01, 137.12, 140.91, 140.99, 142.11, 148.97. HRMS (ESI-TOF) calc for $C_{22}H_{16}O_3S$ $[M+H^+]$ 387.1055 m/z, found : 387.1053 m/z $(M+NH_4^+)$

Compound 8 (phenyl 2-(naphthalen-1-yl) benzenesulfonate)



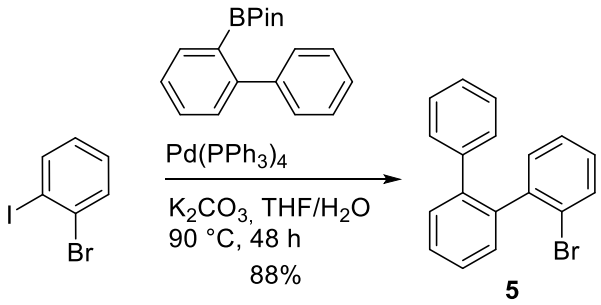
A Schlenk tube under argon atmosphere was charged with phenyl[1,1':2',1"-terphenyl]-2-sulfonate (285 mg, 0.91mmol, 1.00 eq.), 1-naphthylboronic pinacol ester (231 mg, 0.91mmol, 1.00 eq.) and $LiOH \cdot H_2O$ (279 mg, 6.64 mmol, 7.30 eq.). The powders were purged and refilled with nitrogen 3 times, then THF (8 ml) and H_2O (2 ml) were added before argon was bubbled into the mixture for 20 minutes. Finally, $Pd_2(dba)_3$ (46 mg, 0.05 mmol, 0.05 eq) and SPhos (74 mg, 0.18 mmol, 0.20 eq.) were added, the tube was sealed, and the mixture was heated at 50 °C for 72 hours. After cooling at room temperature, the reaction was extracted with CH_2Cl_2 (3 x 20 mL). The combined organic layers were washed with water and NH_4Cl, dried over Na_2SO_4, filtered and the solvent was removed under reduced pressure. The crude was purified by column chromatography (silica gel, toluene: CH_2Cl_2, 8:2 as the eluent) and the desired product was precipitated in pentane to provide the desired compound as a white solid (210 mg, 0.58 mmol, 64%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.23 (dt, *J* = 8.1, 1.0 Hz, 1H), 7.95 (dd, *J* = 8.3, 1.0 Hz, 1H), 7.91 (dd, *J* = 8.2, 1.1 Hz, 1H), 7.77 (td, *J* = 7.5, 1.4 Hz, 1H), 7.65 (td, *J* = 7.8, 1.4 Hz, 1H), 7.55 – 7.44 (m, 4H), 7.40 (dt, *J* = 7.0, 1.2 Hz, 1H), 7.37 – 7.30 (m, 2H), 7.22 – 7.18 (m, 3H), 6.73 – 6.65 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 148.74, 141.00, 135.79, 135.67, 133.59, 133.50, 133.21, 132.36, 130.37, 129.50, 128.60, 128.13, 128.11, 127.43, 126.80, 126.13, 126.00, 125.79, 124.60, 122.06, 77.27, 77.01, 76.76.

HRMS (ESI-TOF) calc for $C_{22}H_{16}O_3S$ $[M+H^+]$ 360.089 m/z, found : 378.122 m/z ($M+NH_4^+$)

Compound 5 (2-bromo-1,1':2',1''-terphenyl)



A Schlenk tube under argon atmosphere was charged with 2-iodobromobenzene (1.13 g, 4.00 mmol, 1.00 eq.), 2-(biphenyl-2-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.12 g, 4.00 mmol, 1.00 eq.) and K_2CO_3 (1,66 g, 12.0 mmol, 3.00 eq.). The powders were purged and refilled with nitrogen 3 times, then dry THF (7.0 mL) and water (1.8 ml) were added before argon was bubbled into the mixture for 20 minutes. Finally, $Pd(PPh_3)_4$ (231 mg, 0.20 mmol, 0.05 eq.) was added, the tube was sealed and the mixture heated at 50 °C for 48 h. After cooling at room temperature, the mixture was extracted with CH_2Cl_2 (3 x 20 mL). The combined organic layers were washed with water and NH_4Cl, dried over Na_2SO_4, filtered and the solvent was removed under reduced pressure.

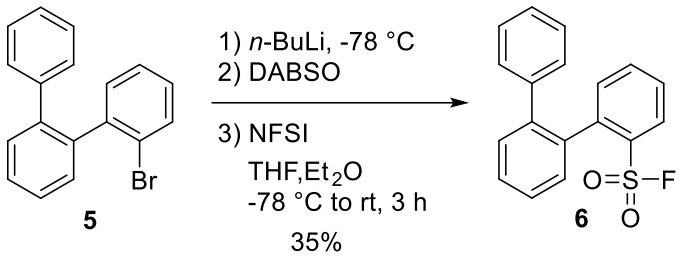
The crude product was purified by column chromatography (silica gel, hexanes:EtOAc, 9:1 to 3:1 as the eluent) and the desired product was obtained as a colorless oil **5** (1.088 g, 3.52 mmol, 88%).

^{1}H NMR (500 MHz, $CDCl_3$). δ 7.58 – 7.56 (m, 1H), 7.52 (dd, *J* = 2.3, 1.0 Hz, 1H), 7.51 (d, *J* = 1.1 Hz, 2H), 7.46 (ddd, *J* = 7.5, 5.4, 3.4 Hz, 1H), 7.39 (dt, *J* = 7.6, 1.1 Hz, 1H), 7.26 – 7.18 (m, 6H), 7.19 – 7.17 (m, 1H), 7.15 – 7.08 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 123.91, 126.81, 126.92, 127.75, 128.19, 128.48, 129.53, 130.82, 132.18, 132.56, 139.70, 141.04, 141.12, 142.38.

HRMS (ESI-TOF) calc for $C_{18}H_{13}Br$ $[M+H^+]$ 309.0279m/z, found : 309.0280 m/z ($M+H^+$)

Compound 6 ([1,1':2',1''-terphenyl]-2-sulfonyl fluoride)



In a round bottom flask equipped with a magnetic stir bar, DABSO[1] (389 mg, 1.62 mmol, 1.00 eq.) was added in portions to a solution of 2-lithium-1,1':2',1"-terphenyl (1.62 mmol, 1.00 eq) in a mixture of THF (15 mL) and Et_2O (15 mL) at -78 °C. The resulting mixture was stirred at room temperature for 45 minutes. Then, the solution was then cooled at 0 °C before NFSI (766 mg, 2.43 mmol, 1.50 eq.) was added gradually while maintaining the same temperature. The mixture was stirred at room temperature for 3 hours before saturated aqueous NH_4Cl (20 mL) was added. The mixture was extracted with EtOAc, followed by a brine wash. The combined organic layers were dried over $MgSO_4$, filtered and the solvent was removed under reduced pressure. The crude product

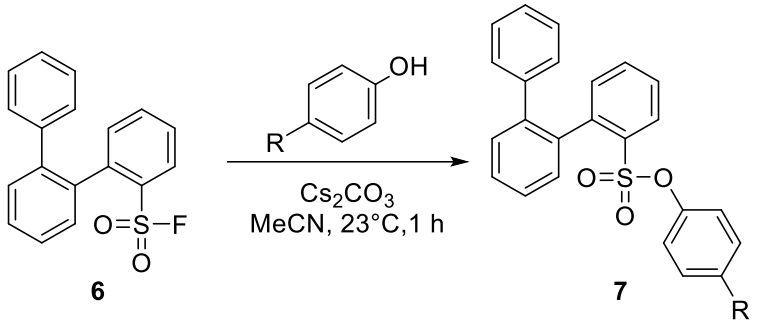
was purified by column chromatography (silica gel, hexanes:CH_2Cl_2, 9:1 as the eluent), yielding the desired compound **6** as a colorless oil (180 mg, 0.57 mmol, 36% yield).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.11 (dd, *J* = 7.9, 1.5 Hz, 1H), 7.53 (ddd, *J* = 7.8, 7.1, 1.5 Hz, 1H), 7.50 – 7.49 (m, 1H), 7.49 – 7.46 (m, 1H), 7.46 – 7.45 (m, 1H), 7.45 – 7.42 (m, 1H), 7.42 – 7.38 (m, 1H), 7.21 – 7.14 (m, 7H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 126.73, 126.80, 127.73, 127.88, 128.98, 129.53, 130.02, 130.06, 130.08, 130.31, 132.60, 132.77, 134.13, 134.19, 136.12, 140.54, 141.01, 142.51.

HRMS (ESI-TOF) calc for $C_{18}H_{13}FO_2S$ [M+H$^+$] 313.0693 m/z, found : 330.0959 m/z (M+NH_4^+).

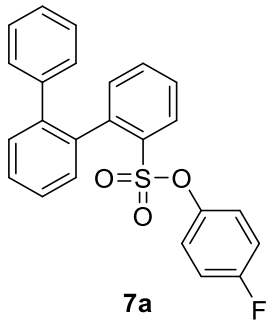
General procedure for the synthesis of phenyl [1,1':2',1''-terphenyl]-2-sulfonate derivatives



6 **7**

A round bottom flask equipped with a magnetic stir bar was charged with compound **9** (100 mg, 0.32 mmol,1.00 eq.), the selected phenol (1.00 mmol, 3.00 eq), cesium carbonate (326 mg, 1 mmol, 3 eq) and acetonitrile (5.0 mL). The resulting mixture was stirred at room temperature for 1 hour before the solvent was removed under reduced pressure. The crude product was extracted with EtOAc and washed with K_2CO_3, and brine. The organic layers were combined, dried over magnesium sulfate ($MgSO_4$), filtered, and the solvent was removed under reduced pressure. The crude product was purified by column chromatography (silica gel) to obtain the desired purified compound.

Compound 7a (4-fluorophenyl [1,1':2',1''-terphenyl]-2-sulfonate)



7a

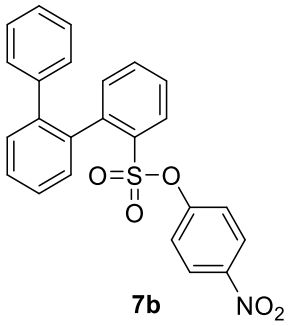
Yield: 116 mg, 89 %

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99 (ddd, *J* = 8.0, 1.4, 0.4 Hz, 1H), 7.62 – 7.57 (m, 2H), 7.57 – 7.47 (m, 3H), 7.46 – 7.37 (m, 3H), 7.23 – 7.12 (m, 6H), 6.94 – 6.89 (m, 2H).

^{13}C NMR (126 MHz, , $CDCl_3$) 76.79, 77.04, 77.25, 77.30, 115.95 (d, $^{2}J_{C-F}$ = 23 Hz, 2C), 116.18, 116.24, 116.43, 124.15(d, $^{3}J_{C-F}$ = 9 Hz, 2C) , 126.39, 126.68, 127.38, 127.83, 128.60, 129.69, 130.24 (d, J_{C-F} = 2 Hz), 130.32, 130.51, 133.20, 134.15, 134.57, 136.98, 140.84, 141.02, 142.17, 144.52 (d, J_{C-F} = 2 Hz), 161.54 (d, $^{1}J_{C-F}$ =247 Hz).

HRMS (ESI-TOF) calc for $C_{24}H_{17}FO_3S$ [M+H$^+$] 405.0960 m/z, found : 405.0956 m/z (M+H$^+$)

Compound 7b (4-nitrophenyl [1,1':2',1''-terphenyl]-2-sulfonate)



7b

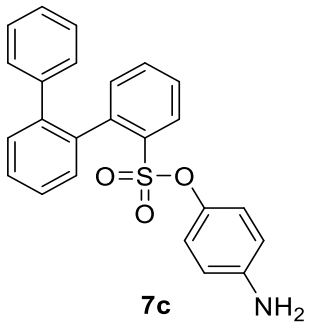
Yield : 116 mg, 84 %

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.21 – 8.14 (m, 2H), 8.01 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.63 – 7.38 (m, 6H), 7.27 – 7.13 (m, 6H), 7.00 – 6.91 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 76.80, 77.06, 77.26, 77.31, 123.24, 125.33, 126.49, 126.81, 127.64, 127.91, 128.85, 129.68, 130.19, 130.38, 130.55, 133.66, 134.33, 134.39, 136.63, 140.70, 141.07, 142.23, 146.13, 153.15.

HRMS (ESI-TOF) calc for $C_{24}H_{17}NO_5S$ [M+H$^+$] 432.0905 m/z, found: 432,0901 m/z (M+H$^+$)

Compound 7c (4-aminophenyl [1,1':2',1''-terphenyl]-2-sulfonate)



7c

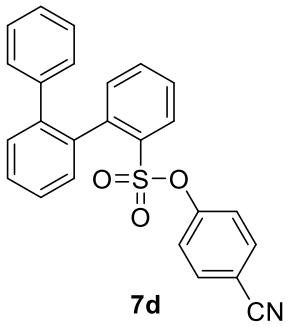
Yield : 104 mg, 81%

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.04 – 7.85 (m, 1H), 7.51 (ddd, *J* = 7.7, 6.8, 1.6 Hz, 1H), 7.48 – 7.43 (m, 2H), 7.43 – 7.38 (m, 2H), 7.37 – 7.32 (m, 1H), 7.22 – 7.18 (m, 2H), 7.16 – 7.13 (m, 2H), 7.12 – 7.08 (m, 1H), 6.68 – 6.61 (m, 2H), 6.57 – 6.49 (m, 2H), 3.73 (d, *J* = 19.9 Hz, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) : δ 145.39, 142.06, 140.95, 140.91, 140.84, 137.24, 134.95, 133.94, 132.81, 130.48, 130.30, 130.13, 129.69, 128.41, 127.76, 127.14, 126.56, 126.32, 123.39, 115.36.

HRMS (ESI-TOF) calc for $C_{24}H_{17}NO_3S$ [M+H$^+$] 402.1164 m/z, found: 402.1159m/z (M+H$^+$)

Compound 7d (4-cyanophenyl [1,1':2',1''-terphenyl]-2-sulfonate)



7d

Yield : 115 mg, 87 %

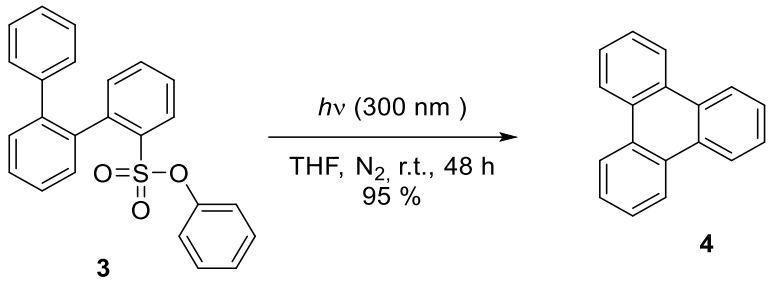
^{1}H NMR (500 MHz, $CDCl_3$): δ 7.99 (ddd, *J* = 8.1, 1.5, 0.4 Hz, 1H), 7.62 – 7.57 (m, 2H), 7.57 – 7.45 (m, 3H), 7.44 – 7.39 (m, 3H), 7.23 – 7.12 (m, 6H), 6.95 – 6.88 (m, 2H).

^{13}C NMR (126 MHz, $CDCl_3$) : δ 141.05, 136.65, 134.33, 133.82, 133.55, 130.51, 130.34, 130.15, 129.65, 128.76, 127.88, 127.57, 126.77, 126.44, 123.47, 111.08, 85.13.

HRMS (ESI-TOF) calc for $C_{25}H_{17}NO_3S$ [M+H$^+$] 412.9368 m/z, found: 412.9372 m/z (M+H$^+$)

Procedure for photosynthesis

Compound 4 (Triphenylene)



3 **4**

A 20 mL quartz tube was charged with compound **3** (868 mg, 2.24 mmol) and dry THF (15 mL). The solution was purged with nitrogen for 15 minutes before it was sealed and irradiated at 300 nm using 16 low-pressure 7.2 W USHIO light bulbs within a Luzchem photochemical reactor.

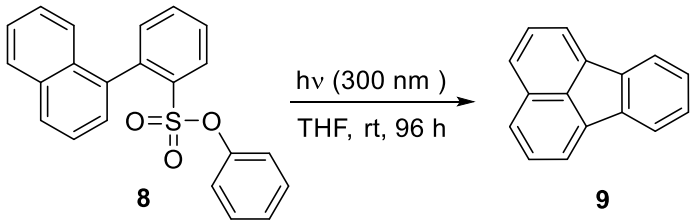
After 48 hours, the mixture was diluted with dichloromethane (CH_2Cl_2, 20 mL) and washed with water (20 mL) and brine (20 mL). The aqueous layers were extracted twice with CH_2Cl_2 (3 x 10 mL). The combined organic layers were dried over Na_2SO_4, and the solvent was reduced under reduced pressure. The crude product was purified using column chromatography (silica gel, hexanes:CH_2Cl_2 9:1 as the eluent) to provide the desired compound as a white crystalline solid (510 mg, 2.25 mmol, 95% yield).

^{1}H NMR (500 MHz, $CDCl_3$): δ 8.68 (dd, *J* = 6.2, 3.4 Hz, 6H), 7.68 (dd, *J* = 6.3, 3.3 Hz, 6H).

^{13}C NMR (101 MHz, $CDCl_3$): δ 123.30, 127.22, 129.79.

HRMS (ESI-TOF) calc for $C_{18}H_{12}$ [M+H$^+$] 229,1017 m/z, found : 229,1014 m/z (M+H$^+$)

Compound 9 (Fluoranthene)



A 20 mL quartz tube was charged with phenyl 2-(naphthalen-1-yl) benzenesulfonate (236 mg, 0.66 mg) dissolved in dry THF (15 mL). The solution was purged with nitrogen for 15 minutes before it was sealed and irradiated at 300 nm using 16 low-pressure 7.2 W USHIO light bulbs within a Luzchem photochemical reactor. After 96 hours, the mixture was diluted with CH_2Cl_2 (20 mL) and washed with water (20 mL) and brine (20 mL). The aqueous layers were extracted twice with CH_2Cl_2 (3 x 10 mL). The combined organic layers were dried over Na_2SO_4 and the solvent was reduced under reduced pressure. The crude product was purified over a short pad of silica (hexanes as eluent) and the resulting solid was analyzed by GC/MS to show the presence of fluoranthene and 1-phenylnaphthalene (1:4).

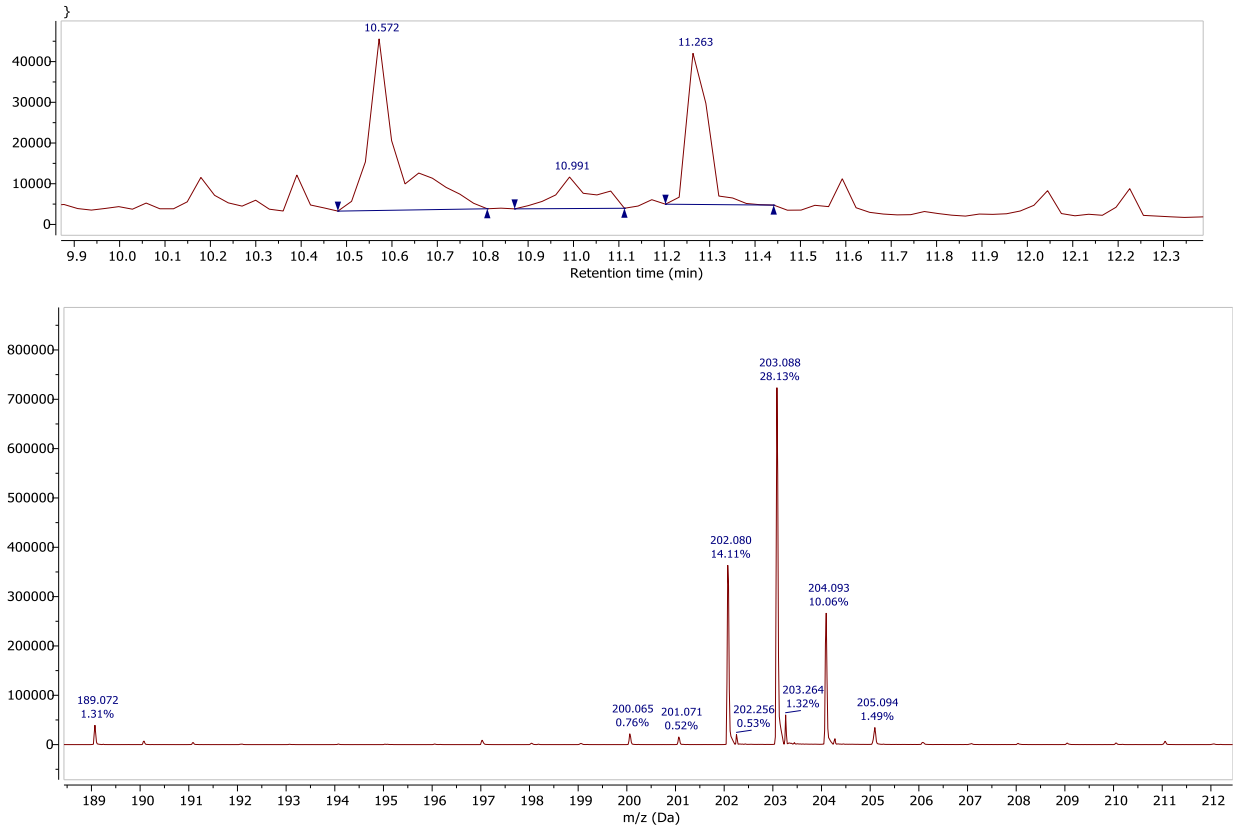
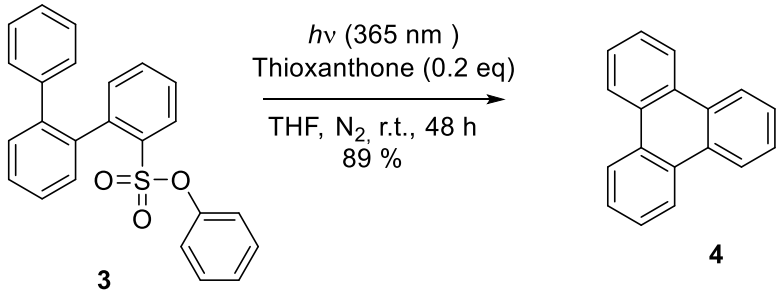


Fig. S1 : GC/MS result for compound **9**

Kinetic Study

Quantitative analysis was conducted using a Thermo Scientific Instrument Trace Ultra as the gas chromatograph coupled with an ITQ 900 Series GC/MS Ion Trap Mass Spectrometer. Calibration of the instrument was achieved using five standard solutions of triphenylene in distilled tetrahydrofuran (THF). Samples were injected into an Agilent J&W DB-5ms fused-silica capillary column (J&W Scientific, Folsom, CA). The column effluent was then introduced into the ion source of the ITQ 900 Series GC/MS Ion Trap Mass Spectrometer. Quantitative kinetic analysis was performed using a flame ionization detector.

Triplet sensitization



In a quartz stirred flask, compound **3** (50 mg, 0.129 mmol, 1 eq) and thioxanthone (5.4 mg, 0.026 mmol) were dissolved in THF. The mixture was stirred for 48h under irradiation at 365 nm. Then the resulting mixture was evaporated under reduced pressure and purified over a short pad of silica (hexanes) to give compound **4** as a white crystalline solid (26 mg, 89%).

TRIPLET QUENCHING

^{1}H and ^{13}C NMR Characterisation data

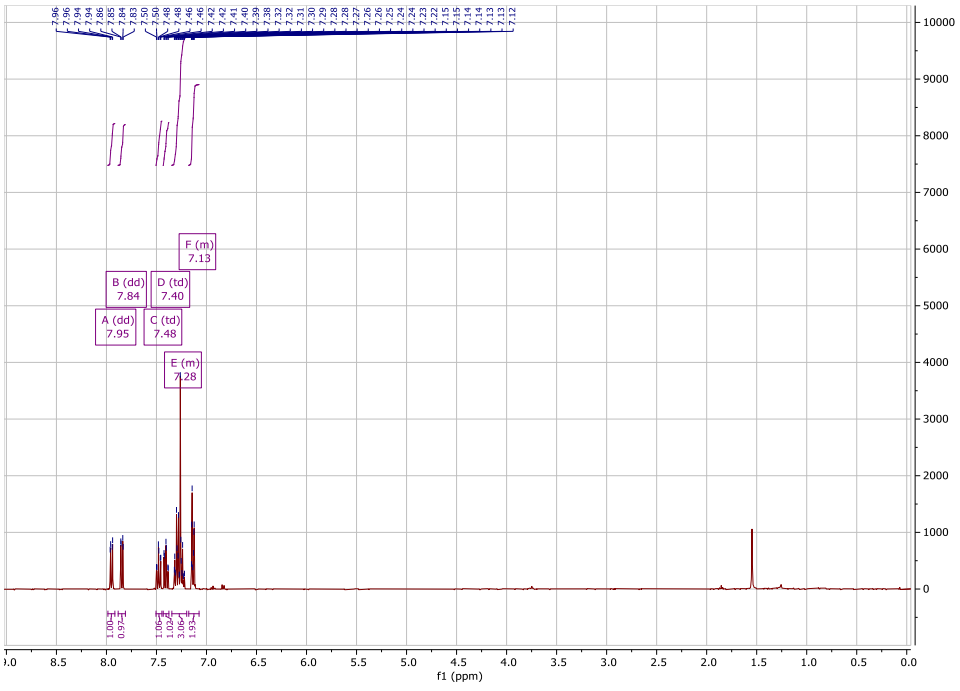


Fig. S2: ^{1}H NMR spectrum of Phenyl 2-bromobenzenesulfonate (**2**)

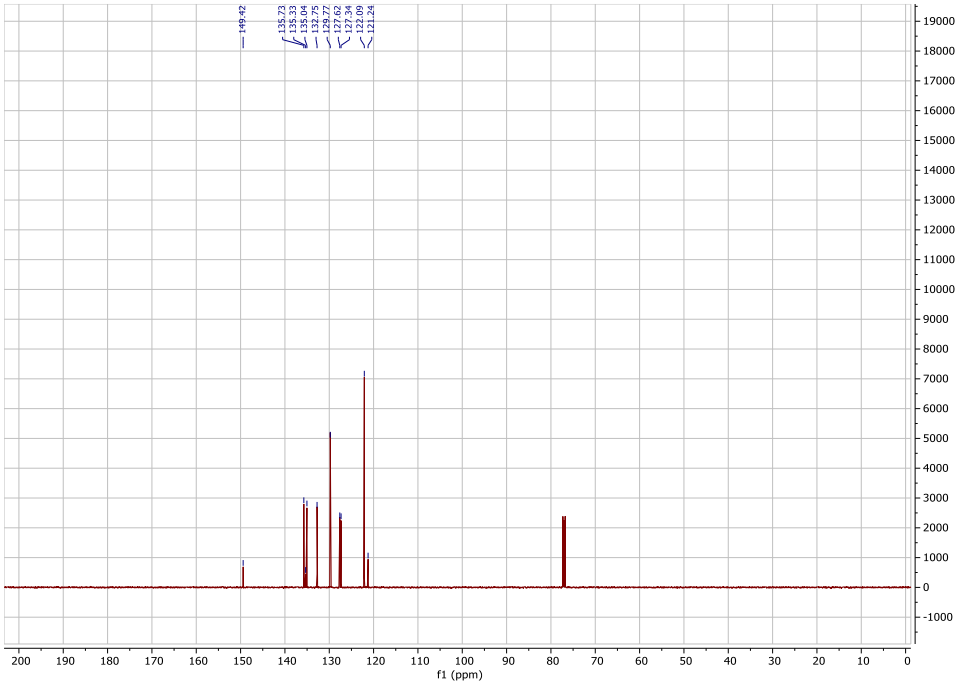


Fig. S3: ^{13}C NMR spectrum of Phenyl 2-bromobenzenesulfonate (**2**)

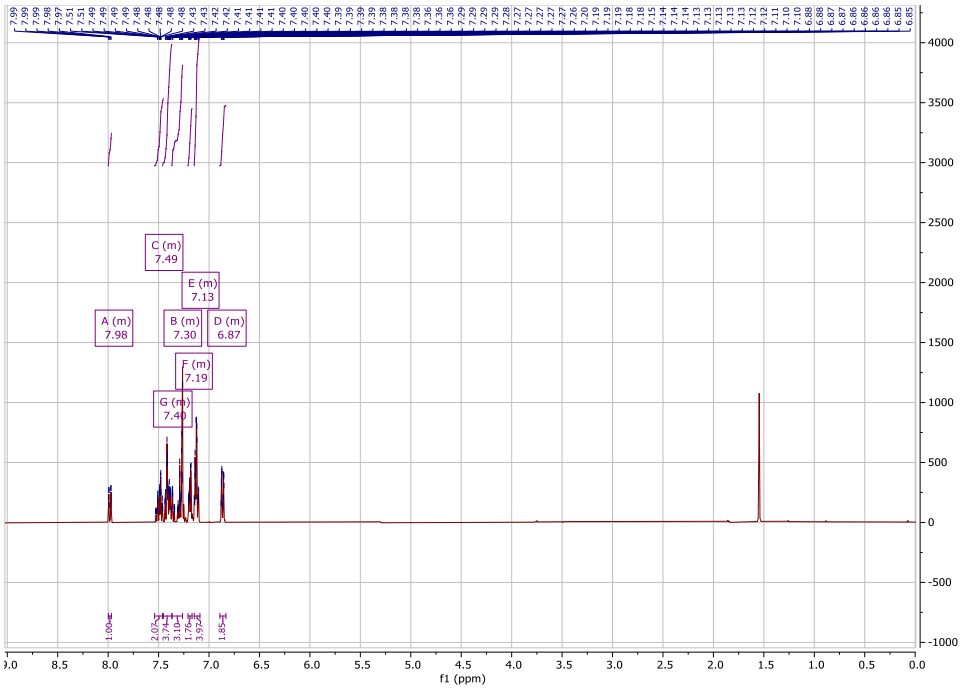


Fig. S4: ^{1}H NMR spectrum of Phenyl [1,1':2',1''-terphenyl]-2-sulfonate (**3**)

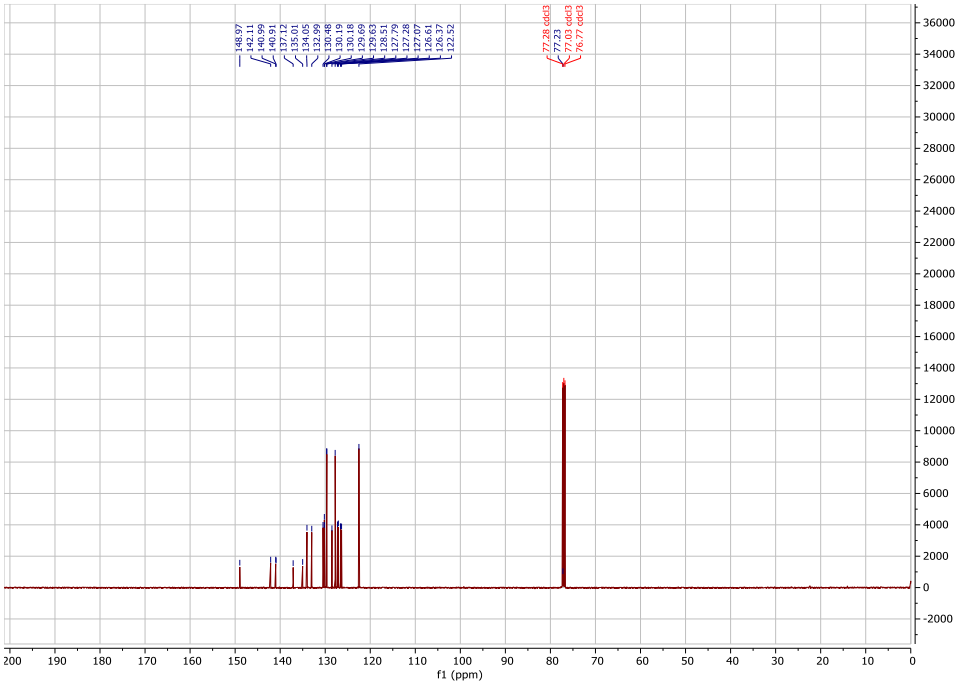


Fig. S5: ^{13}C NMR spectrum of Phenyl [1,1':2',1"-terphenyl]-2-sulfonate (**3**)

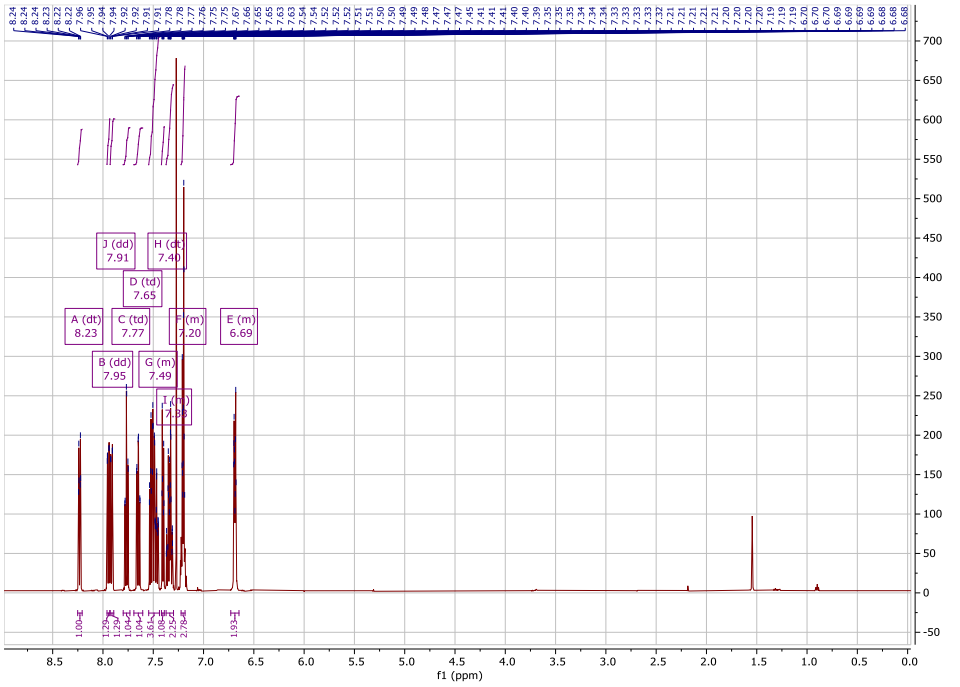


Fig. S6: ^{1}H NMR spectrum of phenyl 2-(naphthalen-1-yl) benzenesulfonate (**8**)

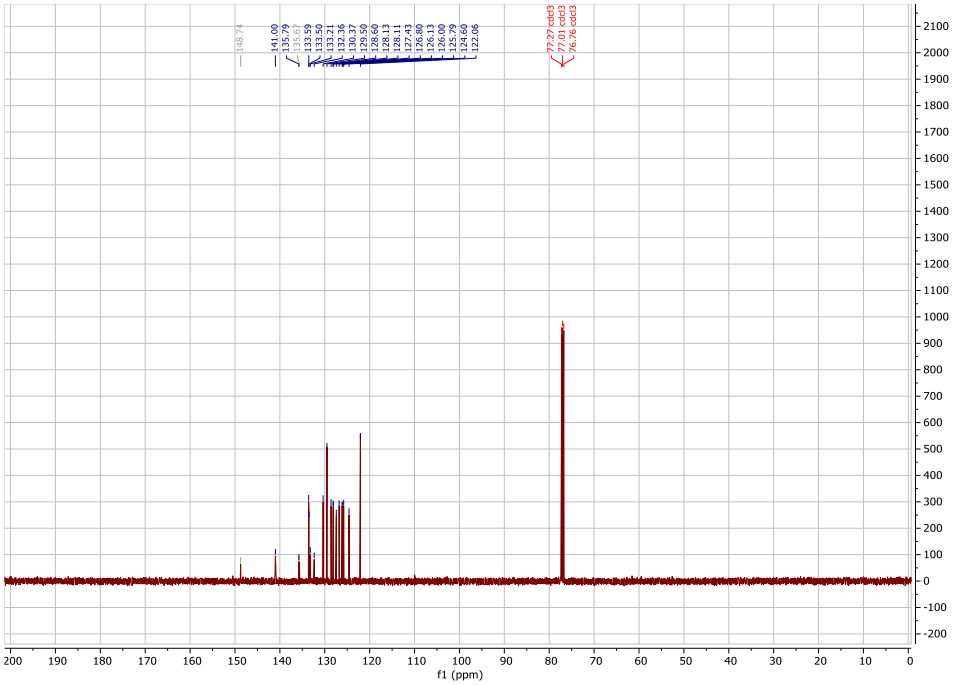


Fig. S7: ^{13}C NMR spectrum of phenyl 2-(naphthalen-1-yl) benzenesulfonate (**8**)

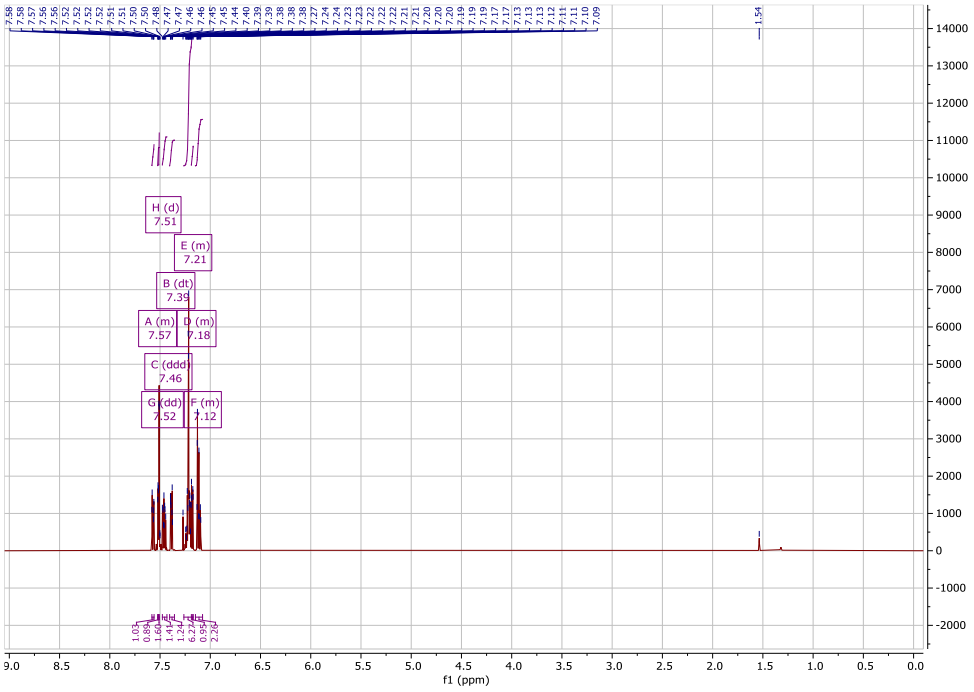


Fig. S8: ^{1}H NMR spectrum of 2-bromo-1,1':2',1''-terphenyl (**5**)

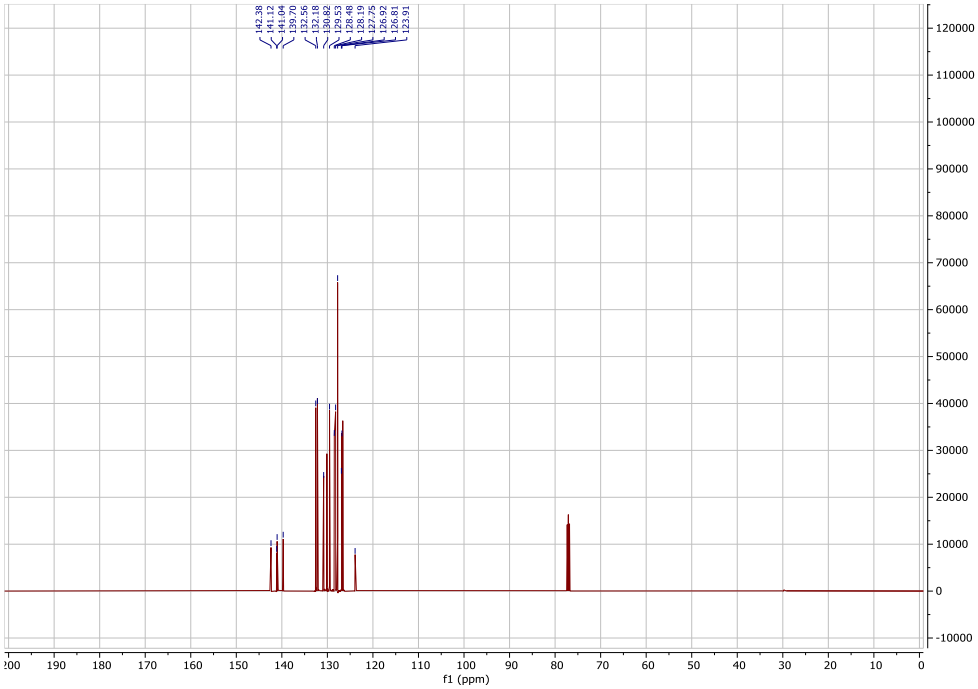


Fig. S9: ^{13}C NMR spectrum of 2-bromo-1,1':2',1"-terphenyl (**5**

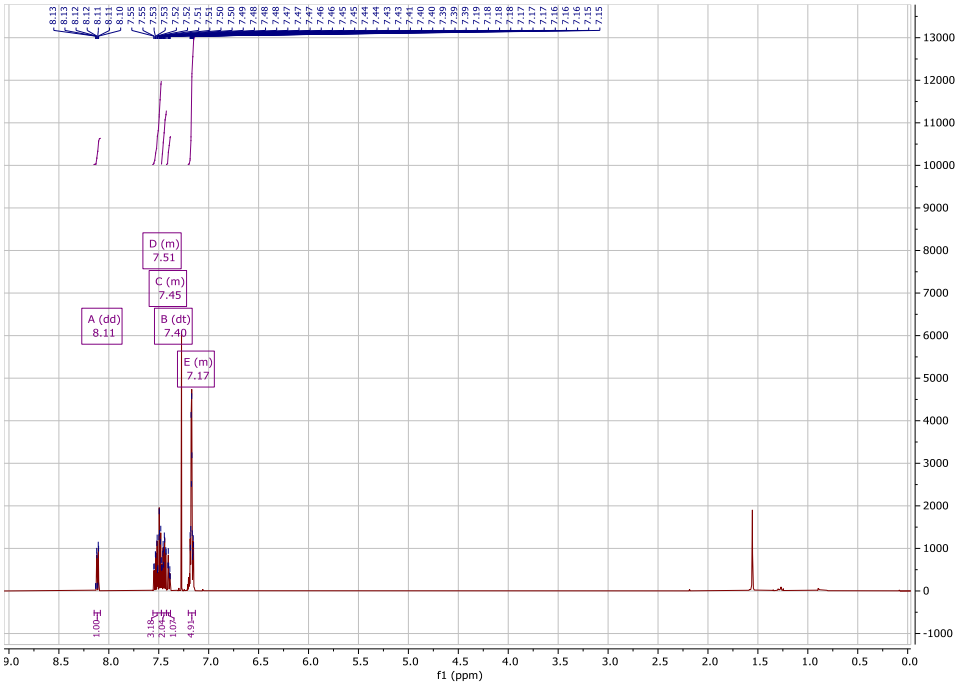


Fig. S10: ^{1}H NMR spectrum of [1,1':2',1"-terphenyl]-2-sulfonyl fluoride (**6**)

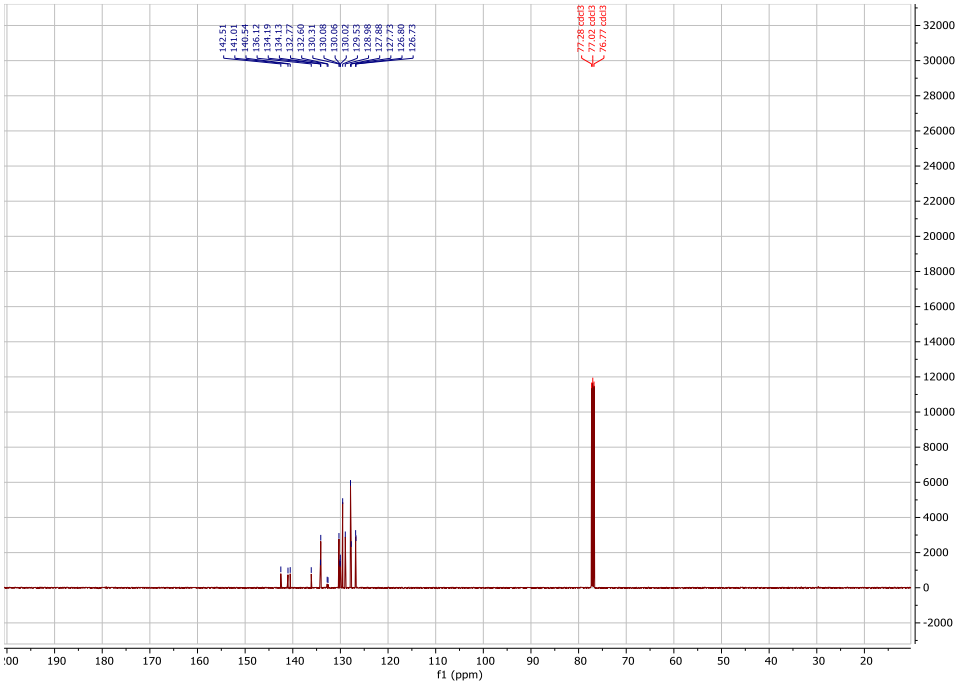


Fig. S11: ^{13}C NMR spectrum of [1,1':2',1"-terphenyl]-2-sulfonyl fluoride (**6**)

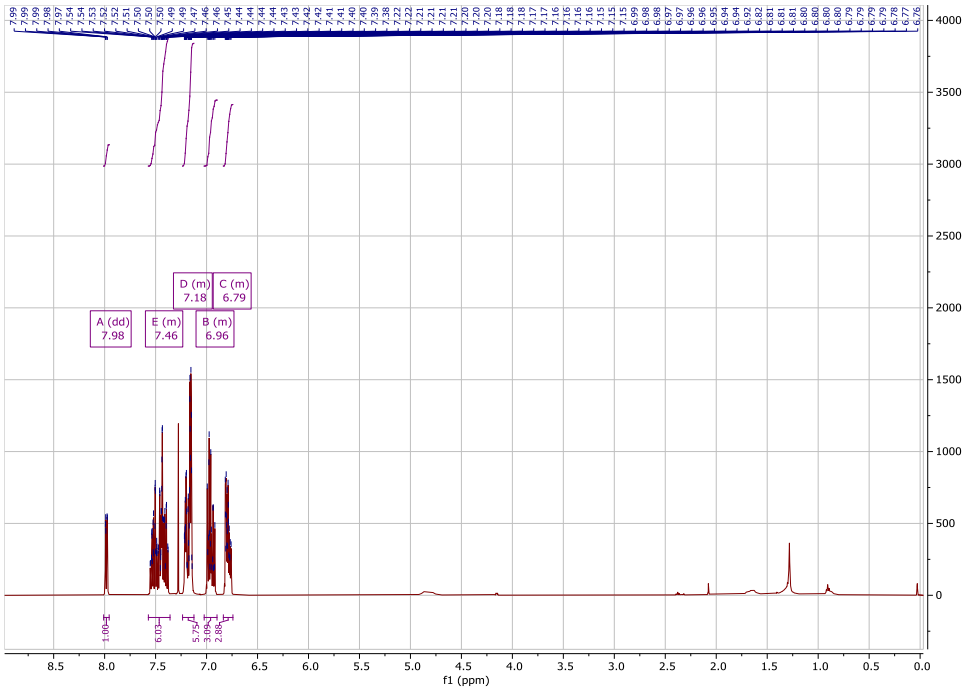


Fig. S12: ^{1}H NMR spectrum of 4-fluorophenyl [1,1':2',1"-terphenyl]-2-sulfonate (**7a**)

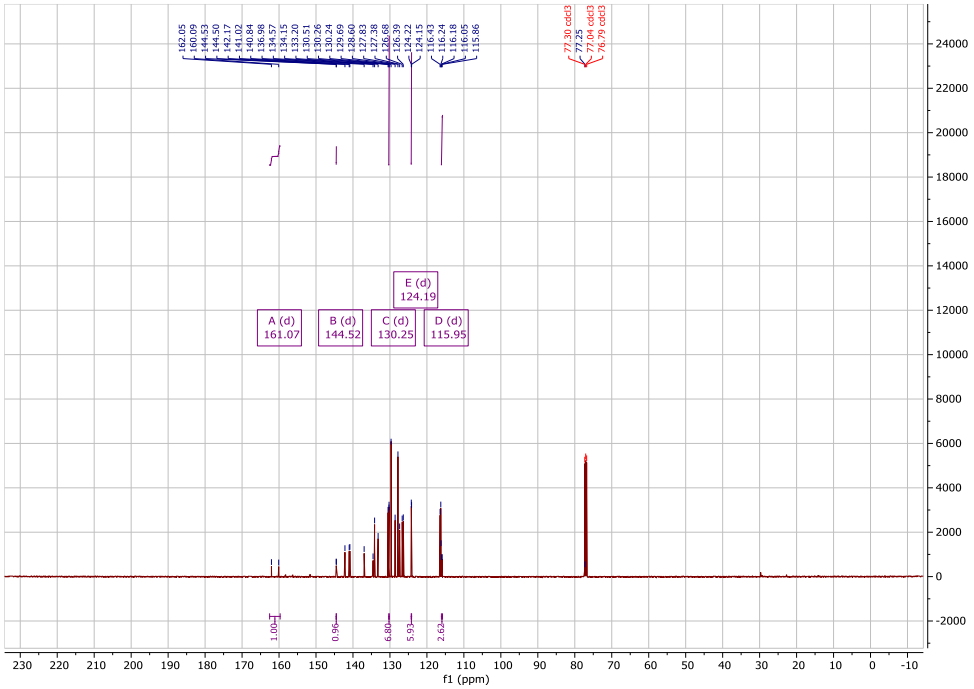


Fig. S13: ^{13}C NMR spectrum of 4-fluorophenyl [1,1':2',1"-terphenyl]-2-sulfonate (**7a**)

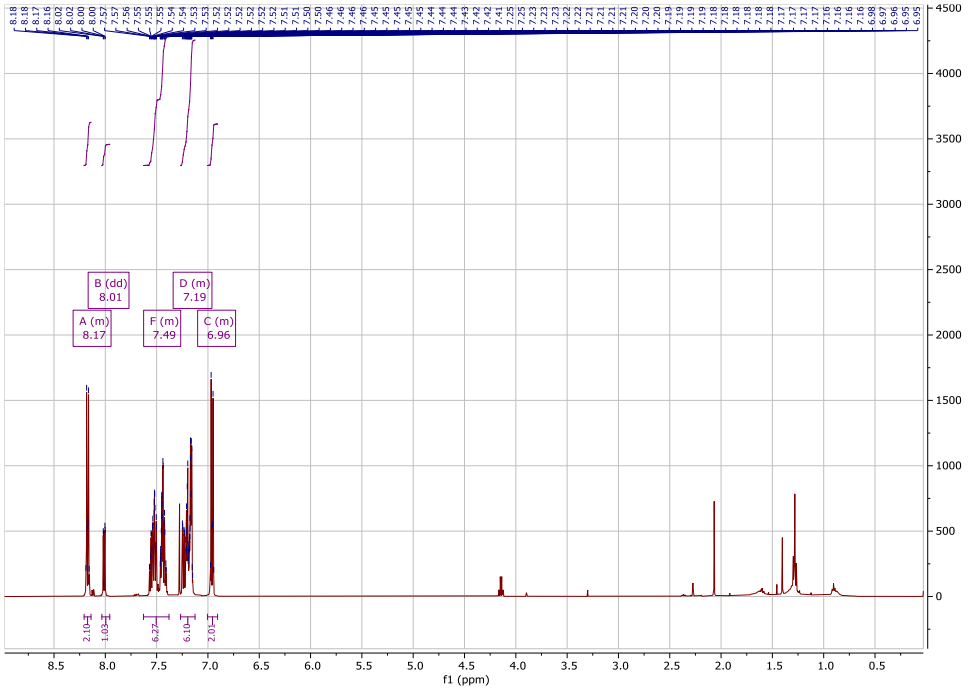


Fig. S14: ^{1}H NMR spectrum of 4-nitrophenyl [1,1':2',1"-terphenyl]-2-sulfonate (**7b**)

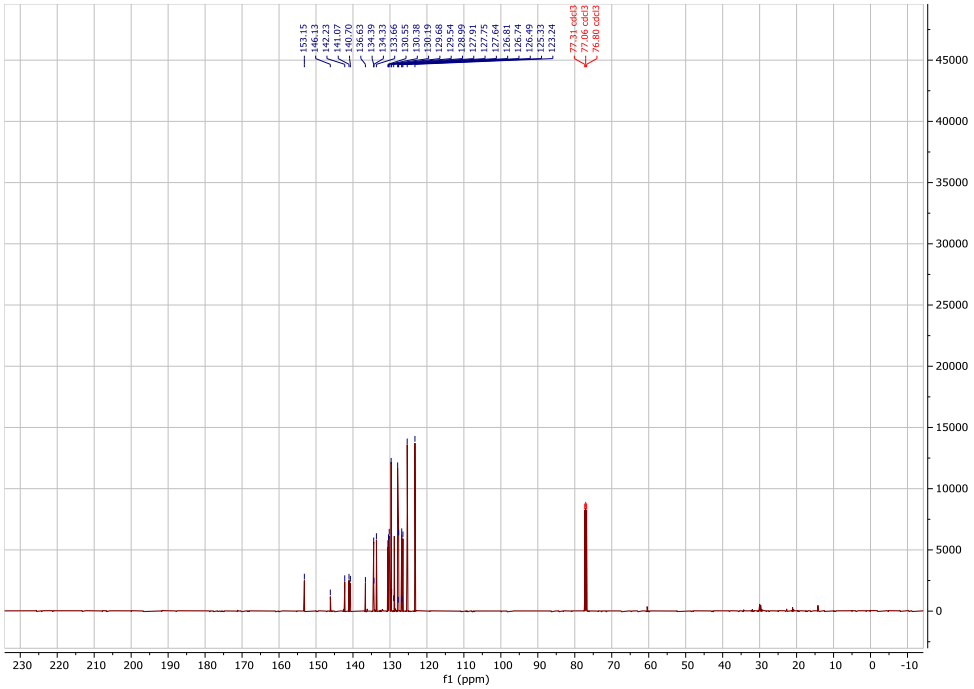


Fig. S15: ^{13}C NMR spectrum of 4-nitrophenyl [1,1':2',1"-terphenyl]-2-sulfonate (**7b**)

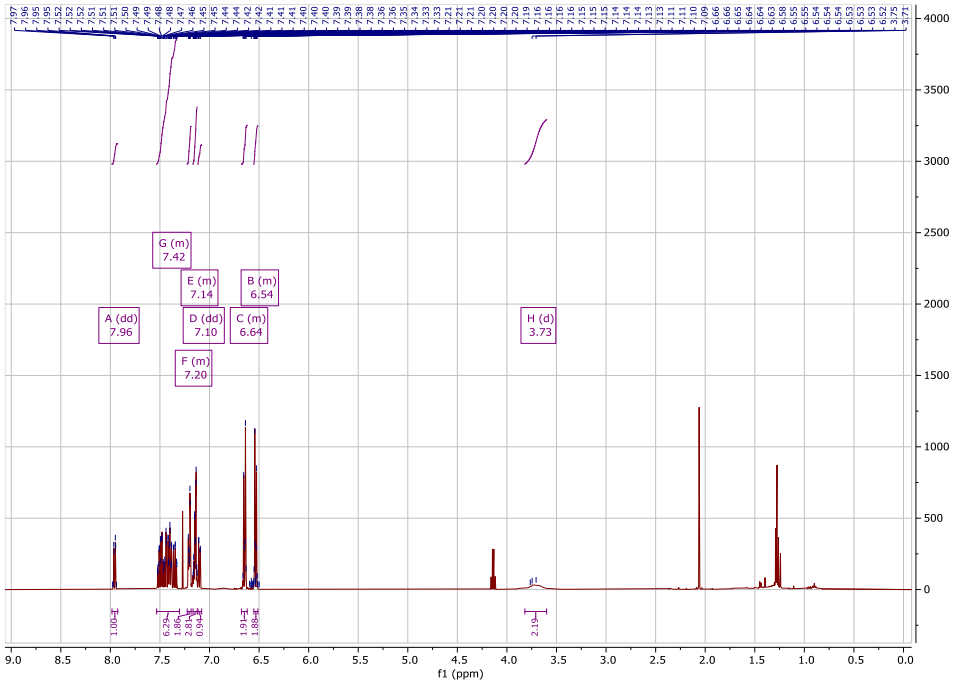


Fig. S16: ^{1}H NMR spectrum of 4-aminophenyl [1,1':2',1"-terphenyl]-2-sulfonate (**7c**)

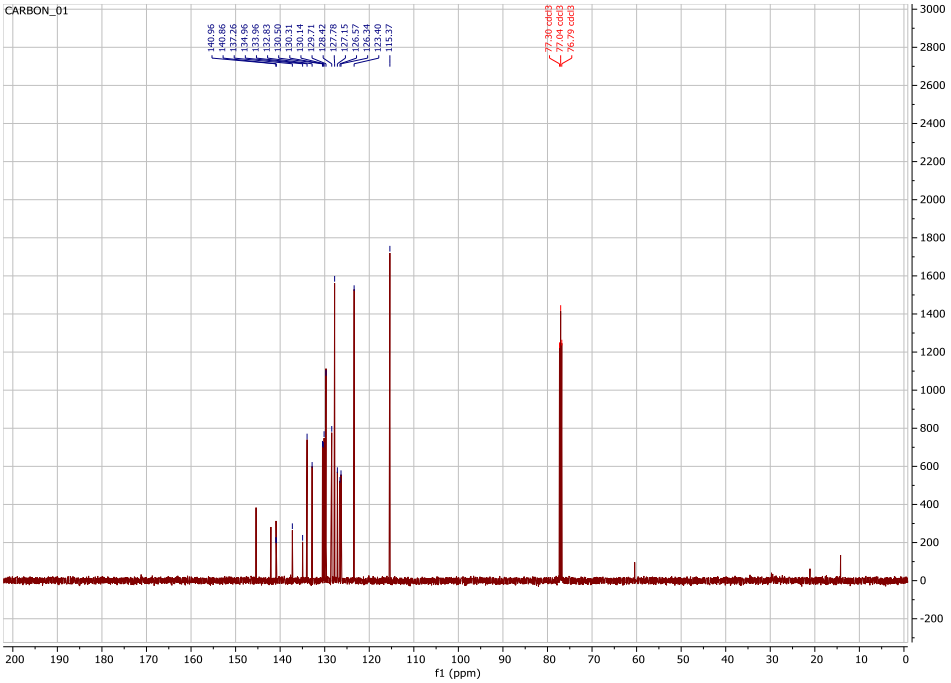


Fig. S17: ^{13}C NMR spectrum of 4-aminophenyl [1,1':2',1"-terphenyl]-2-sulfonate (**7c**)

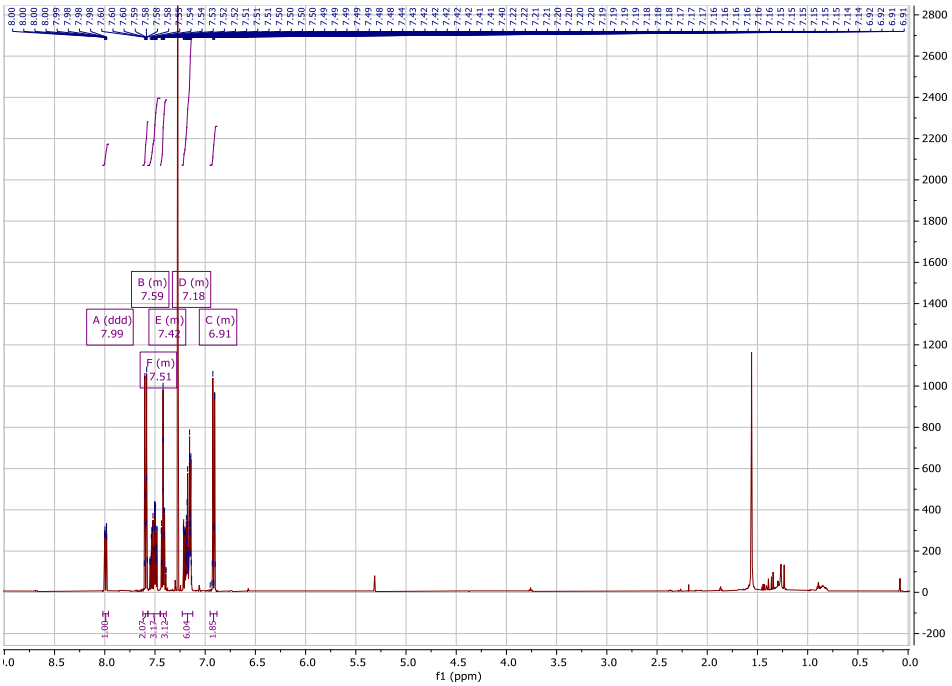


Fig. S18: ^{1}H NMR spectrum of 4-cyanophenyl [1,1':2',1"-terphenyl]-2-sulfonate (**7d**)

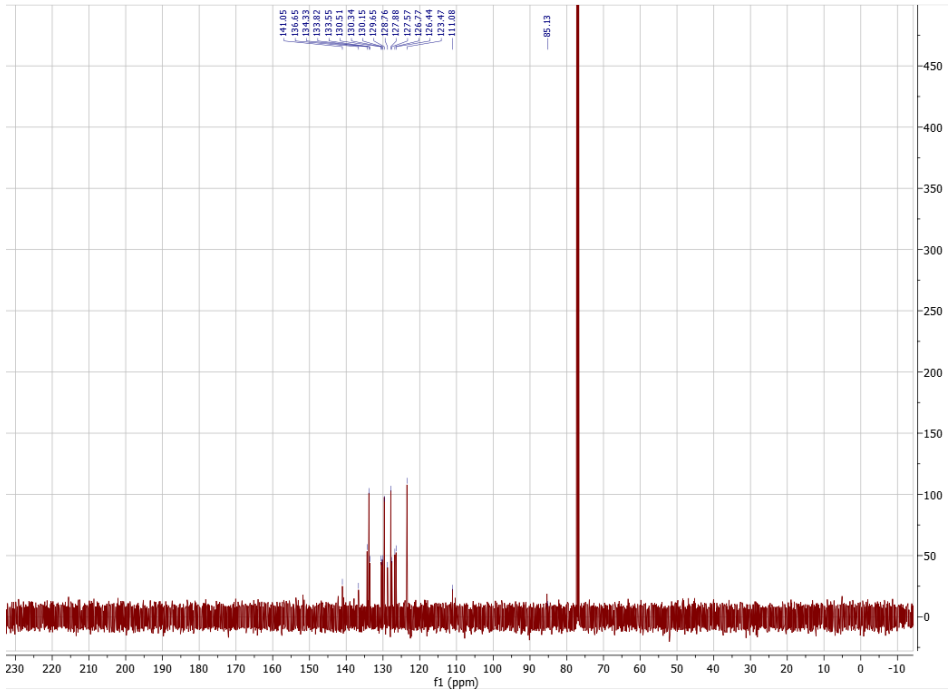


Fig. S19: ^{13}C NMR spectrum of 4-cyanophenyl [1,1':2',1''-terphenyl]-2-sulfonate (**7d**)

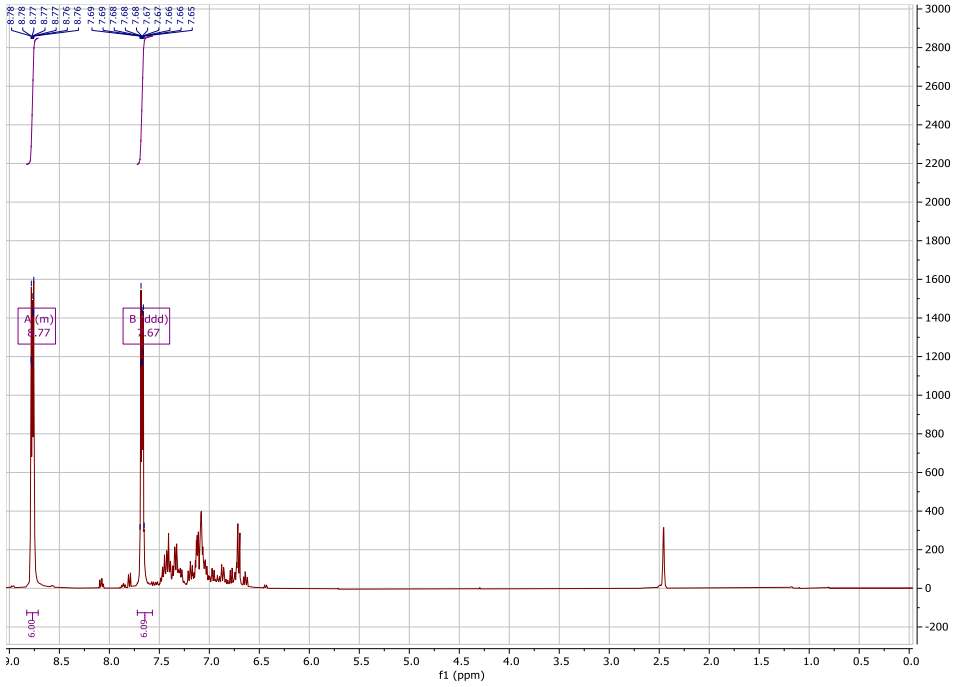


Fig. S20: ^{1}H NMR spectrum of the crude product of (**4**) after photoreaction

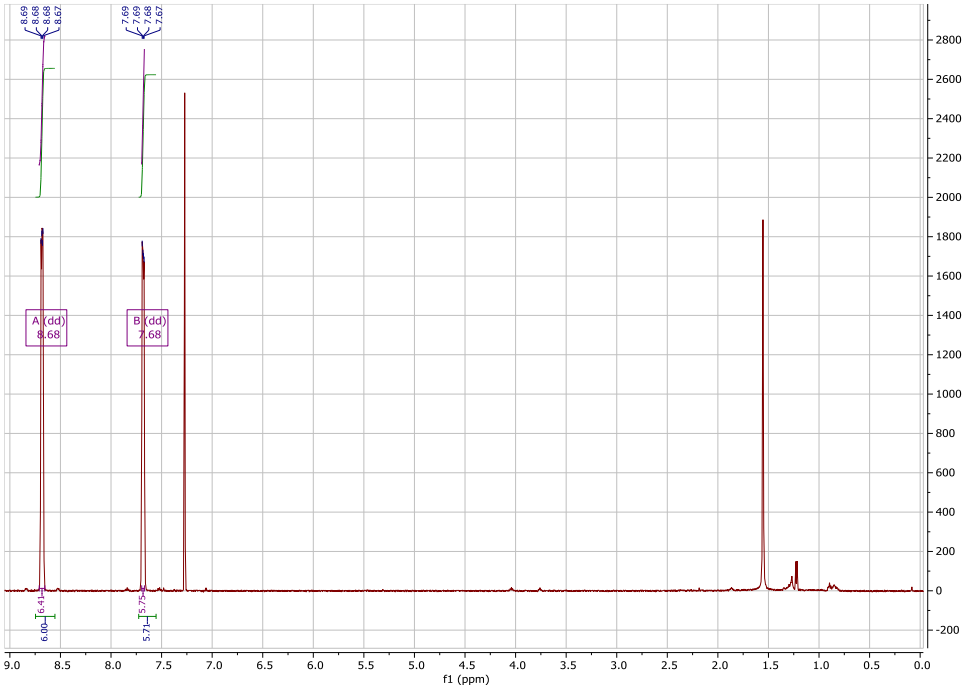


Fig. S21: ^{1}H NMR spectrum of triphenylene (**4**)

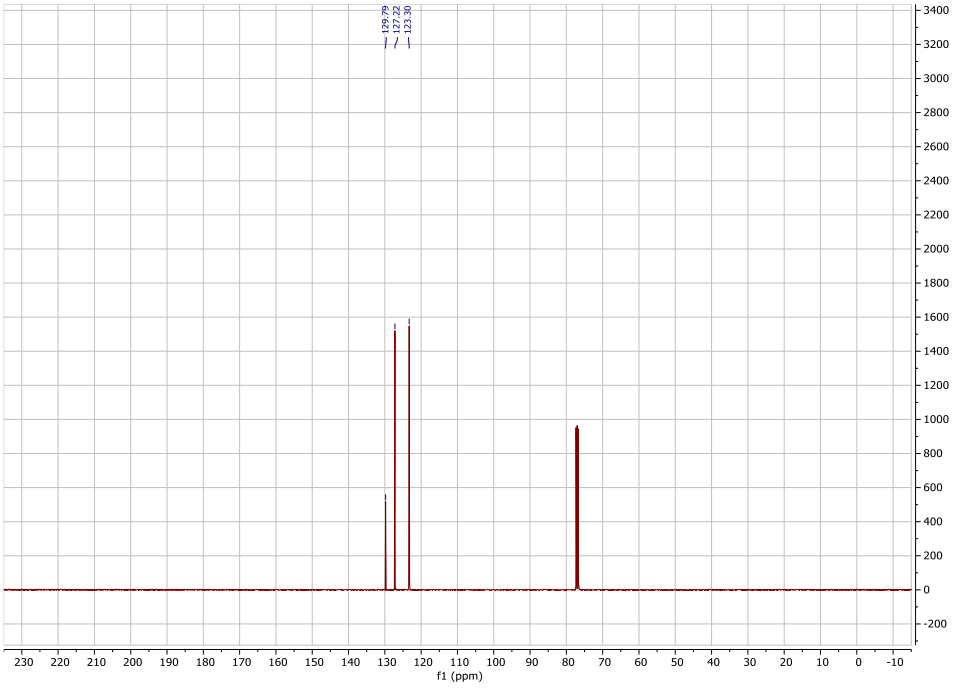


Fig. S22: ^{13}C NMR spectrum of triphenylene (**4**)

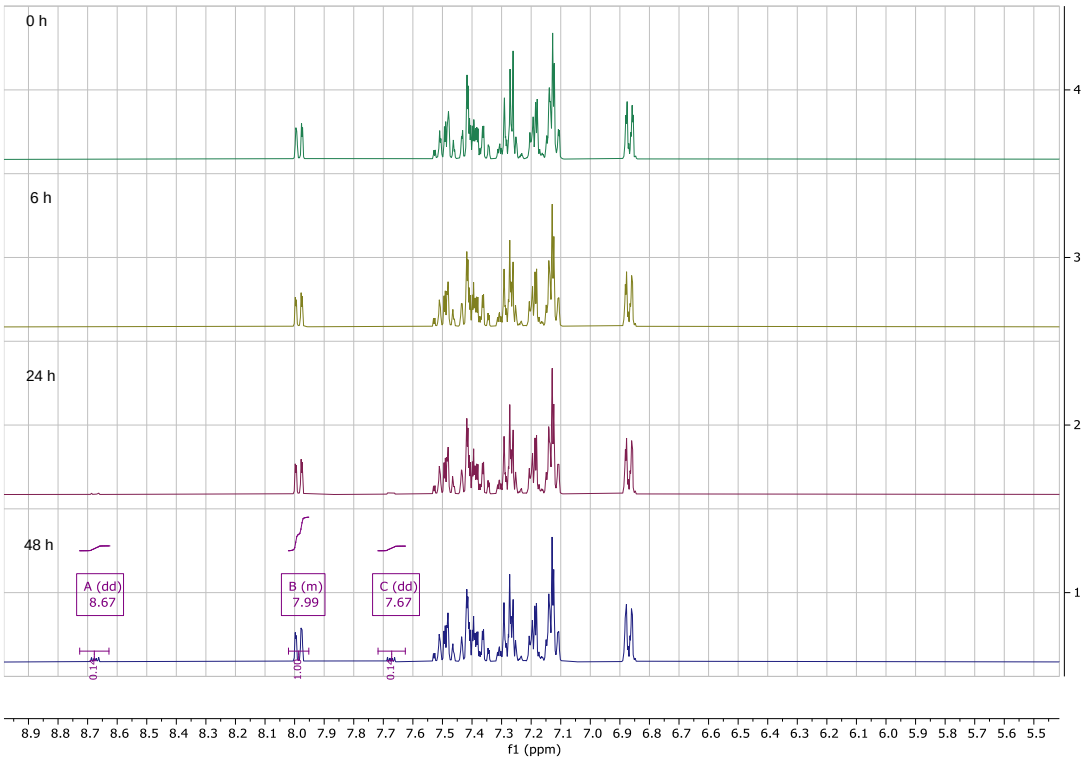


Fig. S23: ^{1}H NMR spectrum of (**3**) at 360 nm without photocatalyst

References

(1) Van Mileghem, S.; De Borggraeve, W. M. *Org. Process Res. Dev.* **2017**, *21*, 785–787.