

# Supporting Information

## Ball Milling-Promoting Difunctionalization of Alkynyl Sulfonium Salts with Sulfinic Acids towards (Z)-1,2-Disulfonylethenes

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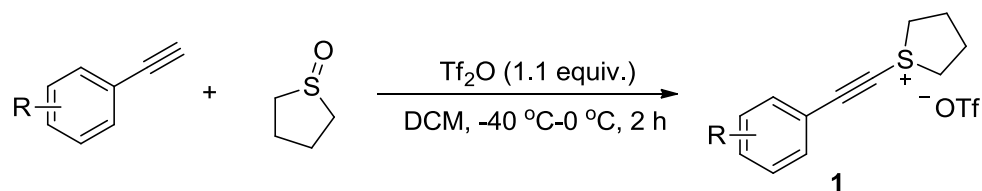
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## 1. General Information

Unless otherwise specified, all reagents and solvents were obtained from commercial suppliers and used without further purification.  $^1\text{H}$ ,  $^{13}\text{C}$  and  $^{19}\text{F}$  NMR spectra were recorded at 400, 101 and 376 MHz, respectively. Chemical shifts were quoted in ppm relative to  $\text{CDCl}_3$  ( $\delta_{\text{H}} = 7.26$ ,  $\delta_{\text{C}} = 77.0$  ppm). Data are reported as follows: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublet, etc. The reactions were monitored by thin-layer chromatography (TLC) using GF254 silica gel-coated TLC plates. Mass spectra were performed on a spectrometer operating on ESI-TOF. Melting points were measured on a melting point apparatus and were uncorrected.

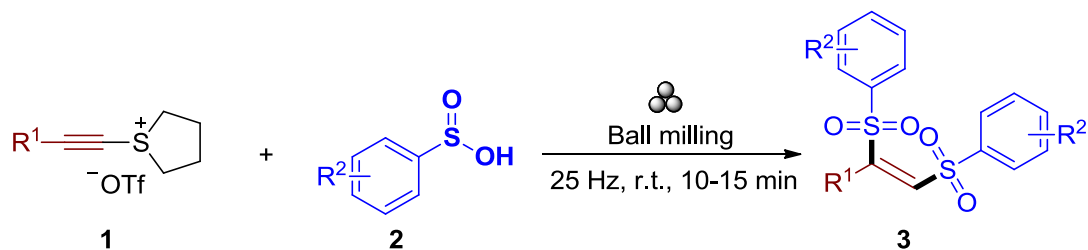
## 2. Experimental Section

### General Procedure for compound 1



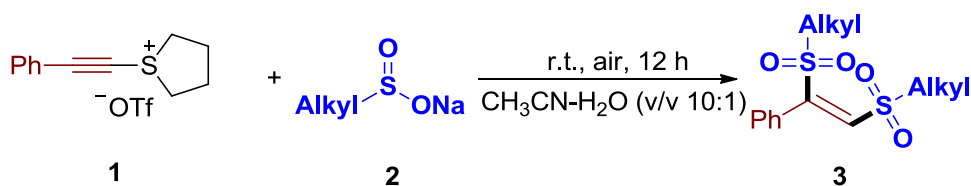
Compound **1** was prepared according to Wen's work.<sup>[1]</sup> Under an argon atmosphere, tetramethylenesulfoxide (1.1 equiv, 5.5 mmol) and anhydrous DCM (20 mL) were added to a 100 mL round bottom flask at  $-40\text{ }^\circ\text{C}$ . The  $\text{Tf}_2\text{O}$  (1.1 equiv, 5.5 mmol) was added dropwise under argon, and then alkene or alkyne (1.0 equiv, 5.0 mmol) was added gradually. The reaction mixture was stirred at  $-40\text{ }^\circ\text{C}$  for 30 min before warming to  $0\text{ }^\circ\text{C}$ . Upon completion monitored by the TLC, the solvent was removed under reduced pressure. The resulted crude product was dissolved in a small amount of anhydrous DCM, which was slowly dropped into anhydrous ether (20 mL) to precipitate out the cyclic sulfonium salts solid. The solid was collected by filtration and washed three times with ether to afford the cyclic sulfonium salt **1**, or the crude product was purified by column chromatography on silica gel (DCM/MeOH, from 50:1 to 20:1) to afford the alkyne sulfonium salt.

### General Procedure for the synthesis of product 3



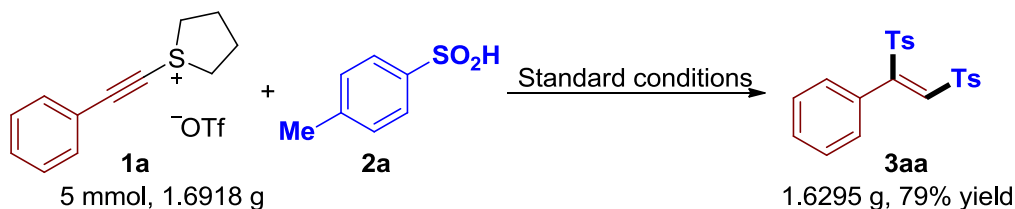
A mixture of alkyne sulfonium salt **1** (0.3 mmol, 1 equiv.), aryl sulfonic acid **2** (0.6 mmol, 2 equiv.) were milled in a stainless steel jar charged with 1 ball (10 mm) of the same material at 25 Hz for 10 – 15 min. After completion, the crude product was directly purified by flash chromatography column over silica gel using a petroleum ether/ethyl acetate eluent mixture (PE/EA 6:1-2:1) to afford the desired product **3**.

### General Procedure for the synthesis of product 3an - 3aq



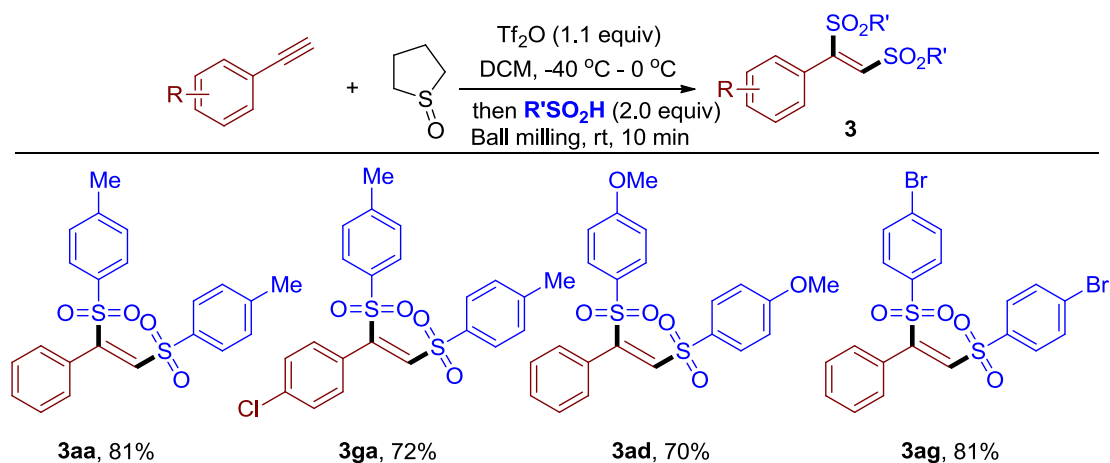
A 10 mL round-bottom flask equipped with a stirring bar was charged with alkynyl sulfonium salt **1a** (0.3 mmol, 1equiv.), sodium alkyl sulfonate **2** (0.6 mmol, 2 equiv.) in CH<sub>3</sub>CN-H<sub>2</sub>O mixture solvent (3 mL, v/v 10:1). The reaction mixture was then stirred at room temperature for about 12 hours, with progress monitored by TLC. Upon completion, 10 mL of H<sub>2</sub>O was added to the mixture, followed by extraction with EtOAc three times (10 mL × 3). The organic phase was subsequently dried over anhydrous sodium sulfate, concentrated under vacuum, and the residue was purified by flash column chromatography using a petroleum ether/ethyl acetate eluent mixture (PE/EA 10:1-6:1) to afford the corresponding product **3** (**3an**, **3ao**, **3ap** and **3aq**).

### Gram-scale synthesis of **3aa**



A mixture of alkynyl sulfonium salt **1a** (1.6918 g, 5 mmol), 4-methylbenzenesulfonic acid **2a** (1.5620 g, 10 mmol) were milled in a stainless steel jar charged with 1 ball (10 mm) of the same material at 25 Hz for 15 min. After completion, the crude product was directly purified by flash chromatography column over silica gel using a petroleum ether/ethyl acetate eluent mixture (PE/EA 6:1-2:1) to afford 1.6295 g of **3aa** in 78% isolated yield.

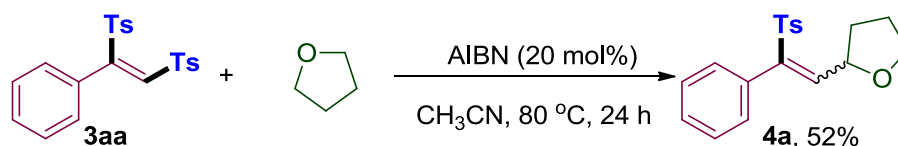
### One-pot synthesis of vinyl sulfone **3**



Under an argon atmosphere, sulfoxide (0.0573 g, 0.55 mmol, 1.1 equiv) and anhydrous DCM (3.0 mL) were added to a 25.0 mL flask at -40 °C. Tf<sub>2</sub>O (0.1551 g, 0.55 mmol, 1.1 equiv) was then added dropwise, followed by the gradual addition of styrene (0.0520 g, 0.5 mmol, 1.0 equiv). The reaction mixture was stirred at -40 °C for 30 min before being warmed to 0 °C. After stirring for 2 h, the reaction mixture was filtered and the solvent was removed under reduced pressure. Then, sulfonic acid **2** (1 mmol, 2.0 equiv.) was added. The reaction mixture was transformed to a stainless steel jar charged with 1 ball (10 mm) of the same material and milled at 25 Hz for 10

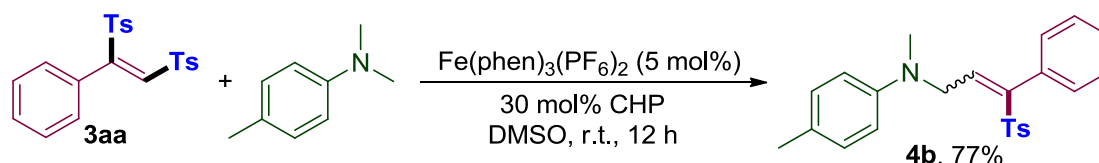
min. After completion, the crude product was directly purified by flash chromatography column over silica gel using a petroleum ether/ethyl acetate eluent mixture (PE/EA 6:1-2:1) to afford the desired product **3aa**, **3ga**, **3ad**, and **3ag** in 81, 72, 70, and 81% yields, respectively.

## Synthesis of 4a



To a stirred solution of **3aa** (0.1238 g, 0.3 mmol, 1.0 equiv.) in  $\text{CH}_3\text{CN}$  (3 mL) was added THF (0.2163 g, 10 equiv.) and AIBN (0.01g, 0.06 mmol). The reaction was heated to 80 °C using an oil bath and stirred for about 24 h. After completion, the reaction was allowed to cool to room temperature, then, 10 mL of  $\text{H}_2\text{O}$  was added to the mixture, followed by extraction with EtOAc three times (10 mL  $\times$  3). The organic phase was then dried with anhydrous sodium sulfate, concentrated under vacuum. The residue was purified by flash column chromatography using a mixture of petroleum ether and ethyl acetate as eluent (PE/EA 10:1-6:1) to obtain 0.0512 g of product **4a** in 52% yield.

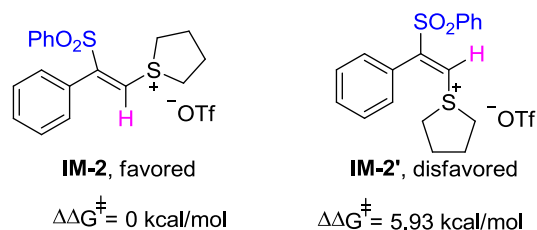
## Synthesis of 4b



Product **4b** was synthesis according to Kang's work.<sup>[2]</sup> A solution of vinyl sulfone **3aa** (206.3 mg, 0.5 mmol) in DMSO (5 mL) was added *N,N*-trimethylaniline (338 mg, 2.5 mmol, 5 equiv.),  $\text{Fe}(\text{phen})_3(\text{PF}_6)_3$  (26 mg, 0.025 mmol, 5 mol%) and cumene hydroperoxide (30  $\mu\text{L}$ , 0.15 mmol, 30 mol %). The reaction mixture was stirred at room temperature for 12 h under  $\text{N}_2$  atmosphere. After completion, silica gel column chromatography of the crude mixture provided the desired product **4b** in 77% yield (150.6 mg).

## Density functional theory calculations

All data in this study were calculated with the Gaussian 16 software package<sup>[3]</sup> and were optimized at the M062X/6-311G(d, p) level of density functional theory (DFT). Vibrational frequency analysis was computed to ensure the points that the minimum have no imaginary frequency and the transition states have only one imaginary frequency. The single-point energy calculations were calculated by using the M062X/6-311++G(d, p). The single-point energy corrected relative free energies in kcal/mol are used for discussion throughout the text. The DFT calculations indicate that the free energy of **IM-2'** is 5.93 kcal/mol higher than that of **IM-2**.



## Cartesian Coordinates for Optimized Structures

**1a**

C	5.12835700	-2.00532800	0.34028700
C	3.80956300	-2.39545700	0.12142200
C	2.82527500	-1.44137200	-0.09451900
C	3.17880200	-0.08452500	-0.08560900
C	4.50097200	0.31135100	0.13559500
C	5.47319200	-0.65585100	0.34693300
H	5.89205300	-2.75586200	0.50604000
H	3.54565100	-3.44577700	0.11662600
H	1.78806300	-1.71049500	-0.26831900
H	4.75563700	1.36403700	0.13817700
H	6.50061500	-0.35835100	0.51687700
C	2.12454100	0.84847600	-0.31035900
C	1.15576000	1.54033400	-0.50035600
C	-1.27514100	1.87197800	0.79313000
C	-0.25302200	3.84823900	-0.70308700
C	-1.34416400	3.27909600	1.39082900
H	-0.66219000	1.15923000	1.33979000
H	-2.23381100	1.41401600	0.55647100
C	-0.21250400	4.11936200	0.79438400
H	-1.14803500	4.25979500	-1.17420800
H	0.62989000	4.14652100	-1.26522500
H	-2.30613000	3.73493700	1.14558300
H	-1.27388700	3.21746000	2.47731200
H	-0.34017000	5.18348200	0.99853500
H	0.75478900	3.80708300	1.19654500
S	-0.42620900	2.02490300	-0.85594000
S	-1.88025000	-1.14133500	-0.86406400
O	-0.53160200	-0.68763800	-0.44999500
O	-1.91532100	-2.23495700	-1.81089300
O	-2.79038800	-0.01400600	-1.10191700
C	-2.52819500	-1.86275500	0.71431400
F	-1.76666700	-2.86959400	1.13305300
F	-2.53052200	-0.92505200	1.67785100
F	-3.77202600	-2.30481600	0.57511200

**IM-2**

C	1.36267900	0.73628000	0.08389300
C	0.15615900	1.29554800	0.01904700
S	-2.29976900	-0.72551000	1.79593500
O	-1.10321000	-1.52238800	1.49597400
O	-3.51196400	-1.13790100	1.09737400
O	-2.04978000	0.72770100	1.78080600
C	-2.61472100	-1.08009900	3.58250200

F	-2.86023500	-2.37121600	3.76975200
F	-1.54266800	-0.74681200	4.30460300
F	-3.65286600	-0.38081800	4.02641300
C	2.13707100	0.69880900	1.34563100
C	3.49148100	1.04820800	1.37250700
C	1.48984000	0.30073500	2.51878200
C	4.18269500	1.01327400	2.57671400
H	3.99135900	1.35247300	0.46110800
C	2.19328000	0.26795600	3.71631300
H	0.45279600	-0.01049100	2.47815100
C	3.53713500	0.62512100	3.74751300
H	5.22877200	1.29386600	2.60030000
H	1.68752500	-0.04695400	4.62106400
H	4.08411800	0.59666900	4.68250200
S	2.08929900	-0.10844000	-1.33866600
O	3.32165200	0.56365700	-1.71820300
O	1.01281500	-0.23406100	-2.32132900
C	2.48124300	-1.70298100	-0.66371800
C	3.77950500	-2.17817700	-0.78744900
C	1.47492900	-2.40949700	-0.01539500
C	4.06992300	-3.43181700	-0.26061700
H	4.53205200	-1.57452200	-1.28000400
C	1.78882400	-3.65161300	0.52049300
H	0.47968100	-1.99531000	0.11533400
C	3.07857200	-4.16170600	0.38960600
H	5.07196600	-3.83278500	-0.34962700
H	1.02393000	-4.20955500	1.04608300
H	3.31621200	-5.13255200	0.80818200
S	-0.89943700	1.43288800	-1.42006700
C	-2.35750200	2.34053500	-0.75436200
C	-1.80202100	-0.16070100	-1.64520400
C	-3.57032000	1.40497600	-0.87494000
H	-2.14720100	2.60744700	0.27959900
C	-3.24541600	0.27663900	-1.86028200
H	-1.64957800	-0.75165500	-0.74334900
H	-1.33151100	-0.64474200	-2.49610800
H	-3.78584900	0.98091200	0.10257500
H	-4.43991700	1.97230600	-1.20866700
H	-3.89539000	-0.58169900	-1.68170500
H	-3.37942600	0.60673600	-2.89409300
H	-2.43321000	3.23389000	-1.37261900
H	-0.27679000	1.75879300	0.89797900

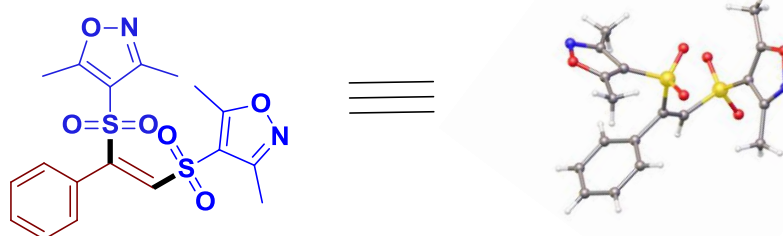
IM-2'

C	2.41501300	0.45177100	-0.59953900
C	1.44086000	1.03622600	-1.28835500
S	-1.94748600	0.11334600	-0.70500700
O	-1.10144000	-0.95911100	-1.22034300
O	-3.38193700	-0.05963200	-0.78072000
O	-1.44706900	1.45794000	-1.07145700
C	-1.58409900	0.10047900	1.11498700
F	-1.98552000	-1.02066200	1.69750800
F	-0.25636200	0.22371800	1.32637700
F	-2.16922000	1.13114400	1.73022700
C	3.30530800	1.10504500	0.38158500
C	4.67979500	1.21776500	0.15083700
C	2.75210100	1.60664200	1.56434500
C	5.48260100	1.85510100	1.08757200
H	5.10589400	0.79814200	-0.75130000
C	3.56594500	2.23456600	2.50126500
H	1.69563700	1.46264500	1.76030100
C	4.92929400	2.36392900	2.26032100
H	6.54613100	1.94876700	0.90459800
H	3.13587500	2.60985500	3.42205800
H	5.56404500	2.85225000	2.99015000
S	2.66658800	-1.32694600	-0.90348800
O	4.11377400	-1.49464800	-0.91749300
O	1.85762700	-1.69001100	-2.04902500
C	2.01558900	-2.08251800	0.56126900
C	2.79448700	-2.07326000	1.71580300
C	0.74338400	-2.63820000	0.51348500
C	2.26469100	-2.63556500	2.86945400
H	3.79245200	-1.65009800	1.69686600
C	0.23470800	-3.20354800	1.67799300
H	0.16657800	-2.59804600	-0.40266200
C	0.98898900	-3.19733400	2.84690800
H	2.84861300	-2.64468800	3.78158100
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H	0.58168000	-3.63581200	3.75042700
S	1.37710600	2.82049500	-1.26551400
C	0.23287300	3.27617000	-2.61293500
C	0.25942300	3.43260300	0.07876200
C	-0.41743300	4.54265400	-2.06234600
H	0.82548500	3.42898600	-3.51287200
C	-0.84466200	4.24411000	-0.61807500
H	0.91454200	4.02028600	0.72083800
H	-0.12341700	2.56078300	0.60109100
H	0.29252700	5.37422400	-2.09653200

H	-1.27439500	4.81024000	-2.68145700
H	-1.02547500	5.16765300	-0.06713000
H	-1.74953500	3.64078000	-0.62275300
H	-0.48007800	2.45866000	-2.71438900
H	0.72891700	0.51295200	-1.92200800

### Crystal data of **3am**

The crystal was obtained by slow evaporation of **3am** in a mixture of petroleum ether/CHCl<sub>3</sub>. CCDC 2393039 (**3am**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre *via* [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif).



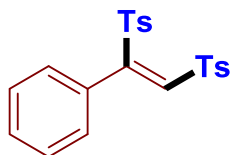
**Table S1 Crystal data and structure refinement of **3am****

Identification code	22
Empirical formula	C <sub>18</sub> H <sub>18</sub> N <sub>2</sub> O <sub>6</sub> S <sub>2</sub>
Formula weight	422.46
Temperature / K	296(2)
Crystal system	monoclinic
Space group	P2 <sub>1</sub> /c
a / Å, b / Å, c / Å	12.909(2), 10.4158(18), 15.096(3)
α/°, β/°, γ/°	90.00, 96.127(2), 90.00
Volume / Å <sup>3</sup>	2018.2(6)
Z	4
ρ <sub>calc</sub> / mg mm <sup>-3</sup>	1.390
μ / mm <sup>-1</sup>	0.301
F(000)	880
Crystal size / mm <sup>3</sup>	0.31 × 0.26 × 0.08
2θ range for data collection	3.18 to 55.18 °
Index ranges	-16 ≤ h ≤ 16, -13 ≤ k ≤ 13, -19 ≤ l ≤ 19
Reflections collected	22291
Independent reflections	4628[R(int) = 0.0366]
Data/restraints/parameters	4628/0/257
Goodness-of-fit on F <sup>2</sup>	1.028

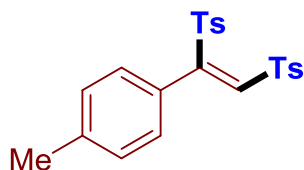


Final R indexes [ $I > 2\sigma$ (I) i.e. $F_o > 4\sigma$ ( $F_o$ )]	R1 = 0.0477, wR2 = 0.1163
Final R indexes [all data]	R1 = 0.0741, wR2 = 0.1316
Largest diff. peak/hole / $e \text{ \AA}^{-3}$	0.237/-0.282
Flack Parameters	N/A
Completeness	0.989

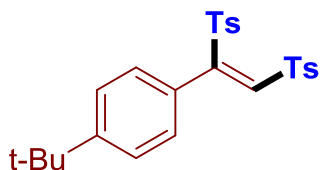
### 3. Characterization data of products



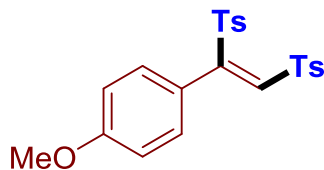
(*Z*)-4,4'-(1-phenylethene-1,2-diyl)disulfonylbis(methylbenzene) (**3aa**): TLC (PE/EtOAc, 3:1),  $R_f$  = 0.52; White solid (105.2 mg, 85%). mp: 113 - 114 °C.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.91 (d,  $J$  = 7.3 Hz, 1H), 7.55 (d,  $J$  = 8.1 Hz, 1H), 7.30 (t,  $J$  = 7.9 Hz, 1H), 7.20 (dd,  $J$  = 8.7, 6.5 Hz, 1H), 7.13 (d,  $J$  = 8.2 Hz, 1H), 6.77 (s, 1H), 2.40 (s, 1H), 2.33 (d,  $J$  = 5.4 Hz, 1H).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.91 (d,  $J$  = 8.0 Hz, 2H), 7.55 (d,  $J$  = 8.0 Hz, 2H), 7.32 - 7.28 (m, 3H), 7.22 - 7.18 (m, 2H), 7.16 - 7.12 (m, 4H), 6.77 (s, 1H), 2.40 (s, 3H), 2.32 (s, 3H).  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  152.1, 145.6, 145.2, 140.9, 138.6, 135.3, 132.7, 130.3, 129.8, 129.7, 129.7, 129.3, 128.4, 128.4, 21.8. HRMS (ESI):  $m/z$  [ $\text{M}+\text{Na}$ ] $^+$  calcd for  $\text{C}_{22}\text{H}_{20}\text{NaO}_4\text{S}_2$ : 435.0695; found: 435.0702.



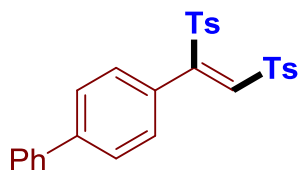
(*Z*)-4,4'-(1-(*p*-tolyl)ethene-1,2-diyl)disulfonylbis(methylbenzene) (**3ba**): TLC (PE/EtOAc, 3:1),  $R_f$  = 0.55; Colorless liquid (110.0 mg, 86%).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.99 (d,  $J$  = 7.9 Hz, 2H), 7.65 (d,  $J$  = 7.9 Hz, 2H), 7.40 (d,  $J$  = 8.0 Hz, 2H), 7.23 (d,  $J$  = 8.0 Hz, 2H), 7.15 (d,  $J$  = 7.9 Hz, 2H), 7.10 (d,  $J$  = 7.9 Hz, 2H), 6.85 (s, 1H), 2.49 (s, 3H), 2.41 (s, 3H), 2.35 (s, 3H);  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  152.3, 145.6, 145.1, 140.8, 140.7, 138.8, 135.5, 129.9, 129.8, 129.7, 129.7, 129.4, 129.1, 128.5, 21.9, 21.4. HRMS (ESI):  $m/z$  [ $\text{M}+\text{Na}$ ] $^+$  calcd for  $\text{C}_{23}\text{H}_{22}\text{NaO}_4\text{S}_2$ : 449.0852; found: 449.0856.



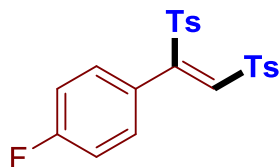
(*Z*)-4,4'-(1-(4-(*tert*-butyl)phenyl)ethene-1,2-diyl)disulfonylbis(methylbenzene) (**3ca**): TLC (PE/EtOAc, 3:1),  $R_f$  = 0.63; White solid (105.4 mg, 75%). mp: 103 - 104 °C.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.96 (d,  $J$  = 8.0 Hz, 2H), 7.65 (d,  $J$  = 8.0 Hz, 2H), 7.37 (d,  $J$  = 8.0 Hz, 2H), 7.28 (d,  $J$  = 8.3 Hz, 2H), 7.21 (d,  $J$  = 8.0 Hz, 2H), 7.16 (d,  $J$  = 8.1 Hz, 2H), 6.83 (s, 1H), 2.46 (s, 3H), 2.39 (s, 3H), 1.27 (s, 9H);  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  153.6, 153.2, 145.7, 145.2, 138.0, 136.3, 133.5, 130.0, 129.8, 129.7, 129.3, 128.3, 124.8, 123.9, 34.9, 31.3, 31.2, 21.9, 21.8. HRMS (ESI):  $m/z$  [ $\text{M}+\text{Na}$ ] $^+$  calcd for  $\text{C}_{26}\text{H}_{28}\text{NaO}_4\text{S}_2$ : 491.1321; found: 491.1324.



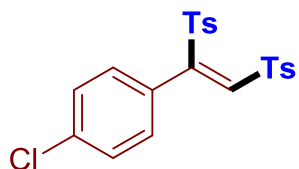
(*Z*)-4,4'-(1-(4-methoxyphenyl)ethene-1,2-diyl)disulfonylbis(methylbenzene) (**3da**): TLC (PE/EtOAc, 3:1),  $R_f = 0.44$ ; Colorless liquid (82.3 mg, 62%).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.89 (d,  $J = 7.8$  Hz, 2H), 7.54 (d,  $J = 7.8$  Hz, 2H), 7.30 (d,  $J = 7.8$  Hz, 2H), 7.15 - 7.12 (m, 4H), 6.75 (s, 1H), 6.71 (d,  $J = 8.2$  Hz, 2H), 3.71 (s, 3H), 2.39 (s, 3H), 2.31 (s, 3H);  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  161.4, 151.9, 145.5, 145.0, 140.2, 138.8, 135.6, 131.5, 129.8, 129.7, 129.3, 128.5, 124.7, 113.9, 55.5, 21.8. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{23}\text{H}_{22}\text{NaO}_5\text{S}_2$ : 465.0801; found: 465.0805.



(*Z*)-4-(1,2-ditosylvinyl)-1,1'-biphenyl (**3ea**): TLC (PE/EtOAc, 3:1),  $R_f = 0.68$ ; White solid (105.5 mg, 72%). mp: 119 - 120 °C.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.92 (d,  $J = 8.0$  Hz, 2H), 7.61 (d,  $J = 8.0$  Hz, 2H), 7.48 (d,  $J = 8.4$  Hz, 2H), 7.43 (d,  $J = 8.0$  Hz, 2H), 7.37 (t,  $J = 7.5$  Hz, 2H), 7.32 (d,  $J = 8.3$  Hz, 2H), 7.24 (d,  $J = 8.0$  Hz, 2H), 7.20 - 7.12 (m, 3H), 6.82 (s, 1H), 2.40 (s, 3H), 2.32 (s, 3H);  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  152.0, 145.7, 145.2, 143.2, 141.0, 139.7, 138.7, 135.5, 131.6, 130.3, 129.9, 129.8, 129.5, 129.1, 128.6, 128.2, 127.2, 127.0, 21.9. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{28}\text{H}_{24}\text{NaO}_4\text{S}_2$ : 511.1008; found: 511.1016.

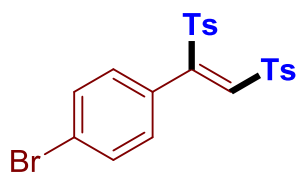


(*Z*)-4,4'-(1-(4-fluorophenyl)ethene-1,2-diyl)disulfonylbis(methylbenzene) (**3fa**): TLC (PE/EtOAc, 3:1),  $R_f = 0.54$ ; Colorless liquid (107.2 mg, 83%).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.97 (d,  $J = 7.8$  Hz, 2H), 7.61 (d,  $J = 7.7$  Hz, 2H), 7.39 (d,  $J = 7.5$  Hz, 2H), 7.22 - 7.21 (m, 4H), 6.99 - 6.94 (m, 2H), 6.84 (d,  $J = 2.4$  Hz, 1H), 2.47 (s, 3H), 2.40 (s, 3H);  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  163.9 (d,  $J_{\text{C-F}} = 252.9$  Hz), 151.1, 145.8, 145.3, 141.3, 138.5, 135.2, 132.0 (d,  $J_{\text{C-F}} = 8.7$  Hz), 129.9, 129.8, 129.3, 128.7 (d,  $J_{\text{C-F}} = 3.3$  Hz), 128.5, 115.6 (d,  $J_{\text{C-F}} = 22.0$  Hz), 21.8.  $^{19}\text{F NMR}$  (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -109.2, -109.2. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{22}\text{H}_{19}\text{FNaO}_4\text{S}_2$ : 453.0601; found: 453.0598.

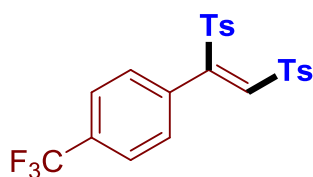


(*Z*)-4,4'-(1-(4-chlorophenyl)ethene-1,2-diyl)disulfonylbis(methylbenzene) (**3ga**): TLC (PE/EtOAc, 3:1),  $R_f = 0.64$ ; White solid (104.6 mg, 78%). mp: 126 - 127 °C.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.97 (d,  $J = 8.1$  Hz, 2H), 7.63 (d,  $J = 8.1$  Hz, 2H), 7.39 (d,  $J = 8.0$  Hz, 2H), 7.27 - 7.22 (m, 4H), 7.17 (d,  $J = 8.3$  Hz, 2H), 6.83 (s, 1H), 2.47 (s, 3H), 2.41 (s, 3H);  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  151.1, 145.9, 145.4, 141.4,

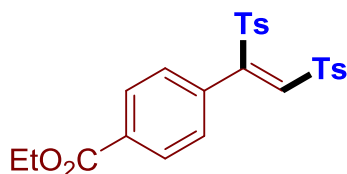
138.4, 136.9, 135.2, 131.2, 131.1, 129.9, 129.9, 129.4, 128.7, 128.6, 21.9, 21.9. HRMS (ESI):  $m/z$   $[M+Na]^+$  calcd for  $C_{22}H_{19}ClNaO_4S_2$ : 469.0305; found: 469.0307.



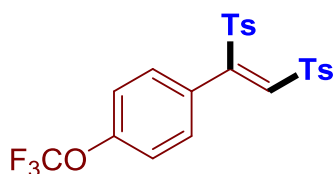
(*Z*)-4,4'-(1-(4-bromophenyl)ethene-1,2-diyl)disulfonylbis(methylbenzene) (**3ha**): TLC (PE/EtOAc, 3:1),  $R_f$  = 0.61; White solid (123.8 mg, 84%). mp: 192 - 193 °C.  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.89 (d,  $J$  = 8.0 Hz, 2H), 7.56 (d,  $J$  = 8.0 Hz, 2H), 7.35 - 7.30 (m, 4H), 7.18 (d,  $J$  = 8.3 Hz, 2H), 7.02 (d,  $J$  = 8.2 Hz, 2H), 6.76 (s, 1H), 2.39 (s, 3H), 2.33 (s, 3H);  $^{13}C$  NMR (101 MHz,  $CDCl_3$ )  $\delta$  151.1, 145.9, 145.4, 141.4, 138.40, 135.1, 131.7, 131.3, 129.9, 129.4, 128.6, 125.2, 21.9, 21.9. HRMS (ESI):  $m/z$   $[M+Na]^+$  calcd for  $C_{22}H_{19}BrNaO_4S_2$ : 512.9800; found: 512.9806.



(*Z*)-4,4'-(1-(4-(trifluoromethyl)phenyl)ethene-1,2-diyl)disulfonylbis(methylbenzene) (**3ia**): TLC (PE/EtOAc, 3:1),  $R_f$  = 0.52; Colorless liquid (116.8 mg, 81%).  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.98 (d,  $J$  = 6.7 Hz, 2H), 7.66 (d,  $J$  = 6.8 Hz, 2H), 7.55 (d,  $J$  = 6.8 Hz, 2H), 7.40 (d,  $J$  = 6.8 Hz, 2H), 7.34 (d,  $J$  = 7.0 Hz, 2H), 7.26 (d,  $J$  = 7.9 Hz, 2H), 6.85 (d,  $J$  = 1.7 Hz, 1H), 2.47 (s, 3H), 2.42 (s, 3H);  $^{13}C$  NMR (101 MHz,  $CDCl_3$ )  $\delta$  150.9, 146.2, 145.6, 141.9, 138.2, 136.4, 135.0, 132.4, 132.1, 130.2, 130.0, 129.5, 128.6, 125.4 (q,  $J_{C-F}$  = 3.4 Hz), 123.6 (q,  $J_{C-F}$  = 339.7 Hz), 21.9, 21.9.  $^{19}F$  NMR (376 MHz,  $CDCl_3$ )  $\delta$  -63.0, -63.0. HRMS (ESI):  $m/z$   $[M+Na]^+$  calcd for  $C_{23}H_{19}F_3NaO_4S_2$ : 503.0569; found: 503.0562.

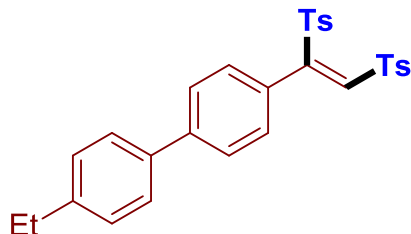


ethyl (*Z*)-4-(1,2-ditosylvinyl)benzoate (**3ja**): TLC (PE/EtOAc, 3:1),  $R_f$  = 0.34; Colorless liquid (107.3 mg, 76%).  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  8.00 (d,  $J$  = 8.0 Hz, 2H), 7.95 (d,  $J$  = 8.0 Hz, 2H), 7.63 (d,  $J$  = 8.0 Hz, 2H), 7.41 (d,  $J$  = 8.0 Hz, 2H), 7.29 (d,  $J$  = 7.8 Hz, 2H), 7.24 (d,  $J$  = 8.0 Hz, 2H), 6.87 (s, 1H), 4.38 (q,  $J$  = 7.1 Hz, 2H), 2.49 (s, 3H), 2.41 (s, 3H), 1.40 (t,  $J$  = 7.0 Hz, 3H);  $^{13}C$  NMR (101 MHz,  $CDCl_3$ )  $\delta$  165.8, 151.4, 146.0, 145.4, 141.6, 138.4, 137.0, 135.1, 132.1, 129.9, 129.9, 129.8, 129.5, 129.5, 128.6, 61.6, 21.9, 14.4. HRMS (ESI):  $m/z$   $[M+Na]^+$  calcd for  $C_{25}H_{24}NaO_6S_2$ : 507.0907; found: 507.0910.

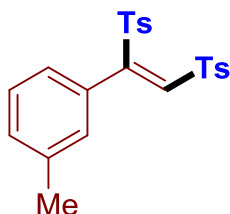


(*Z*)-4,4'-(1-(4-(trifluoromethoxy)phenyl)ethene-1,2-diyl)disulfonylbis(methylbenzene) (**3ka**): TLC (PE/EtOAc, 4:1),  $R_f$  = 0.56; Colorless liquid (95.3 mg, 64%).  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.90 (d,  $J$

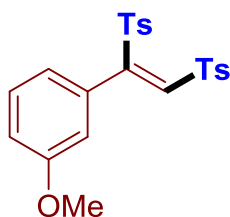
= 8.0 Hz, 2H), 7.57 (d,  $J = 8.0$  Hz, 2H), 7.32 (d,  $J = 7.9$  Hz, 2H), 7.19 - 7.16 (m, 4H), 7.05 (d,  $J = 8.3$  Hz, 2H), 6.77 (s, 1H), 2.40 (s, 3H), 2.34 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  150.9, 150.7, 146.0, 145.4, 141.6, 138.4, 135.2, 131.6, 131.3, 130.0, 129.9, 129.5, 128.6, 120.6, 120.4 (q,  $J_{\text{C-F}} = 259.7$  Hz), 21.9.  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.8. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{25}\text{H}_{24}\text{NaO}_6\text{S}_2$ : 519.0518; found: 519.0522.



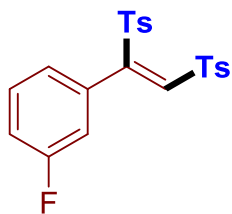
(*Z*)-4-(1,2-ditosylvinyl)-4'-ethyl-1,1'-biphenyl (**3la**): TLC (PE/EtOAc, 3:1),  $R_f = 0.51$ ; Colorless liquid (122.5 mg, 79%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.02 (d,  $J = 8.0$  Hz, 2H), 7.70 (d,  $J = 8.0$  Hz, 2H), 7.53 - 7.50 (m, 4H), 7.42 (d,  $J = 8.1$  Hz, 2H), 7.33 - 7.29 (m, 4H), 7.25 (d,  $J = 8.0$  Hz, 2H), 6.92 (s, 1H), 2.72 (q,  $J = 7.6$  Hz, 2H), 2.50 (s, 3H), 2.42 (s, 3H), 1.29 (t,  $J = 7.6$  Hz, 3H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  152.0, 145.7, 145.2, 144.6, 143.1, 140.8, 138.7, 137.0, 135.5, 131.3, 130.2, 129.9, 129.8, 129.4, 128.6, 128.6, 127.1, 126.8, 28.7, 21.9, 15.7. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{30}\text{H}_{28}\text{NaO}_4\text{S}_2$ : 539.1327; found: 539.1332.



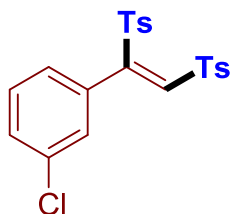
(*Z*)-4,4'-(1-(*m*-tolyl)ethene-1,2-diyldisulfonyl)bis(methylbenzene) (**3ma**): TLC (PE/EtOAc, 3:1),  $R_f = 0.55$ ; White solid (107.5 mg, 84%). mp: 143 - 144 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.97 (d,  $J = 8.0$  Hz, 2H), 7.63 (d,  $J = 8.0$  Hz, 2H), 7.38 (d,  $J = 7.9$  Hz, 2H), 7.28 - 7.11 (m, 5H), 7.03 (s, 1H), 6.97 (d,  $J = 7.1$  Hz, 1H), 6.83 (s, 1H), 2.47 (s, 3H), 2.39 (s, 3H), 2.27 (s, 3H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  152.3, 145.6, 145.1, 140.8, 138.7, 138.2, 135.5, 132.7, 131.1, 130.2, 129.8, 129.7, 129.4, 128.5, 128.3, 126.8, 21.8, 21.3. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{23}\text{H}_{22}\text{NaO}_4\text{S}_2$ : 449.0852; found: 449.0853.



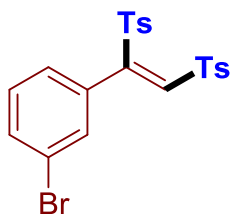
(*Z*)-4,4'-(1-(3-methoxyphenyl)ethene-1,2-diyldisulfonyl)bis(methylbenzene) (**3na**): TLC (PE/EtOAc, 3:1),  $R_f = 0.50$ ; Colorless liquid (96.9 mg, 73%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.97 (d,  $J = 7.4$  Hz, 2H), 7.65 (d,  $J = 7.4$  Hz, 2H), 7.39 (d,  $J = 7.2$  Hz, 2H), 7.25 - 7.16 (m, 3H), 6.91 - 6.86 (m, 2H), 6.78 (s, 1H), 6.72 (d,  $J = 6.8$  Hz, 1H), 3.73 (s, 3H), 2.47 (s, 3H), 2.39 (s, 3H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  156.9, 149.3, 145.1, 145.0, 141.2, 138.7, 135.9, 131.8, 131.1, 129.8, 129.7, 129.1, 128.5, 121.7, 120.3, 110.9, 55.4, 21.9, 21.8. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{23}\text{H}_{22}\text{NaO}_5\text{S}_2$ : 465.0801; found: 465.0804.



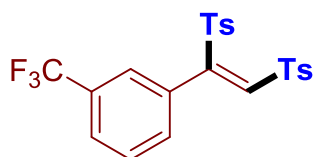
(*Z*)-4,4'-(1-(3-fluorophenyl)ethene-1,2-diyl)disulfonylbis(methylbenzene) (**30a**): TLC (PE/EtOAc, 3:1),  $R_f = 0.56$ ; White solid (96.9 mg, 75%). mp: 130 - 131 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.90 (d,  $J = 7.3$  Hz, 2H), 7.58 (d,  $J = 7.3$  Hz, 2H), 7.32 (d,  $J = 7.7$  Hz, 2H), 7.23 - 7.11 (m, 3H), 7.01 (t,  $J = 8.3$  Hz, 1H), 6.91 (t,  $J = 7.9$  Hz, 2H), 6.78 (s, 1H), 2.40 (s, 3H), 2.33 (s, 3H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  162.1 (d,  $J_{\text{C-F}} = 249.6$  Hz), 150.9 (d,  $J_{\text{C-F}} = 1.9$  Hz), 146.0, 145.4, 141.7, 138.4, 135.2, 134.7 (d,  $J_{\text{C-F}} = 8.1$  Hz), 130.2 (d,  $J_{\text{C-F}} = 8.3$  Hz), 129.9, 129.9, 129.4, 128.6, 125.7 (d,  $J_{\text{C-F}} = 3.2$  Hz), 117.5 (d,  $J_{\text{C-F}} = 21.0$  Hz), 117.0 (d,  $J_{\text{C-F}} = 23.5$  Hz), 21.9.  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -111.5. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{22}\text{H}_{19}\text{FNaO}_4\text{S}_2$ : 453.0601; found: 453.0606.



(*Z*)-4,4'-(1-(3-chlorophenyl)ethene-1,2-diyl)disulfonylbis(methylbenzene) (**3pa**): TLC (PE/EtOAc, 3:1),  $R_f = 0.59$ ; White solid (116.7 mg, 87%). mp: 146 - 147 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.97 (d,  $J = 8.0$  Hz, 2H), 7.65 (d,  $J = 8.0$  Hz, 2H), 7.39 (d,  $J = 8.0$  Hz, 2H), 7.35 (d,  $J = 8.1$  Hz, 1H), 7.30 - 7.16 (m, 5H), 7.10 (d,  $J = 7.7$  Hz, 1H), 6.84 (s, 1H), 2.47 (s, 3H), 2.41 (s, 3H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  150.8, 146.0, 145.4, 141.7, 138.3, 135.1, 134.4, 134.4, 130.5, 129.9, 129.9, 129.7, 129.6, 129.5, 128.615, 128.0, 21.9, 21.9. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{22}\text{H}_{19}\text{ClNaO}_4\text{S}_2$ : 469.0305; found: 469.0302.

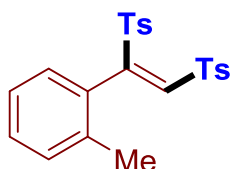


(*Z*)-4,4'-(1-(3-bromophenyl)ethene-1,2-diyl)disulfonylbis(methylbenzene) (**3qa**): TLC (PE/EtOAc, 3:1),  $R_f = 0.60$ ; Colorless liquid (112.0 mg, 76%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.89 (d,  $J = 8.0$  Hz, 2H), 7.57 (d,  $J = 8.0$  Hz, 2H), 7.43 (s, 1H), 7.32 (d,  $J = 8.0$  Hz, 2H), 7.25 (s, 1H), 7.17 (d,  $J = 8.0$  Hz, 2H), 7.08 (d,  $J = 4.5$  Hz, 2H), 6.75 (s, 1H), 2.40 (s, 3H), 2.34 (s, 3H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  150.7, 146.0, 145.4, 141.7, 138.3, 135.1, 134.7, 133.4, 132.4, 129.9, 129.9, 129.5, 128.6, 128.4, 122.3, 21.9, 21.9. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{22}\text{H}_{19}\text{BrNaO}_4\text{S}_2$ : 512.9800; found: 512.9806.

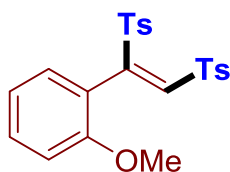


(*Z*)-4,4'-(1-(4-(trifluoromethyl)phenyl)ethene-1,2-diyl)disulfonylbis(methylbenzene) (**3ra**): TLC (PE/

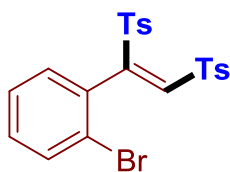
EtOAc, 3:1),  $R_f = 0.51$ ; Colorless liquid (118.2 mg, 82%).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.01 (d,  $J = 7.9$  Hz, 2H), 7.65 - 7.63 (m, 3H), 7.46 - 7.41 (m, 4H), 7.36 (s, 1H), 7.26 (d,  $J = 7.9$  Hz, 2H), 6.88 (s, 1H), 2.49 (s, 3H), 2.43 (s, 3H);  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  150.8, 146.2, 145.5, 141.8, 138.2, 134.9, 133.7, 133.1, 130.0, 129.9, 129.5, 129.1, 128.7, 126.9 (q,  $J_{\text{C-F}} = 3.7$  Hz), 126.5 (q,  $J_{\text{C-F}} = 3.8$  Hz), 123.4 (q,  $J_{\text{C-F}} = 273.7$  Hz), 21.9, 21.8.  $^{19}\text{F NMR}$  (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -62.8. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{23}\text{H}_{19}\text{F}_3\text{NaO}_4\text{S}_2$ : 503.0575; found: 503.0579.



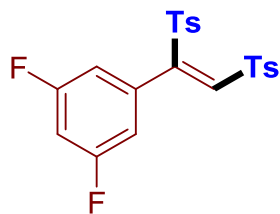
(*Z*)-4,4'-(1-(*o*-tolyl)ethene-1,2-diyl)disulfonylbis(methylbenzene) (**3sa**): TLC (PE/EtOAc, 3:1),  $R_f = 0.56$ ; White solid (99.8 mg, 78%). mp: 141 - 142°C.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.97 (d,  $J = 8.0$  Hz, 2H), 7.62 (d,  $J = 8.0$  Hz, 2H), 7.39 (d,  $J = 8.0$  Hz, 2H), 7.25 - 7.22 (m, 3H), 7.10 - 7.03 (m, 2H), 6.91 (d,  $J = 7.6$  Hz, 1H), 6.76 (s, 1H), 2.47 (s, 3H), 2.42 (s, 3H), 1.94 (s, 3H).  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  151.4, 145.9, 145.2, 141.3, 138.6, 137.2, 134.9, 131.9, 130.5, 130.0, 130.0, 129.9, 129.9, 129.7, 128.4, 125.4, 21.9, 21.8, 19.4. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{23}\text{H}_{22}\text{NaO}_4\text{S}_2$ : 449.0852; found: 449.0854.



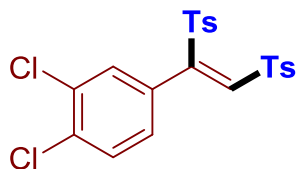
(*Z*)-4,4'-(1-(2-methoxyphenyl)ethene-1,2-diyl)disulfonylbis(methylbenzene) (**3ta**): TLC (PE/EtOAc, 3:1),  $R_f = 0.41$ ; Colorless liquid (96.9 mg, 73%).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.90 (d,  $J = 8.0$  Hz, 2H), 7.52 (d,  $J = 8.0$  Hz, 2H), 7.32 (s, 1H), 7.25 - 7.18 (m, 2H), 7.10 (d,  $J = 8.0$  Hz, 2H), 7.08 - 7.05 (m, 1H), 6.83 - 6.80 (m, 1H), 6.73 (s, 1H), 6.60 (d,  $J = 8.4$  Hz, 1H), 3.36 (s, 3H), 2.40 (s, 3H), 2.32 (s, 3H);  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  156.8, 149.3, 145.1, 145.0, 141.1, 138.7, 135.8, 131.8, 131.0, 129.7, 129.6, 129.1, 128.4, 128.4, 121.6, 120.2, 110.83, 55.3, 21.8, 21.7. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{23}\text{H}_{22}\text{NaO}_5\text{S}_2$ : 465.0801; found: 465.0804.



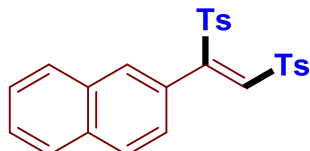
(*Z*)-4,4'-(1-(2-bromophenyl)ethene-1,2-diyl)disulfonylbis(methylbenzene) (**3ua**): TLC (PE/EtOAc, 3:1),  $R_f = 0.58$ ; Colorless liquid (120.9 mg, 82%).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.99 (d,  $J = 7.6$  Hz, 2H), 7.61 (d,  $J = 7.6$  Hz, 2H), 7.45 (d,  $J = 7.9$  Hz, 1H), 7.39 (d,  $J = 7.8$  Hz, 2H), 7.33 - 7.27 (m, 3H), 7.22 (d,  $J = 7.7$  Hz, 2H), 6.83 (s, 1H), 2.48 (s, 3H), 2.42 (s, 3H);  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  149.9, 146.0, 145.3, 143.1, 138.3, 134.8, 133.2, 133.1, 131.7, 131.4, 130.1, 129.9, 129.8, 128.6, 127.1, 124.1, 21.9, 21.9. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{22}\text{H}_{19}\text{BrNaO}_4\text{S}_2$ : 512.9800; found: 512.9803.



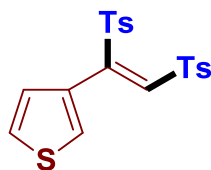
(*Z*)-4,4'-(1-(3,5-difluorophenyl)ethene-1,2-diyldisulfonyl)bis(methylbenzene) (**3va**): TLC (PE/EtOAc, 3:1),  $R_f = 0.71$ ; Colorless liquid (113.0 mg, 84%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.89 (d,  $J = 8.1$  Hz, 2H), 7.61 (d,  $J = 8.1$  Hz, 2H), 7.32 (d,  $J = 8.0$  Hz, 2H), 7.20 (d,  $J = 7.6$  Hz, 2H), 6.79 - 6.76 (m, 2H), 6.74 - 6.68 (m, 2H), 2.40 (s, 3H), 2.35 (s, 3H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  162.3 (dd,  $J_{\text{C-F}} = 252.5$  Hz, 12.7 Hz), 149.8, 146.2, 145.5, 142.1, 138.1, 135.5 (d,  $J_{\text{C-F}} = 20.2$  Hz), 135.5, 134.9, 130.0, 129.9, 129.3, 128.5, 113.1 (d,  $J_{\text{C-F}} = 27.4$  Hz), 113.0 (d,  $J_{\text{C-F}} = 11.4$  Hz), 105.9 (t,  $J_{\text{C-F}} = 25.1$  Hz), 21.8, 21.8.  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -107.8. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{22}\text{H}_{18}\text{F}_2\text{NaO}_4\text{S}_2$ : 471.0512; found: 471.0517.



(*Z*)-4,4'-(1-(3,4-dichlorophenyl)ethene-1,2-diyldisulfonyl)bis(methylbenzene) (**3wa**): TLC (PE/EtOAc, 3:1),  $R_f = 0.75$ ; Colorless liquid (132.9 mg, 92%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.89 (d,  $J = 7.9$  Hz, 2H), 7.59 (d,  $J = 7.9$  Hz, 2H), 7.33 - 7.27 (m, 3H), 7.24 (s, 1H), 7.21 - 7.17 (m, 2H), 7.00 (d,  $J = 8.4$  Hz, 1H), 6.76 (s, 1H), 2.39 (s, 3H), 2.35 (s, 3H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  149.8, 146.2, 145.5, 142.0, 138.2, 135.1, 135.0, 132.8, 132.6, 131.5, 130.5, 130.0, 130.0, 129.4, 129.0, 128.6, 21.9, 21.9. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{22}\text{H}_{18}\text{Cl}_2\text{NaO}_4\text{S}_2$ : 502.9921; found: 502.9917.

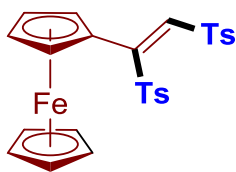


(*Z*)-2-(1,2-ditosylvinyl)naphthalene (**3xa**): TLC (PE/EtOAc, 4:1),  $R_f = 0.31$ ; Colorless liquid (93.0 mg, 67%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.95 (d,  $J = 8.1$  Hz, 2H), 7.75 (d,  $J = 8.2$  Hz, 1H), 7.69 (d,  $J = 8.2$  Hz, 1H), 7.51 (d,  $J = 8.1$  Hz, 2H), 7.45 (d,  $J = 8.4$  Hz, 1H), 7.38 - 7.26 (m, 5H), 7.07 (d,  $J = 7.1$  Hz, 1H), 7.02 (d,  $J = 8.0$  Hz, 2H), 6.84 (s, 1H), 2.40 (s, 3H), 2.26 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  151.2, 145.7, 145.4, 142.2, 138.5, 134.7, 133.3, 131.7, 130.5, 130.0, 129.9, 129.8, 129.5, 128.6, 128.4, 128.3, 127.2, 126.5, 124.9, 124.6, 21.9, 21.8. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{26}\text{H}_{22}\text{NaO}_4\text{S}_2$ : 485.0852; found: 485.0854.

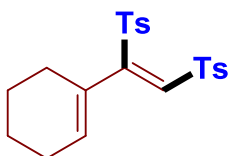


(*Z*)-3-(1,2-ditosylvinyl)thiophene (**3ya**): TLC (PE/EtOAc, 3:1),  $R_f = 0.56$ ; Colorless liquid (76.6 mg, 61%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.90 (d,  $J = 8.1$  Hz, 2H), 7.64 (d,  $J = 8.0$  Hz, 2H), 7.58 - 7.52 (m, 1H), 7.31 (d,  $J = 8.0$  Hz, 2H), 7.20 - 7.14 (m, 3H), 7.06 (d,  $J = 5.0$  Hz, 1H), 6.91 (s, 1H), 2.39 (s, 3H),

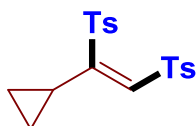
2.32 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  147.2, 145.7, 145.1, 139.6, 138.8, 135.6, 132.4, 129.9, 129.8, 129.6, 129.2, 128.7, 128.5, 126.6, 21.9. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{20}\text{H}_{18}\text{NaO}_4\text{S}_3$ : 441.0259; found: 441.0264.



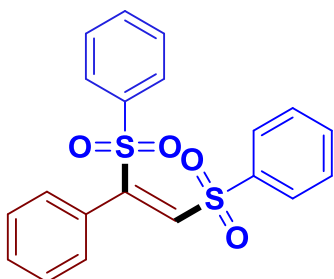
(*Z*)-4,4'-(1-ferrocenyl)ethene-1,2-diylbis(methylbenzene) (**3za**): TLC (PE/EtOAc, 4:1),  $R_f$  = 0.67; brown solid (50.1 mg, 32%). mp: 147 - 148  $^\circ\text{C}$ .  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.88 (d,  $J$  = 8.0 Hz, 2H), 7.82 (d,  $J$  = 7.9 Hz, 2H), 7.36 - 7.26 (m, 3H), 7.21 (d,  $J$  = 8.1 Hz, 2H), 4.63 (s, 2H), 4.37 (s, 2H), 4.01 (s, 5H), 2.39 (s, 3H), 2.33 (s, 3H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  154.7, 145.3, 144.5, 139.5, 136.4, 134.8, 129.7, 129.5, 128.7, 128.0, 76.2, 71.4, 70.9, 70.8, 60.4, 21.7. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{26}\text{H}_{24}\text{FeNaO}_4\text{S}_2$ : 543.0358; found: 543.0367.



(*Z*)-4,4'-(1-cyclohex-1-en-1-yl)ethene-1,2-diylbis(methylbenzene) (**4aa**): TLC (PE/EtOAc, 3:1),  $R_f$  = 0.41; Colorless liquid (63.7 mg, 51%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.93 (d,  $J$  = 6.6 Hz, 2H), 7.84 (d,  $J$  = 6.6 Hz, 2H), 7.36 (d,  $J$  = 7.0 Hz, 2H), 7.31 (d,  $J$  = 6.9 Hz, 2H), 6.70 (d,  $J$  = 1.6 Hz, 1H), 5.62 (s, 1H), 2.45 (m, 6H), 2.09 - 2.05 (m, 2H), 1.96 - 1.91 (m, 2H), 1.49 - 1.43 (m, 2H), 1.29 - 1.26 (m, 2H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  154.9, 145.5, 145.0, 138.8, 138.5, 135.9, 134.4, 132.4, 129.8, 129.7, 129.5, 128.5, 29.1, 25.7, 22.4, 21.9, 21.9, 21.1. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{22}\text{H}_{24}\text{NaO}_4\text{S}_2$ : 439.1008; found: 439.1006.



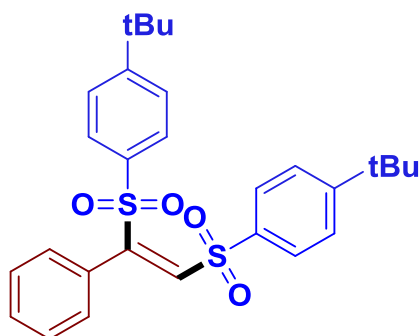
(*Z*)-4,4'-(1-cyclopropylethene-1,2-diyl)bis(methylbenzene) (**4ba**): TLC (PE/EtOAc, 3:1),  $R_f$  = 0.47; White solid (70.0 mg, 62%). mp: 135 - 136  $^\circ\text{C}$ .  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.00 (d,  $J$  = 8.1 Hz, 2H), 7.93 (d,  $J$  = 8.1 Hz, 2H), 7.36 (d,  $J$  = 7.8 Hz, 4H), 6.51 (s, 1H), 2.46 (s, 6H), 0.89 - 0.88 (m, 2H), 0.48 - 0.47 (m, 2H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  156.6, 145.7, 145.0, 138.6, 136.2, 135.0, 130.0, 129.8, 129.4, 128.5, 21.9, 21.8, 15.38, 9.52. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{19}\text{H}_{20}\text{NaO}_4\text{S}_2$ : 399.0695; found: 399.0691.



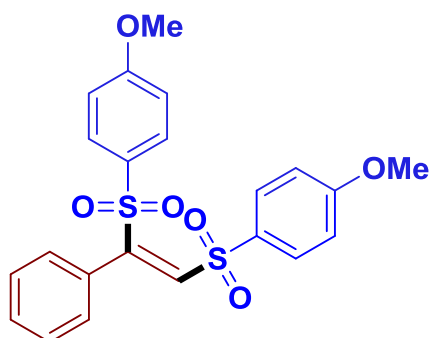
(*Z*)-1-phenylethene-1,2-diylbis(methylbenzene) (**3ab**): TLC (PE/EtOAc, 3:1),  $R_f$  = 0.44; Colorless liquid (85.4 mg, 74%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.10 (d,  $J$  = 7.4 Hz, 2H), 7.73 - 7.67 (m, 3H),



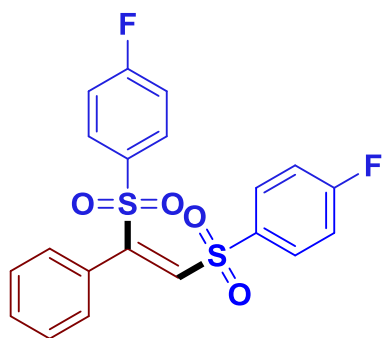
7.62 - 7.56 (m, 3H), 7.44 - 7.35 (m, 3H), 7.29 - 7.25 (m, 2H), 7.20 (d,  $J = 7.4$  Hz, 2H), 6.89 (s, 1H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  152.2, 141.5, 140.9, 138.1, 134.5, 134.1, 132.4, 130.5, 129.7, 129.3, 129.3, 129.1, 128.5, 128.4. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{20}\text{H}_{16}\text{NaO}_4\text{S}_2$ : 407.0382; found: 407.0385.



(*Z*)-4,4'-(1-phenylethene-1,2-diyl)disulfonyl bis(tert-butylbenzene) (**3ac**): TLC (PE/EtOAc, 3:1),  $R_f = 0.72$ ; Colorless liquid (107.3 mg, 72%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.02 (d,  $J = 8.4$  Hz, 2H), 7.65 (d,  $J = 8.3$  Hz, 2H), 7.60 (d,  $J = 8.4$  Hz, 2H), 7.42 (d,  $J = 8.3$  Hz, 2H), 7.37 (d,  $J = 7.2$  Hz, 1H), 7.29 - 7.26 (m, 2H), 7.22 (d,  $J = 7.6$  Hz, 2H), 6.85 (s, 1H), 1.37 (s, 9H), 1.30 (s, 9H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  158.5, 158.1, 152.1, 140.9, 138.4, 135.3, 132.8, 130.3, 129.8, 129.3, 128.4, 128.3, 126.3, 126.1, 35.4, 35.4, 31.2, 31.1. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{28}\text{H}_{32}\text{NaO}_4\text{S}_2$ : 519.1634; found: 519.1641.

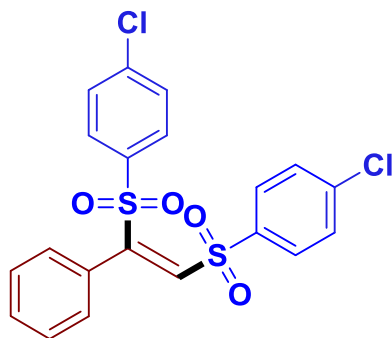


(*Z*)-4,4'-(1-phenylethene-1,2-diyl)disulfonyl bis(methoxybenzene) (**3ad**): TLC (PE/EtOAc, 3:1),  $R_f = 0.30$ ; Colorless liquid (100.0 mg, 75%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.04 (d,  $J = 8.7$  Hz, 2H), 7.68 (d,  $J = 8.7$  Hz, 2H), 7.40 - 7.34 (m, 1H), 7.29 - 7.25 (m, 2H), 7.20 (d,  $J = 7.1$  Hz, 2H), 7.05 (d,  $J = 8.6$  Hz, 2H), 6.87 (d,  $J = 8.7$  Hz, 2H), 6.81 (s, 1H), 3.90 (s, 3H), 3.84 (s, 3H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  164.4, 164.1, 152.0, 140.7, 132.9, 132.9, 131.7, 130.9, 130.2, 129.7, 129.7, 128.4, 114.4, 114.3, 55.9, 55.8. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{22}\text{H}_{20}\text{NaO}_6\text{S}_2$ : 467.0594; found: 467.0598.

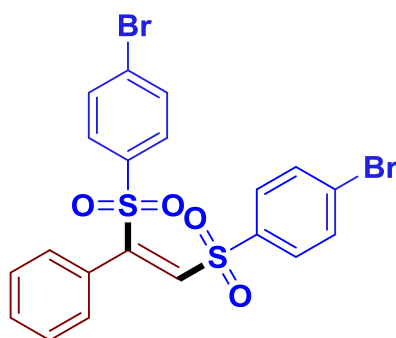


(*Z*)-4,4'-(1-phenylethene-1,2-diyl)disulfonyl bis(fluorobenzene) (**3ae**): TLC (PE/EtOAc, 3:1),  $R_f = 0.61$ ; Colorless liquid (104.7 mg, 83%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.15 - 8.12 (m, 2H), 7.81 - 7.78 (m,

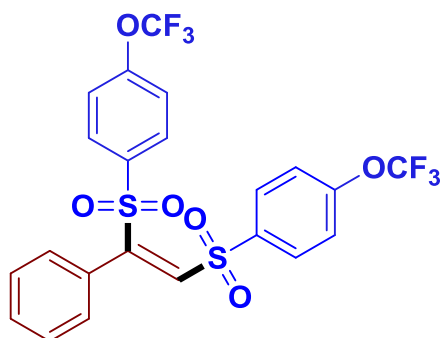
2H), 7.43 - 7.39 (m, 1H), 7.32 - 7.27 (m, 4H), 7.22 (d,  $J = 7.5$  Hz, 2H), 7.13 - 7.09 (m, 2H), 6.88 (s, 1H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  166.4 (d,  $J_{\text{C-F}} = 259.2$  Hz), 166.2 (d,  $J_{\text{C-F}} = 258.3$  Hz), 152.4, 141.0, 137.3 (d,  $J_{\text{C-F}} = 3.1$  Hz), 134.1 (d,  $J_{\text{C-F}} = 3.0$  Hz), 132.4 (d,  $J_{\text{C-F}} = 9.9$  Hz), 132.2, 131.6 (d,  $J_{\text{C-F}} = 9.8$  Hz), 130.7, 129.8, 128.6, 116.6 (d,  $J_{\text{C-F}} = 22.8$  Hz), 116.5 (d,  $J_{\text{C-F}} = 22.8$  Hz);  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -101.45, -102.44. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{20}\text{H}_{14}\text{F}_2\text{NaO}_4\text{S}_2$ : 443.0194; found: 443.0192.



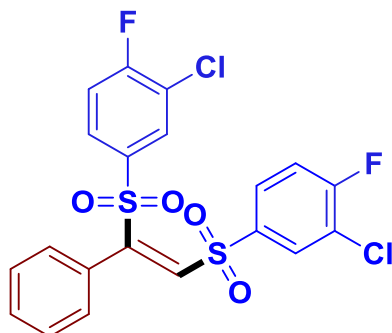
(*Z*)-4,4'-(1-phenylethene-1,2-diyldisulfonyl)bis(chlorobenzene) (**3af**): TLC (PE/EtOAc, 3:1),  $R_f = 0.63$ ; White solid (104.7 mg, 77%). mp: 138 - 139 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.04 (d,  $J = 8.5$  Hz, 2H), 7.69 (d,  $J = 8.5$  Hz, 2H), 7.57 (d,  $J = 8.5$  Hz, 2H), 7.45 - 7.36 (m, 3H), 7.32 - 7.29 (m, 2H), 7.23 (d,  $J = 7.2$  Hz, 2H), 6.88 (s, 1H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  152.4, 141.5, 141.0, 140.9, 139.7, 136.6, 132.0, 130.8, 130.0, 129.7, 129.6, 129.5, 128.7. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{20}\text{H}_{14}\text{Cl}_2\text{NaO}_4\text{S}_2$ : 474.9603; found: 474.9606.



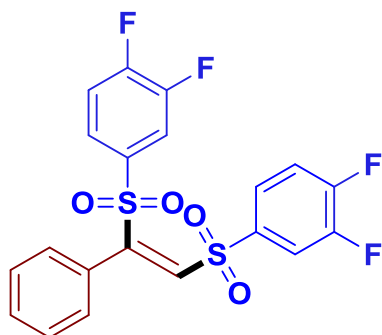
(*Z*)-4,4'-(1-phenylethene-1,2-diyldisulfonyl)bis(bromobenzene) (**3ag**): TLC (PE/EtOAc, 3:1),  $R_f = 0.56$ ; White solid (139.9 mg, 86%). mp: 148 - 149 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.96 (d,  $J = 8.4$  Hz, 2H), 7.74 (d,  $J = 8.3$  Hz, 2H), 7.62 - 7.56 (m, 4H), 7.44 - 7.40 (m, 1H), 7.33 - 7.29 (m, 2H), 7.27 - 7.23 (m, 2H), 6.88 (s, 1H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  152.4, 141.0, 140.3, 137.1, 132.6, 132.5, 132.0, 130.8, 130.3, 130.1, 129.8, 129.7, 128.7. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{20}\text{H}_{14}\text{Br}_2\text{NaO}_4\text{S}_2$ : 562.8592; found: 562.8596.



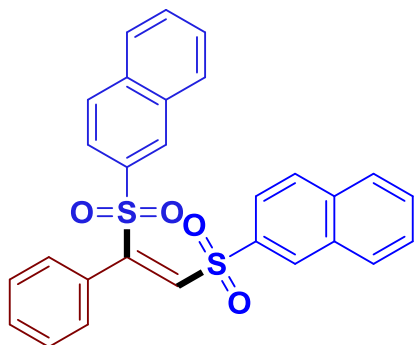
(*Z*)-4,4'-(1-phenylethene-1,2-diyl)disulfonyl)bis((trifluoromethoxy)benzene) (**3ah**): TLC (PE/EtOAc, 3:1),  $R_f = 0.73$ ; Colorless liquid (126.0 mg, 76%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.17 (d,  $J = 8.8$  Hz, 2H), 7.82 (d,  $J = 8.8$  Hz, 2H), 7.46 - 7.40 (m, 3H), 7.33 - 7.30 (m, 2H), 7.26 - 7.24 (m, 4H), 6.90 (s, 1H);  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  153.6, 153.4, 152.6, 140.9, 139.3, 136.1, 132.0, 131.8, 131.0, 130.9, 129.8, 128.7, 120.9, 120.6, 120.3 (q,  $J_{\text{C-F}} = 261.0$  Hz), 120.2 (q,  $J_{\text{C-F}} = 261.2$  Hz);  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -57.60, -57.66. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{22}\text{H}_{14}\text{F}_6\text{NaO}_6\text{S}_2$ : 575.0028; found: 575.0031.



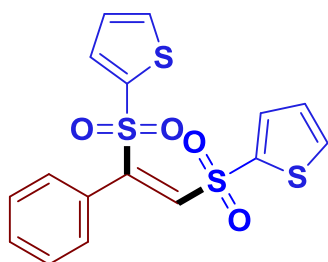
(*Z*)-4,4'-(1-phenylethene-1,2-diyl)disulfonyl)bis(2-chloro-1-fluorobenzene) (**3ai**): TLC (PE/EtOAc, 3:1),  $R_f = 0.56$ ; White solid (108.6 mg, 74%). mp: 127 - 128 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.17 (dd,  $J = 6.5, 1.7$  Hz, 1H), 8.07 - 7.97 (m, 1H), 7.83 (dd,  $J = 6.6, 1.7$  Hz, 1H), 7.71 - 7.64 (m, 1H), 7.47 - 7.43 (m, 1H), 7.41 - 7.31 (m, 3H), 7.27 - 7.25 (m, 2H), 7.22 - 7.18 (m, 1H), 6.90 (s, 1H);  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  161.9 (d,  $J_{\text{C-F}} = 261.5$  Hz), 161.8 (d,  $J_{\text{C-F}} = 260.6$  Hz), 152.6, 140.8, 138.1 (d,  $J_{\text{C-F}} = 3.8$  Hz), 135.0 (d,  $J_{\text{C-F}} = 3.7$  Hz), 132.4, 131.7, 131.1, 130.2 (d,  $J_{\text{C-F}} = 9.1$  Hz), 129.8, 129.4 (d,  $J_{\text{C-F}} = 8.9$  Hz), 129.8, 129.4 (d,  $J_{\text{C-F}} = 8.8$  Hz), 128.8, 122.8 (d,  $J_{\text{C-F}} = 18.9$  Hz), 117.8 (d,  $J_{\text{C-F}} = 9.3$  Hz), 117.6 (d,  $J_{\text{C-F}} = 9.4$  Hz);  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -103.36, -104.38. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{20}\text{H}_{12}\text{Cl}_2\text{F}_2\text{NaO}_4\text{S}_2$ : 510.9414; found: 510.9407.



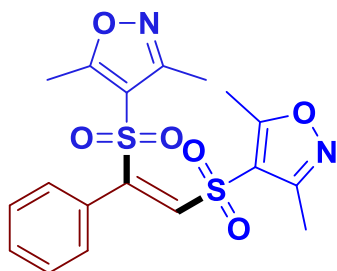
(*Z*)-4,4'-(1-phenylethene-1,2-diyl)disulfonyl)bis(1,2-difluorobenzene) (**3aj**): TLC (PE/EtOAc, 3:1),  $R_f = 0.56$ ; White solid (100.0 mg, 73%). mp: 115 - 116 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.99 - 7.95 (m, 1H), 7.90 (d,  $J = 8.3$  Hz, 1H), 7.68 - 7.64 (m, 1H), 7.58 (d,  $J = 8.2$  Hz, 1H), 7.46 - 7.41 (m, 2H), 7.35 - 7.31 (m, 2H), 7.29 - 7.20 (m, 3H), 6.90 (s, 1H);  $^{13}\text{C}$  NMR (101 MHz, Chloroform-*d*)  $\delta$  154.4 (dd,  $J_{\text{C-F}} = 261.8$  Hz, 12.6 Hz), 154.3 (dd,  $J_{\text{C-F}} = 260.7$  Hz, 12.5 Hz), 152.5, 150.3 (dd,  $J_{\text{C-F}} = 256.6$  Hz, 10.2 Hz), 150.1 (dd,  $J_{\text{C-F}} = 256.6$  Hz, 10.2 Hz), 140.7, 137.8 (dd,  $J_{\text{C-F}} = 4.8$  Hz, 3.7 Hz), 134.7 (dd,  $J_{\text{C-F}} = 4.6$  Hz, 3.5 Hz), 131.7, 131.1, 129.7, 128.8, 127.0 (dd,  $J_{\text{C-F}} = 8.1$  Hz, 4.0 Hz), 126.0 (dd,  $J_{\text{C-F}} = 7.9$  Hz, 4.0 Hz), 119.2 (dd,  $J_{\text{C-F}} = 7.9$  Hz, 4.0 Hz), 119.3 (dd,  $J_{\text{C-F}} = 20.3$  Hz, 2.0 Hz), 118.6 (d,  $J_{\text{C-F}} = 10.1$  Hz), 118.4 (d,  $J_{\text{C-F}} = 10.3$  Hz);  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )  $\delta$  -125.42, -125.48, -126.50, -126.55, -132.57, -132.63, -132.77, -132.83. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{20}\text{H}_{12}\text{F}_4\text{NaO}_4\text{S}_2$ : 479.0005; found: 479.0011.



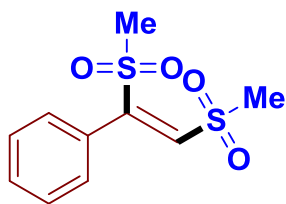
(*Z*)-2,2'-(1-phenylethene-1,2-diyl)disulfonyldinaphthalene (**3ak**): TLC (PE/EtOAc, 3:1),  $R_f = 0.36$ ; White solid (109.0 mg, 75%). mp: 130 - 131 °C.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.71 (s, 1H), 8.25 (s, 1H), 8.06 - 7.99 (m, 3H), 7.95 (d,  $J = 8.1$  Hz, 1H), 7.84 (d,  $J = 8.2$  Hz, 1H), 7.79 (d,  $J = 8.7$  Hz, 1H), 7.74 (d,  $J = 8.1$  Hz, 1H), 7.72 - 7.67 (m, 1H), 7.65 - 7.61 (m, 3H), 7.56 - 7.53 (m, 1H), 7.36 - 7.31 (m, 1H), 7.23 - 7.22 (m, 4H), 6.97 (s, 1H).  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  152.2, 141.0, 138.3, 135.6, 135.1, 132.6, 132.2, 132.0, 131.7, 130.5, 129.8, 129.8, 129.8, 129.7, 129.6, 129.6, 129.2, 128.5, 128.1, 128.0, 127.9, 127.7, 123.5, 123.0. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{28}\text{H}_{20}\text{NaO}_4\text{S}_2$ : 507.0695; found: 507.0698.



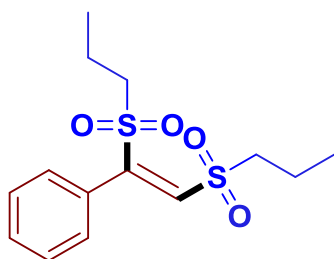
(*Z*)-2,2'-(1-phenylethene-1,2-diyl)dithiophene (**3al**): TLC (PE/EtOAc, 3:1),  $R_f = 0.43$ ; Colorless liquid (78.5 mg, 66%).  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.99 (d,  $J = 3.3$  Hz, 1H), 7.80 (d,  $J = 4.8$  Hz, 1H), 7.71 - 7.68 (m, 2H), 7.43 - 7.39 (m, 1H), 7.35 - 7.26 (m, 4H), 7.22 - 7.19 (m, 1H), 7.08 - 7.06 (m, 1H), 6.90 (s, 1H).  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  152.1, 141.8, 140.2, 138.8, 136.9, 136.5, 135.9, 135.4, 132.4, 130.6, 129.7, 128.5, 128.2, 128.2. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{16}\text{H}_{12}\text{NaO}_4\text{S}_4$ : 418.9511; found: 418.9506.



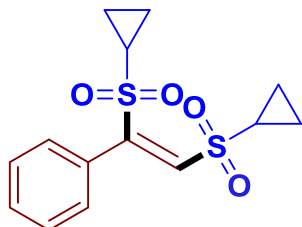
(*Z*)-4,4'-(1-phenylethene-1,2-diyl)bis(3,5-dimethylisoxazole) (**3am**): TLC (PE/EtOAc, 3:1),  $R_f = 0.33$ ; white solid (87.5 mg, 69%). mp: 116 - 117 °C.  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.50 - 7.46 (m, 1H), 7.40 - 7.37 (m, 2H), 7.28 (d,  $J = 7.3$  Hz, 2H), 6.92 (s, 1H), 2.76 (s, 3H), 2.46 (s, 3H), 2.34 (s, 3H), 2.06 (s, 3H);  $^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ )  $\delta$  176.8, 175.5, 158.2, 157.7, 151.9, 141.0, 131.4, 131.1, 129.2, 129.0, 118.0, 114.2, 13.2, 12.5, 11.1, 10.3. HRMS (ESI):  $m/z$   $[\text{M}+\text{H}]^+$  calcd for  $\text{C}_{18}\text{H}_{18}\text{N}_2\text{NaO}_6\text{S}_2$ : 445.0498; found: 445.0502.



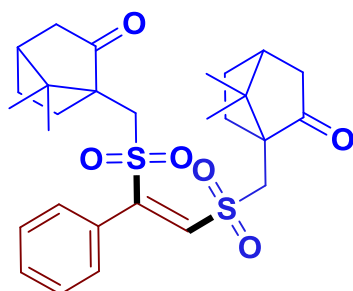
(*Z*)-(1,2-bis(methylsulfonyl)vinyl)benzene (**3an**): TLC (PE/EtOAc, 3:1),  $R_f = 0.56$ ; White solid (52.3 mg, 67%). mp: 102 - 103 °C.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.57 - 7.42 (m, 5H), 6.92 (d,  $J = 1.9$  Hz, 1H), 3.43 (s, 3H), 3.21 (s, 3H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  154.7, 141.2, 132.3, 131.3, 129.5, 129.0, 45.2, 44.1. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{10}\text{H}_{12}\text{NaO}_4\text{S}_2$ : 283.0069; found: 283.0067.



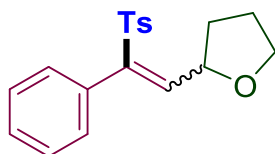
(*Z*)-(1,2-bis(propylsulfonyl)vinyl)benzene (**3ao**): TLC (PE/EtOAc, 3:1),  $R_f = 0.43$ ; Colorless liquid (54.1 mg, 57%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.53 - 7.51 (m, 3H), 7.48 - 7.42 (m, 2H), 6.86 (d,  $J = 3.1$  Hz, 1H), 3.62 - 3.53 (m, 2H), 3.31 - 3.29 (m, 2H), 1.98 - 1.89 (m, 2H), 1.87 - 1.77 (m, 2H), 1.16 - 1.08 (m, 3H), 1.05 - 0.98 (m, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  154.4, 141.2, 132.9, 131.1, 130.0, 129.5, 129.2, 128.9, 128.8, 58.7, 57.8, 16.1, 15.3, 13.2, 13.1. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{14}\text{H}_{20}\text{NaO}_4\text{S}_2$ : 339.0695; found: 339.0698.



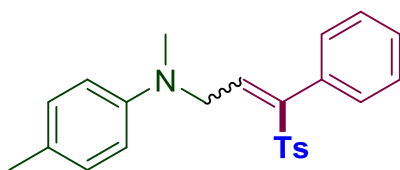
(*Z*)-(1,2-bis(cyclopropylsulfonyl)vinyl)benzene (**3ap**): TLC (PE/EtOAc, 3:1),  $R_f = 0.39$ ; Colorless liquid (57.2 mg, 61%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.53 (d,  $J = 7.6$  Hz, 2H), 7.50 - 7.48 (m, 1H), 7.46 - 7.42 (m, 2H), 6.83 (s, 1H), 3.44 - 3.31 (m, 1H), 2.99 - 2.88 (m, 1H), 1.40 - 1.35 (m, 2H), 1.26 - 1.22 (m, 2H), 1.18 - 1.13 (m, 2H), 1.04 - 0.99 (m, 2H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  153.6, 140.2, 133.2, 130.8, 129.6, 128.8, 32.8, 32.0, 5.9, 5.8. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{14}\text{H}_{16}\text{NaO}_4\text{S}_2$ : 335.0382; found: 335.0387.



(*Z*)-1,1'-((1-phenylethene-1,2-diyldisulfonyl)bis(methylene))bis(7,7-dimethylbicyclo[2.2.1]heptan-2-one) (**3aq**): TLC (PE/EtOAc, 3:1),  $R_f = 0.43$ ; Colorless liquid (100.7 mg, 63%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.70 (d,  $J = 7.4$  Hz, 2H), 7.48 - 7.46 (m, 3H), 7.12 (s, 1H), 4.13 - 4.09 (m, 1H), 3.78 - 3.61 (m, 2H), 3.38 - 3.34 (m, 1H), 2.55 - 2.36 (m, 3H), 2.31 - 2.22 (m, 1H), 2.14 (d,  $J = 4.2$  Hz, 1H), 2.11 - 2.02 (m, 4H), 1.97 - 1.94 (m, 1H), 1.92 - 1.90 (m, 1H), 1.81 - 1.75 (m, 1H), 1.49 - 1.38 (m, 2H), 1.16 (s, 3H), 1.07 (s, 3H), 0.93 (s, 3H), 0.86 (s, 3H);  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  215.1, 214.3, 152.7, 142.1, 133.2, 130.7, 129.6, 128.8, 59.9, 59.7, 55.4, 53.0, 49.1, 48.5, 42.8, 42.6, 42.4, 27.3, 27.2, 26.5, 25.0, 19.9, 19.9, 19.6, 19.6. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{28}\text{H}_{36}\text{NaO}_6\text{S}_2$ : 555.1846; found: 555.1839.



(*Z*)-2-(2-phenyl-2-tosylvinyl)tetrahydrofuran (**5a**): TLC (PE/EtOAc, 3:1),  $R_f = 0.6$ ; Colorless liquid (51.2 mg, 52%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.51 (d,  $J = 8.1$  Hz, 2H), 7.31 - 7.18 (m, 6H), 6.25 (s, 1H), 5.69 - 5.64 (m, 1H), 3.96 - 3.84 (m, 2H), 2.61 - 2.51 (m, 1H), 2.38 (s, 3H), 2.06 - 1.98 (m, 2H), 1.87 - 1.78 (m, 1H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  147.2, 144.4, 141.3, 137.1, 134.9, 130.2, 129.6, 128.8, 128.1, 128.1, 75.2, 68.7, 33.7, 26.4, 23.5, 21.7. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{19}\text{H}_{20}\text{NaO}_3\text{S}$ : 351.1031; found: 351.1033.



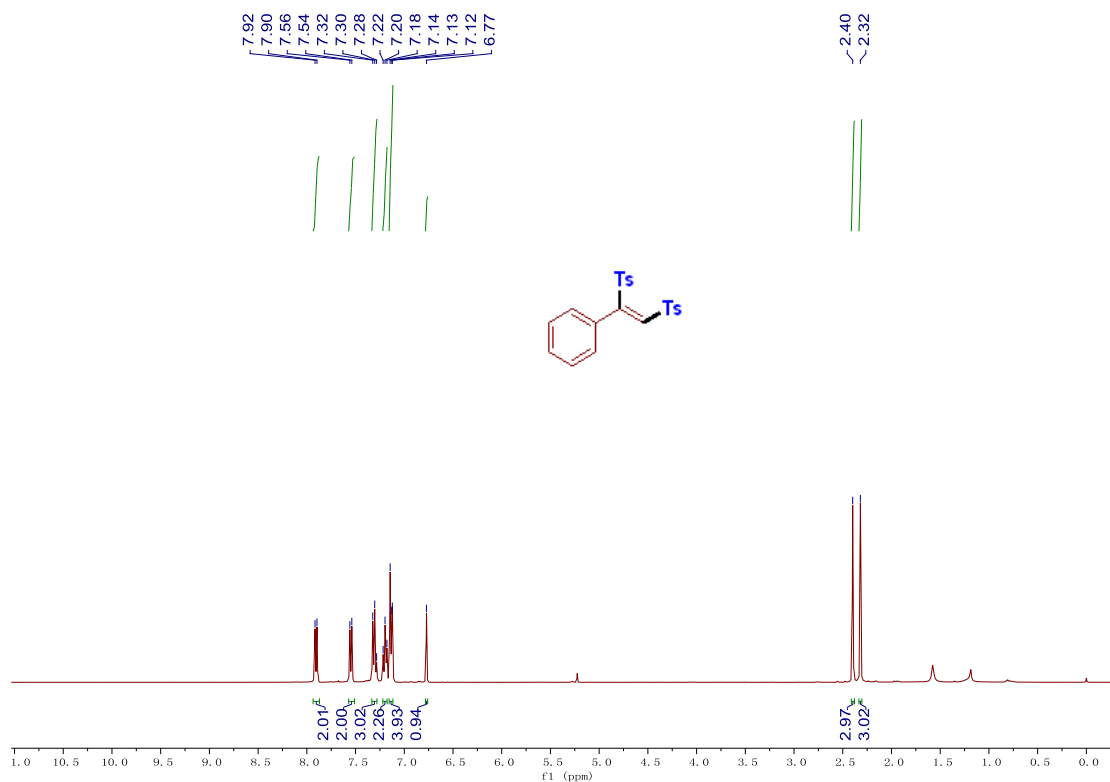
(*E*)-*N*,4-dimethyl-*N*-(3-phenyl-3-tosylallyl)aniline (**5b**): TLC (PE/EtOAc, 3:1),  $R_f = 0.52$ ; Colorless liquid (90.4 mg, 77%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.60 (d,  $J = 8.1$  Hz, 2H), 7.33 - 7.20 (m, 7H), 7.07 (d,  $J = 8.3$  Hz, 2H), 6.67 (d,  $J = 8.4$  Hz, 2H), 6.18 - 6.16 (m, 1H), 4.78 (d,  $J = 5.4$  Hz, 2H), 3.02 (s, 3H), 2.43 (s, 3H), 2.29 (s, 3H).  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ )  $\delta$  146.7, 144.5, 144.4, 143.7, 137.4, 135.0, 130.0, 129.9, 129.6, 128.8, 128.1, 128.0, 126.6, 113.4, 51.5, 39.1, 21.7, 20.3. HRMS (ESI):  $m/z$   $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{24}\text{H}_{25}\text{NNaO}_2\text{S}$ : 414.1504; found: 414.1510.

#### 4. References

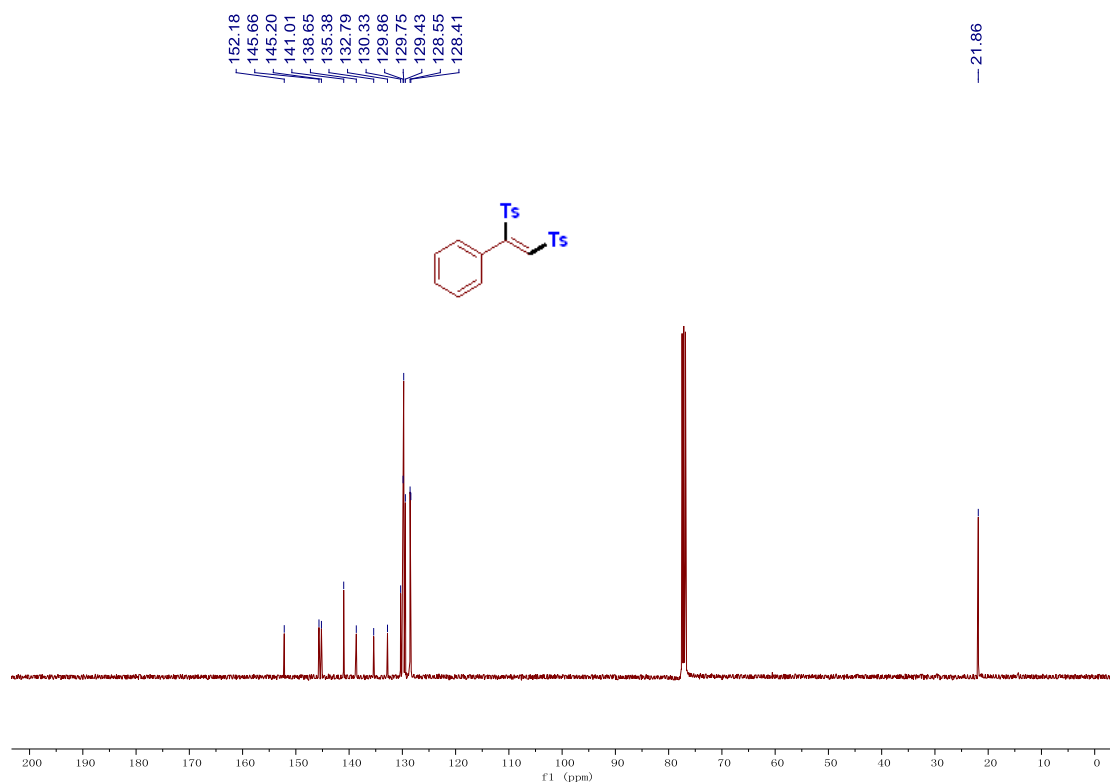
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- Gaussian 16, Revision C.01, M. J. Frisch, G. W. Trucks, H. B. Schlegel, G. E. Scuseria, M. A. Robb, J. R. Cheeseman, G. Scalmani, V. Barone, G. A. Petersson, H. Nakatsuji, X. Li, M. Caricato, A. V. Marenich, J. Bloino, B. G. Janesko, R. Gomperts, B. Mennucci, H. P. Hratchian, J. V. Ortiz, A. F. Izmaylov, J. L. Sonnenberg, D. Williams-Young, F. Ding, F. Lipparini, F. Egidi, J. Goings, B. Peng, A. Petrone, T. Henderson, D. Ranasinghe, V. G. Zakrzewski, J. Gao, N. Rega, G. Zheng, W. Liang, M. Hada, M. Ehara, K. Toyota, R. Fukuda, J. Hasegawa, M. Ishida, T. Nakajima, Y. Honda, O. Kitao, H. Nakai, T. Vreven, K. Throssell, J. A. Montgomery, Jr., J. E. Peralta, F. Ogliaro, M. J. Bearpark, J. J. Heyd, E. N. Brothers, K. N. Kudin, V. N. Staroverov, T. A. Keith, R. Kobayashi, J. Normand, K. Raghavachari, A. P. Rendell, J. C. Burant, S. S. Iyengar,

J. Tomasi, M. Cossi, J. M. Millam, M. Klene, C. Adamo, R. Cammi, J. W. Ochterski, R. L. Martin, K. Morokuma, O. Farkas, J. B. Foresman, and D. J. Fox, Gaussian, Inc., Wallingford CT, 2019.

#### 4. NMR spectra of products

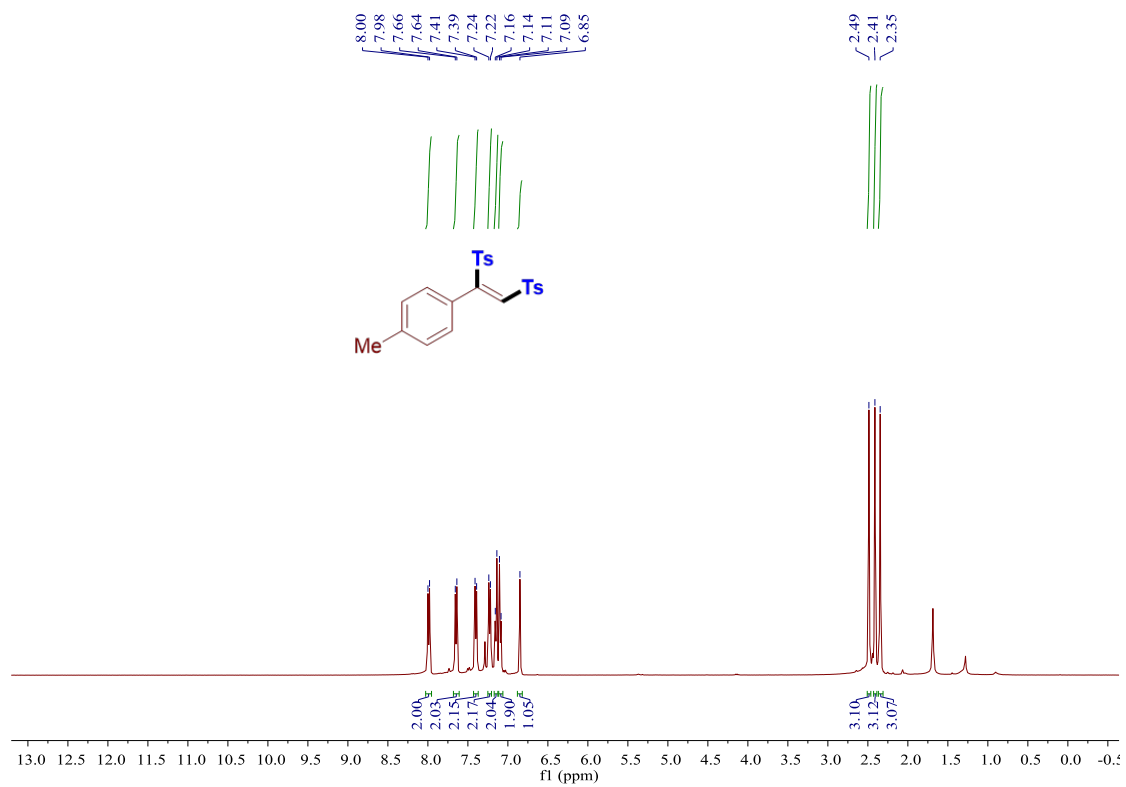


<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectra of **3aa**

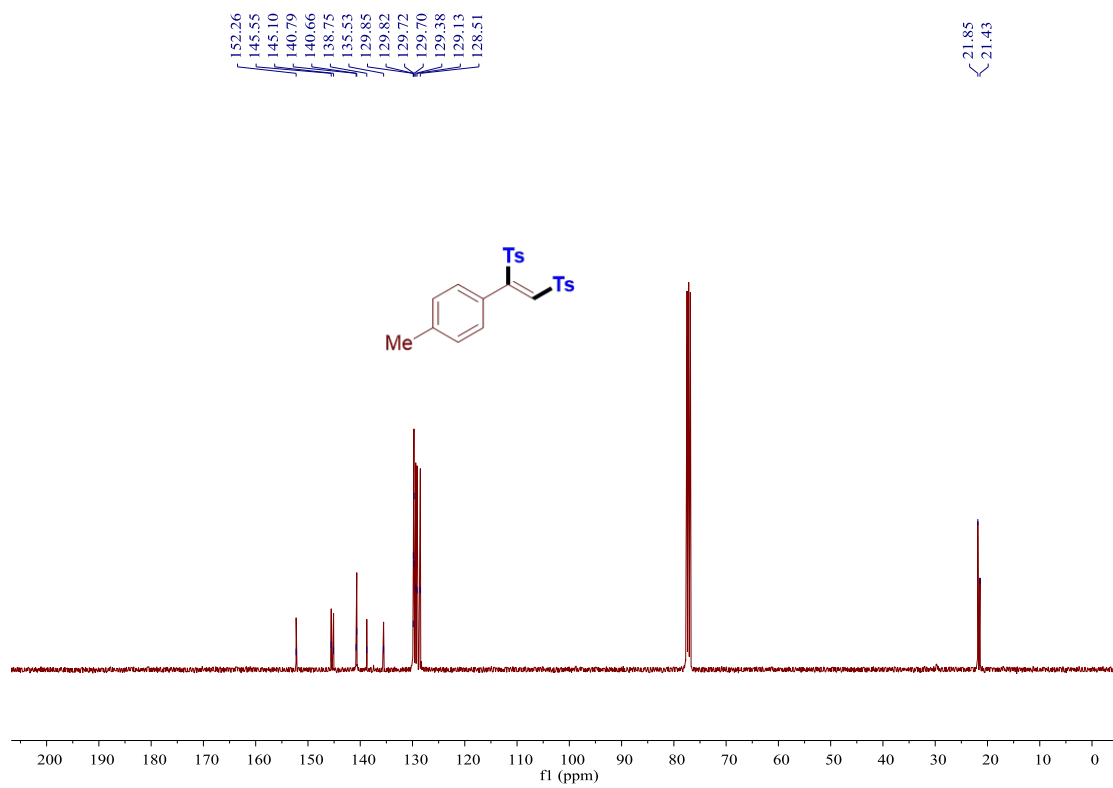


<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of **3aa**

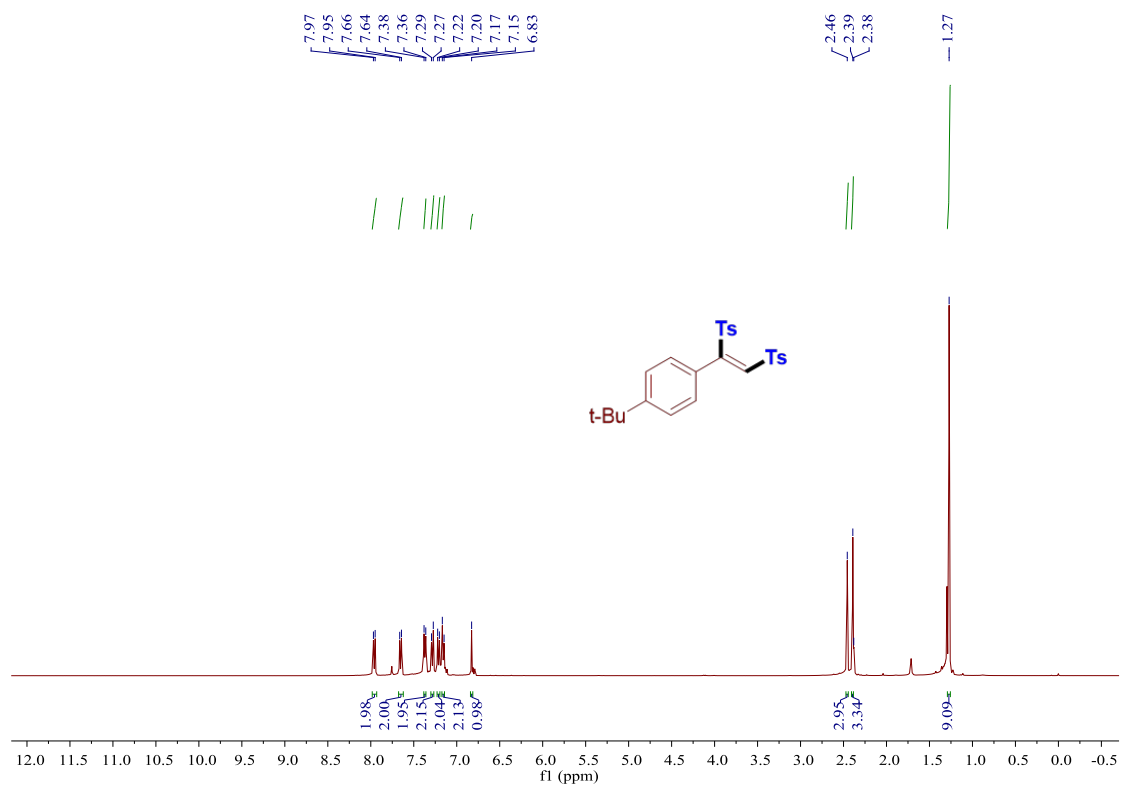




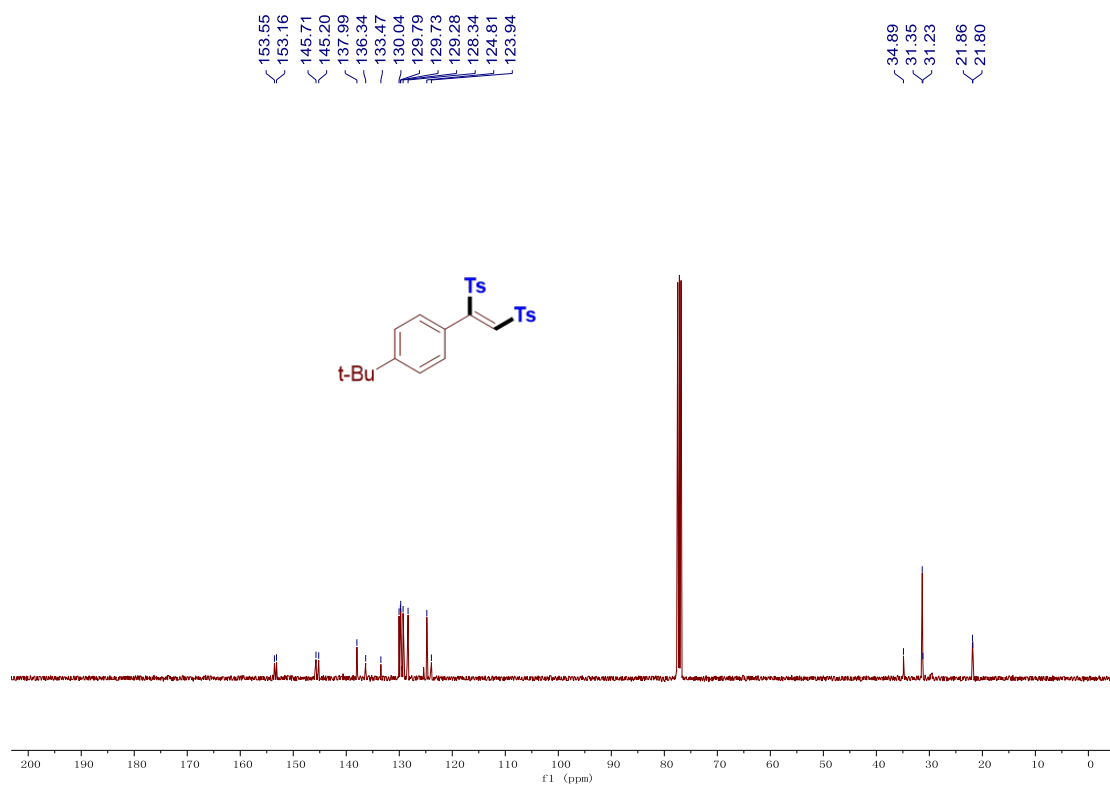
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectra of **3ba**



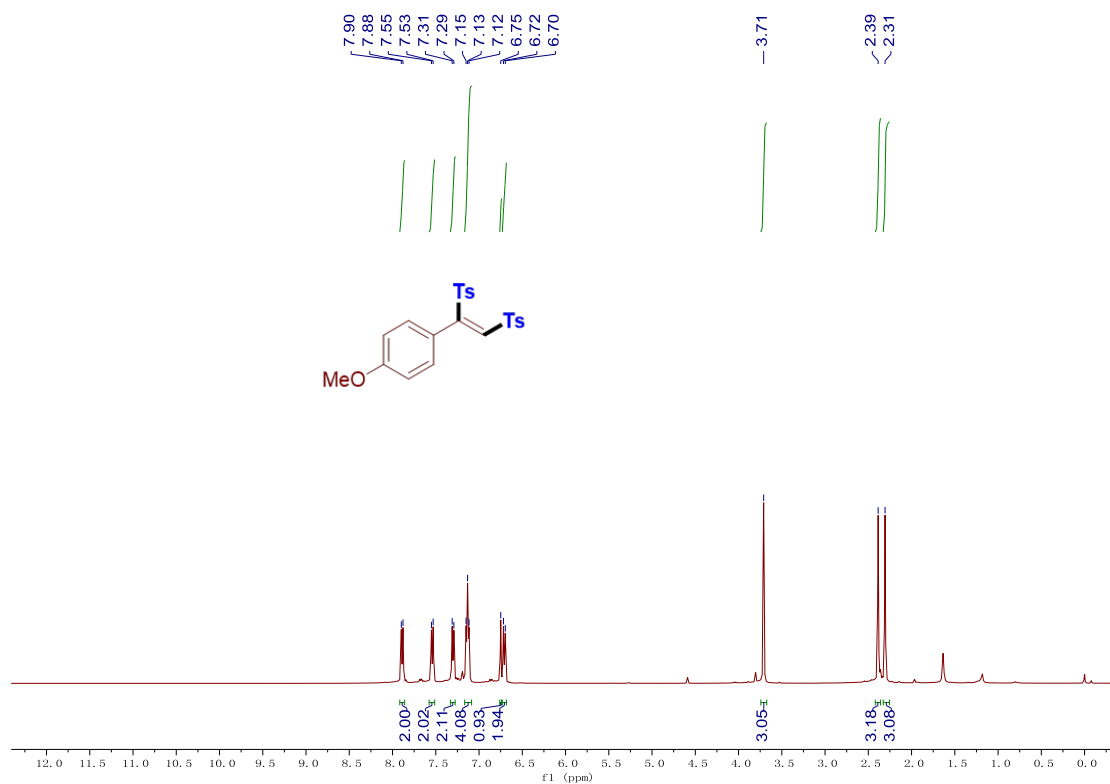
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of **3ba**



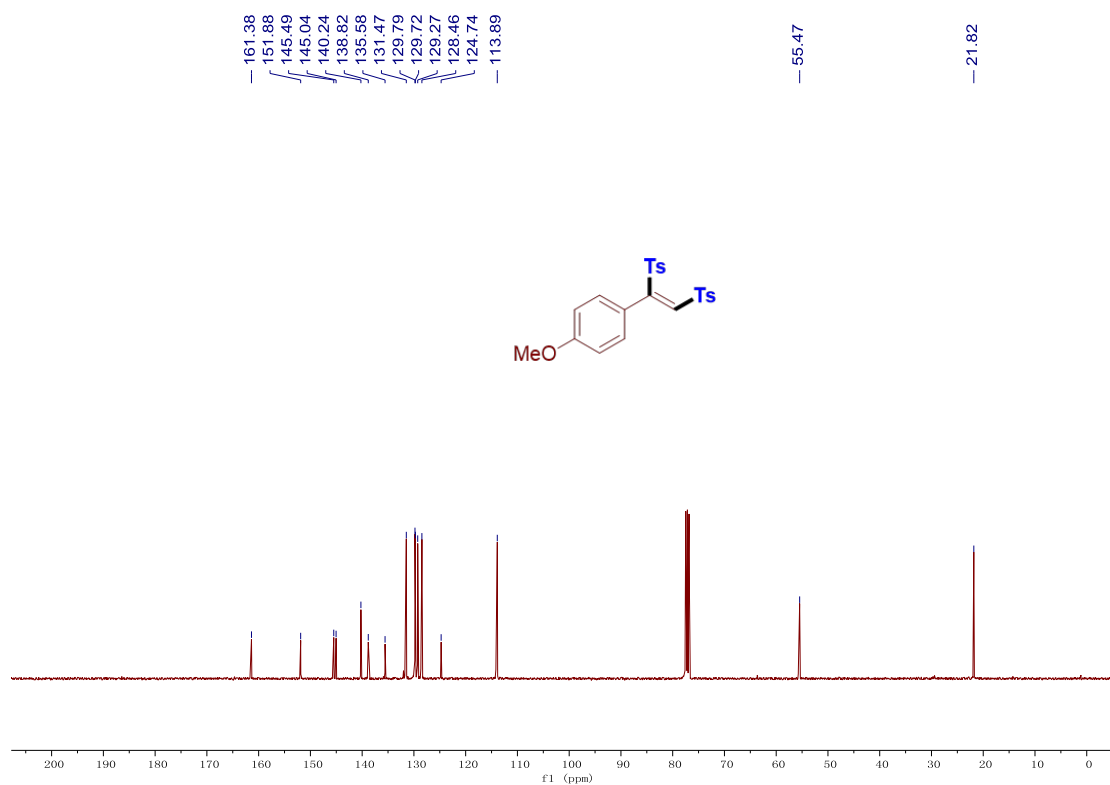
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectra of **3ca**



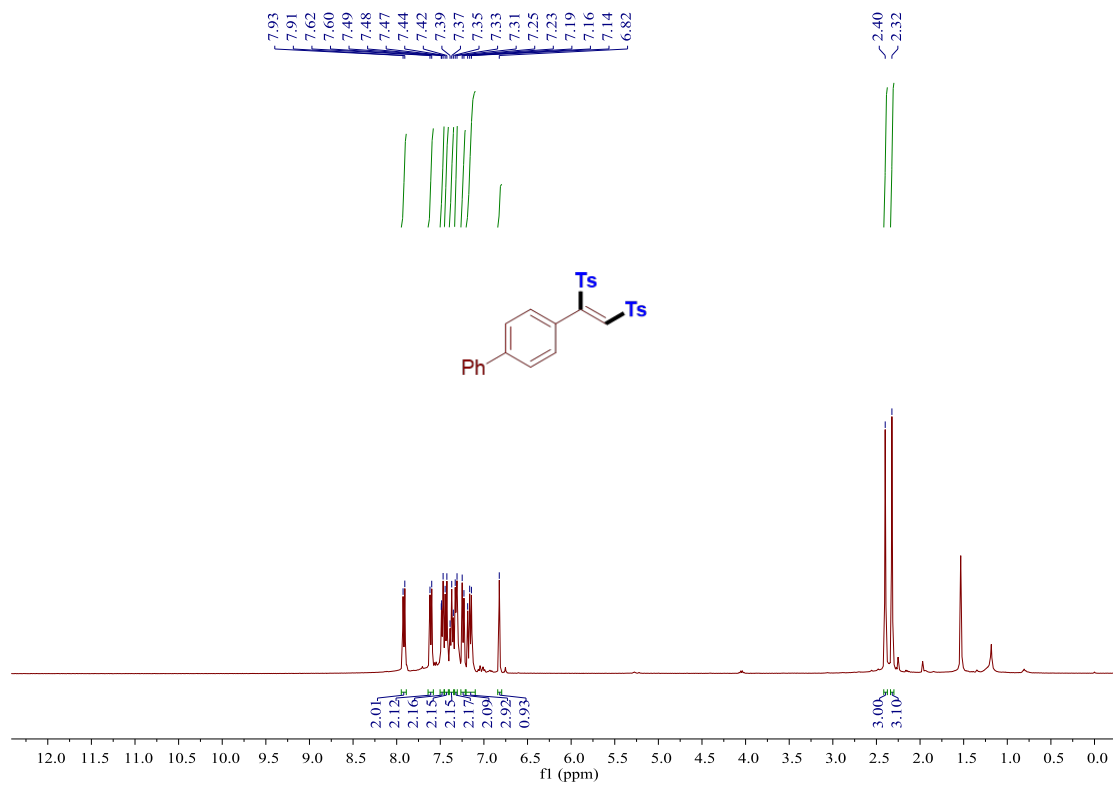
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of **3ca**



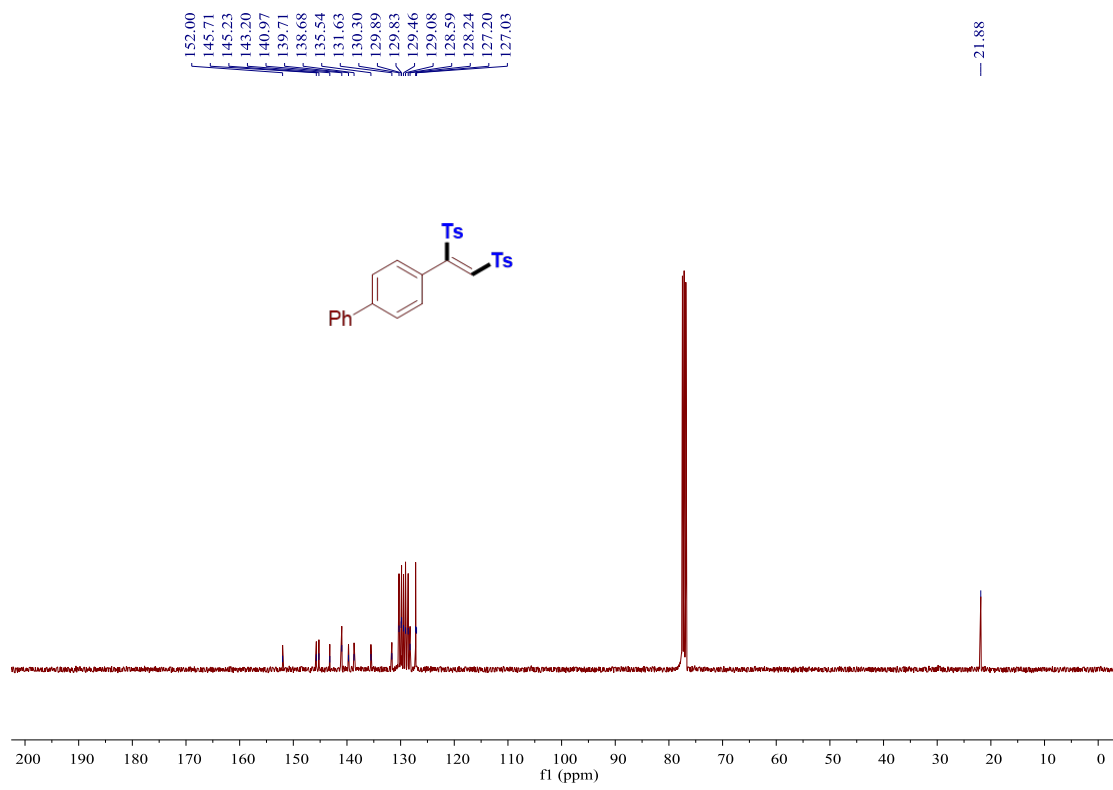
$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ) spectra of **3da**



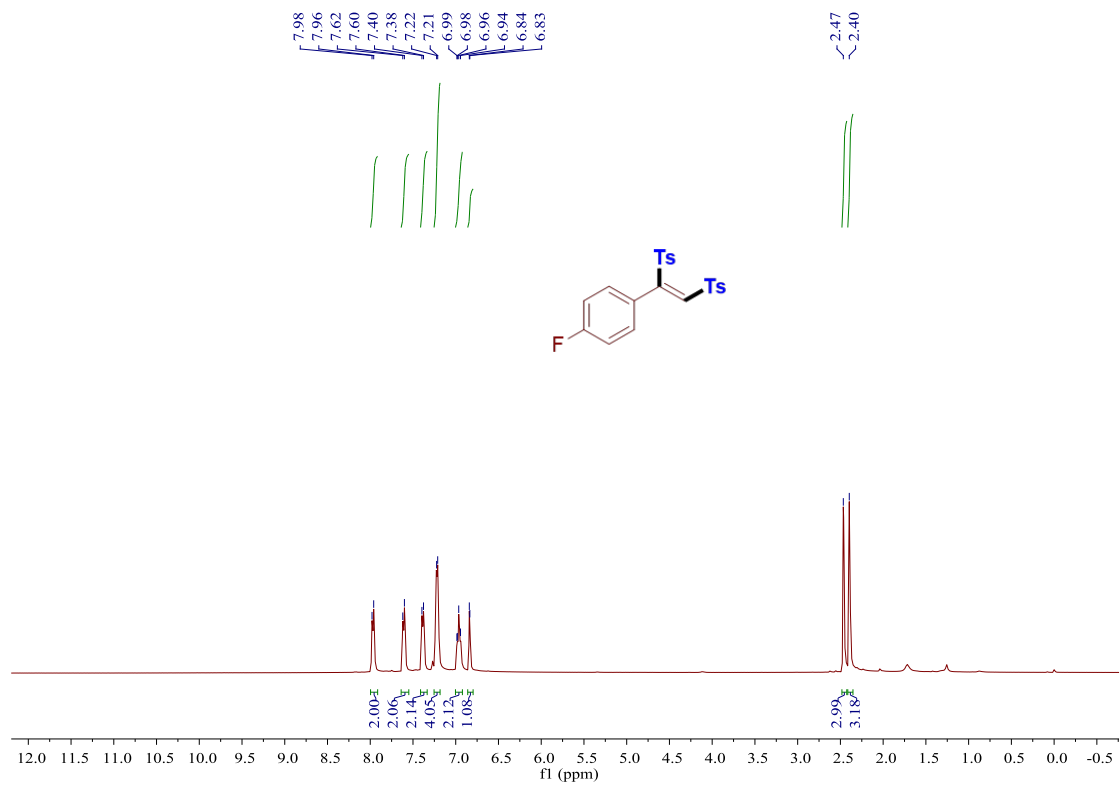
$^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ ) spectra of **3da**



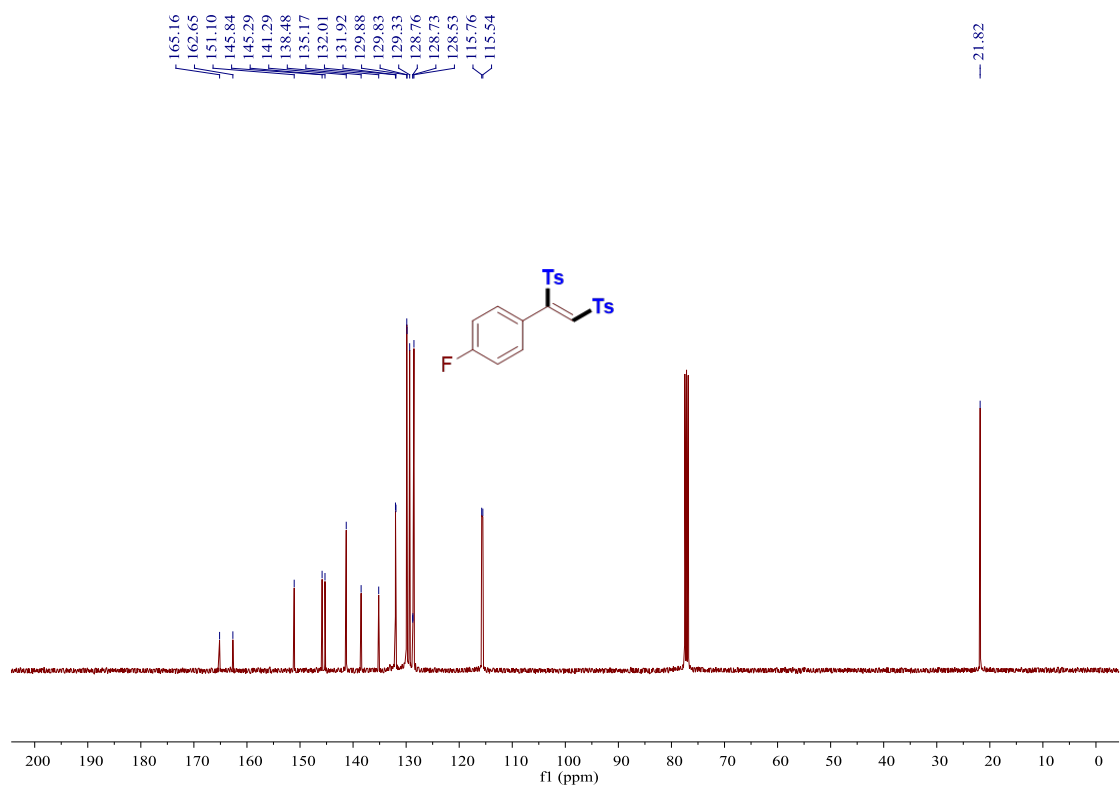
$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ) spectra of **3ea**



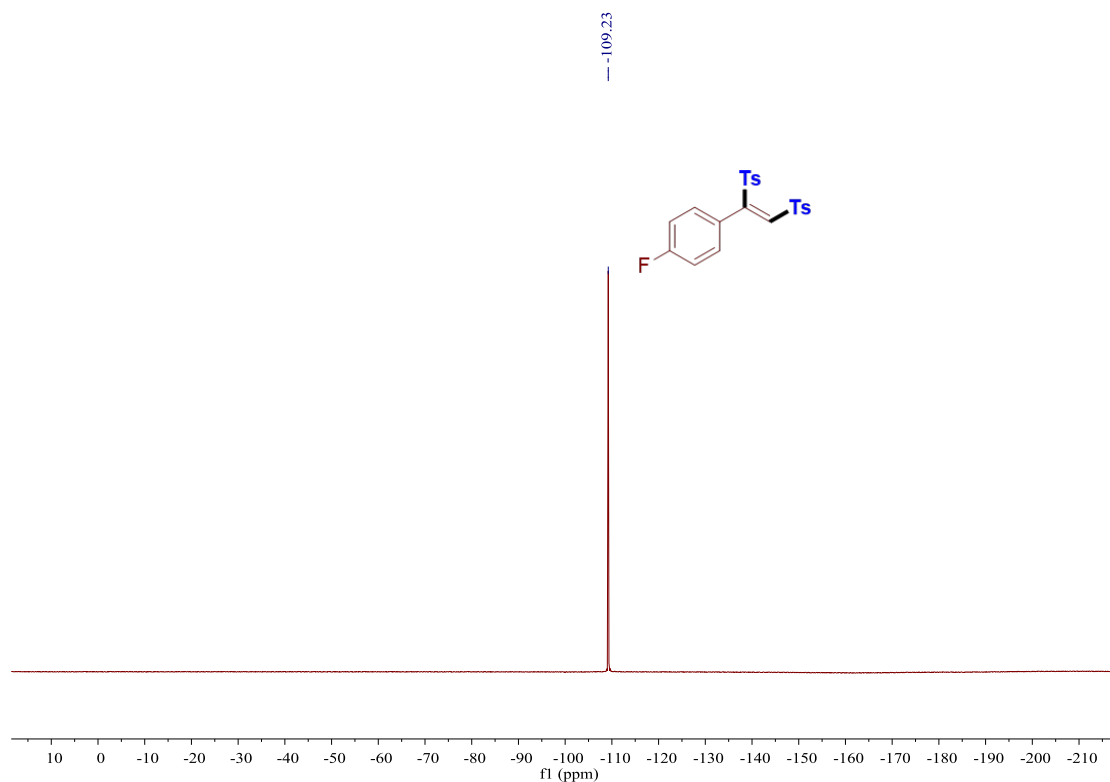
$^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ ) spectra of **3ea**



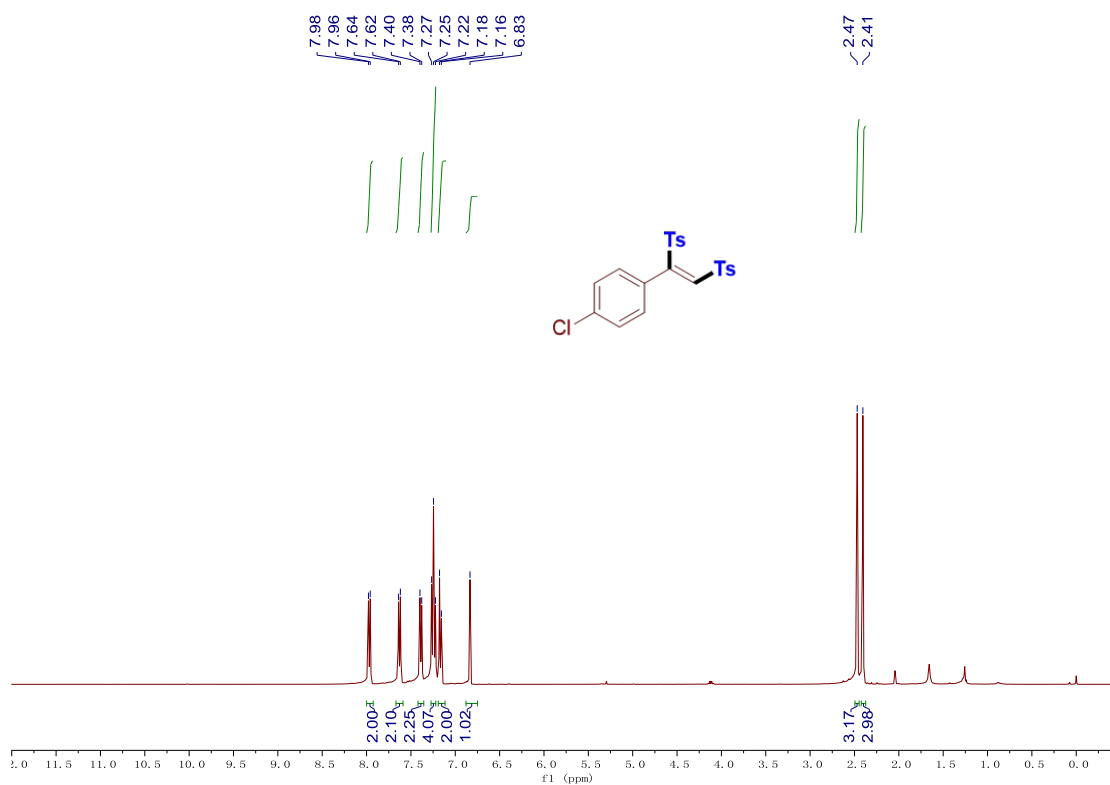
$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ) spectra of **3fa**



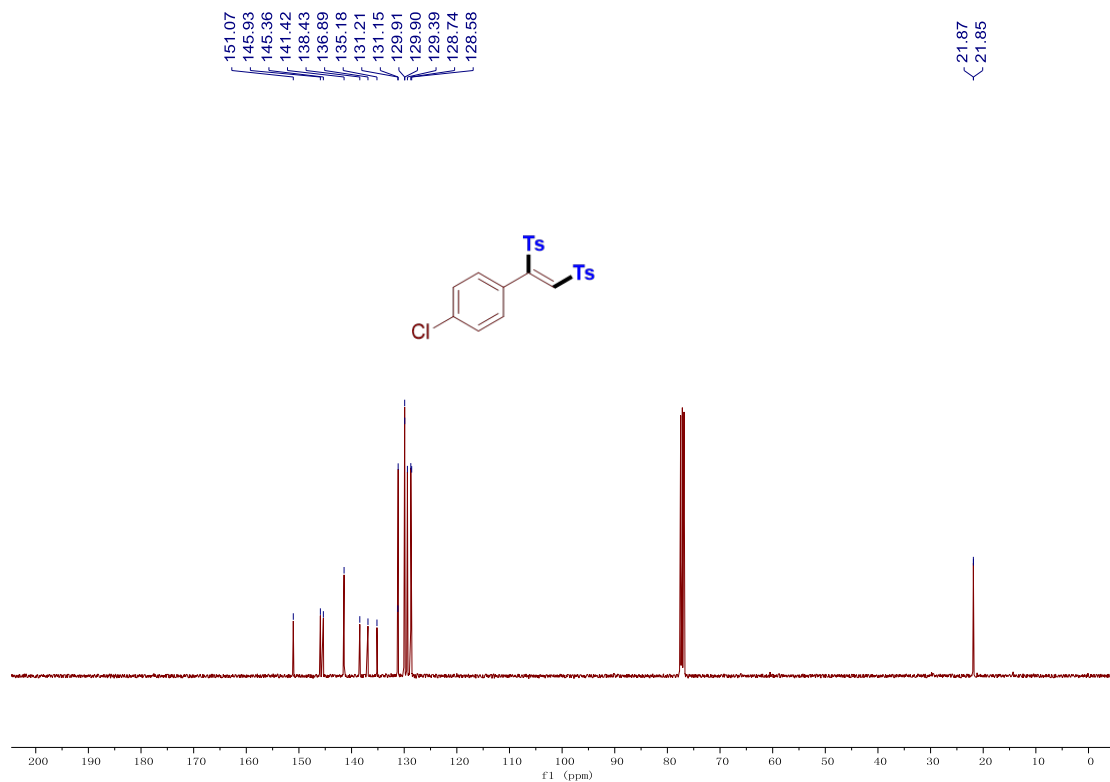
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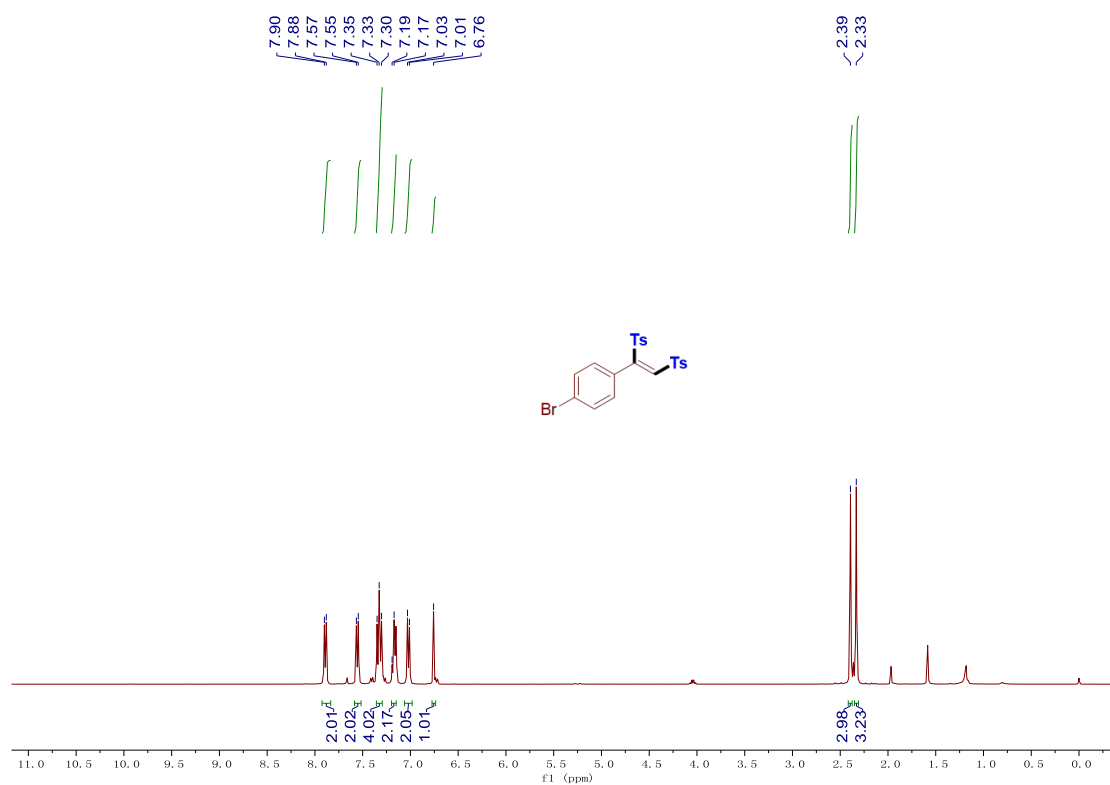
$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ ) spectra of **3fa**



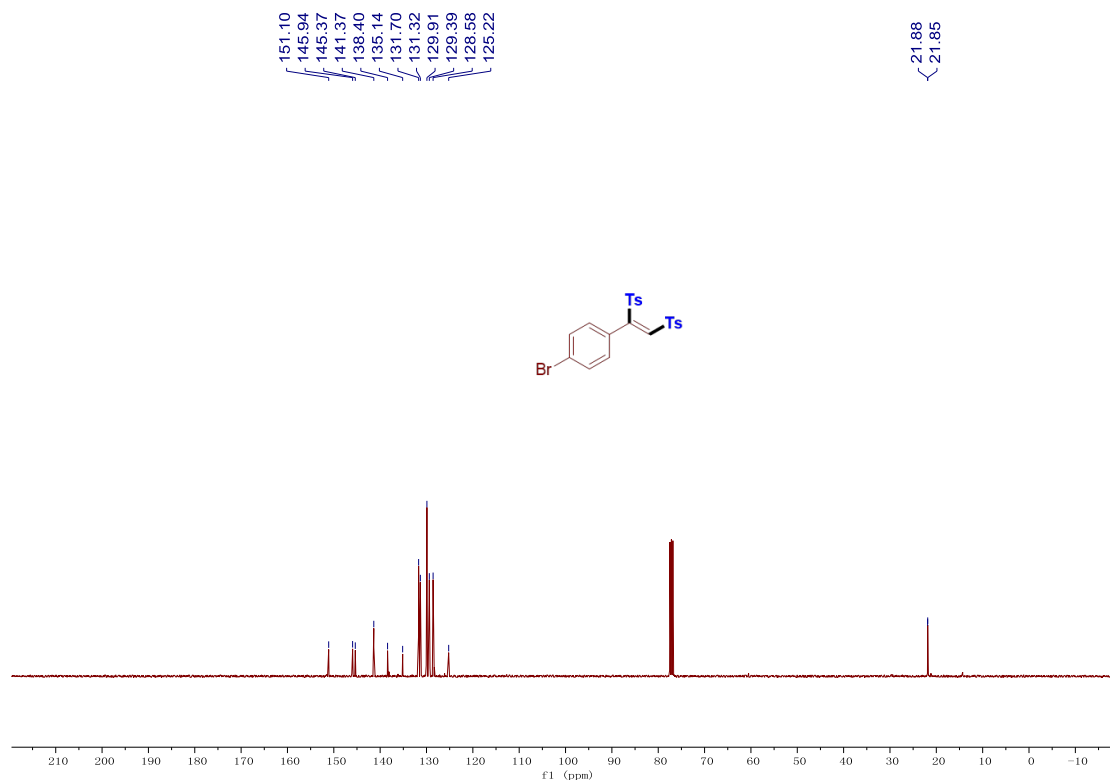
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) spectra of **3ga**



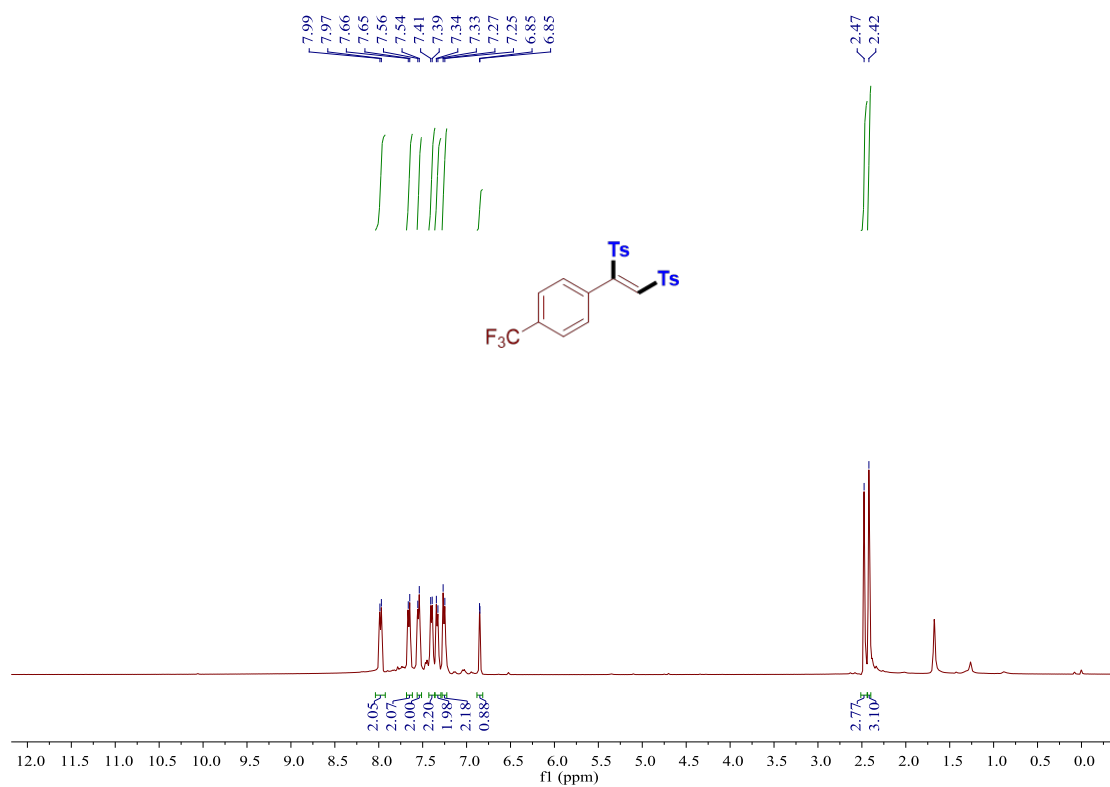
$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectra of **3ga**



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) spectra of **3ha**

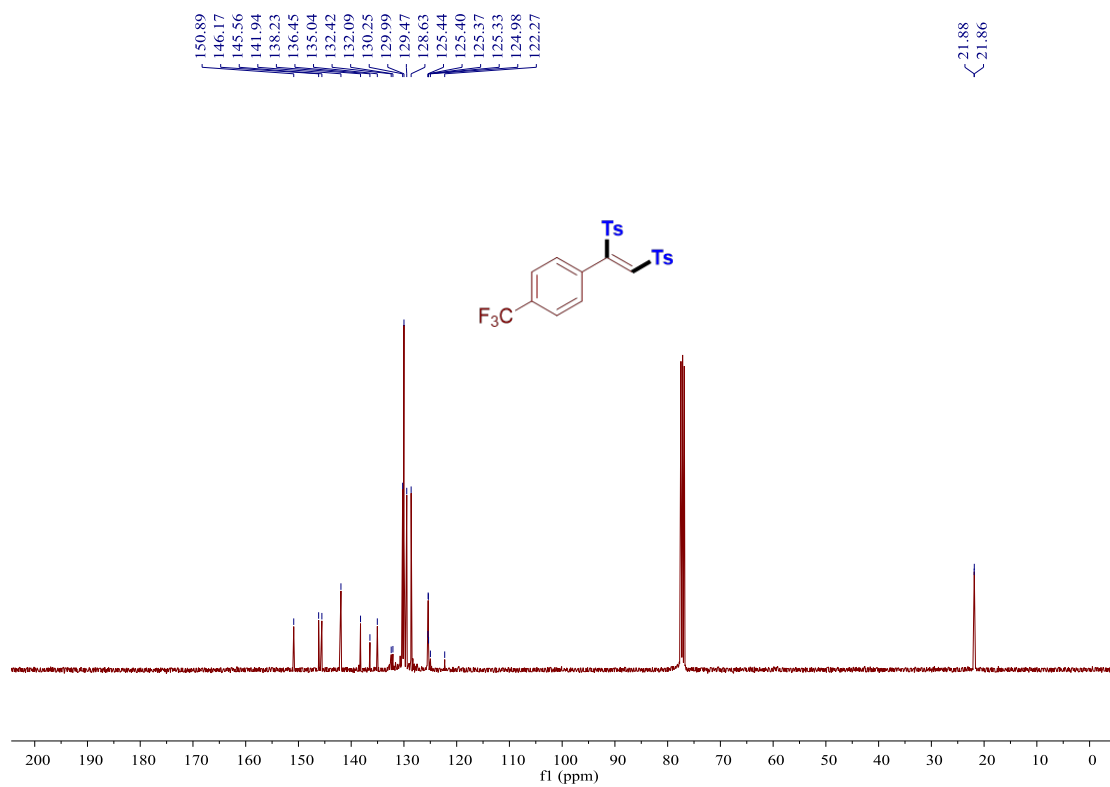


$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectra of **3a**

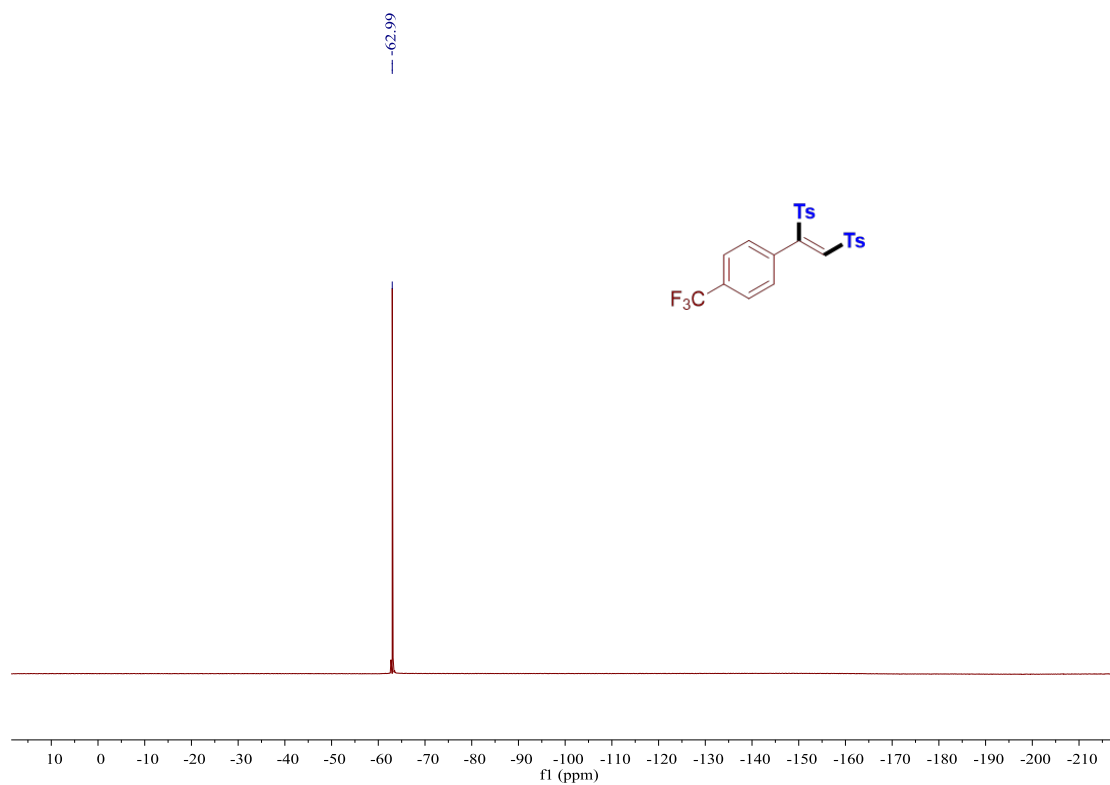


$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) spectra of **3ia**

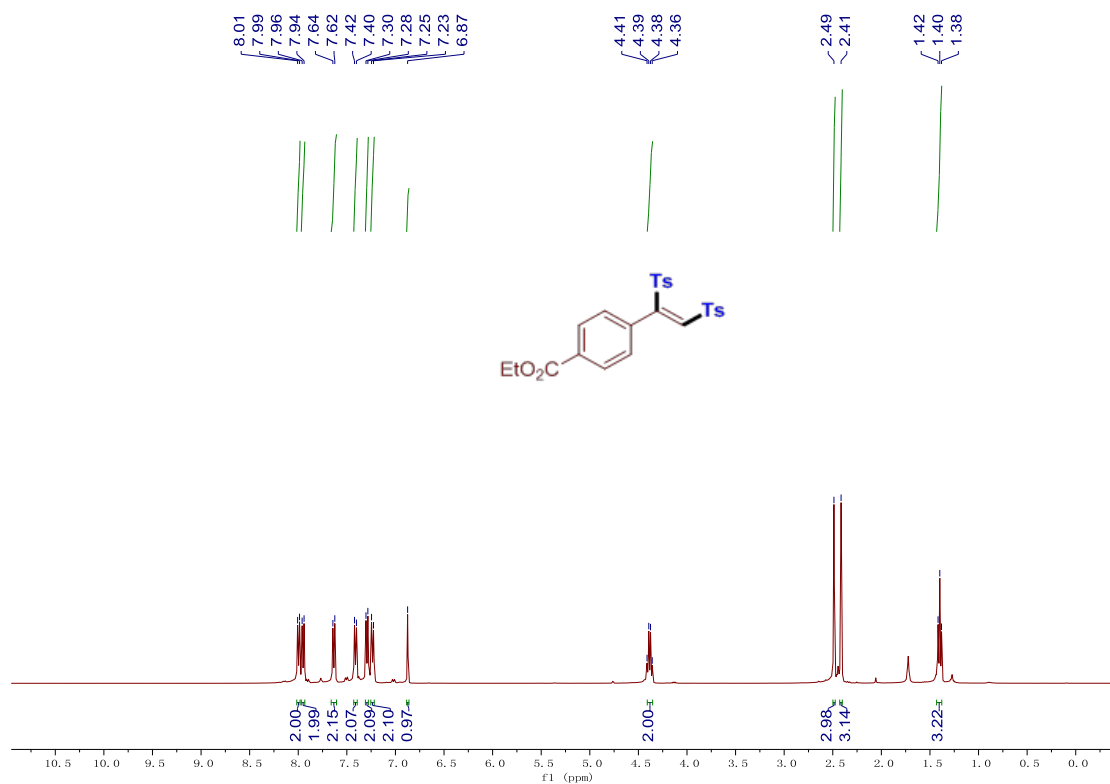




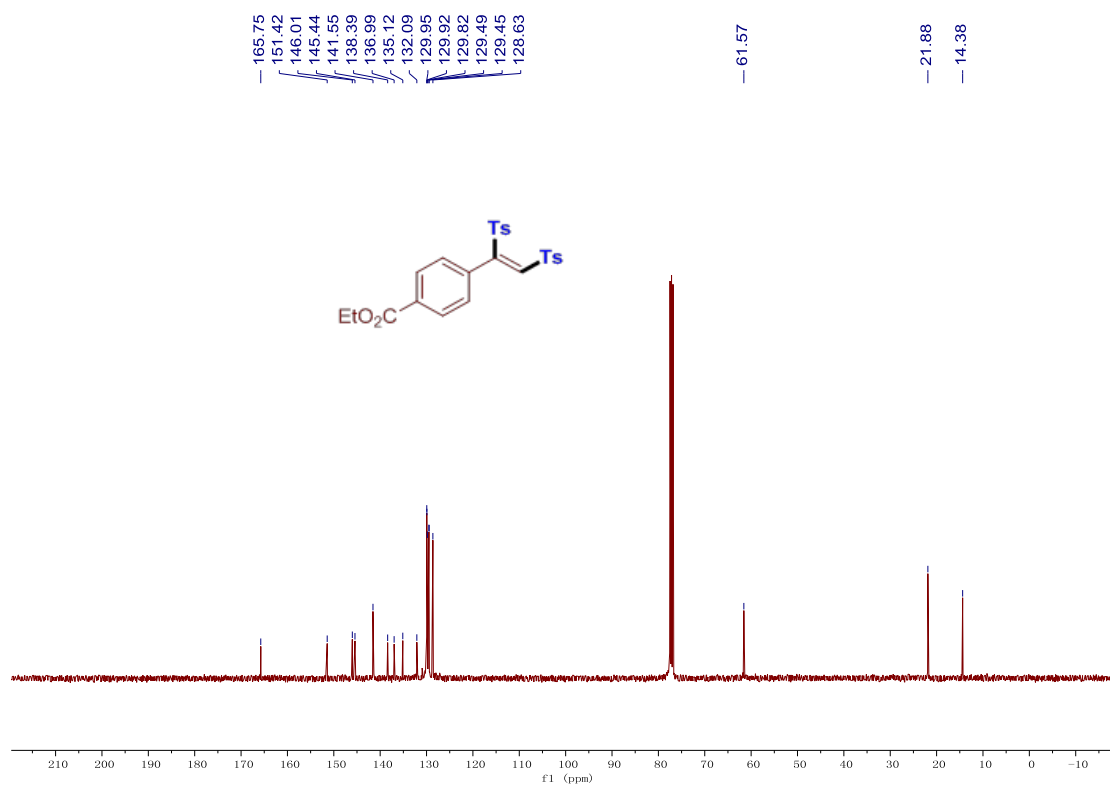
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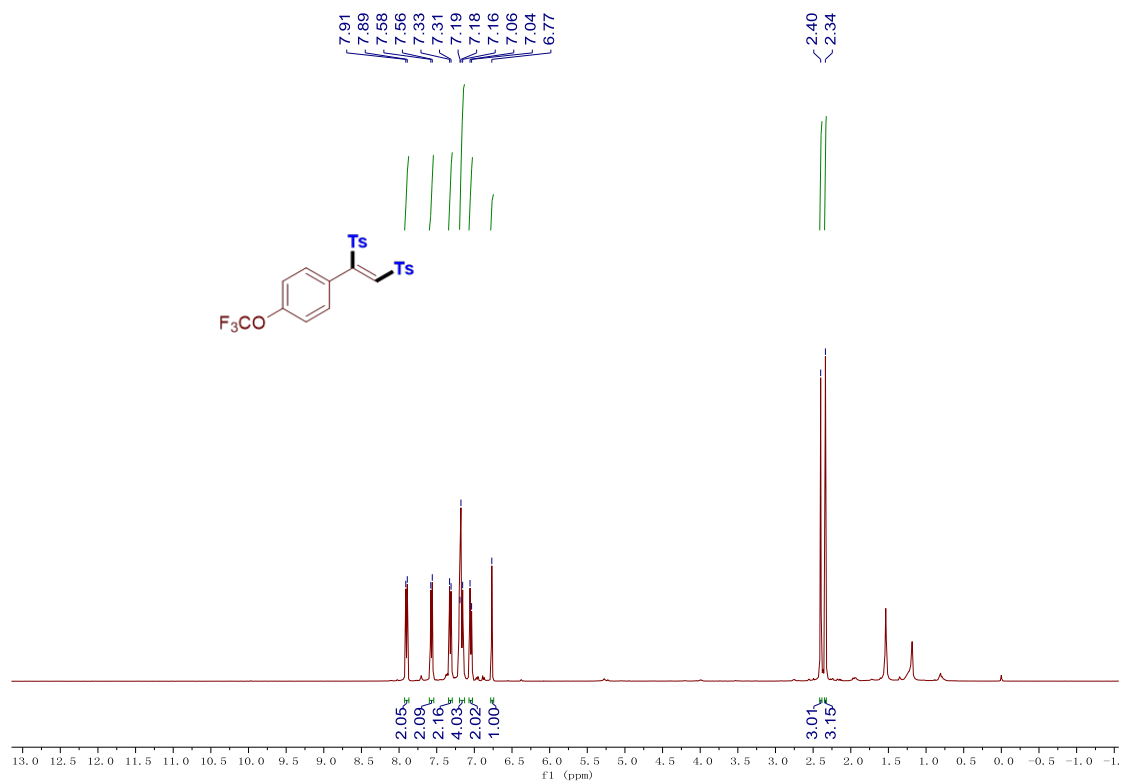
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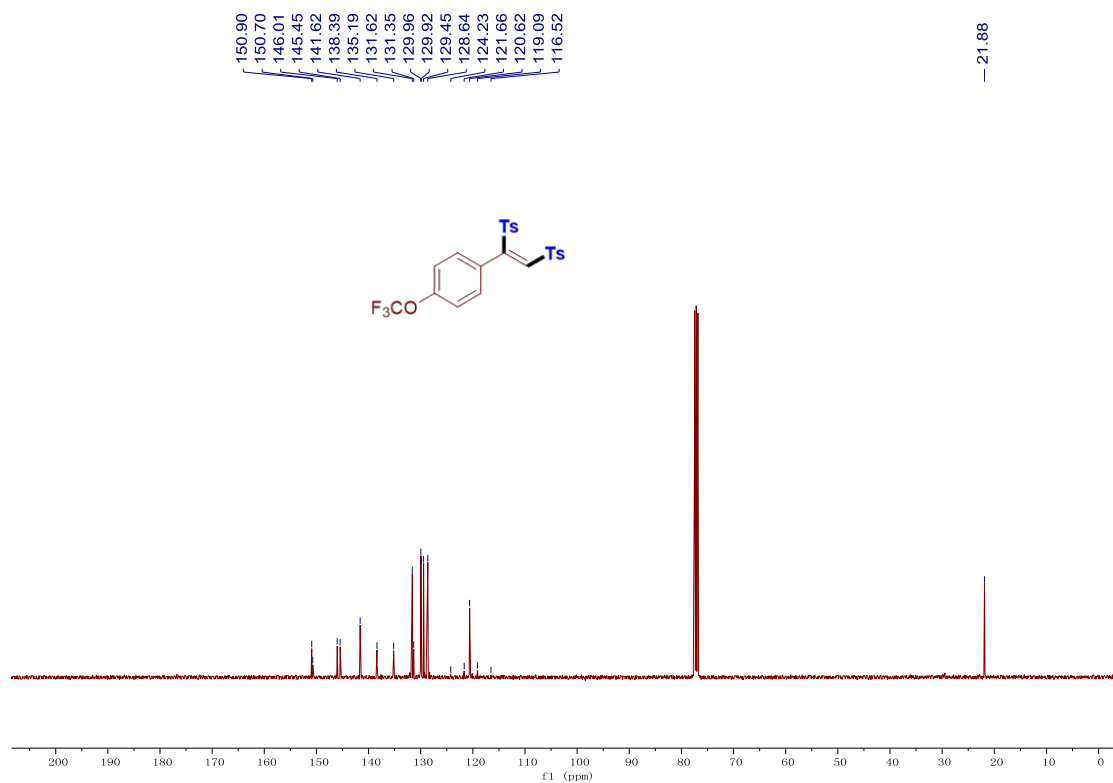
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectra of **3ja**



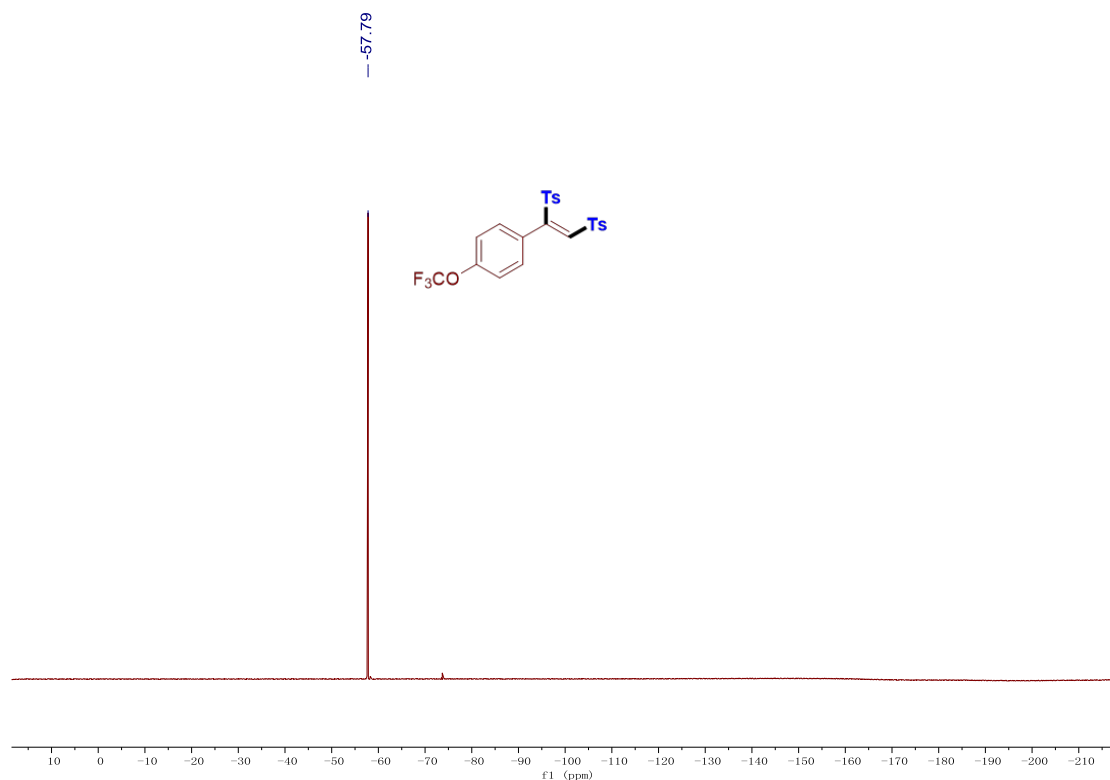
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of **3ja**



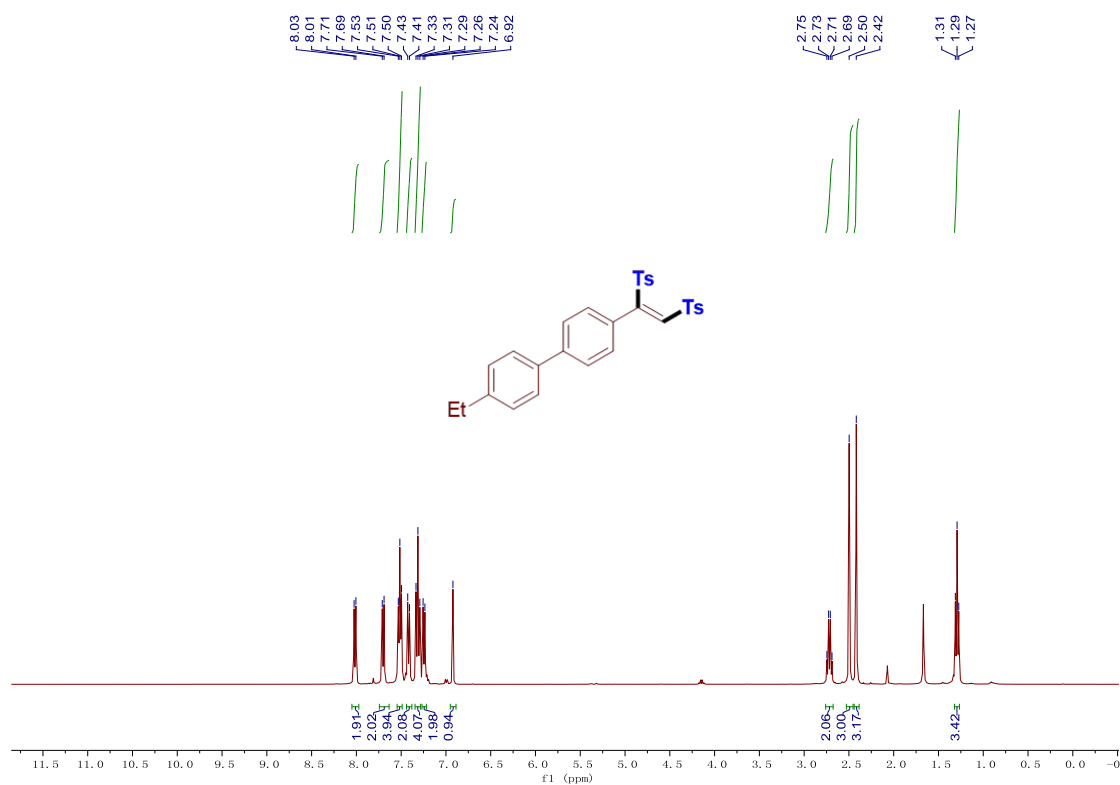
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectra of **3ka**



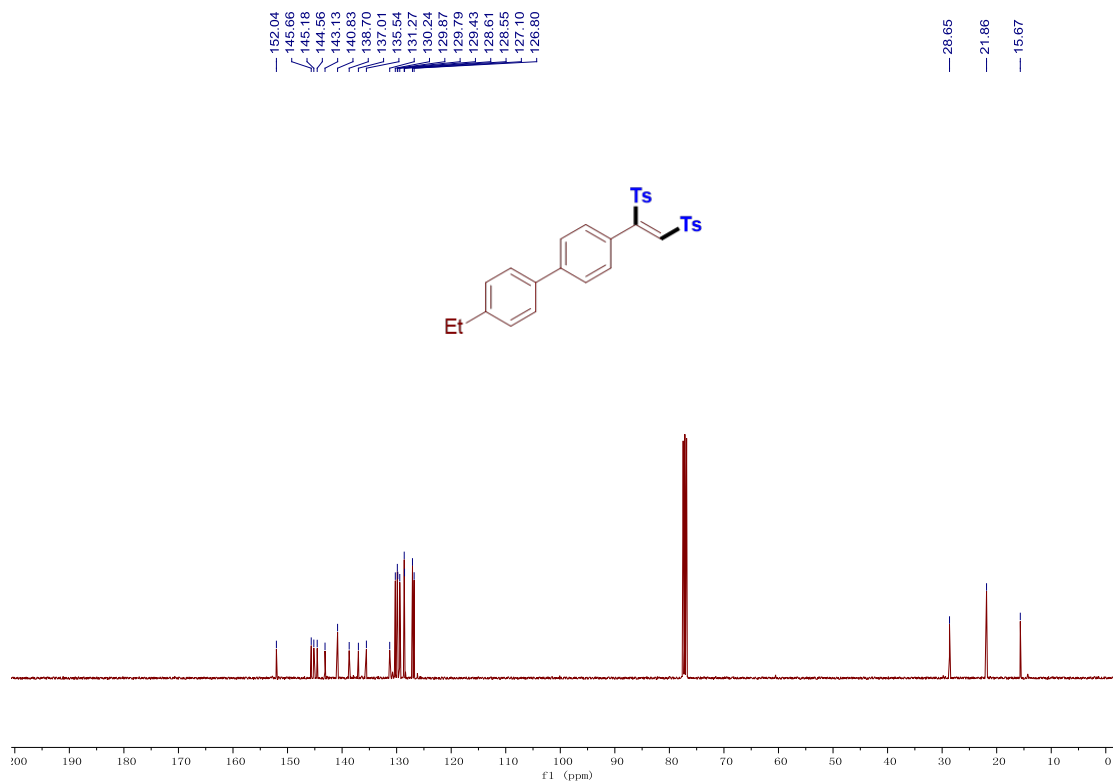
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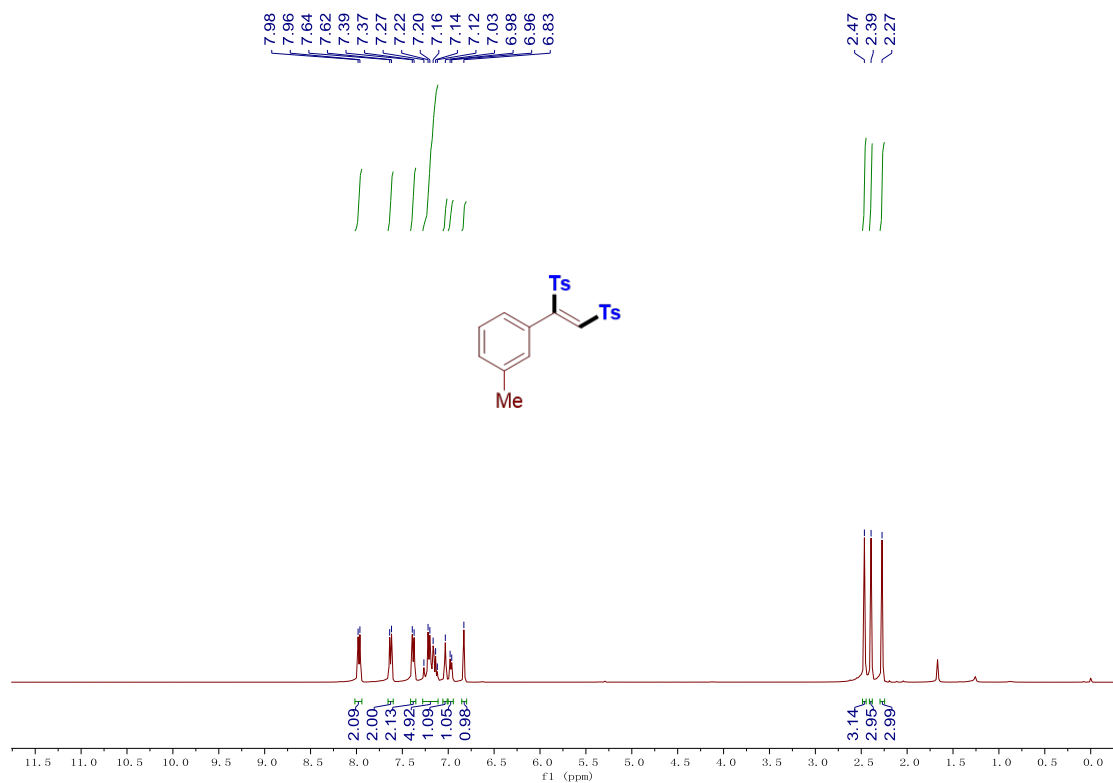
$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ ) spectra of **3ka**



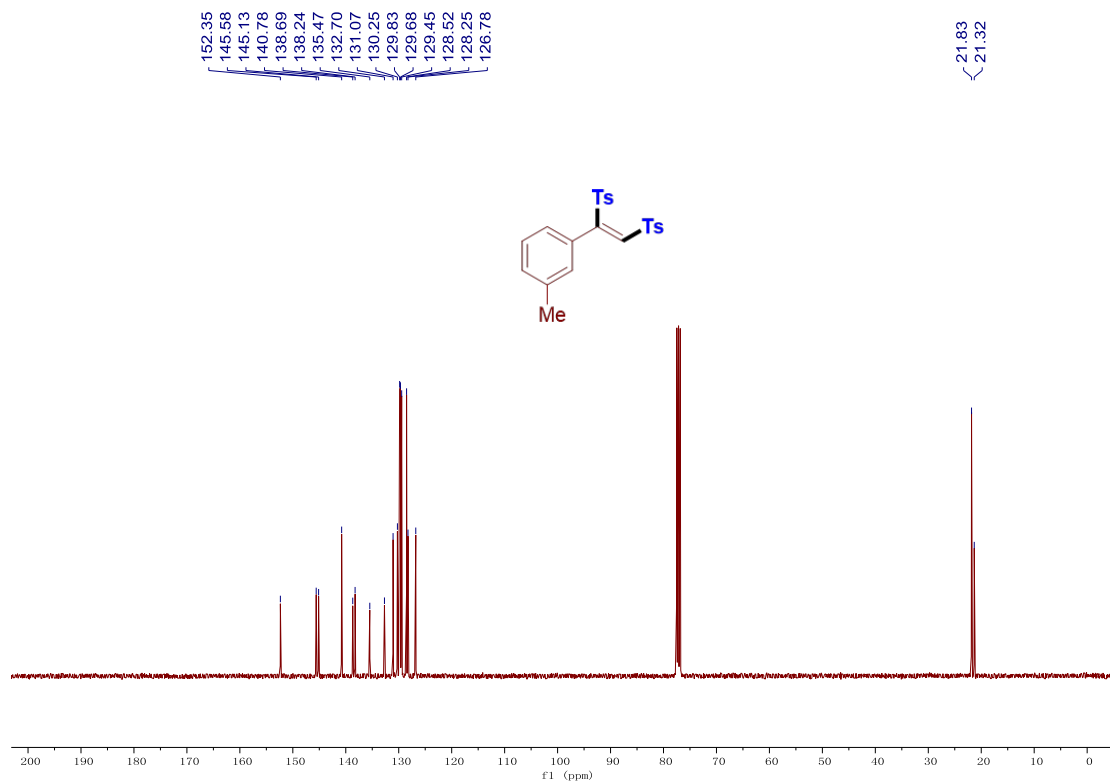
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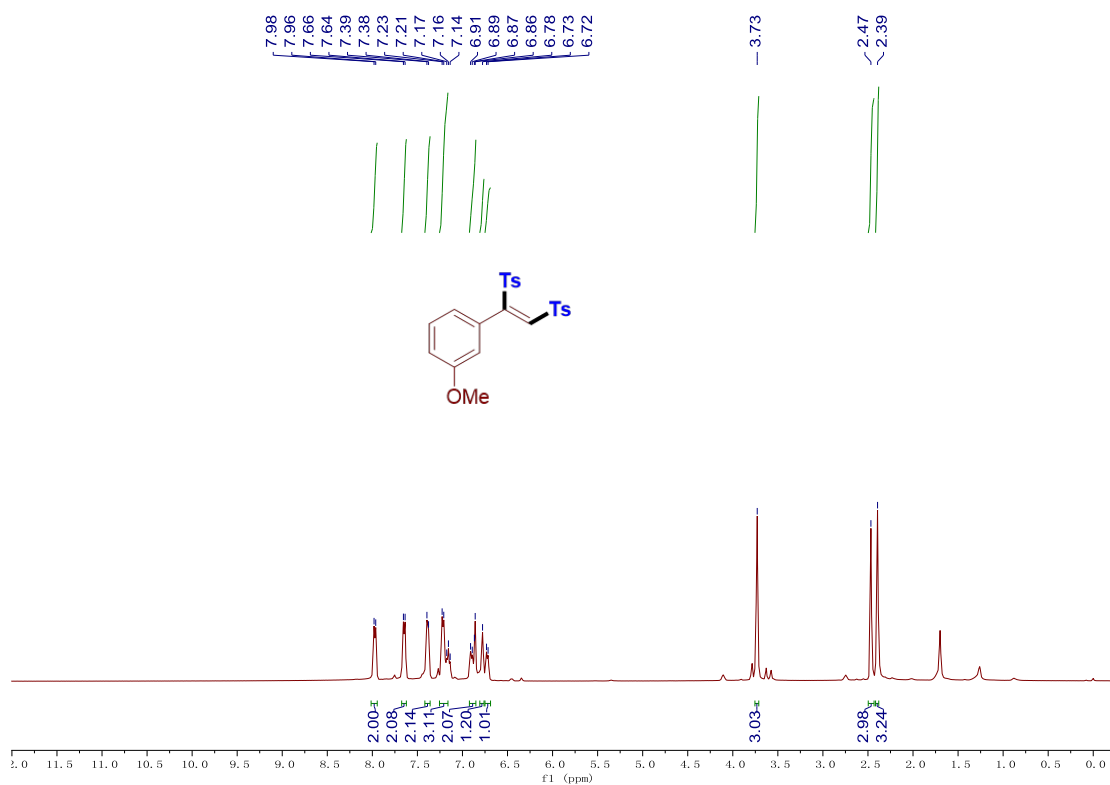
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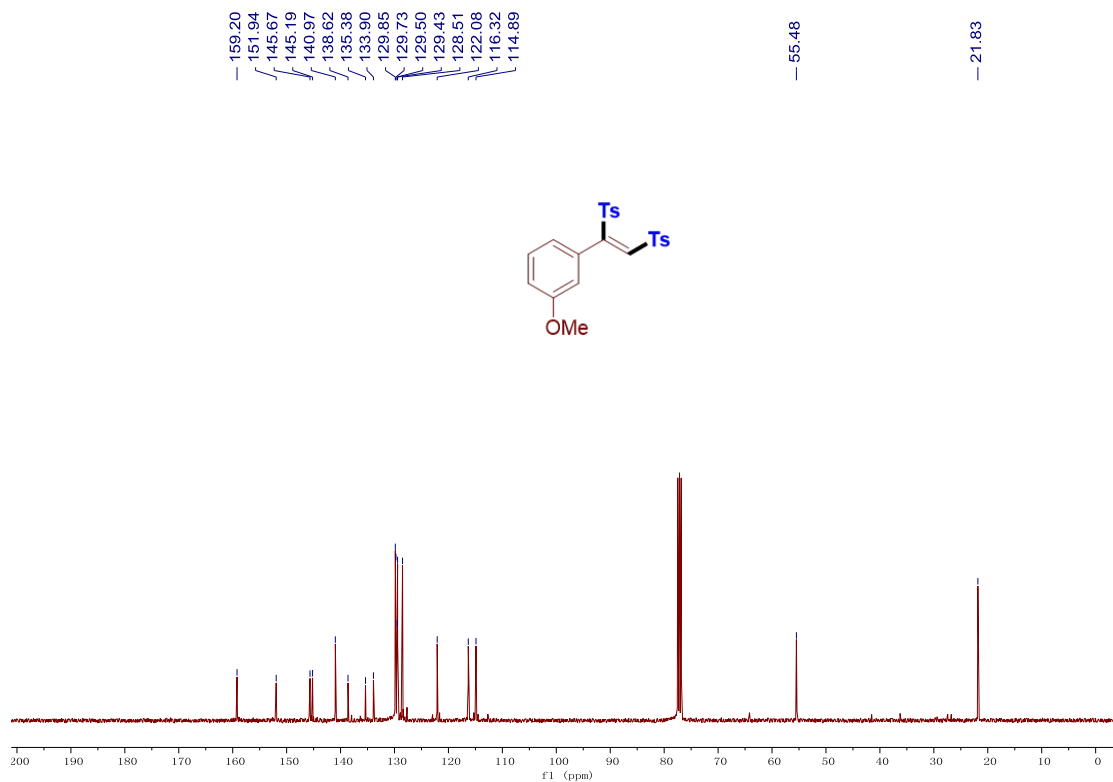
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) spectra of **3ma**



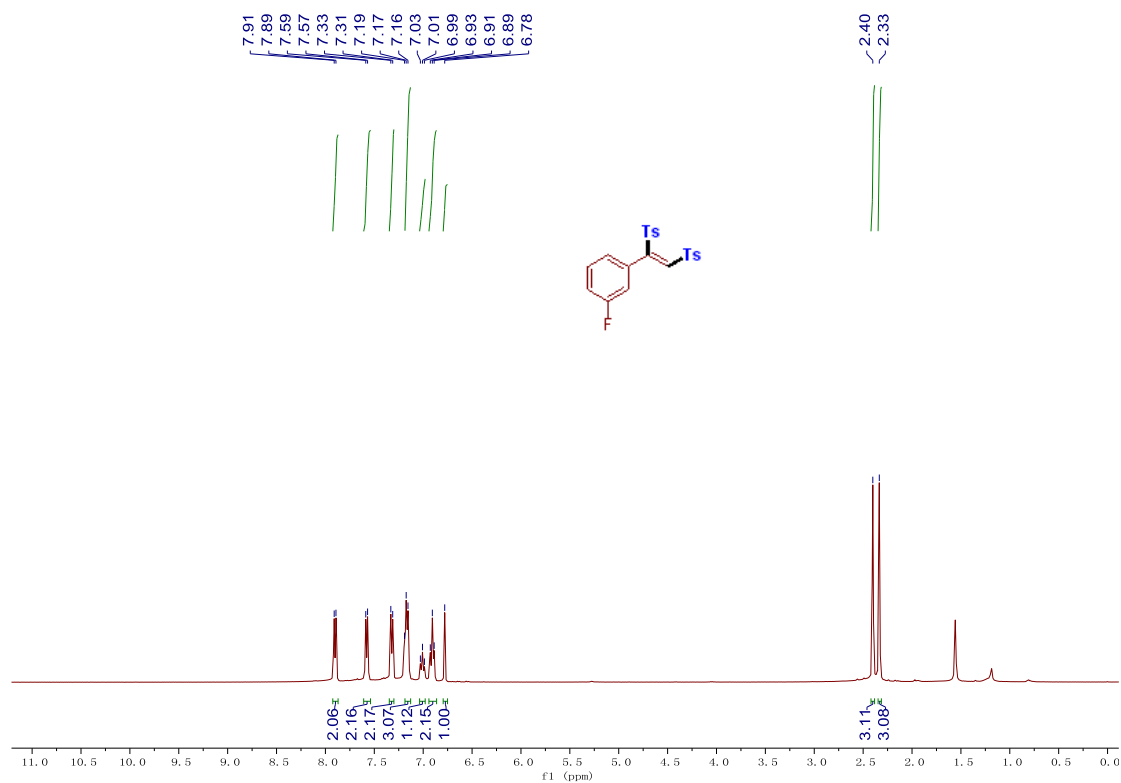
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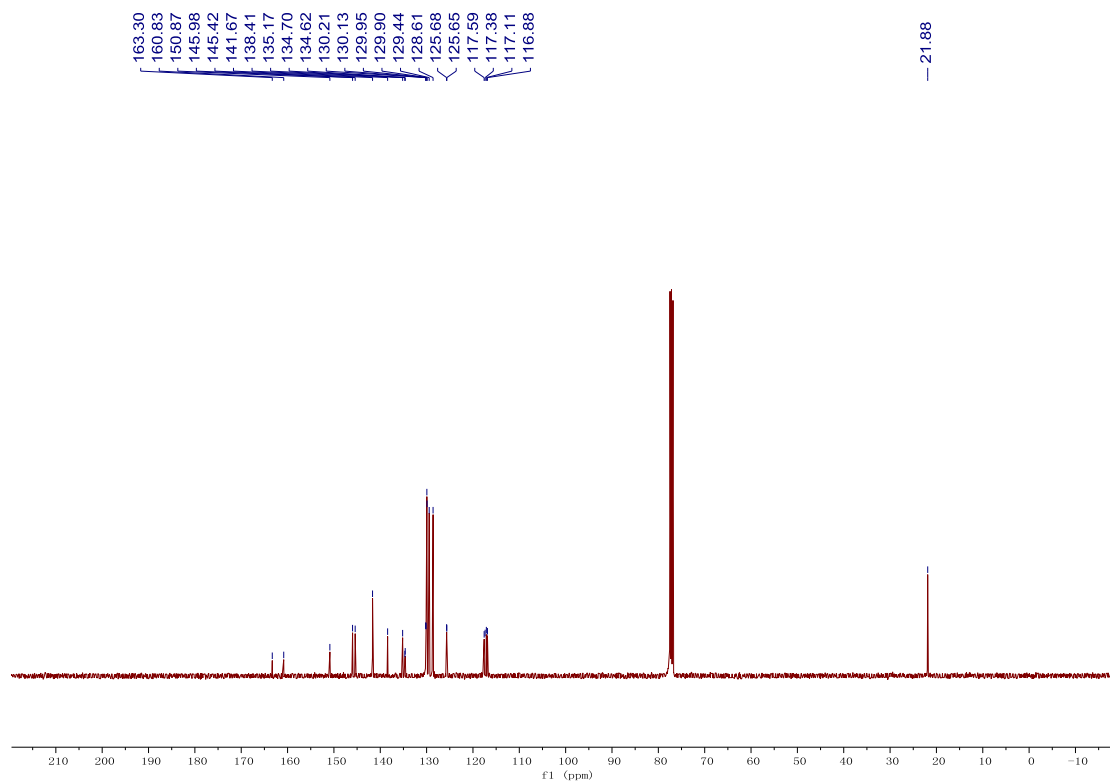
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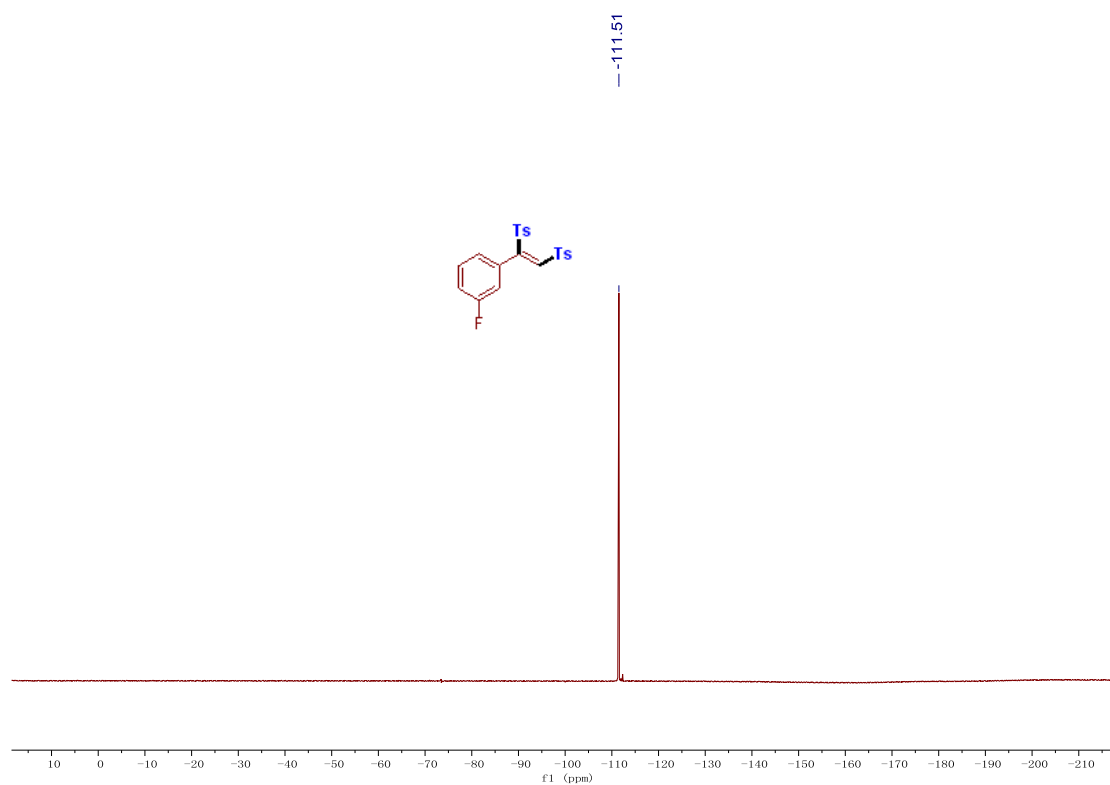
$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectra of **3na**



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) spectra of **3oa**

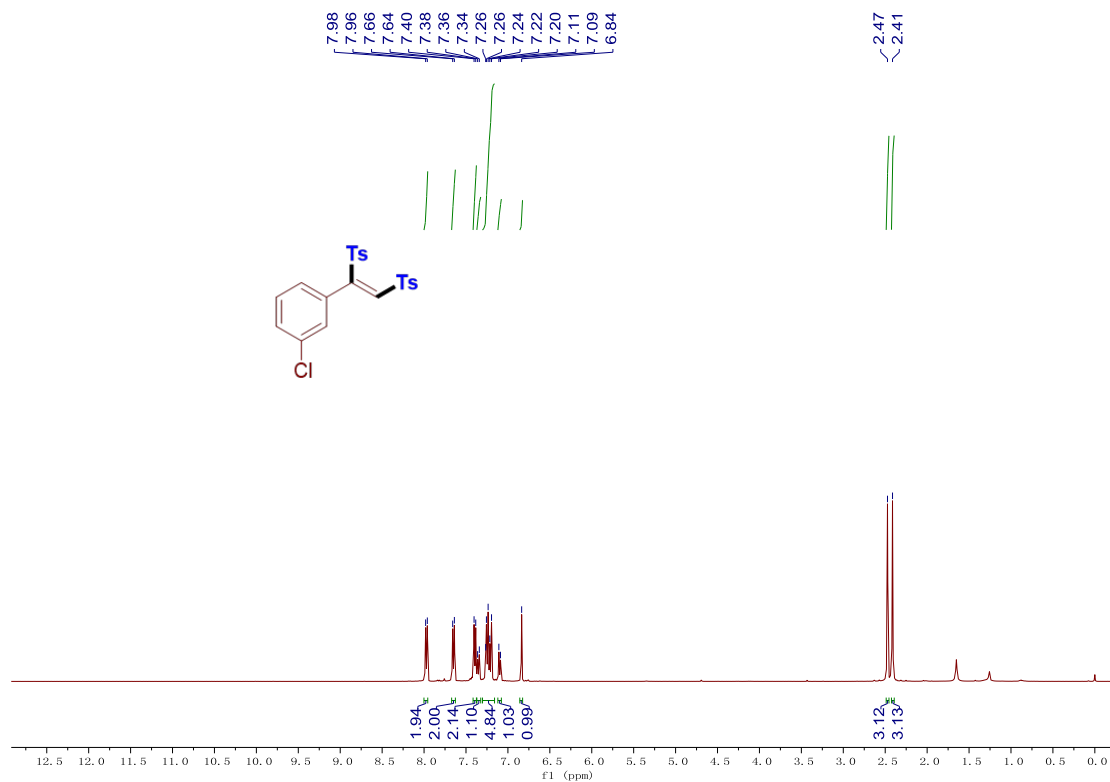


$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectra of **30a**

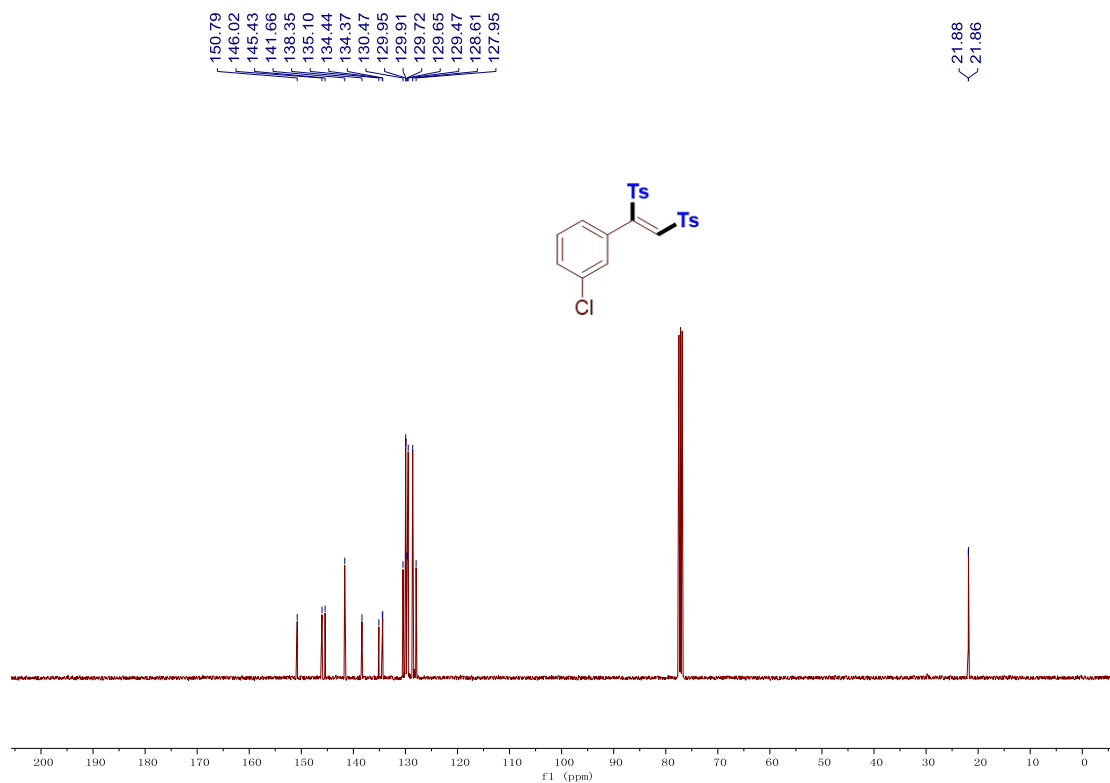


$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ ) spectra of **30a**

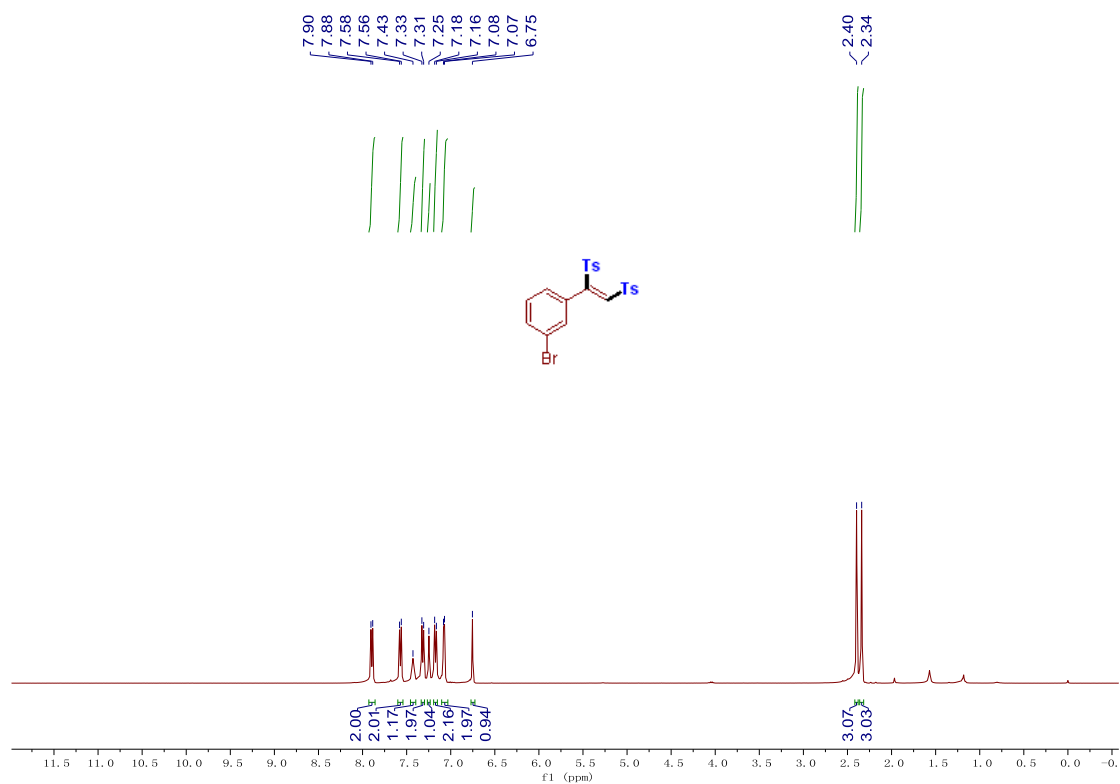




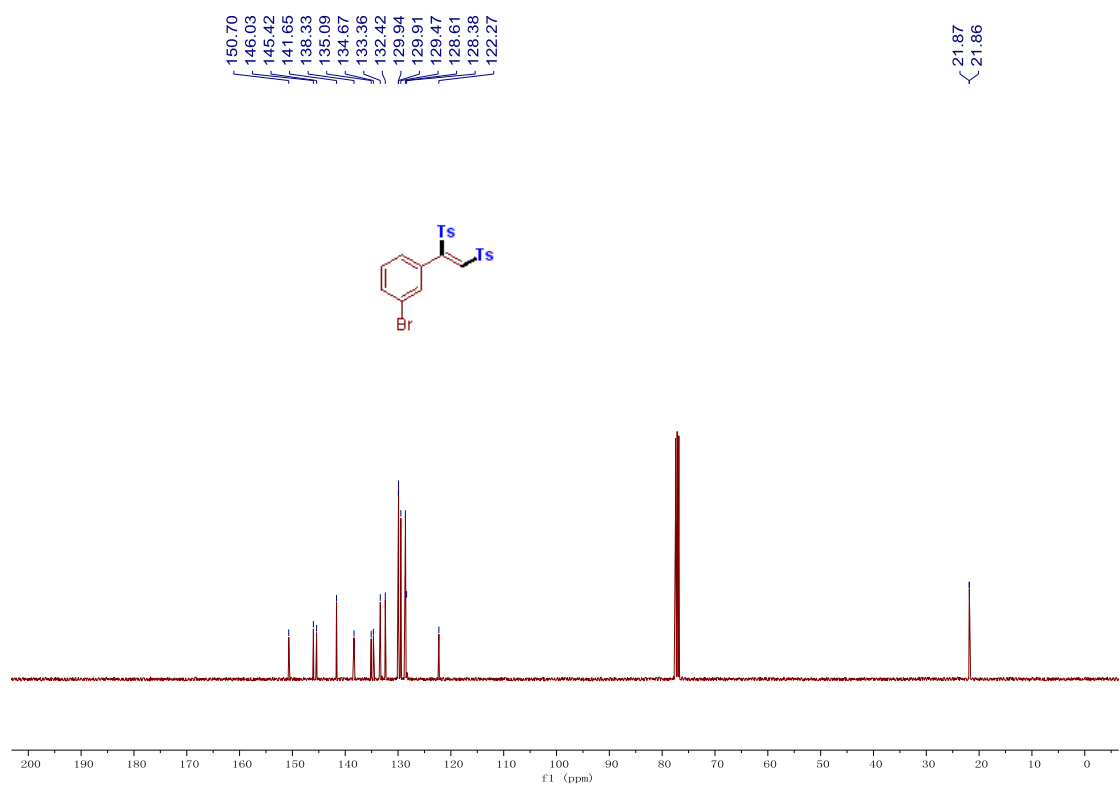
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectra of 3pa**



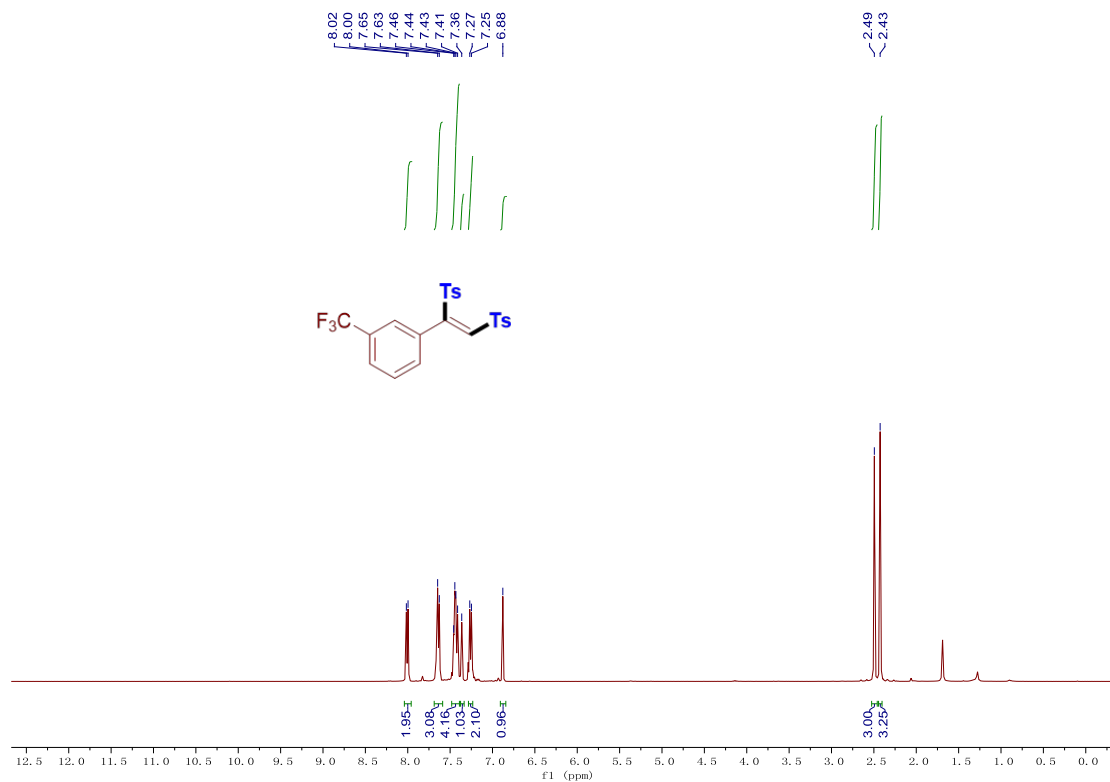
**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of 3pa**



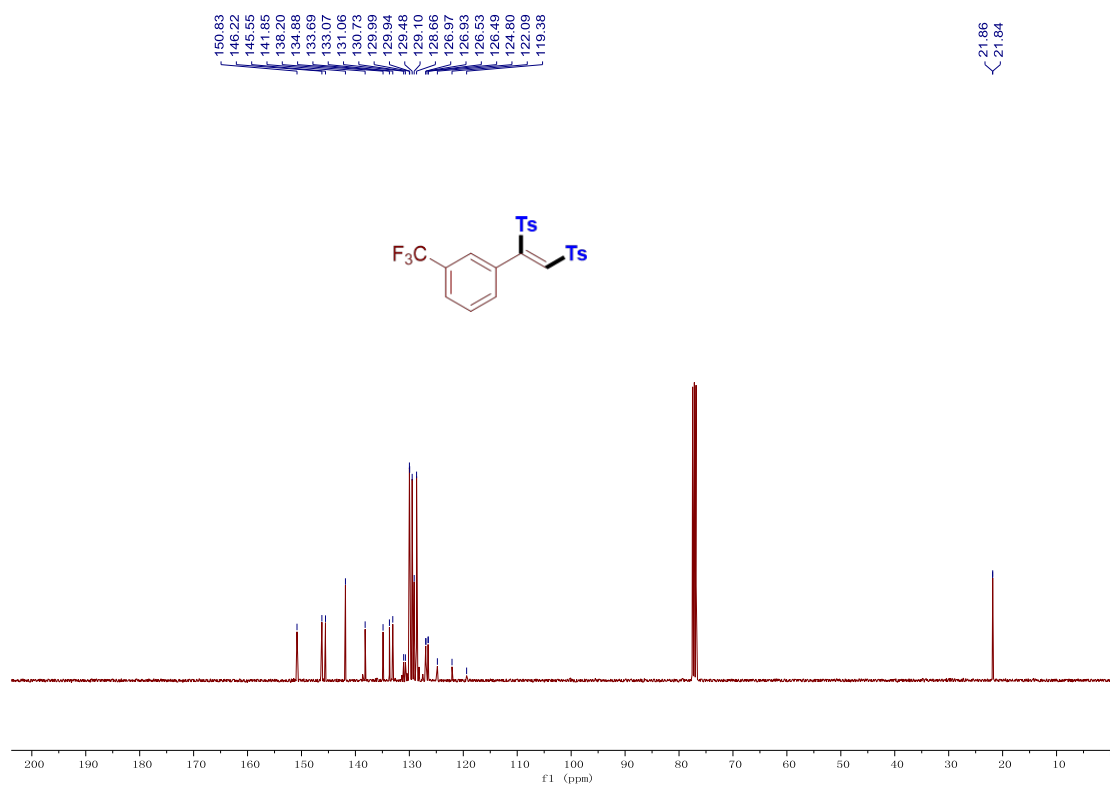
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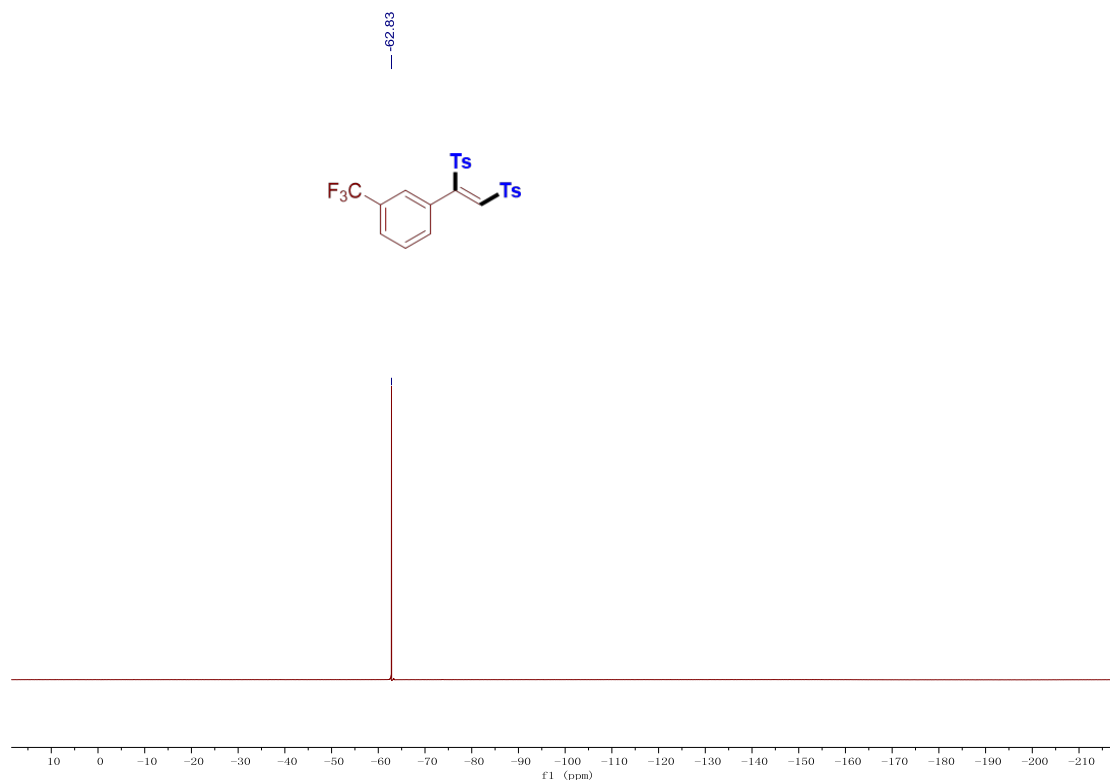
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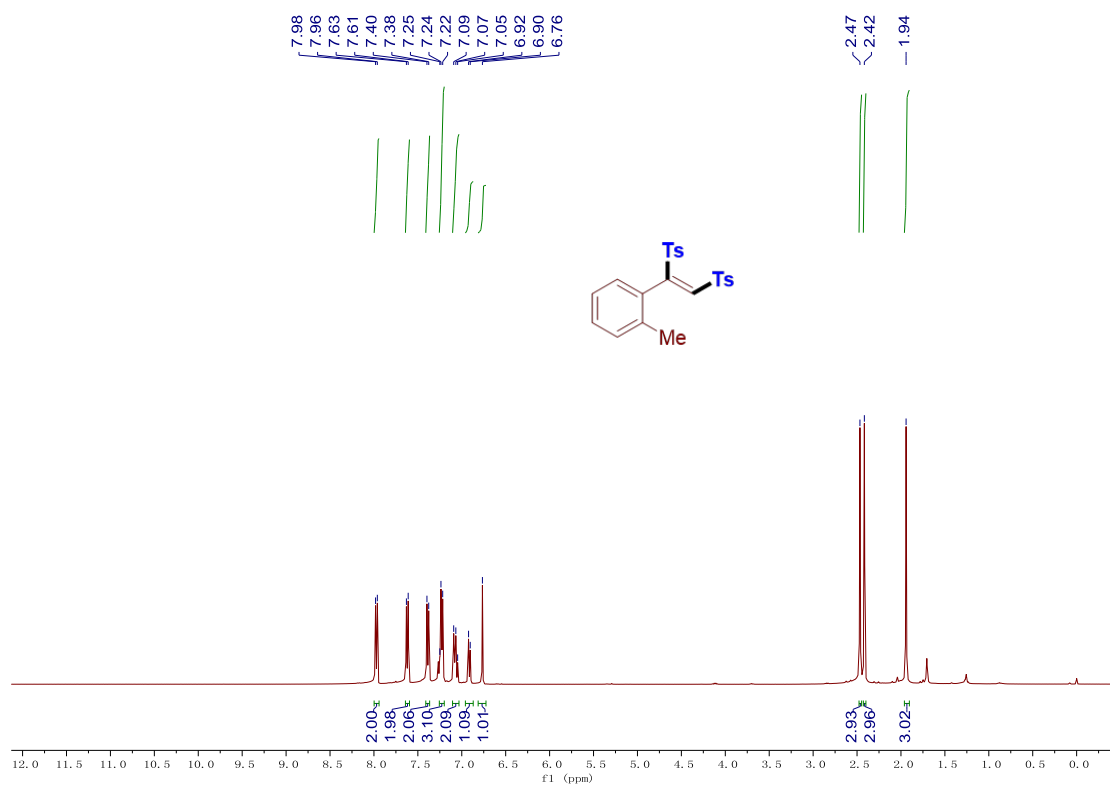
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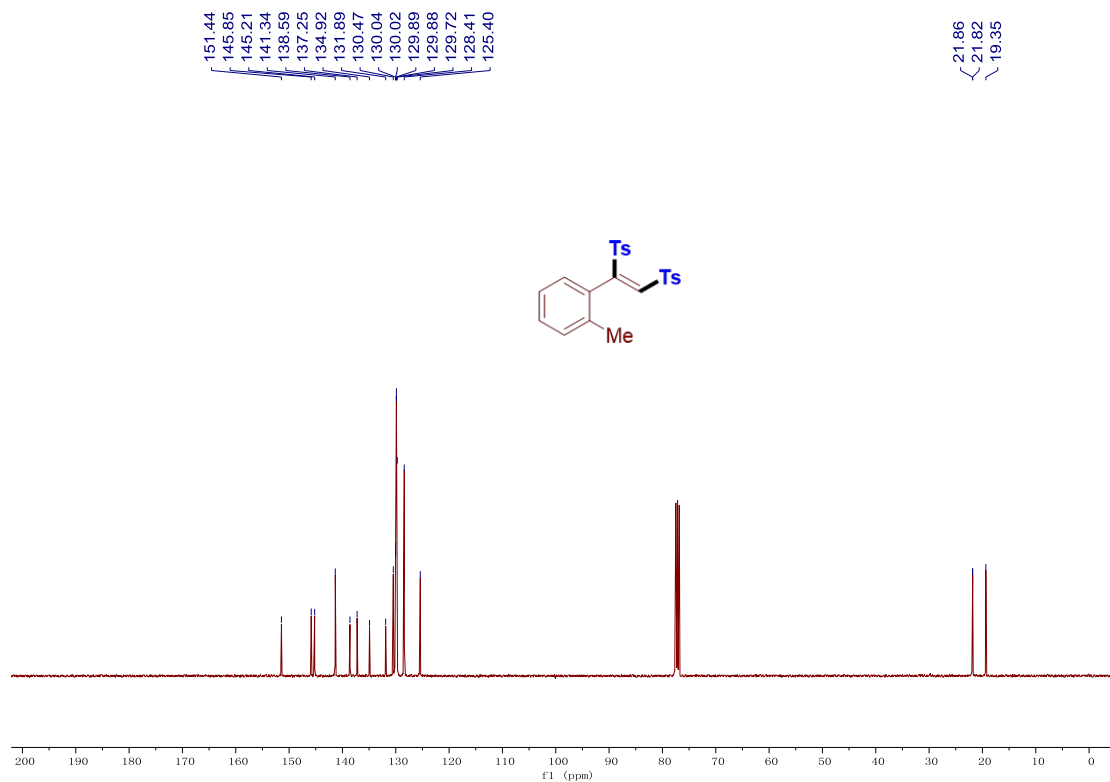
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of **3ra**



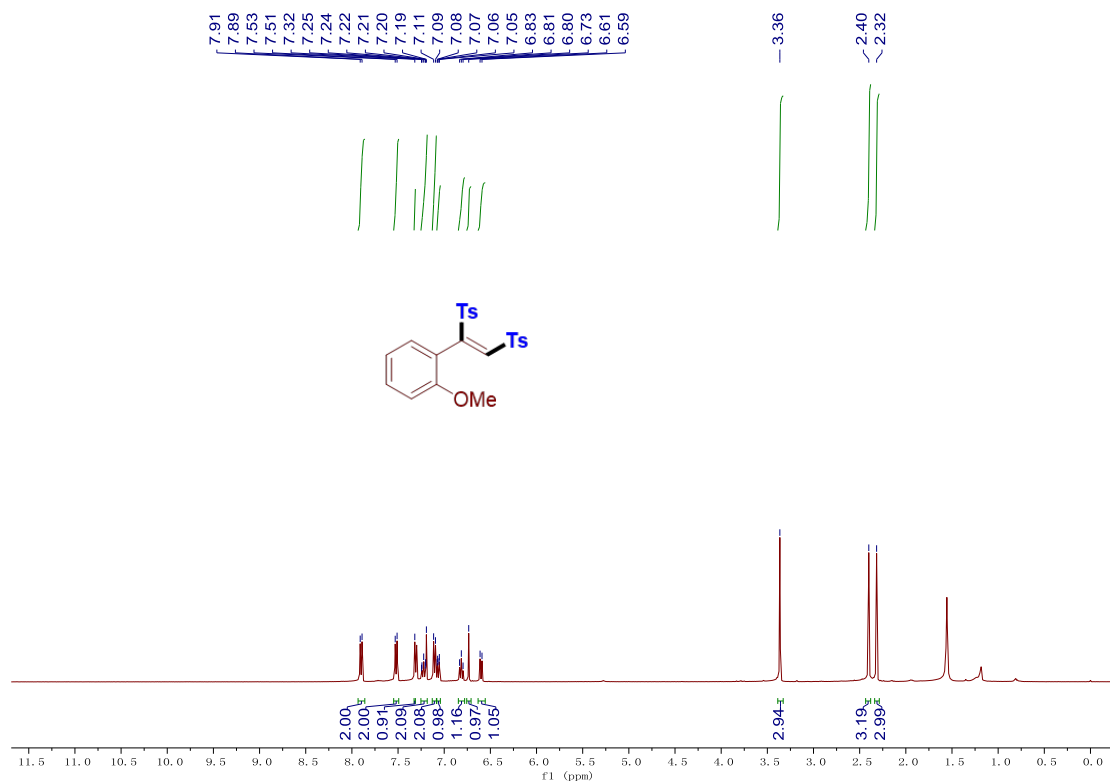
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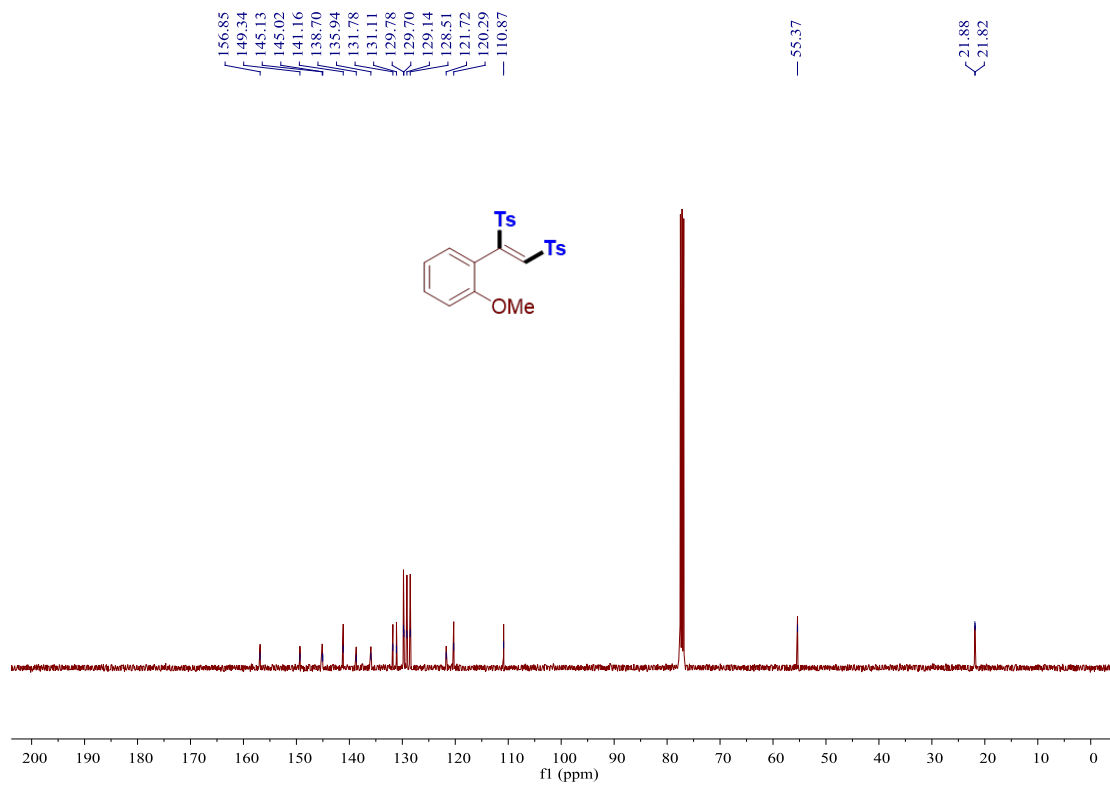
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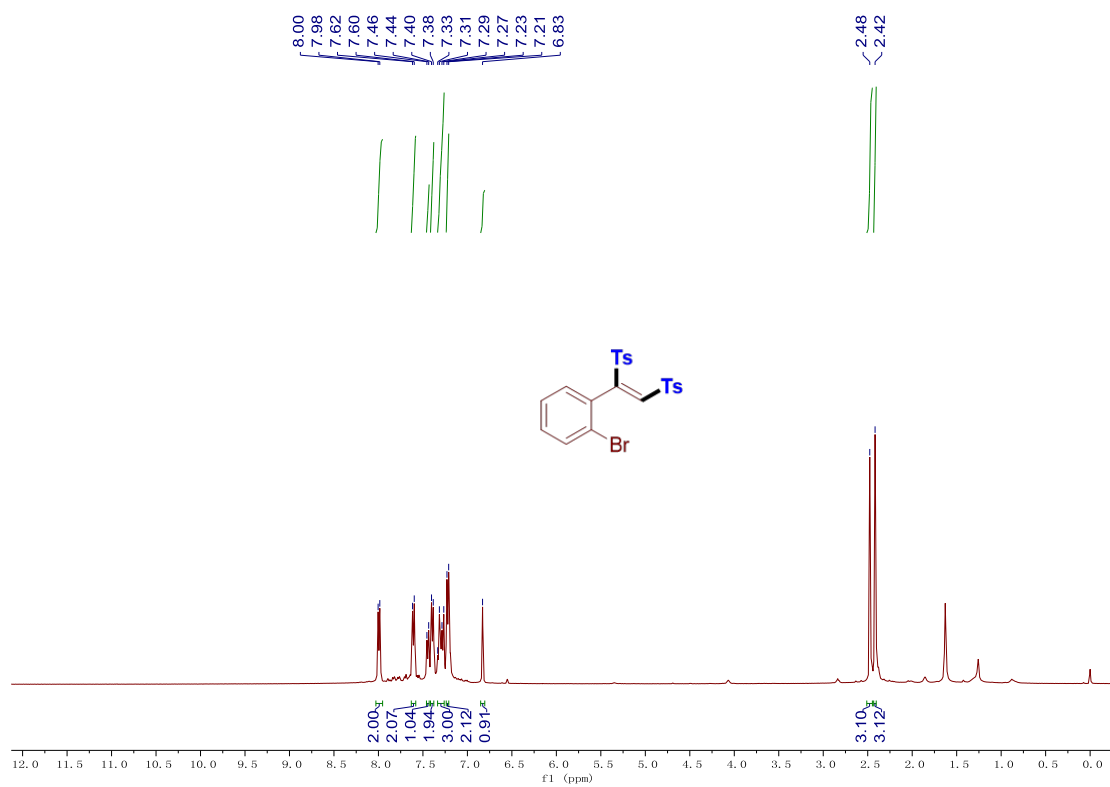
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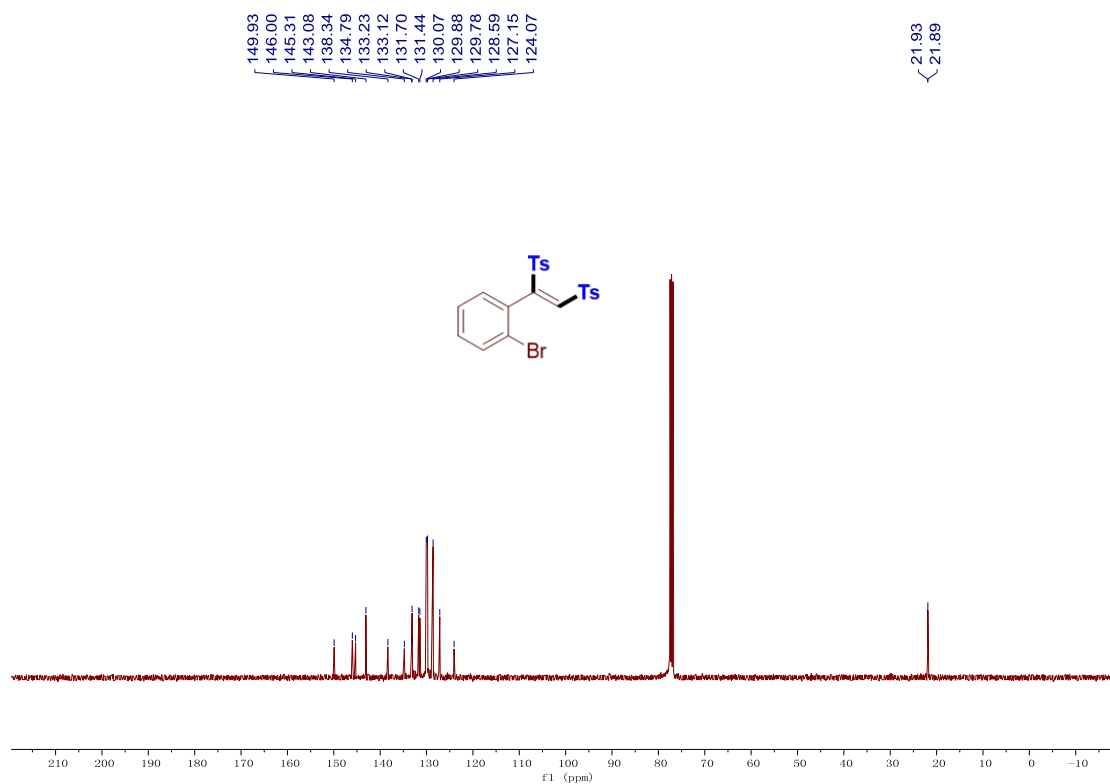
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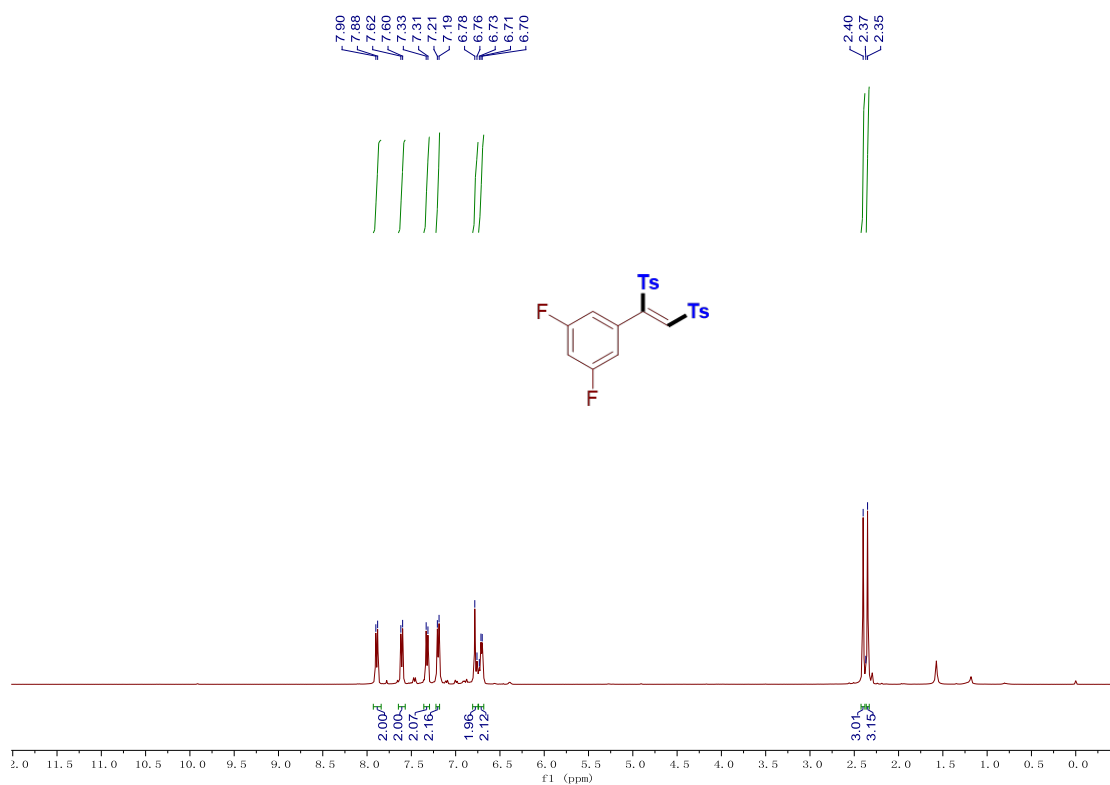
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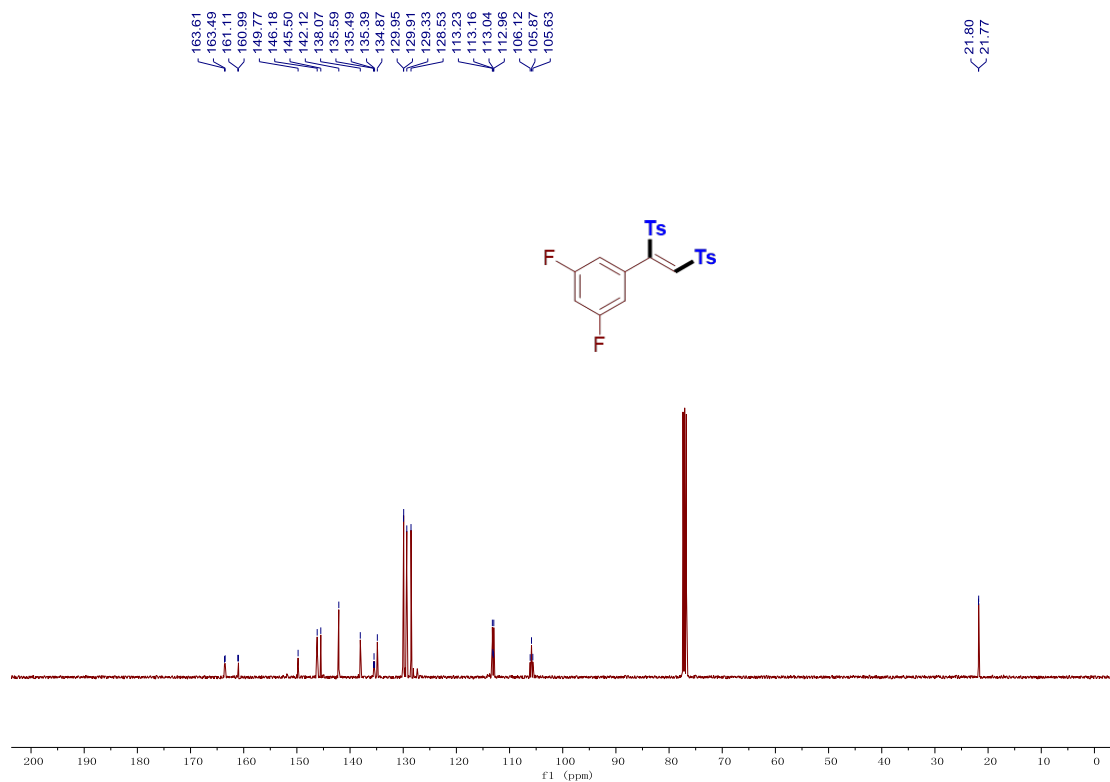
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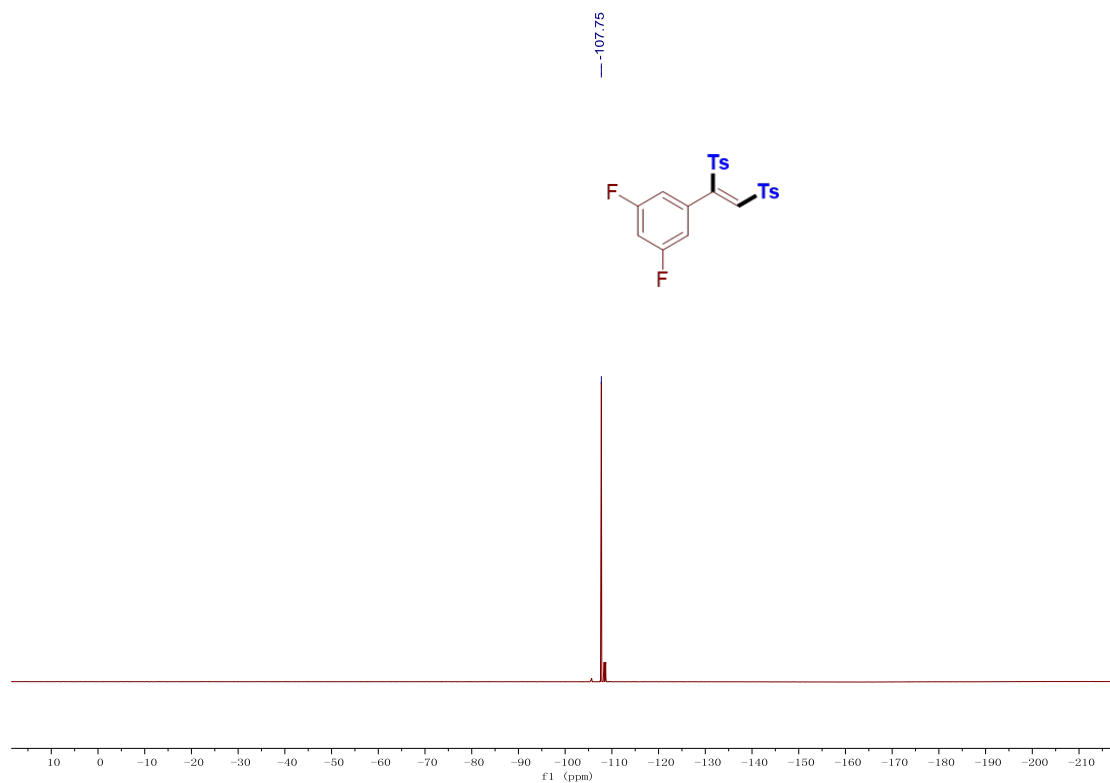
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$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) spectra of **3va**

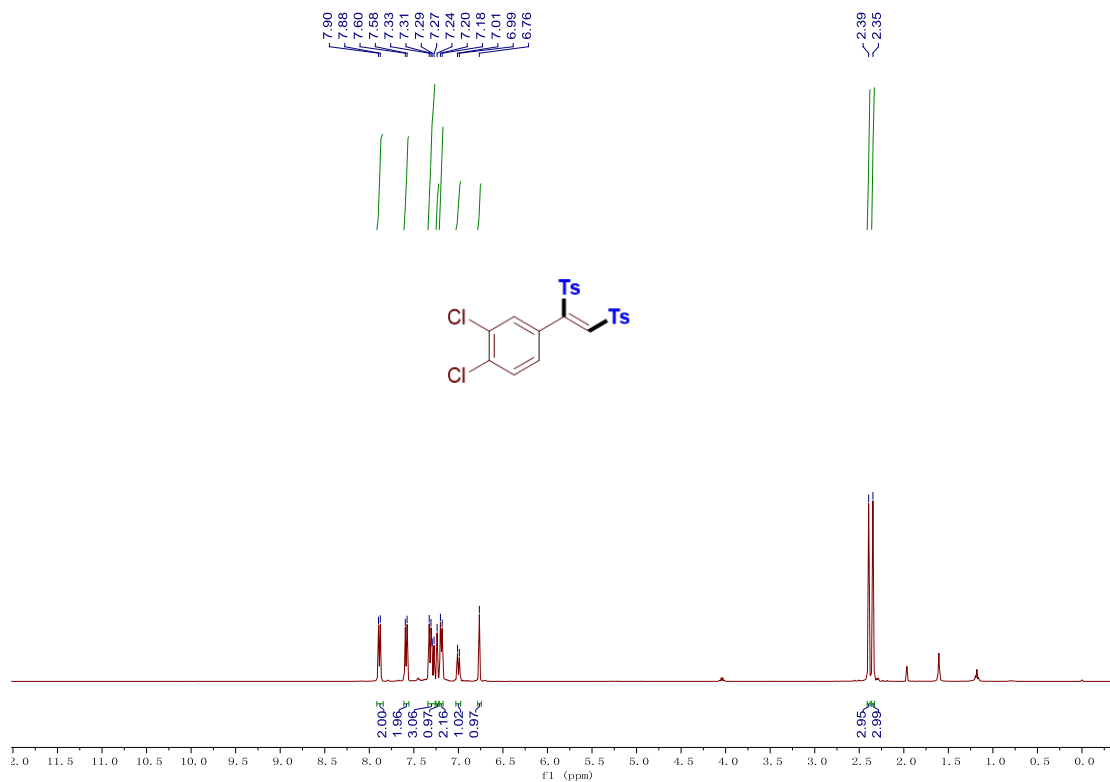


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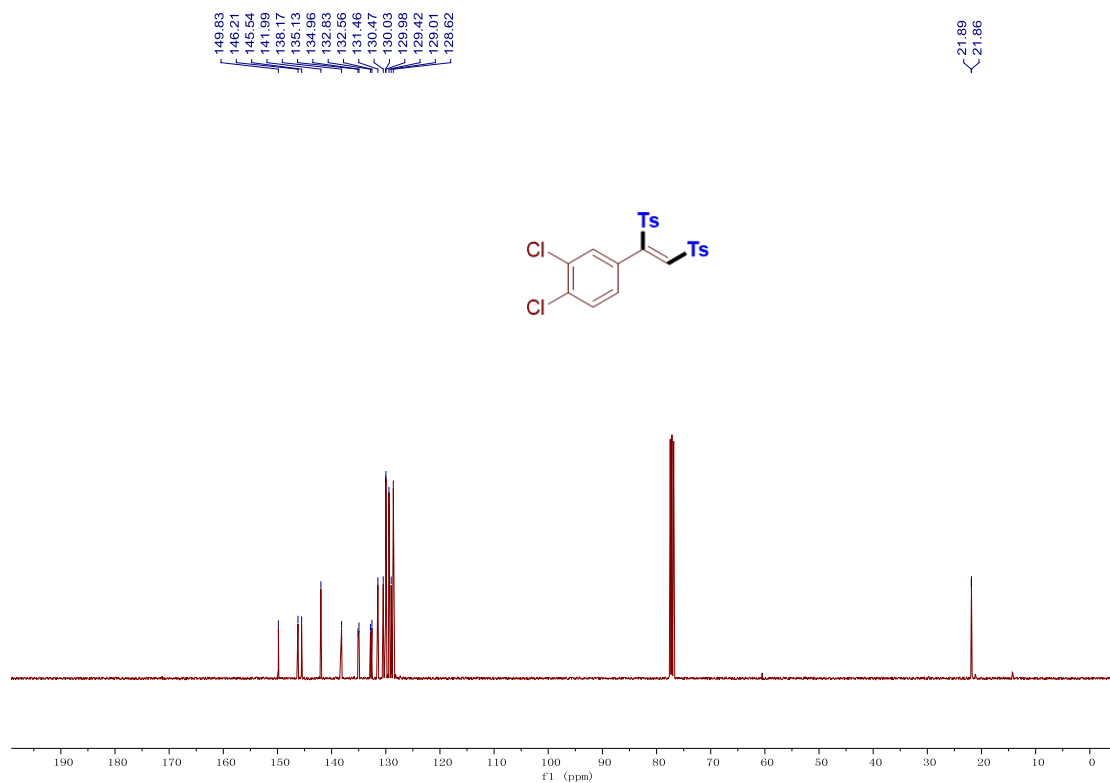


$^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ ) spectra of **3va**

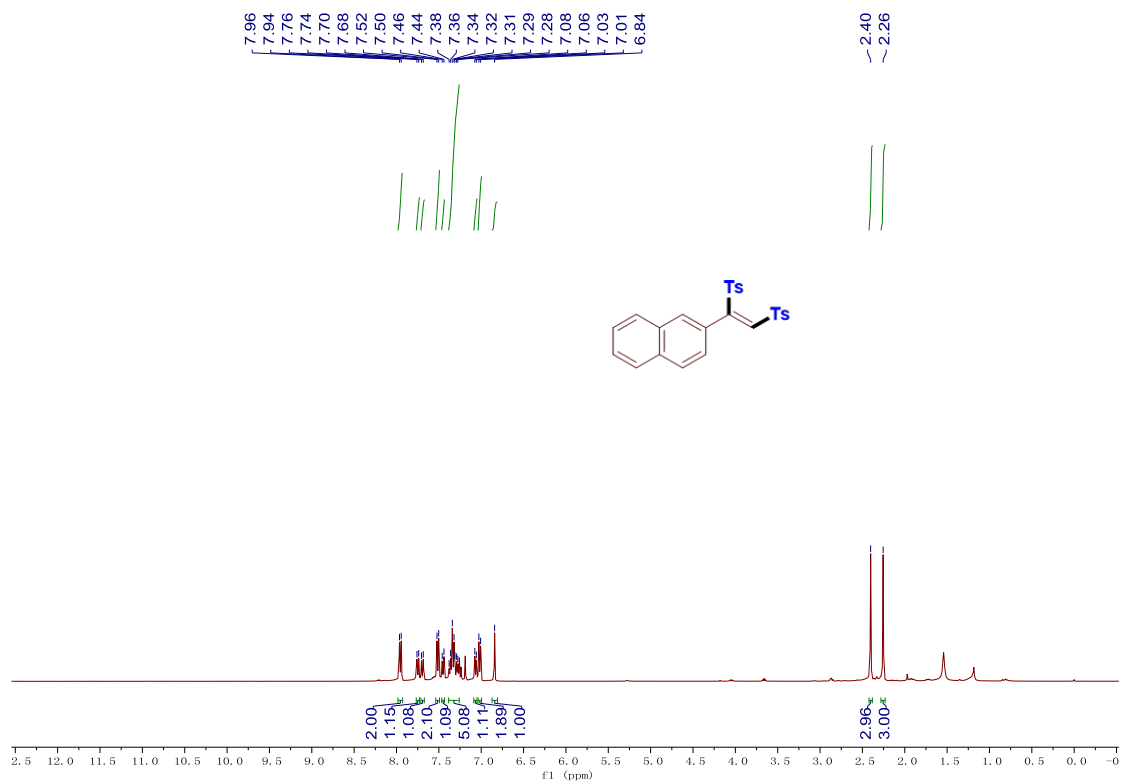




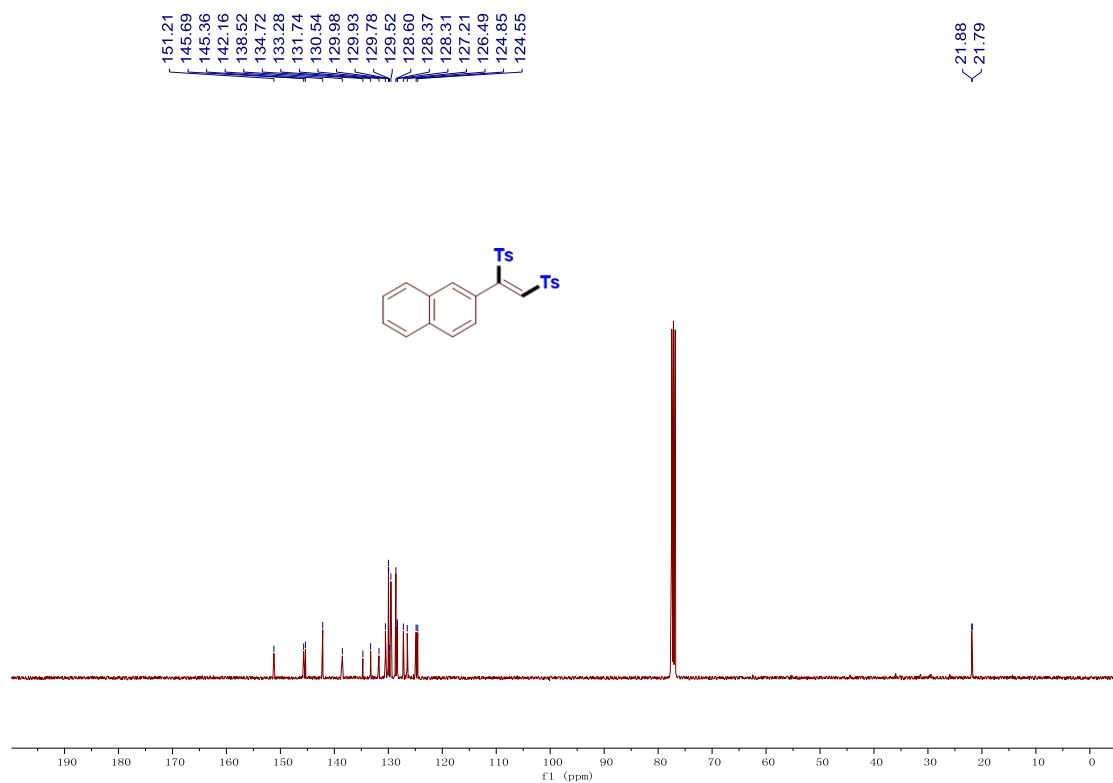
**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectra of **3wa****



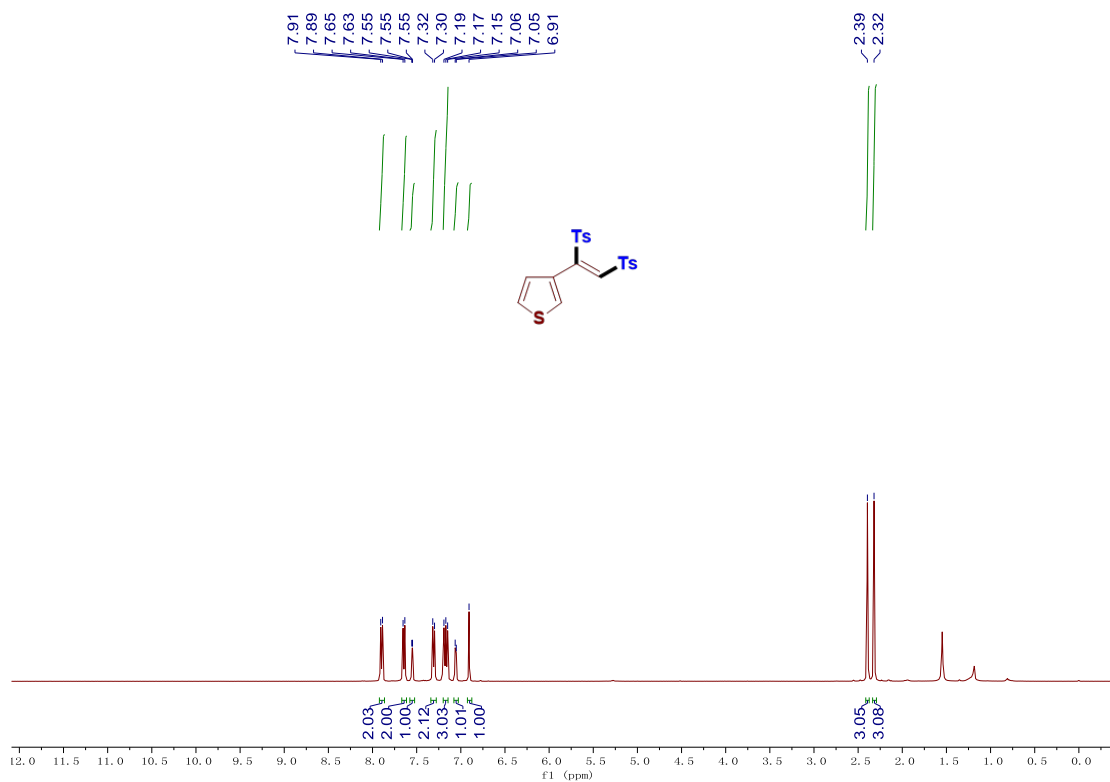
**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of **3wa****



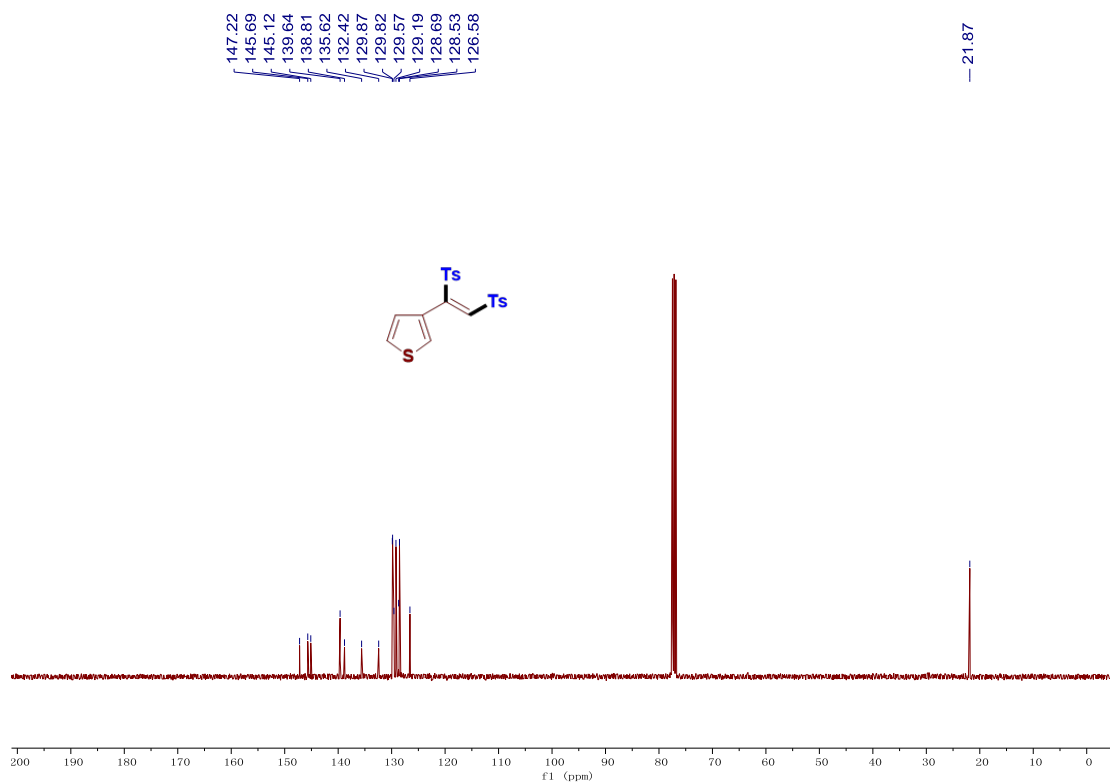
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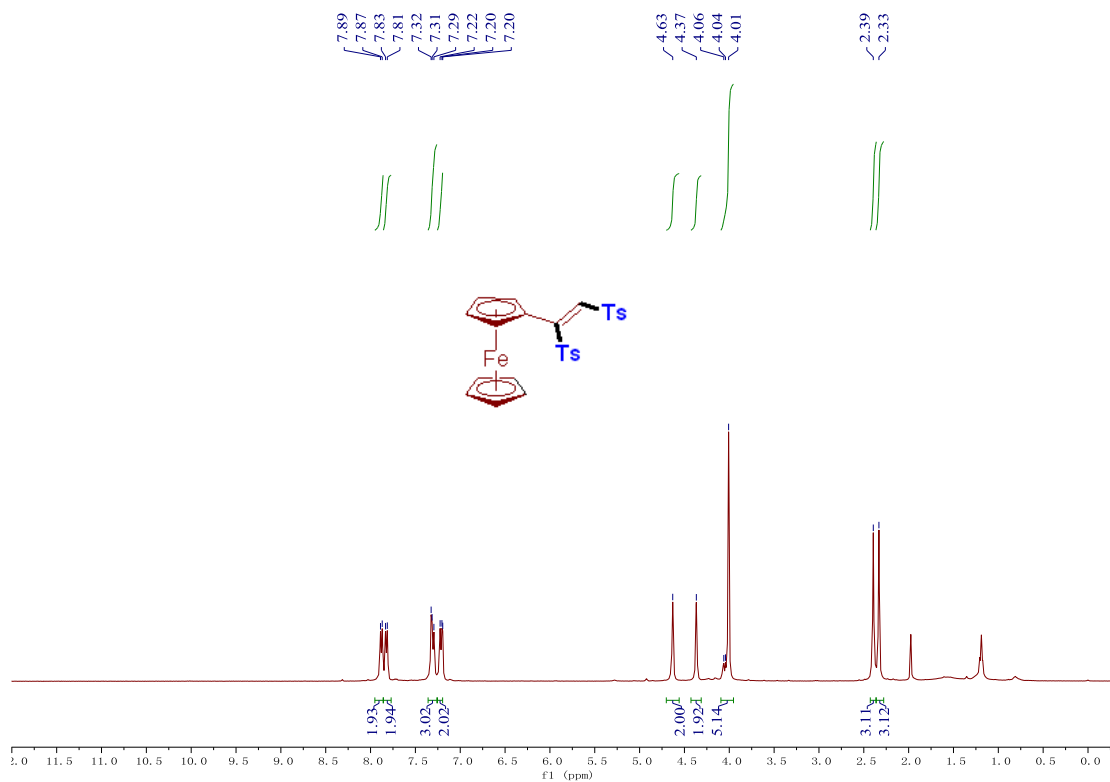
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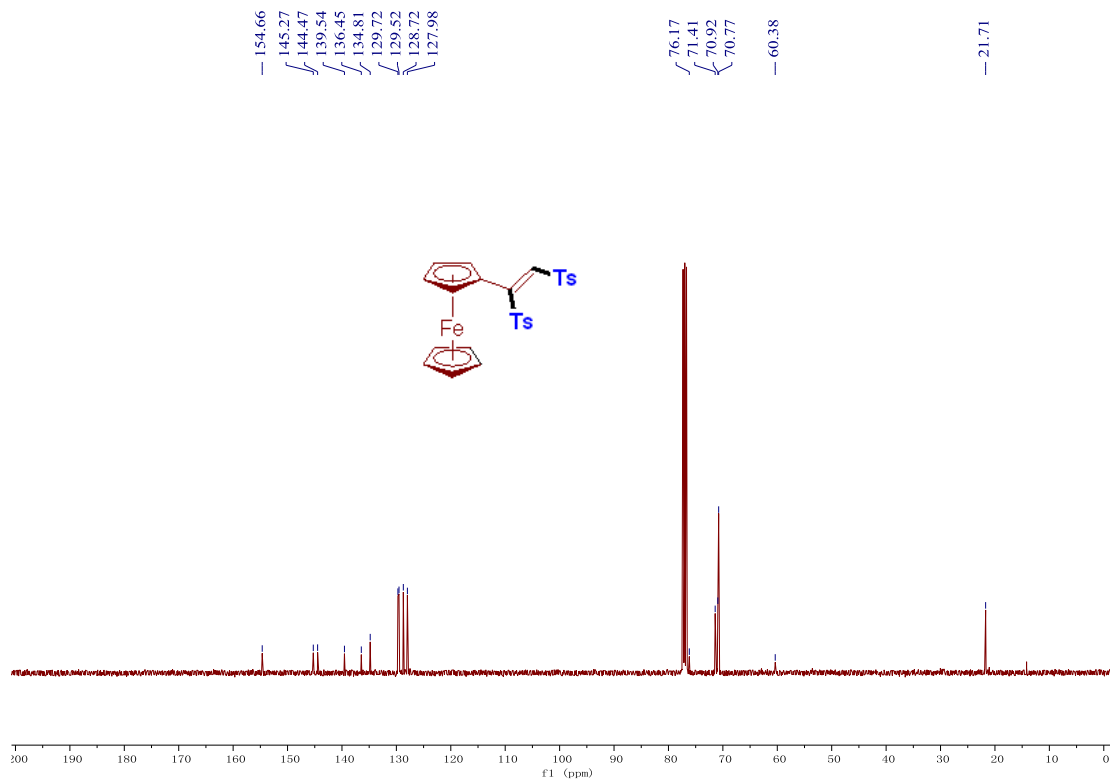
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectra of **3ya**



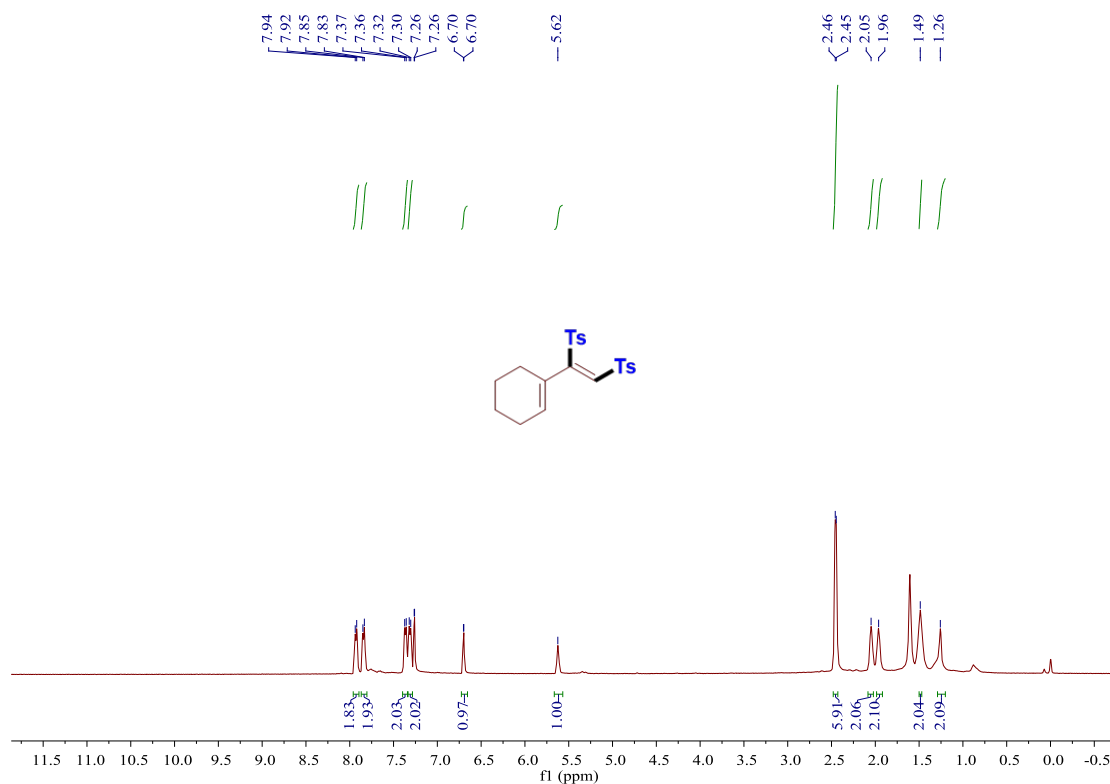
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of **3ya**



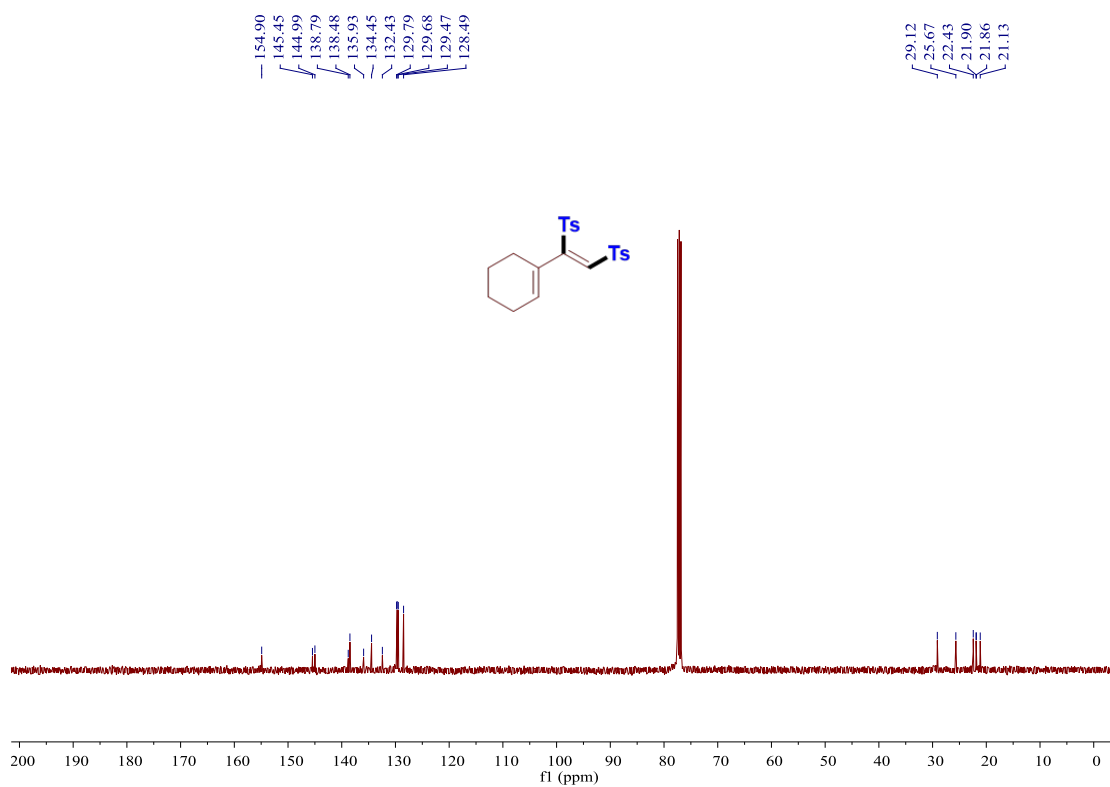
$^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ) spectra of **3za**



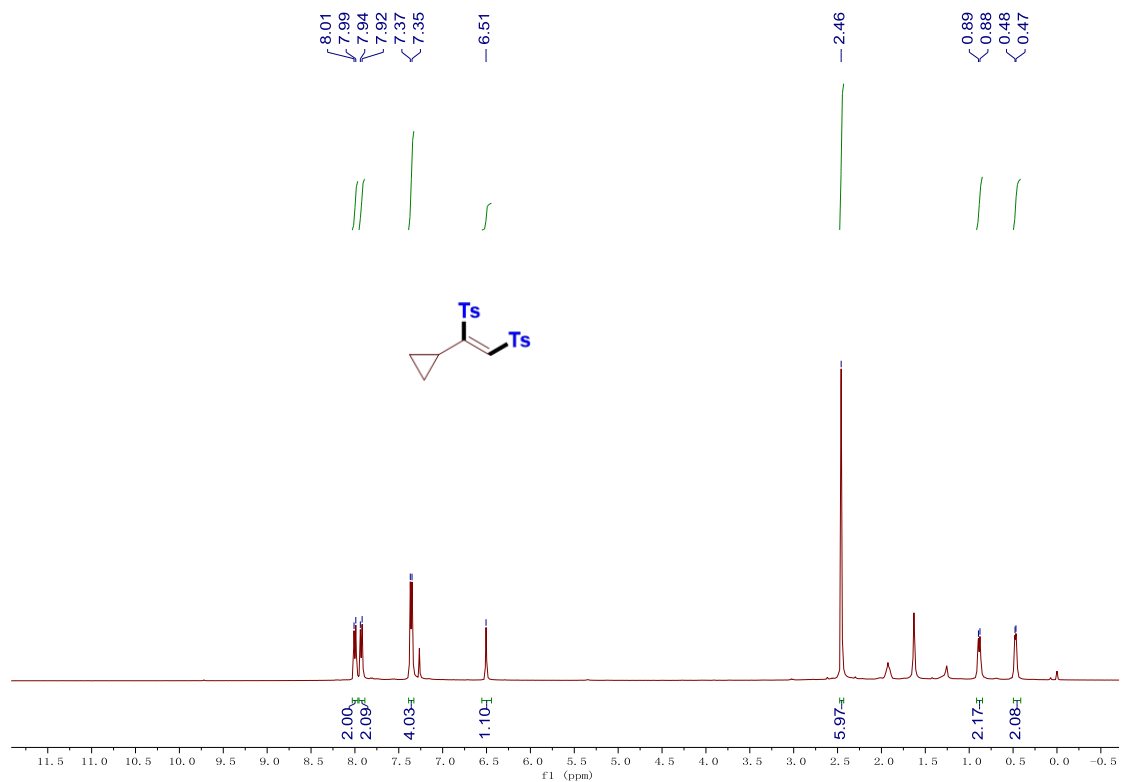
$^{13}\text{C NMR}$  (101 MHz,  $\text{CDCl}_3$ ) spectra of **3za**



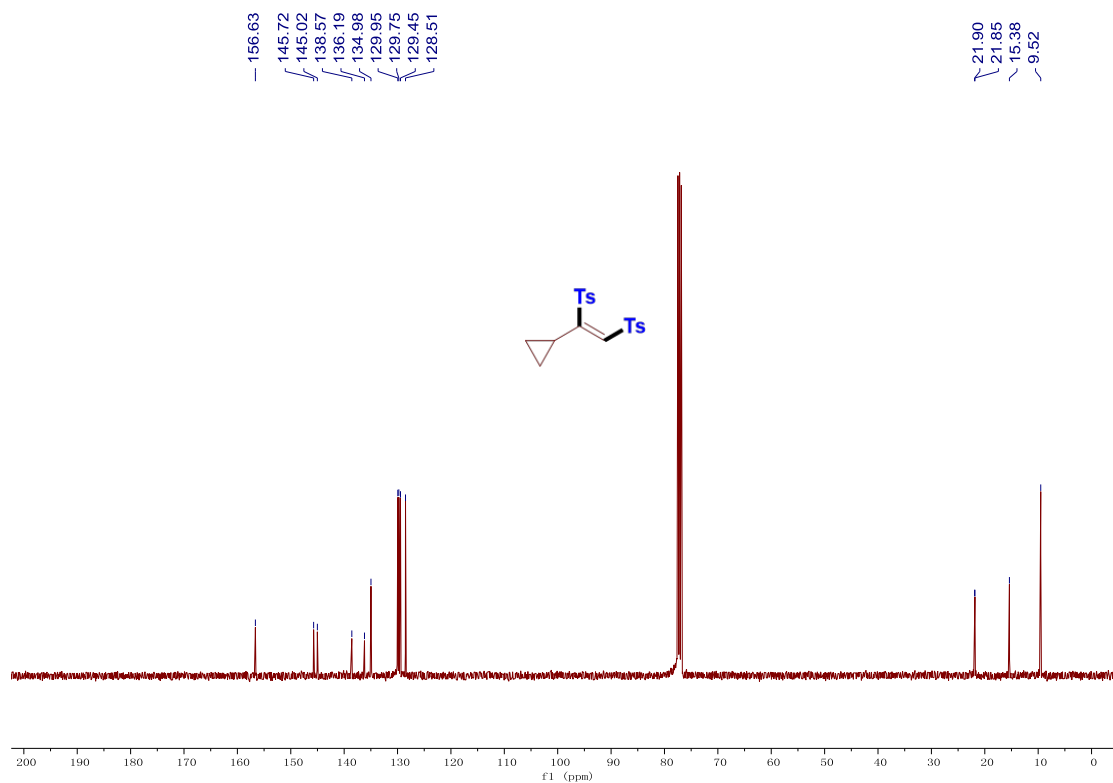
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectra of **4aa**



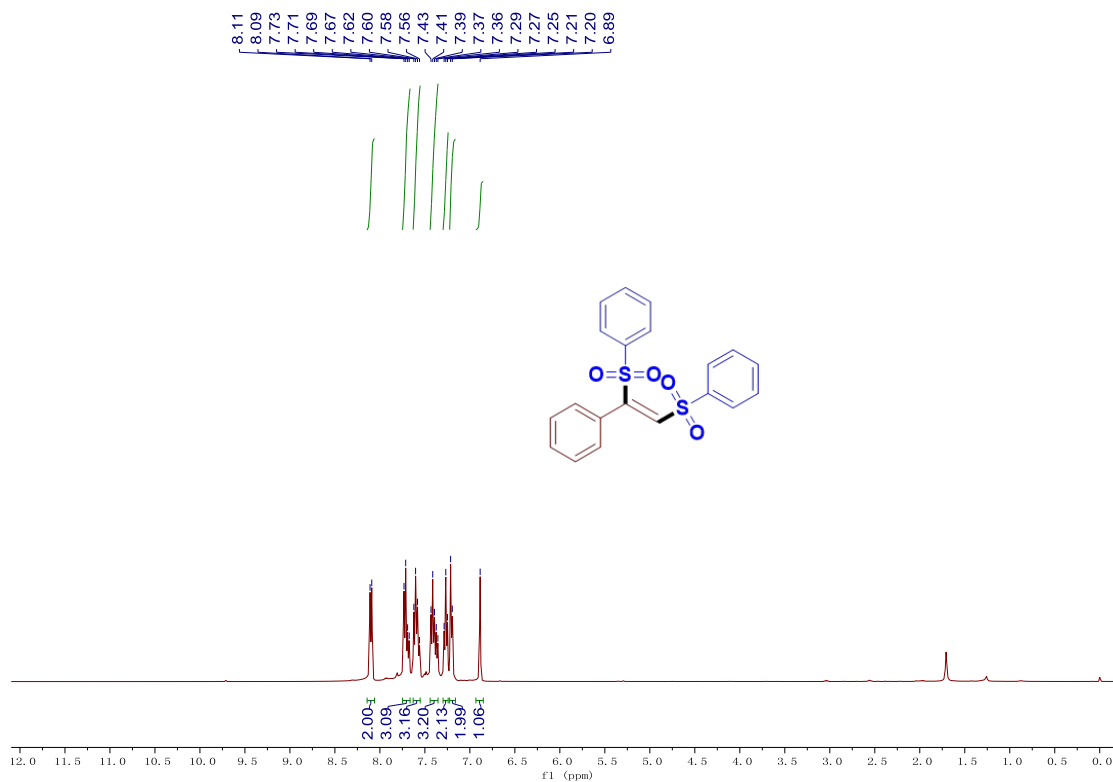
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of **4aa**



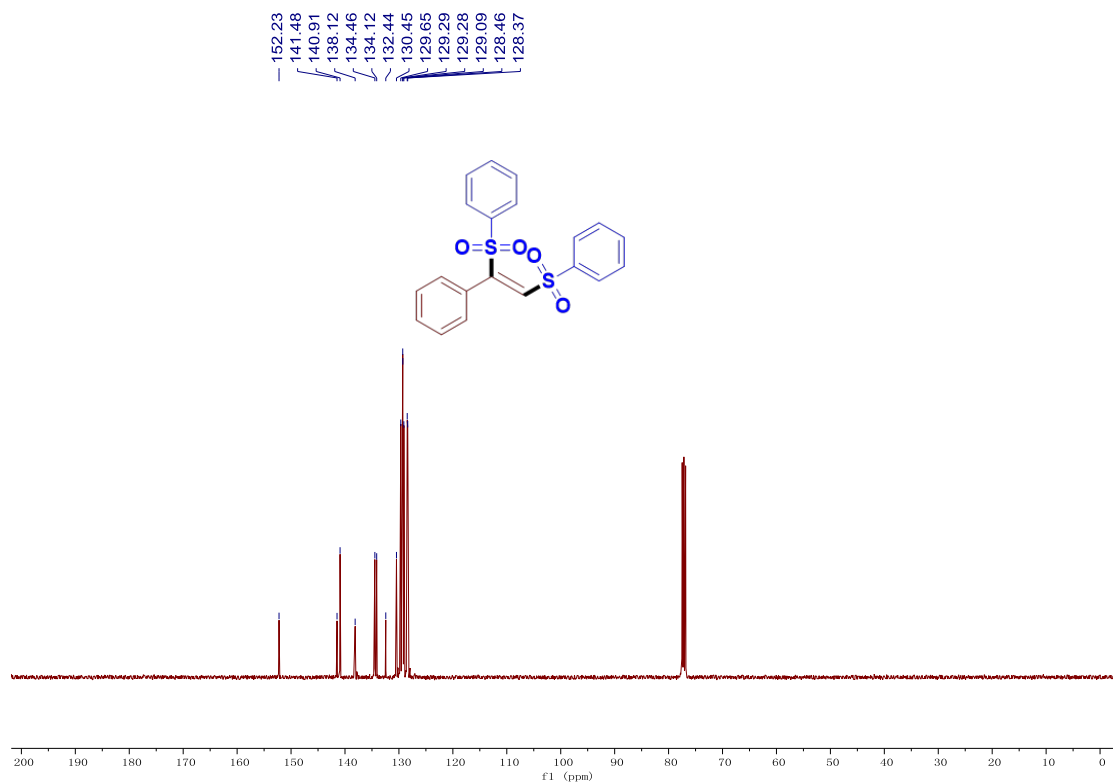
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) spectra of **4ba**



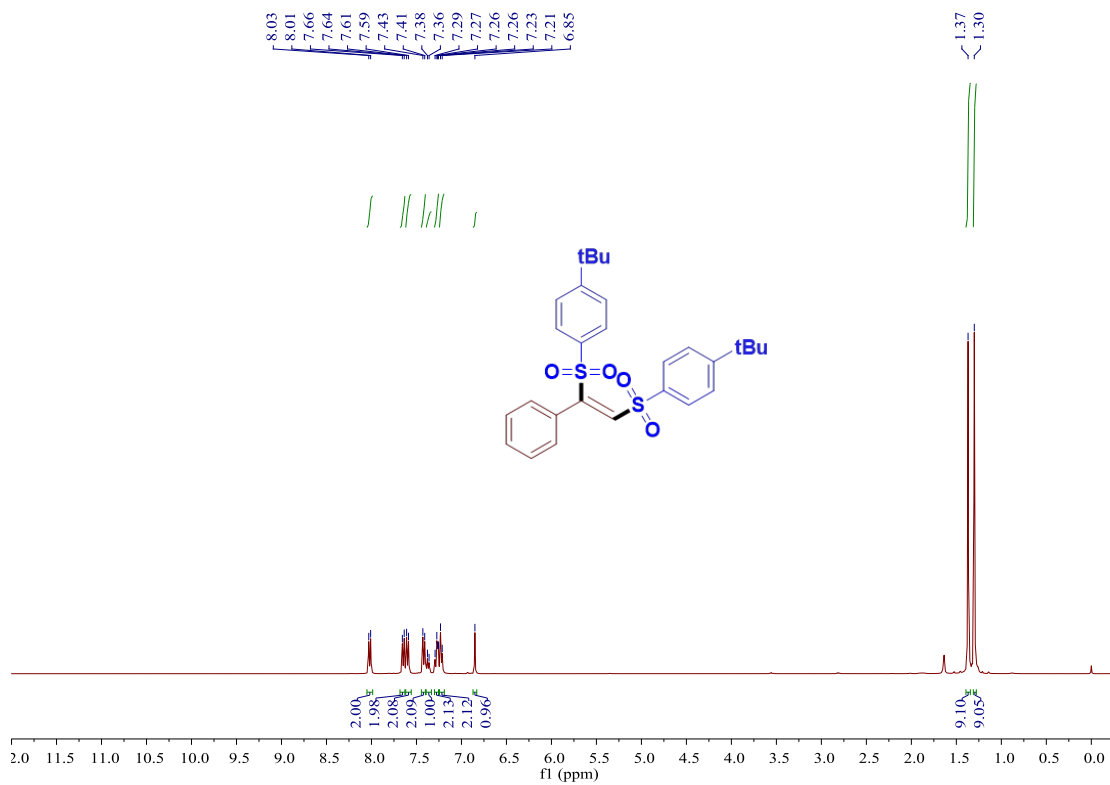
$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectra of **4ba**



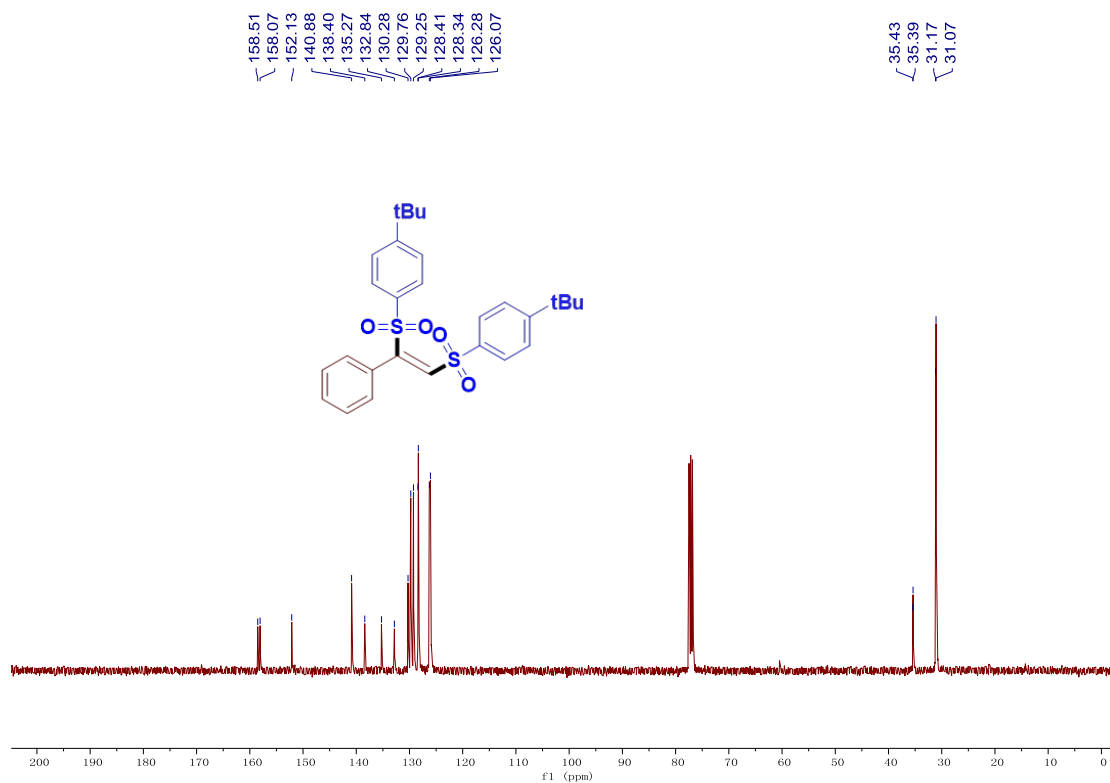
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectra of **3ab**



<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of **3ab**

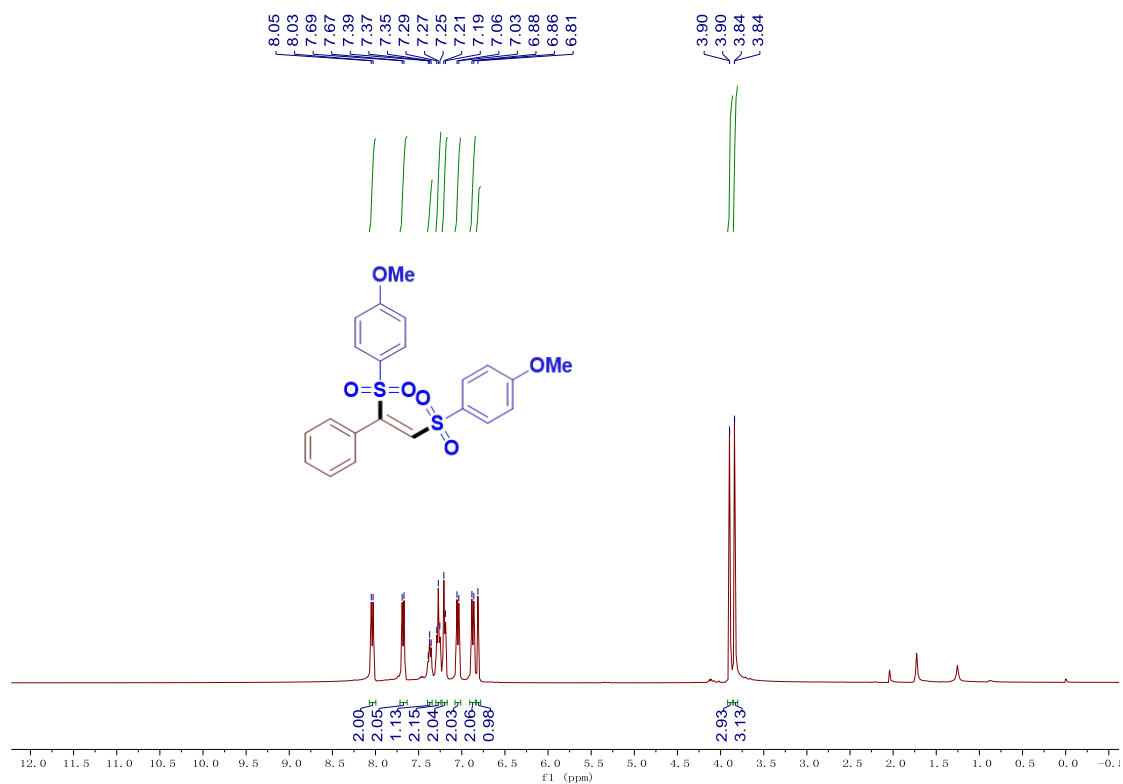


<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectra of **3ac**

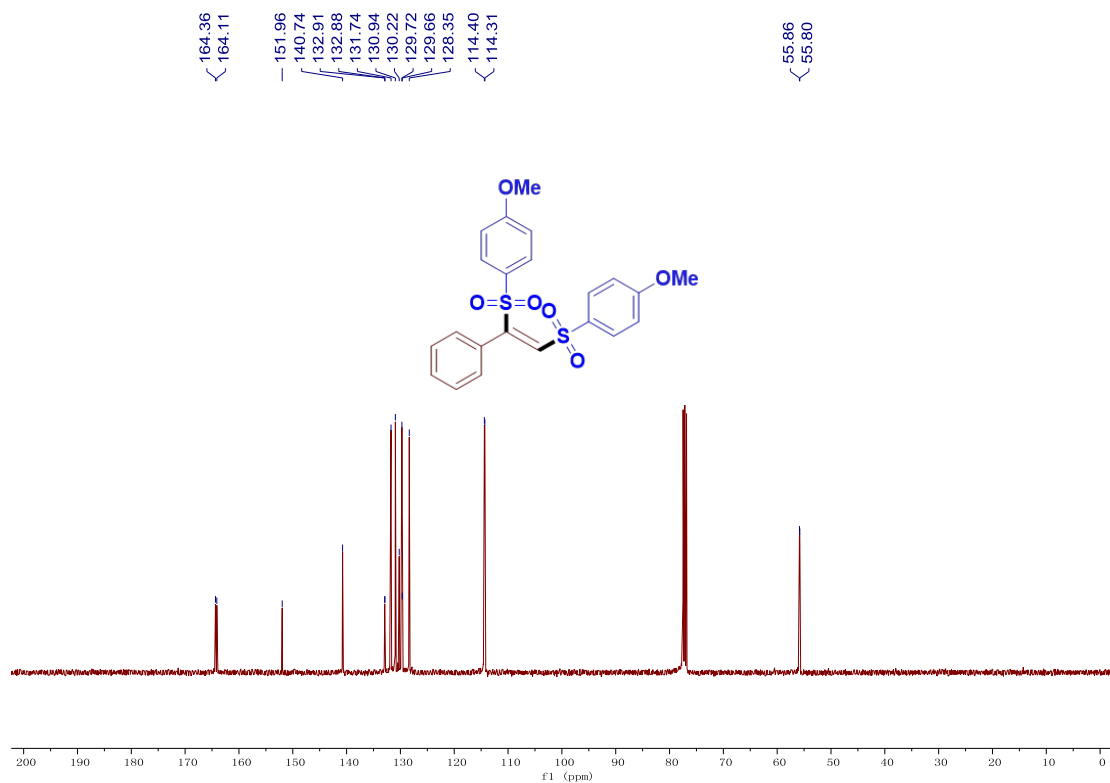


<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of **3ac**

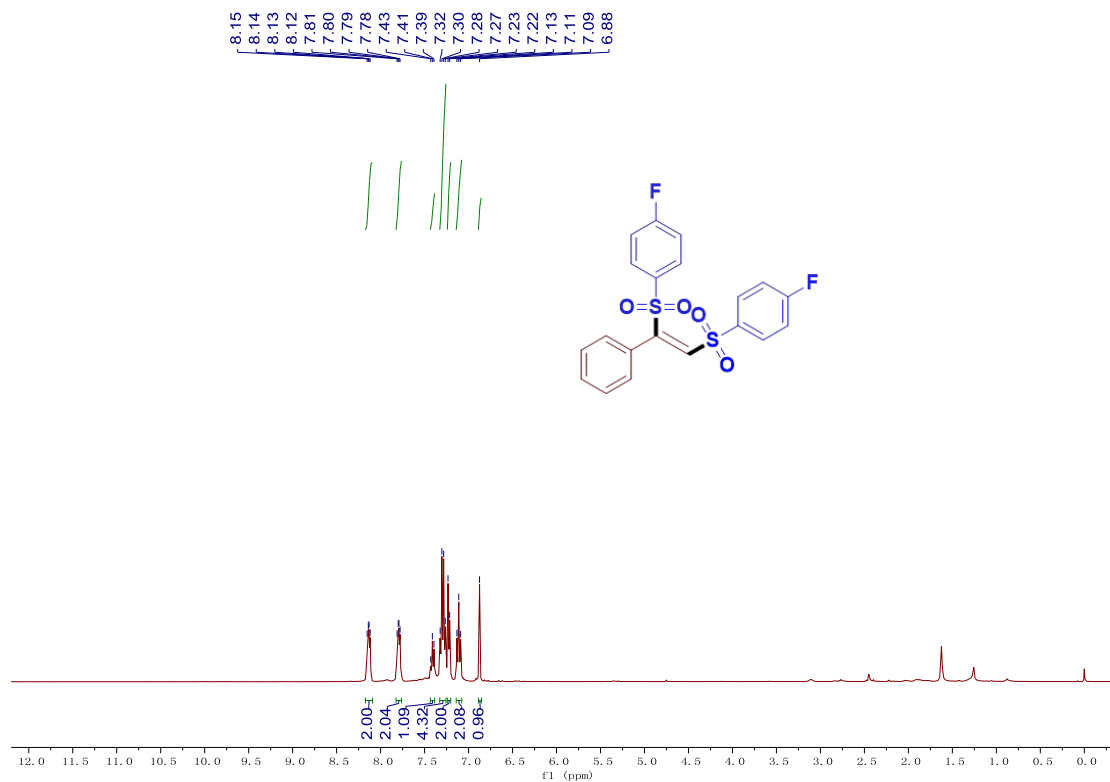




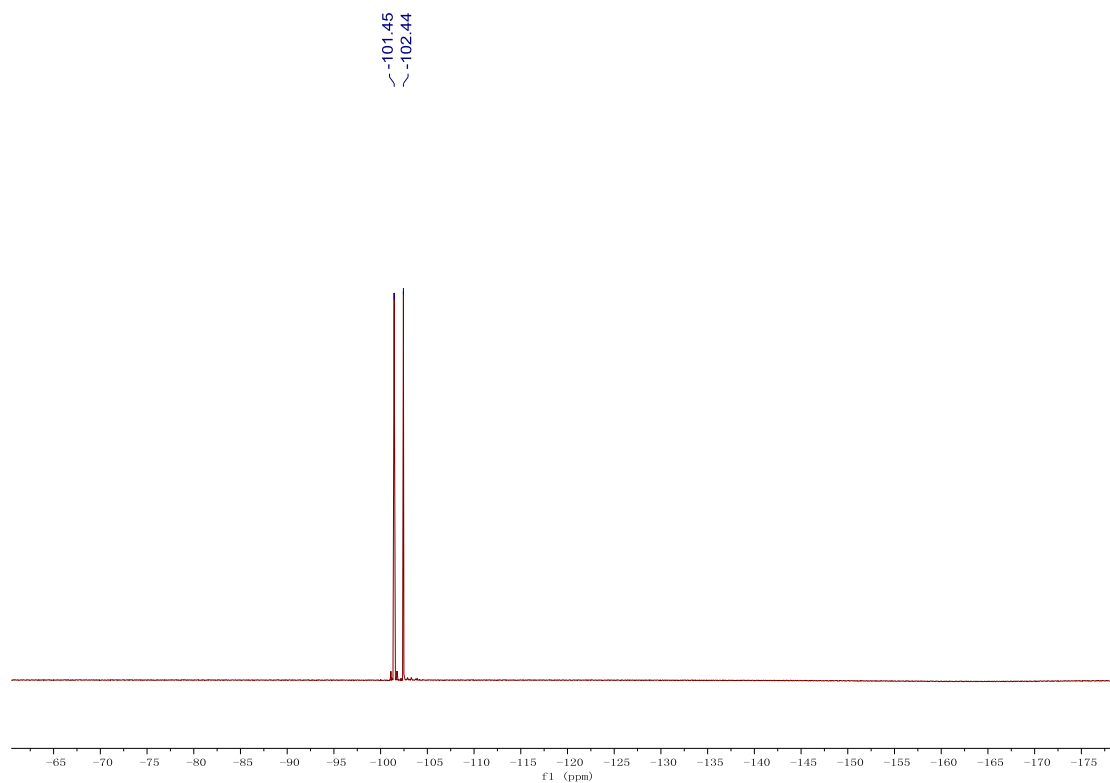
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectra of **3ad**



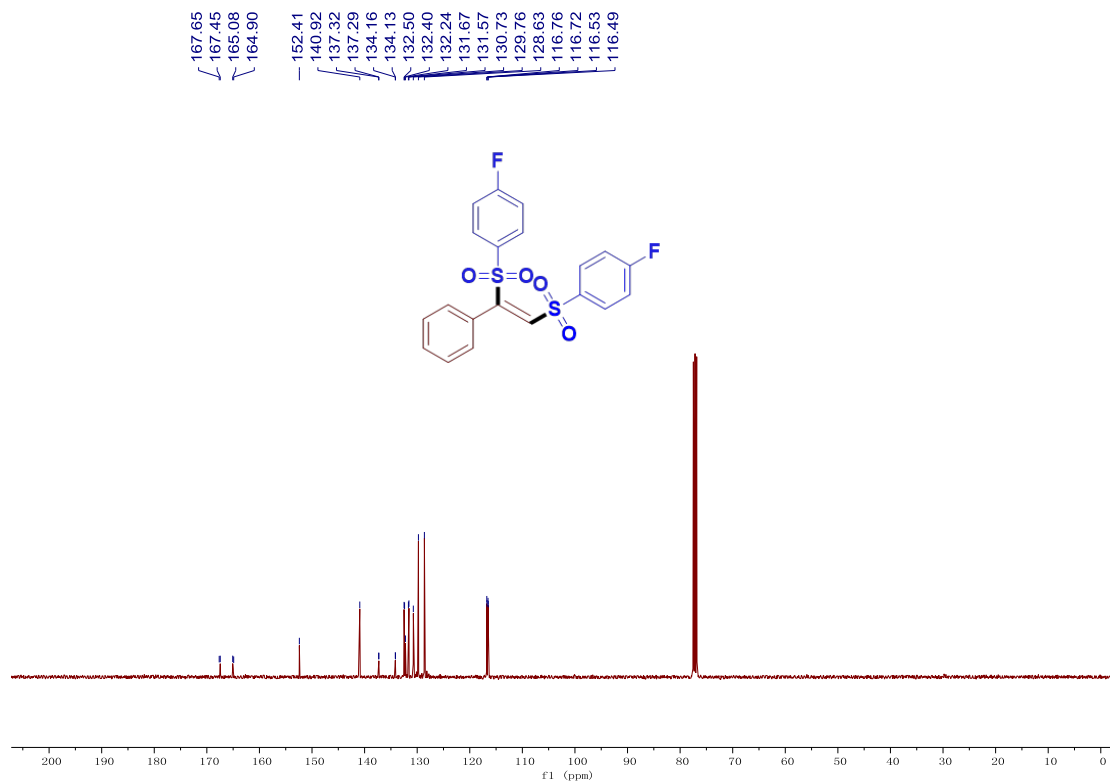
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of **3ad**



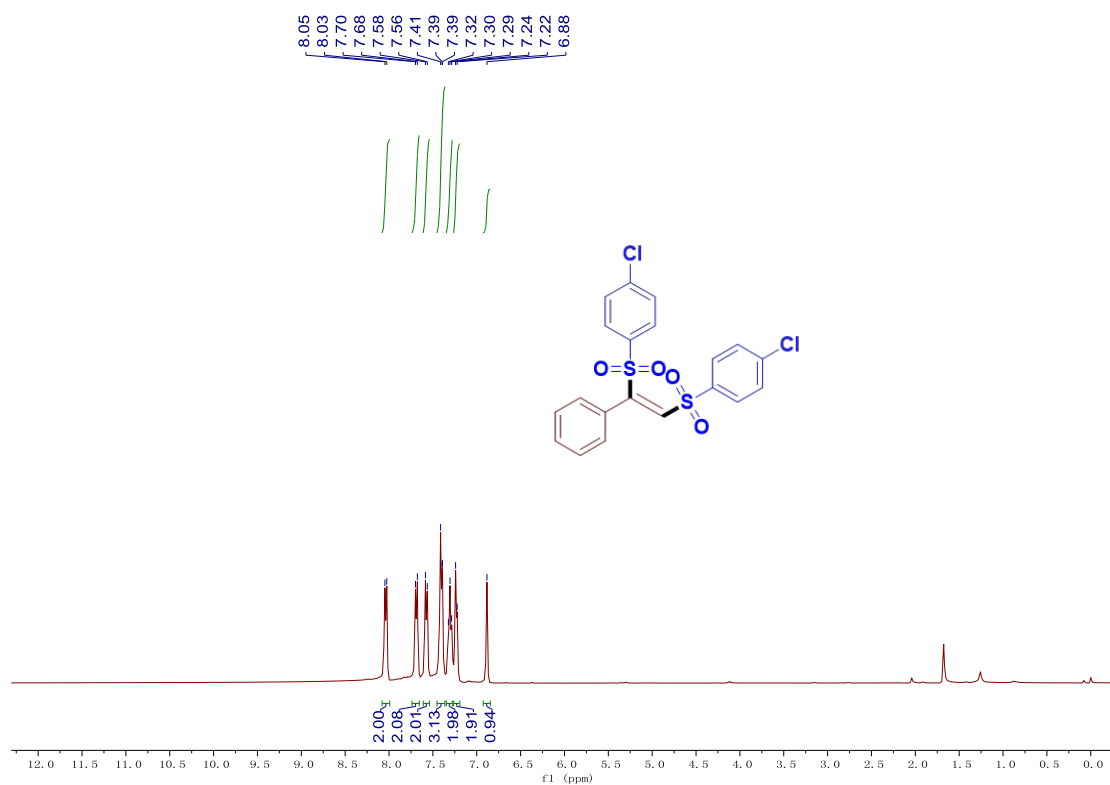
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectra of **3ae**



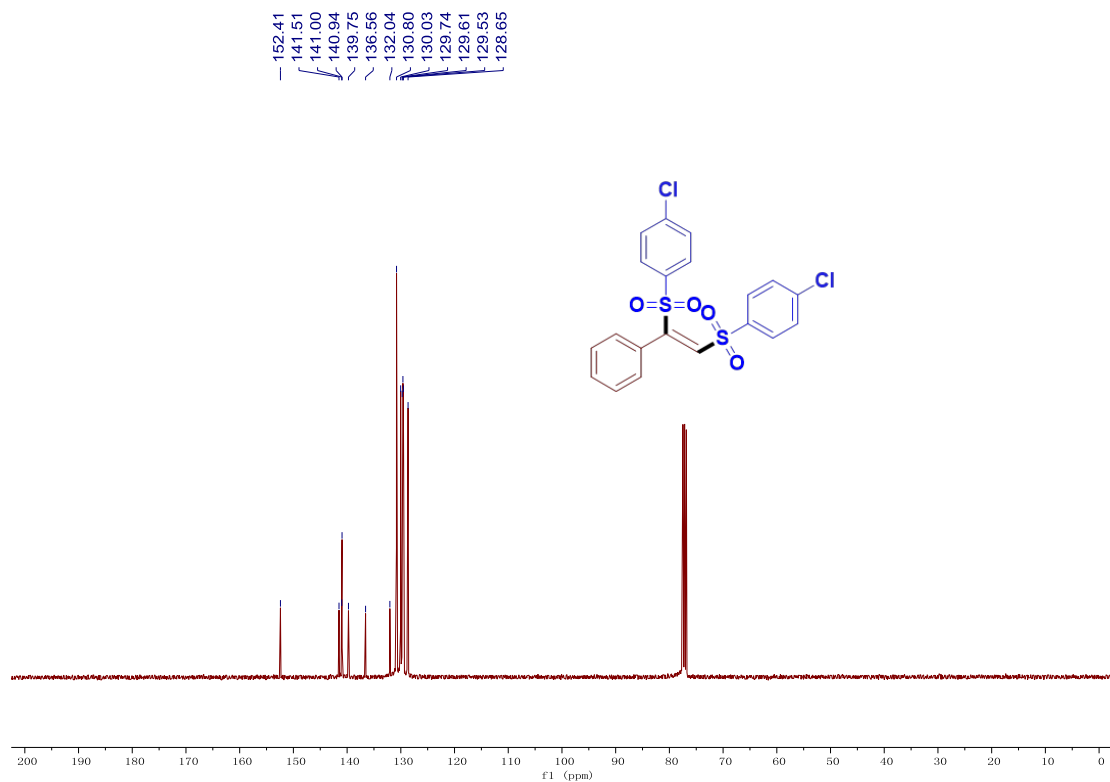
<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) spectra of **3ae**



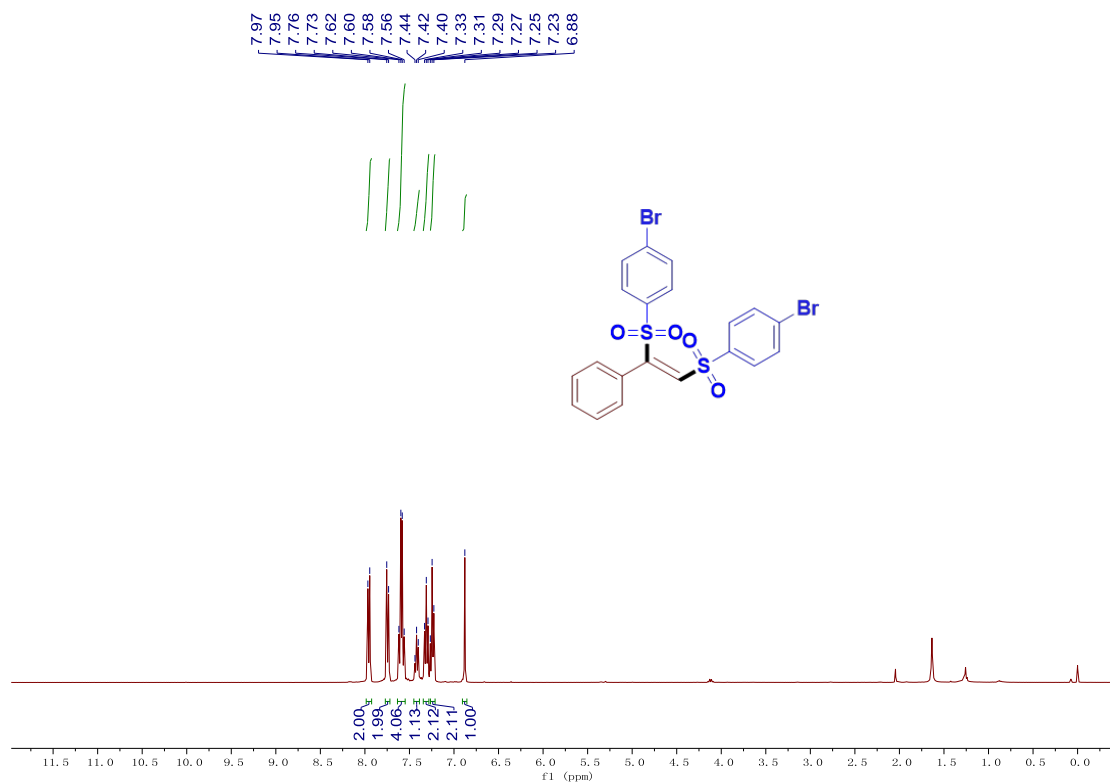
$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectra of **3ae**



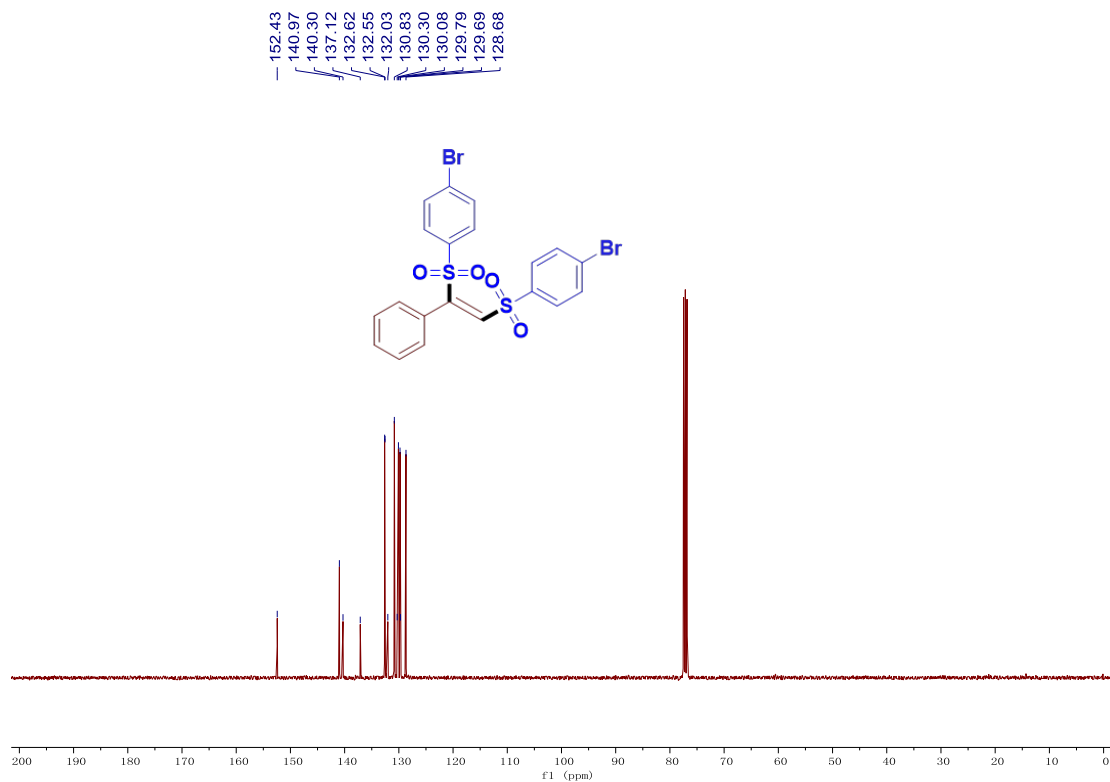
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) spectra of **3af**



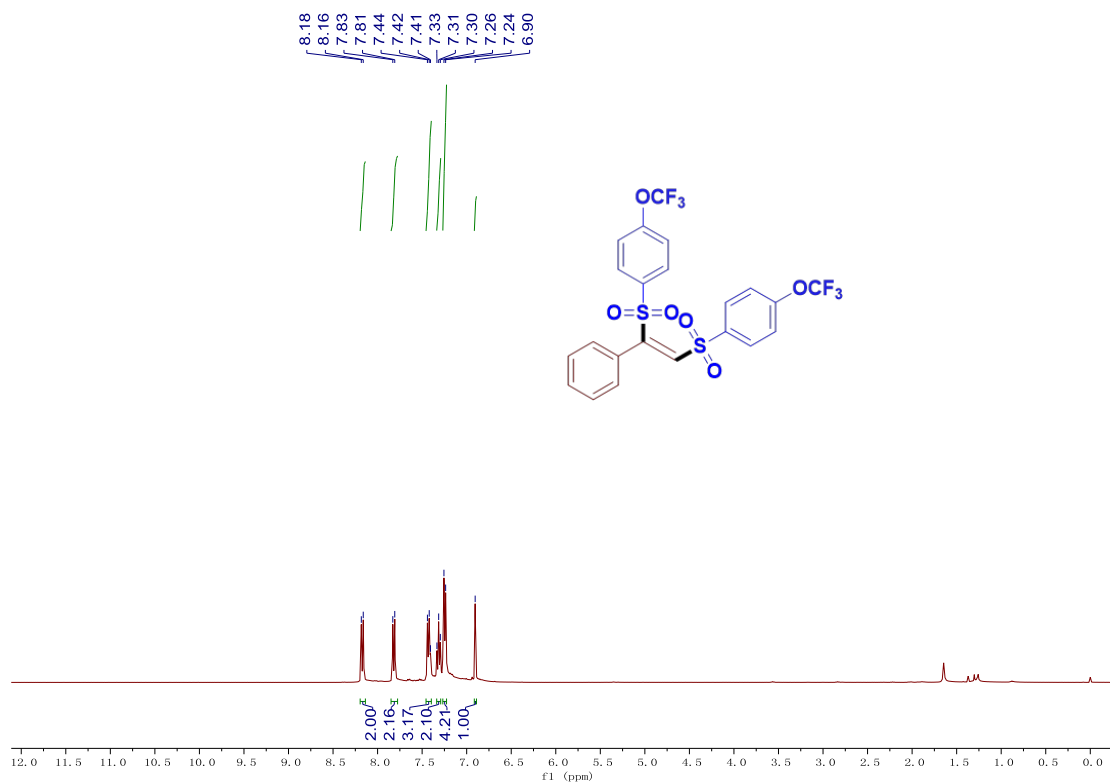
$^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectra of **3af**



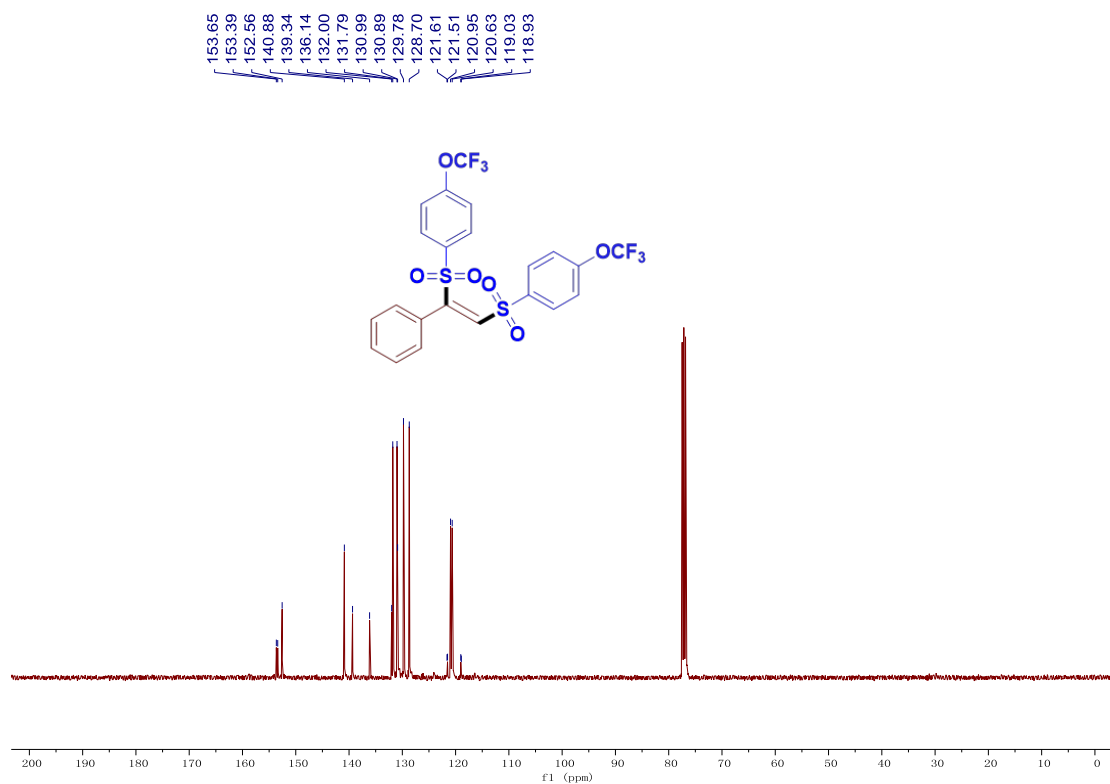
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) spectra of **3ag**



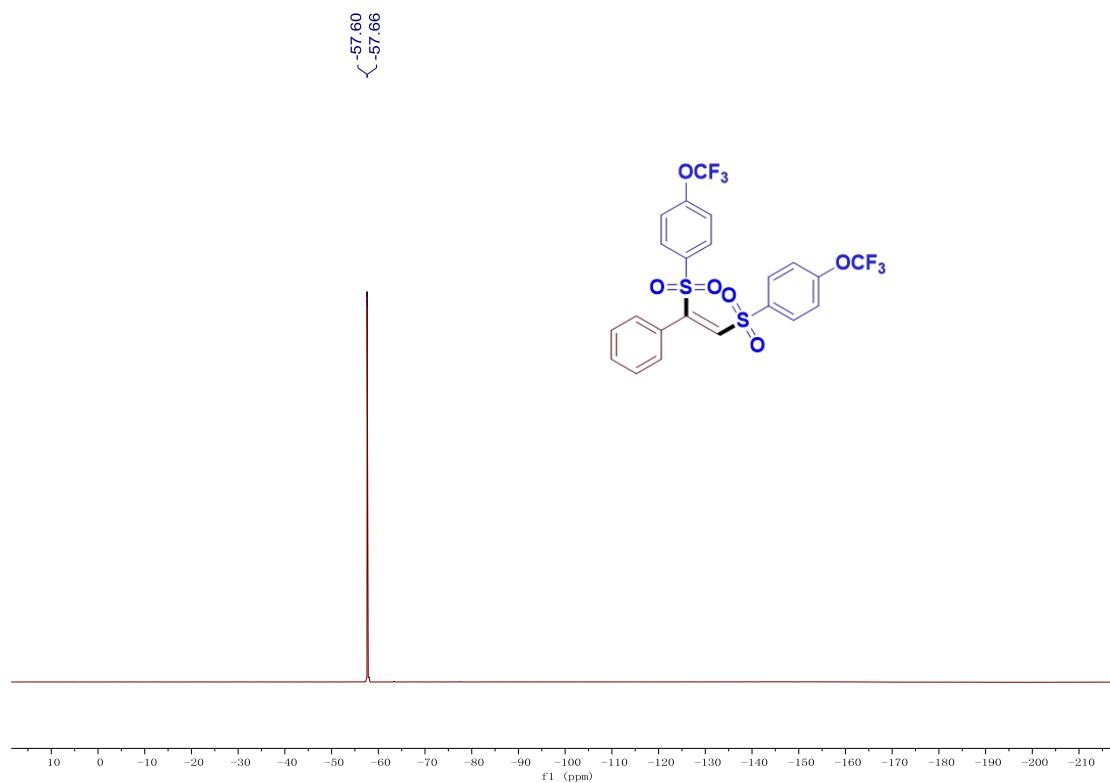
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of **3ag**



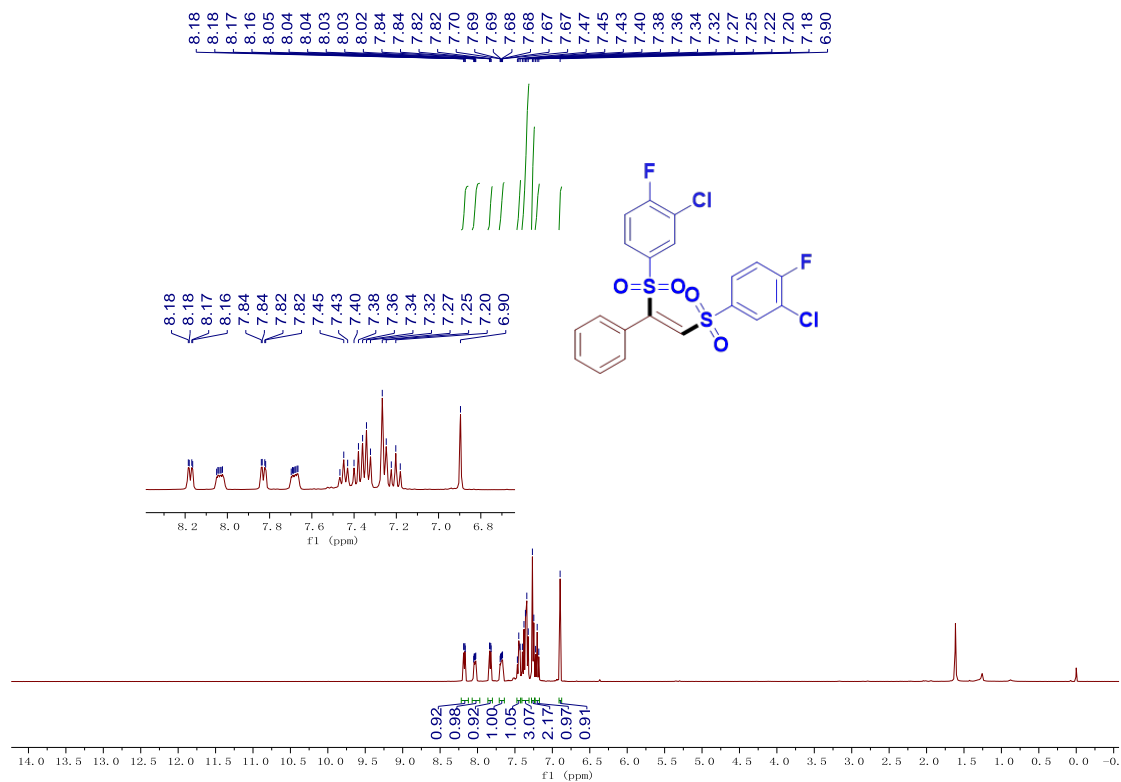
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectra of **3ah**



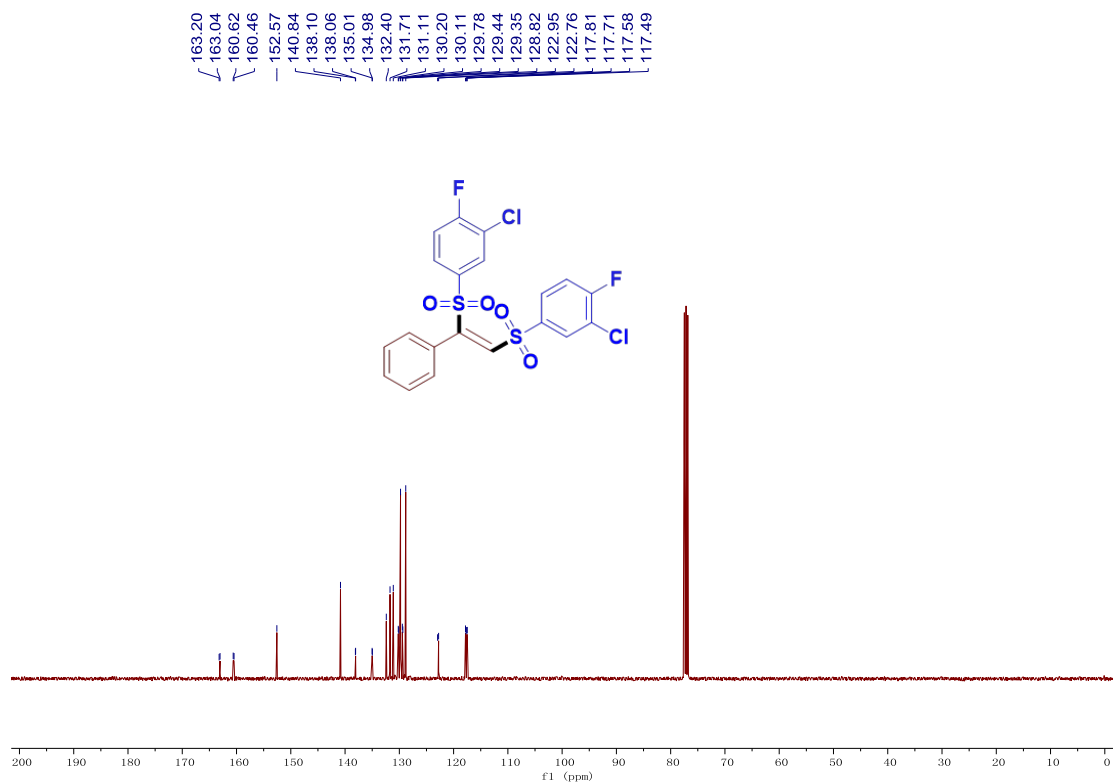
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of **3ah**



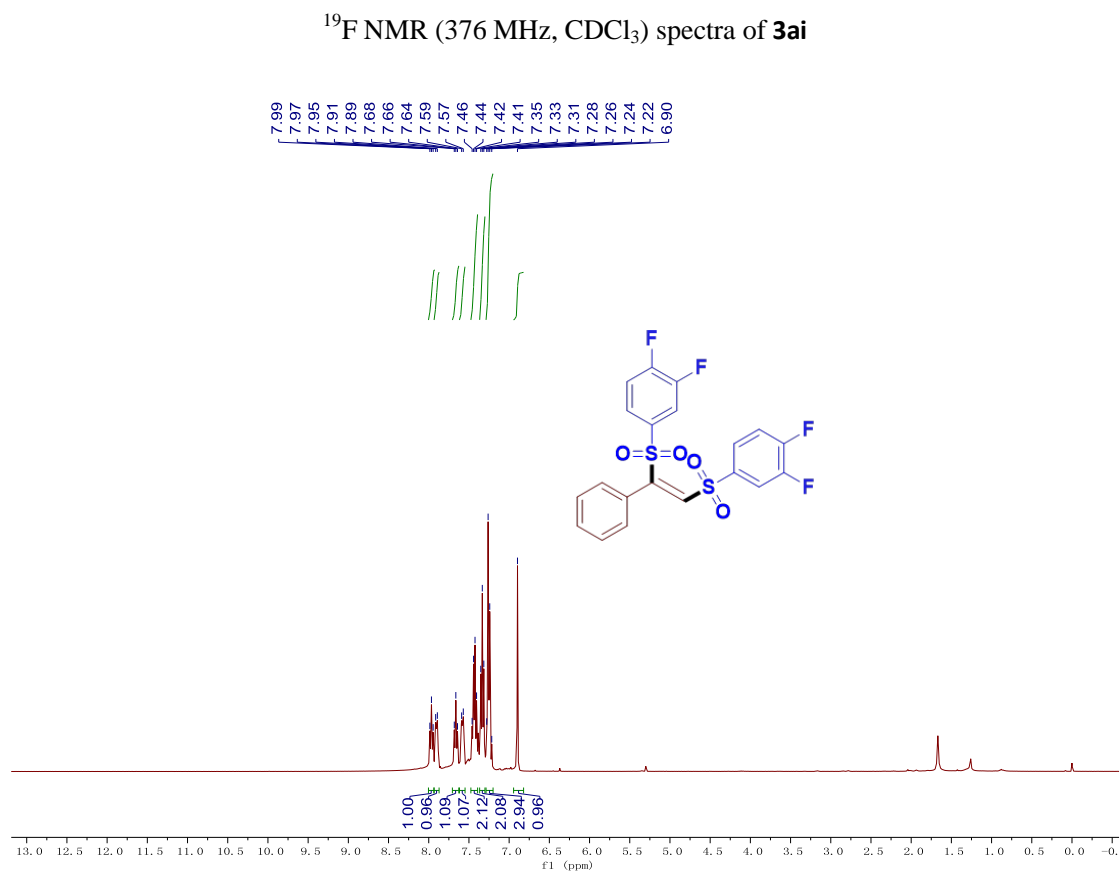
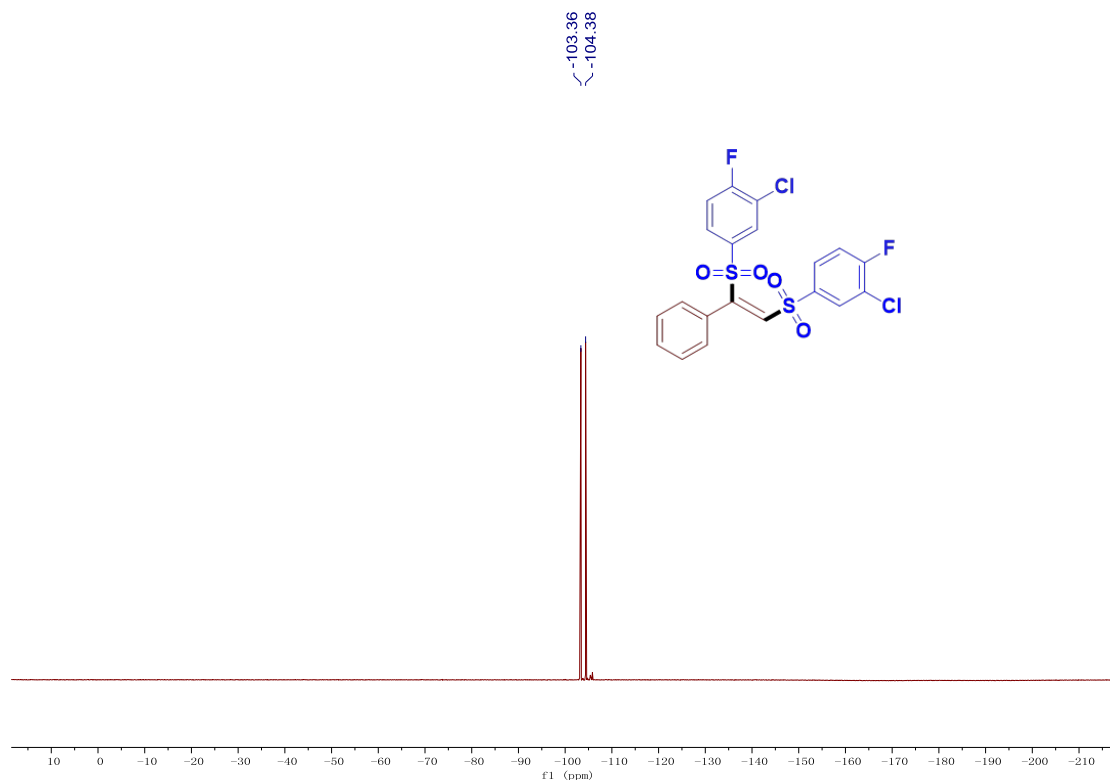
<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) spectra of **3ah**



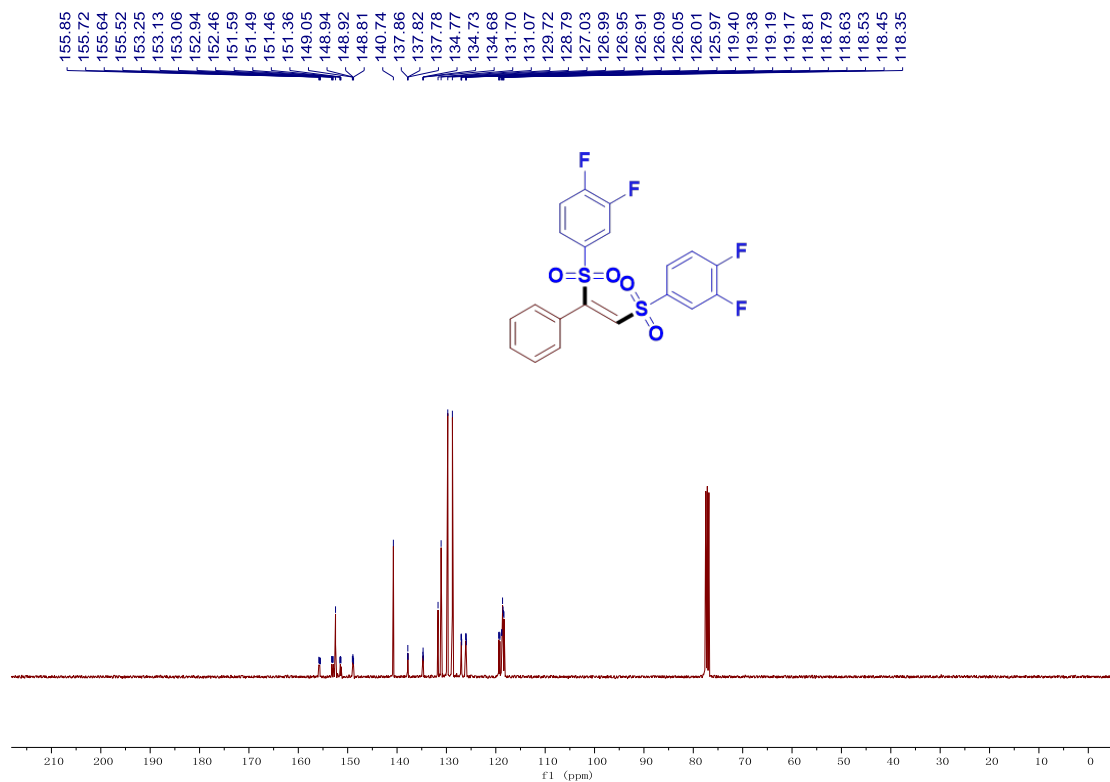
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectra of **3ai**



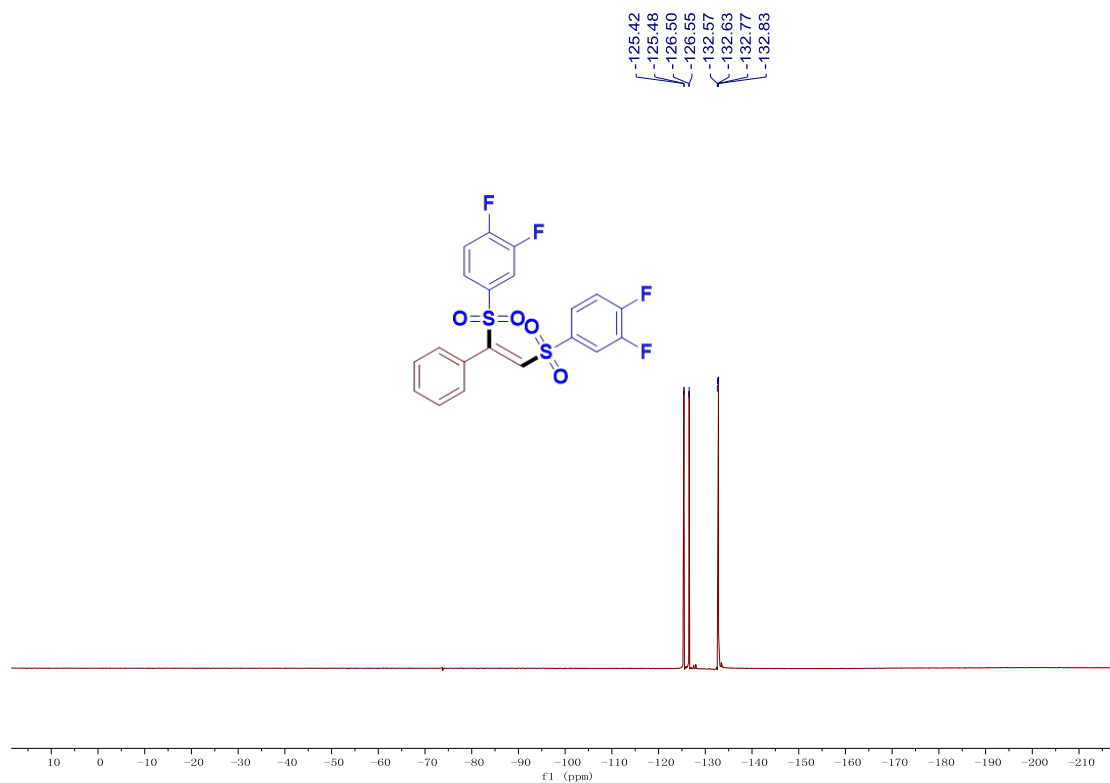
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of **3ai**



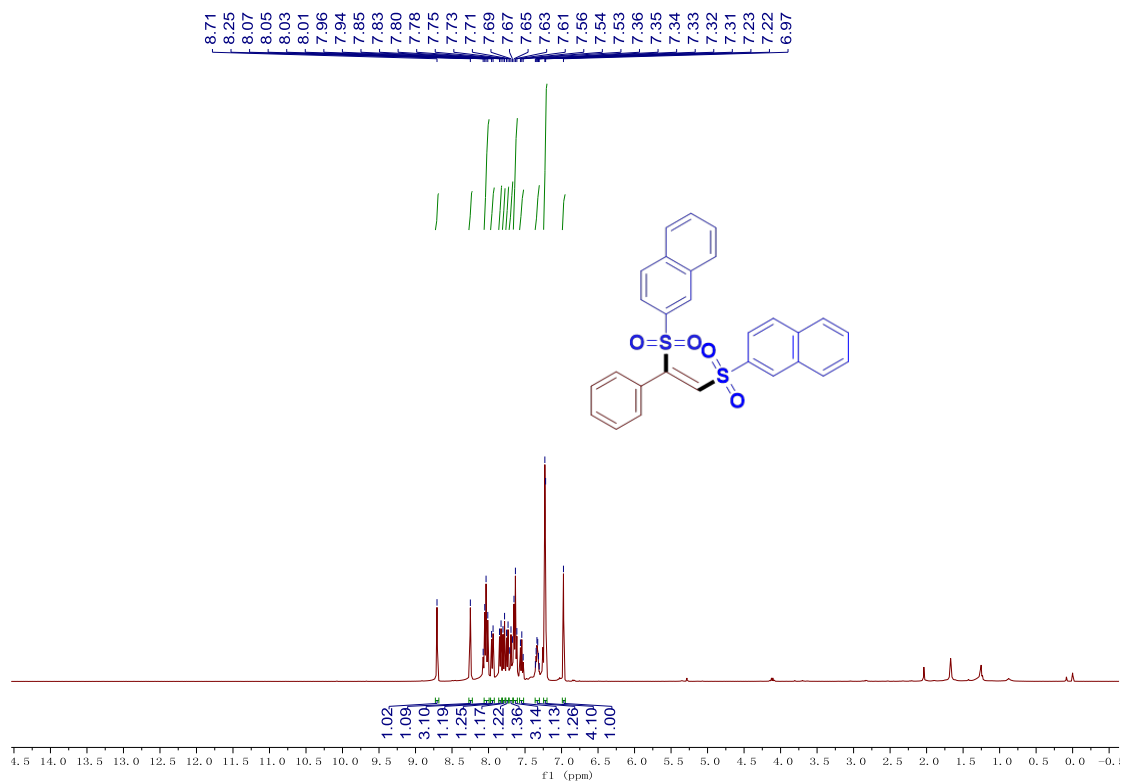




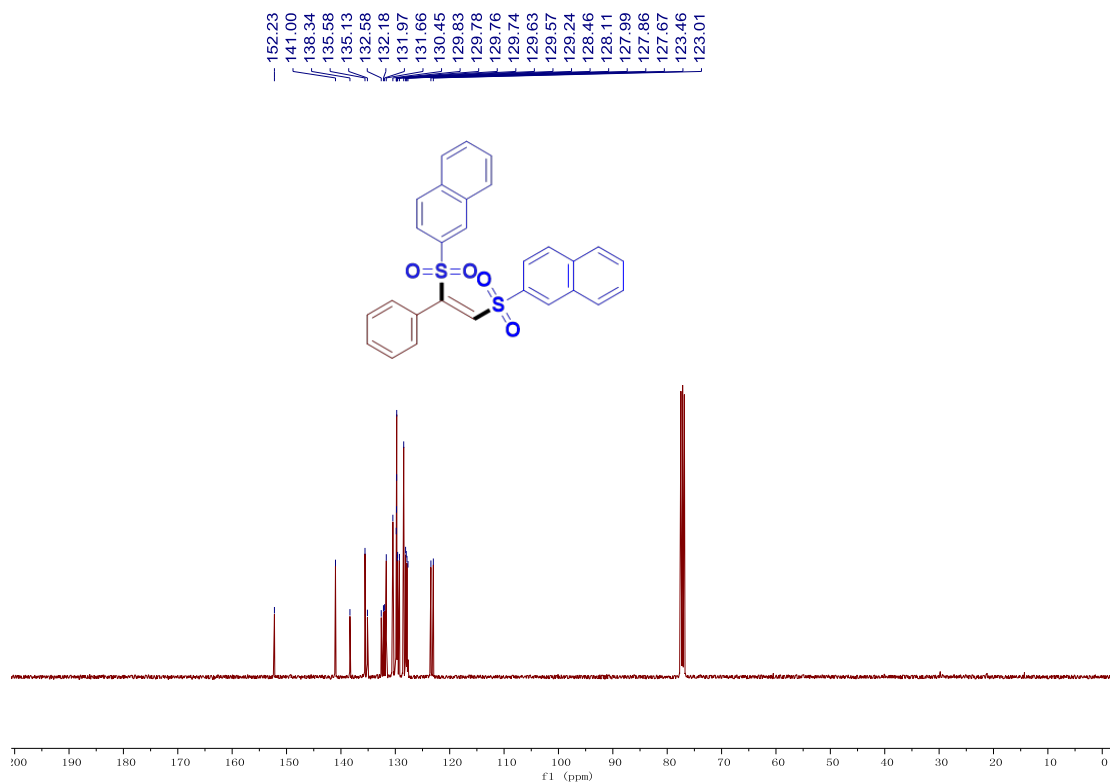
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of **3aj**



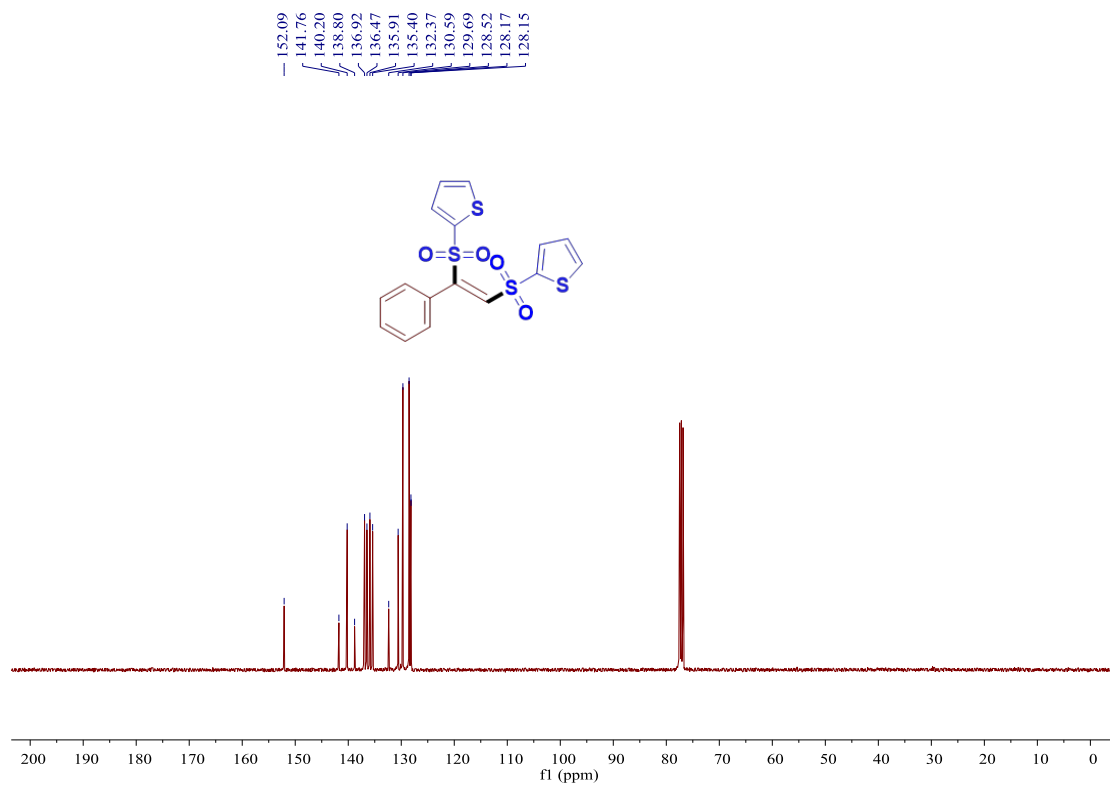
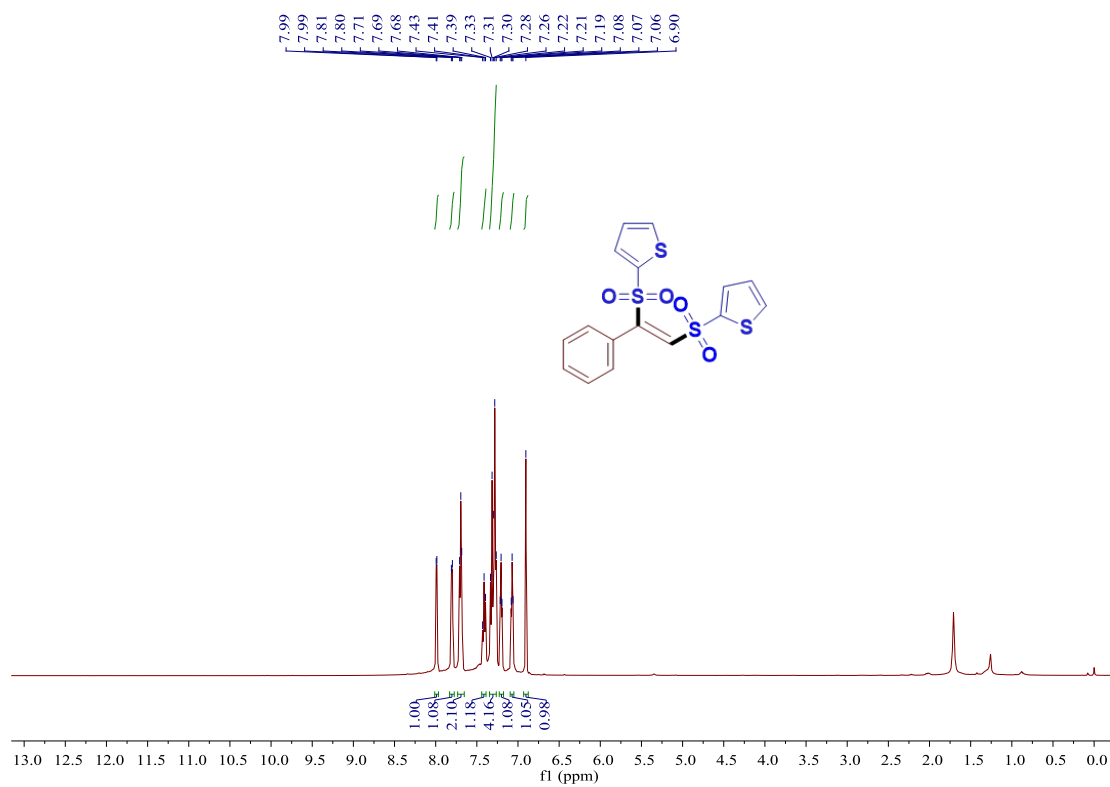
<sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>) spectra of **3aj**

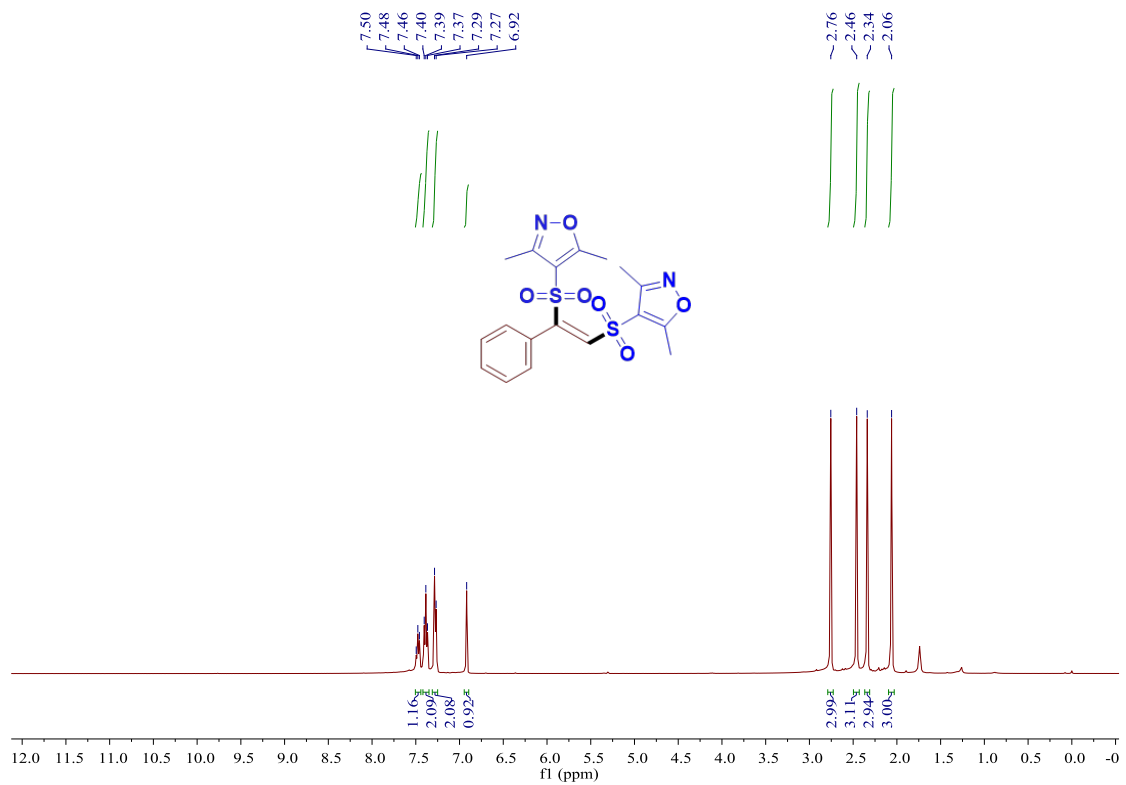


<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectra of **3ak**

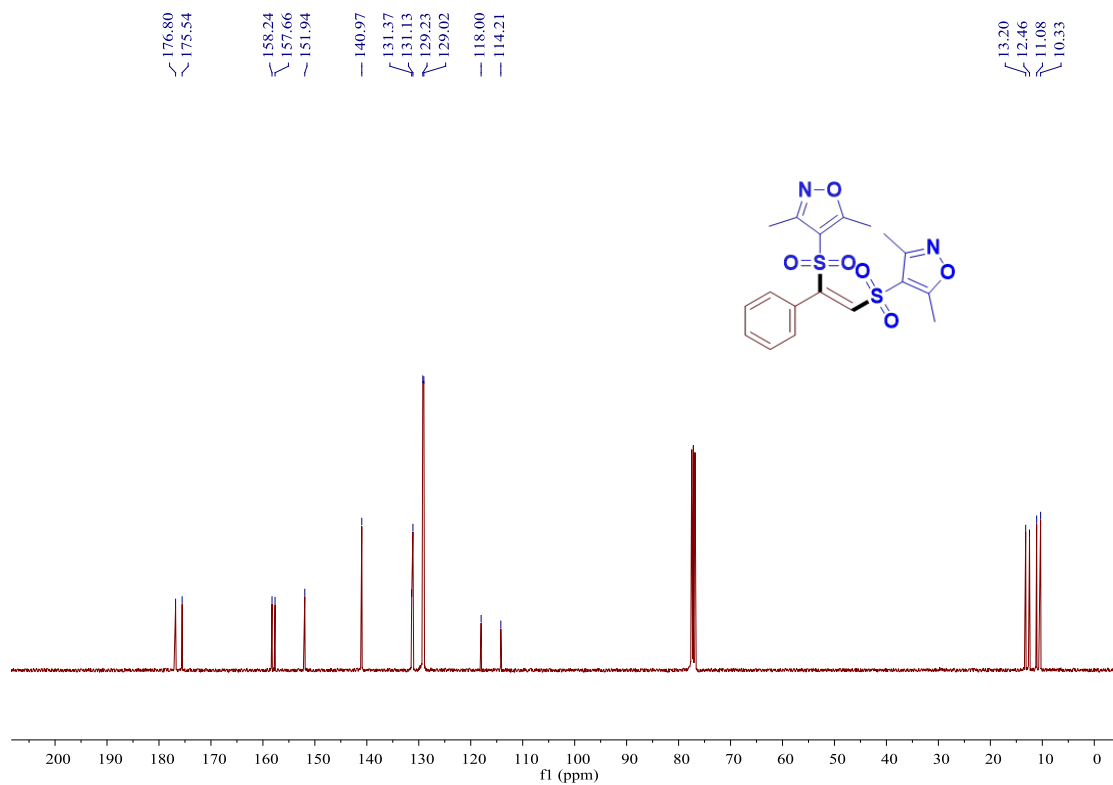


<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of **3ak**

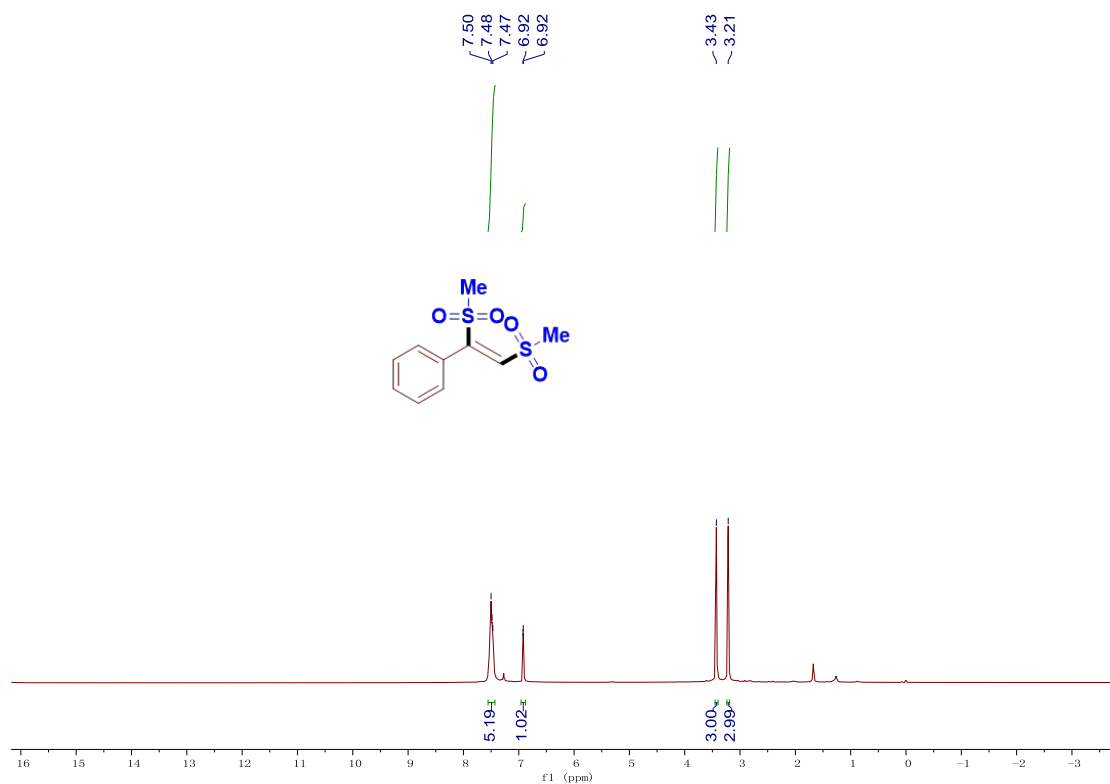




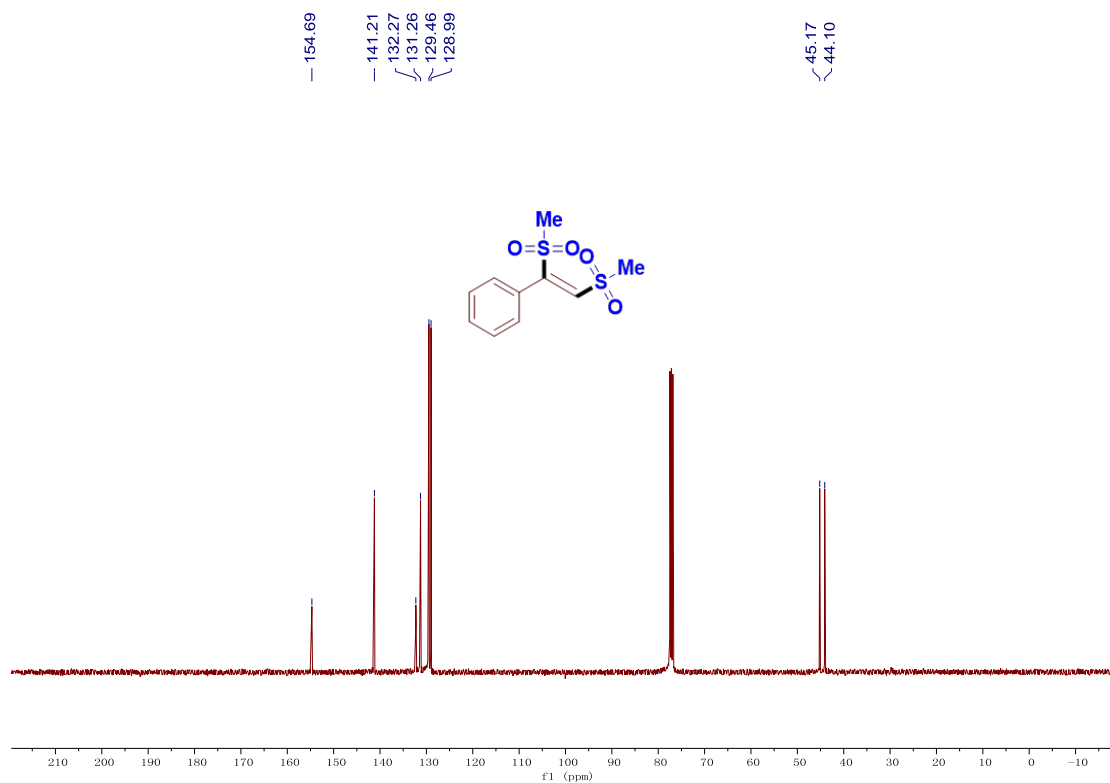
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectra of **3am**



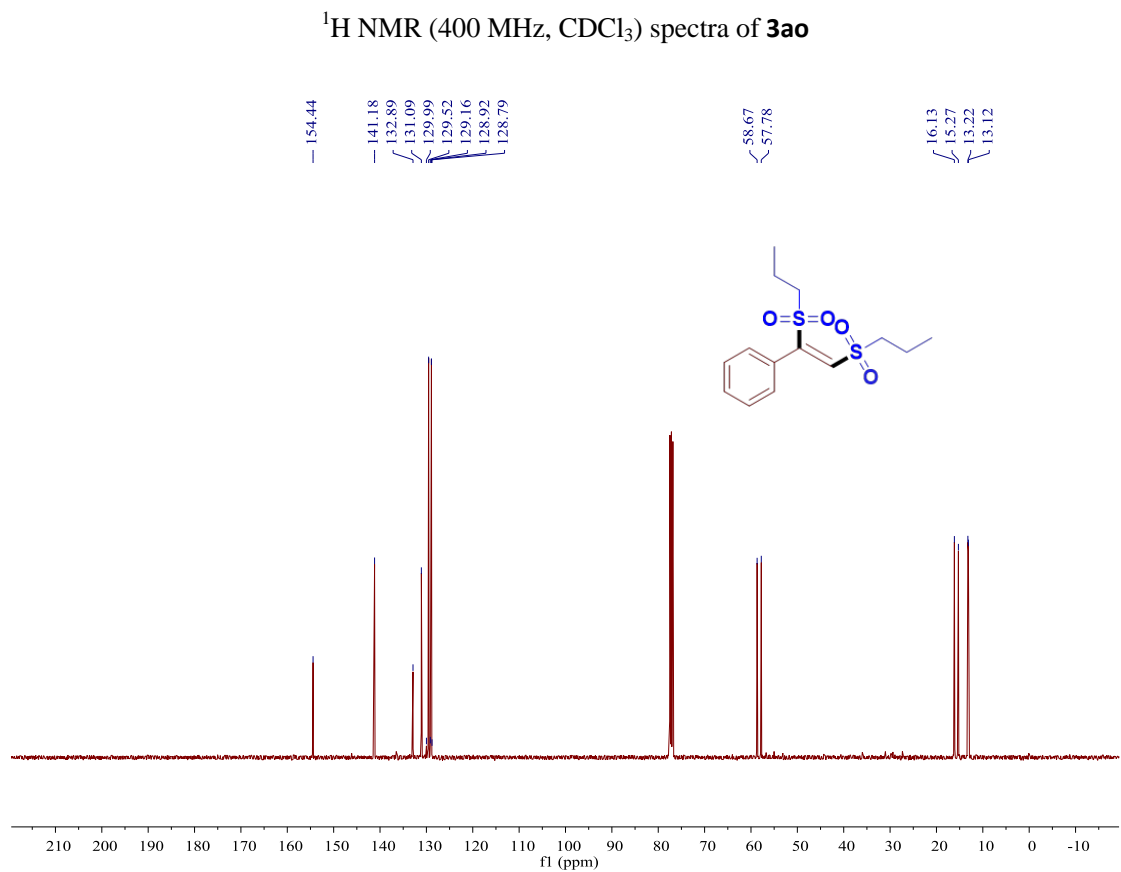
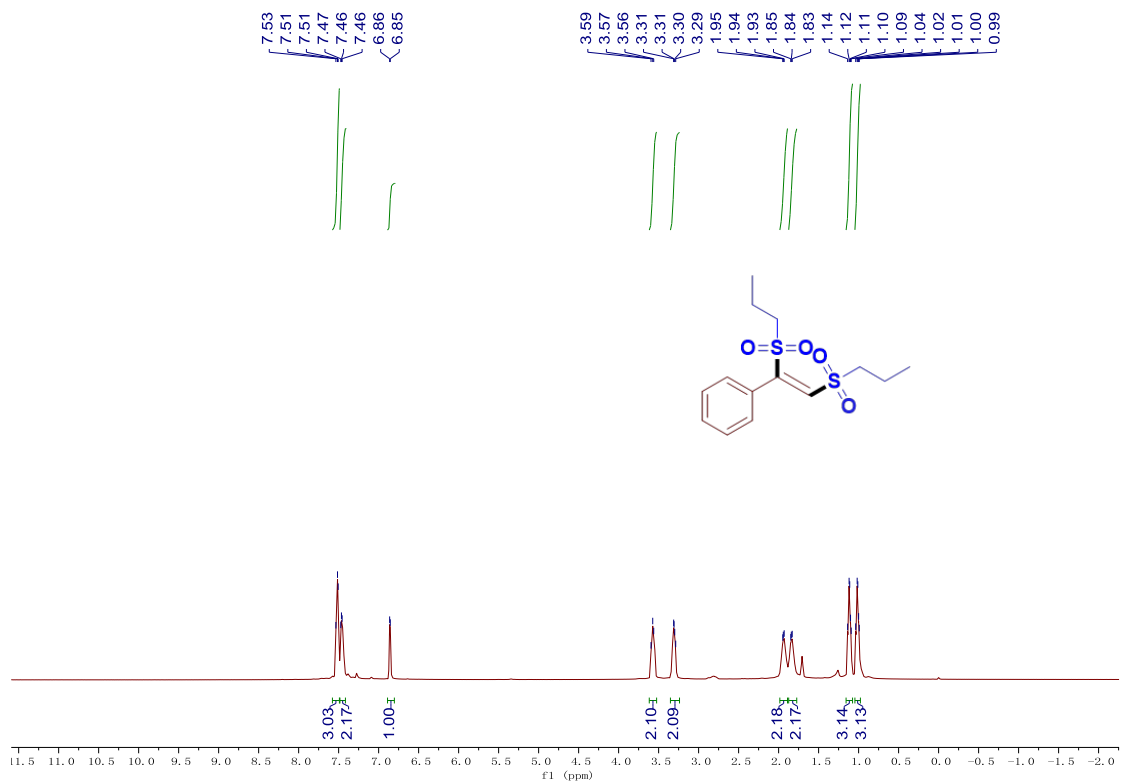
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of **3am**

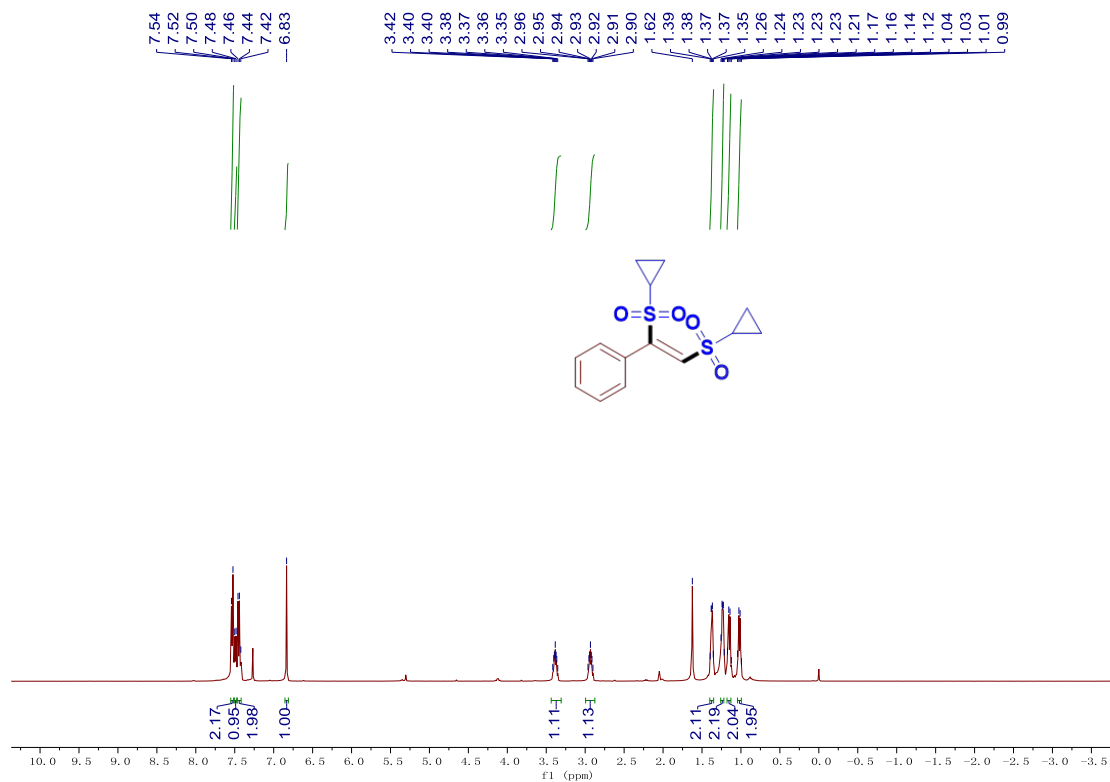


<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectra of **3an**

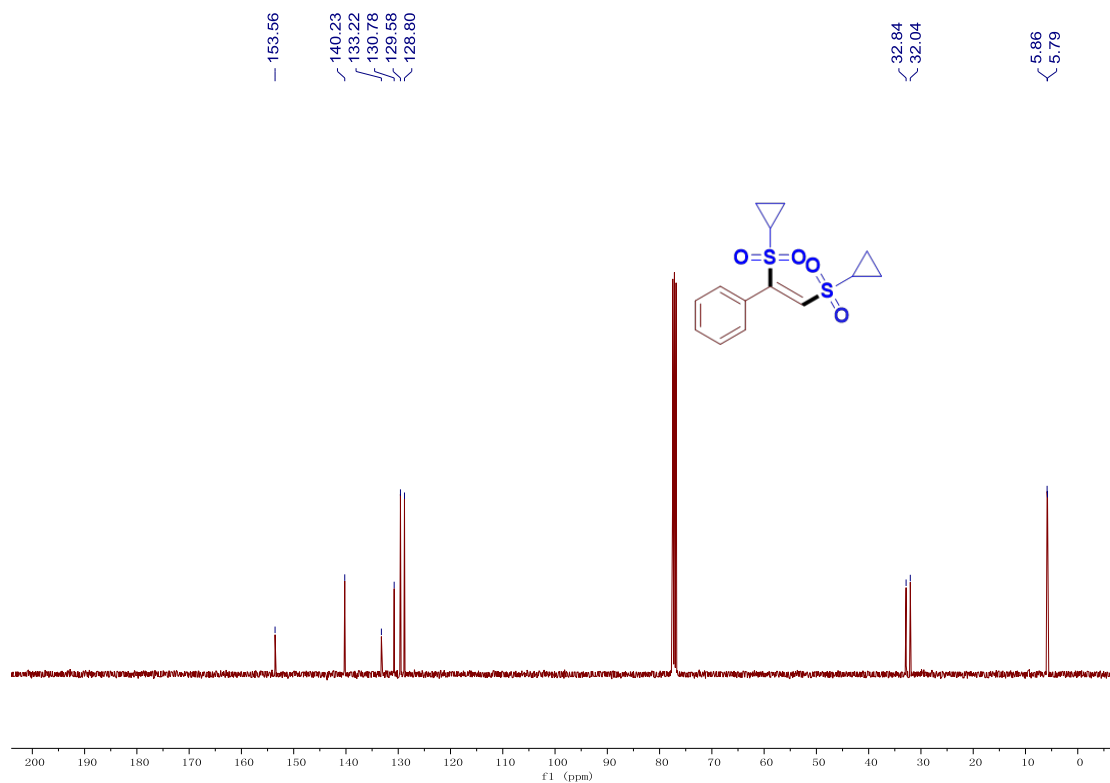


<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of **3an**

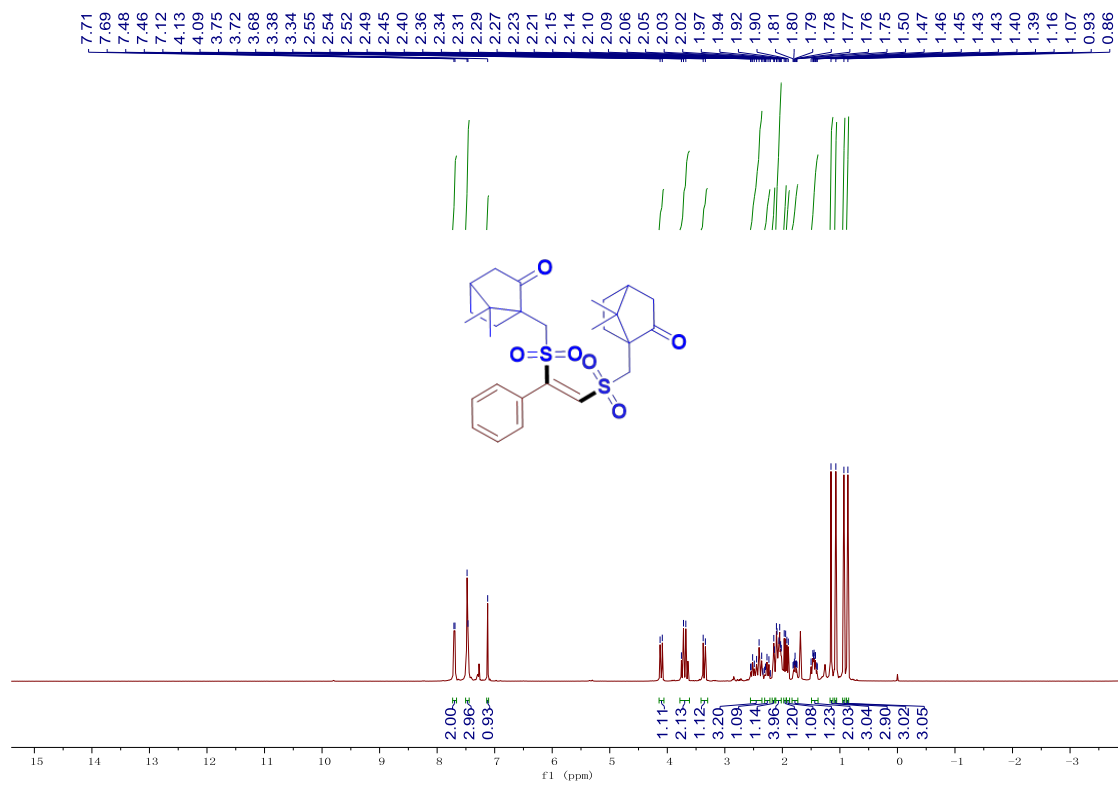




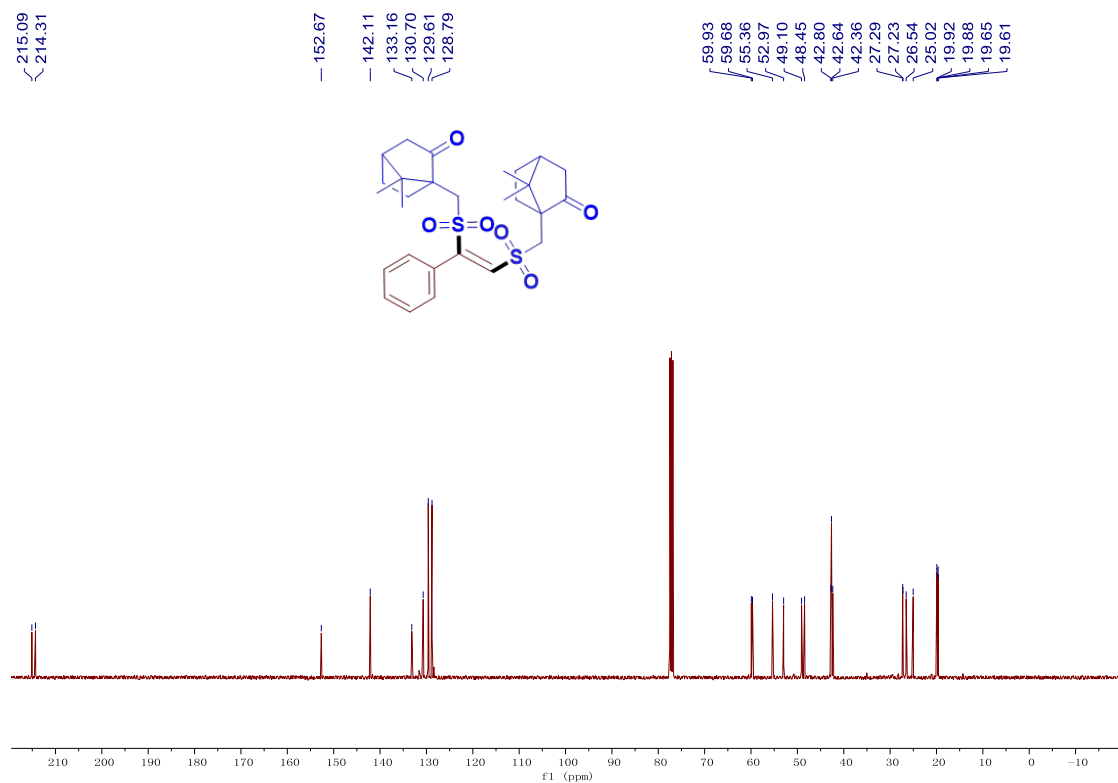
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectra of **3ap**



<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of **3ap**

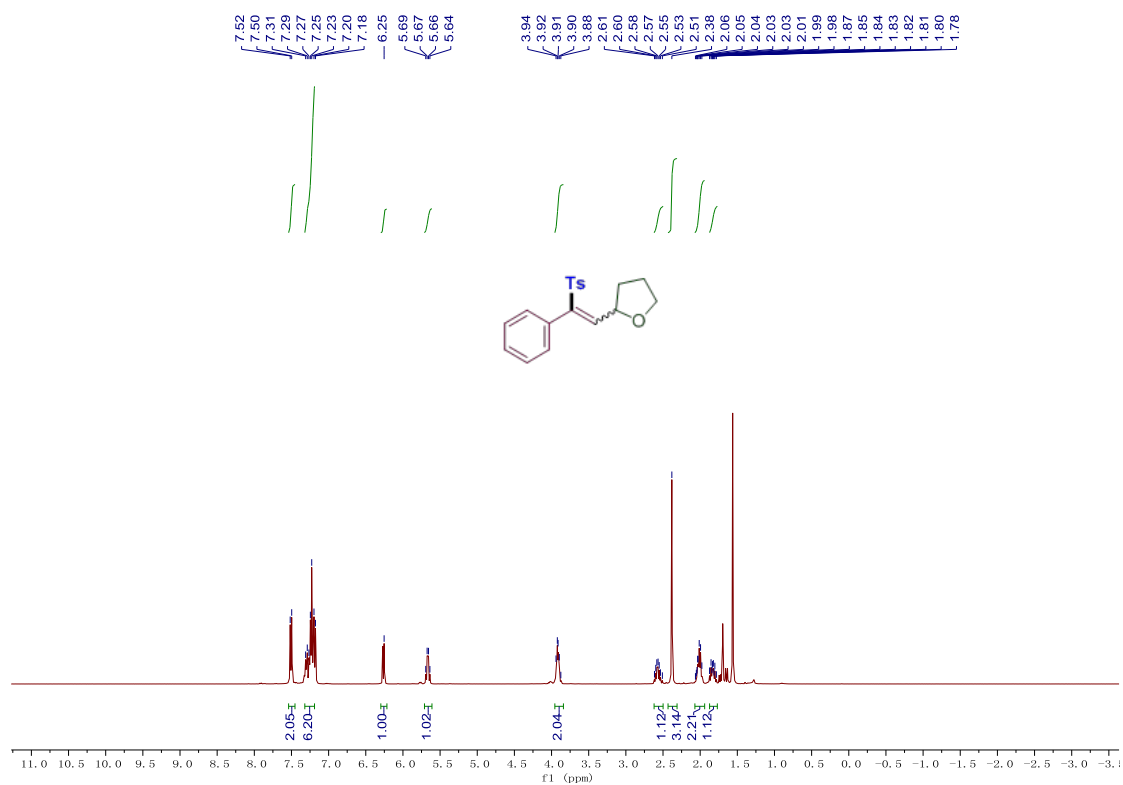


**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectra of **3aq****

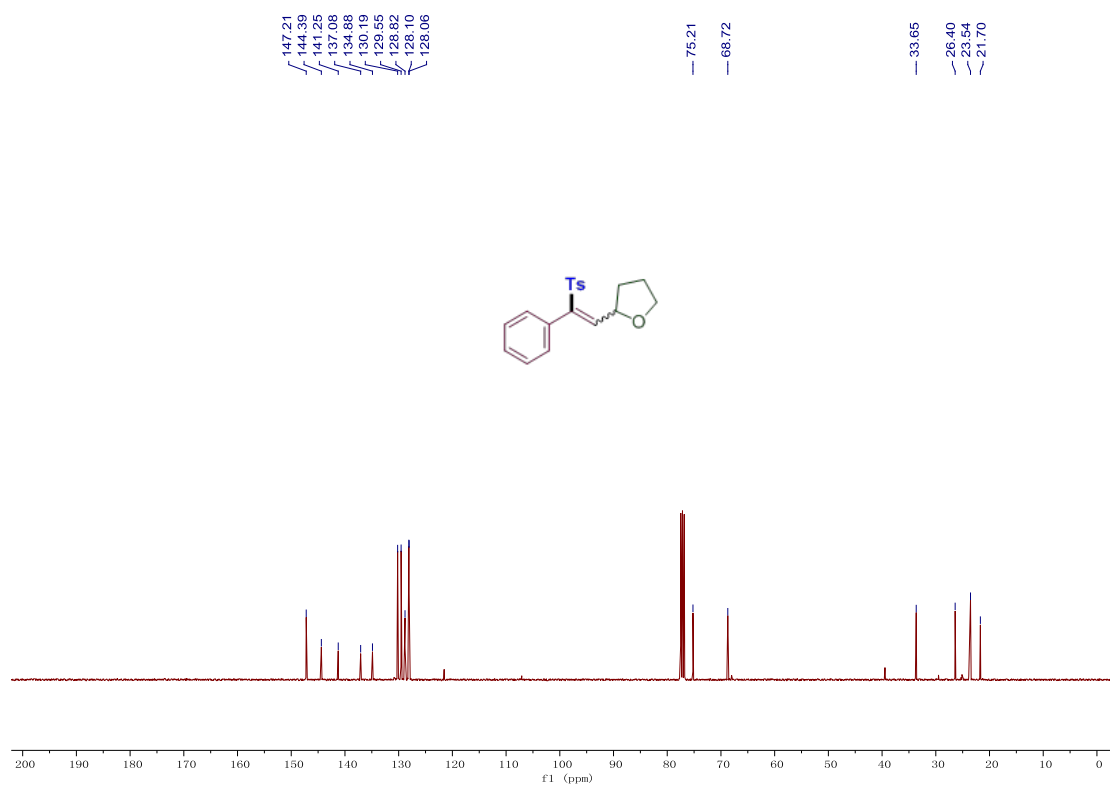


**<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of **3aq****

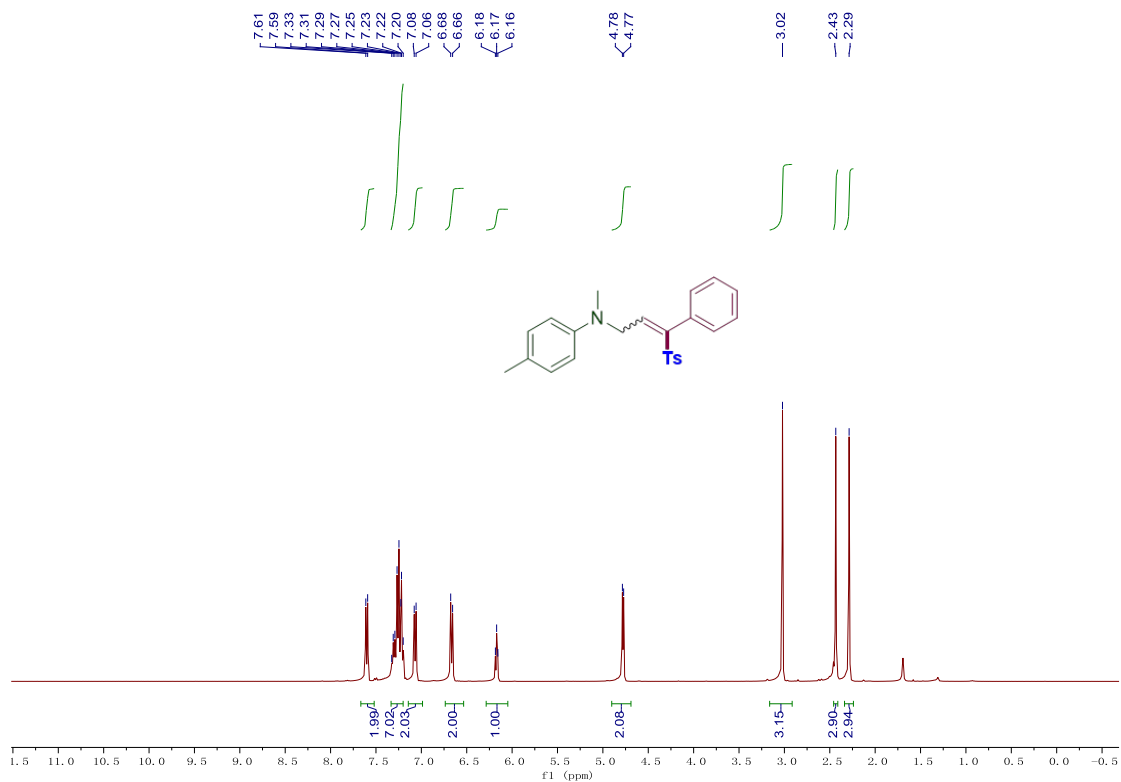




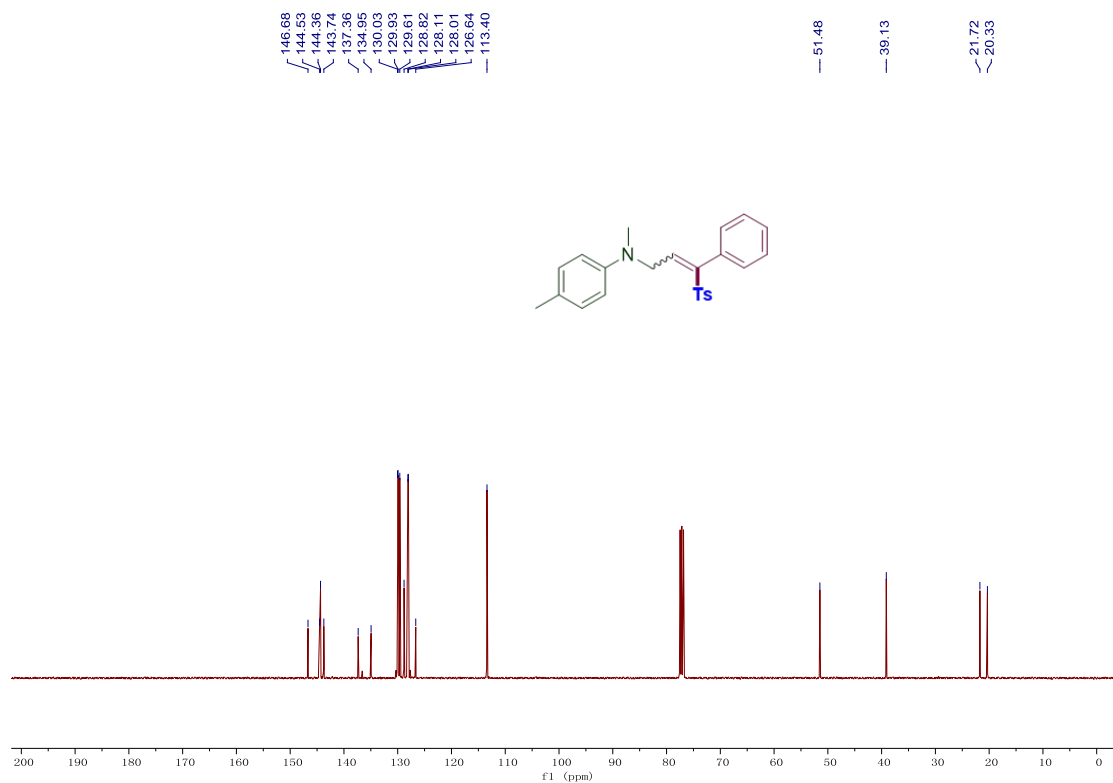
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectra of **5a**



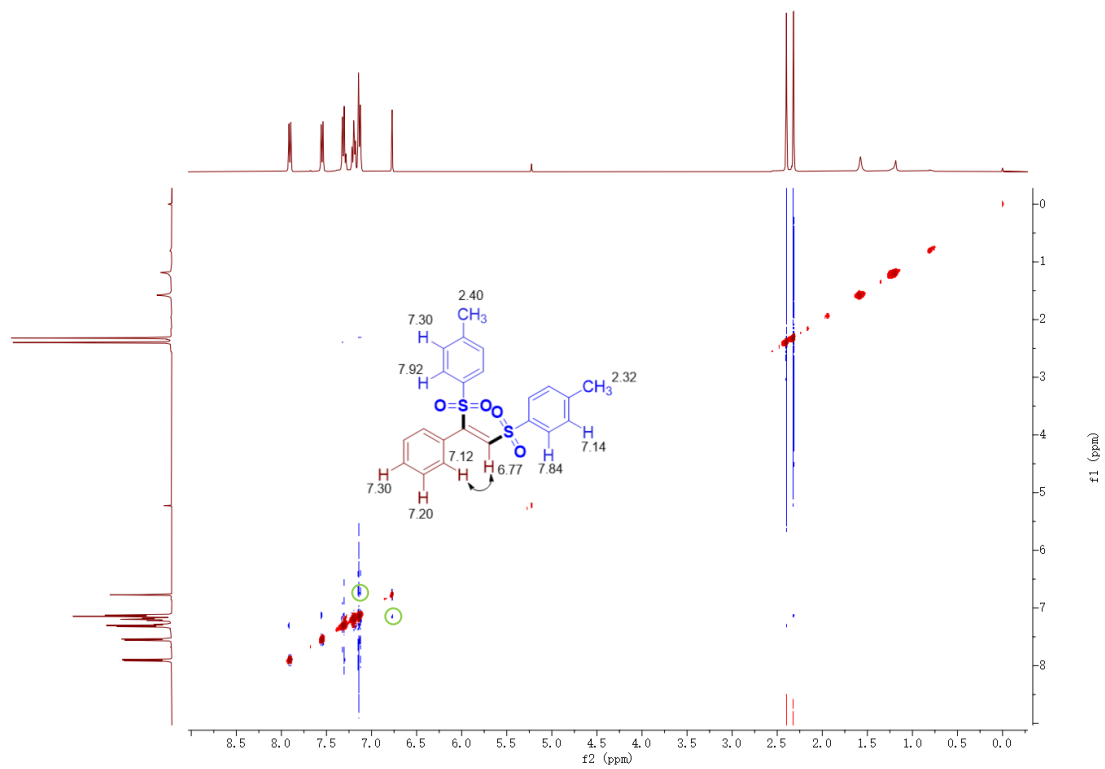
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of **5a**



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) spectra of **5b**



<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) spectra of **5b**



2D-NOESY spectrum of compound **3aa** (400 MHz, CDCl<sub>3</sub>)