Supporting Information

β-Trifluorosulfinylesters: Tuneable Reagents for Switchable Trifluoromethylsulfinylation and C-H Trifluoromethylthiolation

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Table of Contents

I. General information

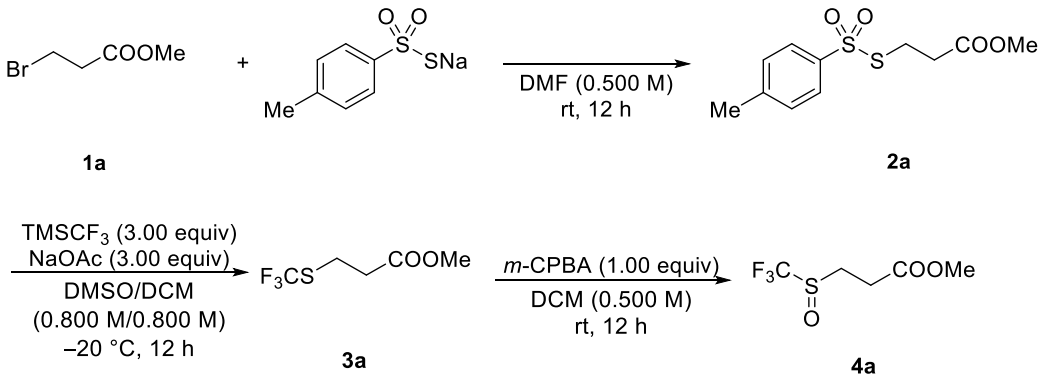
General procedures. General information unless specifically stated, all reagents were commercially obtained and where appropriate, purified prior to use. For example, dichloromethane (DCM) was freshly distilled from CaH_2. Other commercially available reagents and solvents were used directly without purification. Reactions were monitored by thin layer chromatography (TLC) using silica gel plates. Flash column chromatography was performed over silica (200 – 300 mesh). ^{1}H, ^{13}C, ^{19}F NMR spectra were recorded on a Bruker 400 MHz or 500 MHz spectrometer in $CDCl_3$ or d_6-DMSO. Multiplicities were given as: s (singlet); d (doublet); dd (doublets of doublet); t (triplet); q (quartet); td (triplet of doublets); tt (triplet of triplets) ddd (doublet of doublet of doublets) or m (multiplets). High resolution mass spectra (**HRMS**) of the products were obtained on a Bruker Daltonics micro TOF-spectrometer. High resolution mass spectra (**HRMS**) of the products were obtained on an Agilent Technologies micro Q-TOF-spectrometer.

Reagents. The following chemicals were used as received: Benzene (Hushi), *m*-Chloroperbenzoic acid (Energy-Chemical), 2-Chlorophenylboronic acid (Leyan), 2-Methoxyphenylboronic acid (Leyan), Iodobenzene (Energy-Chemical), Trifluoromethanesulfonic acid (Energy-Chemical), Trifluoromethanesulfonic anhydride (Adamas), 3-Bromopropionic acid (ALDRICH), Ethyl 3-bromopropionate (Leyan), Methyl 3-bromopropionate (Energy-Chemical), Isopropanol (Hushi), Sodium *p*-tolylsulfinate (Leyan), 1-Bromo-4-iodobenzene (Energy-Chemical), 1-Chloro-4-iodobenzene (Energy-Chemical), 4-Iodoanisole (Energy-Chemical), 2-Chloroanisole (Leyan), 3-Tolylboronic acid (Leyan), 1,2,3-Trimethoxybenzene (Leyan), Mesitylene (Energy-Chemical), 3-Chlorobenzophenone (Energy-Chemical), 2-Methylbenzophenone (Leyan), 3,4-Dimethylbenzophenone (Energy-Chemical), 4-Benzoylbiphenyl (Leyan), 4,4'-Dimethylbenzophenone (Energy-Chemical), 4,4'-Difluorobenzophenone (Leyan), 4,4'-Dibromobenzophenone (Leyan), 4,4'-Dimethoxybenzophenone (Leyan), 1,1-Diphenylethylene (Energy-Chemical), Triphenylphosphine (Energy-Chemical), Potassium *tert*-butoxide (Leyan), Sodiu

m acetate anhydrous (3A), Trimethyl(trifluoromethyl)silane (Energy-Chemical), 1-Bro mo-2-methoxybenzene (Energy-Chemical), 1,3-Benzodioxole (Leyan), 1,2-Dimethox ybenzene (Leyan), 1,3-Dimethoxybenzene (Energy-Chemical), Naphthalene (Energy-Chemical), 1-Methoxynaphthalene (Leyan), 2-Methoxynaphthalene (Leyan), 1,3,5-Tri methoxybenzene (Energy-Chemical), β-Estradiol (Heowns), Ethyl bromoacetate (Ener gy-Chemical), Sodium thiocyanate (3A), Benzyl bromide (Energy-Chemical).

II. Synthesis of starting materials

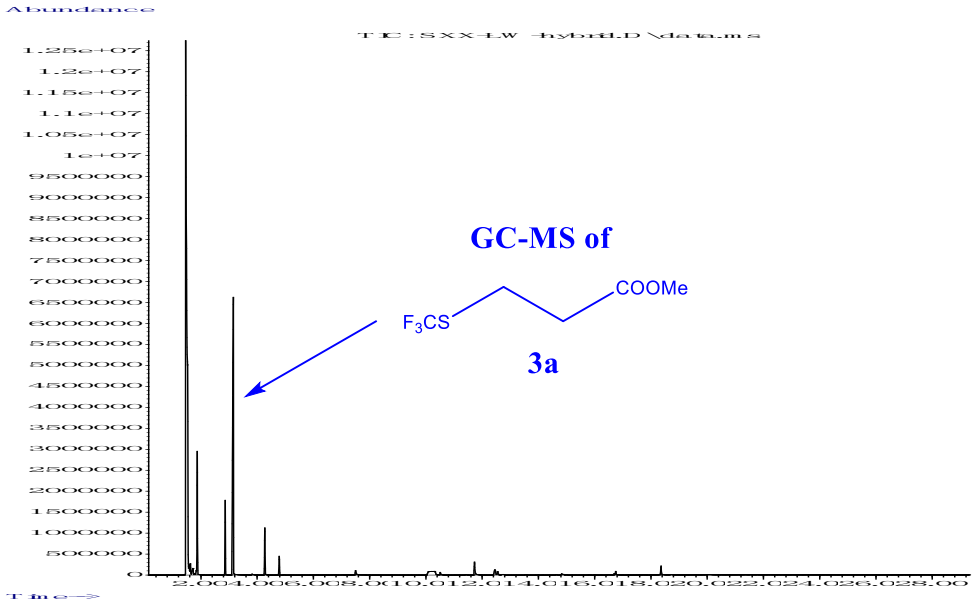
1. Synthesis of difunctional EWG reagents 4a-4e:

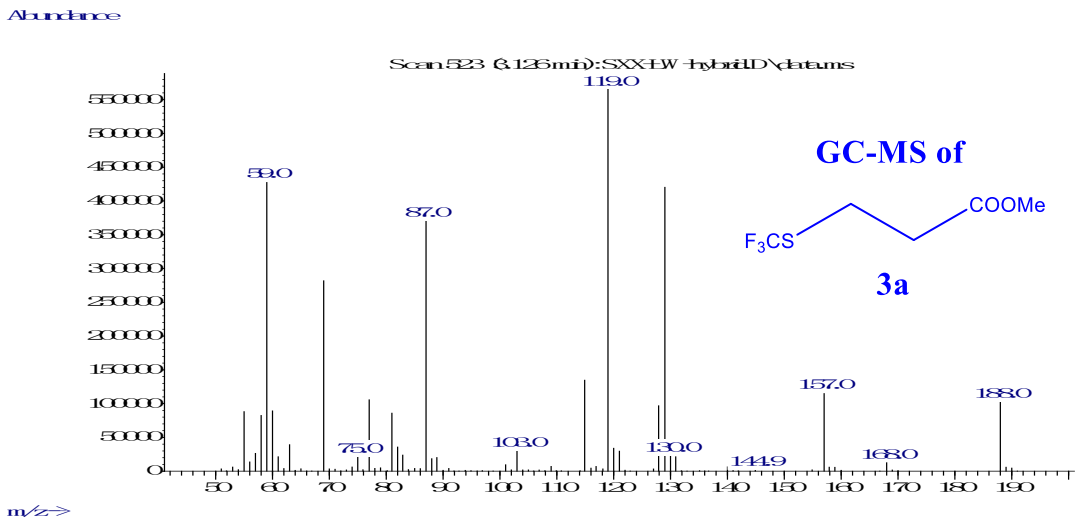


An oven-dried 300-mL round-bottom flask, equipped with a stir bar, was charged with sodium 4-methylbenzenesulfonothioate (10.5 g, 50.0 mmol, 1.00 equiv), methyl 3-bromopropanoate (8.35 g, 50.0 mmol, 1.00 equiv) and anhydrous DMF (100 mL) was added under N_2 atmosphere. The mixture was allowed to stir at room temperature for 12 h, then was poured into brine (200 mL) and extracted with EtOAc (50.0 mL × 3), the combined organic layers were extracted with brine (100 mL × 5). The combined organic layers were dried over by Na_2SO_4 then filtered. The solvent was removed by rotary evaporation. The residues were recrystallized from petroleum ether (50.0 mL)[1]. The product was filtered and washed by petroleum ether to give the desired product **2a** as a white solid (11.8 g, 43.2 mmol, 86.3% yield). **M.p.** = 53.9 – 54.3 °C; **IR (thin film)** 2921 (w), 1729 (s), 1434 (m), 1370 (m), 1319 (s), 1201 (s), 1160 (s), 1074 (s), 976 (m), 939 (m), 817 (s), 657 (s) cm^{-1}; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.80 (2H, d, J = 6.5 Hz),

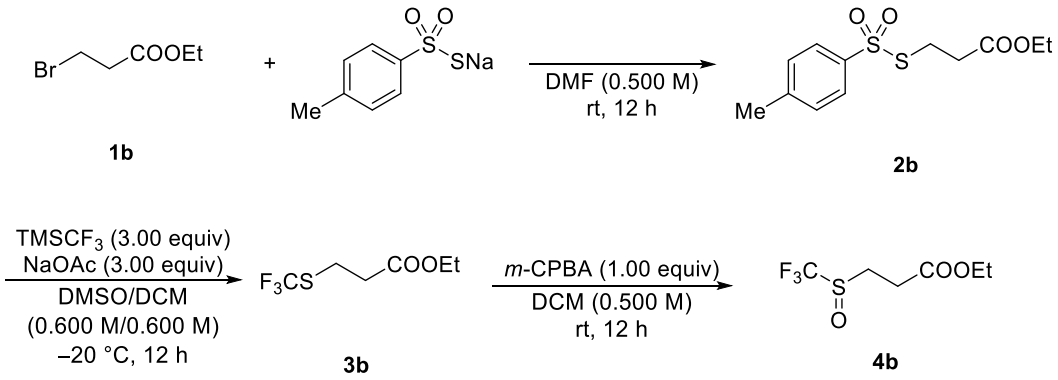
7.34 (2H, d, J = 7.9 Hz), 3.66 (3H, s), 3.17 (2H, t, J = 7.0 Hz), 2.72 (2H, t, J = 7.0 Hz), 2.44 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 171.6, 145.1, 141.7, 130.1, 127.2, 52.1, 34.0, 30.6, 21.8; **HRMS** (ESI^+) $[M+Na]^+$ calc'd for $C_{11}H_{14}O_4S_2Na$: 297.0226, found: 297.0224.

An oven-dried 200-mL round-bottom flask, equipped with a stir bar, was charged with **2a** (11.0 g, 40.0 mmol, 1.00 equiv), NaOAc (9.84 g, 120 mmol, 3.00 equiv) and anhydrous DMSO (50.0 mL) and DCM (50.0 mL) was added under N_2 atmosphere. The mixture was cooled to –20 ºC. Then $TMSCF_3$ (17.1 g, 120 mmol, 3.00 equiv) was added dropwise to the mixture. The mixture was allowed to stir at –20 ºC for 12 h. The mixture was poured into brine (100 mL) and extracted with DCM (50.0 mL × 3), the combined organic layers were washed by brine (50.0 mL × 5). The combined organic layers were dried over by Na_2SO_4 then filtered. The solvent was removed by rotary evaporation and the residue was used to next step directly without further purification[2]. The compound **3a** was detected and confirmed by GC-MS.





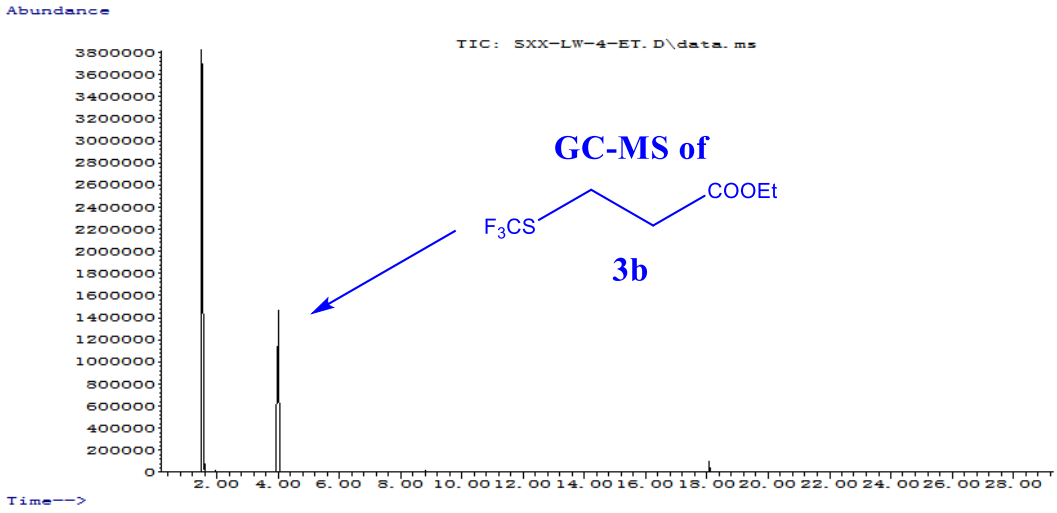
An oven-dried 200-mL round-bottom flask, equipped with a stir bar, was charged with methyl 3-((trifluoromethyl)sulfinyl)propanoate (40.0 mmol, 1.00 equiv), *m*-CPBA (85.0 wt%, 8.12 g, 40.0 mmol, 1.00 equiv) and anhydrous DCM (80.0 mL) was added under N_2 atmosphere and the mixture was stirred at room temperature for 12 h. The mixture was filtered to remove most of solids. Then the mixture was diluted with 50.0 mL saturated Na_2CO_3 solution and extracted with DCM (30.0 mL × 3), the combined organic layers were washed by brine (40.0 mL × 3). The combined organic layers were dried over by Na_2SO_4 then filtered. The solvent was removed by rotary evaporation and purified by reduced pressure distillation to give desired product **4a** (5.76 g, 28.2 mmol, 70.5% yield) which was a known compound. The target product can be further purified by flash silica gel chromatography (Eluent: 100:1 to 5:1 petroleum ether: ethyl acetate). The spectral data match those previously reported[3].



An oven-dried 100-mL round-bottom flask, equipped with a stir bar, was charged with sodium 4-methylbenzenesulfonothioate (3.15 g, 15.0 mmol, 1.00 equiv), ethyl 3-

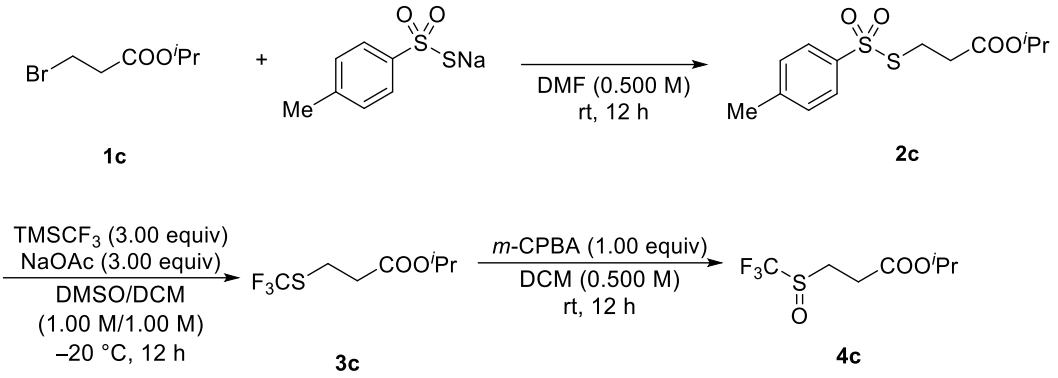
bromopropanoate (2.72 g, 15.0 mmol, 1.00 equiv) and anhydrous DMF (30.0 mL) was added under N_2 atmosphere. The mixture was allowed to stir at room temperature for 12 h then was poured into brine (40.0 mL) and extracted with EtOAc (20.0 mL × 3). The combined organic layers were dried over by Na_2SO_4 then filtered. The solvent was removed by rotary evaporation and the residue was purified by flash silica gel chromatography (Eluent: 100:1 to 10:1 petroleum ether: ethyl acetate) to give the desired product **2b** as a white solid (3.37 g, 11.7 mmol, 77.9 % yield). **M.p. =** 41.6 – 42.5 ºC; **IR (thin film)** 2921 (w), 1726 (s), 1404 (m), 1377 (s), 1315 (m), 1222 (s), 1197 (s), 1132 (s), 1075 (s), 1014 (m), 925 (s), 818 (m), 702 (m), 652 (s) cm^{-1}; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.81 (2H, d, J = 8.3 Hz), 7.35 (2H, d, J = 8.3 Hz), 4.13 (2H, q, J = 7.1 Hz), 3.18 (2H, t, J = 7.0 Hz), 2.71 (2H, t, J = 7.0 Hz), 2.45 (3H, s), 1.24 (3H, t, J = 7.1 Hz); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 171.1, 145.1, 141.8, 130.1, 127.2, 61.2, 34.2, 30.7, 21.8, 14.3; **HRMS** (ESI^+) $[M+H]^+$ calc'd for $C_{12}H_{17}O_4S_2$: 289.0563, found: 289.0563.

An oven-dried 100-mL round-bottom flask, equipped with a stir bar, was charged with **2b** (2.56 g, 8.90 mmol, 1.00 equiv), NaOAc (2.19 g, 26.7 mmol, 3.00 equiv) and anhydrous DMSO (15.0 mL) and DCM (15.0 mL) was added under N_2 atmosphere. The mixture was cooled to –20 ºC. Then $TMSCF_3$ (3.80 g, 26.7 mmol, 3.00 equiv) was added dropwise to the mixture and the mixture was allowed to stir at –20 ºC for 12 h. The mixture was poured into brine (50.0 mL) and extracted with DCM (30.0 mL × 3). The combined organic layers were washed by brine (40.0 mL × 5), and dried over by Na_2SO_4 then filtered. The solvent was removed by rotary evaporation and the residue was used to next step directly without purification. The compound **3b** was detected and confirmed by GC-MS.



An oven-dried 100-mL round-bottom flask, equipped with a stir bar, was charged with ethyl 3-((trifluoromethyl)thio)propanoate (8.90 mmol, 1.00 equiv), *m*-CPBA (85.0 wt%, 1.81 g, 8.90 mmol, 1.00 equiv) and anhydrous DCM (18.0 mL) was added under N_2 atmosphere. The mixture was stirred at room temperature for 12 h. The mixture was filtered to remove most of solids. Then the mixture was diluted with 20.0 mL saturated Na_2CO_3 solution and extracted with DCM (20.0 mL × 3), the combined organic layers were washed by brine (20.0 mL × 3). The combined organic layers were dried over by Na_2SO_4 then filtered. The solvent was removed by rotary evaporation, and the residue was purified by flash silica gel chromatography (Eluent: 100:1 to 5:1 petroleum ether: ethyl acetate) to give the desired product **4b** as a colorless liquid (1.00 g, 4.64 mmol, 51.6% yield). **IR (thin film)** 2987 (w), 1732 (s), 1376 (m), 1173 (s), 1139 (s), 1076 (s) cm^{-1}; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 4.20 (2H, q, J =

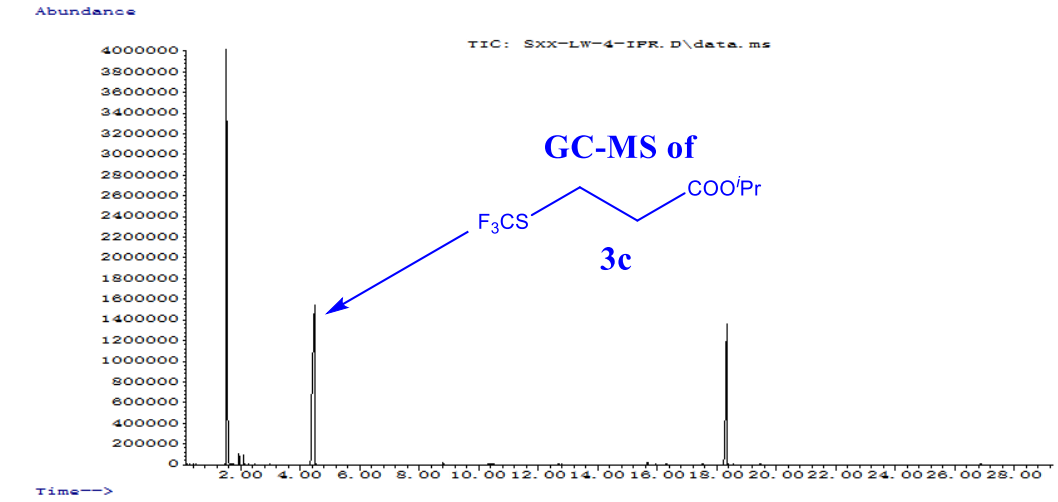
7.1 Hz), 3.26 (2H, m), 3.04 – 2.77 (2H, m), 1.28 (3H, t, J = 7.1 Hz); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 170.5, 125.5 (q, $J_{C\text{-}F}$ = 335.0 Hz), 61.8, 43.5, 25.9, 14.2; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –73.7; **HRMS** (ESI^+) $[M+Na]^+$ calc'd for $C_6H_9F_3NaO_3S$: 241.0117, found: 241.0119.

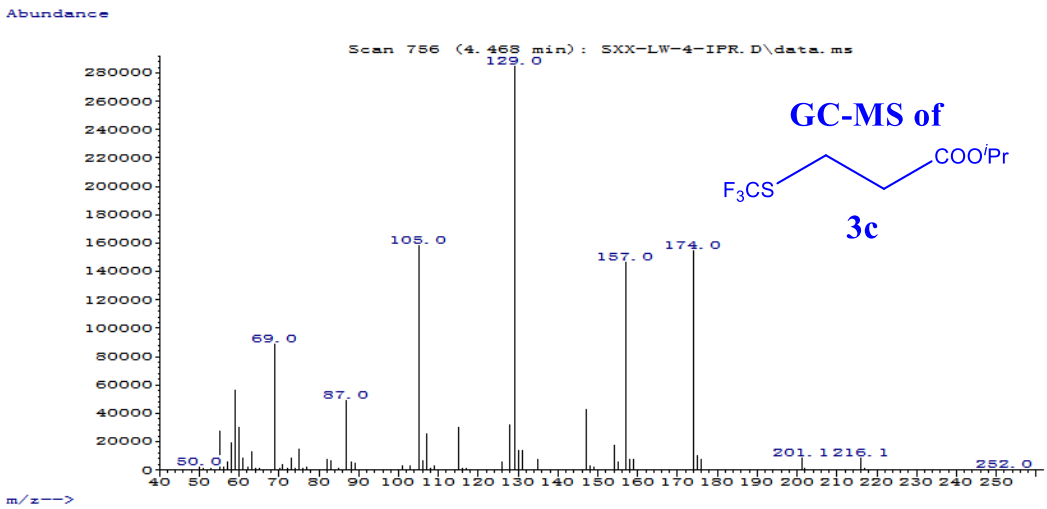


An oven-dried 100-mL round-bottom flask, equipped with a stir bar, was charged with sodium 4-methylbenzenesulfonothioate (3.15 g, 15.0 mmol, 1.00 equiv), isopropyl 3-bromopropanoate (2.93 g, 15.0 mmol, 1.00 equiv) and anhydrous DMF (15.0 mL) was added under N_2 atmosphere. The mixture was allowed to stir at room temperature for 12 h, then was poured into brine (40.0 mL) and extracted with EtOAc (20.0 mL × 3), the combined organic layers were extracted with brine (40.0 mL × 5). The combined organic layers were dried over by Na_2SO_4 then filtered. The solvent was removed by rotary evaporation, and the residue was purified by flash silica gel chromatography (Eluent: 100:1 to 5:1 petroleum ether: ethyl acetate) to give the desired product **2c** as a colorless oil (2.76 g, 9.14 mmol, 60.9% yield). **IR (thin film)** 2992 (w), 1724 (m), 1374 (m), 1324 (m), 1137 (s), 1104 (s), 1076 (s), 813 (m), 703 (m), 652 (s) cm^{-1}; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.81 (2H, d, J = 8.2 Hz), 7.35 (2H, d, J = 8.0 Hz), 4.98 (1H, hept, J = 6.3 Hz), 3.17 (2H, t, J = 7.0 Hz), 2.67 (2H, t, J = 7.0 Hz), 2.45 (3H, s), 1.21 (6H, d, J = 6.3 Hz); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 170.4, 144.9, 141.6, 129.9, 127.0, 68.5, 34.2, 30.6, 21.7, 21.6; **HRMS** (ESI^+) $[M+H]^+$ calc'd for $C_{13}H_{19}O_4S_2$: 303.0719, found: 303.0721.

An oven-dried 100-mL round-bottom flask, equipped with a stir bar, was charged with **2c** (2.80 g, 9.00 mmol, 1.00 equiv), NaOAc (2.21 g, 27.0 mmol, 3.00 equiv) and anhydrous DMSO (9.00 mL) and DCM (9.00 mL) was added under N_2 atmosphere.

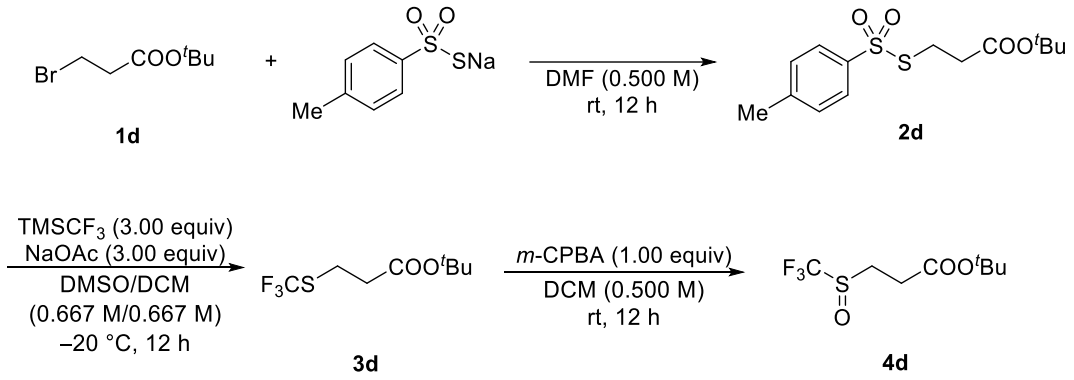
The mixture was cooled to –20 °C. Then $TMSCF_3$ (3.84 g, 27.0 mmol, 3.00 equiv) was added dropwise to the mixture and the mixture was allowed to stir at –20 °C for 12 h. The mixture was poured into brine (50.0 mL) and extracted with DCM (30.0 mL × 3), the combined organic layers were washed by brine (40.0 mL × 5). The combined organic layers were dried over by Na_2SO_4 then filtered. The solvent was removed by rotary evaporation and the residue was used to next step directly without purification. The compound **3c** was detected and confirmed by GC-MS.





An oven-dried 100-mL round-bottom flask, equipped with a stir bar, was charged with isopropyl 3-((trifluoromethyl)thio)propanoate (9.00 mmol, 1.00 equiv), *m*-CPBA (85.0 wt%, 1.83 g, 9.00 mmol, 1.00 equiv) and anhydrous DCM (20.0 mL) was added under N_2 atmosphere. The mixture was stirred at room temperature for 12 h. The mixture was filtered to remove most of solids. Then the mixture was diluted with

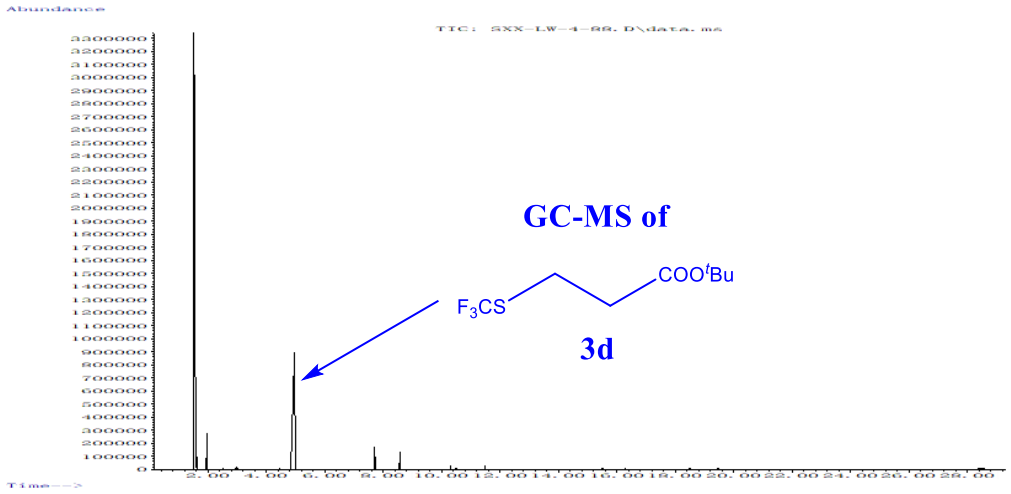
20.0 mL saturated Na_2CO_3 solution and extracted with DCM (20.0 mL × 3), the combined organic layers were washed by brine (20.0 mL × 3). The combined organic layers were dried over by Na_2SO_4 then filtered. The solvent was removed by rotary evaporation and purified by flash silica gel chromatography (Eluent: 100:1 to 5:1 petroleum ether: ethyl acetate) to give the desired product **4c** as a colorless oil (1.11 g, 4.79 mmol, 53.2% yield). **IR (thin film)** 2989 (w), 1728 (s), 1376 (s), 1324 (m), 1174 (s), 1141 (s), 1106 (s), 1077 (s) cm^{-1}; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 5.06 (1H, hept, J = 6.2 Hz), 3.33 – 3.19 (2H, m), 2.98 – 2.75 (2H, m), 1.26 (6H, d, J = 6.3 Hz); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 169.9, 125.5 (q, $J_{C\text{-}F}$ = 335.0 Hz), 69.5, 43.5, 26.2, 21.8; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –73.6; **HRMS** (ESI^+) $[M+Na]^+$ calc'd for $C_7H_{11}O_3SF_3Na$: 255.0273, found: 255.0273.

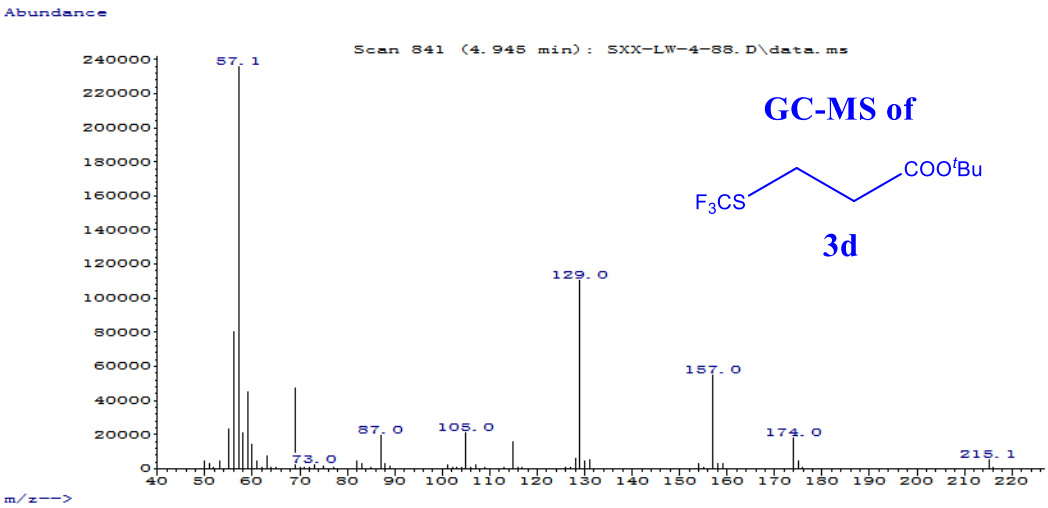


An oven-dried 100-mL round-bottom flask, equipped with a stir bar, was charged with sodium 4-methylbenzenesulfonothioate (4.20 g, 20.0 mmol, 1.00 equiv), *tert*-butyl 3-bromopropanoate (4.18 g, 20.0 mmol, 1.00 equiv) and anhydrous DMF (40.0 mL) was added under N_2 atmosphere. The mixture was allowed to stir at room temperature for 12 h. The mixture was poured into brine (40.0 mL) and extracted with EtOAc (20.0 mL × 3), the combined organic layers were extracted with brine (40.0 mL × 5). The combined organic layers were dried over by Na_2SO_4 then filtered. The solvent was removed by rotary evaporation, and the residue was purified by flash silica gel chromatography (Eluent: 100:1 to 5:1 petroleum ether: ethyl acetate) to give the desired product **2d** as a colorless oil (4.30 g, 13.6 mmol, 67.9% yield). **IR (thin film)** 2893 (w), 1724 (s), 1366 (m), 1324 (s), 1234 (m), 1137 (s), 1077 (s), 813 (m), 703 (m), 652 (s) cm^{-1}; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.81 (2H, d, J = 8.3 Hz), 7.34 (2H, d, J =

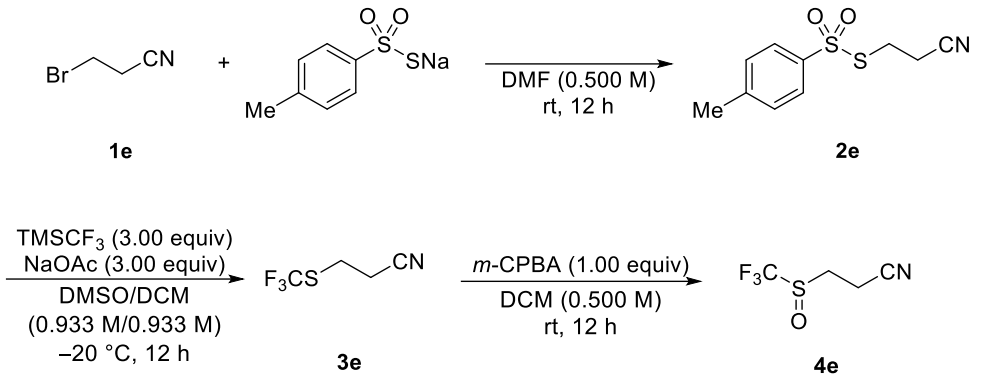
8.1 Hz), 3.13 (2H, t, J = 6.9 Hz), 2.61 (2H, t, J = 6.9 Hz), 2.45 (3H, s), 1.41 (9H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 170.3, 145.0, 141.9, 130.0, 127.2, 81.7, 35.2, 30.9, 28.1, 21.8; **HRMS** (ESI^+) $[M+H]^+$ calc'd for $C_{14}H_{21}O_4S_2$: 317.0876, found: 317.0876.

An oven-dried 100-mL round-bottom flask, equipped with a stir bar, was charged with **2d** (3.16 g, 10.0 mmol, 1.00 equiv), NaOAc (2.46 g, 30.0 mmol, 3.00 equiv) and anhydrous DMSO (15.00 mL) and DCM (15.00 mL) was added under N_2 atmosphere. The mixture was cooled to –20 °C. Then the $TMSCF_3$ (4.27 g, 30.0 mmol, 3.00 equiv) was added dropwise to the mixture and the mixture was allowed to stir at –20 °C for 12 h. The mixture was poured into brine (50.0 mL) and extracted with DCM (30.0 mL × 3). The combined organic layers were washed by brine (40.0 mL × 5) and dried over by Na_2SO_4 then filtered. The solvent was removed by rotary evaporation and the residue was used to next step directly without purification. The compound **3d** was detected and confirmed by GC-MS.





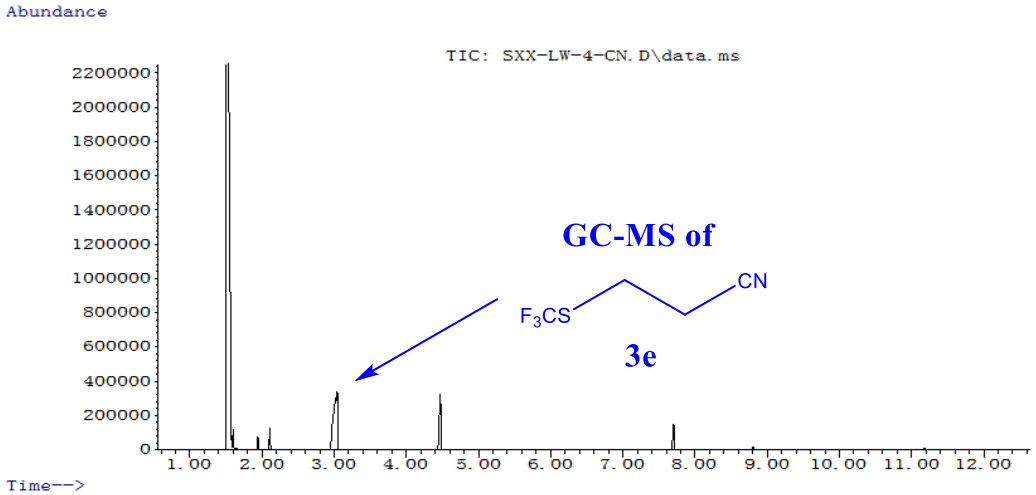
An oven-dried 100-mL round-bottom flask, equipped with a stir bar, was charged with *tert*-butyl 3-((trifluoromethyl)thio)propanoate (10.0 mmol, 1.00 equiv), *m*-CPBA (85.0 wt%, 2.03 g, 10.0 mmol, 1.00 equiv) and anhydrous DCM (20.0 mL) was added under N_2 atmosphere and the mixture was stirred at room temperature for 12 h. The mixture was filtered to remove most of solids. Then the mixture was diluted with 20.0 mL saturated Na_2CO_3 solution and extracted with DCM (20.0 mL × 3), the combined organic layers were washed by brine (20.0 mL × 3). The combined organic layers were dried over by Na_2SO_4 then filtered. The solvent was removed by rotary evaporation and purified by flash silica gel chromatography (Eluent: 100:1 to 10:1 petroleum ether: ethyl acetate) to give the desired product **4d** as a colorless oil (1.62 g, 6.59 mmol, 65.9% yield). **IR (thin film)** 2974 (w), 1727 (s), 1368 (m), 1137 (s), 1078 (s), 840 (m) cm^{-1}; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.28 – 3.13 (2H, m), 2.93 – 2.71 (2H, m), 1.47 (9H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 169.6, 125.5 (q, $J_{C\text{-}F}$ = 335.0 Hz), 82.4, 43.7, 28.1, 27.0; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –73.7; **HRMS** (ESI^+) $[M+H]^+$ calc'd for $C_8H_{14}O_3F_3S$: 247.0610, found: 247.0601.

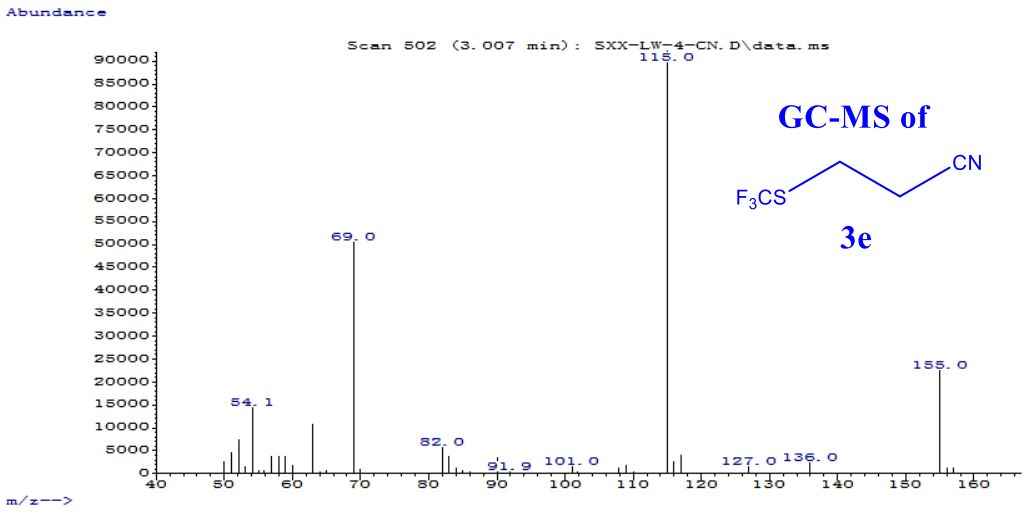


An oven-dried 100-mL round-bottom flask, equipped with a stir bar, was charged with sodium 4-methylbenzenesulfonothioate (4.20 g, 20.0 mmol, 1.00 equiv), 3-bromopropanenitrile (2.68 g, 20.0 mmol, 1.00 equiv) and anhydrous DMF (40.0 mL) was added under N_2 atmosphere and the mixture was allowed to stir at room temperature for 12 h. The mixture was poured into brine (40.0 mL) and extracted with EtOAc (20.0 mL × 3), the combined organic layers were extracted with brine (40.0 mL × 5). The combined organic layers were dried over by Na_2SO_4 then filtered. The solvent was removed by rotary evaporation and the residue was purified by flash silica gel chromatography (Eluent: 100:1 to 5:1 petroleum ether: ethyl acetate) to give the desired product **2e** as a colorless oil (2.04 g, 8.45 mmol, 42.2% yield). **IR (thin film)** 2995 (w), 1729 (s), 1376 (s), 1174 (s), 1140 (s), 1137 (s), 1106 (s), 1077 (s) cm^{-1}; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.82 (2H, d, J = 8.1 Hz), 7.38 (2H, d, J = 8.1 Hz), 3.19 (2H, t, J = 7.2 Hz), 2.81 (2H, t, J = 7.2 Hz), 2.47 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 145.8, 141.1, 130.3, 127.2, 117.3, 31.0, 21.8, 18.7; **HRMS** (ESI^+) $[M+H]^+$ calc'd for $C_{10}H_{12}O_2S_2$: 242.0304, found: 242.0304.

An oven-dried 100-mL round-bottom flask, equipped with a stir bar, was charged with **2e** (2.03 g, 8.40 mmol, 1.00 equiv), NaOAc (2.07 g, 25.2 mmol, 3.00 equiv) and anhydrous DMSO (9.00 mL) and DCM (9.00 mL) was added under N_2 atmosphere and the mixture was cooled to –20 °C. Then the $TMSCF_3$ (3.58 g, 25.2 mmol, 3.00 equiv) was added dropwise to the mixture and the mixture was allowed to stir at –20 °C for 12 h. The mixture was poured into brine (50.0 mL) and extracted with DCM (30.0 mL × 3). The combined organic layers were washed by brine (40.0 mL × 5). The combined organic layers were dried over by Na_2SO_4 then filtered. The solvent was removed by

rotary evaporation and the residue was used to next step directly without purification. The compound **3e** was detected and confirmed by GC-MS.

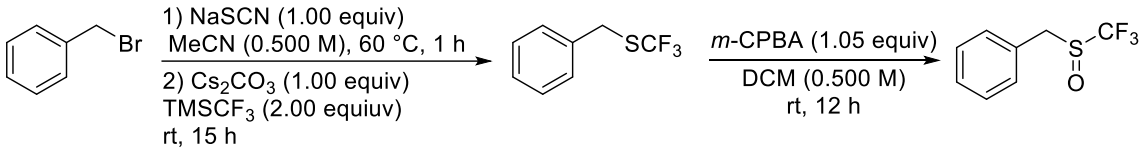




An oven-dried 100-mL round-bottom flask, equipped with a stir bar, was charged with 3-((trifluoromethyl)thio)propanenitrile (7.50 mmol, 1.00 equiv), *m*-CPBA (85.0 wt%, 1.52 g, 7.50 mmol, 1.00 equiv) and anhydrous DCM (20.0 mL) was added under N_2 atmosphere and the mixture was stirred at room temperature for 12 h. The mixture was filtered to remove most of solids. Then mixture was diluted with 20.0 mL saturated Na_2CO_3 solution and extracted with DCM (20.0 mL × 3), the combined organic layers were washed by brine (20.0 mL × 3). The combined organic layers were dried over by Na_2SO_4 then filtered. The solvent was removed by rotary evaporation and the residue purified by flash silica gel chromatography (Eluent: 100:1 to 5:1 petroleum ether: ethyl acetate) to give the desired product **4e** as a colorless oil (0.470 g, 2.75 mmol,

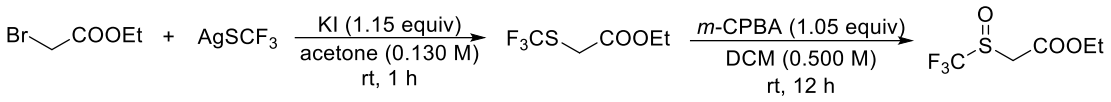
36.7% yield). **IR (thin film)** 1177 (s), 1138 (s), 1072 (s) cm^{-1}; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 3.26 – 3.23 (2H, m), 3.06 – 2.86 (2H, m); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 125.1 (q, $J_{C\text{-}F}$ = 335.7 Hz), 116.26, 43.45, 10.45; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –72.5; **HRMS** (EI) calc'd for $C_4H_4OF_3SN$: 170.9956, found: 170.9960.

2. Synthesis of (((trifluoromethyl)sulfinyl)methyl)benzene 4f:



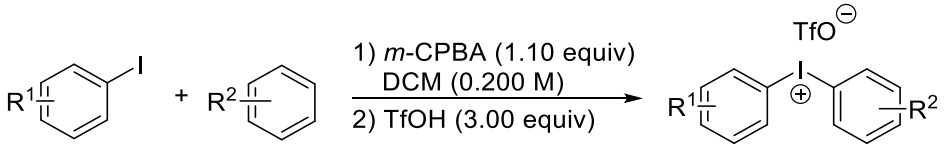
An oven-dried 100 mL round-bottom flask, equipped with a stir bar, was charged with NaSCN (1.62 g, 20.0 mmol, 1.00 equiv). After exchanging the atmosphere three times with N_2, MeCN (40.0 mL) and benzyl bromide (3.42 g, 20.0 mmol, 1.00 equiv) were added to flask. The reaction mixture was allowed to heat at 60 ºC for 1 h. Then the reaction mixture was cooled to room temperature, Cs_2CO_3 (6.52 g, 20.0 mmol, 1.00 equiv) and $TMSCF_3$ (5.69 g, 40.0 mmol, 2.00 equiv) were added to the mixture. The reaction mixture was allowed to stir at room temperature for 15 h. When the reaction completed, the mixture was extracted with Et_2O (50.0 ml ×3), the combined organic layers were washed by water (80.0 mL × 2) and brine (100 mL × 1). The organic layer was dried over by Na_2SO_4 then filtered. The solvent was removed by rotary evaporation and the residue purified by flash silica gel chromatography (Eluent: 200:1 to 50:1 petroleum ether: ethyl ether) to give the corresponding sulfide as a colorless oil. The sulfide was dissolved in DCM (22.0 mL) followed by addition of *m*-CPBA (85.0 wt%, 2.34 g, 11.6 mmol, 1.05 equiv). The reaction mixture was allowed to stir at room temperature for 12 h. The reaction was then quenched with saturated aqueous Na_2CO_3 and extracted with DCM (50.0 mL × 3). The combined organic layers were dried with Na_2SO_4 and the solvent was removed by rotary evaporation. The residue was purified by column chromatography (Eluent: 100:1 to 5:1 petroleum ether: ethyl acetate) to give the desired product as a white solid (1.52 g, 7.31 mmol, 36.6% yield). The spectral data match those previously reported [4].

3. Synthesis of ethyl 2-((trifluoromethyl)sulfinyl)acetate 4g:



An oven-dried 200 mL round-bottom flask was equipped with a stir bar, was charged with $AgSCF_3$ (1.89 g, 9.00 mmol, 1.20 equiv) and KI (1.43 g, 8.63 mmol, 1.15 equiv). After exchanging the atmosphere three times with N_2, acetone (56.0 mL) was added to it. The mixture was stirred at room temperature until the solid completely dissolved. The ethyl bromoacetate (1.25 g, 7.50 mmol, 1.00 equiv) was added to the mixture in one portion and the reaction was allowed to stir at room temperature for 1 h. The reaction mixture was removed by rotary evaporation and the residue was treated with Et_2O (50.0 mL). The precipitate was removed by filtration and the organic layer was concentrated to give the corresponding sulfide as a red oil. An oven-dried 100 mL round-bottom flask was equipped with a stir bar, was charged with *m*-CPBA (85 wt%, 1.60 g, 1.05 equiv). After exchanging the atmosphere three times with N_2, DCM (15.0 mL) was added to flask. When the solid completely dissolved, the red oil was added to the mixture. The reaction was allowed to stir at room temperature for 12 h. The reaction was then quenched with saturated aqueous Na_2CO_3 and extracted with DCM (50.0 mL × 3). The combined organic layers were dried with Na_2SO_4 and the solvent was removed by rotary evaporation. The residue was purified by column chromatography (Eluent: 100:1 to 10:1 petroleum ether: ethyl acetate) to give the desired product as a yellow liquid (0.909 g, 4.45 mmol, 59.3% yield). The spectral data match those previously reported [4].

4. <u>General Method A:</u> Synthesis of diaryliodonium salts 5a-c, 5j:

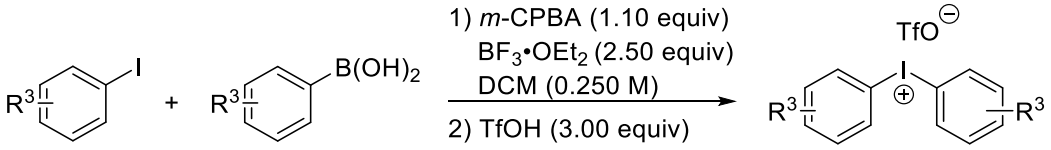


An oven-dried 200-mL round-bottom flask, equipped with a stir bar, was charged with *m*-CPBA (85.0 wt%, 0.670 g, 3.30 mmol, 1.10 equiv) and aryl(hetero) iodides (3.00 mmol, 1.00 equiv) were dissolved in dry DCM (15.0 mL) under N_2 atmosphere.

Then the corresponding arene (3.30 mmol, 1.10 equiv) was added to the mixture and the solution was cooled to 0 °C followed by dropwise addition of triflic acid (1.35 g, 9.00 mmol, 3.00 equiv) and further stirred for 10 minutes. Then the mixture was allowed to stir at room temperature for 30 minutes. Concentrated the mixture under reduced pressure. Et_2O (100 mL) was added to the residue and the mixture was stirred at room temperature for 30 minutes to precipitate an off-white solid. The flask was stored in a freezer for 1 h, after that time the solid was filtered off, washed with cold Et_2O and dried under vacuum[5].

Compound **5a**: white solid, 1.08 g, 2.51 mmol, 80.8% yield[5]; compound **5b**: white solid, 1.34 g, 2.69 mmol, 75.9% yield[6]; compound **5c**: white solid, 0.816 g, 1.39 mmol, 54.5% yield[6]; compound **5j**: white solid, 0.995 g, 2.24 mmol, 74.6% yield[6]. The spectral data match those previously reported.

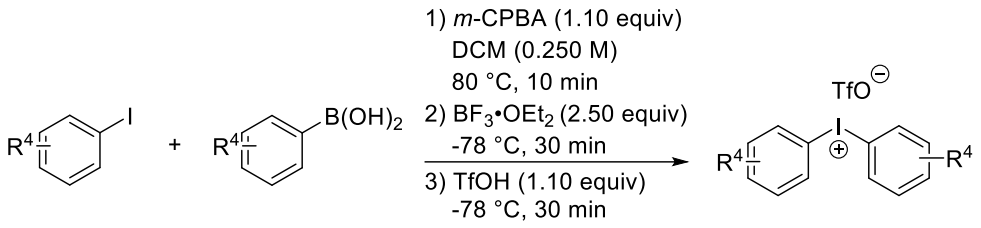
5. General Method B: Synthesis of diaryliodonium salts 5e, 5f, 5h:



An oven-dried 200-mL round-bottom flask, equipped with a stir bar, was charged with *m*-CPBA (85.0 wt%, 0.690 g, 3.30 mmol, 1.10 equiv), aryl(hetero) iodides (3.00 mmol, 1.00 equiv) and DCM (15.0 mL) was added under N_2 atmosphere. The $BF_3 \cdot OEt_2$ (1.06 g, 7.50 mmol, 2.50 equiv) was added to the mixture at room temperature and the solution was allowed to stir at room temperature for 1 h. Then the corresponding boronic acid (3.30 mmol, 1.10 equiv) was added to the mixture at 0 °C and stirred for 15 minutes. Then the mixture was allowed to stirred at room temperature for 1 h. The triflic acid (0.495 g, 3.30 mmol, 1.10 equiv) was added dropwise to the mixture at 0 °C and stirred for 15 minutes, then stirred at room temperature for 30 minutes. Concentrate the mixture under reduced pressure. Et_2O (50.0 mL) was added and the mixture was stirred at room temperature for 30 minutes to precipitate an off-white solid. The flask was stored in a freezer for 1 h, after that time the solid was filtered off, washed with cold Et_2O and dried under vacuum [7].

Compound **5e**: white solid, 0.906 g, 1.98 mmol, 65.9% yield[7]; compound **5f** (prepared on 5.00 mmol scale): white solid, 1.63 g, 3.27 mmol, 65.4% yield[8]; compound **5h** (prepared on 5.00 mmol scale): white solid, 0.997 g, 3.96 mmol, 79.1% yield[9]. The spectral data match those previously reported.

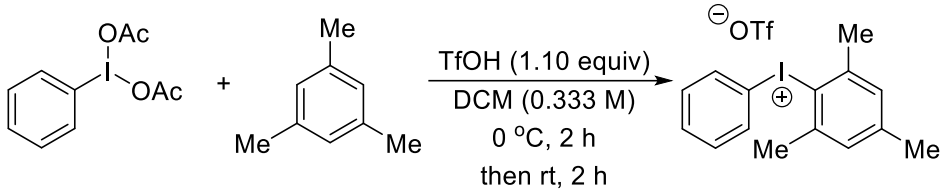
6. General Method C: Synthesis of diaryliodonium salts 5d, 5g:



An oven-dried 200-mL round-bottom flask, equipped with a stir bar, was charged with aryl(hetero) iodides (5.00 mmol, 1.00 equiv), 3-chloroperbenzoic acid (85.0 wt%, 1.12 g, 5.50 mmol, 1.10 equiv) and anhydrous DCM (20.0 mL) was added under N_2 atmosphere. The mixture was allowed to stir at 80 ºC for 10 min. Then the mixture was cooled to –78 ºC. The corresponding boronic acid (0.836 g, 5.50 mmol, 1.10 equiv) and BF_3•OEt_2 (1.78 g, 12.5 mmol, 2.50 equiv) were added to DCM (20.0 mL) and was dropwise to the mixture. The mixture was stirred for 30 min at –78 ºC then warmed to room temperature. The triflic acid (0.826 g, 5.50 mmol, 1.10 equiv) was added dropwise to the mixture and the mixture was stirred for 30 min. The solvent was removed by rotary evaporation. Et_2O (100 mL) was added to the residue and the mixture was stirred at room temperature for 30 minutes to precipitate a grey solid. The precipitate was filtered and washed with Et_2O to give the desired product.

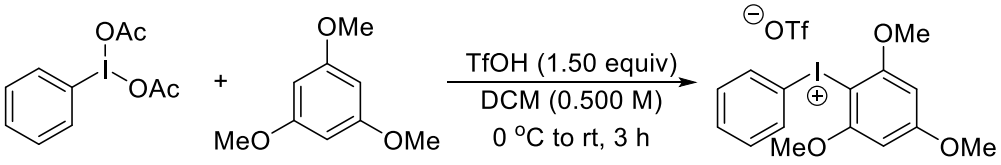
Compound **5d**: grey solid**,** 0.973 g, 0.198 mmol, 39.7% yield[10]; compound **5g**: white solid, 1.45 g, 2.96 mmol, 59.2% yield[11]; **M.p.** 148.7 –149.5 ºC; **IR (thin film)** 3242 (w), 1476 (m), 1278 (s), 1239 (s), 1163 (s), 1024 (s), 768 (s), 751 (s), 633 (s) cm^{-1}; **^{1}H NMR** (400 MHz, DMSO-d_6) δ 8.16 (2H, d, J = 6.5 Hz), 7.61 (2H, dd, J = 7.1, 8.6 Hz), 7.27 (2H, d, J = 9.2 Hz), 7.04 (2H, dd, J = 7.9, 7.4 Hz), 3.92 (6H, s); **^{13}C NMR** (126 MHz, DMSO-d_6) δ 156.6, 137.3, 134.6, 123.2, 112.9, 105.9, 57.0; **^{19}F NMR** (471 MHz, DMSO-d_6) δ –77.8. Spectra were consistent with literature data.

7. Synthesis of diaryliodonium salt 5i:



An oven-dried 100-mL round-bottom flask, equipped with a stir bar, was charged with phenyliodine(III) diacetate (1.61 g, 5.00 mmol, 1.00 equiv), mesitylene (0.661 g, 5.50 mmol, 1.10 equiv) and DCM (15.0 mL) was added under N_2 atmosphere. The triflic acid (0.825 g, 5.50 mmol, 1.10 equiv) was slowly added to the mixture at 0 °C and the mixture was allowed to stir at 0 °C for 2 h, then warmed up to room temperature and it was stirred for another 2 hours. The solvent was removed by rotary evaporation. Et_2O (100 mL) was added to the residue and the mixture was stirred at room temperature for 30 minutes to precipitate an grey solid. The precipitate was filtered and washed with Et_2O to give the desired compound **5i** (2.23 g, 4.72 mmol, 94.4% yield) as a white solid. The spectral data match those previously reported[12].

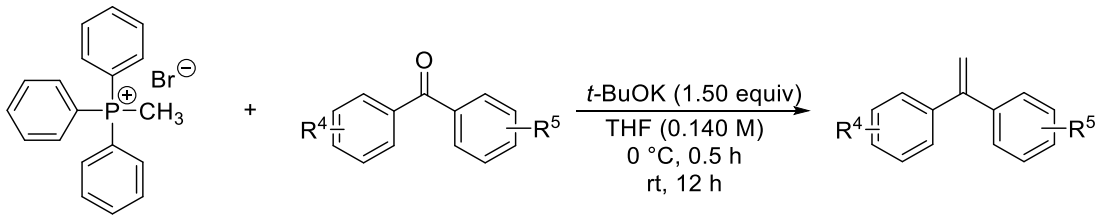
8. Synthesis of diaryliodonium salt 5k:



An oven-dried 200-mL round-bottom flask, equipped with a stir bar, was charged with phenyliodine(III) diacetate (1.61 g, 5.00 mmol, 1.00 equiv), 1,3,5-trimethoxybenzene (0.925 g, 5.50 mmol, 1.10 equiv) and DCM (10.0 mL) was added under N_2 atmosphere. The triflic acid (1.13 g, 7.50 mmol, 1.50 equiv) was dissolved in water (10.0 mL) and slowly added to the mixture at 0 °C. The solution was warmed up to room temperature and it was allowed to stir for 3 hours. The resulting mixture was extracted with DCM (40.0 mL × 5), and the combined organic layer were dried over by Na_2SO_4 then filtered. The solvent was removed by rotary evaporation. Et_2O (80.0 mL) was added to the residue and the mixture was stirred at room temperature for 30 minutes to precipitate an off-white solid. The precipitate was filtered and washed with Et_2O to

give the desired **5k** (1.95 g, 3.75 mmol, 74.9% yield) as a white solid. The spectral data match those previously reported[13].

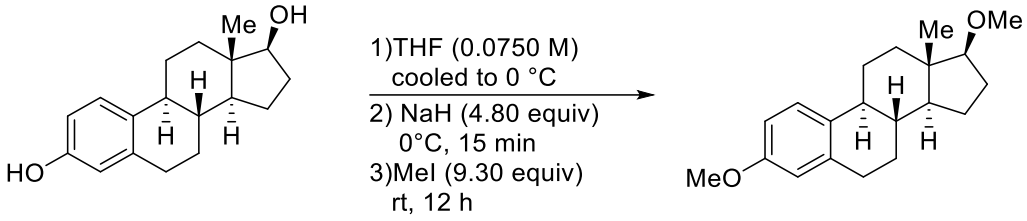
9. General Method D: Synthesis of alkenes 7b-7i:



An oven-dried 100-mL two-necked round-bottom flask, equipped with a stir bar, was charged with methyltriphenylphosphonium bromide (2.68 g, 7.50 mmol, 1.50 equiv) in THF (20.0 mL), *t*-BuOK (0.842 g, 7.50 mmol, 1.50 equiv) was added to the mixture under N_2 atmosphere at 0 ºC and the mixture was allowed to stir for 30 minutes. A solution of corresponding methanone (5.00 mmol, 1.00 equiv) in THF (15.0 mL) was added dropwise to the mixture at 0 ºC and the mixture was allowed to stir at room temperature for 12 h. The mixture was quenched by water (50.0 mL) then extracted with EtOAc (30.0 mL ×3). The combined organic layers were dried over by Na_2SO_4 then filtered. The solvent was removed by rotary evaporation and purified by flash silica gel chromatography (Eluent: petroleum ether: ethyl acetate) to give desired products.

Compound **7b** : colorless liquid, 0.955 g, 4.58 mmol, 91.7% yield[14]; compound **7c**: colorless liquid, 0.762 g, 3.06 mmol, 61.2% yield [15]; compound **7d**: colorless liquid, 1.05 g, 3.10 mmol, 62.1% yield[15]; compound **7e**: colorless liquid, 0.983 g, 4.54 mmol, 90.9% yield[15]; compound **7f**: colorless liquid, 1.06 g, 4.41 mmol, 88.2% yield[16]; compound **7g**: colorless liquid, 0.919 g, 4.28 mmol, 85.6% yield[17]; compound **7h**: colorless liquid, 1.03 g, 4.90 mmol, 98.0% yield[17]; compound **7i**: white solid, 1.116 g, 4.35 mmol, 87.1% yield[18]; compound **7j**: colorless liquid, 1.02 g, 4.75 mmol, 95.0% yield[18]; compound **7k**: colorless liquid, 0.884 g, 4.55 mmol, 91.0% yield[17]; compound **7l**: colorless liquid, 1.00 g, 4.80 mmol, 96.0% yield[19]. The spectral data match those previously reported.

10. Synthesis of Estra-1,3,5(10)-triene 11:



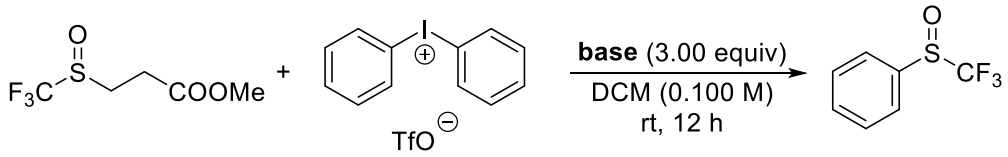
11

An oven-dried 100-mL two-necked round-bottom flask, equipped with a stir bar, was charged with β-Estradiol (0.545 g, 2.00 mmol, 1.00 equiv) in dry THF (26.8 mL) was added under under N_2 atmosphere, the mixture was cooled to 0 ºC. NaH (60.0 wt%, 0.384 g, 9.60 mmol, 4.80 equiv) was added to the mixture. The reaction mixture was allowed to stir at 0 ºC for 15 min. The MeI (2.64 g, 18.6 mmol, 9.30 equiv) was added to the mixture, and the mixture was allowed to stir at room temperature for 12 h. The ice-water was poured carefully into the mixture. Until the mixture was no bubbles coming out, the mixture was extracted with EtOAc (50.0 mL × 3), the combined organic layers were washed with aqueous $NaHCO_3$ (50.0 mL × 3). The combined organic layers were dried over by $NaSO_4$ then filtered. The solvent was removed by rotary evaporation to afford Estra-1,3,5(10)-triene as a white solid, 0.493 g, 1.64 mmol, 82.0% yield[20]. The spectral data match the previously reported.

III. Optimization of the reaction conditions

1. Optimization of the reaction between 5a with reagent 4a

Table S1. Evaluation of different bases

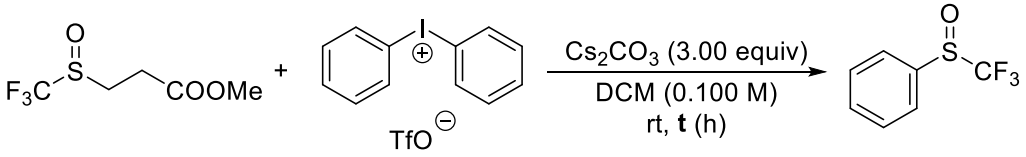


4a **5a** **6a**

entry	**base**	^{19}F NMR yield of **6a** (%)
1	KOH	36
2	*t*-BuOLi	<5
3	Cs_2CO_3	73
4	K_2CO_3	9
5	NaH	50
6	DBU	<5
7	*t*-BuOK	<5
8	$LiOH•H_2O$	6
9	Et_3N	15
10	Na_2CO_3	<5

Reaction condition: **4a** (0.100 mmol), **5a** (0.110 mmol), **base** (3.00 equiv), DCM (1.00 mL) at rt for 12 h. Yield was determined by ^{19}F NMR spectroscopy in the presence of $PhCF_3$ (99.0 wt%,14.8 mg, 0.100 mmol) as an internal standard.

Table S2. Evaluation of different time

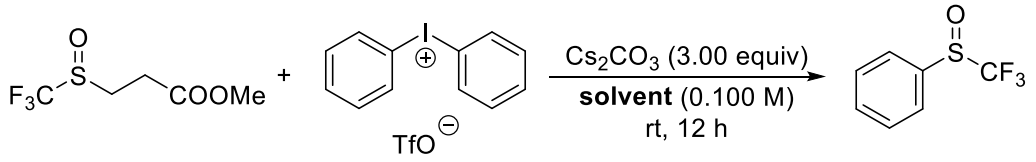


4a **5a** **6a**

entry	**t** (h)	^{19}F NMR yield of **6a** (%)
1	12	84
2	6	73
3	4	69
4	2	63

Reaction condition: **4a** (0.100 mmol), **5a** (0.110 mmol), Cs_2CO_3 (3.00 equiv), DCM (1.00 mL) stirred at rt for **t** h. Yield was determined by ^{19}F NMR spectroscopy in the presence of $PhCF_3$ (99.0 wt%,14.8 mg, 0.100 mmol) as an internal standard.

Table S3. Evaluation of different solvents

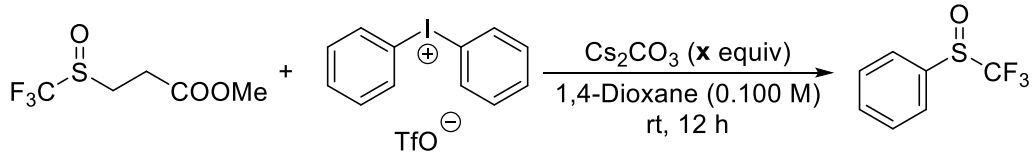


4a **5a** **6a**

entry	**solvent**	^{19}F NMR yield of **6a** (%)
1	Toluene	51
2	1,4-Dioxane	85
3	MeCN	69
4	2-MeTHF	55
5	DCE	76
6	Diglyme	75
7	CCl_3H	80
8	THF	72
9	DMSO	24
10	DMF	36
11	DME	68
12	EA	69
13	PhCl	80
14	Pyridine	32
15	CH_3OH	64

Reaction condition: **4a** (0.100 mmol), **5a** (0.110 mmol), Cs_2CO_3 (3.00 equiv), **solvent** (1.00 mL) at rt for 12 h. Yield was determined by ^{19}F NMR spectroscopy in the presence of $PhCF_3$ (99.0 wt%,14.8 mg, 0.100 mmol) as an internal standard.

Table S4. Evaluation of different amounts of bases

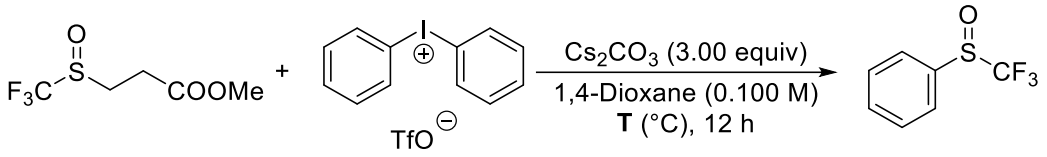


4a **5a** **6a**

entry	**x**	^{19}F NMR yield of **6a** (%)
1	4.00	85
2	3.00	84
3	2.00	80
4	1.50	76
5	1.00	36

Reaction condition: **4a** (0.100 mmol), **5a** (0.110 mmol), Cs_2CO_3 (**x** equiv), 1,4-Dioxane (1.00 mL) at rt for 12 h. Yield was determined by ^{19}F NMR spectroscopy in the presence of $PhCF_3$ (99.0 wt%,14.8 mg, 0.100 mmol) as an internal standard.

Table S5. Evaluation of different temperature

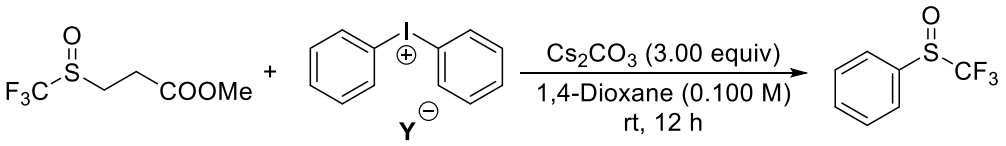


4a **5a** **6a**

entry	**T** (°C)	^{19}F NMR yield of **6a** (%)
1	0	50
2	22 (rt)	84
3	60	62

Reaction condition: **4a** (0.100 mmol), **5a** (0.110 mmol), Cs_2CO_3 (3.00 equiv), 1,4-Dioxane (1.00 mL) at **T** (°C) for 12 h. Yield was determined by ^{19}F NMR spectroscopy in the presence of $PhCF_3$ (99.0 wt%,14.8 mg, 0.100 mmol) as an internal standard.

Table S6. Evaluation of different counteracting anions



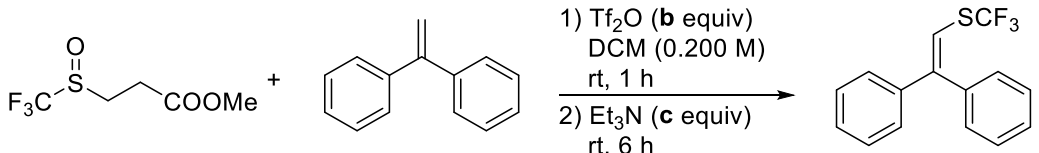
4a **6a**

entry	**Y**	^{19}F NMR yield of **6a** (%)
1	$BF_4^{\ominus}$	74
2	$PF_6^{\ominus}$	86

Reaction condition: **4a** (0.100 mmol), **Y** (0.110 mmol), Cs_2CO_3 (3.00 equiv), 1,4-Dioxane (1.00 mL) at rt for 12 h. Yield was determined by ^{19}F NMR spectroscopy in the presence of $PhCF_3$ (99.0 wt%,14.8 mg, 0.100 mmol) as an internal standard.

2. Optimization of the reaction between 7a with reagent 4a

Table S1. Evaluation of different amount of reagent 4a, Tf_2O and Et_3N



4a
a equiv

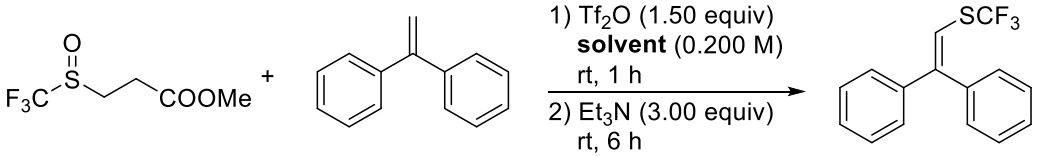
7a
1.00 equiv

8a

entry	**a**	**b**	**c**	^{19}F NMR yield of **8a** (%)
1	1.2	1.5	3.5	78
2	1.5	1.5	3.5	84
3	2.0	2.0	3.5	65
4	1.5	1.5	2.0	68
5	1.5	1.5	2.5	85
6	1.5	1.5	3.0	93
7	1.5	1.5	4.0	80
8	1.5	1.5	4.5	85
9	1.5	1.5	5.0	74

Reaction condition: **4a** (**a** equiv), **7a** (0.200 mmol), DCM (1.00 mL), then Tf_2O (**b** equiv) was added dropwise to the mixture at rt for 1 h. Then the Et_3N (**c** equiv) was added to it under N_2 atmosphere, and the mixture was stirred at rt for 6 h. Yield was determined by ^{19}F NMR spectroscopy in the presence of $PhCF_3$ (99.0 wt%, 14.8 mg, 0.100 mmol) as an internal standard.

Table S2. Evaluation of different solvents

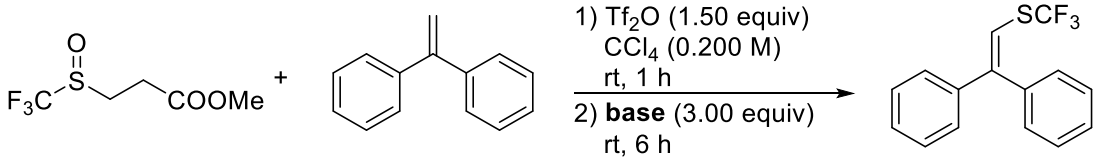


4a **7a** **8a**

entry	**solvent**	^{19}F NMR yield of **8a** (%)
1	EA	64
2	DMF	<5
3	1,4-Dioxane	14
4	MeOH	<5
5	THF	<5
6	$CHCl_3$	65
7	DCE	81
8	MeCN	69
9	DMSO	<5
10	CCl_4	>99

Reaction condition: **4a** (0.300 mmol), **7a** (0.200 mmol), **solvent** (1.00 mL), then Tf_2O (1.50 equiv) was added dropwise to the mixture at rt for 1 h.. Then the Et_3N (3.00 equiv) was added to it under N_2 atmosphere, and stirred at rt for 6 h. Yield was determined by ^{19}F NMR spectroscopy in the presence of $PhCF_3$ (99.0 wt%, 14.8 mg, 0.100 mmol) as an internal standard.

Table S3. Evaluation of different bases

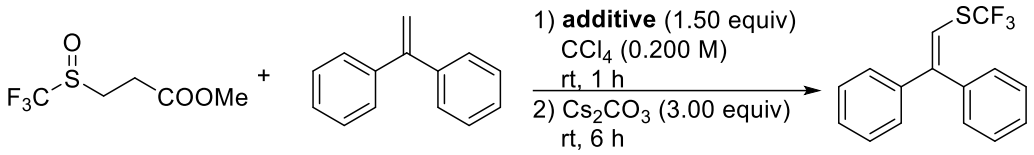


4a **7a** **8a**

entry	**base**	^{19}F NMR yield of **8a** (%)
1	DMAP	86
2	DBU	>99
3	K_2CO_3	24
4	Cs_2CO_3	>99
5	NaOH	6
6	*t*-BuOK	54
7	Pyridine	93
8	DIPEA	90
9	K_3PO_4	87
10	no	<5

Reaction condition: **4a** (0.300 mmol), **7a** (0.200 mmol), CCl_4 (1.00 mL), then Tf_2O (1.50 equiv) was added dropwise to the mixture at rt for 1 h. Then the **base** (3.00 equiv) was added to it under N_2 atmosphere, and stirred at rt for 6 h. Yield was determined by ^{19}F NMR spectroscopy in the presence of $PhCF_3$ (99.0 wt%, 14.8 mg, 0.100 mmol) as an internal standard.

Table S4. Evaluation of different additives



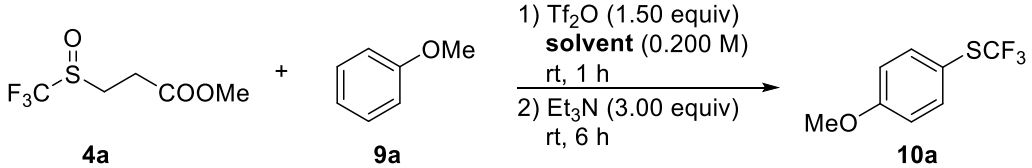
4a **7a** **8a**

entry	**additive**	^{19}F NMR yield of **8a** (%)
1	TFAA	64
2	TfOH	>99

Reaction condition: **4a** (0.300 mmol), **7a** (0.200 mmol), CCl_4 (1.00 mL), dropwise **additive** (1.50 equiv) at rt for 1 h. Then Cs_2CO_3 (3.00 equiv) was added to it under N_2 atmosphere, and stirred at rt for 6 h. Yield was determined by ^{19}F NMR spectroscopy in the presence of $PhCF_3$ (99.0 wt%, 14.8 mg, 0.100 mmol) as an internal standard.

3. Optimization of the reaction between 9a with reagent 4a

Table S1. Evaluation of different solvents

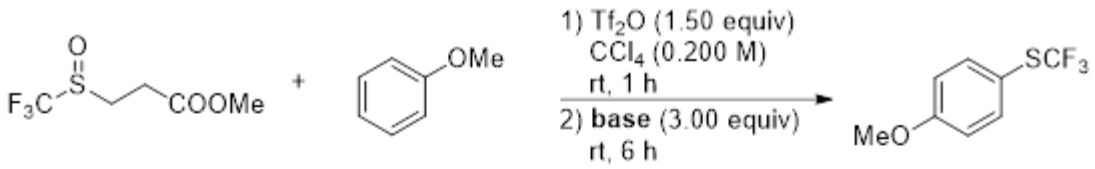


4a **9a** **10a**

entry	**solvent**	^{19}F NMR yield of **10a** (%)
1	DCM	62
2	$CHCl_3$	39
3	DCE	72
4	EA	54
5	MeCN	47
6	1,4-Dioxane	10
7	CCl_4	80

Reaction condition: **4a** (0.300 mmol), **7a** (0.200 mmol), **solvent** (1.00 mL), then Tf_2O (1.50 equiv) was added dropwise to the mixture at rt for 1 h. Then Et_3N (3.00 equiv) was added to it under N_2 atmosphere, and stirred at rt for 6 h. Yield was determined by ^{19}F NMR spectroscopy in the presence of $PhCF_3$ (99.0 wt%, 14.8 mg, 0.100 mmol) as an internal standard.

Table S2. Evaluation of different bases



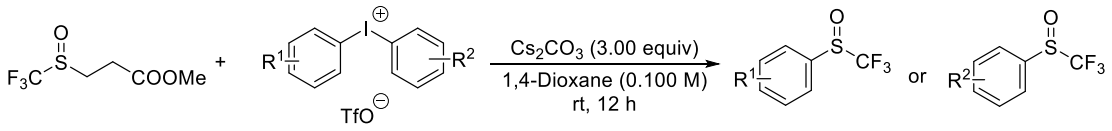
4a **9a** **10a**

entry	base	^{19}F NMR yield of **10a** (%)
1	Cs_2CO_3	72
2	DMAP	64
3	DBU	73
4	*t*-BuOK	52
5	Pyridine	74
6	DIPEA	68
7	K_3PO_4	11

Reaction condition: **4a** (0.300 mmol), **9a** (0.200 mmol), CCl_4 (1.00 mL), then Tf_2O (1.50 equiv) was added dropwise to the mixture at rt for 1 h. Then **base** (3.00 equiv) was added to it under N_2 atmosphere, and stirred at rt for 6 h. Yield was determined by ^{19}F NMR spectroscopy in the presence of $PhCF_3$ (99.0 wt%, 14.8 mg, 0.100 mmol) as an internal standard.

IV. Substrate scope

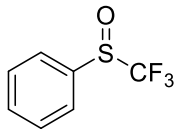
1. General Method E



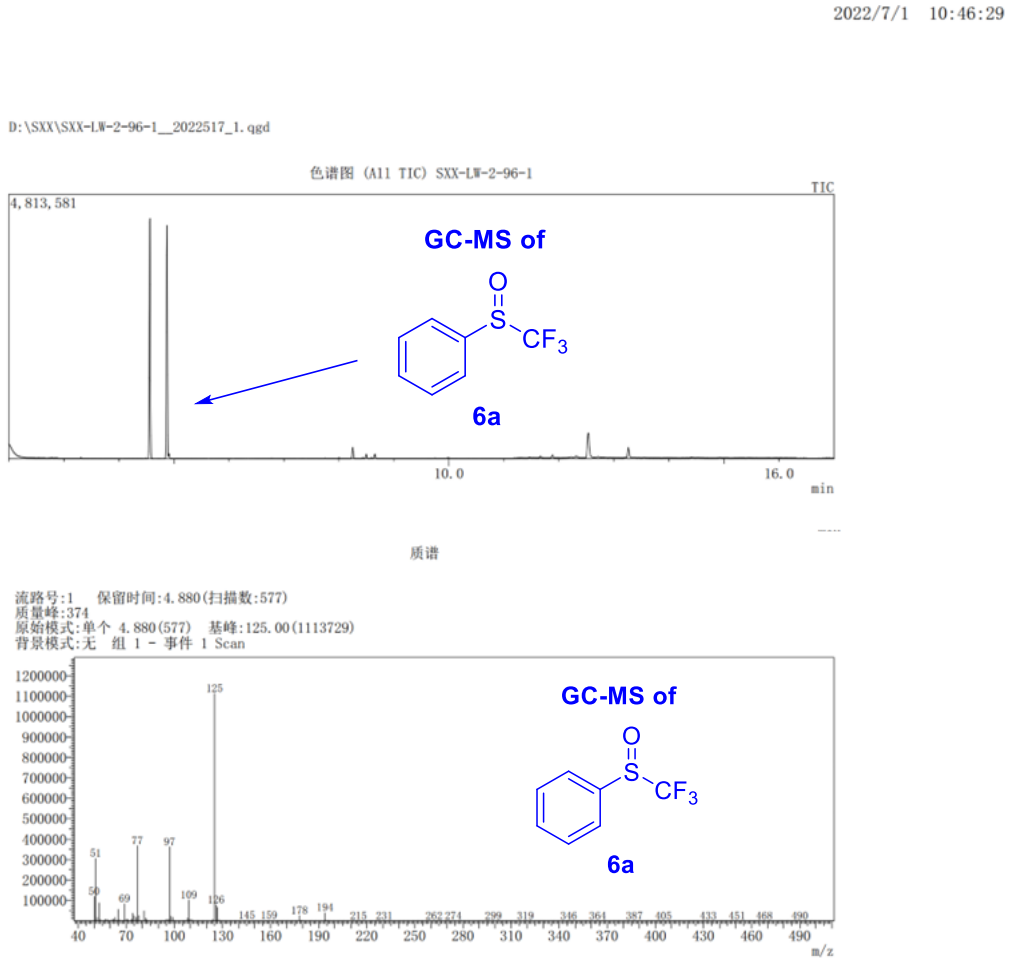
4a **5a-5k** **6a-6k**

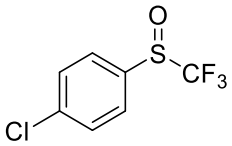
An oven-dried 25-mL glass schlenck, equipped with a stirring bar, was charged with diaryliodonium trifluoromethanesulfonate (0.550 mmol, 1.10 equiv), Cs_2CO_3 (488 mg, 1.50 mmol, 3.00 equiv). The mixture was evacuated and backfilled with nitrogen for three times. Then methyl 3-((trifluoromethyl)sulfinyl)propanoate **4a** (102 mg, 0.500 mmol, 1.00 equiv) and dry 1,4-Dioxane (5.00 mL) was added under N_2 atmosphere and the mixture was allowed to stir at room temperature for 12 h. The mixture was diluted with brine (25.0 mL) and extracted with DCM (15.0 mL × 3). The combined organic layers were washed with brine (30.0 mL × 5), then dried over by

Na_2SO_4 then filtered. The solvent was removed by rotary evaporation. The solvent was removed by rotary evaporation and the residue was purified by flash silica gel chromatography.

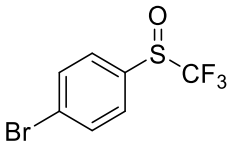


((Trifluoromethyl)sulfinyl)benzene 6a: Prepared according to **General Method E** and $PhCF_3$ (99.0 wt%, 73.1 mg, 0.500 mmol) was added directly to the reaction mixture, ^{19}F NMR yield of compound **6a** was 85.0% yield, and compound **6a** was further confirmed by GC-MS[21].

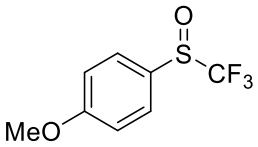




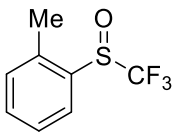
Chloro-4-((trifluoromethyl)sulfinyl)benzene 6b: Prepared according to **General Method E** (Eluent: 120:1 to 50:1 petroleum ether: ethyl acetate) and the title compound was isolated as a colorless liquid (92.6 mg, 0.405 mmol, 81.0% yield)[21]; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.72 (2H, d, J = 8.4 Hz), 7.58 (2H, d, J = 8.6Hz); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 140.3, 134.1, 130.1, 127.3, 124.6 (q, $J_{C\text{-}F}$ = 336.5 Hz); **^{19}F NMR** (471 MHz, $CDCl_3$) δ –74.5.



1-Bromo-4-((trifluoromethyl)sulfinyl)benzene 6c: Prepared according to **General Method E** (Eluent: 120:1 to 50:1 petroleum ether: ethyl acetate) and the title compound was isolated as a white solid (107.7 mg, 0.394 mmol, 78.9% yield)[3]; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.75 (2H, d, J = 8.6 Hz), 7.66 (2H, d, J = 8.3 Hz); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 134.8, 133.1, 128.8, 127.4, 124.5 (q, $J_{C\text{-}F}$ = 336.7 Hz); **^{19}F NMR** (471 MHz, $CDCl_3$) δ –74.4.

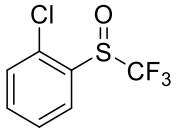


1-Methoxy-4-((trifluoromethyl)sulfinyl)benzene 6d: Prepared according to **General Method E** (Eluent: 120:1 to 30:1 petroleum ether: ethyl acetate) and the title compound was isolated as a yellowish liquid (106.9 mg, 0.477 mmol, 95.4% yield)[21]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.70 (2H, d, J = 8.8 Hz), 7.06 (2H, d, J = 8.9 Hz), 3.85 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 164.1, 128.1, 126.2 (d, $J_{C\text{-}F}$ = 1.26 Hz), 124.7 (q, $J_{C\text{-}F}$ = 336.0 Hz), 115.2, 55.7; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –75.4.

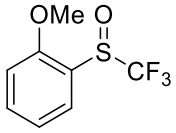


1-Methyl-2-((trifluoromethyl)sulfinyl)benzene 6e: Prepared according to **General**

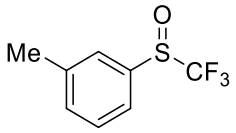
Method E (Eluent: 120:1 to 50:1 petroleum ether: ethyl acetate) and the title compound was isolated as a colorless liquid (80.9 mg, 0.389 mmol, 77.7% yield)[22]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.02 (1H, d, J = 7.7 Hz), 7.54 – 7.45 (2H, m), 7.31 (1H, d, J = 7.4 Hz), 2.47 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.0, 134.2, 133.2, 131.4, 127.3, 125.9, 125.3 (q, $J_{C\text{-}F}$ = 337.1 Hz), 18.3; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –73.6.



1-Chloro-2-((trifluoromethyl)sulfinyl)benzene 6f: Prepared according to **General Method E** (Eluent: 120:1 to 50:1 petroleum ether: ethyl acetate) and the title compound was isolated as a yellow liquid (66.2 mg, 0.290 mmol, 57.9% yield)[22]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.08 – 7.97 (1H, m), 7.60 – 7.55 (2H, m), 7.50 – 7.47 (1H, m); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 134.7, 134.6, 133.3, 130.5, 128.2, 127.9, 125.2 (q, $J_{C\text{-}F}$ = 339.4 Hz); **^{19}F NMR** (471 MHz, $CDCl_3$) δ –72.7.

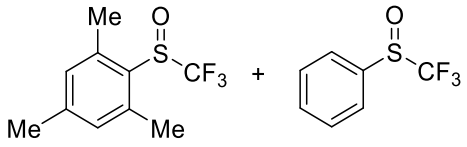


1-Methoxy-2-((trifluoromethyl)sulfinyl)benzene 6g: Prepared according to **General Method E** (Eluent: 120:1 to 50:1 petroleum ether: ethyl acetate) and the title compound was isolated as a yellow liquid (97.2 mg, 0.434 mmol, 84.7% yield); **IR (thin film)** 2874 (w), 2756 (w), 1590 (m), 1479 (s), 1278 (s), 1247 (m), 1173 (s), 1130 (s), 1075 (s), 1049 (s), 1016 (s), 795 (m), 755 (s) cm^{-1}; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.89 (1H, d, J = 9.1 Hz), 7.57 (1H, dd, J = 8.5, 8.9 Hz), 7.19 (1H, dd, J = 7.8, 7.4 Hz), 6.99 (1H, d, J = 8.3 Hz), 3.88 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 157.8, 134.9, 126.8, 125.2 (q, $J_{C\text{-}F}$ = 340.2 Hz), 123.5, 121.8, 111.5, 56.1; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –74.0; **HRMS** (ESI$^+$) $[M+H]^+$ calc'd for $C_8H_7F_3O_2SNa$: 247.0011, found: 247.0014.

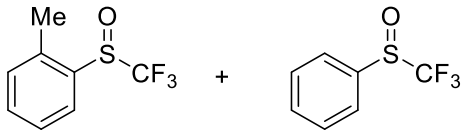


1-Methyl-3-((trifluoromethyl)sulfinyl)benzene 6h: Prepared according to **General Method E** (Eluent: 120:1 to 50:1 petroleum ether: ethyl acetate) and the title compound

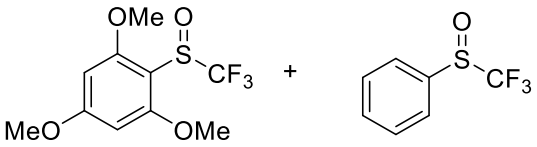
was isolated as a colorless liquid (61.8 mg, 0.297 mmol, 59.4% yield)[22]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.60 (1H, s), 7.56 (1H, d, J = 7.0 Hz), 7.56 (2H, m), 2.44 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 140.1, 135.5, 134.5, 129.5, 126.1, 124.8 (q, $J_{C\text{-}F}$ = 336.3 Hz), 123.2, 21.4; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –74.6.



1,3,5-Trimethyl-2-((trifluoromethyl)sulfinyl)benzene 6i &((trifluoromethyl)sulfinyl)benzene 6a： Prepared according to **General Method E** (Eluent: 120:1 to 50:1 petroleum ether: ethyl acetate) and the title compound **6i** was isolated as a yellow liquid (98.5 mg, 0.417 mmol, 83.4% yield), ^{19}F NMR yield of product **6i: 6a** = 4: 1[23]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.92 (2H, s), 2.54 (6H, brs), 2.31 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 143.7, 132.7, 131.6, 130.5, 128.3, 126.2 (q, $J_{C\text{-}F}$ = 262.6 Hz), 121.6, 21.2, 19.3; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –70.1.



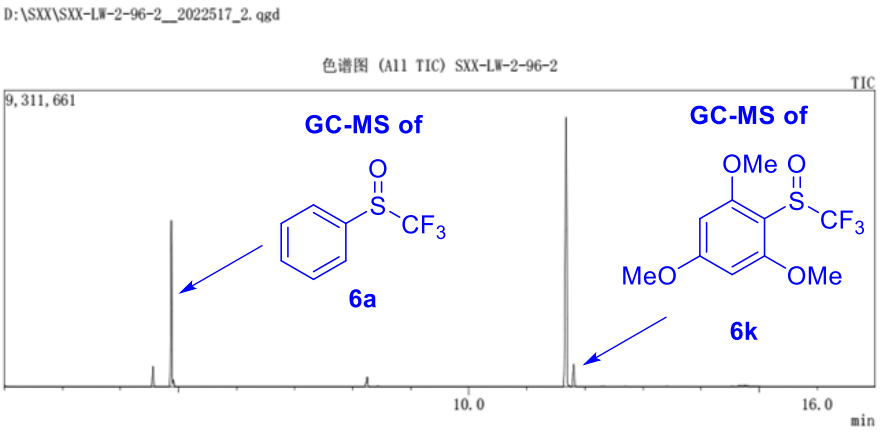
1-Methyl-2-((trifluoromethyl)sulfinyl)benzene 6j & ((trifluoromethyl)sulfinyl)benzene 6a: Prepared according to **General Method E** (Eluent: 120:1 to 50:1 petroleum ether: ethyl acetate) and the title compound was isolated as a colorless liquid (79.4 mg, 0.381 mmol, 76.3% yield), ^{19}F NMR rate of **6j: 6a** = 5: 1[23]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.00 (2H, d, J = 7.7 Hz), 7.78 (0.4H, d, J = 7.6 Hz), 7.67 – 7.58 (0.6H, m), 7.53 – 7.44 (m, 2H), 7.29 (1H, d, J = 7.4 Hz), 2.45 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.1, 135.7, 134.3, 133.6, 133.3, 131.4, 129.7, 127.3, 125.9, 125.3 (q, $J_{C\text{-}F}$ = 337.1 Hz), 18.3; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –73.6, –74.6.



1-methyl-2-((trifluoromethyl)sulfinyl)benzene 6k & ((trifluoromethyl)sulfinyl)benzene 6a: Prepared according to **General Method E** and

the $PhCF_3$ (99.0 wt%, 73.1 mg, 0.500 mmol) was added directly to the mixture, ^{19}F NMR yield of product **6a**+**6k**: 81.0%, **6a**: **6k** = 13 : 1 and were confirmed by GC-MS[21];

2022/7/1 10:47



质谱

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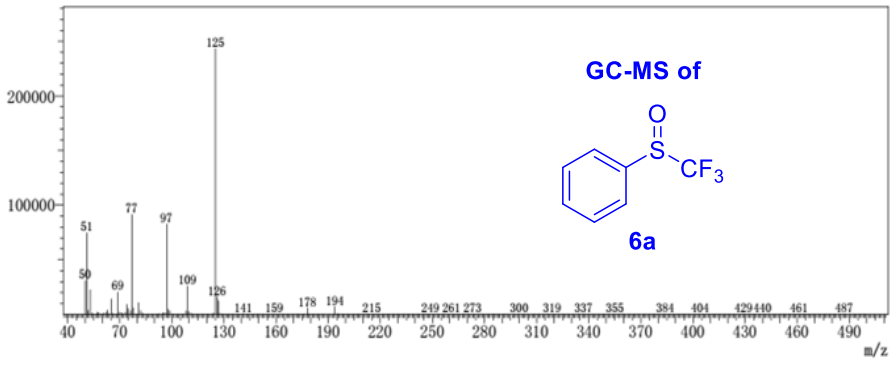

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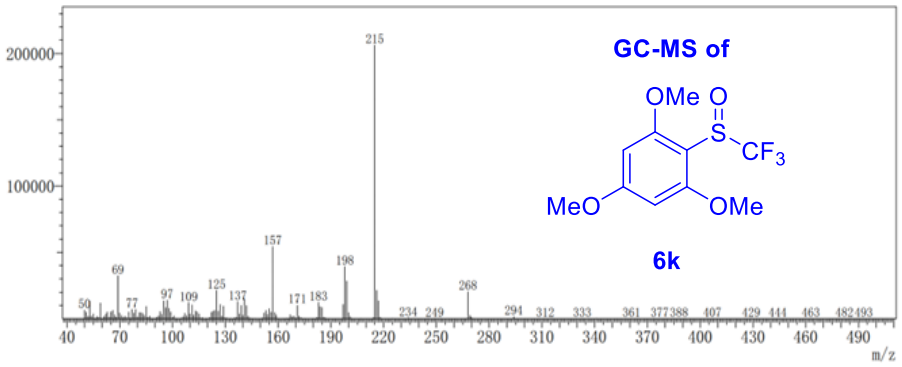
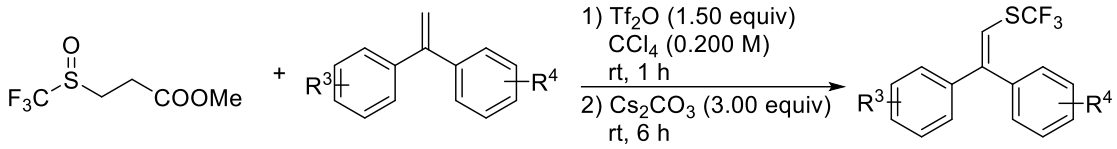
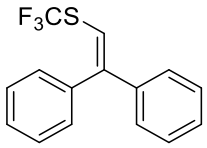


2. General Method F

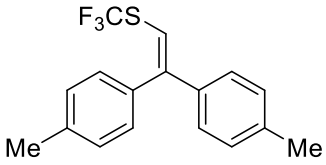


4a **7a-k** **8a-k**

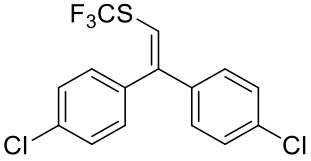
An oven-dried 25-mL glass schlenck, equipped with a stirring bar, and was evacuated and backfilled with N_2 for three times. And was charged with alkene (0.500 mmol, 1.00 equiv), methyl 3-((trifluoromethyl)sulfinyl)propanoate **4a** (153 mg, 0.750 mmol, 1.50 equiv), CCl_4 (2.50 mL) was added under N_2 atmosphere. Then the Tf_2O (212 mg, 0.750 mmol, 1.50 equiv) was added dropwise to the mixture, and the mixture was allowed to stir at room temperature for 1 h. Then Cs_2CO_3 (488 mg, 1.50 mmol, 3.00 equiv) was added to the mixture under N_2 atmosphere, then the mixture was allowed to stir for 6 h at room temperature. The mixture was diluted with brine (25.0 mL) and extracted with DCM (25.0 mL × 3). The combined organic layers were washed with brine (30.0 mL × 3), dried over by Na_2SO_4 then filtered. The solvent was removed by rotary evaporation and the residue was purified by flash silica gel chromatography.



(2,2-Diphenylvinyl)(trifluoromethyl)sulfane 8a: Prepared according to **General Method F** (Eluent: petroleum ether) and the title compound was isolated as a colorless liquid (116.4 mg, 0.415 mmol, 83.1% yield)[24]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.51–7.44 (3H, m), 7.38 (3H, d, J = 4.4 Hz), 7.31 (4H, dd, J = 7.1, 6.8 Hz), 6.79 (1H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 146.5, 140.4, 138.2, 130.0 (q, $J_{C\text{-}F}$ = 308.9 Hz), 129.5, 128.8, 128.7, 128.6, 128.5, 127.5, 112.2 (q, $J_{C\text{-}F}$ = 3.3 Hz); **^{19}F NMR** (471 MHz, $CDCl_3$) δ –42.6.

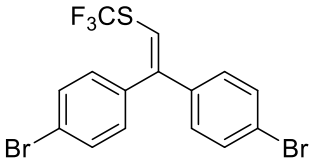


(2,2-Di-*p*-tolylvinyl)(trifluoromethyl)sulfane 8b: Prepared according to **General Method F** (Eluent: petroleum ether) and the title compound was isolated as a colorless liquid (139.2 mg, 0.451 mmol, 90.3% yield)[24]; **^{1}H NMR** (500 MHz, $CDCl_3$) δ 7.28 (2H, d, J = 7.8 Hz), 7.24 – 7.13 (6H, m), 6.69 (1H, s), 2.46 (3H, s), 2.41 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 146.8, 138.6, 138.4, 137.9, 135.4, 130.0 (q, $J_{C\text{-}F}$ = 309.0 Hz), 129.4, 129.4, 129.3, 127.5, 110.7 (q, $J_{C\text{-}F}$ = 3.3 Hz), 21.5, 21.3; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –42.7.

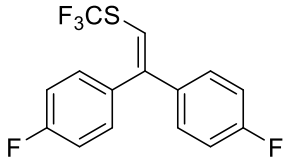


(2,2-Bis(4-chlorophenyl)vinyl)(trifluoromethyl)sulfane 8c: Prepared according to **General Method F** (Eluent: petroleum ether) and the title compound was isolated as a colorless liquid (136.6 mg, 0.391 mmol, 78.2% yield)[24]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.42 (2H, d, J = 8.4 Hz), 7.31 (2H, d, J = 8.5 Hz), 7.18 (4H, d, J = 8.4 Hz), 6.73 (1H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.1, 138.5, 136.1, 134.9, 134.7, 130.8, 129.7 (q, $J_{C\text{-}F}$ = 309.2 Hz), 129.2, 128.9, 128.7, 113.4 (q, $J_{C\text{-}F}$ = 3.4 Hz); **^{19}F NMR** (471 MHz,

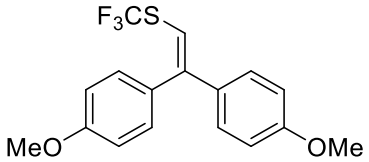
$CDCl_3$) δ –42.6.



(2,2-Bis(4-chlorophenyl)vinyl)(trifluoromethyl)sulfane 8d: Prepared according to **General Method F** (Eluent: petroleum ether) and the title compound was isolated as a colorless liquid (166.4 mg, 0.380 mmol, 76.0% yield)[24]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.58 (2H, d, J = 8.4 Hz), 7.46 (2H, d, J = 8.5 Hz), 7.11 (4H, d, J = 8.3 Hz), 6.73 (1H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 144.1, 138.8, 136.4, 132.2, 131.9, 131.1, 129.6 (q, $J_{C\text{-}F}$ = 309.3 Hz), 129.0, 123.2, 123.0, 113.5 (q, $J_{C\text{-}F}$ = 3.4 Hz); **^{19}F NMR** (471 MHz, $CDCl_3$) δ –42.3.

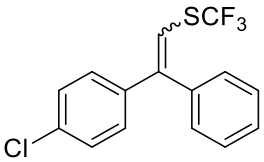


(2,2-Bis(4-fluorophenyl)vinyl)(trifluoromethyl)sulfane 8e: Prepared according to **General Method F** (Eluent: petroleum ether) and the title compound was isolated as a colorless liquid (141.5 mg, 0.447 mmol, 89.5% yield)[24]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.21 – 7.17 (4H, m), 7.10 (2H, dd, J = 8.6, 8.7 Hz), 7.00 (2H, dd, J = 8.5, 8.7 Hz), 6.64 (1H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 163.0 (d, $J_{C\text{-}F}$ = 250.0 Hz), 162.9 (d, $J_{C\text{-}F}$ = 249.8 Hz), 144.8, 136.5 (d, $J_{C\text{-}F}$ = 3.2 Hz), 134.0 (d, $J_{C\text{-}F}$ = 3.5 Hz), 131.4 (d, $J_{C\text{-}F}$ = 8.2 Hz), 129.8 (q, $J_{C\text{-}F}$ = 309.1 Hz), 129.3 (d, $J_{C\text{-}F}$ = 8.4 Hz), 115.9 (d, $J_{C\text{-}F}$ = 31.6 Hz), 115.7 (d, $J_{C\text{-}F}$ = 31.6 Hz), 112.4; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –42.6 (3F, s), –112.1 – –112.1 (1F, m), –112.8 – –112.9 (1F, m).

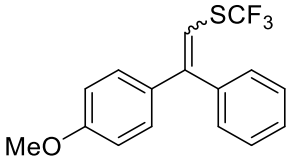


(2,2-Bis(4-methoxyphenyl)vinyl)(trifluoromethyl)sulfane 8f: Prepared according to **General Method F** (Eluent: 100:1 petroleum ether: ethyl acetate) and the title compound was isolated as a colorless liquid (151.3 mg, 0.445 mmol, 88.9% yield)[24]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.24 – 7.18 (4H, m), 6.98 (2H, d, J = 8.5 Hz), 6.88 (2H, d, J = 8.7 Hz), 6.57 (1H, s), 3.87 (3H, s), 3.84 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ

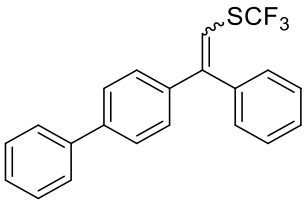
160.0, 159.8, 146.8, 133.4, 130.9, 130.6, 130.0 (q, $J_{C\text{-}F}$ = 308.8 Hz), 129.0, 114.0, 113.9, 109.1 (q, $J_{C\text{-}F}$ = 3.1 Hz), 55.3, 55.3; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –43.9.



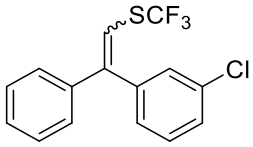
(*Z*)-(2-(4-Chlorophenyl)-2-phenylvinyl)(trifluoromethyl)sulfane & (*E*)-(2-(4-Chlorophenyl)-2-phenylvinyl)(trifluoromethyl)sulfane 8g: Prepared according to **General Method F** (Eluent: petroleum ether) and the title compound was isolated as a colorless liquid (134.1 mg, 0.426 mmol, 85.2% yield, E/Z = 1:1)[25]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.41 – 7.36 (2.5H, m), 7.31 – 7.25 (2.5H, m), 7.22 – 7.14 (4H, m), 6.70 (0.5H, s), 6.68 (0.5H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 145.6, 145.1, 140.1, 138.8, 137.8, 136.6, 134.7, 134.7, 134.5, 134.5, 130.9, 129.9 (q, $J_{C\text{-}F}$ = 309.2 Hz), 129.8 (q, $J_{C\text{-}F}$ = 309.0 Hz), 129.4, 129.1, 128.9, 128.8, 128.8, 128.7, 127.5, 112.9 (q, $J_{C\text{-}F}$ = 3.2 Hz), 112.7 (q, $J_{C\text{-}F}$ = 3.4 Hz); **^{19}F NMR** (471 MHz, $CDCl_3$) δ –42.5, –42.5.



(*Z*)-(2-(4-Methoxyphenyl)-2-phenylvinyl)(trifluoromethyl)sulfane & (*E*)-(2-(4-Methoxyphenyl)-2-phenylvinyl)(trifluoromethyl)sulfane 8h: Prepared according to **General Method F** (Eluent: petroleum ether) and the title compound was isolated as a colorless liquid (140.7 mg, 0.454 mmol, 90.7% yield, E/Z = 2:1)[25]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.41 – 7.39 (1H, m), 7.37 – 7.33 (2H, m), 7.30 – 7.27 (1H, m), 7.24 – 7.23 (0.5H, m), 7.21 – 7.19 (2H, m), 7.17 – 7.13 (3H, m), 6.92 (1H, d, J = 8.7 Hz), 6.82 (2H, d, J = 8.7 Hz), 6.61 (0.5H, s), 6.59 (1H, s), 3.80 (1.5H, s), 3.76 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 160.0, 159.9, 147.0, 146.4, 140.8, 138.4, 133.1, 130.9, 130.4, 130.0 (q, $J_{C\text{-}F}$ = 308.8 Hz), 129.5, 128.8, 128.7, 128.5, 128.4, 127.7, 114.1, 113.9, 111.4 (q, $J_{C\text{-}F}$ = 3.3 Hz), 109.8 (q, $J_{C\text{-}F}$ = 3.1 Hz), 55.3, 55.3; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –42.7, –42.7.

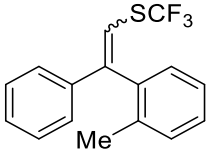


(*Z*)-(2-([1,1'-Biphenyl]-4-yl)-2-phenylvinyl)(trifluoromethyl)sulfane & (*E*)-(2-([1,1'-Biphenyl]-4-yl)-2-phenylvinyl)(trifluoromethyl)sulfane 8i: Prepared according to **General Method F** (Eluent: petroleum ether) and the title compound was isolated as a colorless liquid (130.0 mg, 0.365 mmol, 73.0% yield) (It was difficult to identify the E and Z structure, but the ratio was determined by ^{19}F NMR spectroscopy as 1:2); **M.p.** = 75.7 – 76.4 ºC; **IR (thin film)** 3003 (w), 3000 (w), 1598 (w), 1497 (w), 1432 (w), 1107 (s), 758 (s), 690 (s) cm^{-1}; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.73 – 7.70 (2H, m), 7.67 – 7.61 (4H, m), 7.55 – 7.33 (15H, m), 6.85 (1H, s), 6.80 (0.5H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 146.2, 146.1, 141.5, 141.2, 140.5, 140.5, 140.4, 139.2, 138.1, 137.1, 130.0, 129.9 (q, $J_{C\text{-}F}$ = 309.5 Hz), 129.5, 129.0, 128.8, 128.7, 128.6, 128.5, 127.9, 127.7, 127.7, 127.7, 127.4, 127.2, 127.2, 127.1, 112.2 (q, $J_{C\text{-}F}$ = 3.4 Hz);**^{19}F NMR** (471 MHz, $CDCl_3$) δ –42.5, –42.5; **HRMS** (ESI$^+$) $[M+H]^+$ calc'd for $C_{17}H_{16}F_3S$: 356.0919, found: 903.0919.

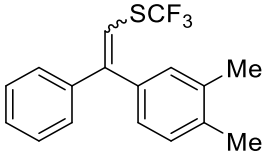


(*E*)-(2-(3-Chlorophenyl)-2-phenylvinyl)(trifluoromethyl)sulfane & (*Z*)-(2-(3-Chlorophenyl)-2-phenylvinyl)(trifluoromethyl)sulfane 8j: Prepared according to **General Method F** (Eluent: petroleum ether) and the title compound was isolated as a colorless liquid (108.6 mg, 0.345 mmol, 69.0% yield) (It was difficult to identify the E and Z structure, but the ratio was determined by ^{19}F NMR spectroscopy as 1:2.5); **IR (thin film)** 3203 (w), 1587 (w), 1103 (s), 773 (m), 753 (m), 694 (m) cm^{-1}; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.48 – 7.44 (1H, m), 7.48 – 7.44 (0.4H, m), 7.42 – 7.39 (1H, m), 7.42 – 7.39 (0.4H, m), 7.37 – 7.24 (5H, m), 7.37 – 7.24 (2H, m), 7.18 – 7.15 (1H, m), 7.18 – 7.15 (0.4H, m),6.77 (1H, s), 6.77 (0.4H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 145.3, 144.7, 142.2, 139.9, 139.8, 137.5, 134.8, 134.7, 130.1, 129.8, 129.8 (q, $J_{C\text{-}F}$ = 308.7 Hz),

129.8 (q, $J_{C\text{-}F}$ = 309.2 Hz), 129.5, 129.4, 129.0, 129.0, 128.9, 128.8, 128.5, 127.7, 127.5, 127.5, 125.7, 113.9 (q, $J_{C\text{-}F}$ = 3.5 Hz), 113.2 (q, $J_{C\text{-}F}$ = 3.1Hz); **^{19}F NMR** (471 MHz, $CDCl_3$) δ –42.2, –43.0. **HRMS** (EI) calc'd for $C_{15}H_{10}F_3SCl$: 314.0134, found: 314.0138.



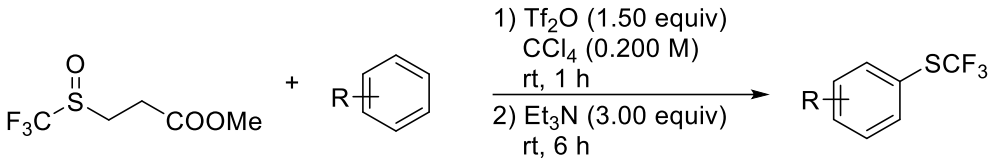
(*E*)-(2-Phenyl-2-(*o*-tolyl)vinyl)(trifluoromethyl)sulfane & (*Z*)-(2-Phenyl-2-(*o*-tolyl)vinyl)(trifluoromethyl)sulfane 8k: Prepared according to **General Method F** (Eluent: petroleum ether) and the title compound was isolated as a colorless liquid (91.7 mg, 0.312 mmol, 62.3% yield) (the ratio of E and Z was determined by ^{19}F NMR spectroscopy as 1:2.4); **IR (thin film)** 2923 (m), 2802 (m), 1572 (w), 1465 (w), 1112 (s), 751 (m), 731 (m), 694 (m) cm^{-1}; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.43 – 7.39 (1H, m), 7.43 – 7.39 (0.4H, m), 7.37 – 7.33 (4H, m), 7.37 – 7.33 (1.6H, m), 7.32 – 7.25 (3H, m), 7.32 – 7.25 (1.2H, m), 7.24 – 7.18 (1H, m), 7.24 – 7.18 (0.4H, m), 6.94 (1H, s), 6.46 (0.4H, s), 2.17 (3H, s), 2.13 (1.2H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 146.8, 145.3, 141.3, 138.7, 138.3, 137.3, 136.4, 136.3, 134.6, 131.6, 130.8, 130.8, 130.1, 130.0 (q, $J_{C\text{-}F}$ = 309.0 Hz), 129.6, 128.8, 128.8, 128.6, 128.5, 128.5, 128.4, 126.4, 126.2, 126.0, 113.6 (q, $J_{C\text{-}F}$ = 3.1 Hz), 113.0 (q, $J_{C\text{-}F}$ = 3.3 Hz), 20.4, 19.4; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –42.2, –43.0; **HRMS** (ESI^+) calc'd for $C_{16}H_{13}F_3S$: 294.0686, found: 294.0685.



(*E*)-(2-(3,4-Dimethylphenyl)-2-phenylvinyl)(trifluoromethyl)sulfane & (*Z*)-(2-(3,4-Dimethylphenyl)-2-phenylvinyl)(trifluoromethyl)sulfane 8l: Prepared according to **General Method F** (Eluent: petroleum ether) and the title compound was isolated as a colorless liquid (103.0 mg, 0.334 mmol, 66.8% yield) (the ratio of E and Z was determined by ^{19}F NMR spectroscopy as 1:1.5); **IR (thin film)** 3031 (w), 2898 (w), 1465 (w), 1217 (m), 1103 (s), 755 (m), 696 (m) cm^{-1}; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.49 – 7.42 (2H, m), 7.49 – 7.42 (1.2H, m), 7.37 – 7.27 (3H, m), 7.37 – 7.27 (1.8H, m), 7.24 (0.6H, d, J = 8.0 Hz), 7.14 (1H, d, J = 7.8 Hz), 7.09 – 7.10 (2H, m), 7.09 –

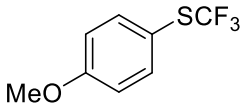
7.10 (1.2H, m), 6.72 (1H, s), 6.72 (0.6H, s), 2.36 (3H, s), 2.33 (3H, s), 2.32 (3H, s), 2.29 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 147.1, 146.6, 140.7, 138.5, 138.1, 137.3, 137.3, 137.1, 136.8, 135.7, 130.5, 130.0, 130.0 (q, $J_{C\text{-}F}$ = 308.8 Hz), 129.9, 129.5, 128.7, 128.6, 128.6, 128.4, 127.6, 125.1, 111.8 (q, $J_{C\text{-}F}$ = 3.3 Hz), 111.0 (q, $J_{C\text{-}F}$ = 3.1 Hz), 19.9, 19.8, 19.6; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –42.6, –42.7; **HRMS** (ESI^+) $[M+H]^+$ calc'd for $C_{17}H_{16}F_3S$: 309.0919, found: 309.0921.

3. General Method G:

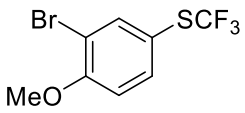


4a **9a-k** **10a-k**

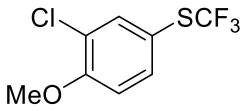
An oven-dried 25-mL glass schlenck, equipped with a stirring bar, and was evacuated and backfilled with nitrogen for three times. And was charged with arene **9a-k** (0.500 mmol, 1.00 equiv), methyl 3-((trifluoromethyl)sulfinyl)propanoate **4a** (153 mg, 0.750 mmol, 1.50 equiv), CCl_4 (2.50 mL) was added under N_2. Then the Tf_2O (212 mg, 0.750 mmol, 1.50 equiv) was added dropwise to the mixture, and the mixture was allowed to stir at room temperature for 1 h. Then Et_3N (152 mg, 1.50 mmol, 3.00 equiv) was added to the mixture under N_2 atmosphere, the mixture was allowed to stir at room temperature for 6 h. The solvent was removed by rotary evaporation and the residue was purified by flash silica gel chromatography.



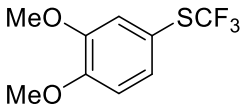
(4-Methoxyphenyl)(trifluoromethyl)sulfane 10a: Prepared according to **General Method G** (Eluent: petroleum ether) and the title compound was isolated as a colorless liquid (85.2 mg, 0.409 mmol, 81.8% yield)[4]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.58 (2H, d, J = 8.7 Hz), 6.93 (2H, d, J = 8.9 Hz), 3.84 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 162.0, 138.4, 129.8 (q, $J_{C\text{-}F}$ = 309.3 Hz), 115.1, 114.9 (q, $J_{C\text{-}F}$ = 2.0 Hz), 55.5; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –44.0.



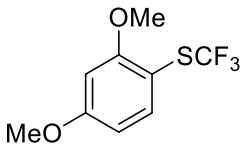
(3-Bromo-4-methoxyphenyl)(trifluoromethyl)sulfane 10b: Prepared according to **General Method G** (Eluent: petroleum ether) and the title compound was isolated as a colorless liquid (90.3 mg, 0.315 mmol, 62.9% yield)[4]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.83 (1H, s), 7.56 (1H, d, J = 8.6 Hz), 6.90 (1H, d, J = 8.6 Hz), 3.91 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.3, 141.0, 137.4, 129.5 (q, $J_{C\text{-}F}$ = 309.5 Hz), 116.1 (d, $J_{C\text{-}F}$ = 2.1 Hz), 112.3, 112.3, 56.4; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –43.6.



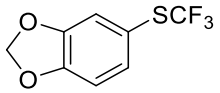
(3-Chloro-4-methoxyphenyl)(trifluoromethyl)sulfane 10c: Prepared according to **General Method G** (Eluent: Eluent: petroleum ether) and the title compound was isolated as colorless liquid (96.2 mg, 0.396 mmol, 79.3% yield)[26]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.67 (1H, d, J = 2.1 Hz), 7.53 (1H, dd, J = 8.6, 2.1 Hz), 6.95 (1H, d, J = 8.6 Hz), 3.94 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 157.5, 138.1, 136.7, 129.5 (q, $J_{C\text{-}F}$ = 309.8 Hz), 123.4, 115.7 (d, $J_{C\text{-}F}$ = 2.1 Hz), 112.5, 56.4; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –43.6.



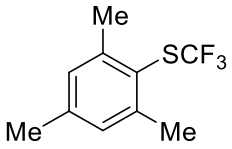
(3,4-Dimethoxyphenyl)(trifluoromethyl)sulfane 10d: Prepared according to **General Method G** (Eluent: 200:1 to 50:1 petroleum ether: ethyl acetate) and the title compound was isolated as a colorless liquid (116.3 mg, 0.488 mmol, 97.6% yield)[4]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.22 (1H, dd, J = 8.3, 2.0 Hz), 7.09 (1H, d, J = 1.9 Hz), 6.85 (1H, d, J = 8.4 Hz), 3.87 (3H, s), 3.87 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 151.6, 149.3, 130.2, 129.7 (q, $J_{C\text{-}F}$ = 309.4 Hz), 118.9, 114.9 (q, $J_{C\text{-}F}$ = 2.0 Hz), 111.5, 56.1, 55.9; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –43.8.



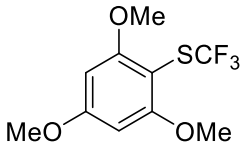
(2,4-Dimethoxyphenyl)(trifluoromethyl)sulfane 10e: Prepared according to **General Method G** (Eluent: petroleum ether) and the title compound was isolated as colorless liquid (117.1 mg, 0.492 mmol, 98.3% yield)[4]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.52 (1H, d, J = 9.2 Hz), 6.51 (1H, d, J = 7.0 Hz), 6.50 (1H, s), 3.87 (3H, s), 3.82 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 164.1, 162.2, 140.3, 129.7 (q, $J_{C\text{-}F}$ = 310.3 Hz), 105.7, 103.1 (d, $J_{C\text{-}F}$ = 1.3 Hz), 99.3, 56.0, 55.5; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –43.7.



5-((Trifluoromethyl)thio)benzo[d][1,3]dioxole 10f: Prepared according to **General Method G** (Eluent: petroleum ether) and the title compound was isolated as a colorless liquid (77.2 mg, 0.348 mmol, 69.5% yield)[4]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.17 (1H, d, J = 9..2 Hz), 7.09 (1H, s), 6.83 (1H, d, J = 8.1 Hz), 6.04 (2H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 150.5, 148.4, 131.7, 129.7 (q, $J_{C\text{-}F}$ = 309.4 Hz), 116.4, 116.1 (q, $J_{C\text{-}F}$ = 2.0 Hz), 109.2, 102.1; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –43.9.

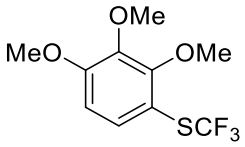


Mesityl(trifluoromethyl)sulfane 10g: Prepared according to **General Method E** (Eluent: petroleum ether) and the title compound was isolated as colorless liquid (52.5 mg, 0.238 mmol, 47.7% yield)[27]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.03 (2H, s), 2.56 (6H, s), 2.32 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 145.4, 141.5, 130.3 (q, $J_{C\text{-}F}$ = 310.4 Hz), 129.7, 120.2 (d, $J_{C\text{-}F}$ = 1.3 Hz), 22.2, 21.3; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –41.9.

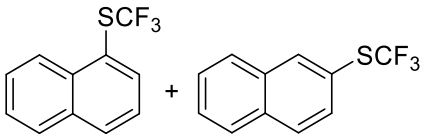


(Trifluoromethyl)(2,4,6-trimethoxyphenyl)sulfane 10h: Prepared according to **General Method G** (Eluent: 100:1 to 20:1 petroleum ether: ethyl acetate) and the title

compound was isolated as a white solid (126.3 mg, 0.471 mmol, 94.1% yield)[4]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 6.13 (2H, s), 3.84 (3H, s), 3.81 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 164.6, 163.5, 129.6 (q, $J_{C\text{-}F}$ = 311.7 Hz), 91.4 (q, $J_{C\text{-}F}$ = 1.8 Hz), 91.0, 56.2, 55.4; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –43.5.

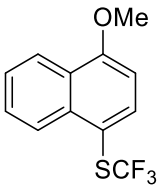


(Trifluoromethyl)(2,3,4-trimethoxyphenyl)sulfane 10i: Prepared according to **General Method G** (Eluent: Eluent: 100:1 to 20:1 petroleum ether: ethyl acetate) and the title compound was isolated as colorless liquid (120.4 mg, 0.449 mmol, 89.8% yield); **IR (thin film)** 2968 (w), 2786 (w), 1578 (m), 1484 (m), 1461 (m), 1408 (s), 1296 (m), 1089 (s), 1012 (s), 799 (m) cm^{-1}; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.33 (1H, d, J = 8.8 Hz), 6.69 (1H, d, J = 8.8 Hz), 3.94 (3H, s), 3.87 (3H, s), 3.85 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 156.9, 156.0, 142.9, 133.5, 129.6 (q, $J_{C\text{-}F}$ = 309.9 Hz), 109.4 (d, $J_{C\text{-}F}$ = 1.4 Hz), 107.7, 61.6, 61.0, 56.2; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –43.3; **HRMS** (EI) calc'd for $C_{10}H_{11}F_3O_3S$: 268.0374, found: 268.0376.

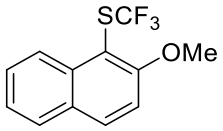


10j **10j'**

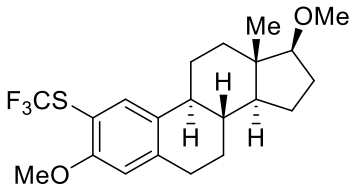
Naphthalen-1-yl(trifluoromethyl)sulfane 10j & Naphthalen-2-yl(trifluoromethyl)sulfane 10j': Prepared according to **General Method G** (Eluent: petroleum ether) and the title compound was isolated as white solid (72.7 mg, 0.319 mmol, 63.7% yield, **10j**:**10j'** = 7:1)[4]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.58 (1H, d, J = 8.5 Hz), 8.22 (0.14H, s), 8.01 (1H, dd, J = 9.4, 8.3 Hz), 7.91 (1H, d, J = 8.2 Hz), 7.92 – 7.87 (0.42H, m), 7.68 (1H, dd, J = 8.1, 7.3 Hz), 7.70 – 7.66 (0.14H, m), 7.59 (1H, dd, J = 7.1, 7.1 Hz), 7.61 – 7.57 (0.28H, m), 7.52 (1H, dd, J = 7.6, 7.9 Hz); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 138.0, 137.2, 135.5, 134.4, 134.0, 133.5, 132.5, 131.9, 129.8 (q, $J_{C\text{-}F}$ = 309.0 Hz), 129.4, 128.7, 128.3, 128.1, 127.9, 127.8, 127.1, 126.9, 126.0, 125.7, 121.7 (d, $J_{C\text{-}F}$ = 1.6 Hz); **^{19}F NMR** (471 MHz, $CDCl_3$) δ –42.2, –42.4.



(4-Methoxynaphthalen-1-yl)(trifluoromethyl)sulfane 10k: Prepared according to **General Method G** (Eluent: petroleum ether) and the title compound was isolated as colorless liquid (100.6 mg, 0.390 mmol, 77.9% yield)[4]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.46 (1H, d, J = 8.5 Hz), 8.29 (1H, d, J = 8.4 Hz), 7.85 (1H, d, J = 8.1 Hz), 7.62 (1H, dd, J = 7.1, 7.2 Hz), 7.52 (1H, dd, J = 8.1, 7.1 Hz), 6.76 (1H, d, J = 8.1 Hz), 3.96 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.8, 139.1, 136.3, 129.9 (q, $J_{C\text{-}F}$ = 310.8 Hz), 128.2, 126.5, 126.1, 125.8, 122.7, 112.3 (d, $J_{C\text{-}F}$ = 1.5 Hz), 104.0, 55.8; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –43.2.



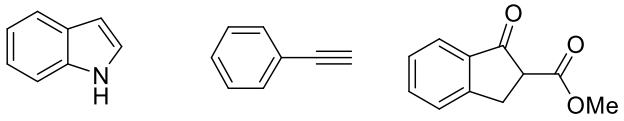
(2-Methoxynaphthalen-1-yl)(trifluoromethyl)sulfane 10l: Prepared according to **General Method G** (Eluent: petroleum ether) and the title compound was isolated as white solid (69.8 mg, 0.270 mmol, 54.1% yield)[28]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.56 (1H, d, J = 8.6 Hz), 8.00 (1H, d, J = 9.1 Hz), 7.81 (1H, d, J = 8.1 Hz), 7.64 (1H, dd, J = 8.2, 8.2 Hz), 7.44 (1H, dd, J = 7.8, 7.1 Hz), 7.31 (1H, d, J = 9.1 Hz), 4.04 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 160.8, 137.0, 134.3, 129.8 (q, $J_{C\text{-}F}$ = 311.7 Hz), 129.4, 128.4, 128.3, 125.1, 124.4, 113.1, 105.2 (d, $J_{C\text{-}F}$ = 1.2 Hz), 56.9; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –41.8.



(3-Chloro-4-methoxyphenyl)(trifluoromethyl)sulfane 12: Prepared according to **General Method G** (Eluent: Eluent: petroleum ether) and the title compound was isolated as colorless liquid (145 mg, 0.362 mmol, 72.4% yield)[4]; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.50 (1H, s), 6.67 (1H, s), 3.86 (3H, s), 3.38 (3H, s), 3.32 (1H, t, J = 8.3 Hz),

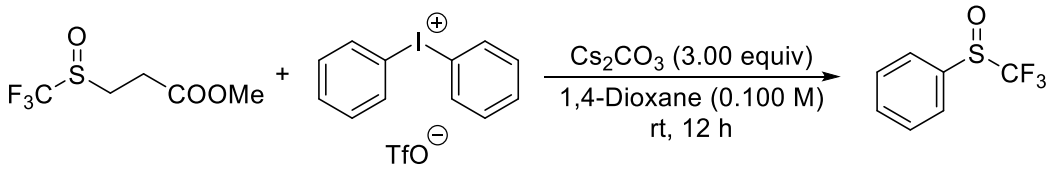
2.88 (2H, m), 2.34 – 2.23 (1H, m), 2.23 – 2.12 (1H, m), 2.11 – 2.03 (2H, m), 1.94 – 1.86 (1H, m), 1.75 – 1.64 (1H, m), 1.56– 1.14 (7H, m), 0.79 (3H, s); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 158.5, 142.6, 136.2, 133.7, 129.8 (d, $J_{C\text{-}F}$ = 310.2 Hz), 112.1, 109.0, 90.8, 58.0, 56.2, 50.3, 43.7, 43.3, 38.4, 38.0, 30.1, 27.9, 27.1, 26.4, 23.1, 11.7; **^{19}F NMR** (471 MHz, $CDCl_3$) δ –42.7.

Reactions of substrates such as indole, alkyne, and *beta*-keto ester with our reagents were not successful, low yields of desired products were obtained according to ^{19}F NMR and GC-MS. Further optimization of the reaction conditions is needed.



V. Gram-scale reaction

1. Synthesis of ((trifluoromethyl)sulfinyl)benzene 6a

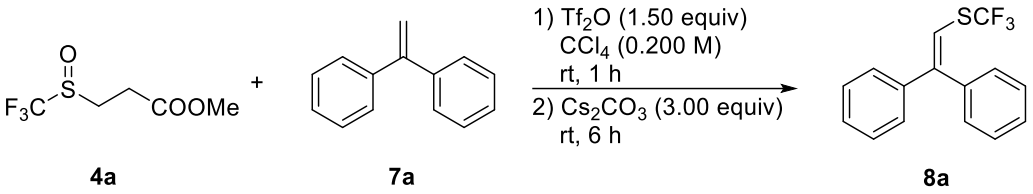


4a **5a** **6a**

An oven-dried 200-mL round bottom flask, equipped with a stirring bar, was charged with diphenyliodonium trifluoromethanesulfonate (2.37 g, 5.50 mmol, 1.10 equiv), Cs_2CO_3 (4.88 g, 15.0 mmol, 3.00 equiv). The mixture was evacuated and backfilled with nitrogen for three times. Then methyl 3-((trifluoromethyl)sulfinyl)propanoate **4a** (1.02 g, 5.00 mmol, 1.00 equiv) and dry 1,4-Dioxane (50.0 mL) was added under N_2 atmosphere, the mixture was allowed to stir at room temperature for 12 h. The mixture was diluted with brine (150 mL) and extracted with ethyl acetate (40.0 mL × 3). The combined organic layers were washed with brine (150 mL × 5), dried over by Na_2SO_4 then filtered. The solvent was removed by rotary evaporation and the residue was purified by flash silica gel chromatography (Eluent: 100:1 to 50:1 petroleum ether:

ethyl acetate) to provide the desired product **6a** as a colorless liquid (0.598 g, 2.53 mmol, 50.5% yield).

2. Synthesis of ((trifluoromethyl)sulfinyl)benzene 8a

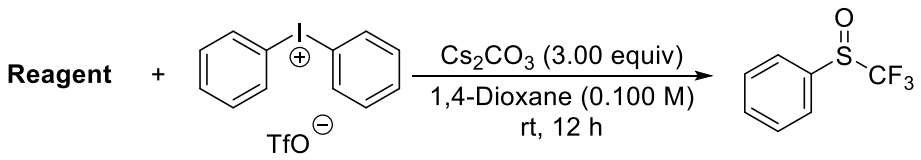


4a **7a** **8a**

An oven-dried 100-mL two-neck round bottom flask, equipped with a stirring bar, and was evacuated and backfilled with nitrogen for three times. And was charged with ethene-1,1-diarene (0.901 g, 5.00 mmol, 1.00 equiv), methyl 3-((trifluoromethyl)sulfinyl)propanoate **4a** (1.53 g, 7.50 mmol, 1.50 equiv), CCl_4 (25.0 mL) was added under N_2 atmosphere. Then the Tf_2O (2.12 g, 7.50 mmol, 1.50 equiv) was added dropwise to the mixture, and the mixture was allowed to stir at room temperature for 1 h. Then Cs_2CO_3 (4.88 g, 15.0 mmol, 3.00 equiv) was added to the mixture under N_2 atmosphere, the mixture was allowed to stir at room temperature for 6 h. The mixture was diluted with brine (25.0 mL) and extracted with DCM (50.0 mL × 3). The combined organic layers were washed with brine (80.0 mL × 3), dried over by Na_2SO_4 then filtered. The solvent was removed by rotary evaporation and the residue was purified by flash silica gel chromatography (Eluent: petroleum ether) to provide the desired product **8a** as a yellow liquid (1.10 g, 3.92 mmol, 78.3% yield).

VI. Mechanistic study

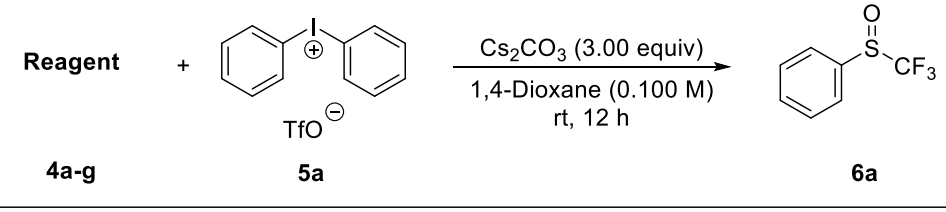
1. Effect of different reagents for trifluoromethylsulfinylation

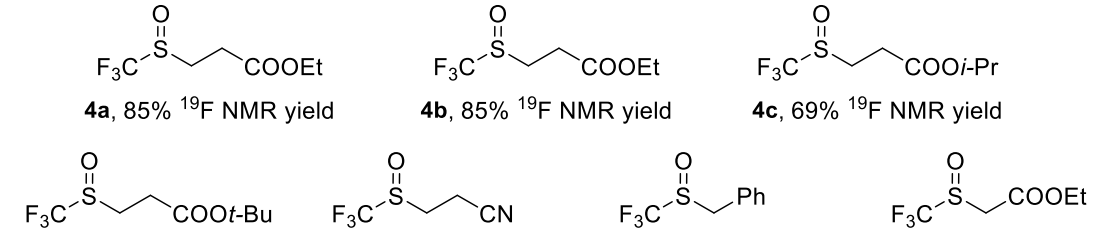


4a-g **5a** **6a**

An oven-dried 25-mL glass schlenck, equipped with a stirring bar, was charged with

diphenyliodonium trifluoromethanesulfonate (47.3 mg, 0.110 mmol, 1.10 equiv), Cs_2CO_3 (97.7 mg, 0.300 mmol, 3.00 equiv). The mixture was evacuated and backfilled with N_2 for three times. Then the reagent (0.100 mmol, 1.00 equiv) and dry 1,4-Dioxane (1.00 mL) was added under N_2 atmosphere and the mixture was allowed to stir at room temperature for 12 h. When the reaction completed, the yield was determined by ^{19}F NMR spectroscopy in the presence of $PhCF_3$ (99.0 wt%, 14.8 mg, 0.100 mmol) as an internal standard.



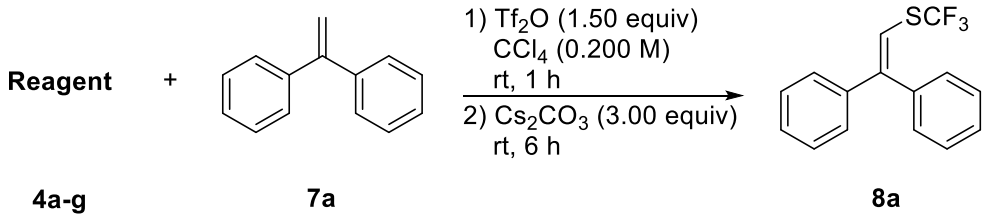


4a, 85% ^{19}F NMR yield **4b**, 85% ^{19}F NMR yield **4c**, 69% ^{19}F NMR yield

4d, 30% ^{19}F NMR yield **4e**, 76% ^{19}F NMR yield **4f**, <5% ^{19}F NMR yield **4g**, <5% ^{19}F NMR yield

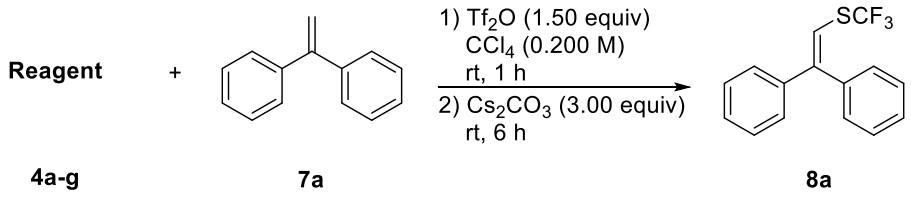
Reaction condition: **4a-g** (0.100 mmol), **5a** (0.110 mmol), Cs_2CO_3 (3.00 equiv), 1,4-Dioxane (1.00 mL) stirred at rt for 12 h. Yield was determined by ^{19}F NMR spectroscopy in the presence of $PhCF_3$ (99.0 wt%, 14.8 mg, 0.100 mmol) as an internal standard.

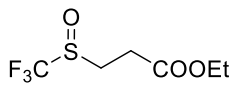
2. Effect of different reagents for C−H trifluoromethylthiolation of 1,1-diphenylethylene



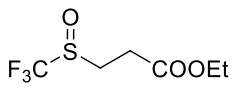
An oven-dried 25-mL glass schlenck, equipped with a stirring bar, and was evacuated and backfilled with nitrogen for three times. And was charged with 1,1-Diphenylethylene (18.0 mg, 0.100 mmol, 1.00 equiv), different EWG reagent (0.150 mmol, 1.50 equiv), CCl_4 (0.500 mL) were added under N_2 atmosphere. Then the Tf_2O (42.3 mg, 0.150 mmol, 1.50 equiv) was added dropwise to the mixture, and the

mixture was allowed to stir at room temperature for 1 h. Then Cs_2CO_3 (97.7 mg, 0.300 mmol, 3.00 equiv) was added under N_2 atmosphere, the mixture was allowed to stir at room temperature for 6 h. When the reaction completed, yield was determined by ^{19}F NMR spectroscopy in the presence of $PhCF_3$ (99.0 wt%, 14.8 mg, 0.100 mmol) as an internal standard.

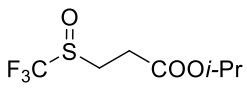




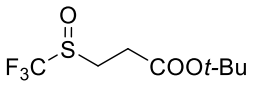
4a, 99% ^{19}F NMR yield



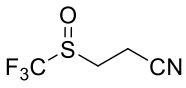
4b, 98% ^{19}F NMR yield



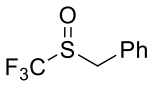
4c, 64% ^{19}F NMR yield



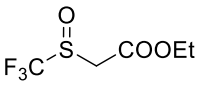
4d, 66% ^{19}F NMR yield



4e, 5% ^{19}F NMR yield



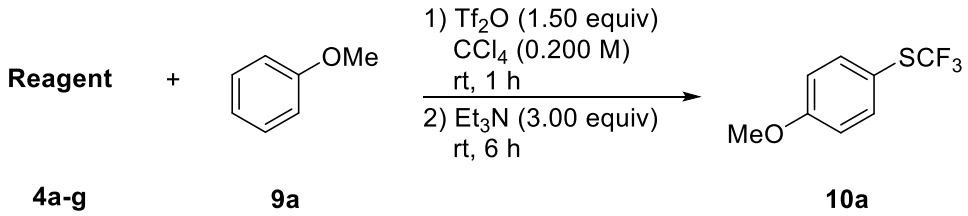
4f, <5% ^{19}F NMR yield



4g, 34% ^{19}F NMR yield

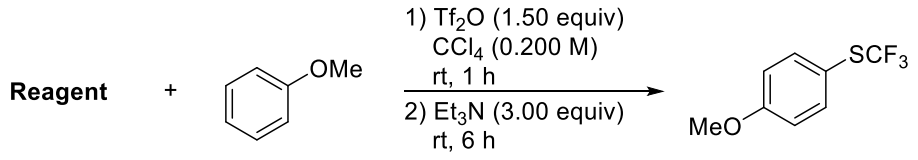
Reaction condition: **4a-g** (0.150 mmol), **7a** (0.100 mmol), CCl_4 (0.500 mL), Tf_2O (1.50 equiv) was addded dropwise to the mixture at rt for 1 h. Then Cs_2CO_3 (3.00 equiv) was added to the mixture under N_2 atmosphere, and stirred at rt for 6 h. Yield was determined by ^{19}F NMR spectroscopy in the presence of $PhCF_3$ (99.0 wt%, 14.8 mg, 0.100 mmol) as an internal standard.

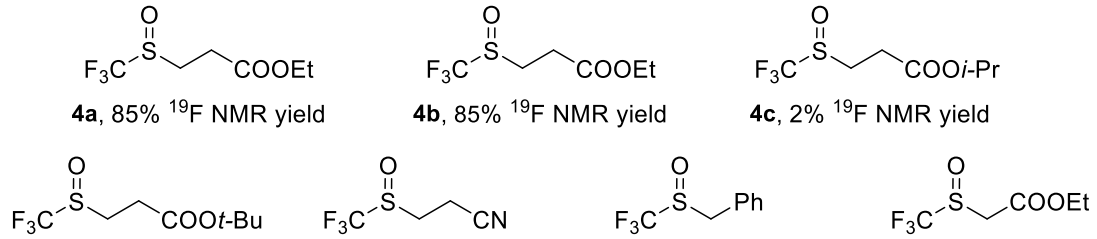
3. Effect of different reagents for C−H trifluoromethylthiolation of anisole



An oven-dried 25-mL glass schlenck, equipped with a stirring bar, and was evacuated and backfilled with nitrogen for three times. And was charged with anisole (10.8 mg, 0.100 mmol, 1.00 equiv), different EWG reagent (0.150 mmol, 1.50 equiv), CCl_4 (0.500 mL) were added under N_2 atmosphere. Then Tf_2O (42.3 mg, 0.150 mmol, 1.50 equiv) was added dropwise to the mixture, and the mixture was allowed to stir at room temperature for 1 h. Then Et_3N (30.4 mg, 0.300 mmol, 3.00 equiv) was added to the mixture under N_2 atmosphere, the mixture was allowed to stir at room temperature

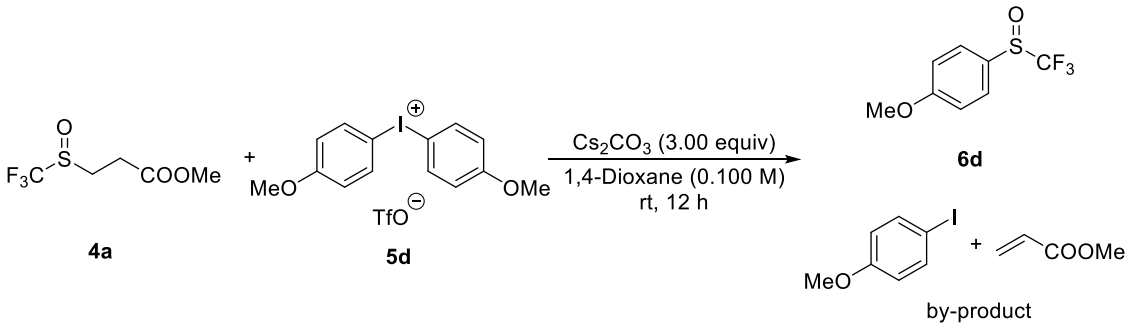
for 6 h. When the reaction completed, the yield was determined by ^{19}F NMR spectroscopy in the presence of $PhCF_3$ (99.0 wt%, 14.8 mg, 0.100 mmol) as an internal standard.





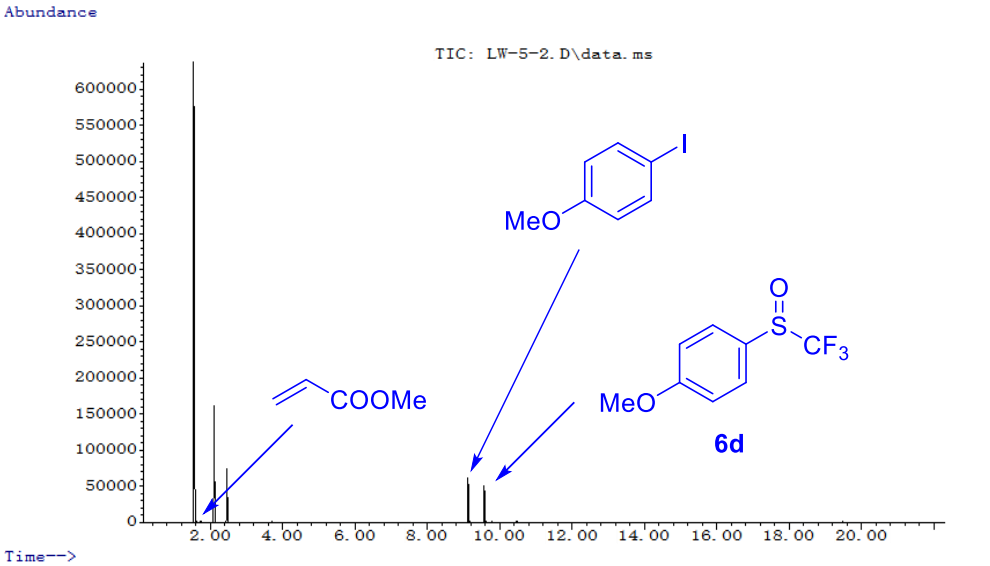
Reaction condition: **4a-g** (0.150 mmol), **9a** (0.100 mmol), CCl_4 (1.00 mL), Tf_2O (1.50 equiv) was added dropwise to rhe mixture at rt for 1 h. Then Et_3N (3.00 equiv) was added to the mixture under N_2 atmosphere, and stirred at rt for 6 h. Yield was determined by ^{19}F NMR spectroscopy in the presence of $PhCF_3$ (99.0 wt%, 14.8 mg, 0.100 mmol) as an internal standard.

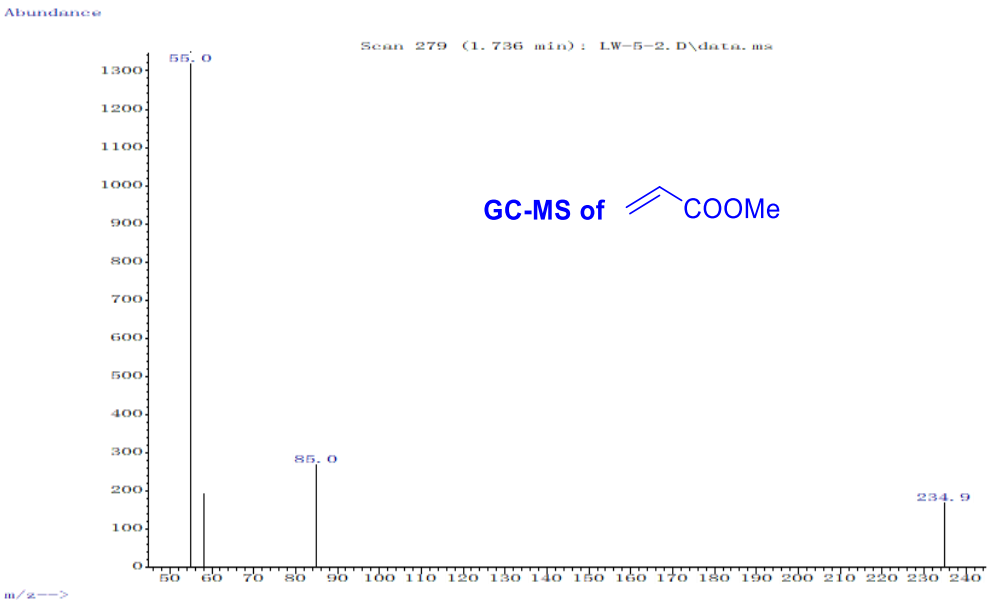
4. Observation of by-product for the reaction between 5d with reagent 4a

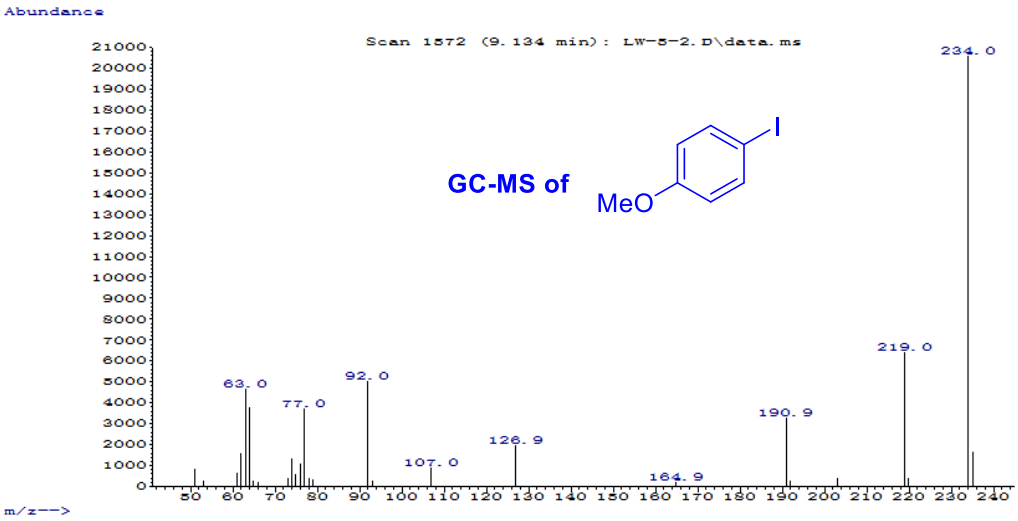


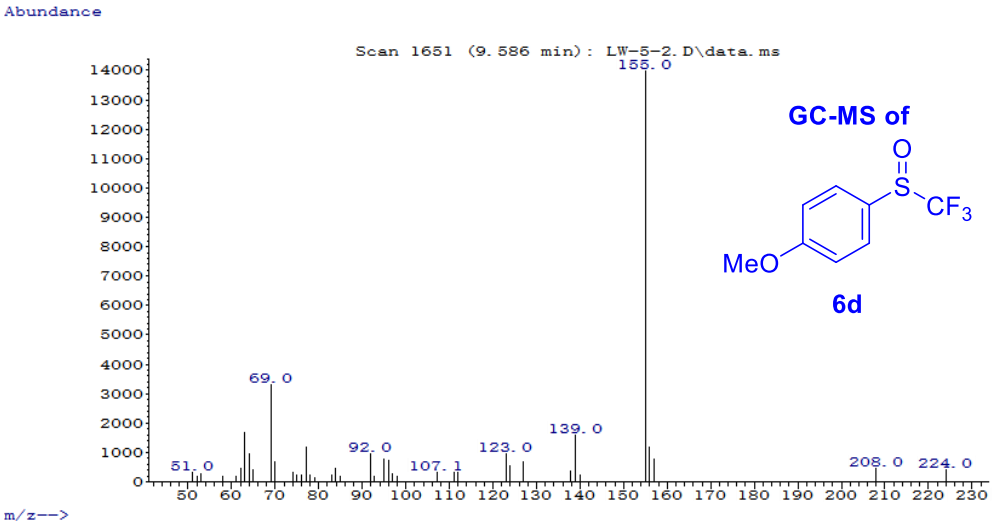
An oven-dried 25-mL glass schlenck, equipped with a stirring bar, was charged with bis(4-methoxyphenyl)iodonium trifluoromethanesulfonate **5d** (107.9 mg, 0.220 mmol, 1.10 equiv), Cs_2CO_3 (195.5 mg, 0.600 mmol, 3.00 equiv). The mixture was evacuated and backfilled with nitrogen for three times. Then methyl 3-((trifluoromethyl)sulfinyl)propanoate **4a** (40.8 mg, 0.200 mmol, 1.00 equiv) and dry 1,4-Dioxane (1.00 mL) was added under N_2 atmosphere and the mixture was allowed to stir at room temperature for 12 h. When the reaction completed, the crude mixture

was monitored by GC-MS.

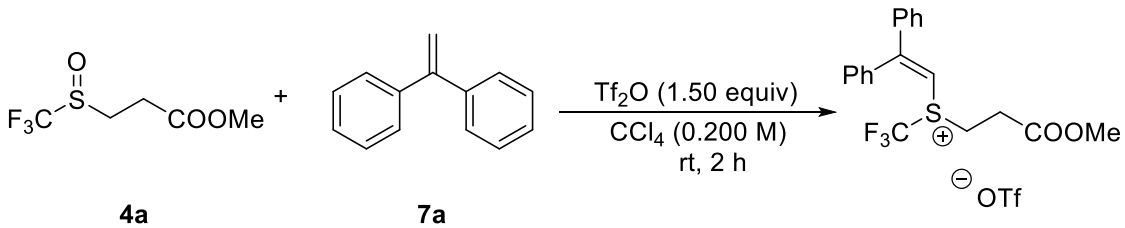








5. Observation of intermediate for the reaction between 7a or 9a with reagent 4a

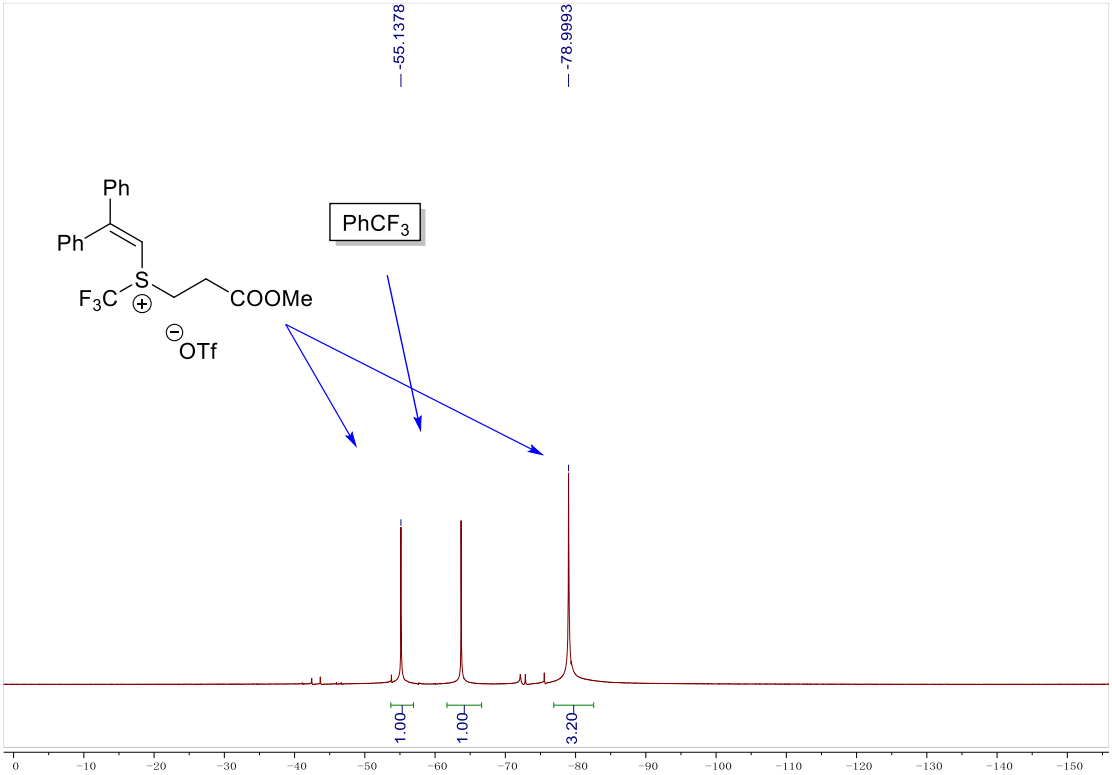


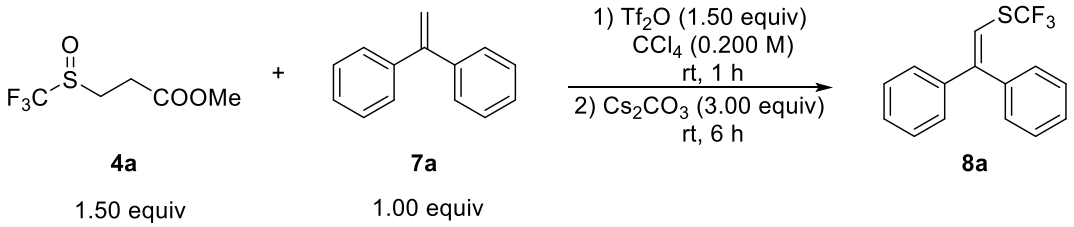
4a
1.50 equiv

7a
1.00 equiv

An oven-dried 25-mL glass schlenck, equipped with a stirring bar, and was evacuated and backfilled with nitrogen for three times, and were charged with 1,1-diphenylethylene (36.1 mg, 0.200 mmol, 1.00 equiv), methyl 3-((trifluoromethyl)sulfinyl)propanoate (61.3 mg, 0.300 mmol, 1.50 equiv), CCl_4

(1.00 mL) was added under N_2 atmosphere. Then the Tf_2O (84.6 mg, 0.300 mmol, 1.50 equiv) was added dropwise to the mixture, and the mixture was allowed to stir at room temperature for 2 h. The crude mixture was subjected to ^{19}F NMR spectroscopy in the presence of $PhCF_3$ (99.0 wt%, 29.5 mg, 0.200 mmol) as an internal standard.





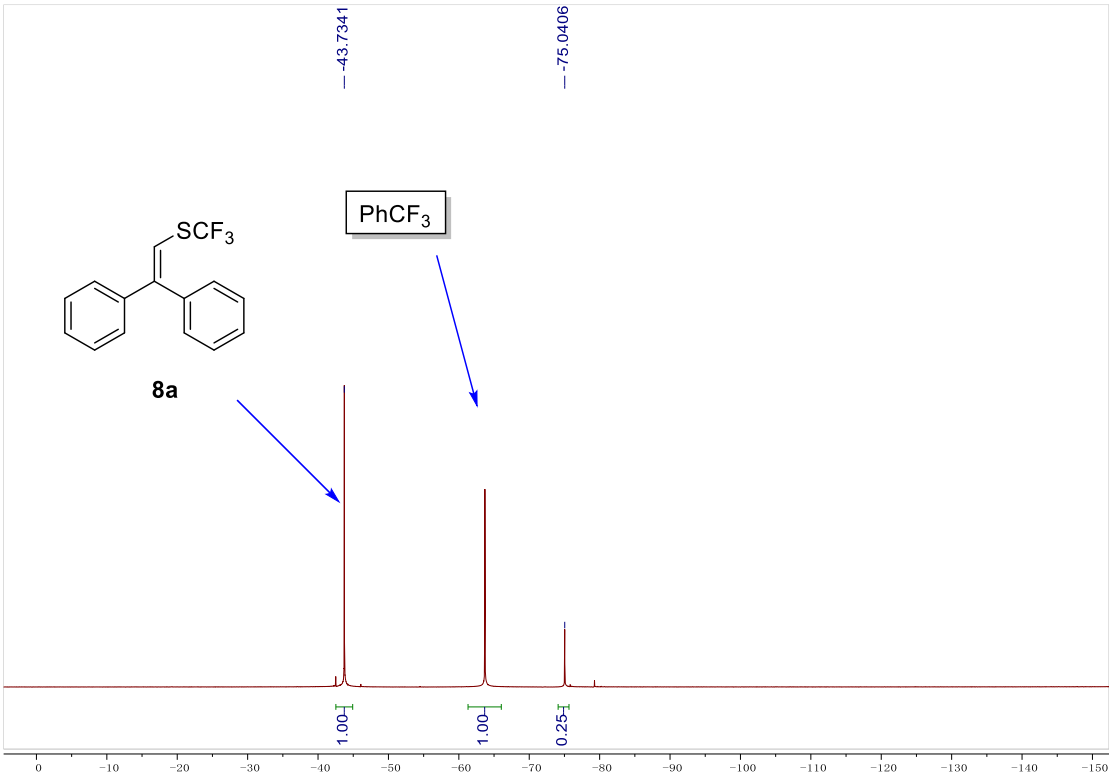
4a 1.50 equiv

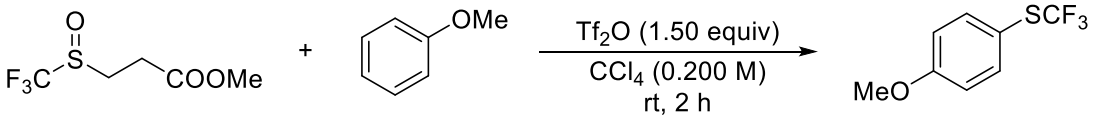
7a 1.00 equiv

8a

An oven-dried 25-mL glass schlenck, equipped with a stirring bar, and was evacuated and backfilled with nitrogen for three times, and were charged with 1,1-diphenylethylene (36.1 mg, 0.200 mmol, 1.00 equiv), methyl 3-((trifluoromethyl)sulfinyl)propanoate (61.3 mg, 0.300 mmol, 1.50 equiv), CCl_4 (1.00 mL) was added under N_2 atmosphere. Then the Tf_2O (84.6 mg, 0.300 mmol, 1.50 equiv) was added dropwise to the mixture, and the mixture was allowed to stir at room temperature for 2 h. Then Cs_2CO_3 (97.7 mg, 0.300 mmol, 3.00 equiv) was added under N_2 atmosphere, the mixture was allowed to stir at room temperature for 6 h. When the reaction completed, the crude mixture was subjected to ^{19}F NMR spectroscopy in

the presence of $PhCF_3$ (99.0 wt%, 29.5 mg, 0.200 mmol) as an internal standard.



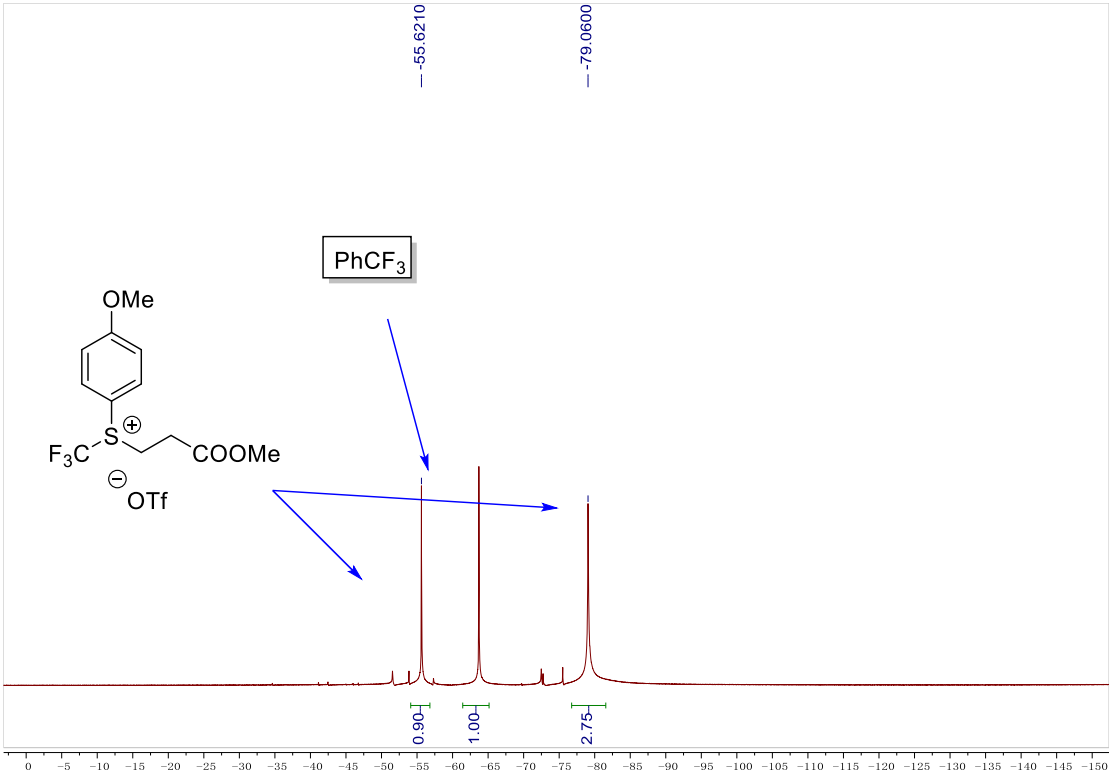


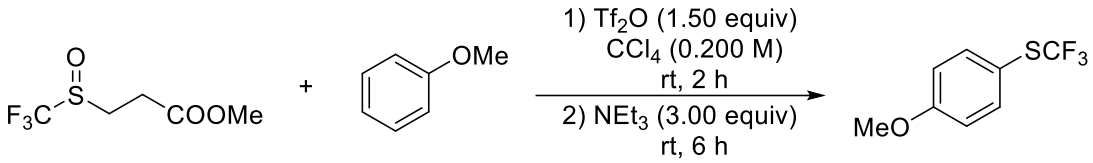
4a
1.50 equiv

9a
1.00 equiv

10a

An oven-dried 25-mL glass schlenck, equipped with a stirring bar, and was evacuated and backfilled with nitrogen for three times, and were charged with anisole (21.6 mg, 0.200 mmol, 1.00 equiv), methyl 3-((trifluoromethyl)sulfinyl)propanoate (61.3 mg, 0.300 mmol, 1.50 equiv), CCl_4 (1.00 mL) was added under N_2 atmosphere. Then Tf_2O (84.6 mg, 0.300 mmol, 1.50 equiv) was added dropwise to the mixture, and the mixture was allowed to stir at room temperature for 2 h. The crude mixture was subjected to ^{19}F NMR spectroscopy in the presence of $PhCF_3$ (99.0 wt%, 29.5 mg, 0.200 mmol) as an internal standard.



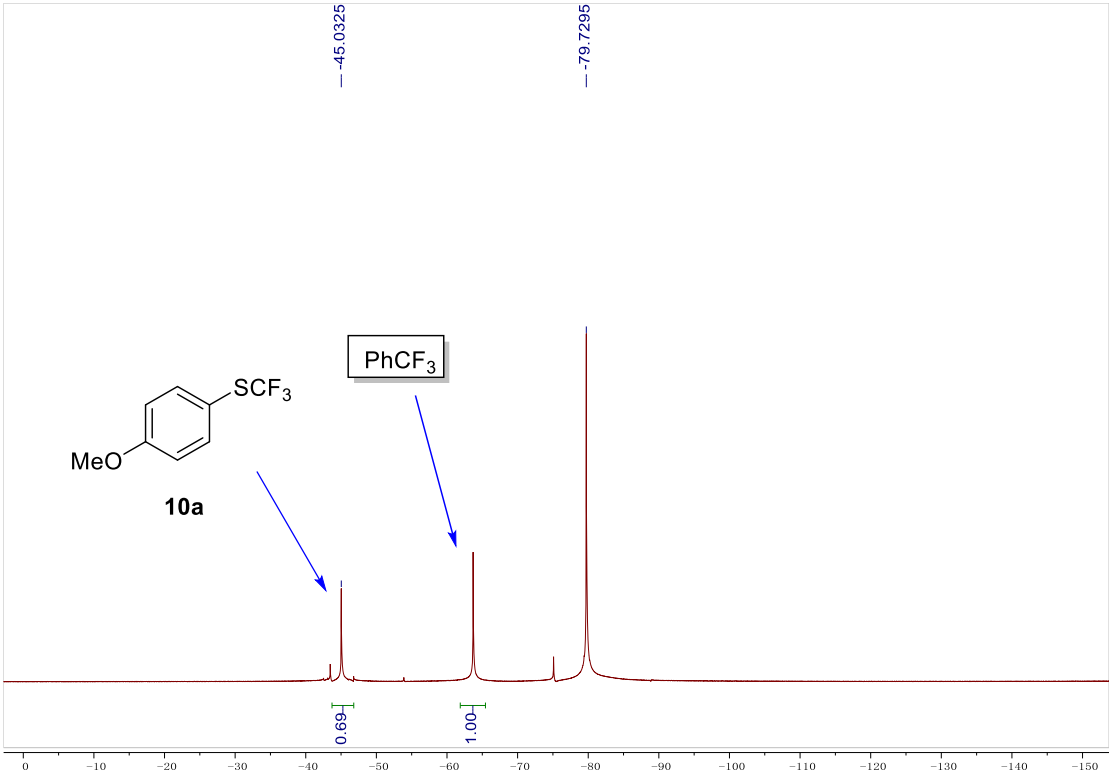


4a 1.50 equiv

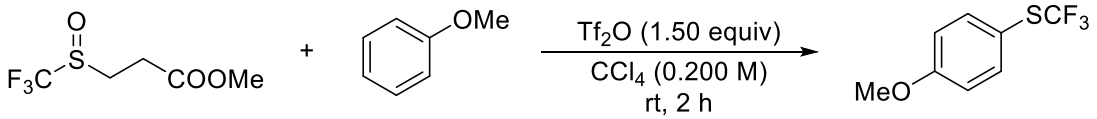
9a 1.00 equiv

10a

An oven-dried 25-mL glass schlenck, equipped with a stirring bar, and was evacuated and backfilled with nitrogen for three times, and were charged with anisole (21.6 mg, 0.200 mmol, 1.00 equiv), methyl 3-((trifluoromethyl)sulfinyl)propanoate (61.3 mg, 0.300 mmol, 1.50 equiv), CCl_4 (1.00 mL) was added under N_2 atmosphere. Then Tf_2O (84.6 mg, 0.300 mmol, 1.50 equiv) was added dropwise to the mixture, and the mixture was allowed to stir at room temperature for 2 h. Then Et_3N (60.7 mg, 0.600 mmol, 3.00 equiv) was added to the mixture under N_2 atmosphere, the mixture was allowed to stir at room temperature for 6 h. When the reaction completed, the crude mixture was subjected to ^{19}F NMR spectroscopy in the presence of $PhCF_3$ (99.0 wt%, 29.5 mg, 0.200 mmol) as an internal standard.



6. Observation of intermediate (3-methoxy-3-oxopropyl)(4-methoxyphenyl)(trifluoromethyl)sulfonium trifluoromethanesulfonate:



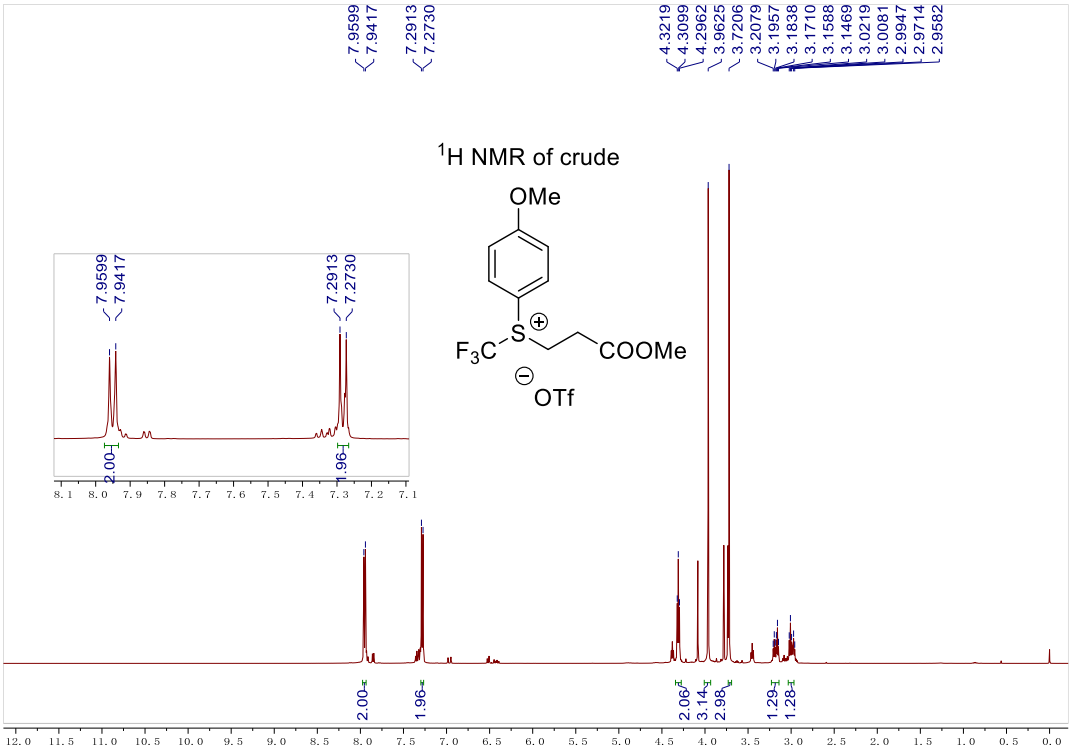
4a 1.50 equiv

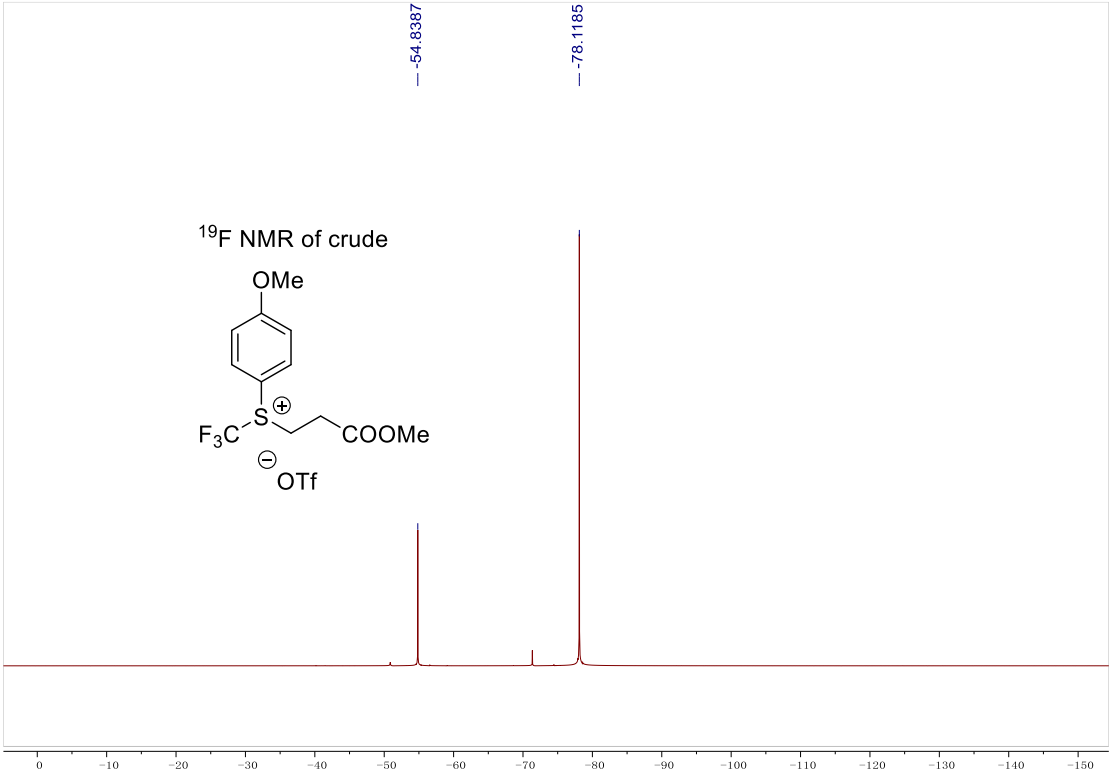
9a 1.00 equiv

10a

An oven-dried 25-mL glass schlenck, equipped with a stirring bar, and was evacuated and backfilled with nitrogen for three times, and were charged with anisole (21.6 mg, 0.200 mmol, 1.00 equiv), methyl 3-((trifluoromethyl)sulfinyl)propanoate (61.3 mg, 0.300 mmol, 1.50 equiv), CCl_4 (1.00 mL) was added under N_2 atmosphere. Then Tf_2O (84.6 mg, 0.300 mmol, 1.50 equiv) was added dropwise to the mixture, and the mixture was allowed to stir at room temperature for 2 h. Then poured out the colorless liquid of the mixture, the black residue was washed by CCl_4 (3.00 mL × 3). The black residue was redissolved in the $CDCl_3$ to give the crude ^1H NMR and ^{19}F NMR spectroscopy .**^{1}H**

NMR (500 MHz, CDCl3) δ 7.95 (2H, d, *J* = 9.1 Hz), 7.28 (2H, d, *J* = 9.2 Hz), 4.31 (2H, t, *J* = 6.4 Hz), 3.96 (3H, s), 3.72 (3H, s), 3.21 – 3.15 (1H, m), 3.03 – 2.97 (1H, m). ^{19}F NMR (471 MHz, CDCl3) δ –54.8, –78.1.





VII. References

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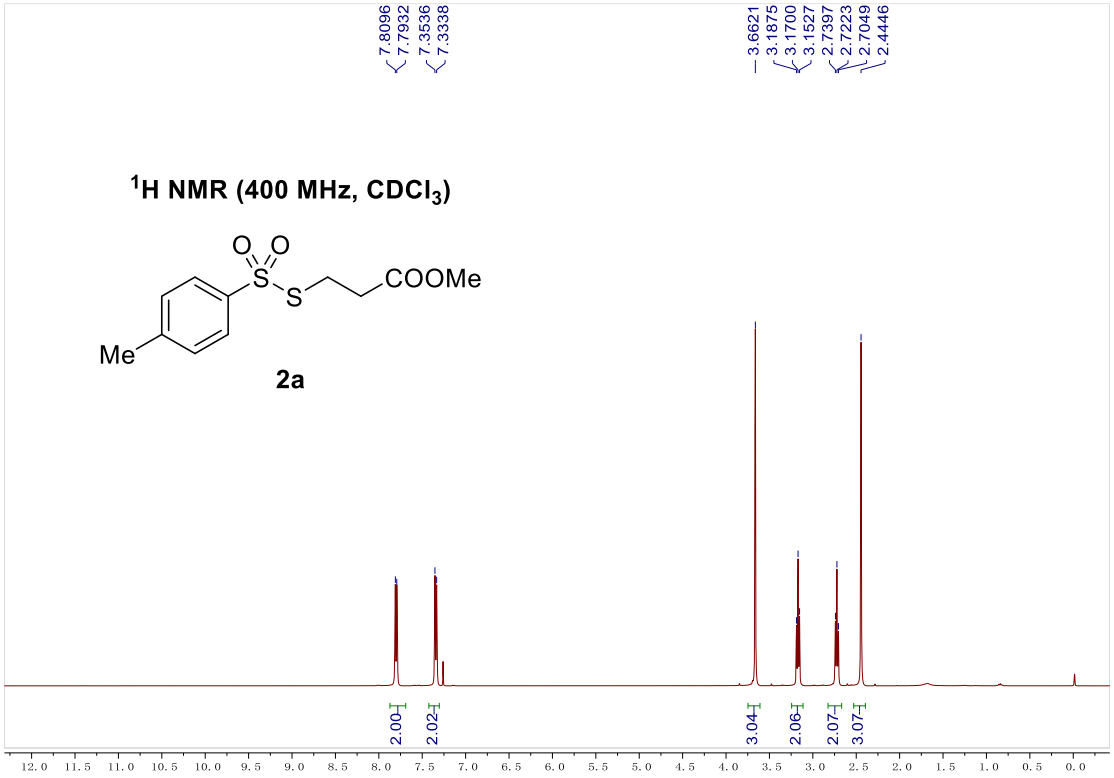
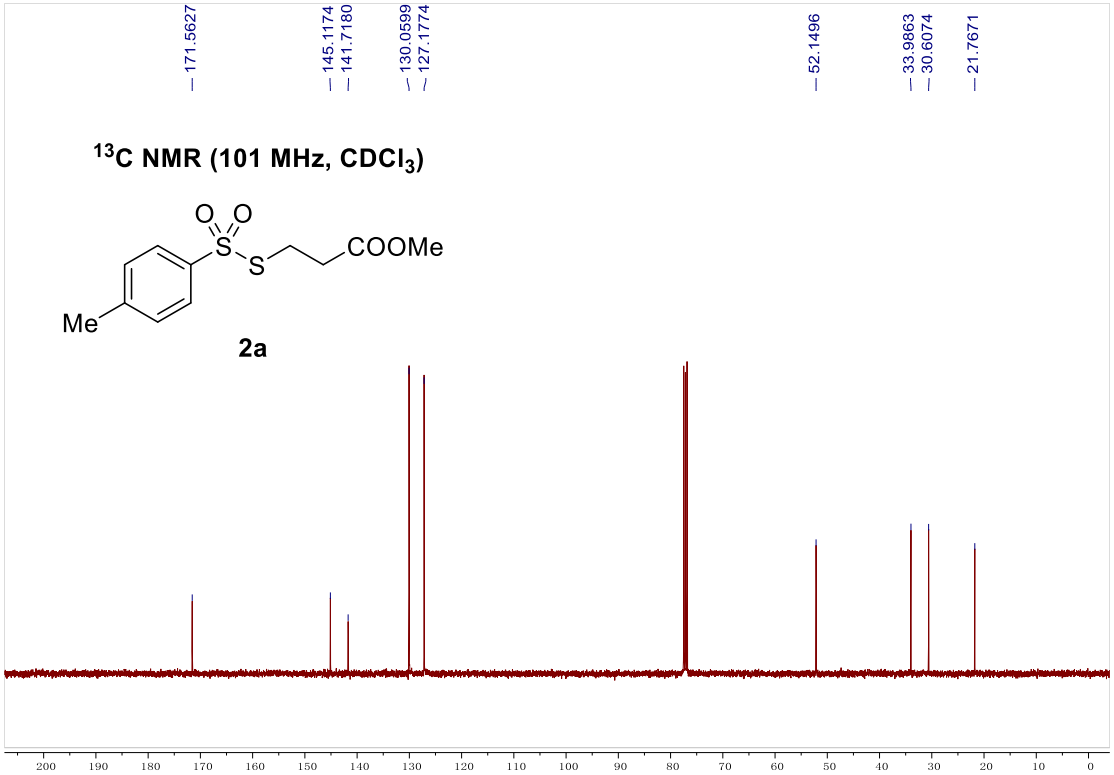
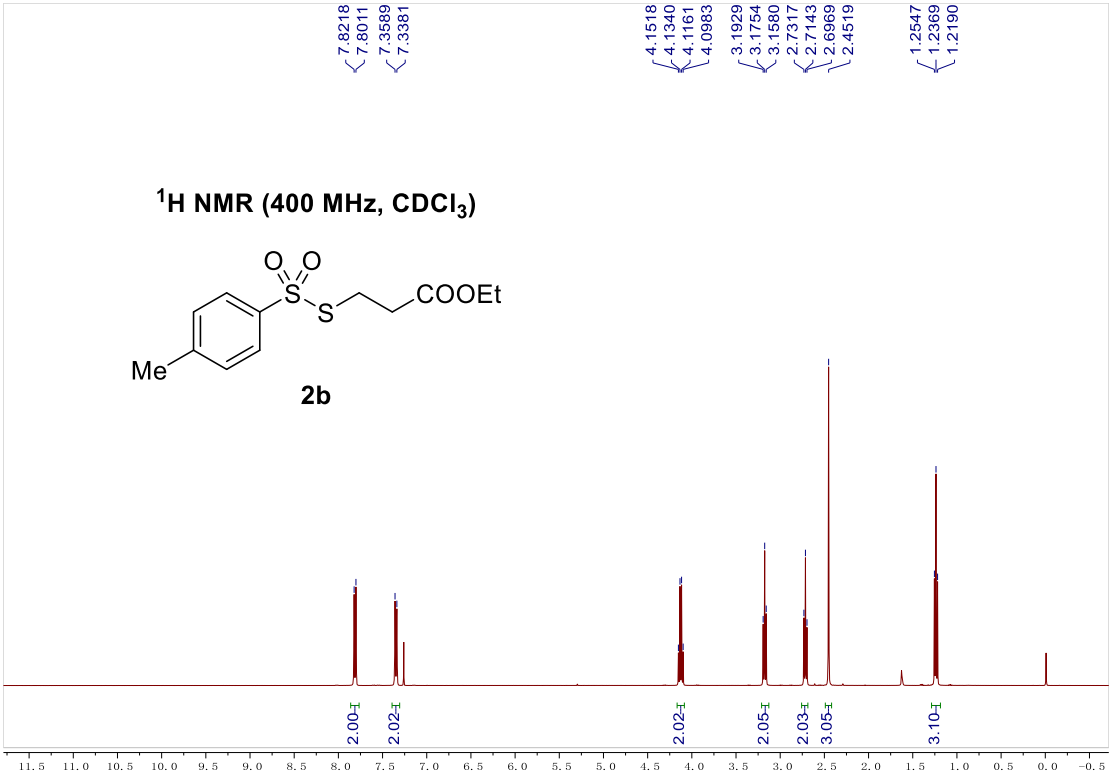
VIII. NMR spectra



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7.3381
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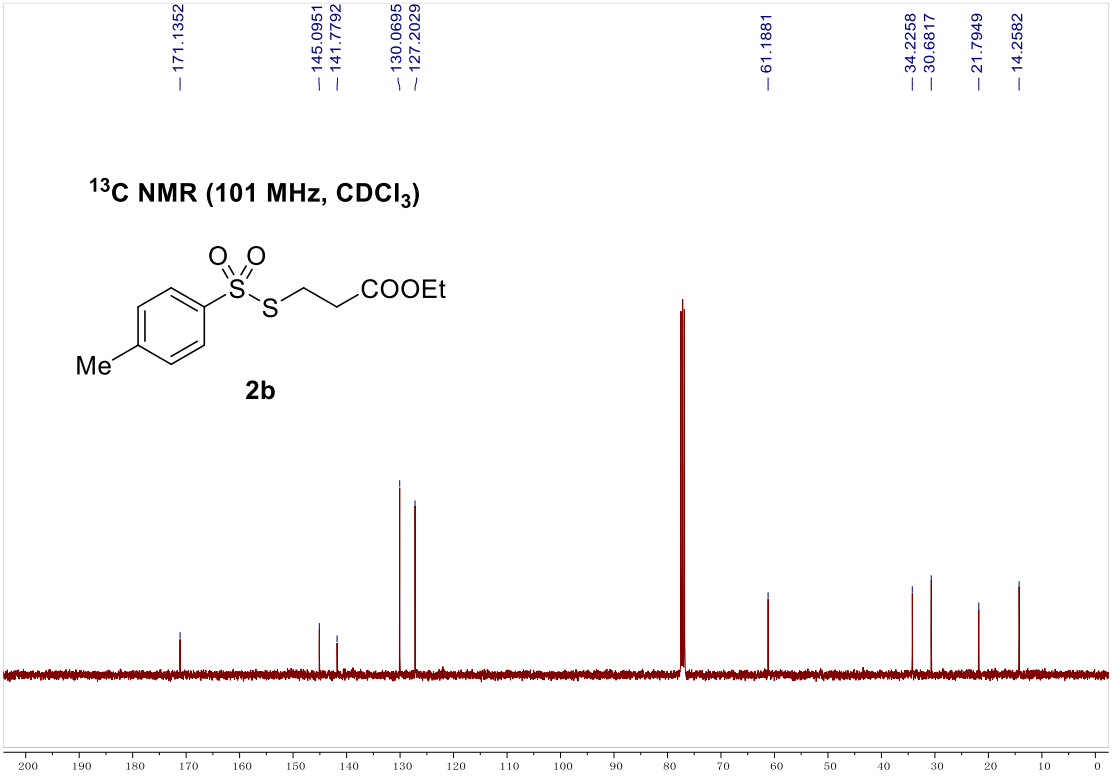
4.1340
4.1161
4.0983
3.1929
3.1754
3.1580
2.7317
2.7143
2.6969
2.4519
1.2547
1.2369
1.2190
1H NMR (400 MHz, CDCl3)
O
O
S
S
COOEt
Me
2b
2.00
2.02
2.02
2.05
2.03
3.05
3.10
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5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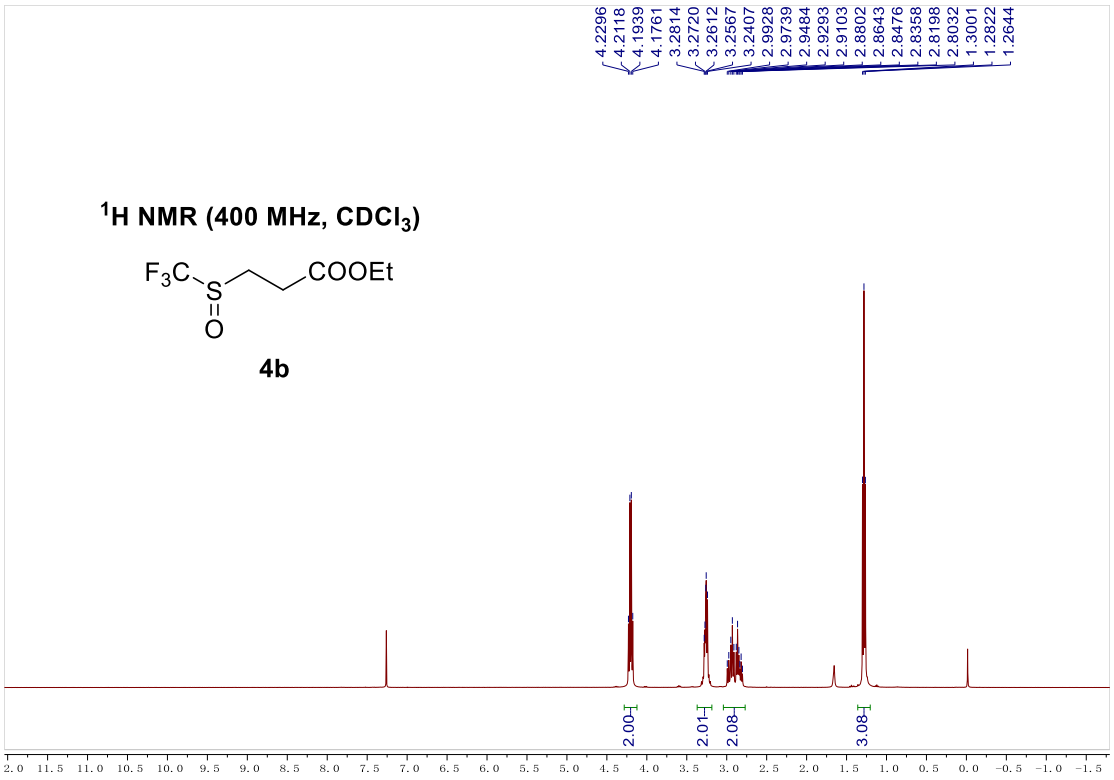
145.0951
141.7792
130.0695
127.2029
61.1881
34.2258
30.6817
21.7949
14.2582
13C NMR (101 MHz, CDCl3)
O
O
S
S
COOEt
Me
2b
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0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4.1939
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3.2612
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3.2407
2.9928
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2.9484
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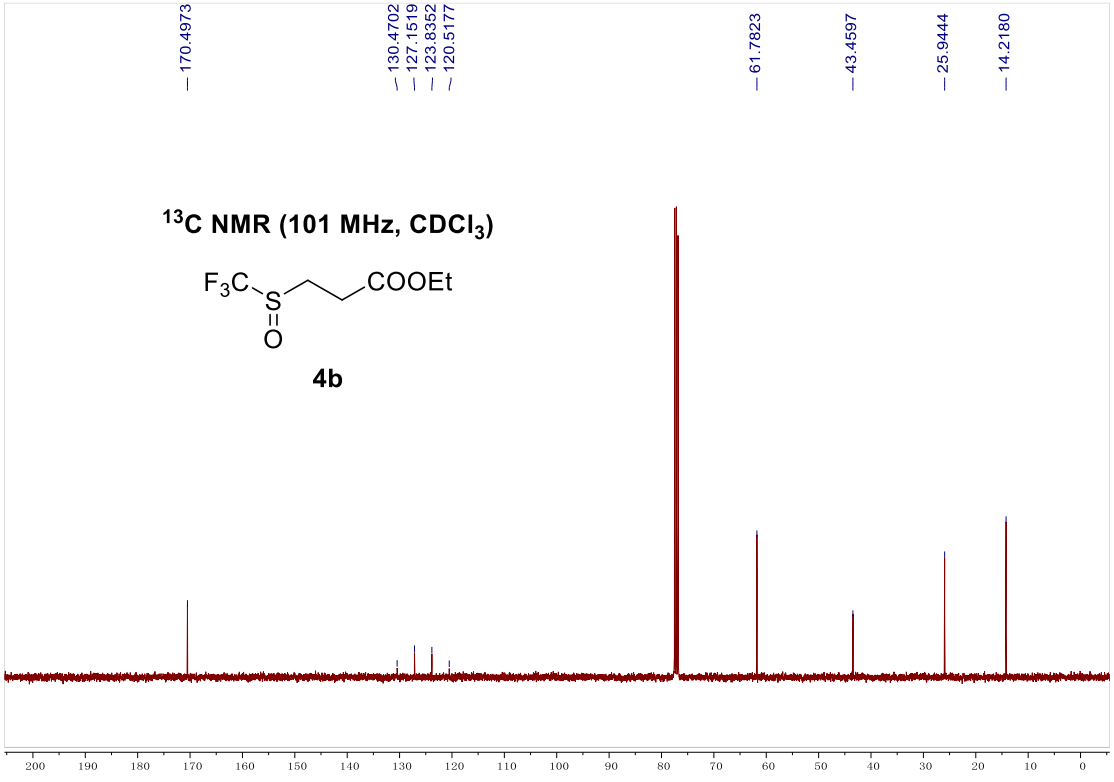
2.8358
2.8198
2.8032
1.3001
1.2822
1.2644
1H NMR (400 MHz, CDCl3)
F3C
S
O
COOEt
4b
2.00
2.01
2.08
3.08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123.8352
120.5177
61.7823
43.4597
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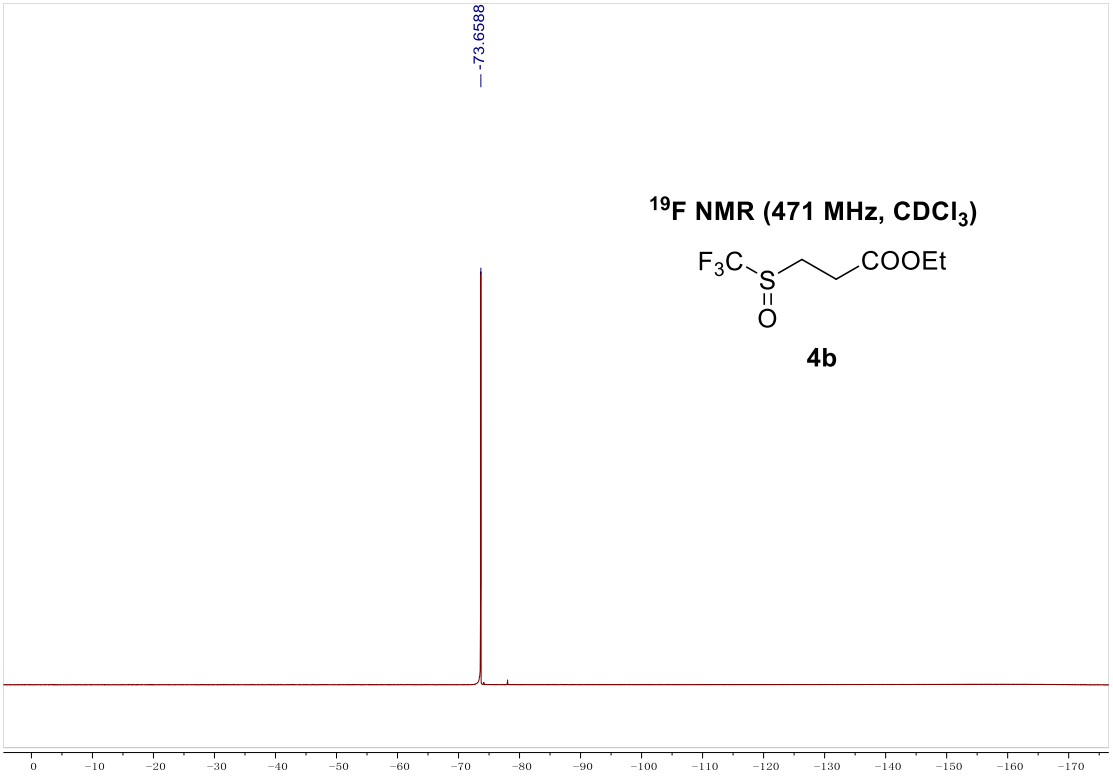
14.2180
13C NMR (101 MHz, CDCl3)
F3C
S
O
COOEt
4b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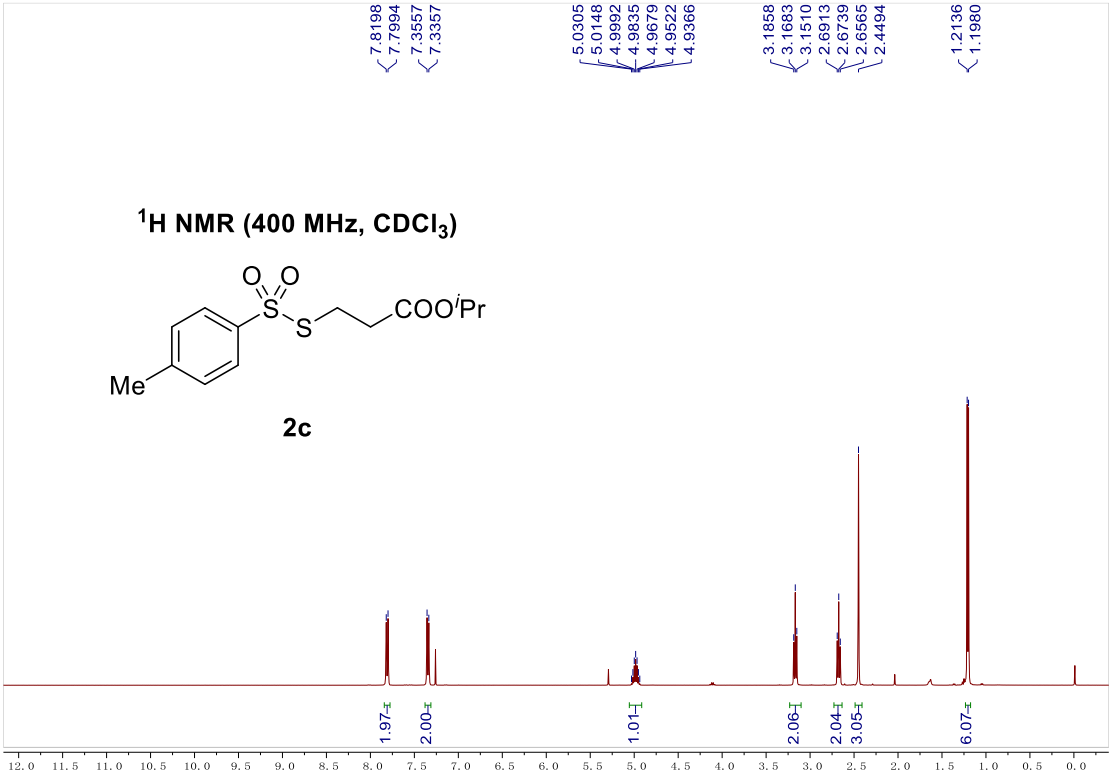
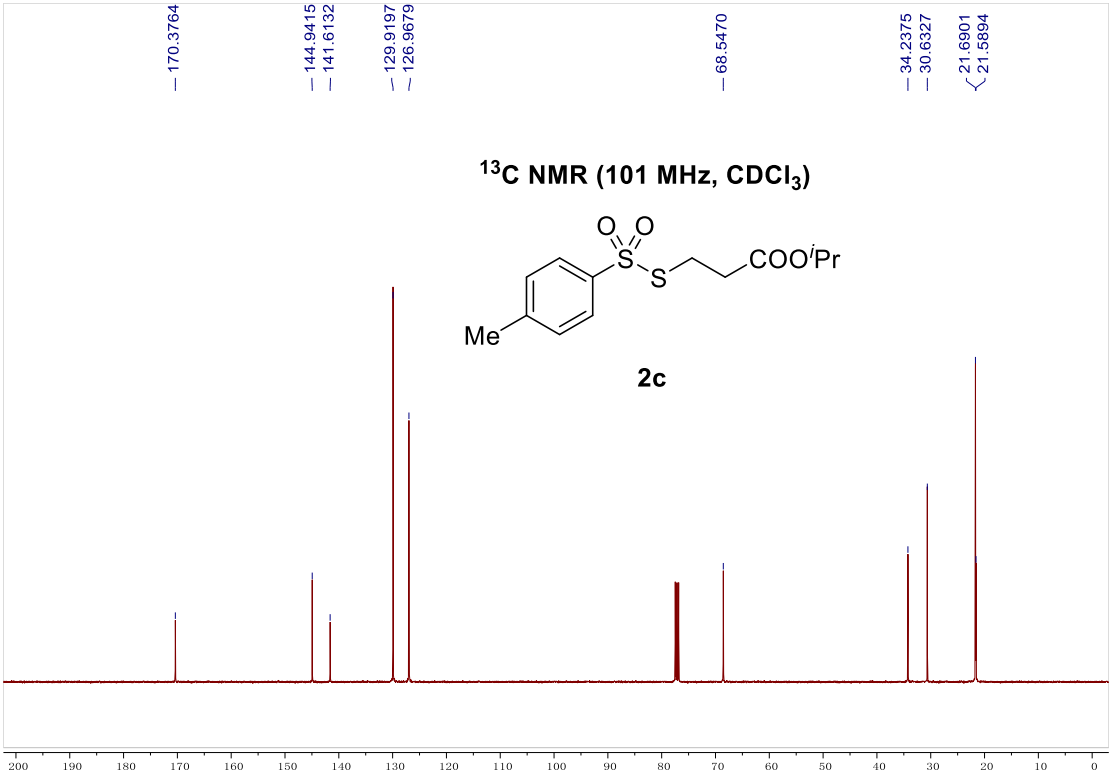
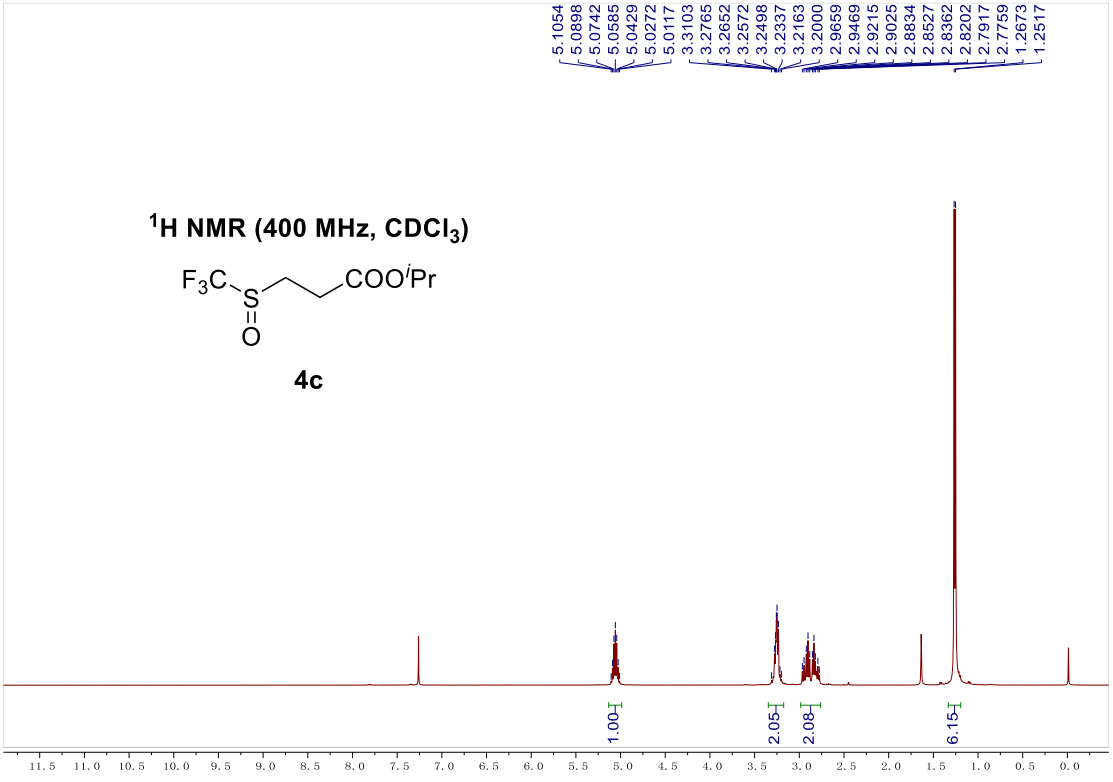
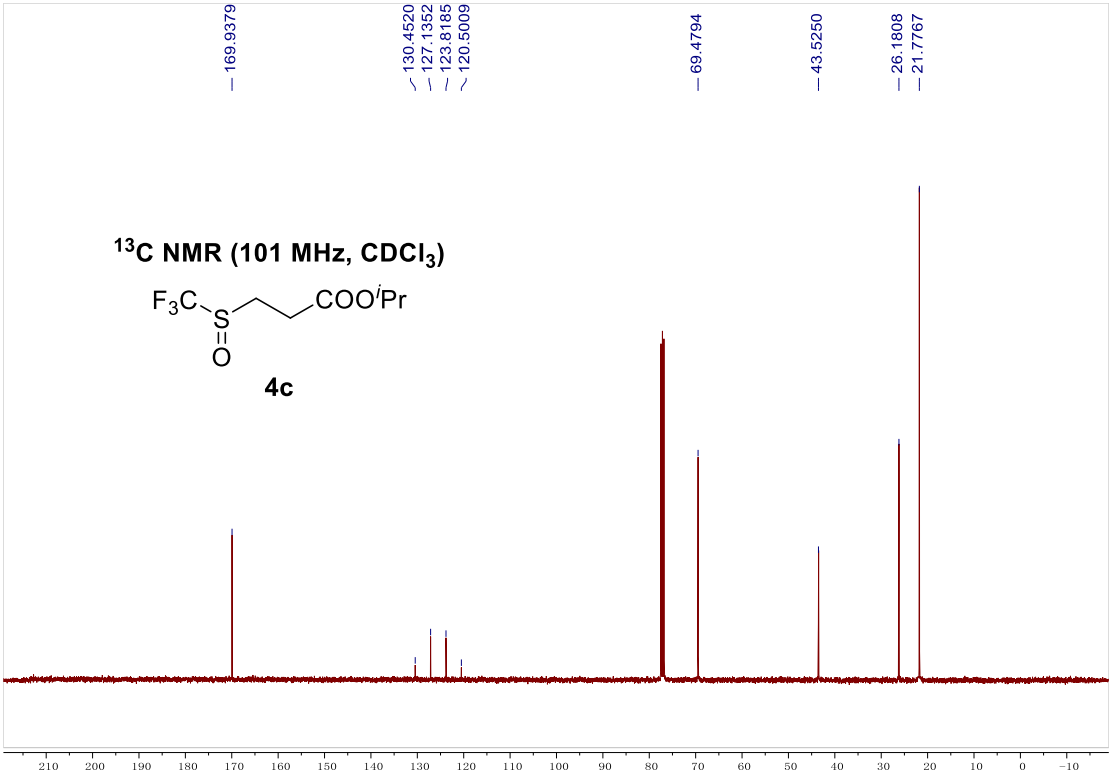
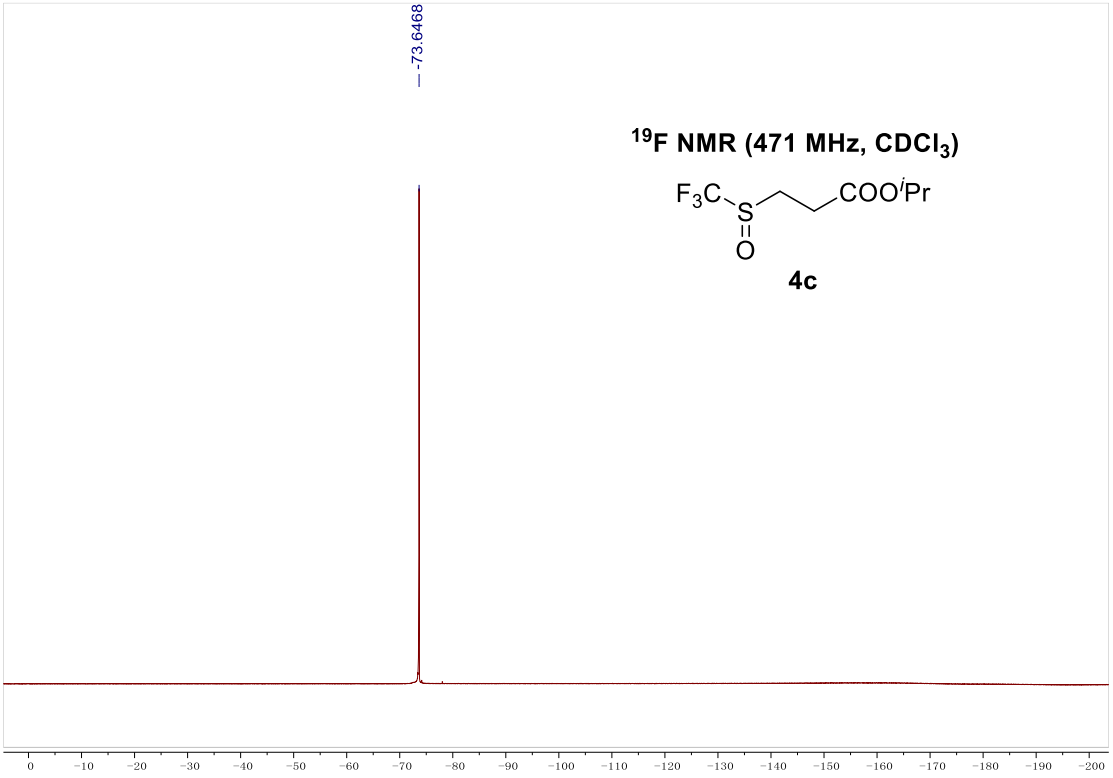
19F NMR (471 MHz, CDCl3)
F3C
S
O
COOEt
4b

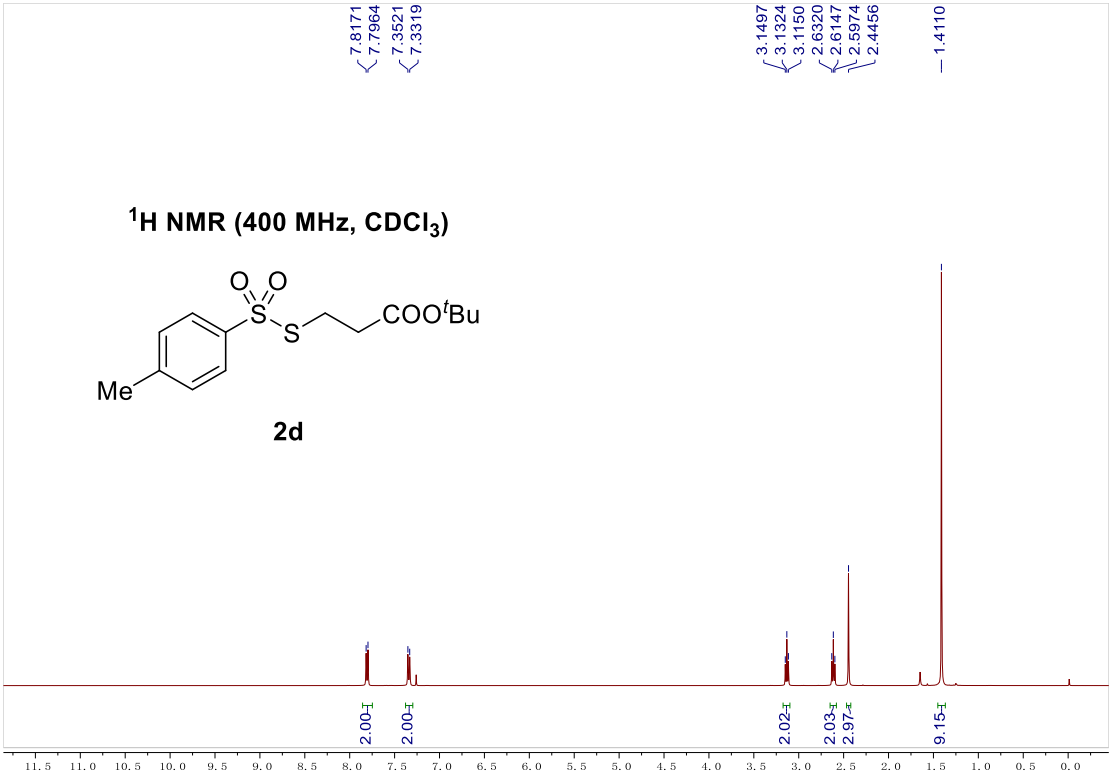

7.8198
7.7994
7.3557
7.3357
5.0305
5.0148
4.9992
4.9835
4.9679
4.9522
4.9366
3.1858
3.1683
3.1510
2.6913
2.6739
2.6565
2.4494
1.2136
1.1980
1H NMR (400 MHz, CDCl3)
O
O
S
S
COOiPr
Me
2c
1.97
2.00
1.01
2.06
2.04
3.05
6.07


170.3764
144.9415
141.6132
129.9197
126.9679
68.5470
34.2375
30.6327
21.6901
21.5894
13C NMR (101 MHz, CDCl3)
O
O
S
S
COOiPr
Me
2c


5.1054
5.0898
5.0742
5.0585
5.0429
5.0272
5.0117
3.3103
3.2765
3.2652
3.2572
3.2498
3.2337
3.2163
3.2000
2.9659
2.9469
2.9215
2.9025
2.8834
2.8527
2.8362
2.8202
2.7917
2.7759
2.2673
1.2517
1H NMR (400 MHz, CDCl3)
F3C
S
O
COOiPr
4c
1.00
2.05
2.08
6.15

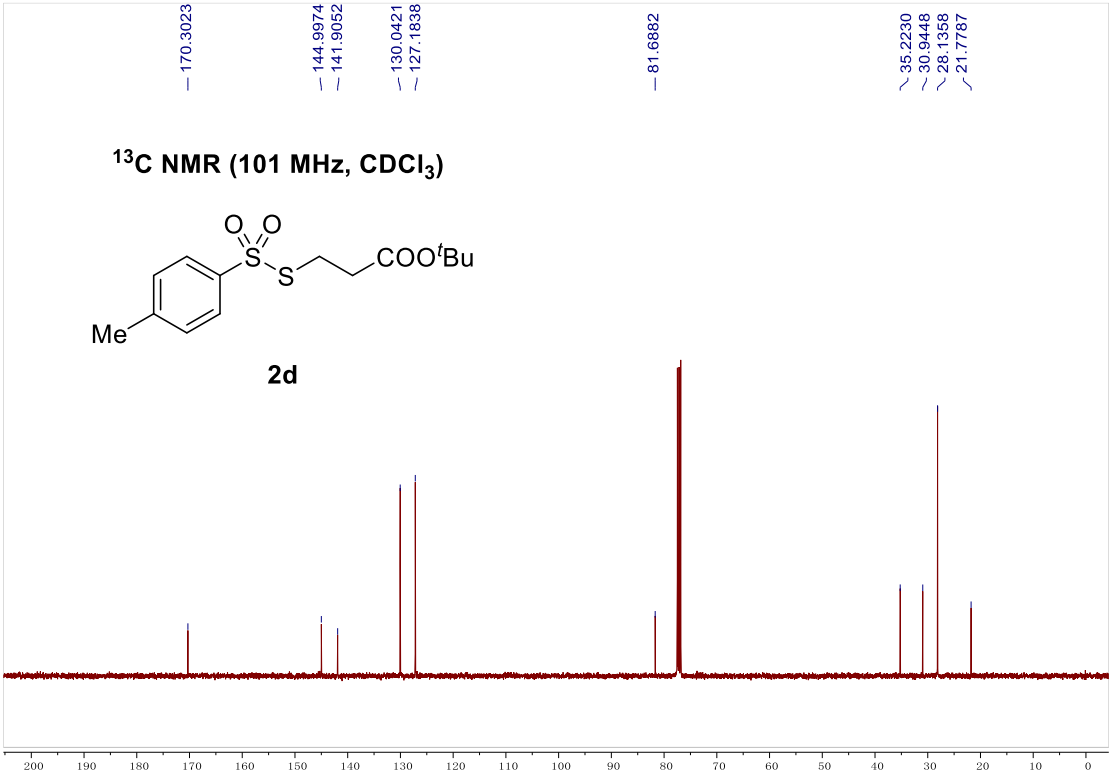

169.9379
130.4520
127.1352
123.8185
120.5009
69.4794
43.5250
26.1808
21.7767
13C NMR (101 MHz, CDCl3)
F3C
S
O
COOiPr
4c


-73.6468
19F NMR (471 MHz, CDCl3)
F3C
S
O
COOiPr
4c



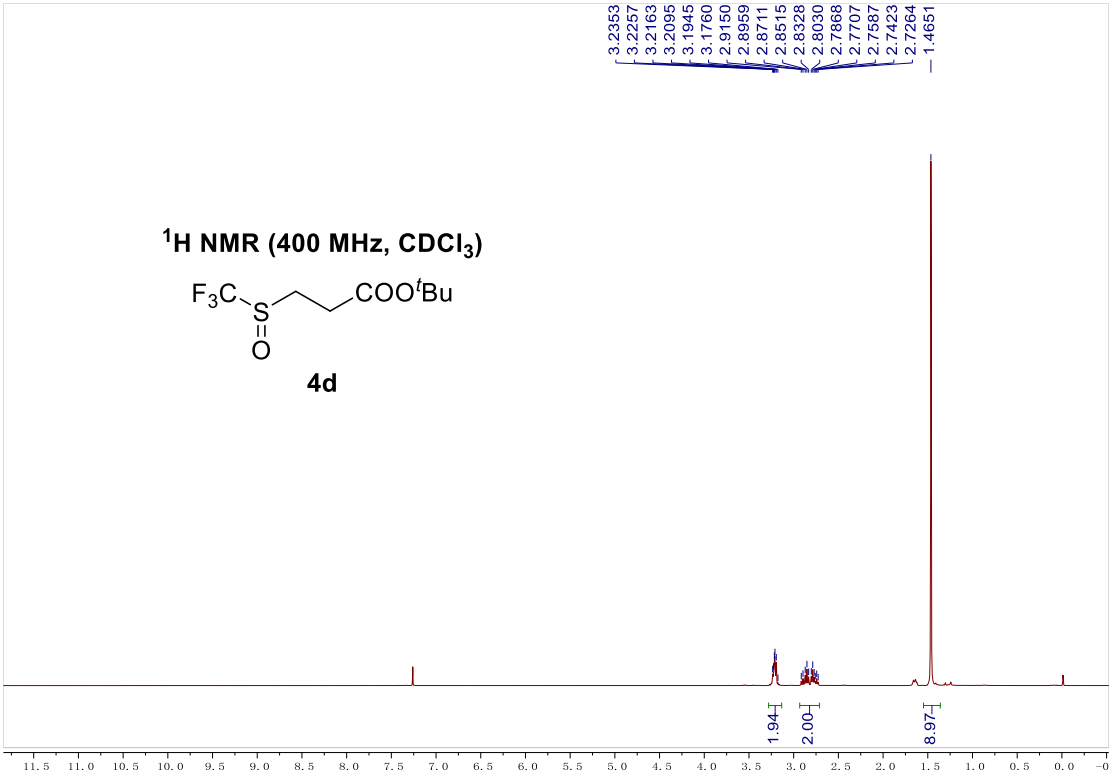
^{1}H NMR (400 MHz, $CDCl_3$)

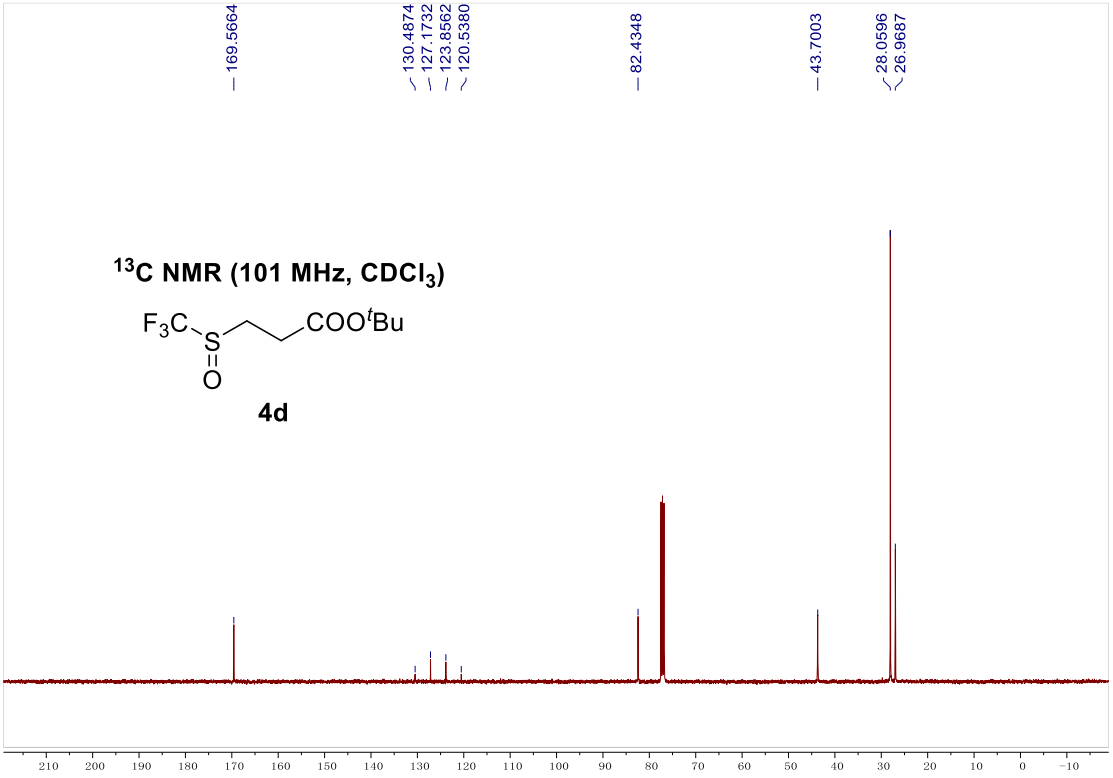
2d

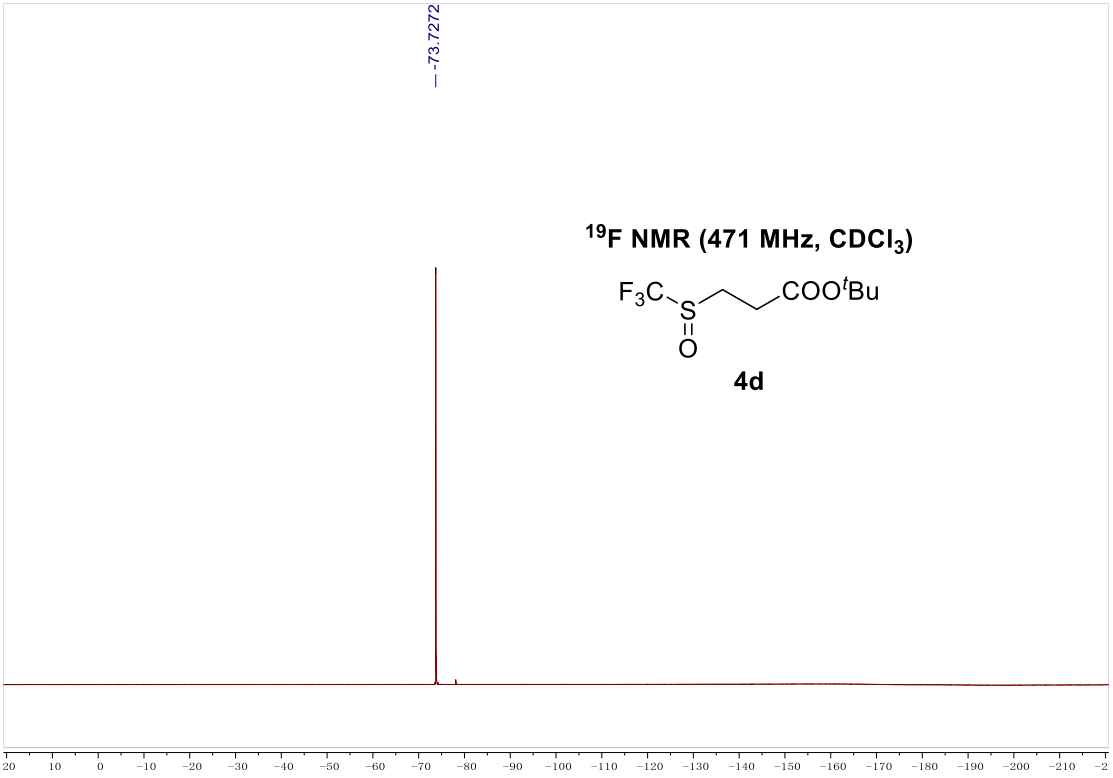


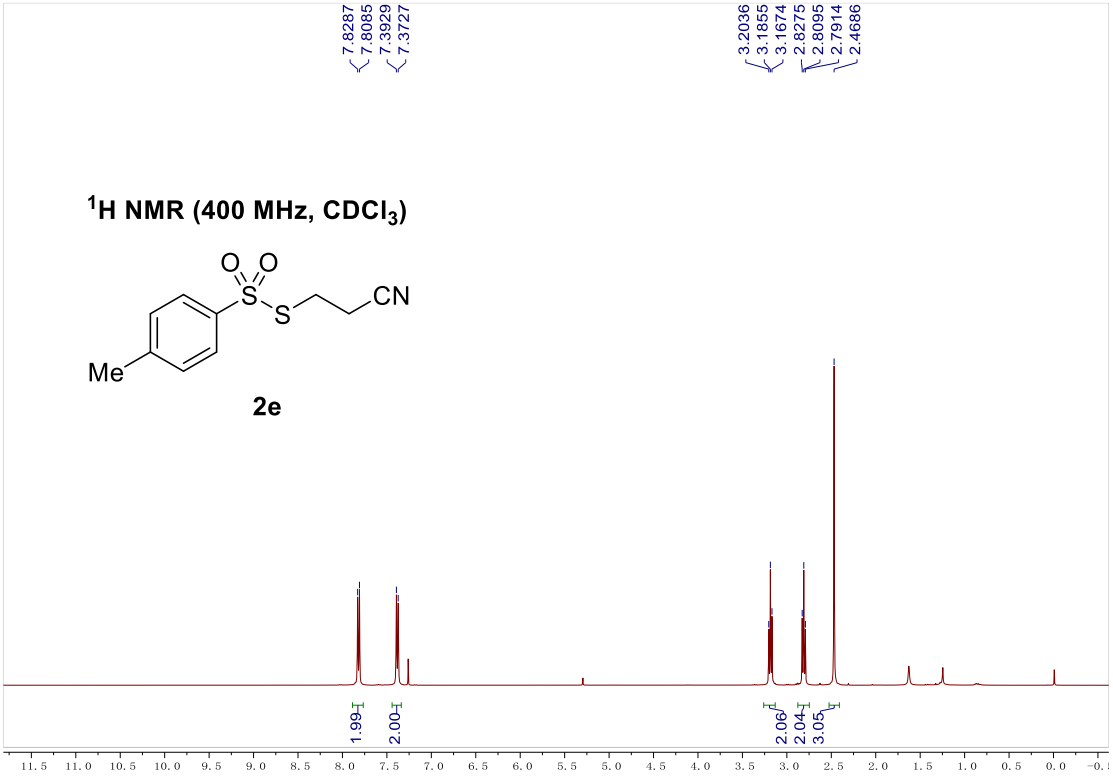
^{13}C NMR (101 MHz, $CDCl_3$)

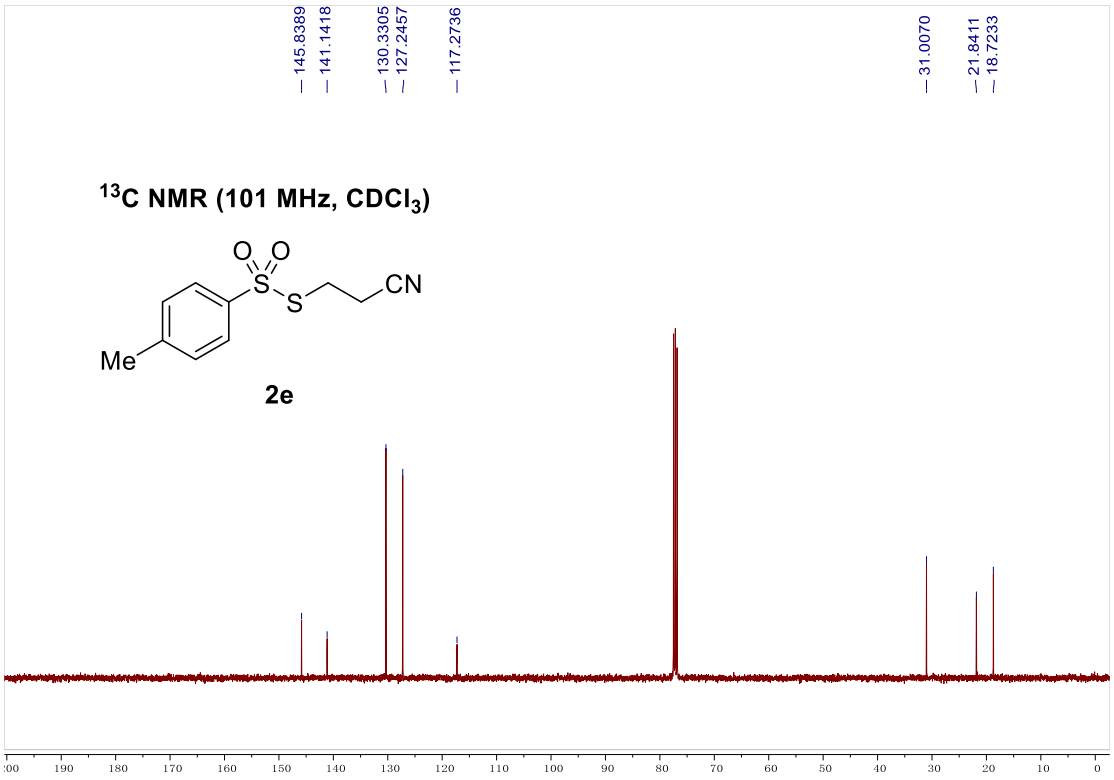
2d

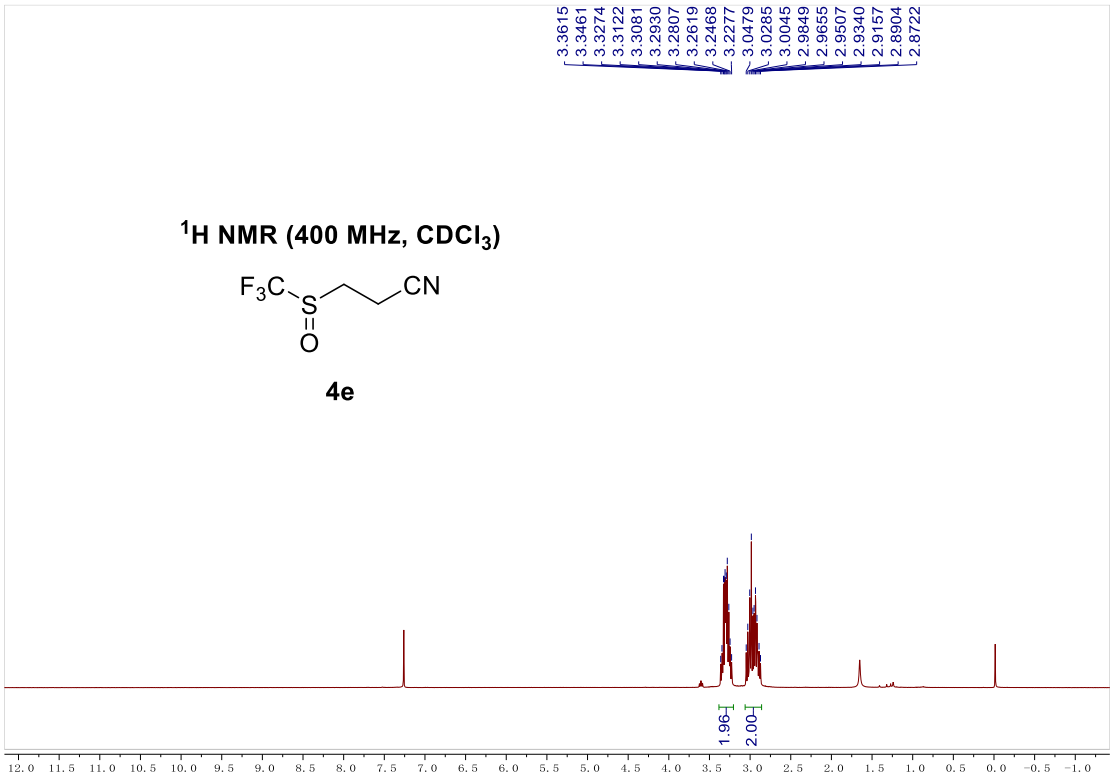

3.2353
3.2257
3.2163
3.2095
3.1945
3.1760
2.9150
2.8959
2.8711
2.8515
2.8328
2.8030
2.7868
2.7707
2.7587
2.7423
2.7264
1.4651
1H NMR (400 MHz, CDCl3)
F3C
S
O
COOtBu
4d
1.94
2.00
8.97

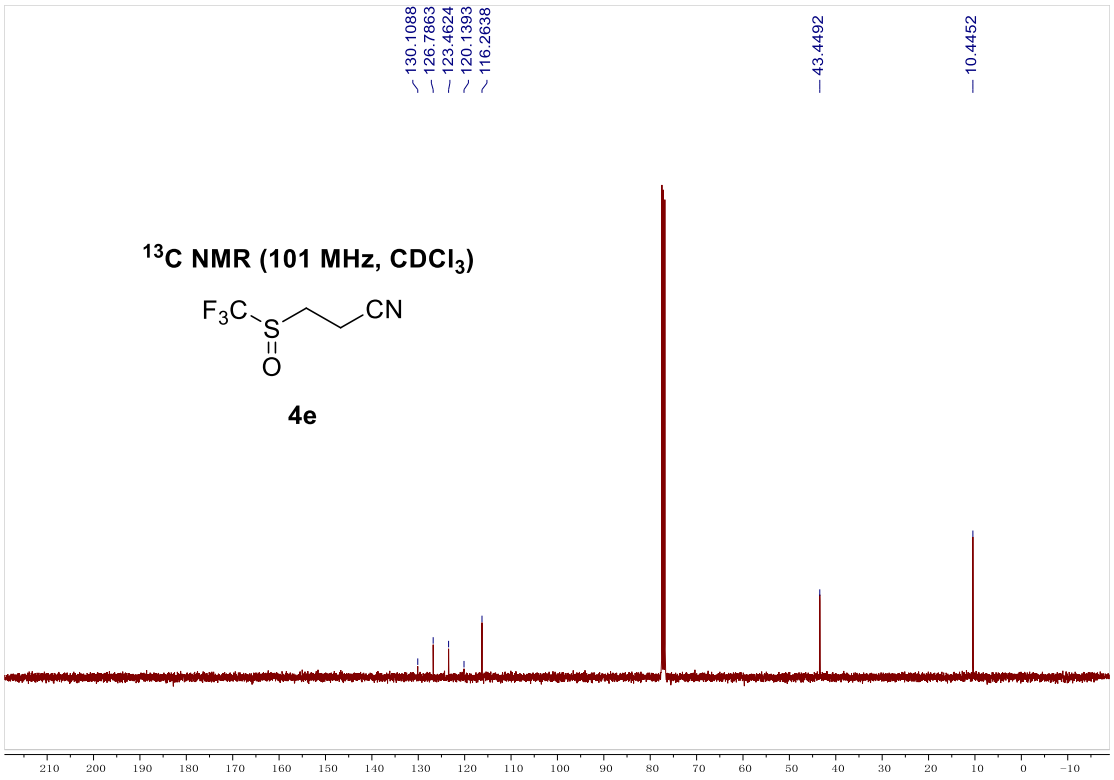

169.5664
130.4874
127.1732
123.8562
120.5380
82.4348
43.7003
28.0596
26.9687
13C NMR (101 MHz, CDCl3)
F3C
S
O
COOtBu
4d

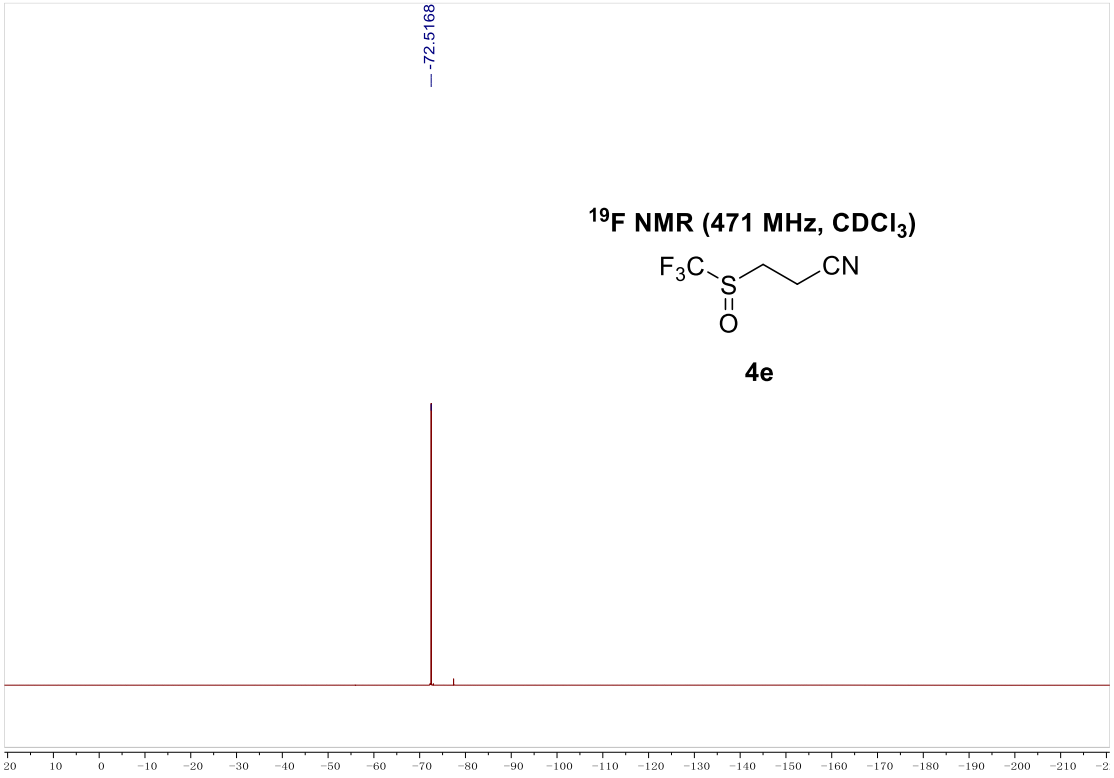

-73.7272
19F NMR (471 MHz, CDCl3)
F3C
S
O
COOtBu
4d

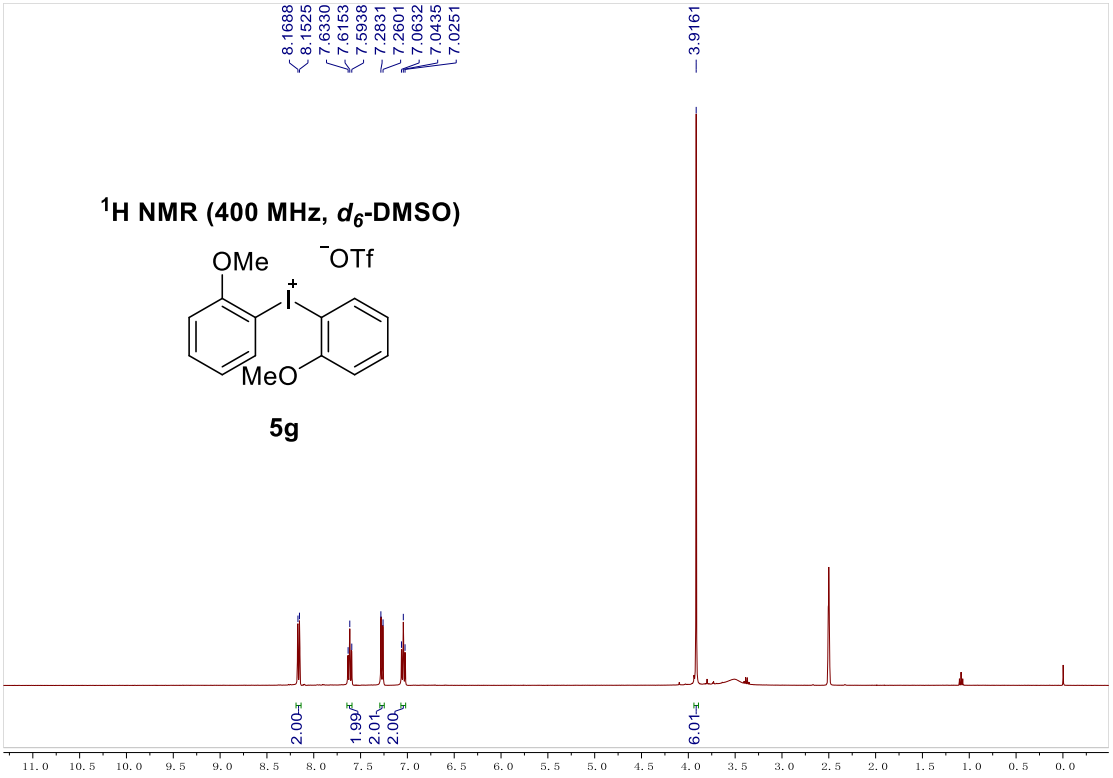

7.8287
7.8085
7.3929
7.3727
3.2036
3.1855
3.1674
2.8275
2.8095
2.7914
2.4686
1H NMR (400 MHz, CDCl3)
O
O
S
S
CN
Me
2e
1.99
2.00
2.06
2.04
3.05


145.8389
141.1418
130.3305
127.2457
117.2736
31.0070
21.8411
18.7233
13C NMR (101 MHz, CDCl3)
O
O
S
S
CN
Me
2e


3.3615
3.3461
3.3274
3.3122
3.3081
3.2930
3.2807
3.2619
3.2468
3.2277
3.0479
3.0285
3.0045
2.9849
2.9655
2.9507
2.9340
2.9157
2.8904
2.8722
1H NMR (400 MHz, CDCl3)
F3C
S
O
CN
4e
1.96
2.00

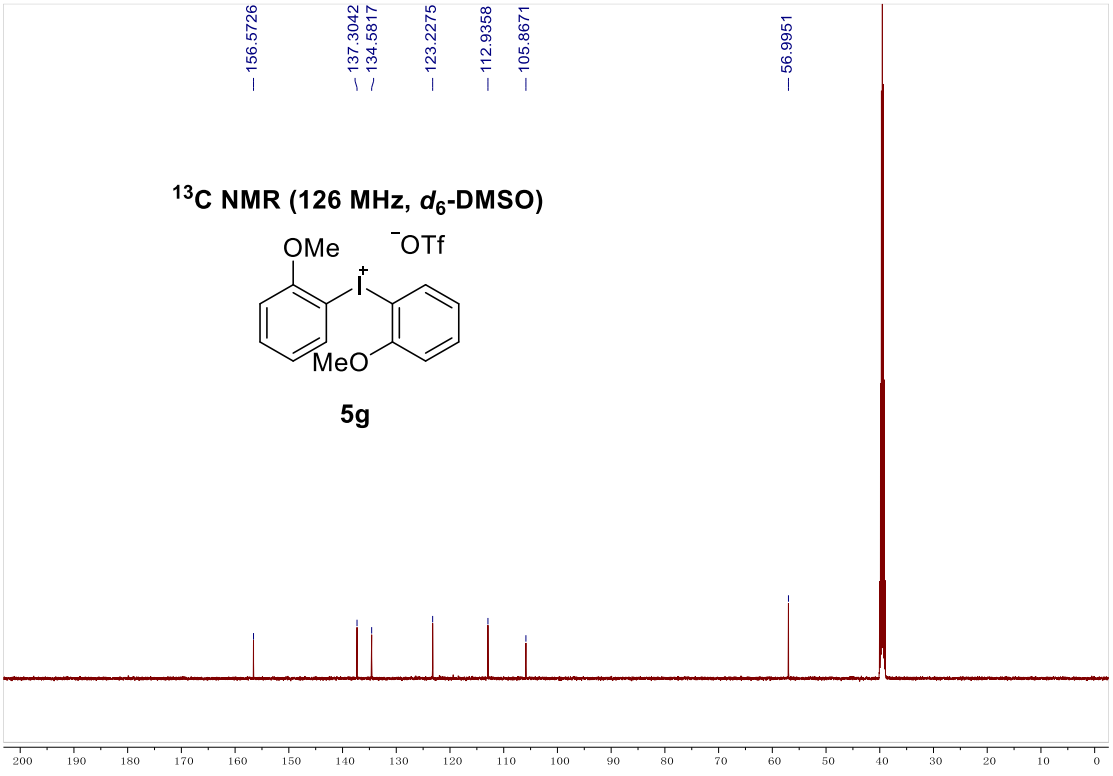

130.1088
126.7863
123.4624
120.1393
116.2638
43.4492
10.4452
13C NMR (101 MHz, CDCl3)
F3C
S
O
CN
4e


-72.5168
19F NMR (471 MHz, CDCl3)
F3C
S
O
CN
4e



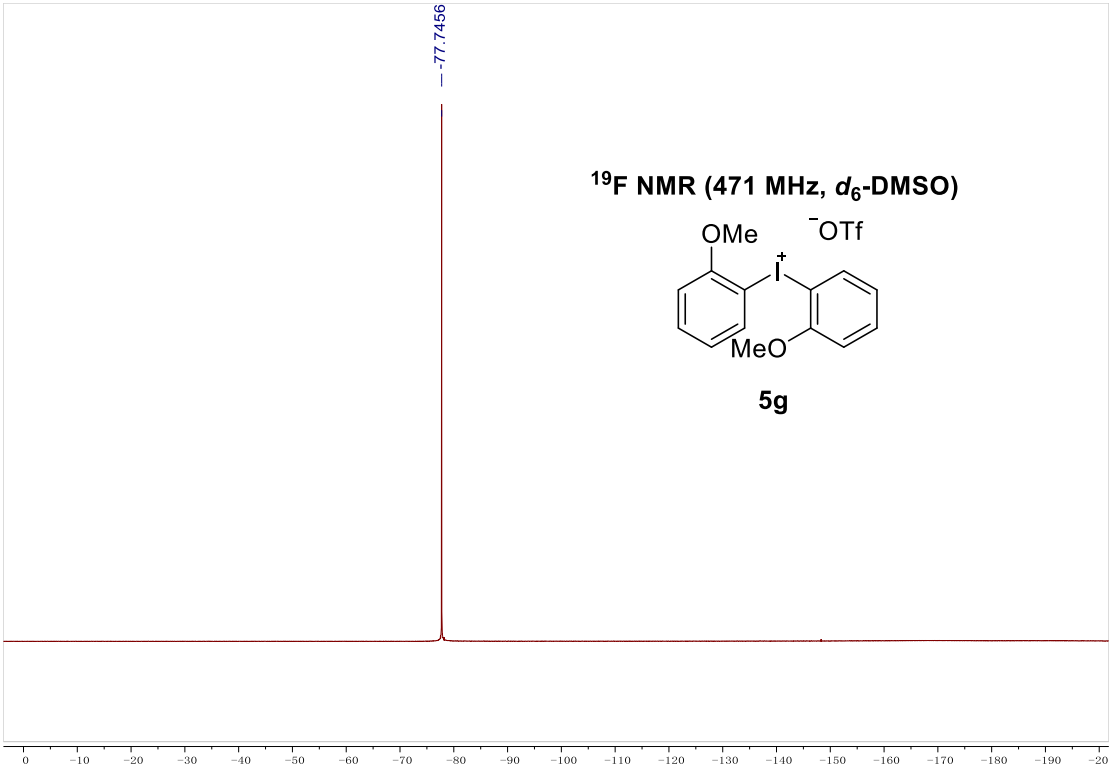
^{1}H NMR (400 MHz, d_6-DMSO)

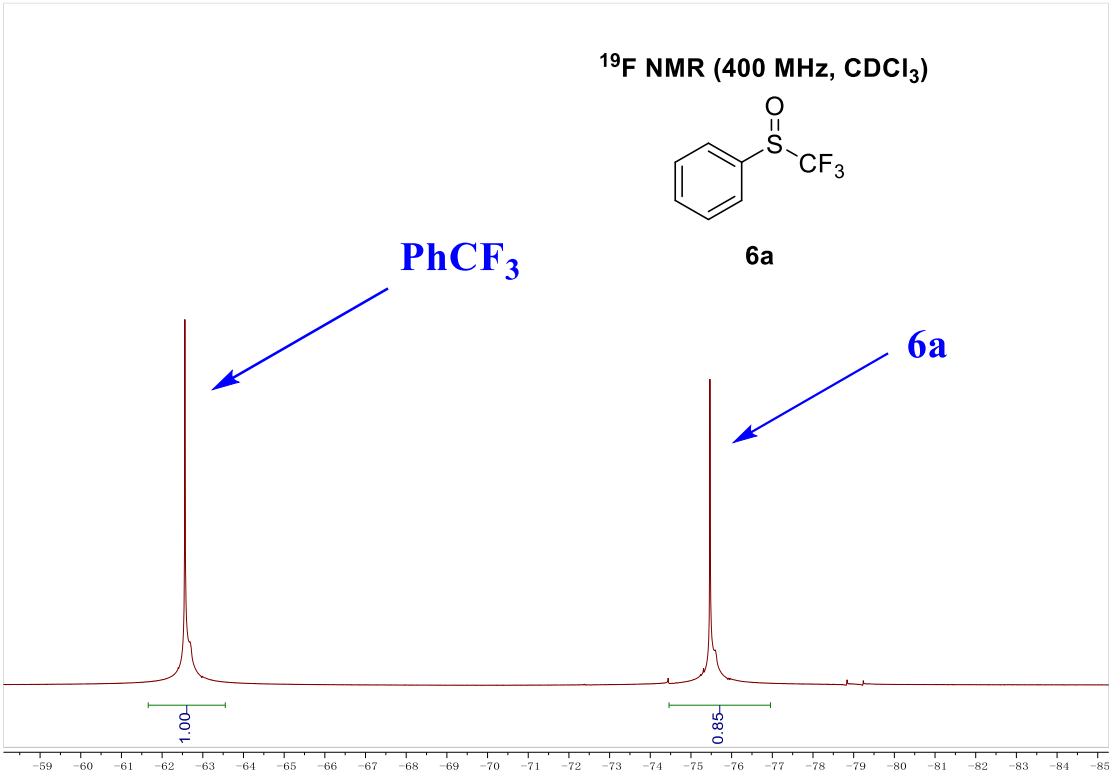
5g

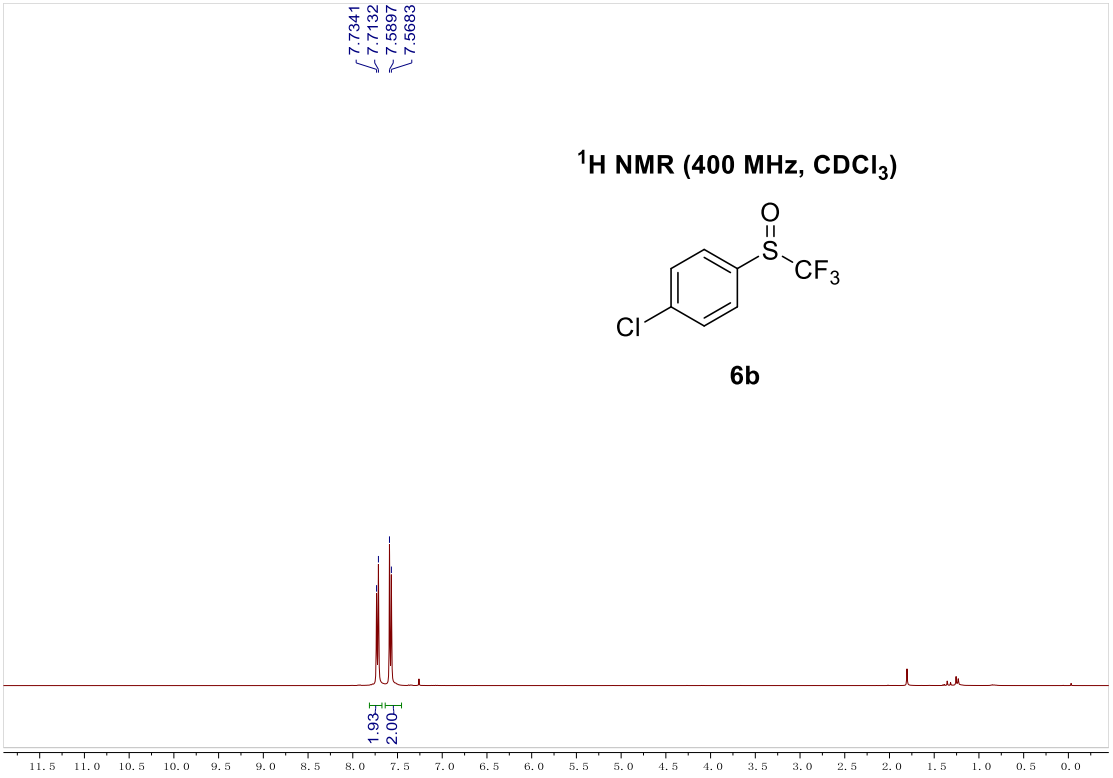


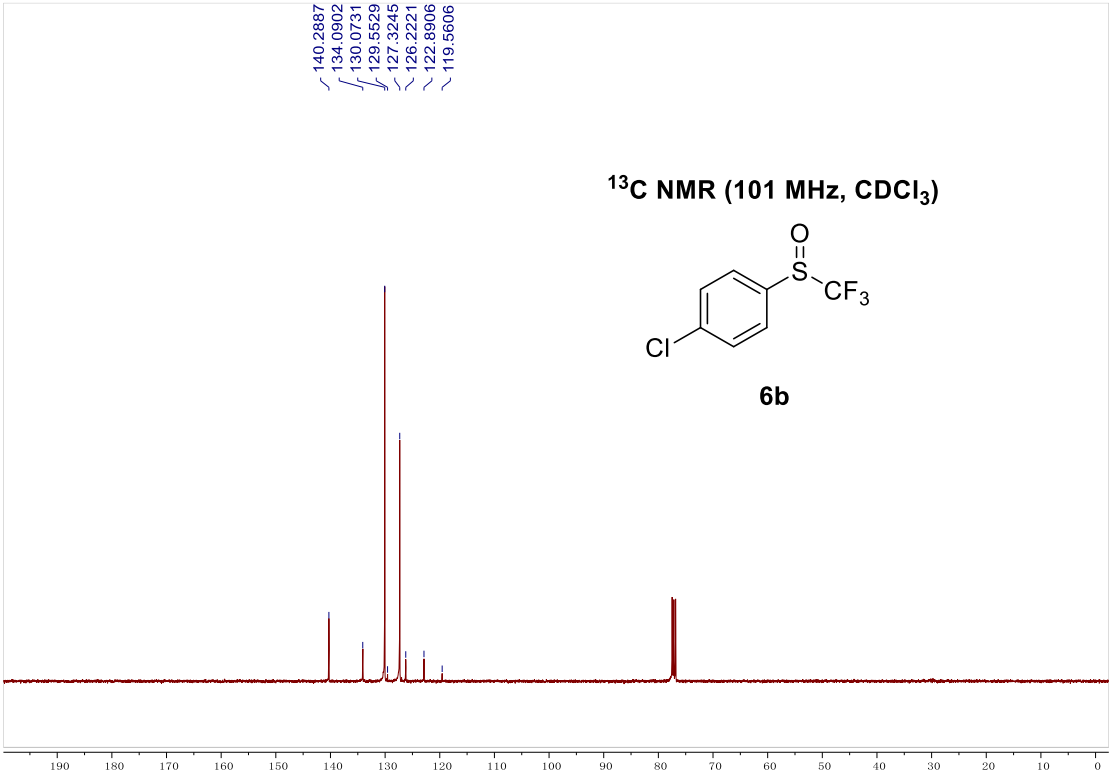
^{13}C NMR (126 MHz, d_6-DMSO)

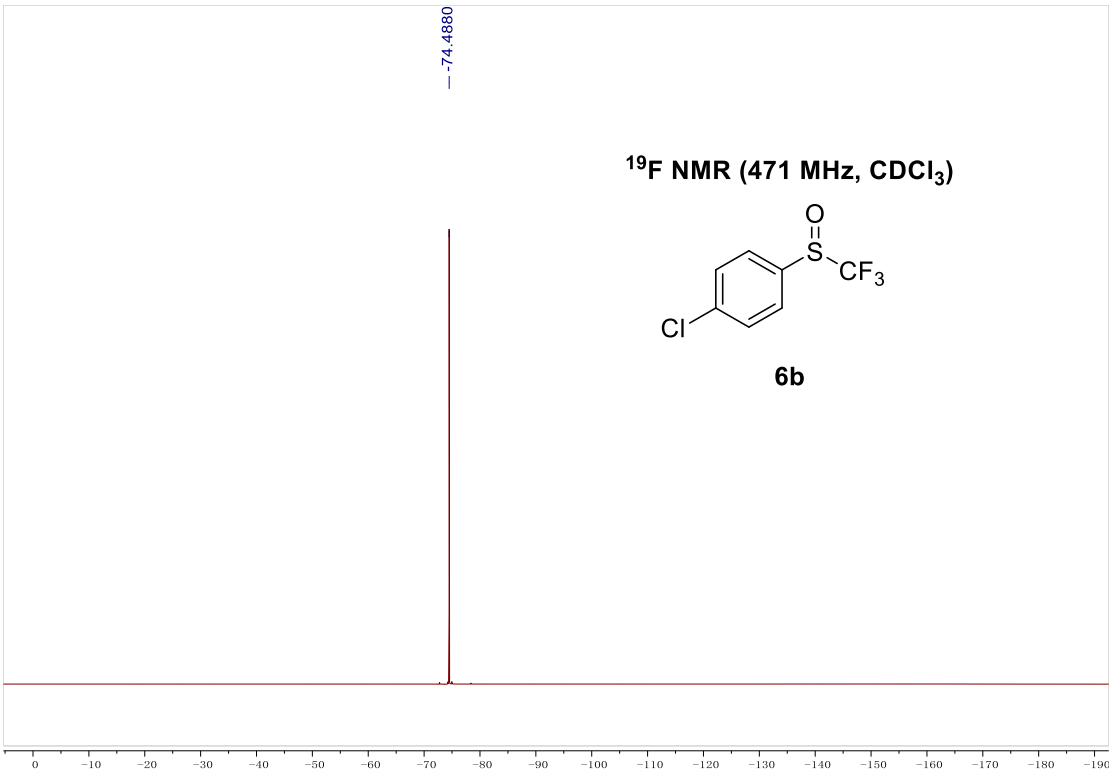
5g

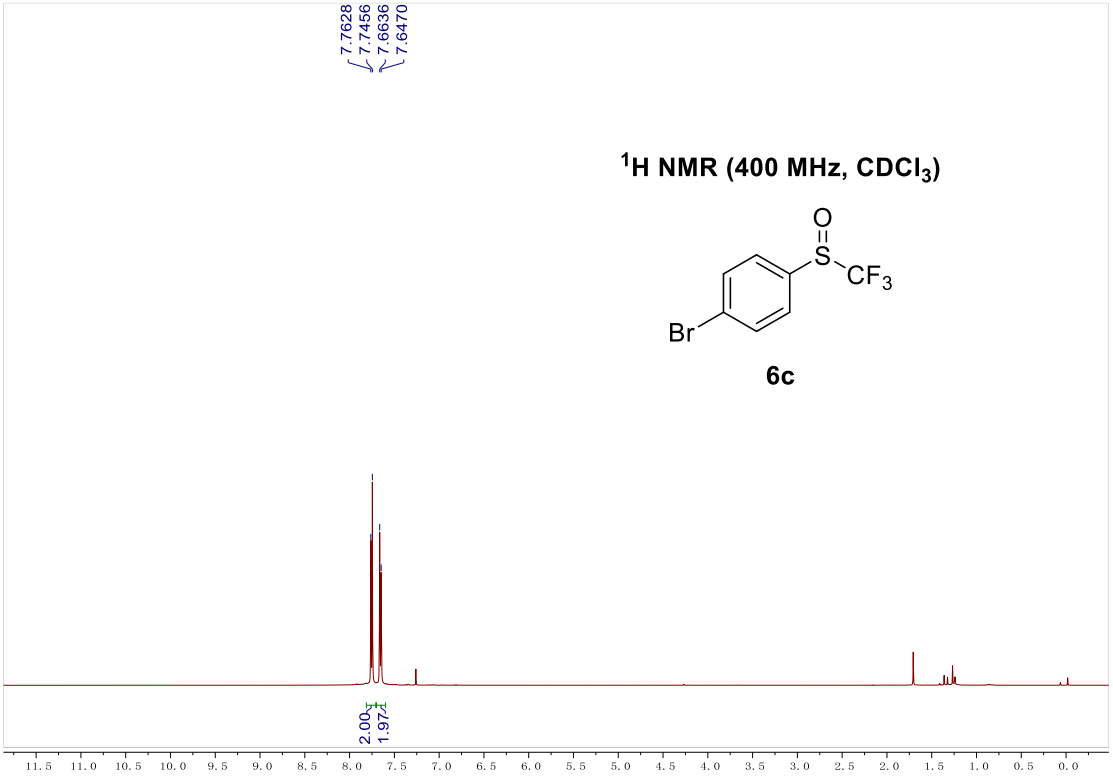

-77.7456
^{19}F NMR (471 MHz, d_6-DMSO)
OMe
$^{-}$OTf
MeO
5g

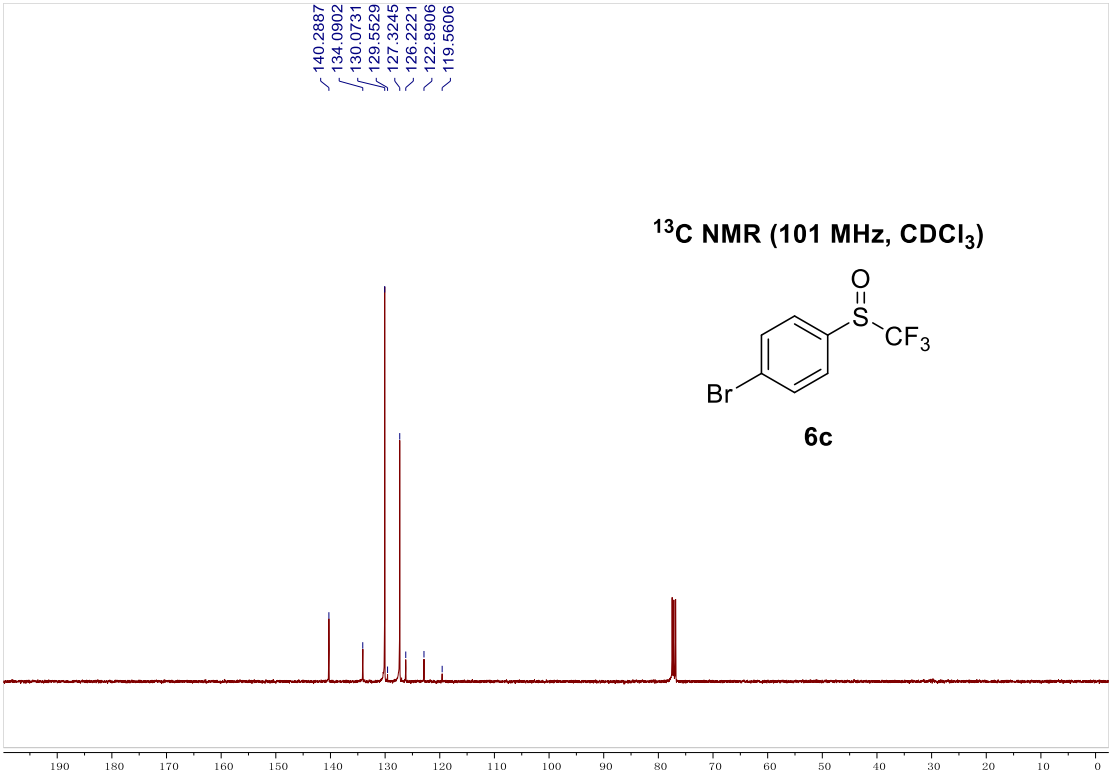

^{19}F NMR (400 MHz, $CDCl_3$)
O
S
CF_3
6a
PhCF3
6a
1.00
0.85


7.7341
7.7132
7.5897
7.5683
1H NMR (400 MHz, CDCl3)
O
S
CF3
Cl
6b
1.93
2.00
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0


140.2887
134.0902
130.0731
129.5529
127.3245
126.2221
122.8906
119.5606
13C NMR (101 MHz, CDCl3)
O
S
CF3
Cl
6b
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

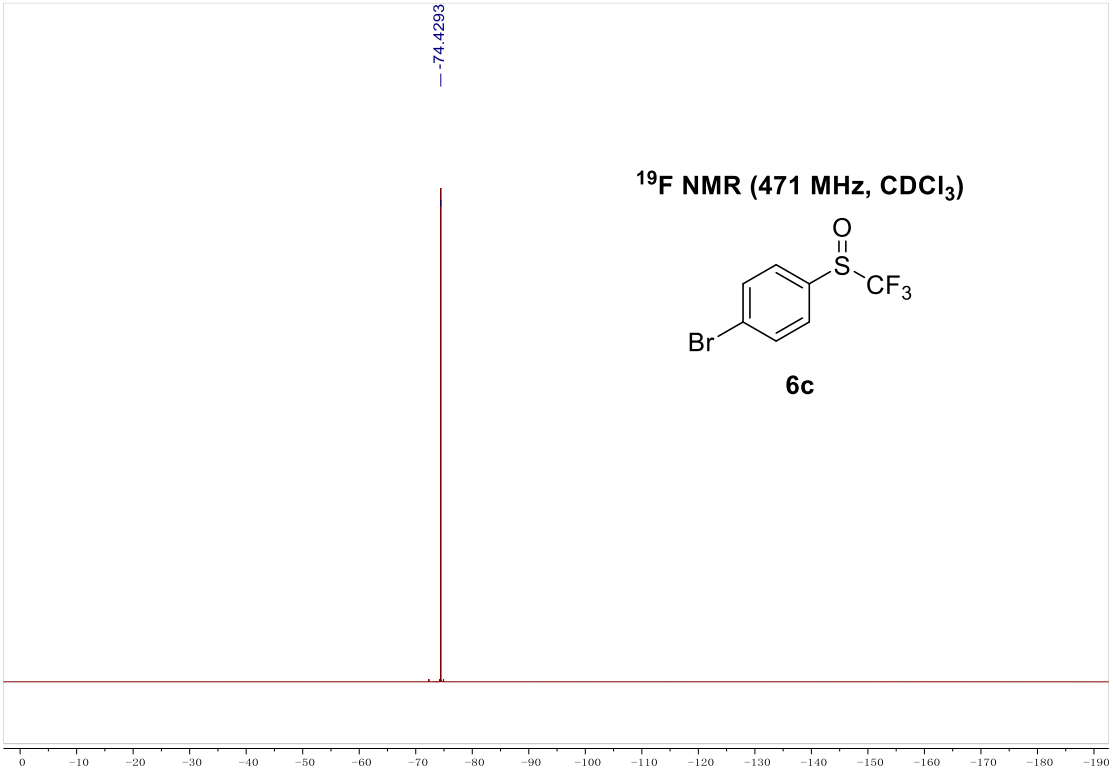

-74.4880
^{19}F NMR (471 MHz, CDCl$_3$)
O
S
CF$_3$
Cl
6b
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190


7.7628
7.7456
7.6636
7.6470
^{1}H NMR (400 MHz, CDCl$_3$)
O
S
CF$_3$
Br
6c
2.00
1.97
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0



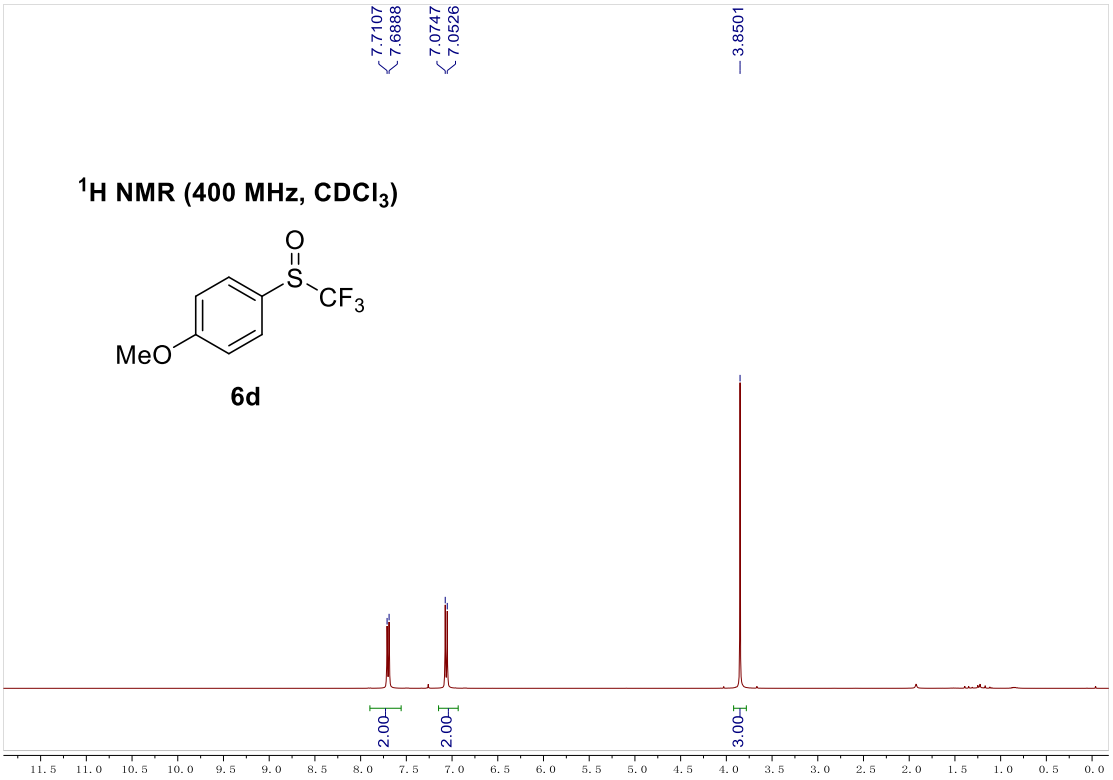
^{13}C NMR (101 MHz, $CDCl_3$)

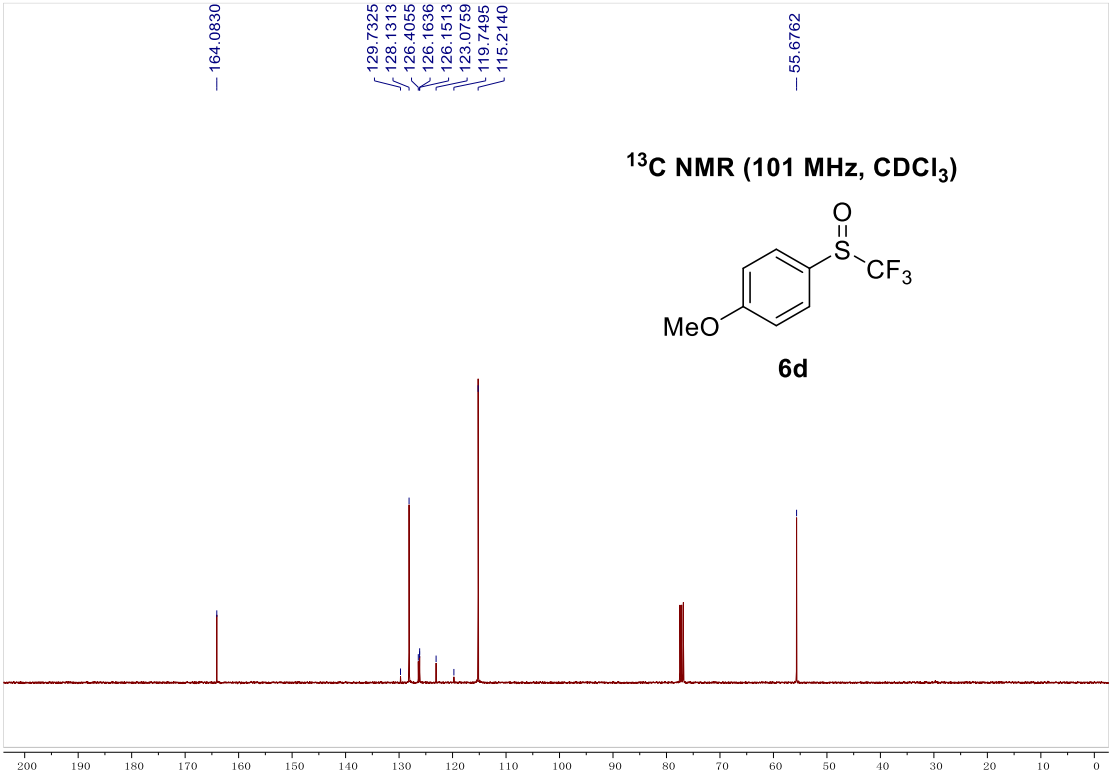
6c

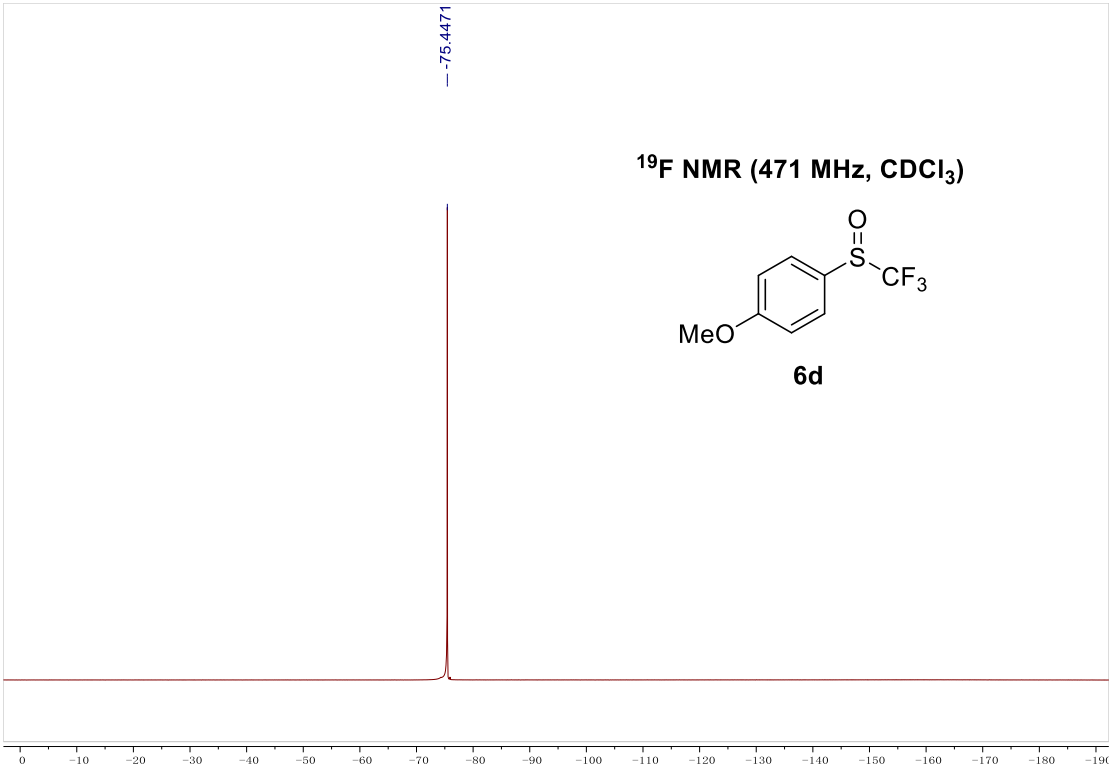


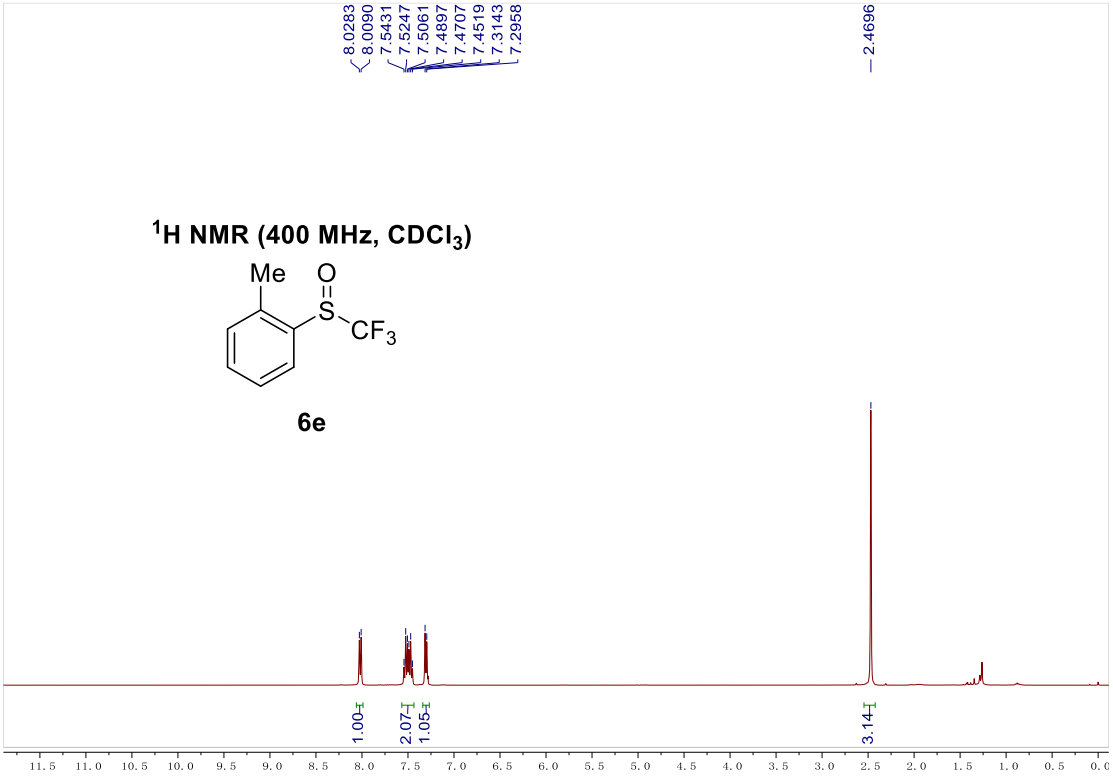
^{19}F NMR (471 MHz, $CDCl_3$)

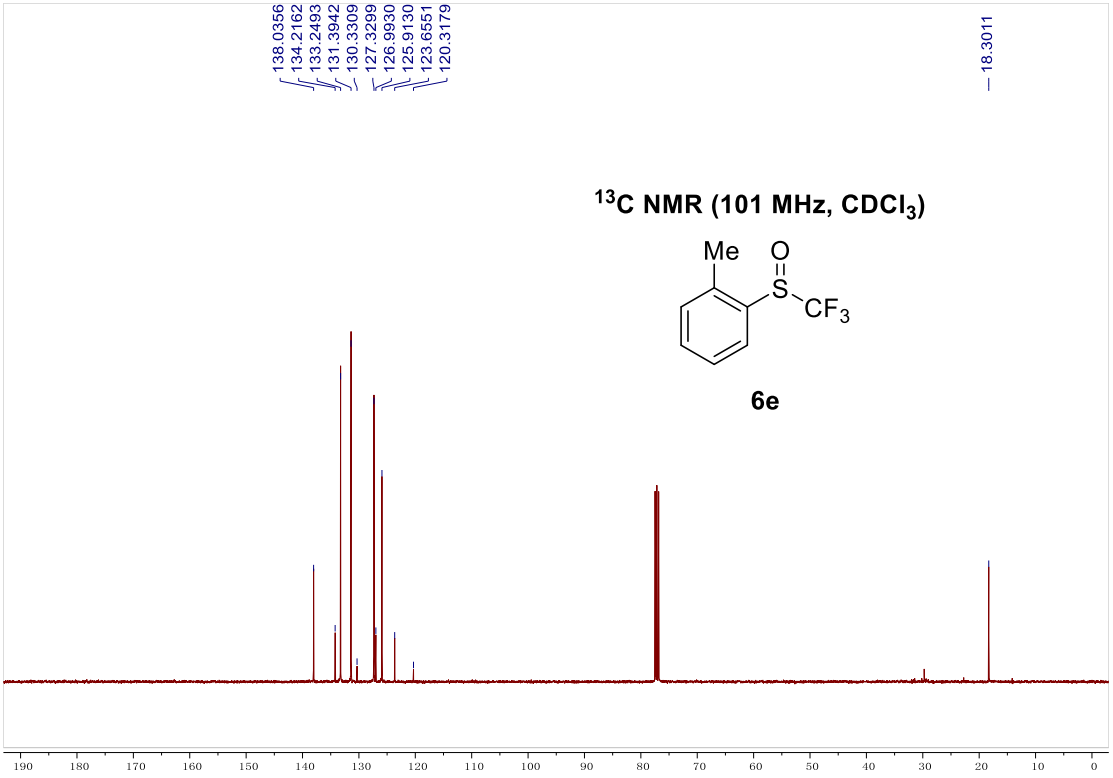
6c

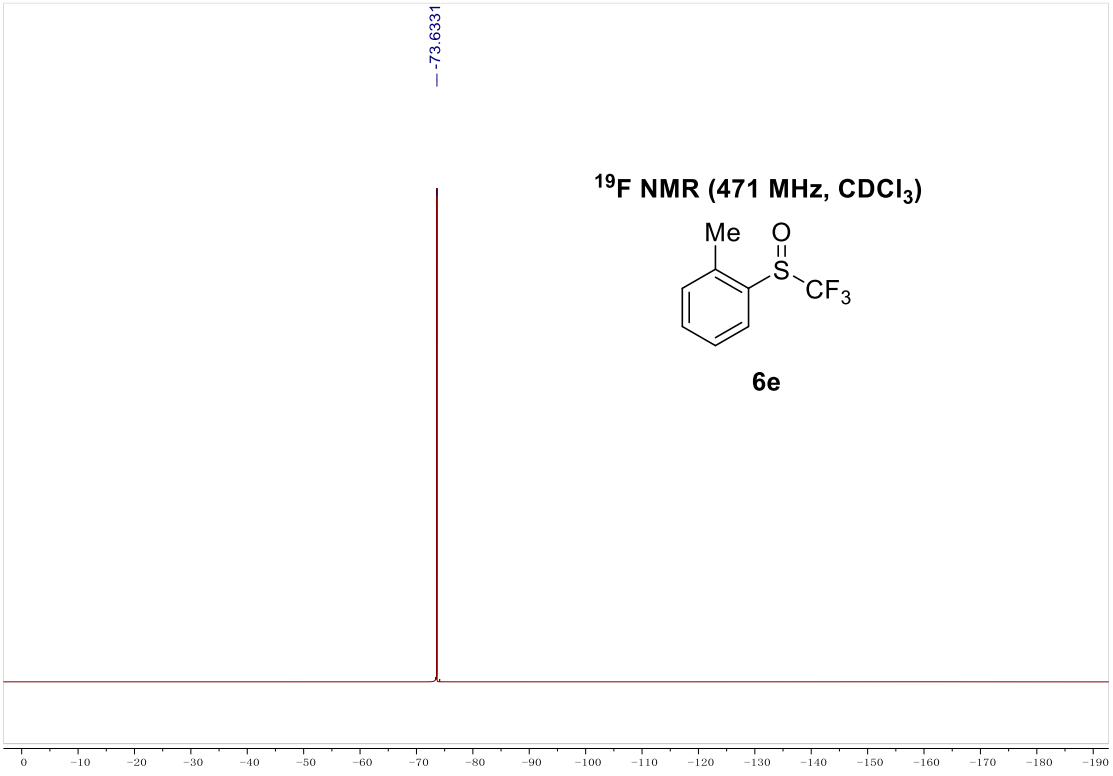

7.7107
7.6888
7.0747
7.0526
3.8501
1H NMR (400 MHz, CDCl3)
O
S
CF3
MeO
6d
2.00
2.00
3.00
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

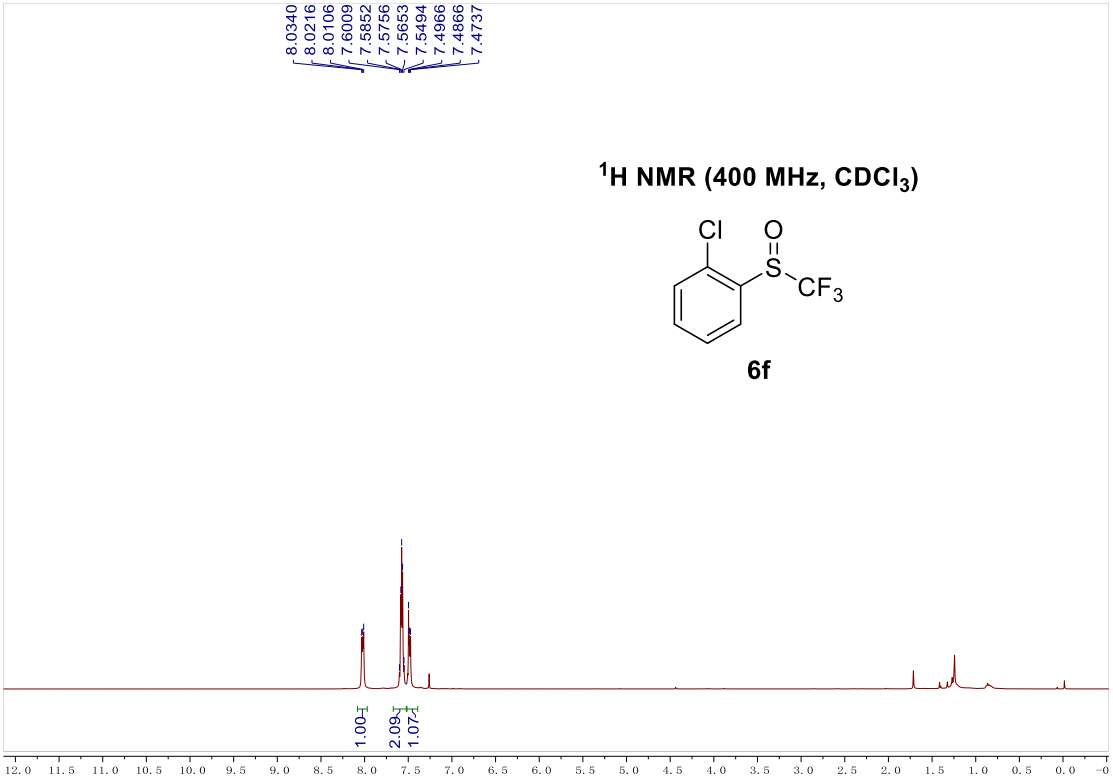

164.0830
129.7325
128.1313
126.4055
126.1636
126.1513
123.0759
119.7495
115.2140
55.6762
13C NMR (101 MHz, CDCl3)
O
S
CF3
MeO
6d
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

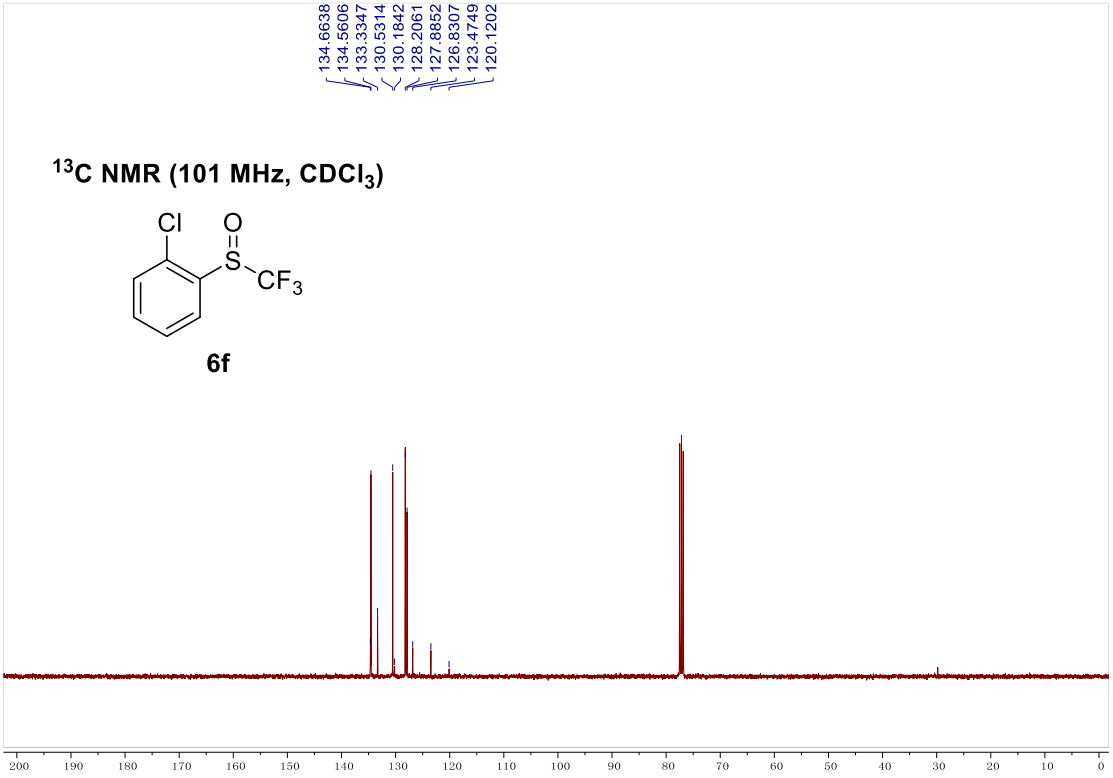

-75.4471
19F NMR (471 MHz, CDCl3)
O
S
CF3
MeO
6d
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180

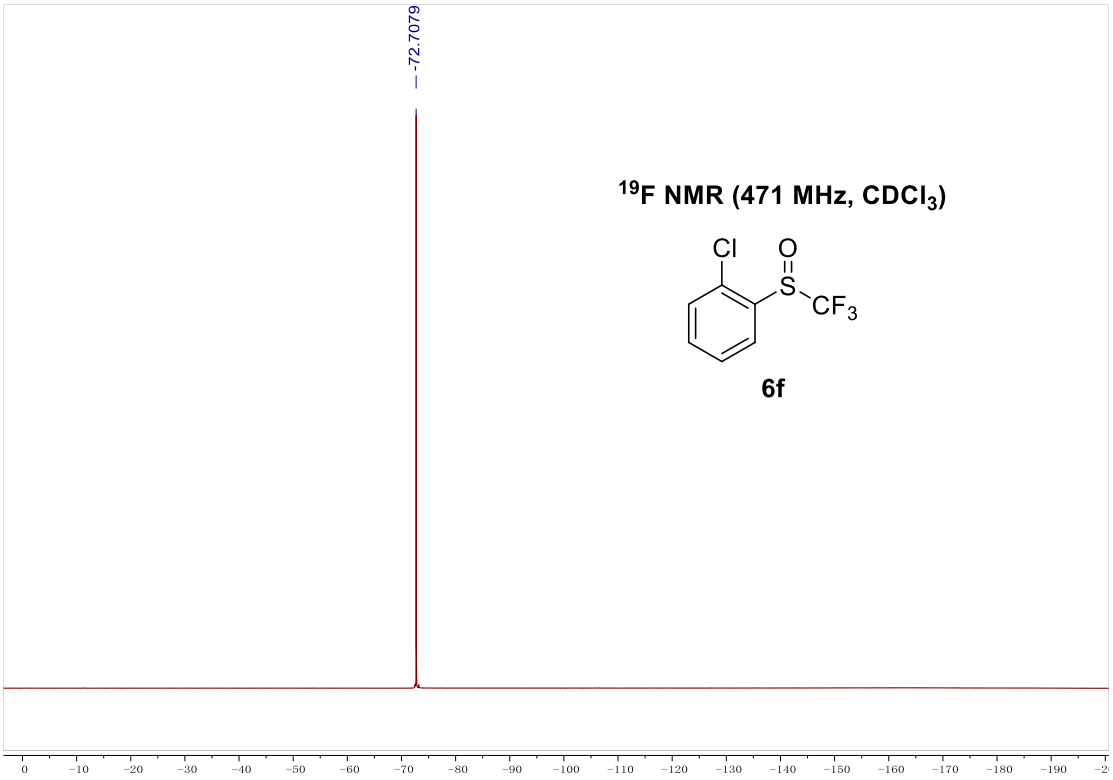

8.0283
8.0090
7.5431
7.5247
7.5061
7.4897
7.4707
7.4519
7.3143
7.2958
2.4696
1H NMR (400 MHz, CDCl3)
Me
O
S
CF3
6e
1.00
2.07
1.05
3.14
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5

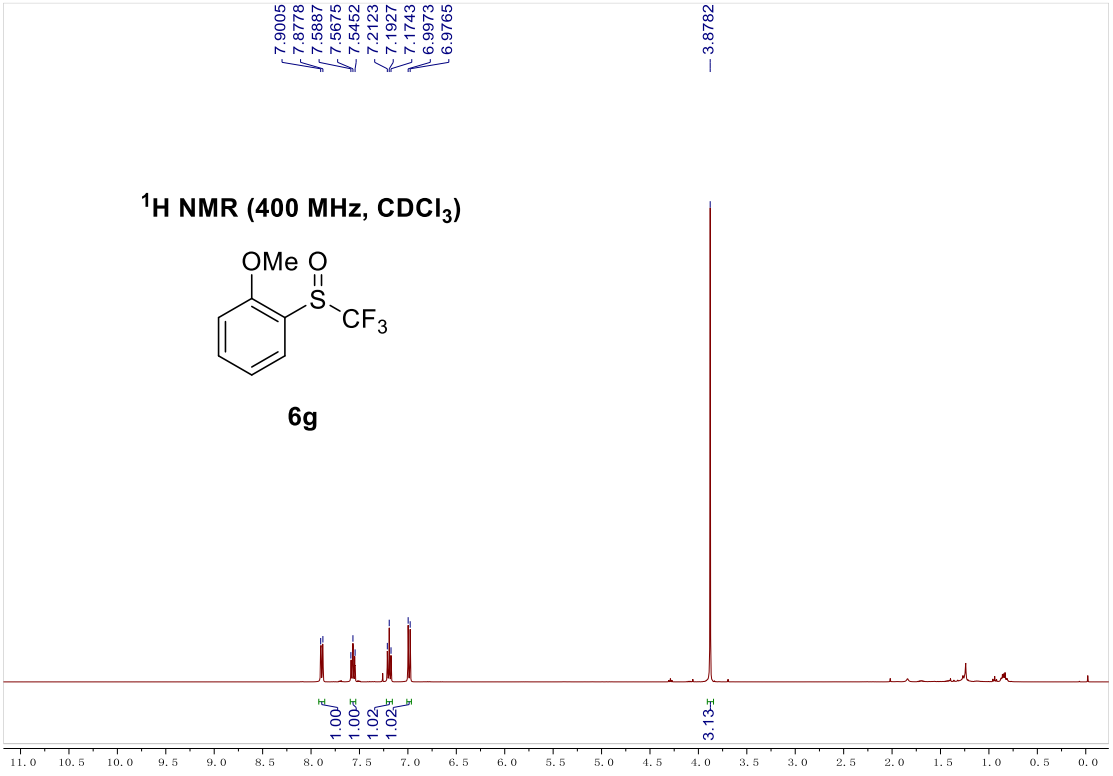

138.0356
134.2162
133.2493
131.3942
130.3309
127.3299
126.9930
125.9130
123.6551
120.3179
18.3011
13C NMR (101 MHz, CDCl3)
Me
O
S
CF3
6e
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

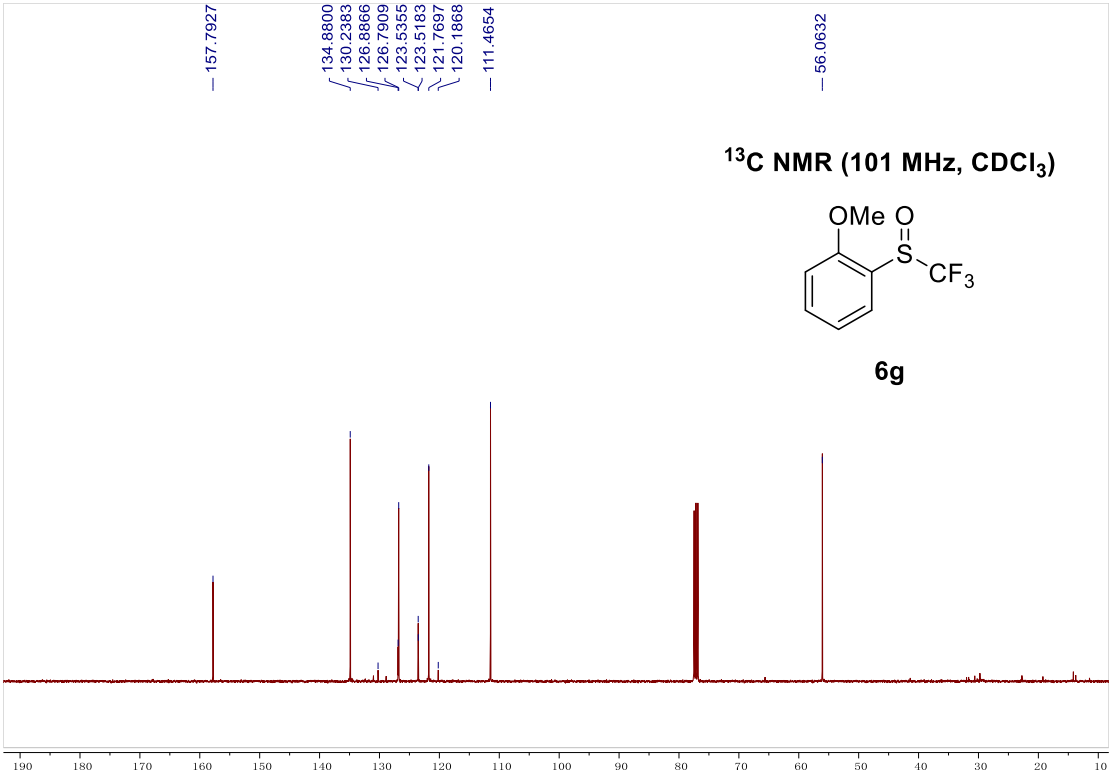

-73.6331
19F NMR (471 MHz, CDCl3)
Me
O
S
CF3
6e
0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190

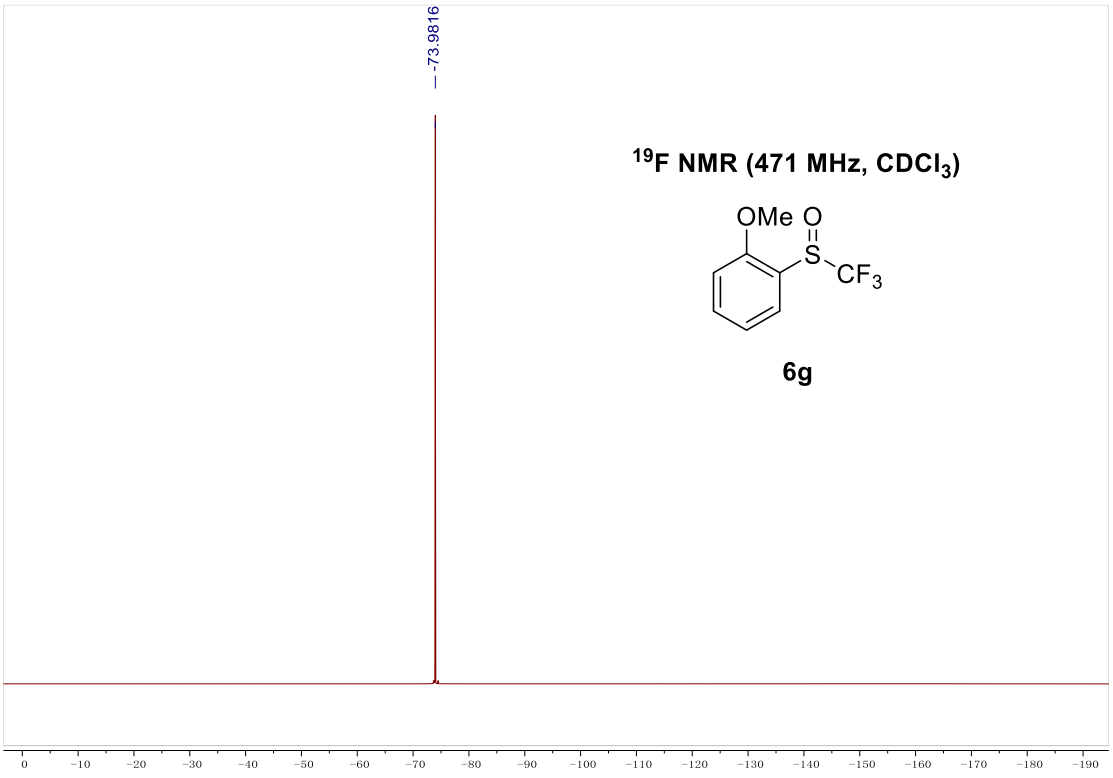

8.0340
8.0216
8.0106
7.6009
7.5852
7.5756
7.5653
7.5494
7.4966
7.4866
7.4737
1H NMR (400 MHz, CDCl3)
Cl
O
S
CF3
6f
1.00
2.09
1.07
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

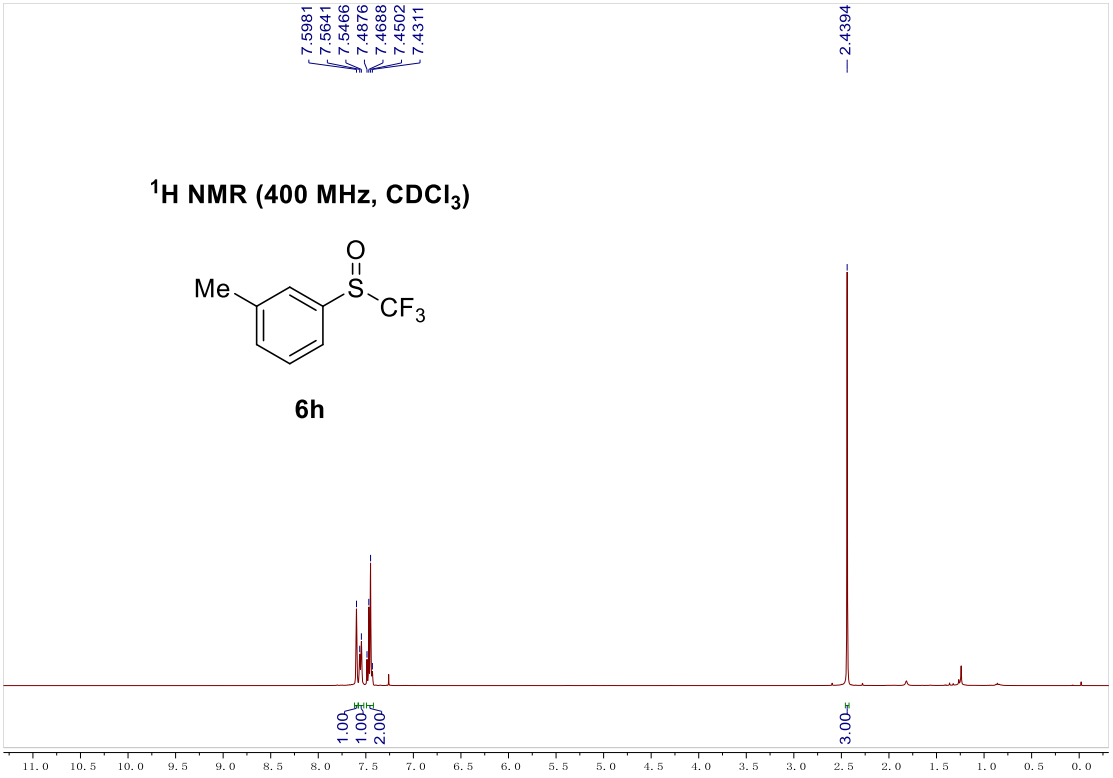

134.6638
134.5606
133.3347
130.5314
130.1842
128.2061
127.8852
126.8307
123.4749
120.1202
13C NMR (101 MHz, CDCl3)
Cl
O
S
CF3
6f
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

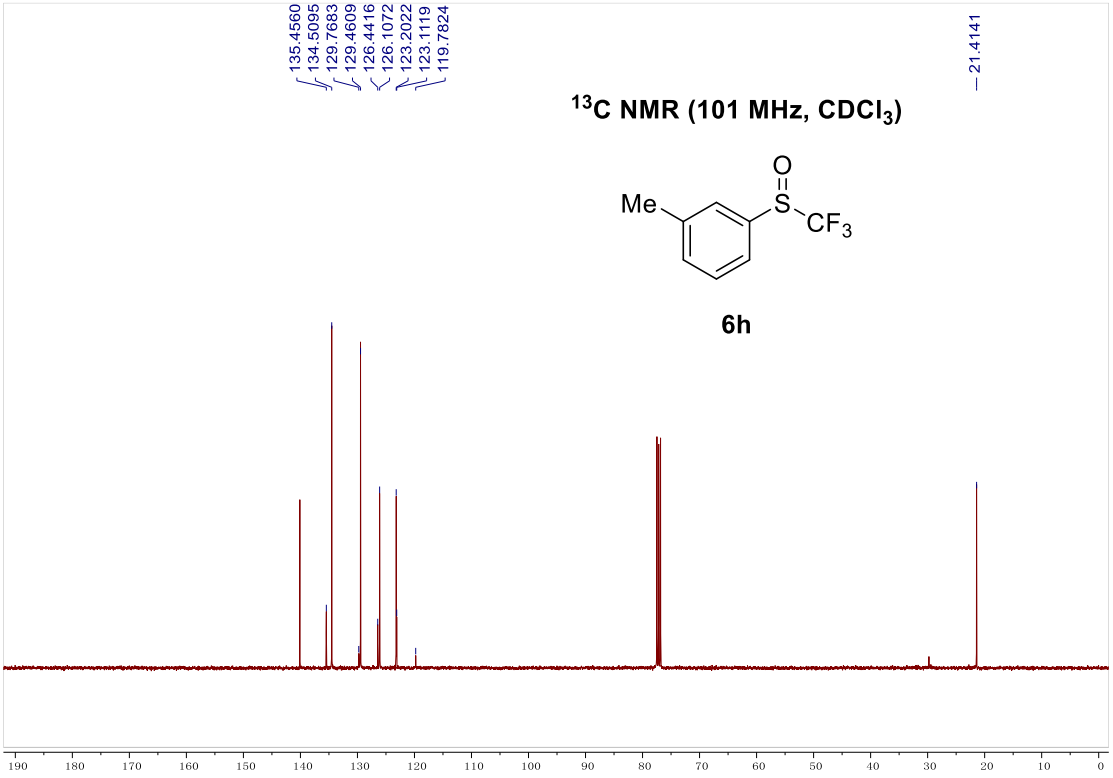

-72.7079
^{19}F NMR (471 MHz, CDCl$_3$)
Cl
O
S
CF$_3$
6f

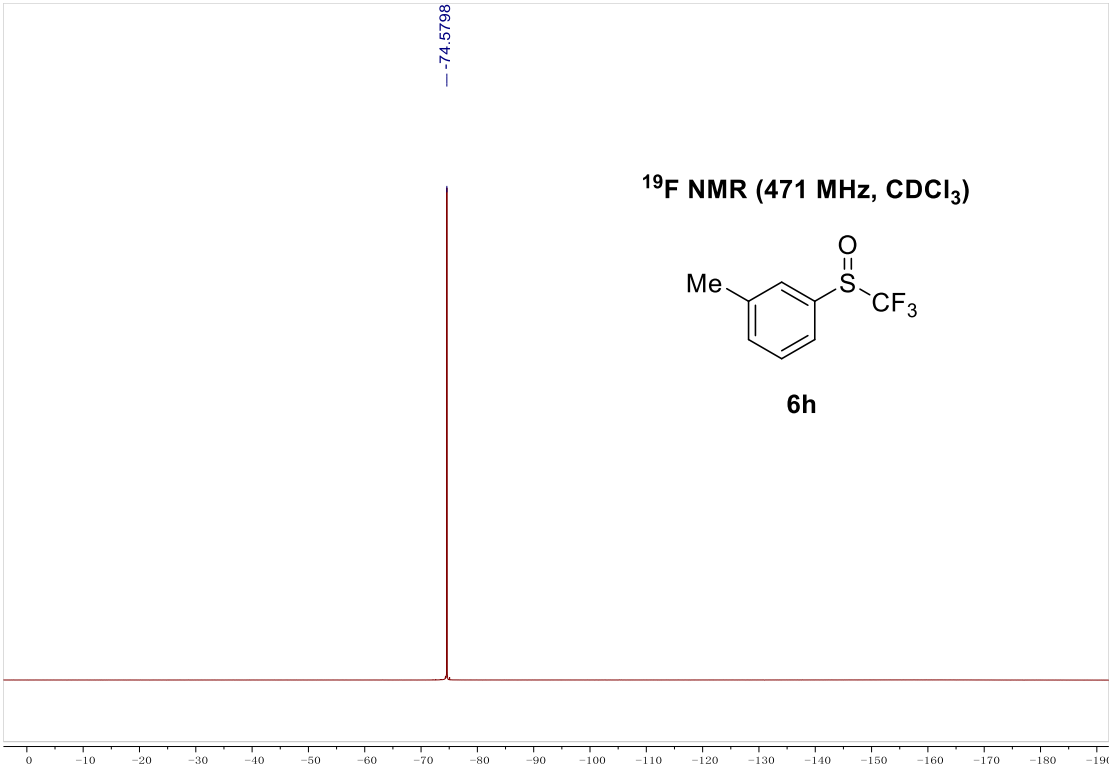

7.9005
7.8778
7.5887
7.5675
7.5452
7.2123
7.1927
7.1743
6.9973
6.9765
3.8782
^{1}H NMR (400 MHz, CDCl$_3$)
OMe
O
S
CF$_3$
6g
1.00
1.00
1.02
1.02
3.13

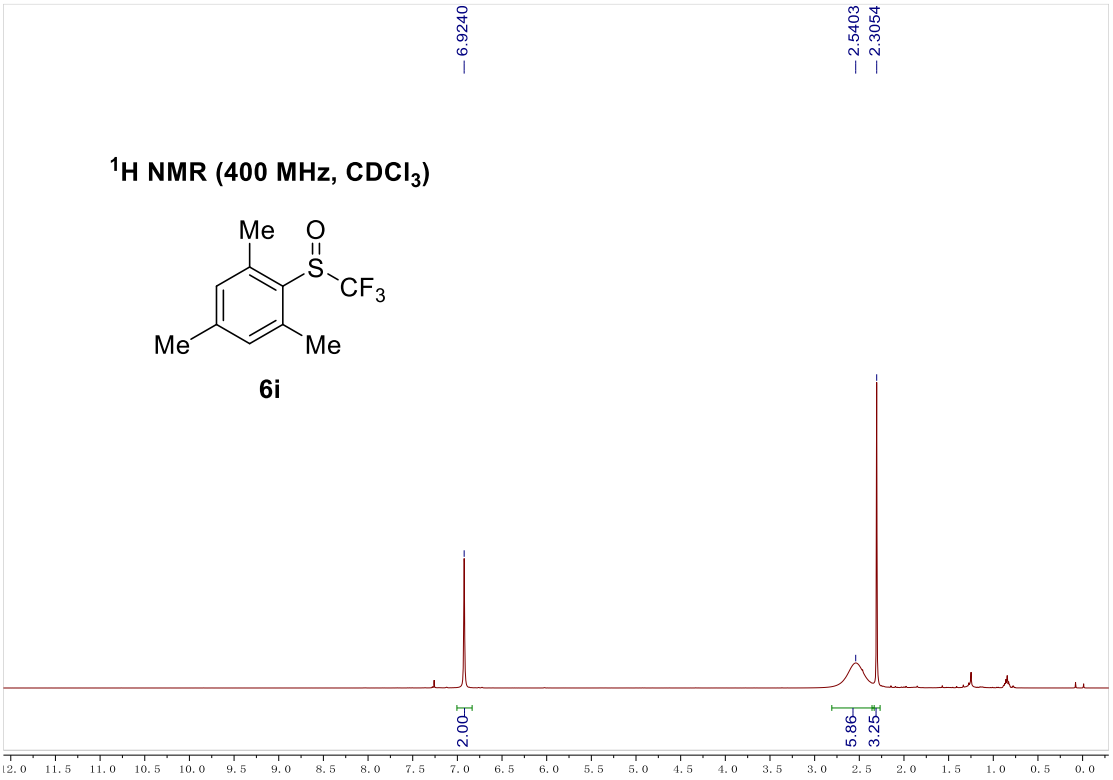

157.7927
134.8800
130.2383
126.8866
126.7909
123.5355
123.5183
121.7697
120.1868
111.4654
56.0632
13C NMR (101 MHz, CDCl3)
OMe O
S
CF3
6g
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

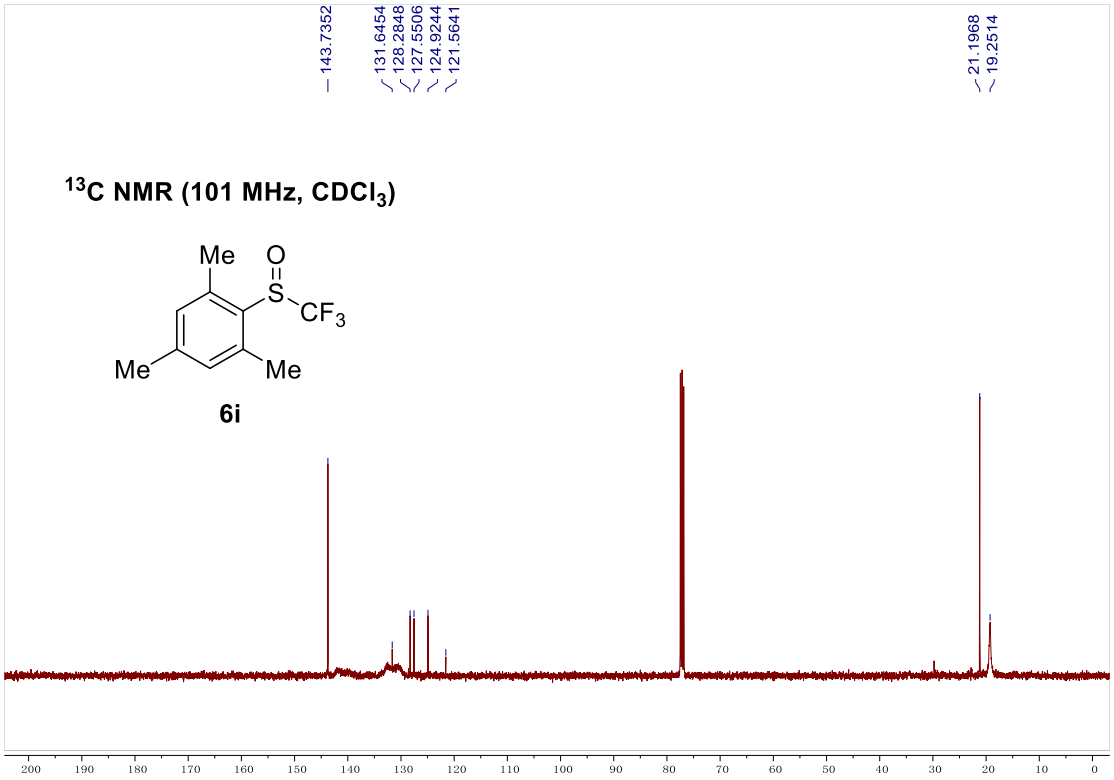

-73.9816
19F NMR (471 MHz, CDCl3)
OMe O
S
CF3
6g
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190

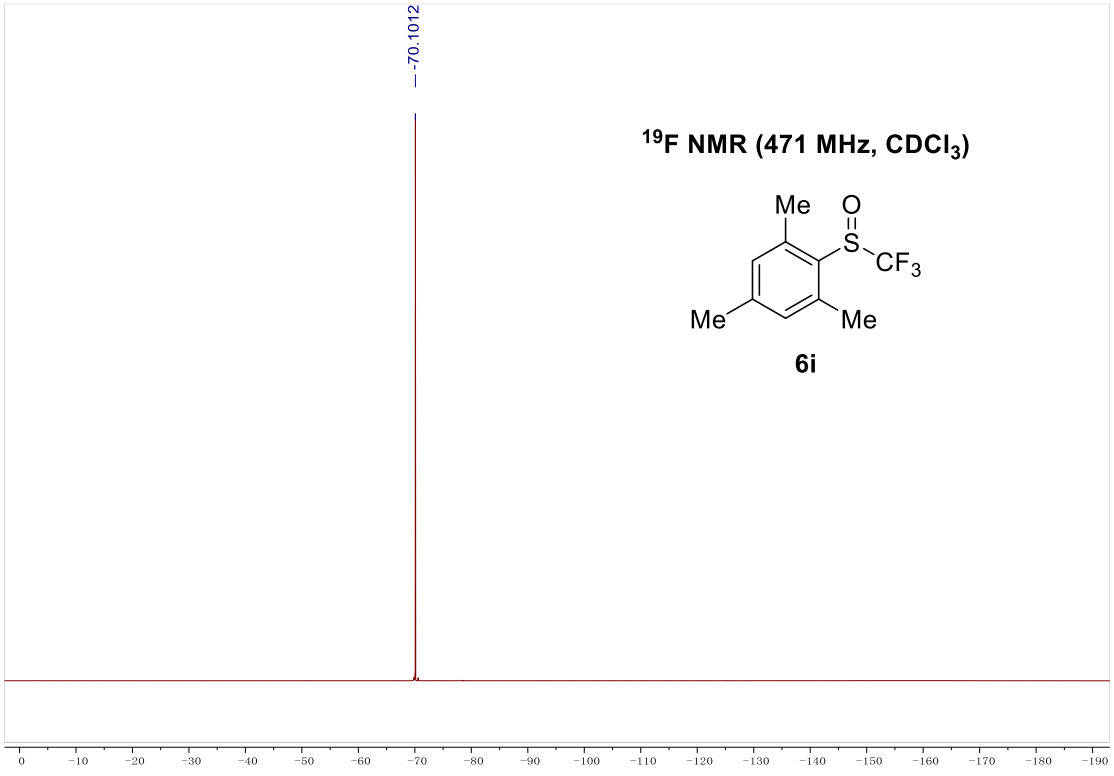

7.5981
7.5641
7.5466
7.4876
7.4688
7.4502
7.4311
2.4394
1H NMR (400 MHz, CDCl3)
Me
S
O
CF3
6h
1.00
1.00
2.00
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

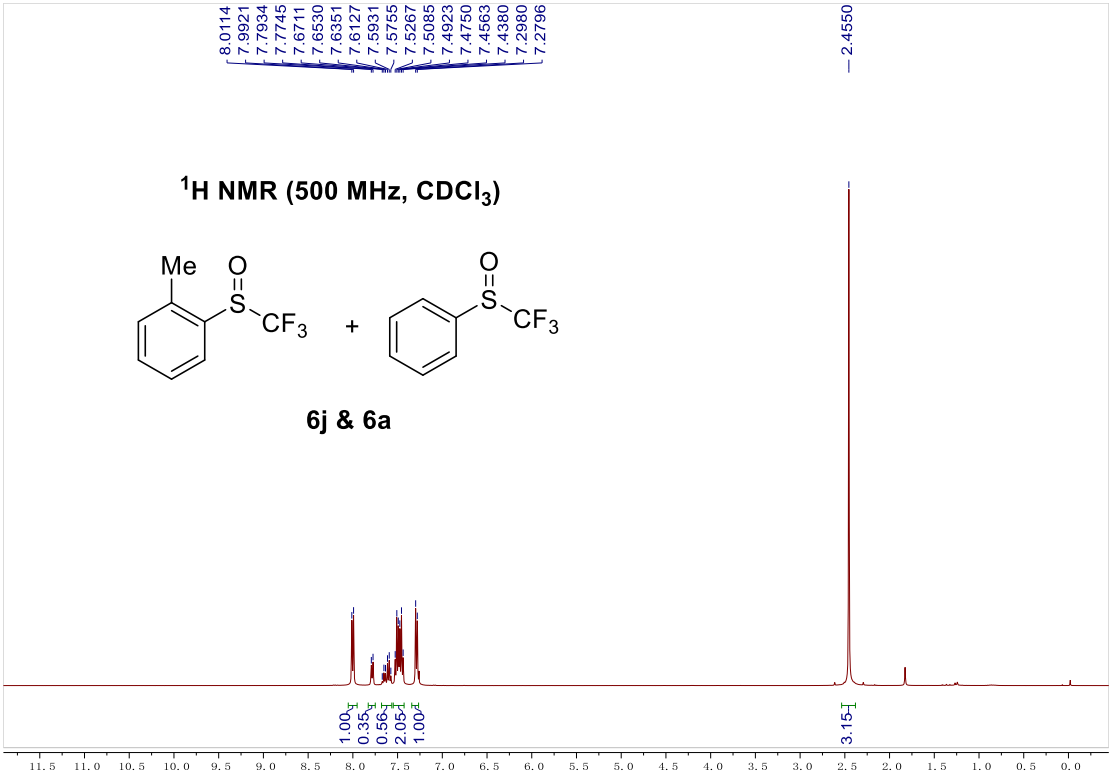

135.4560
134.5095
129.7683
129.4609
126.4416
126.1072
123.2022
123.1119
119.7824
21.4141
13C NMR (101 MHz, CDCl3)
Me
S
O
CF3
6h
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

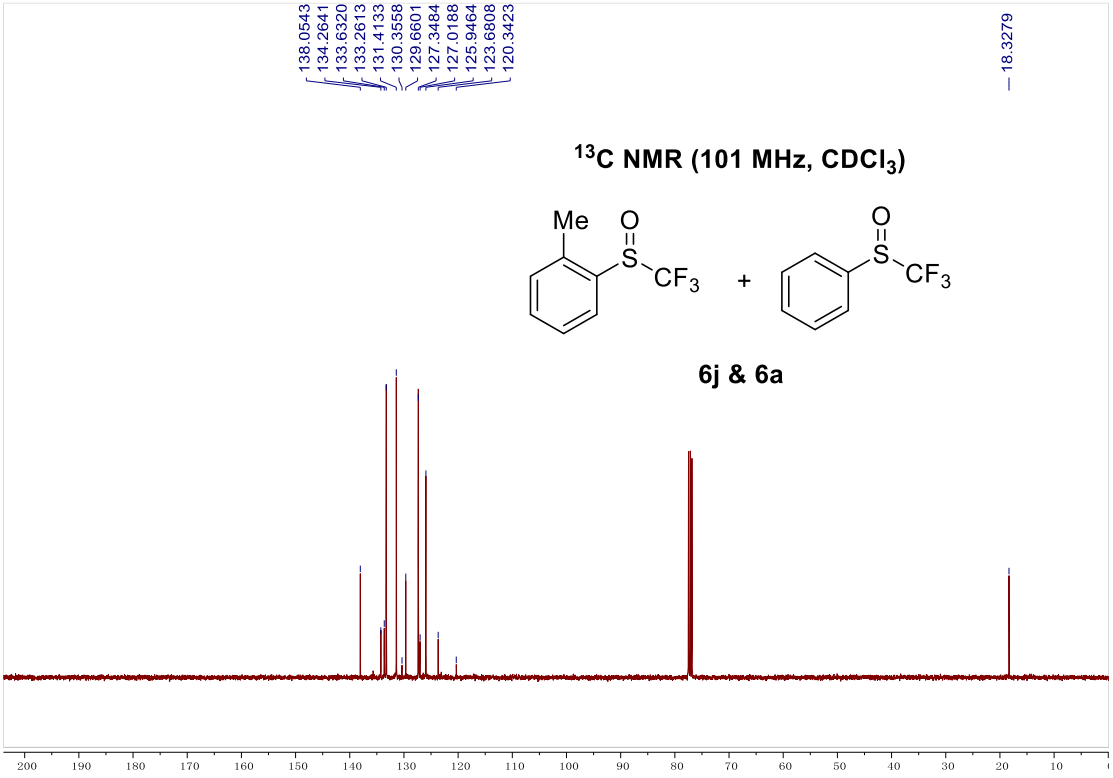

-74.5798
19F NMR (471 MHz, CDCl3)
Me
O
S
CF3
6h

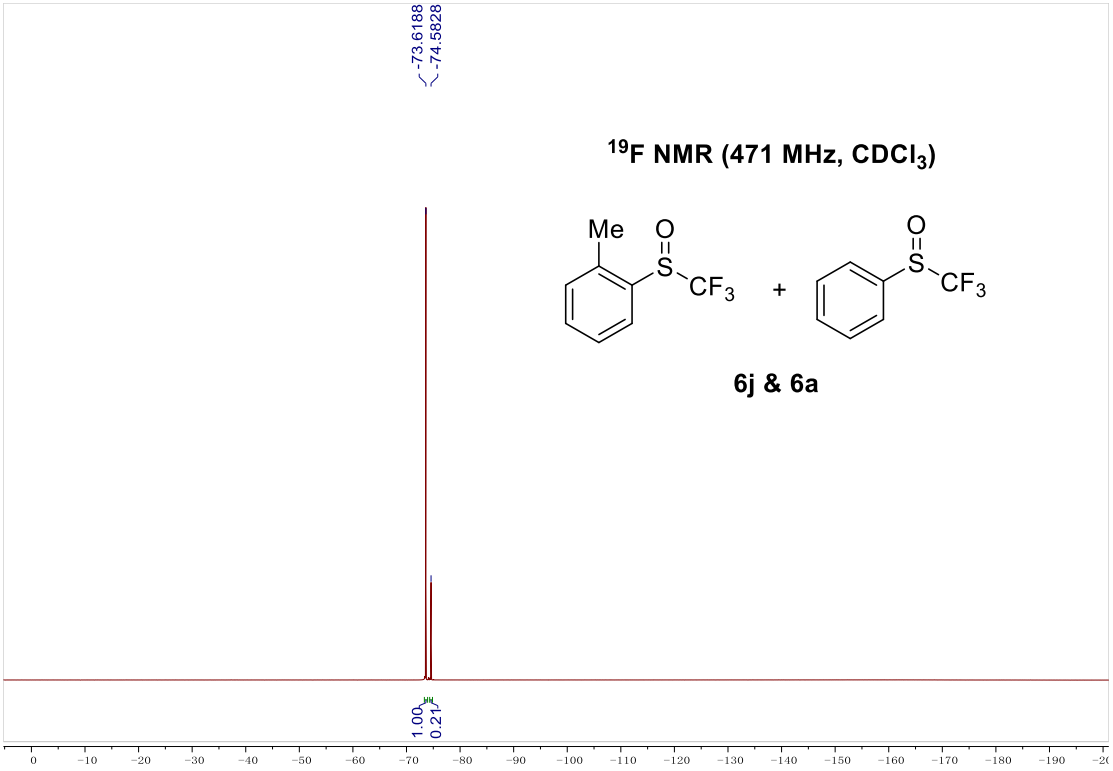

6.9240
2.5403
2.3054
1H NMR (400 MHz, CDCl3)
Me
O
S
CF3
Me
Me
6i
2.00
5.86
3.25

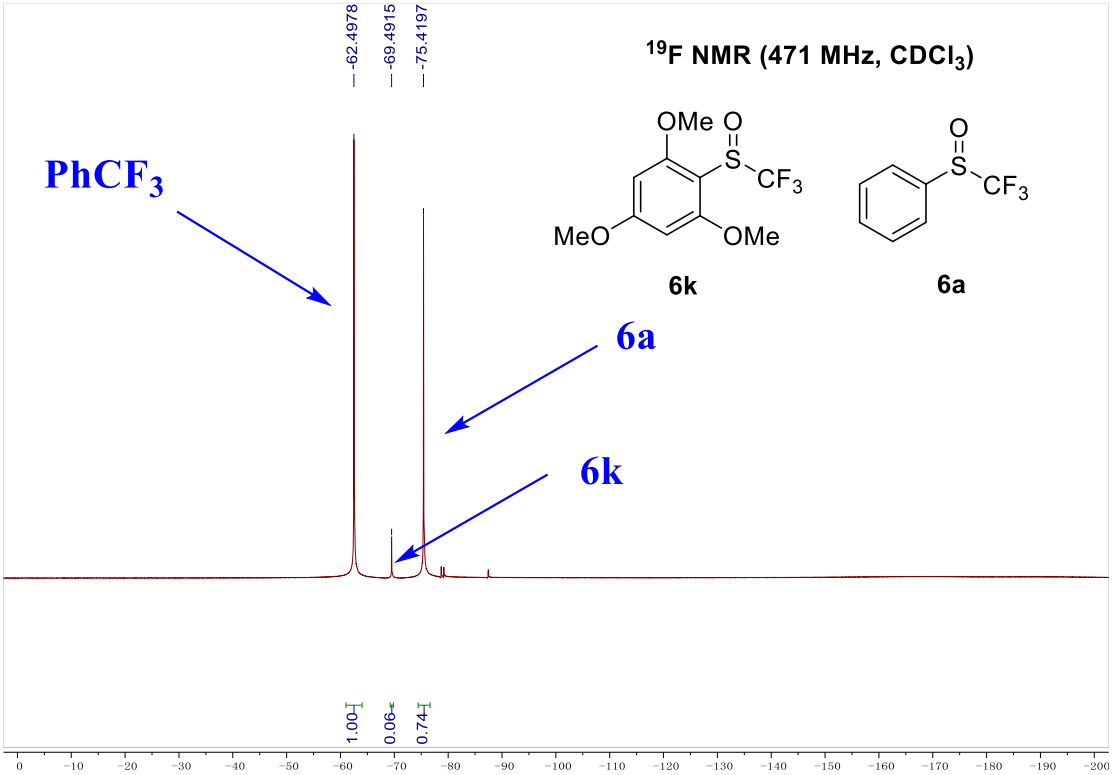

143.7352
131.6454
128.2848
127.5506
124.9244
121.5641
21.1968
19.2514
13C NMR (101 MHz, CDCl3)
Me
O
S
CF3
Me
Me
6i
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

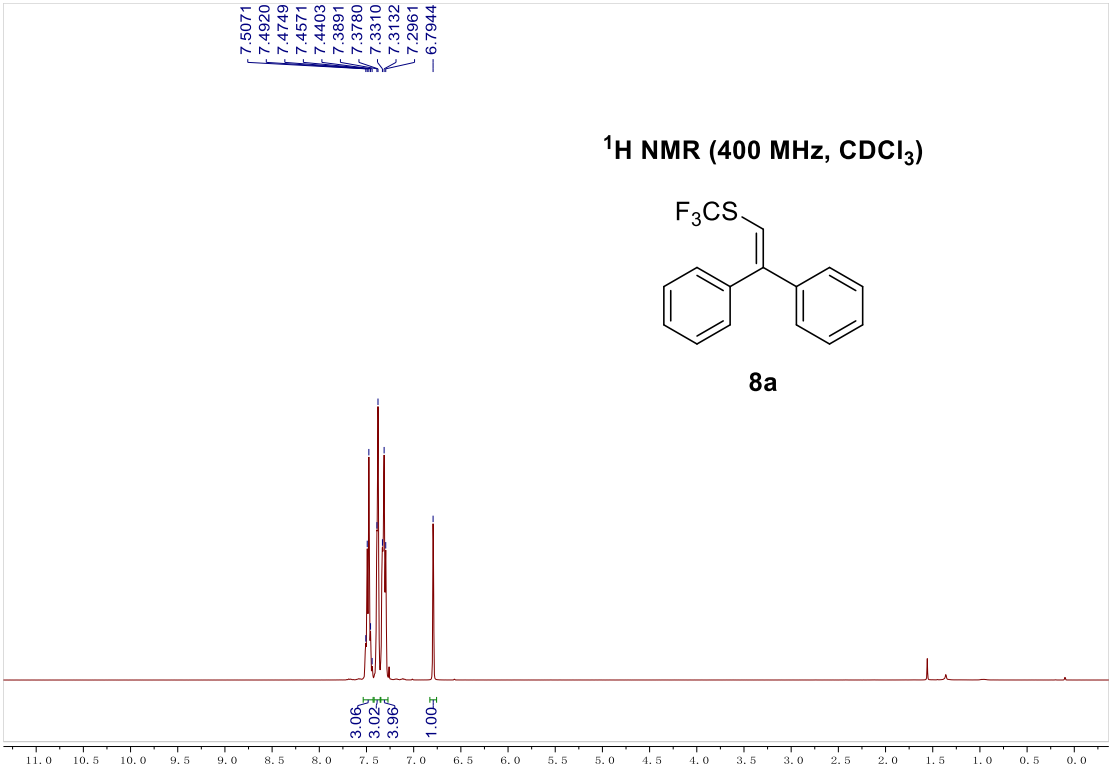

-70.1012
19F NMR (471 MHz, CDCl3)
Me
O
S
CF3
Me
Me
6i
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190

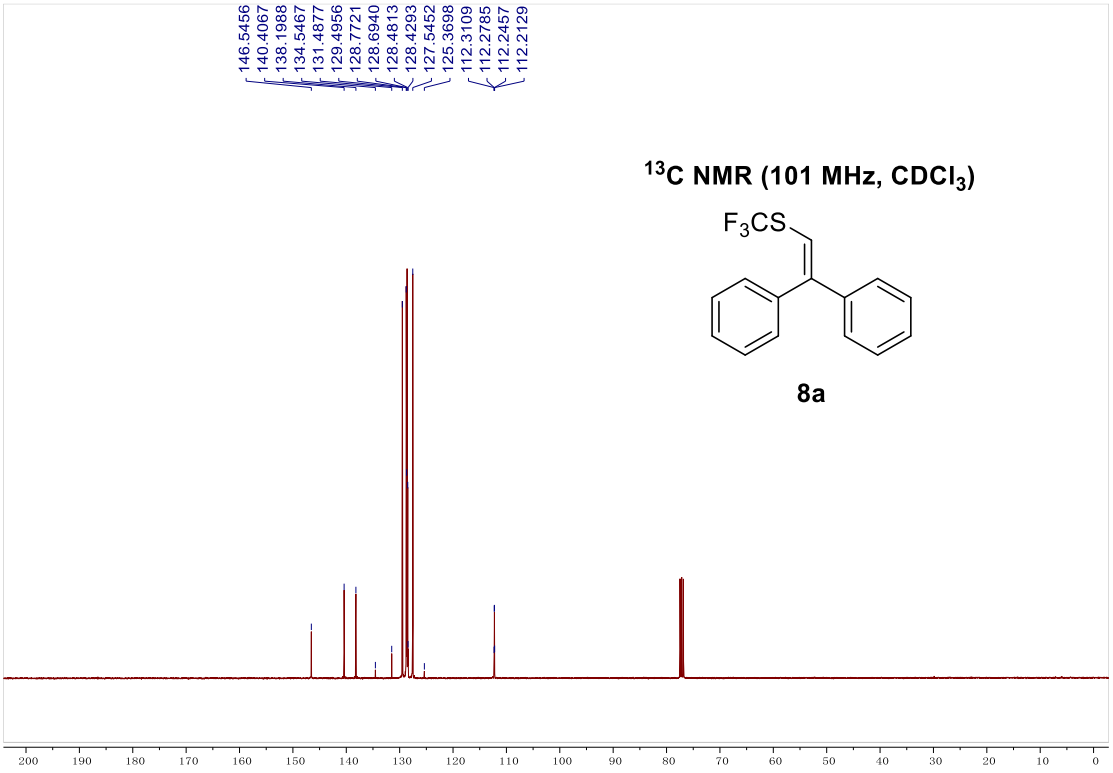

8.0114
7.9921
7.7934
7.7745
7.6711
7.6530
7.6351
7.6127
7.5931
7.5755
7.5267
7.5085
7.4923
7.4750
7.4563
7.4380
7.2980
7.2796
2.4550
1H NMR (500 MHz, CDCl3)
Me
O
S
CF3
+
O
S
CF3
6j & 6a
1.00
0.35
0.56
2.05
1.00
3.15

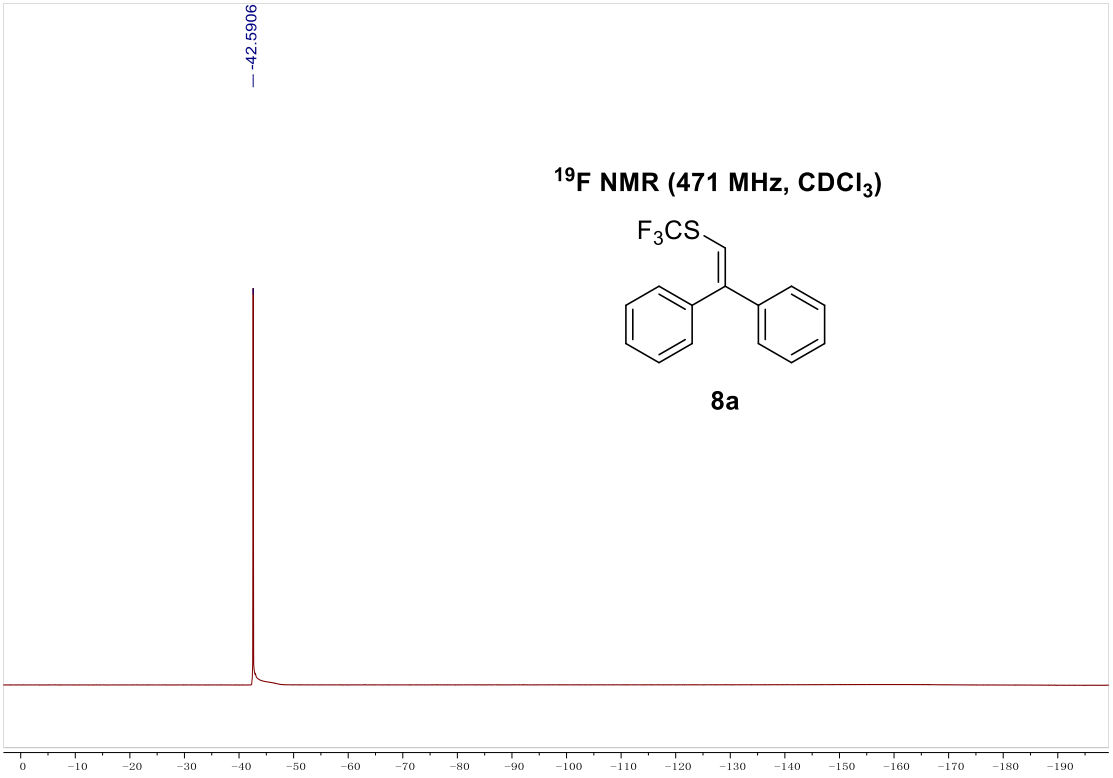

138.0543
134.2641
133.6320
133.2613
131.4133
130.3558
129.6601
127.3484
127.0188
125.9464
123.6808
120.3423
18.3279
13C NMR (101 MHz, CDCl3)
Me
O
S
CF3
+
O
S
CF3
6j & 6a

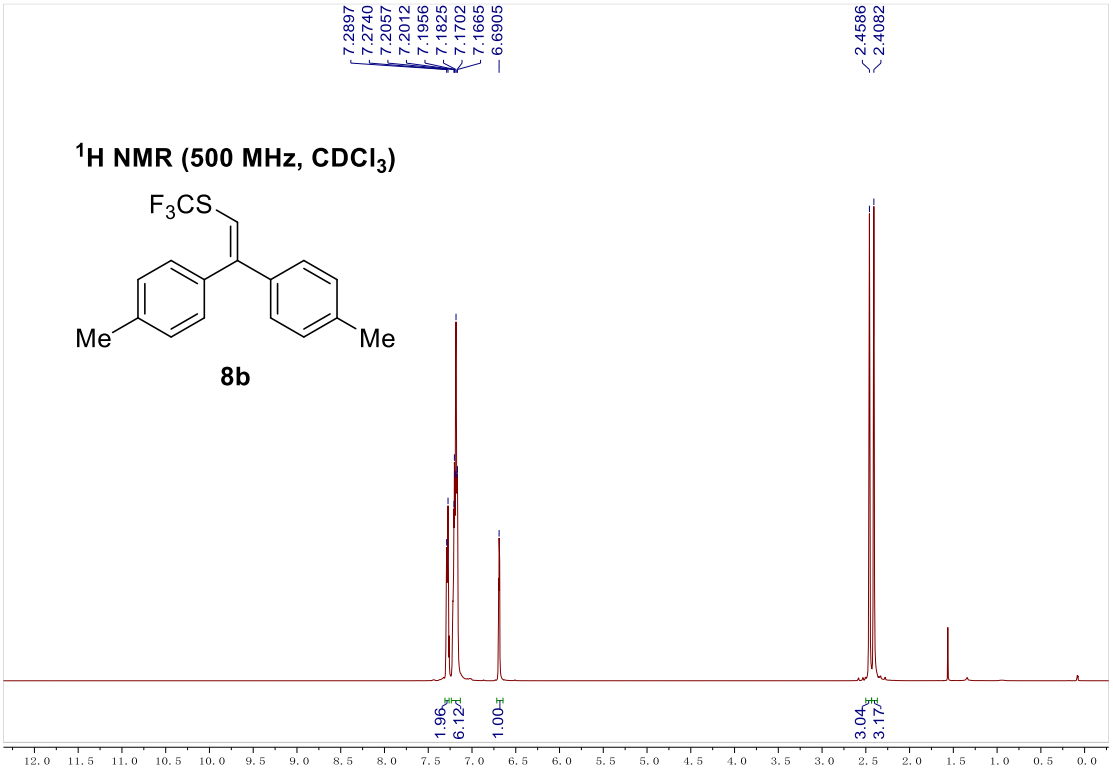

-73.6188
-74.5828
19F NMR (471 MHz, CDCl3)
Me
O
S
CF3
+
O
S
CF3
6j & 6a
1.00
0.21

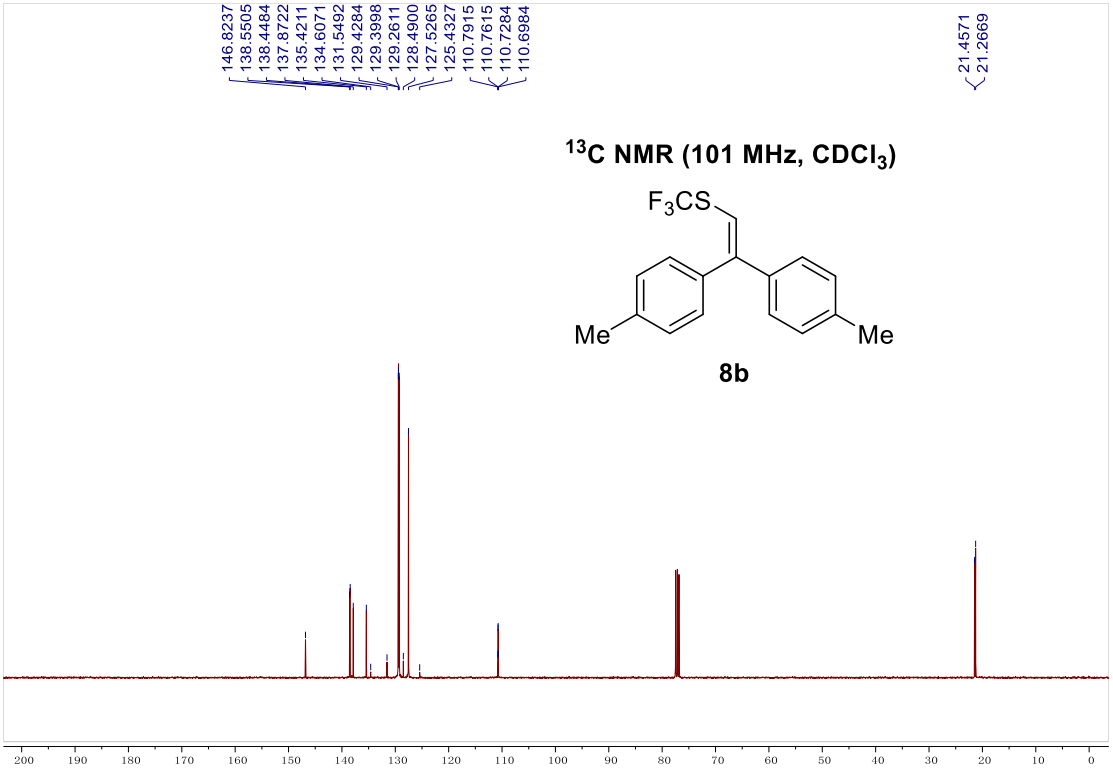

-62.4978
-69.4915
-75.4197
19F NMR (471 MHz, CDCl3)
PhCF3
OMe
O
S
CF3
MeO
OMe
6k
O
S
CF3
6a
6a
6k
1.00
0.06
0.74

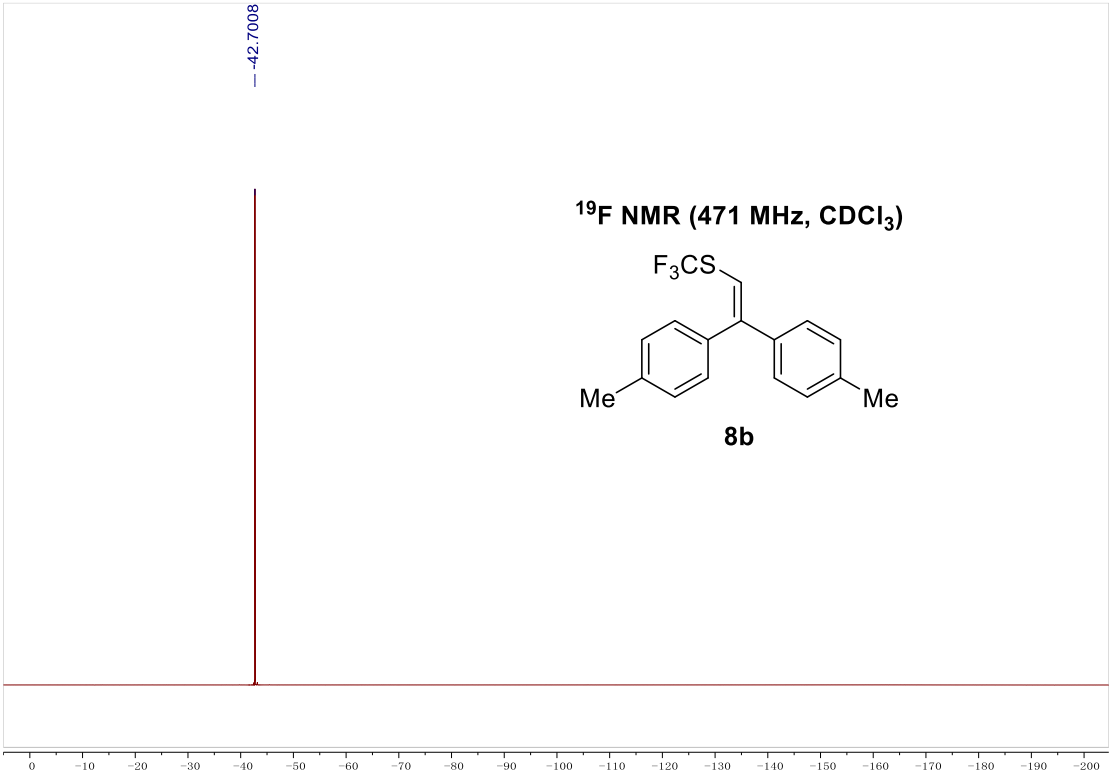

7.5071
7.4920
7.4749
7.4571
7.4403
7.3891
7.3780
7.3310
7.3132
7.2961
6.7944
^{1}H NMR (400 MHz, CDCl$_3$)
F$_3$CS
8a
3.06
3.02
3.96
1.00

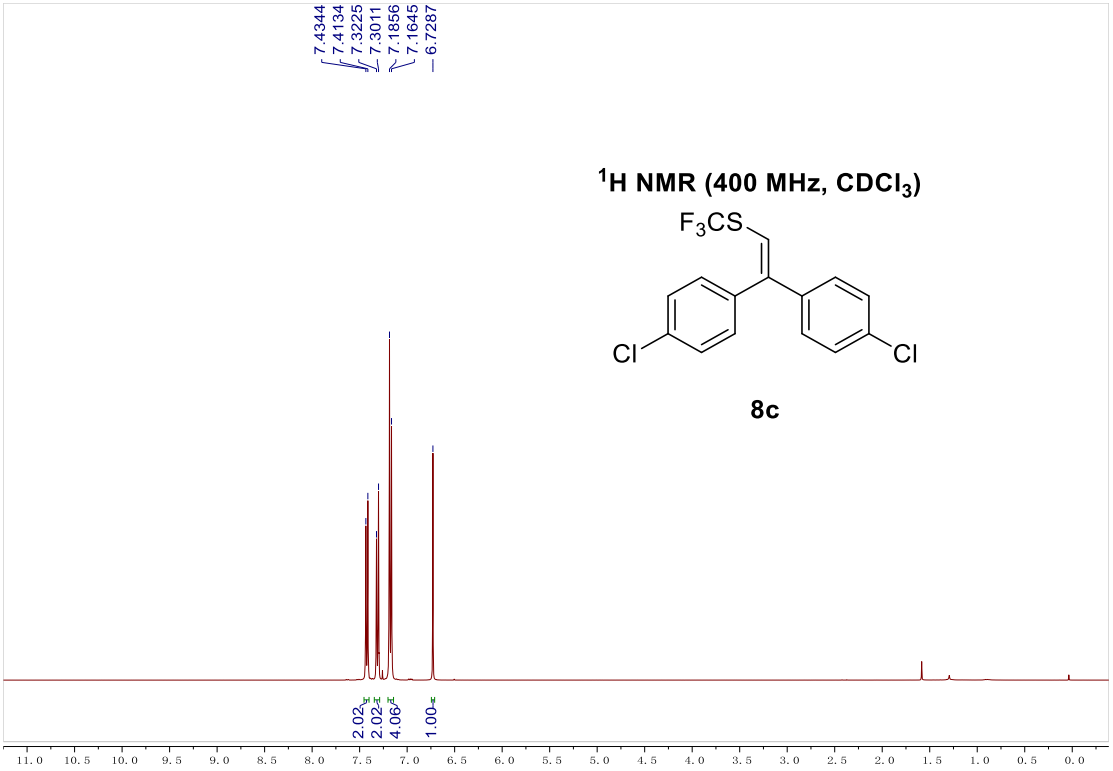

146.5456
140.4067
138.1988
134.5467
131.4877
129.4956
128.7721
128.6940
128.4813
128.4293
127.5452
125.3698
112.3109
112.2785
112.2457
112.2129
^{13}C NMR (101 MHz, CDCl$_3$)
F$_3$CS
8a


-42.5906
19F NMR (471 MHz, CDCl3)
F3CS
8a


7.2897
7.2740
7.2057
7.2012
7.1956
7.1825
7.1702
7.1665
6.6905
2.4586
2.4082
1H NMR (500 MHz, CDCl3)
F3CS
Me
Me
8b
1.96
6.12
1.00
3.04
3.17

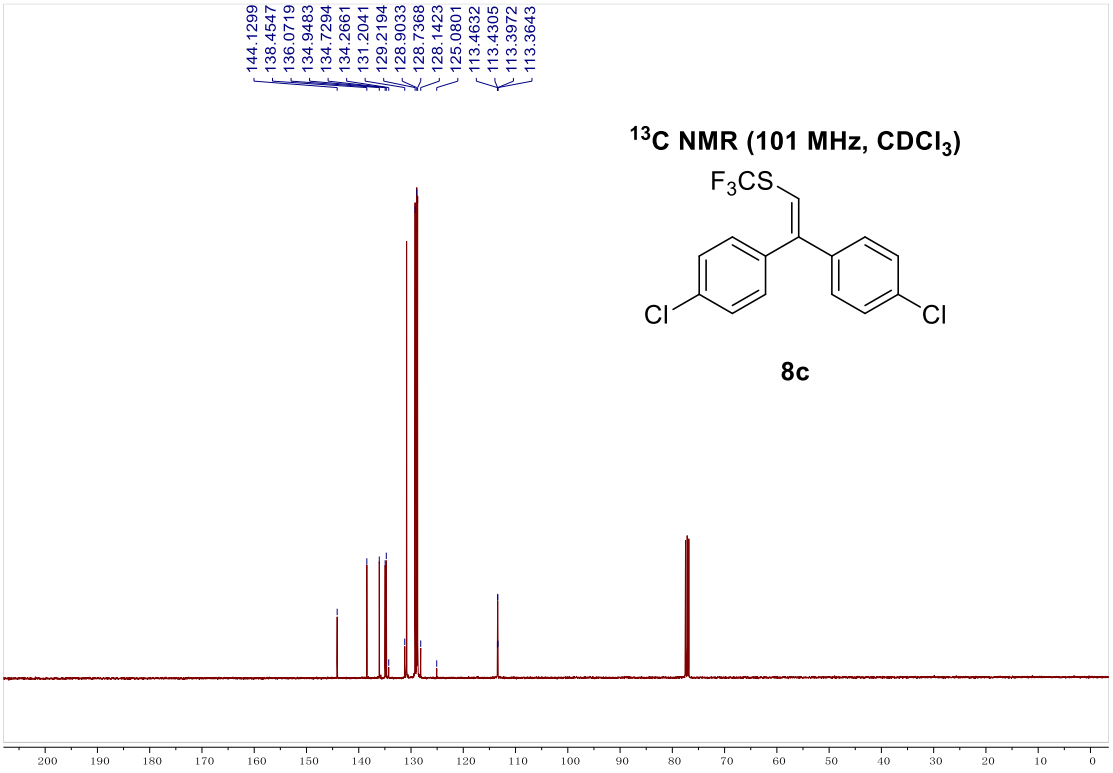

146.8237
138.5505
138.4484
137.8722
135.4211
134.6071
131.5492
129.4284
129.3998
129.2611
128.4900
127.5265
125.4327
110.7915
110.7615
110.7284
110.6984
21.4571
21.2669
13C NMR (101 MHz, CDCl3)
F3CS
Me
Me
8b
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


-42.7008
19F NMR (471 MHz, CDCl3)
F3CS
Me
Me
8b
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200



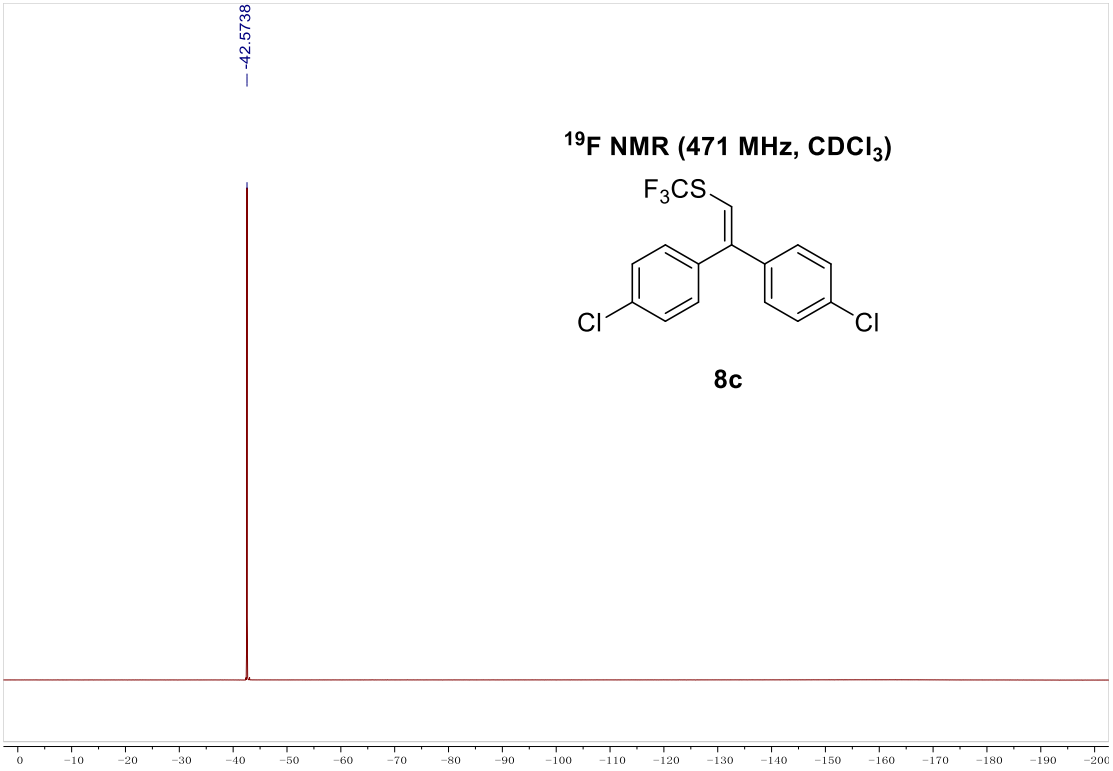
^{1}H NMR (400 MHz, $CDCl_3$)

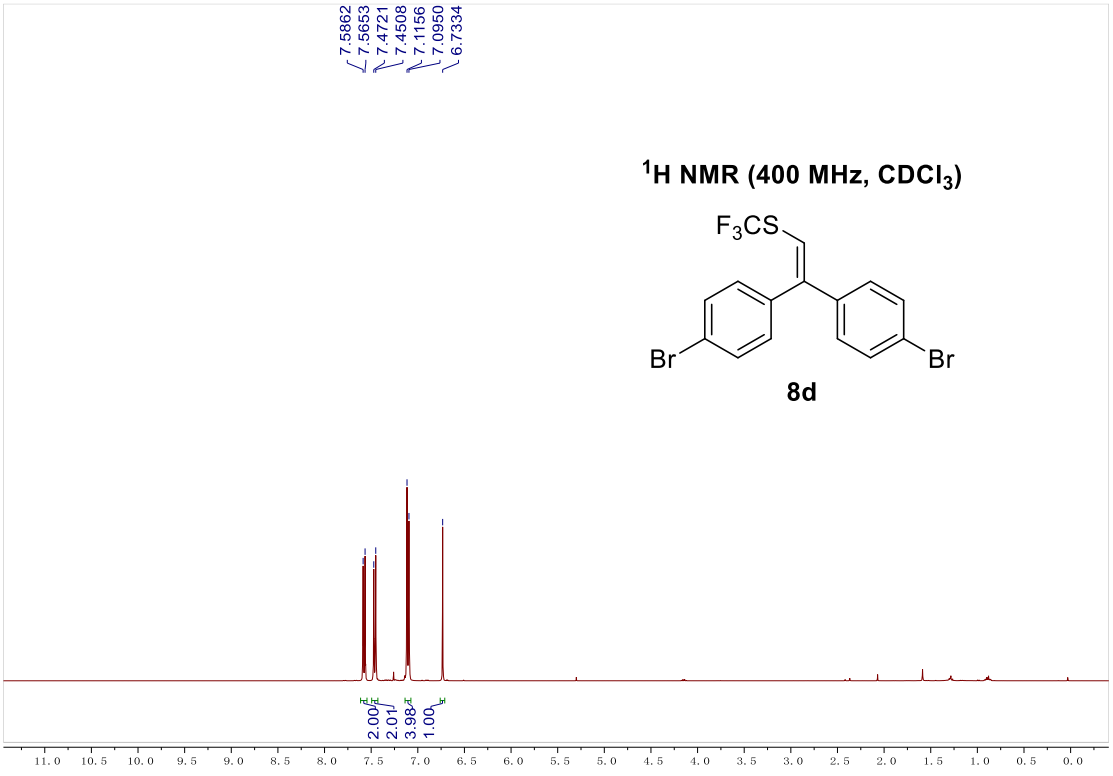
8c

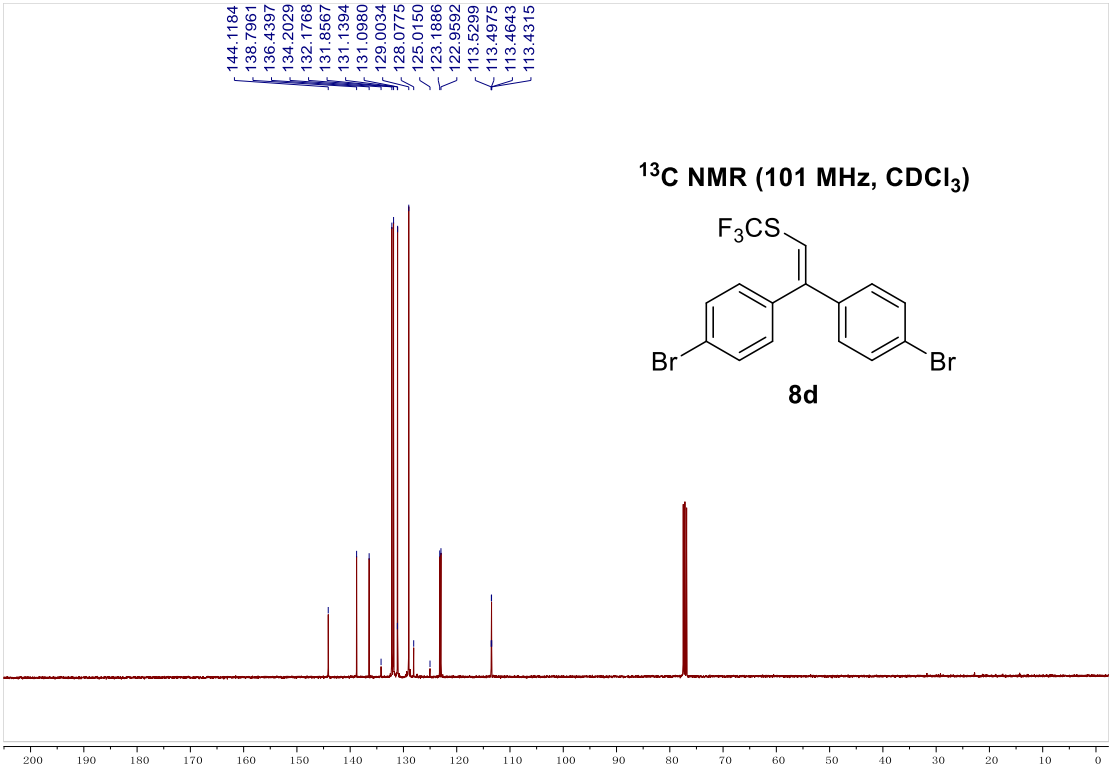


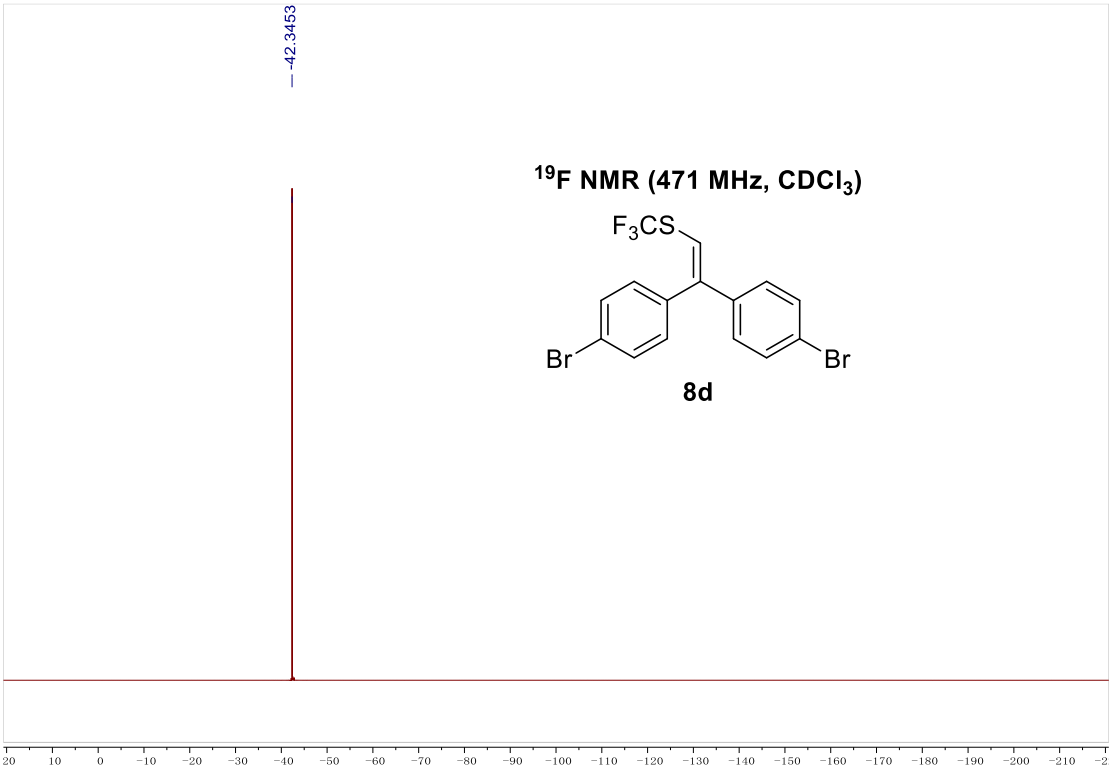
^{13}C NMR (101 MHz, $CDCl_3$)

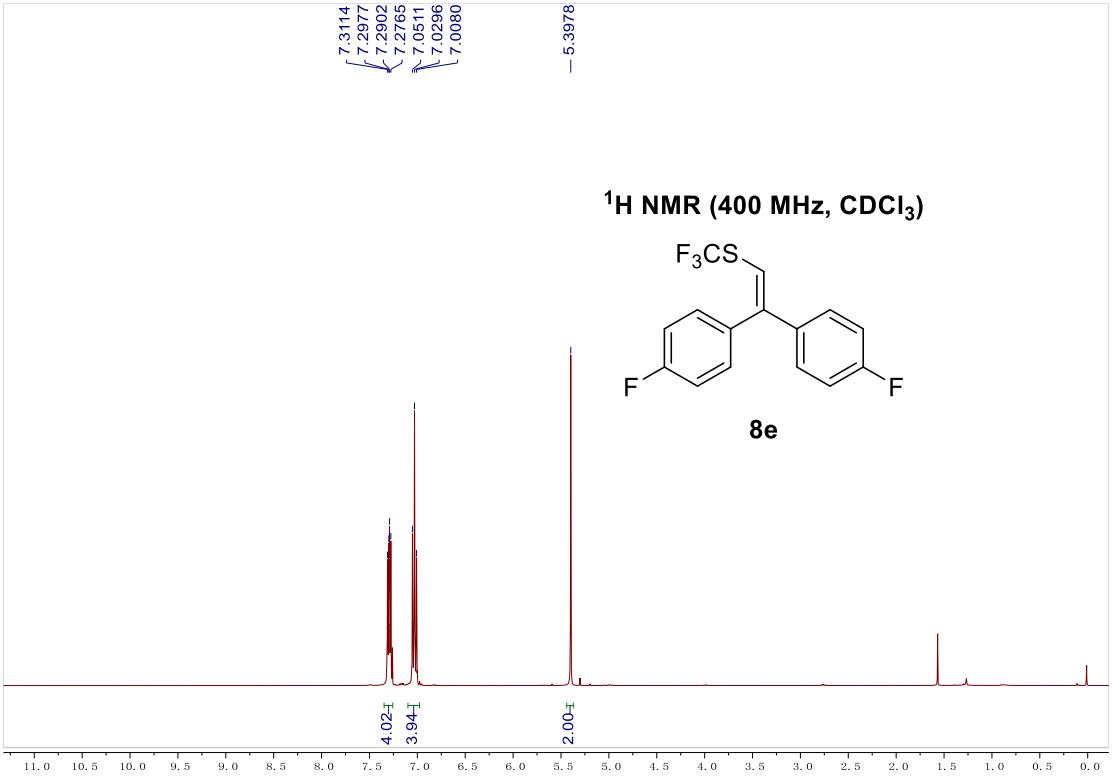
8c

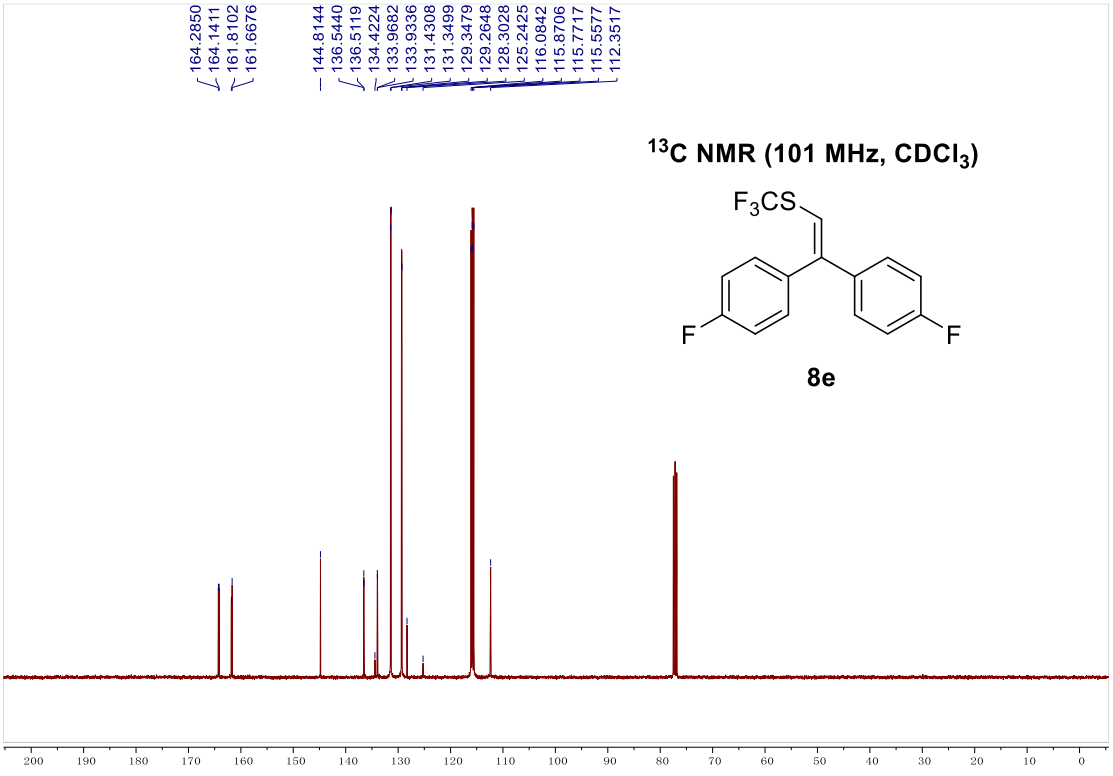

-42.5738
19F NMR (471 MHz, CDCl3)
F3CS
Cl
Cl
8c

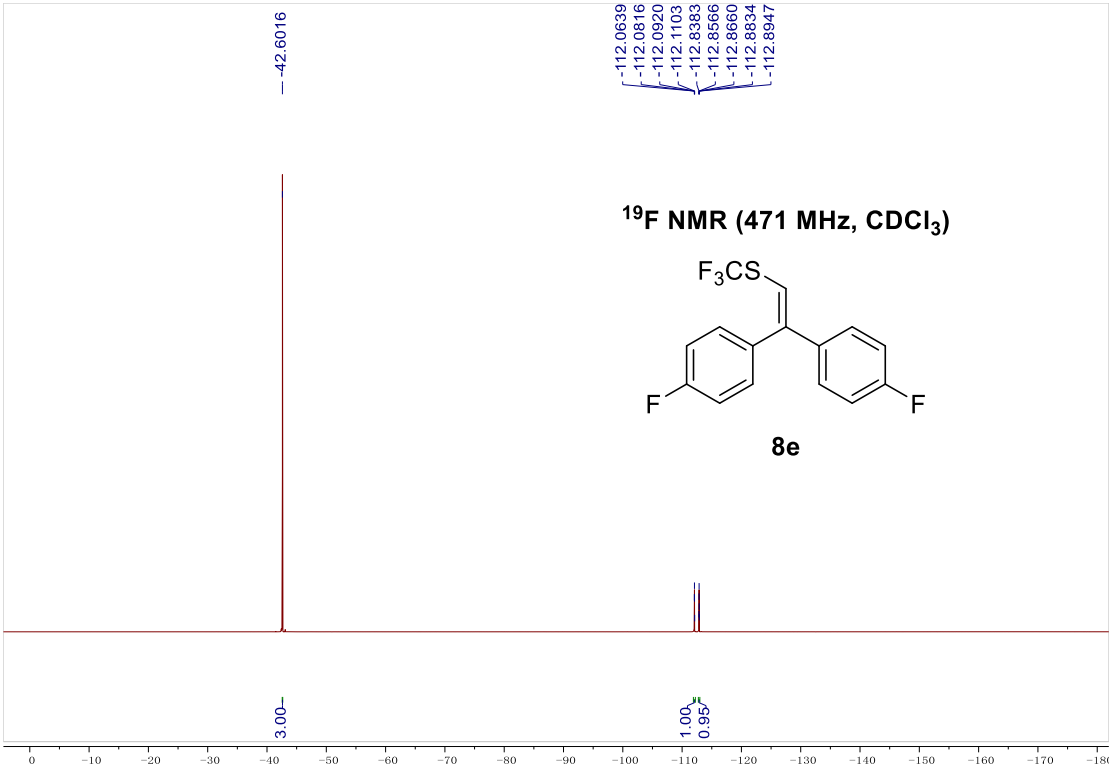

7.5862
7.5653
7.4721
7.4508
7.1156
7.0950
6.7334
1H NMR (400 MHz, CDCl3)
F3CS
Br
Br
8d
2.00
2.01
3.98
1.00

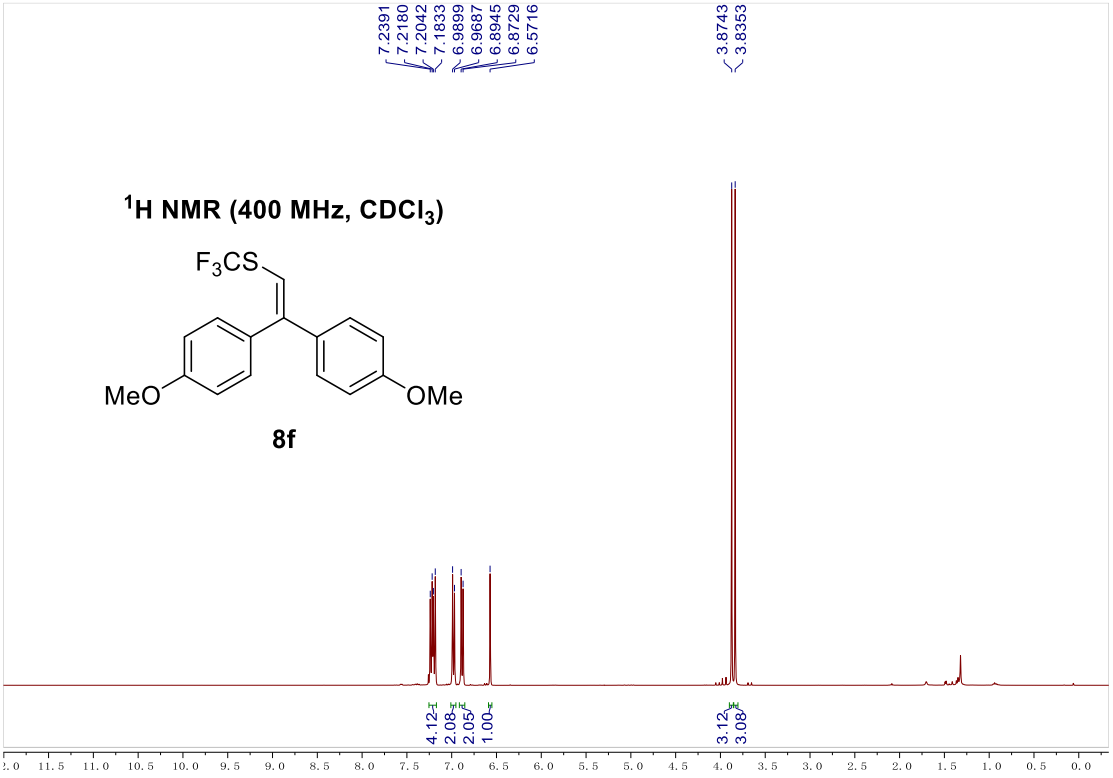

144.1184
138.7961
136.4397
134.2029
132.1768
131.8567
131.1394
131.0980
129.0034
128.0775
125.0150
123.1886
122.9592
113.5299
113.4975
113.4643
113.4315
13C NMR (101 MHz, CDCl3)
F3CS
Br
Br
8d
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

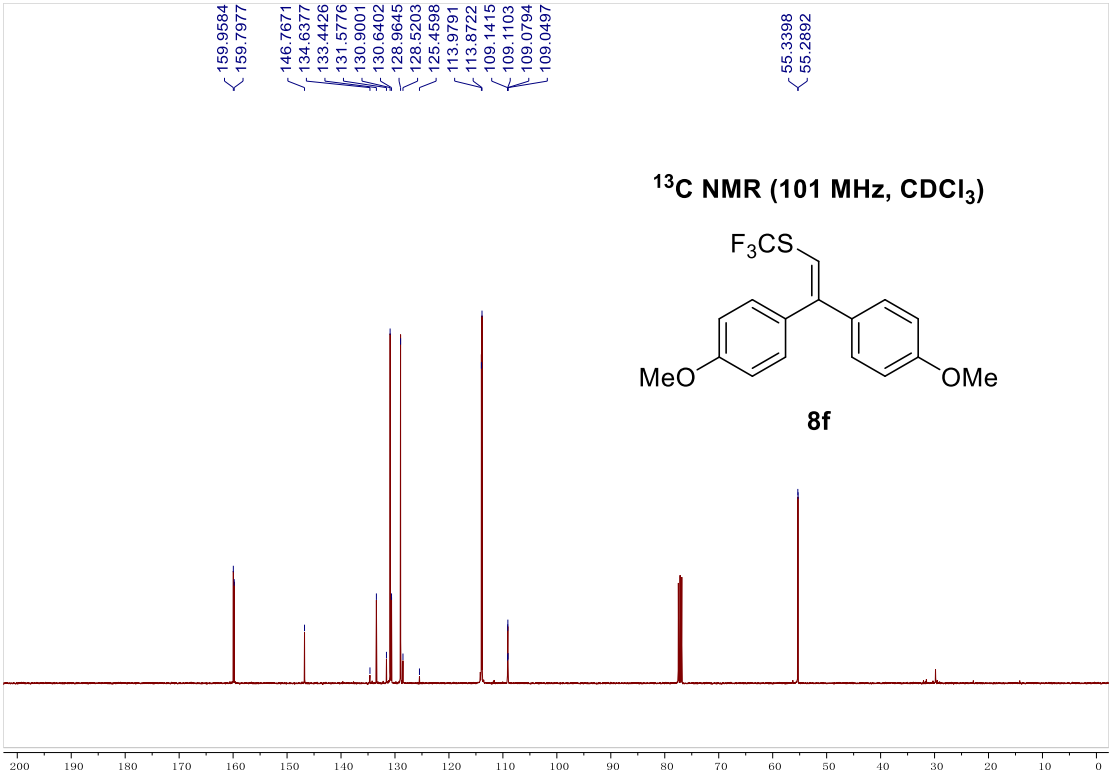

-42.3453
19F NMR (471 MHz, CDCl3)
F3CS
Br
Br
8d
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210

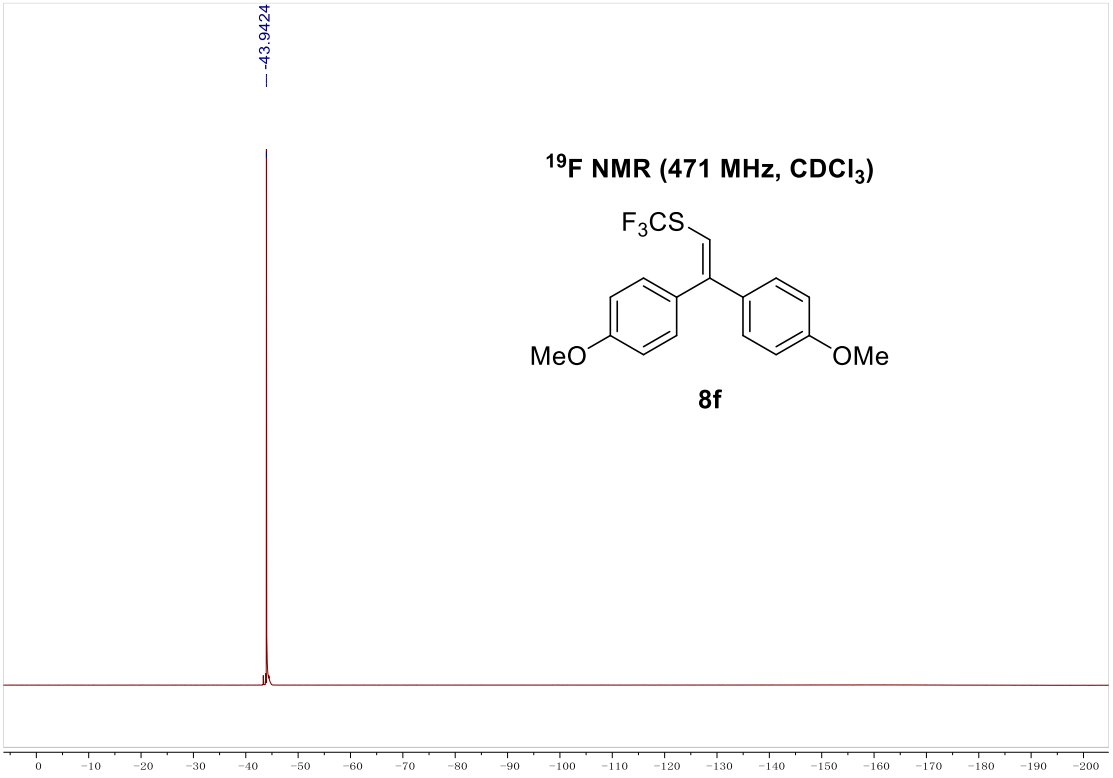

7.3114
7.2977
7.2902
7.2765
7.0511
7.0296
7.0080
5.3978
^{1}H NMR (400 MHz, $CDCl_3$)
F_3CS
F
F
8e
4.02
3.94
2.00

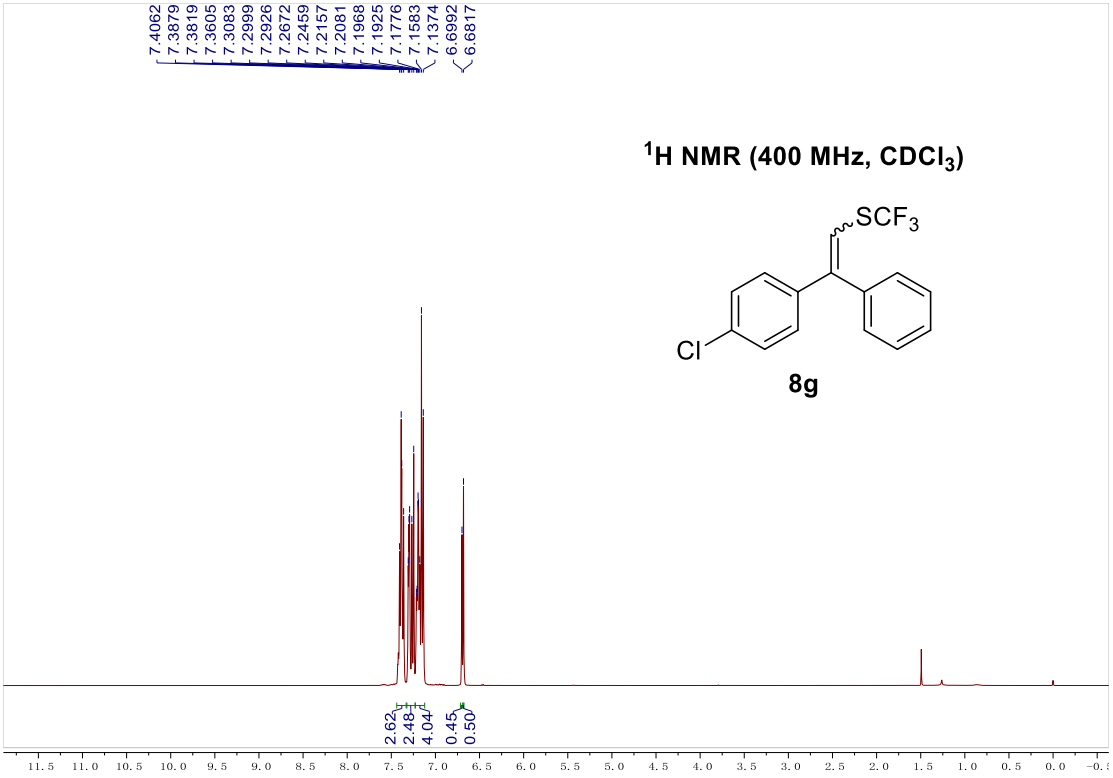

164.2850
164.1411
161.8102
161.6676
144.8144
136.5440
136.5119
134.4224
133.9682
133.9336
131.4308
131.3499
129.3479
129.2648
128.3028
125.2425
116.0842
115.8706
115.7717
115.5577
112.3517
^{13}C NMR (101 MHz, $CDCl_3$)
F_3CS
F
F
8e

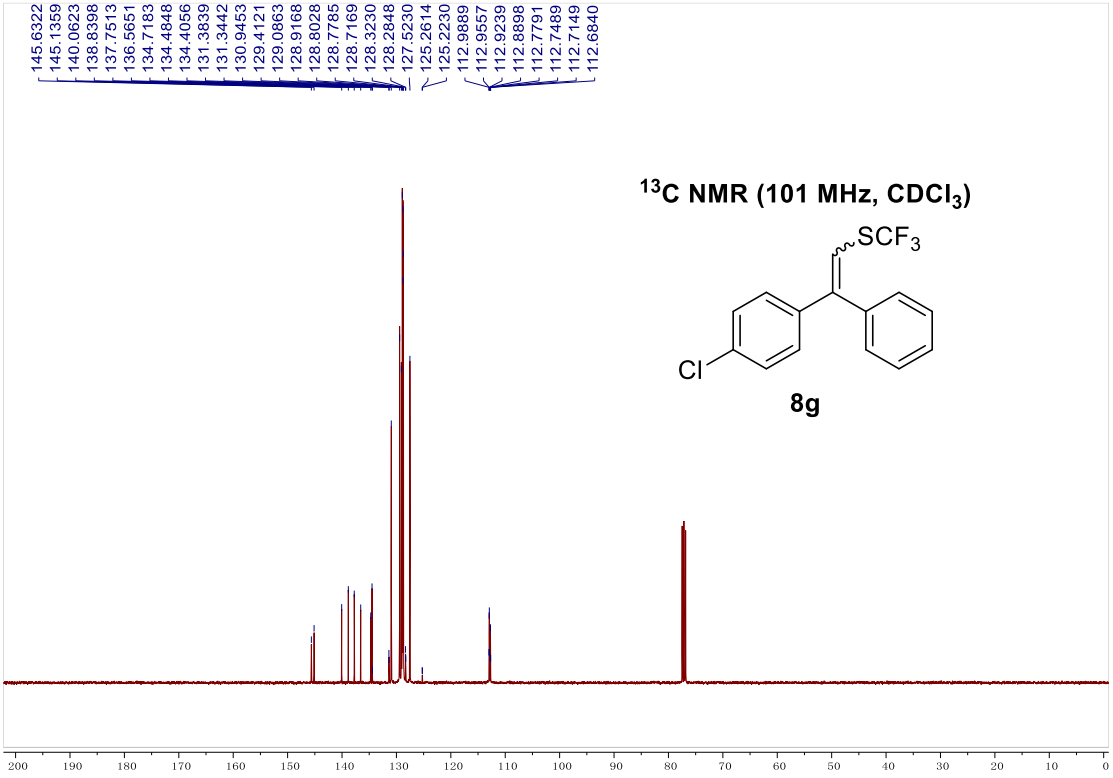

-42.6016
-112.0639
-112.0816
-112.0920
-112.1103
-112.8383
-112.8566
-112.8660
-112.8834
-112.8947
^{19}F NMR (471 MHz, CDCl$_3$)
F$_3$CS
F
F
8e
3.00
1.00
0.95

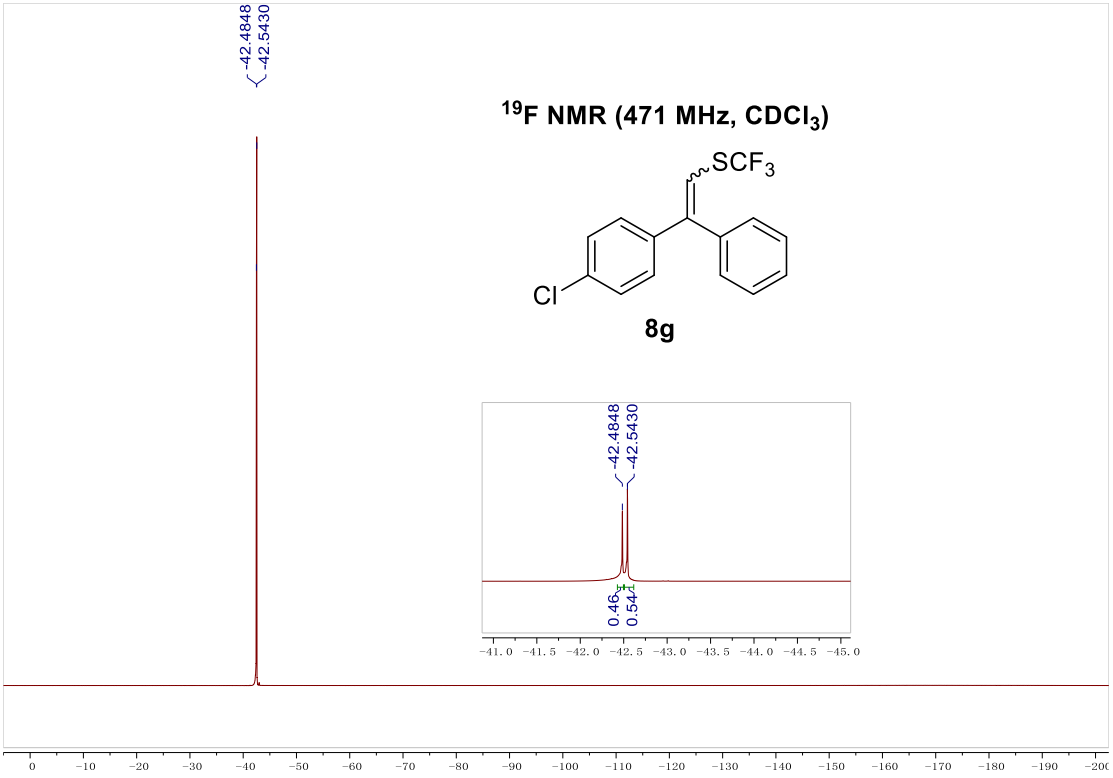

7.2391
7.2180
7.2042
7.1833
6.9899
6.9687
6.8945
6.8729
6.5716
3.8743
3.8353
^{1}H NMR (400 MHz, CDCl$_3$)
F$_3$CS
MeO
OMe
8f
4.12
2.08
2.05
1.00
3.12
3.08

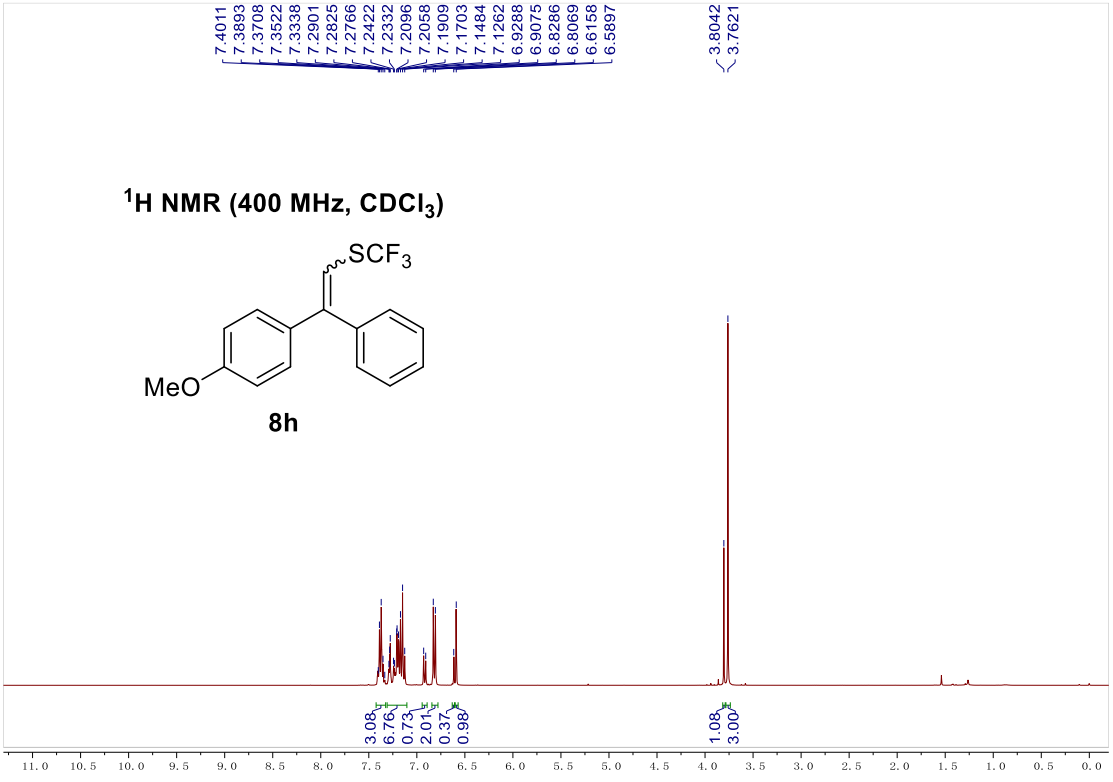

159.9584
159.7977
146.7671
134.6377
133.4426
131.5776
130.9001
130.6402
128.9645
128.5203
125.4598
113.9791
113.8722
109.1415
109.1103
109.0794
109.0497
55.3398
55.2892
13C NMR (101 MHz, CDCl3)
F3CS
MeO
OMe
8f
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


-43.9424
19F NMR (471 MHz, CDCl3)
F3CS
MeO
OMe
8f
0
−10
−20
−30
−40
−50
−60
−70
−80
−90
−100
−110
−120
−130
−140
−150
−160
−170
−180
−190
−200


7.4062
7.3879
7.3819
7.3605
7.3083
7.2999
7.2926
7.2672
7.2459
7.2157
7.2081
7.1968
7.1925
7.1776
7.1583
7.1374
6.6992
6.6817
^{1}H NMR (400 MHz, CDCl$_3$)
SCF$_3$
Cl
8g
2.62
2.48
4.04
0.45
0.50


145.6322
145.1359
140.0623
138.8398
137.7513
136.5651
134.7183
134.4848
134.4056
131.3839
131.3442
130.9453
129.4121
129.0863
128.9168
128.8028
128.7785
128.7169
128.3230
128.2848
127.5230
125.2614
125.2230
112.9889
112.9557
112.9239
112.8898
112.7791
112.7489
112.7149
112.6840
^{13}C NMR (101 MHz, CDCl$_3$)
SCF$_3$
Cl
8g


-42.4848
-42.5430
19F NMR (471 MHz, CDCl3)
SCF3
Cl
8g
0.46
0.54
0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200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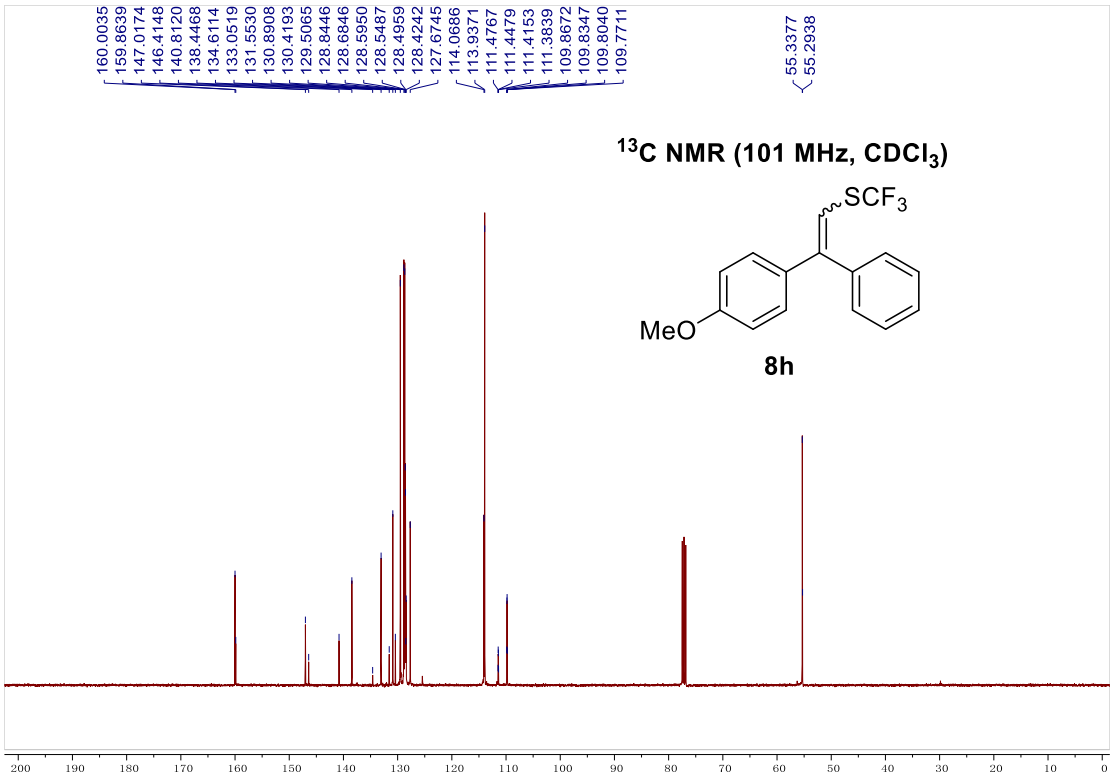
7.3893
7.3708
7.3522
7.3338
7.2901
7.2825
7.2766
7.2422
7.2332
7.2096
7.2058
7.1909
7.1703
7.1484
7.1262
6.9288
6.9075
6.8286
6.8069
6.6158
6.5897
3.8042
3.7621
1H NMR (400 MHz, CDCl3)
SCF3
MeO
8h
3.08
6.76
0.73
2.01
0.37
0.98
1.08
3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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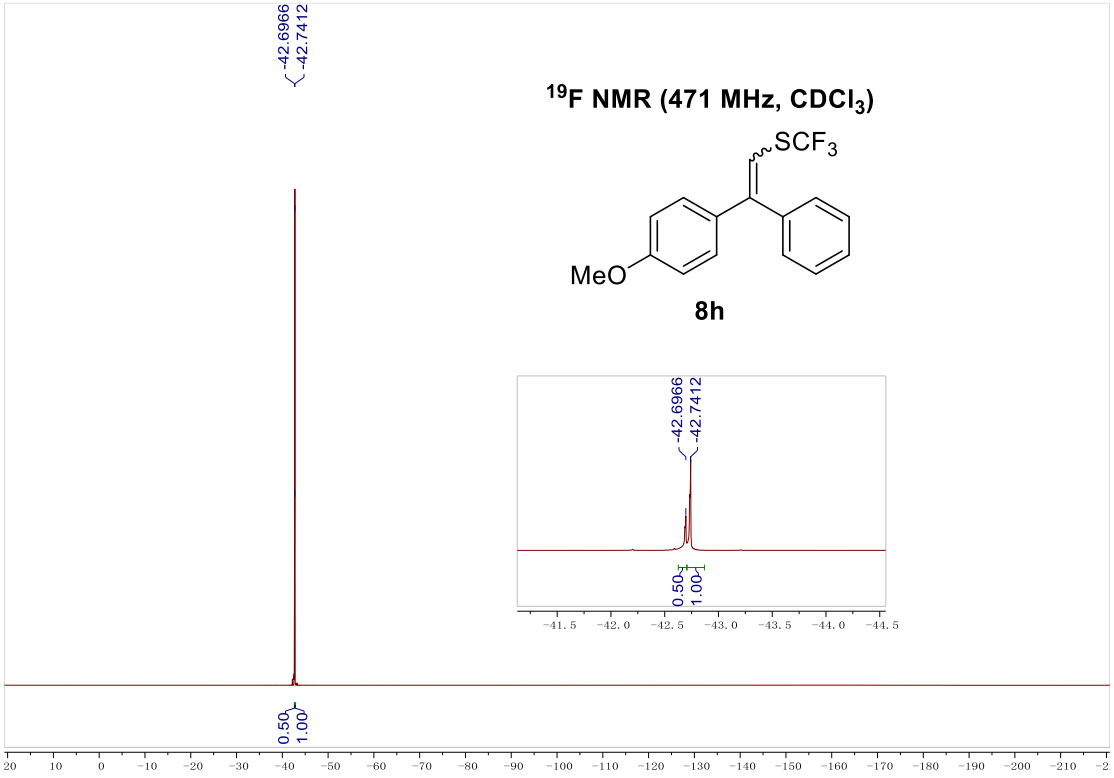
159.8639
147.0174
146.4148
140.8120
138.4468
134.6114
133.0519
131.5530
130.8908
130.4193
129.5065
128.8446
128.6846
128.5950
128.5487
128.4959
128.4242
127.6745
114.0686
113.9371
111.4767
111.4479
111.4153
111.3839
109.8672
109.8347
109.8040
109.7711
55.3377
55.2938
13C NMR (101 MHz, CDCl3)
SCF3
MeO
8h
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


-42.6966
-42.7412
19F NMR (471 MHz, CDCl3)
SCF3
MeO
8h
-42.6966
-42.7412
0.50
1.00
-41.5
-42.0
-42.5
-43.0
-43.5
-44.0
-44.5
0.50
1.00
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
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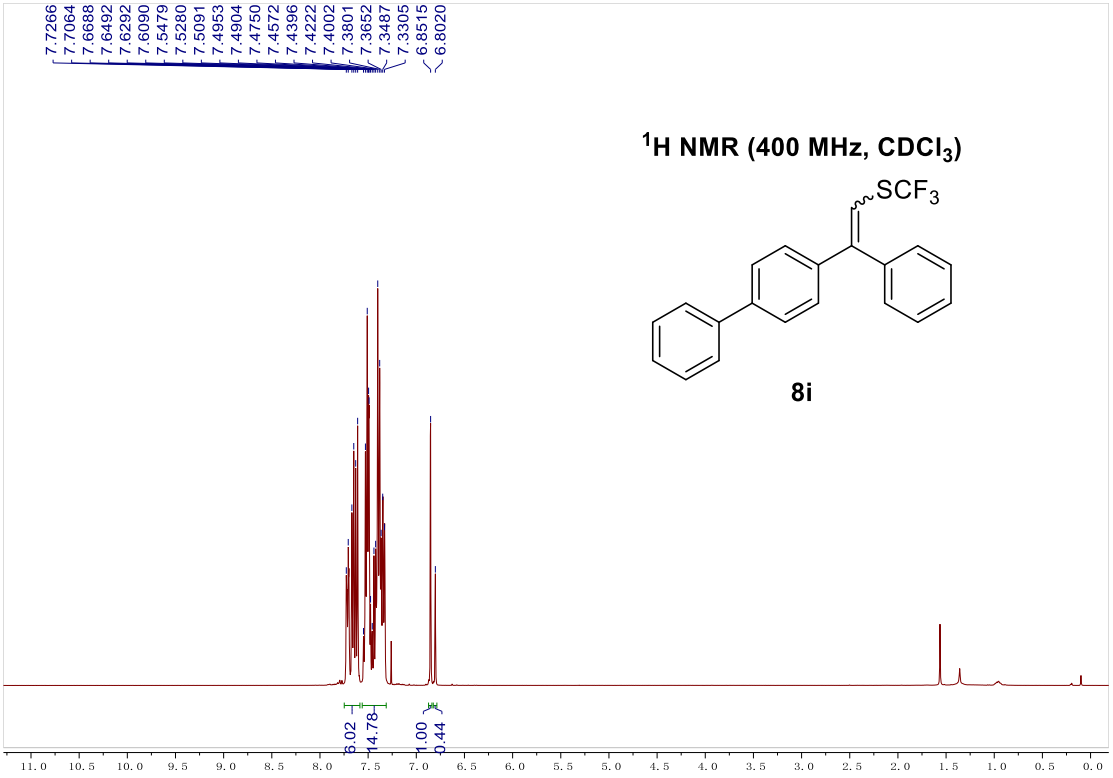
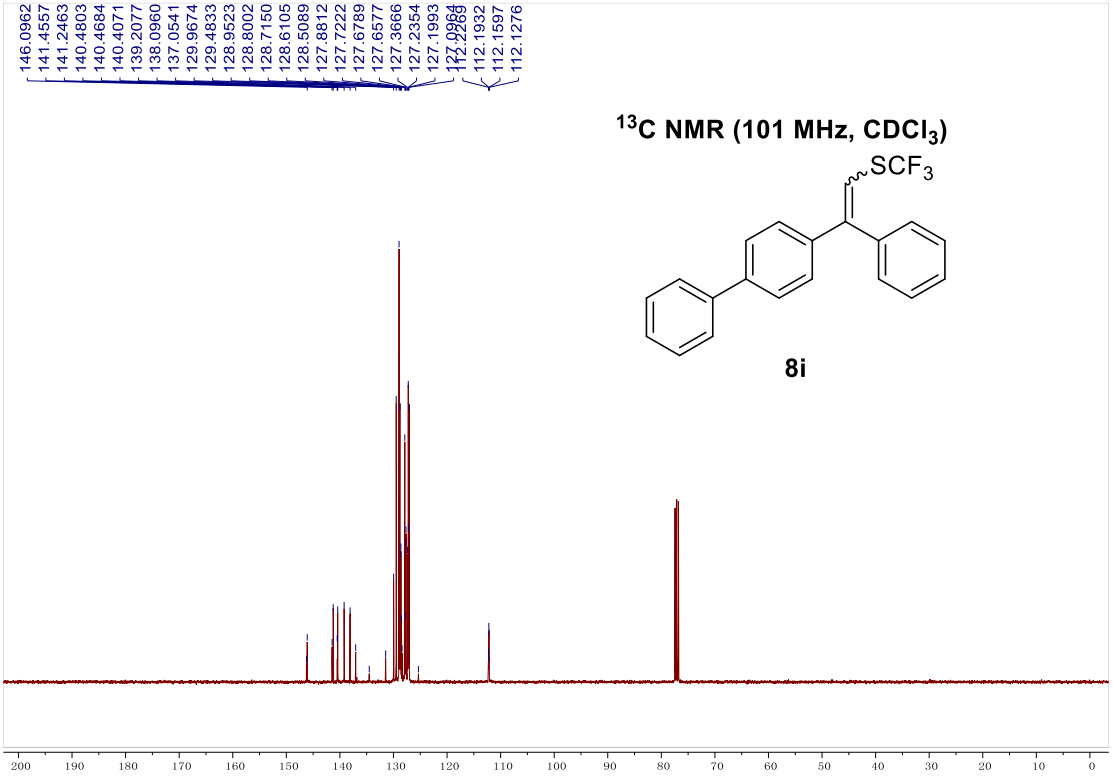
-150
-160
-170
-180
-190
-200
-210


7.7266
7.7064
7.6688
7.6492
7.6292
7.6090
7.5479
7.5280
7.5091
7.4953
7.4904
7.4750
7.4572
7.4396
7.4222
7.4002
7.3801
7.3652
7.3487
7.3305
6.8515
6.8020
^{1}H NMR (400 MHz, CDCl$_3$)
SCF$_3$
8i
6.02
14.78
1.00
0.44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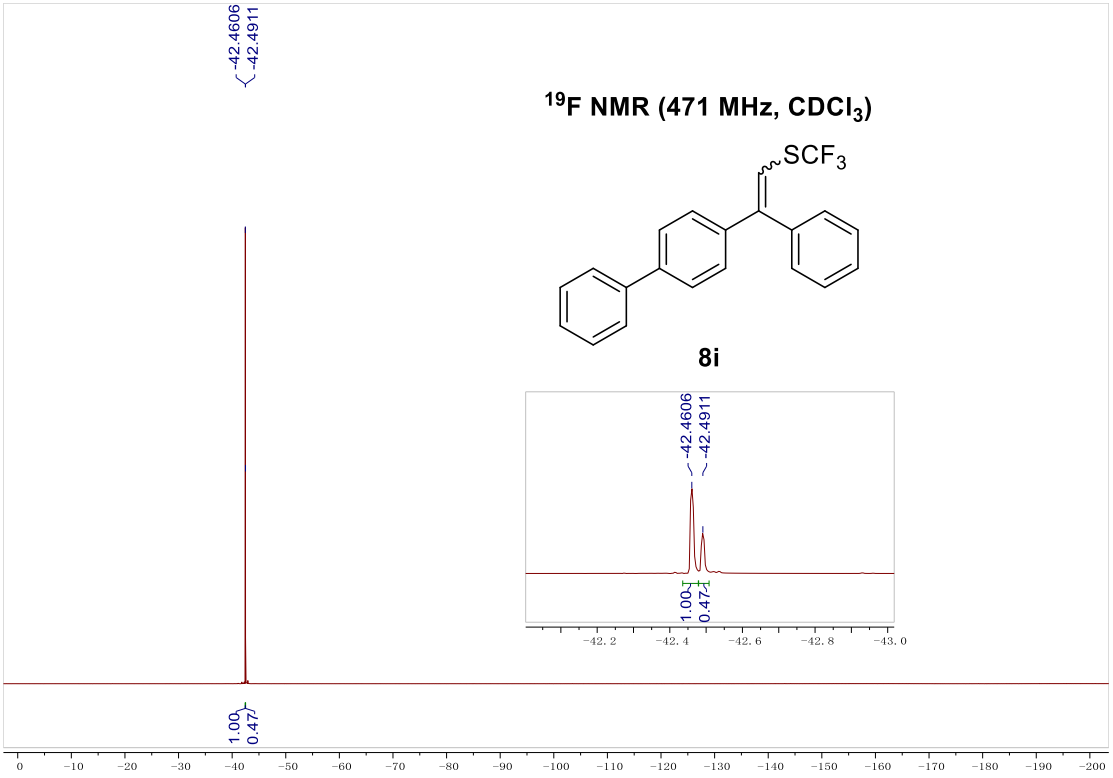
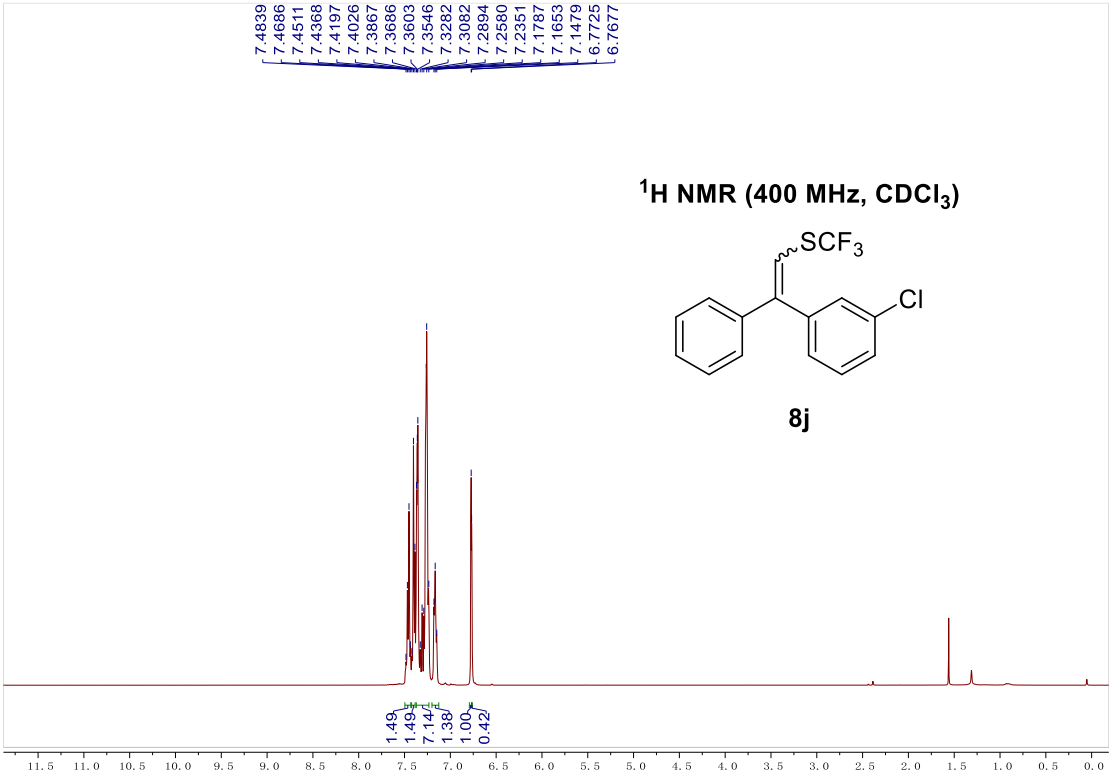
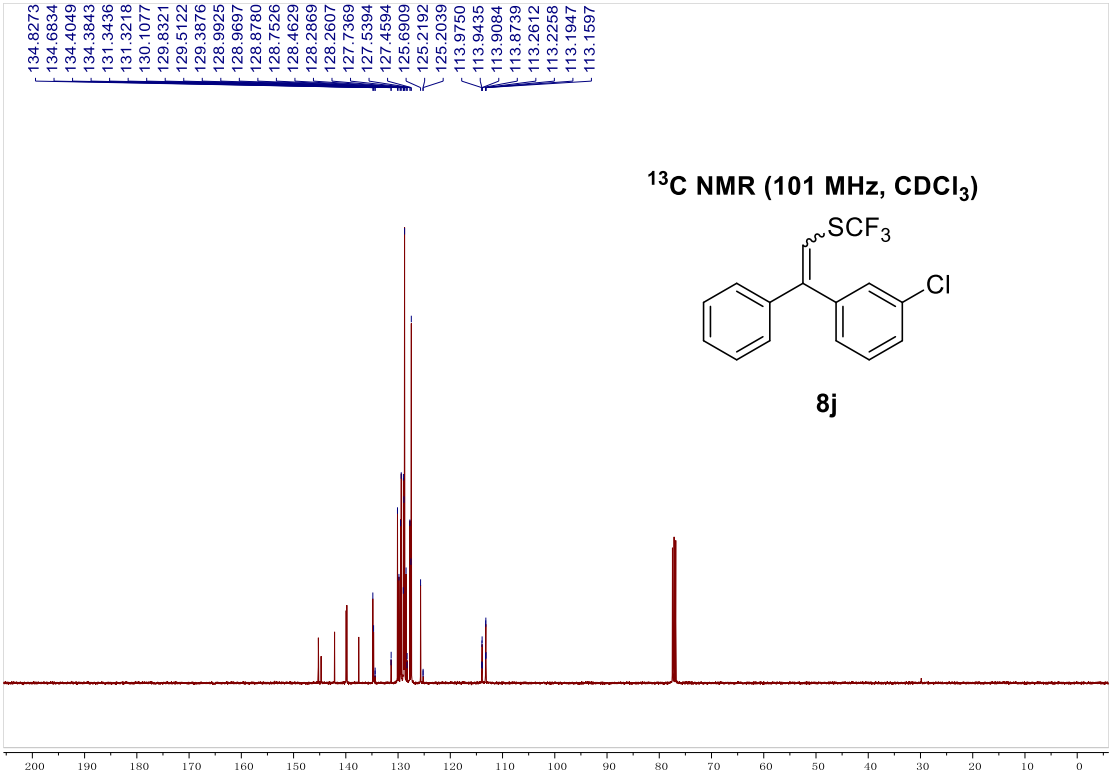
141.4557
141.2463
140.4803
140.4684
140.4071
139.2077
138.0960
137.0541
129.9674
129.4833
128.9523
128.8002
128.7150
128.6105
128.5089
127.8812
127.7222
127.6789
127.6577
127.3666
127.2354
127.1993
112.1932
112.1597
112.1276
^{13}C NMR (101 MHz, CDCl$_3$)
SCF$_3$
8i

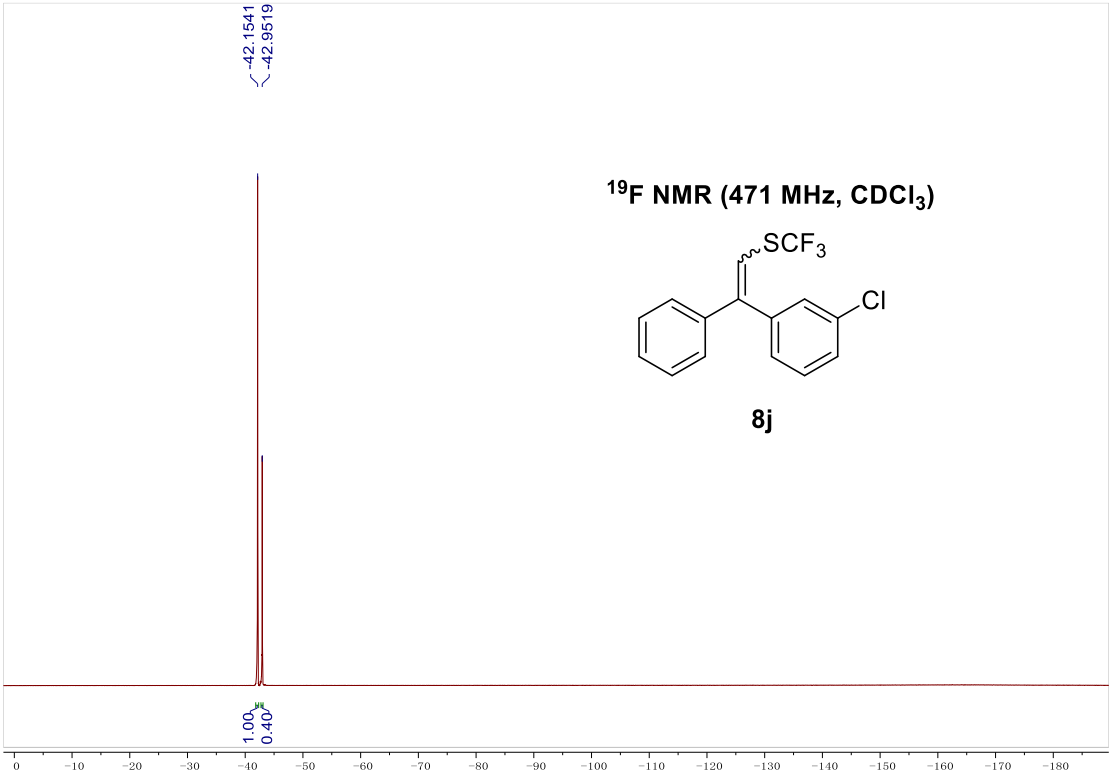

-42.4606
-42.4911
19F NMR (471 MHz, CDCl3)
SCF3
8i
1.00
0.47


7.4839
7.4686
7.4511
7.4368
7.4197
7.4026
7.3867
7.3686
7.3603
7.3546
7.3282
7.3082
7.2894
7.2580
7.2351
7.1787
7.1653
7.1479
6.7725
6.7677
1H NMR (400 MHz, CDCl3)
SCF3
Cl
8j
1.49
1.49
7.14
1.38
1.00
0.42



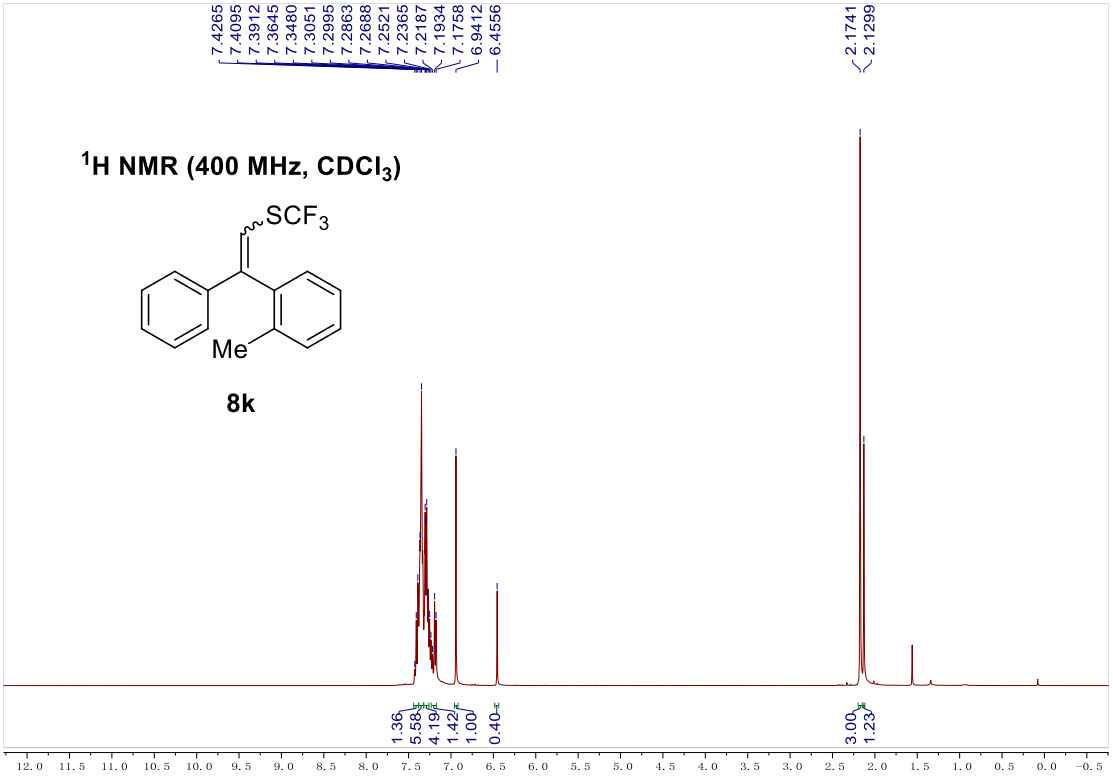
^{13}C NMR (101 MHz, $CDCl_3$)

8j



^{19}F NMR (471 MHz, $CDCl_3$)

8j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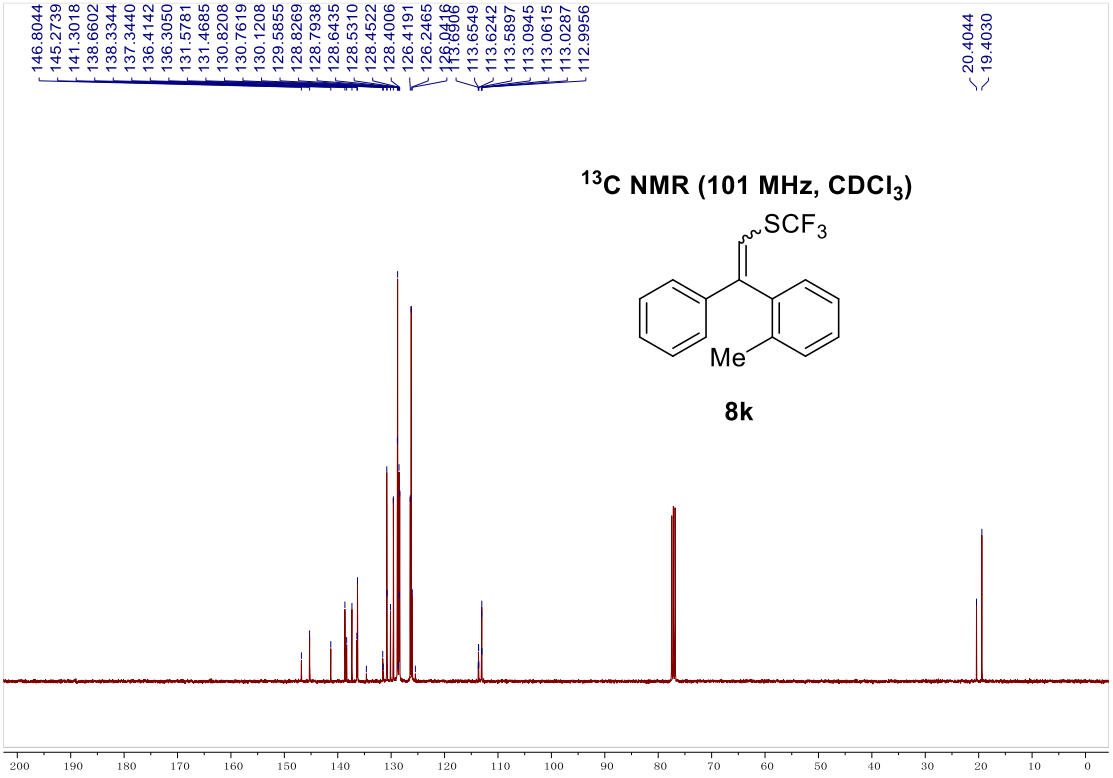
7.4095
7.3912
7.3645
7.3480
7.3051
7.2995
7.2863
7.2688
7.2521
7.2365
7.2187
7.1934
7.1758
6.9412
6.4556
2.1741
2.1299
1H NMR (400 MHz, CDCl3)
SCF3
Me
8k
1.36
5.58
4.19
1.42
1.00
0.40
3.00
1.23
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5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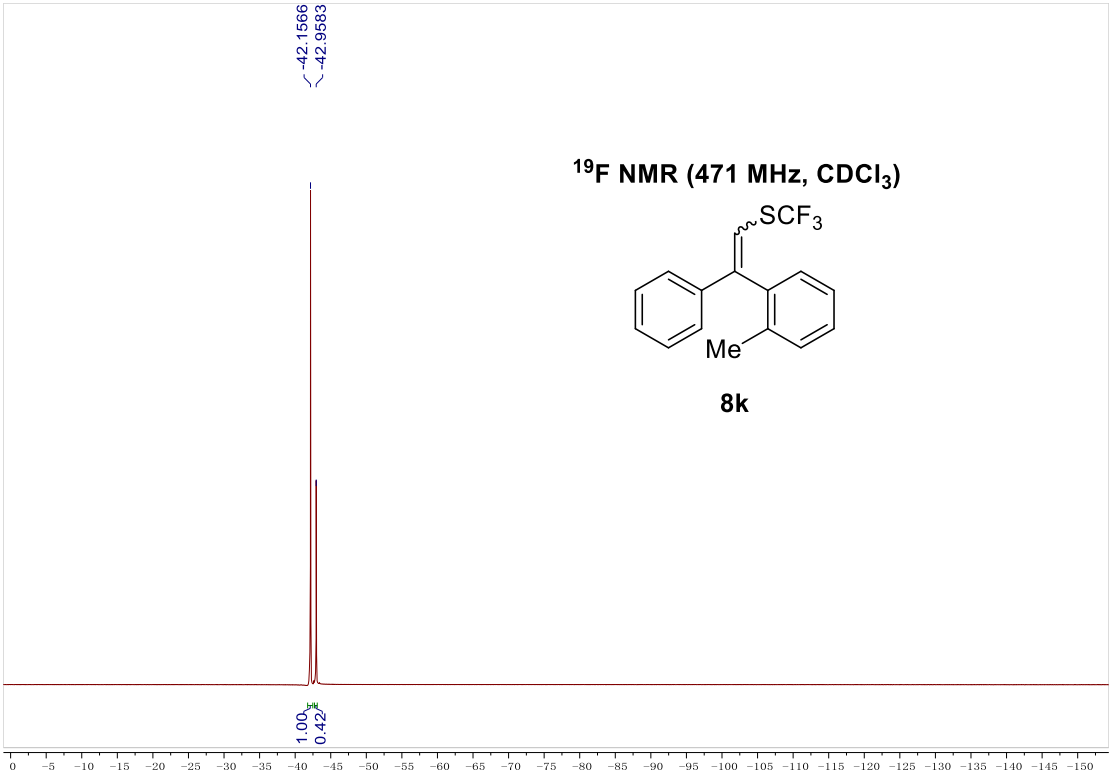
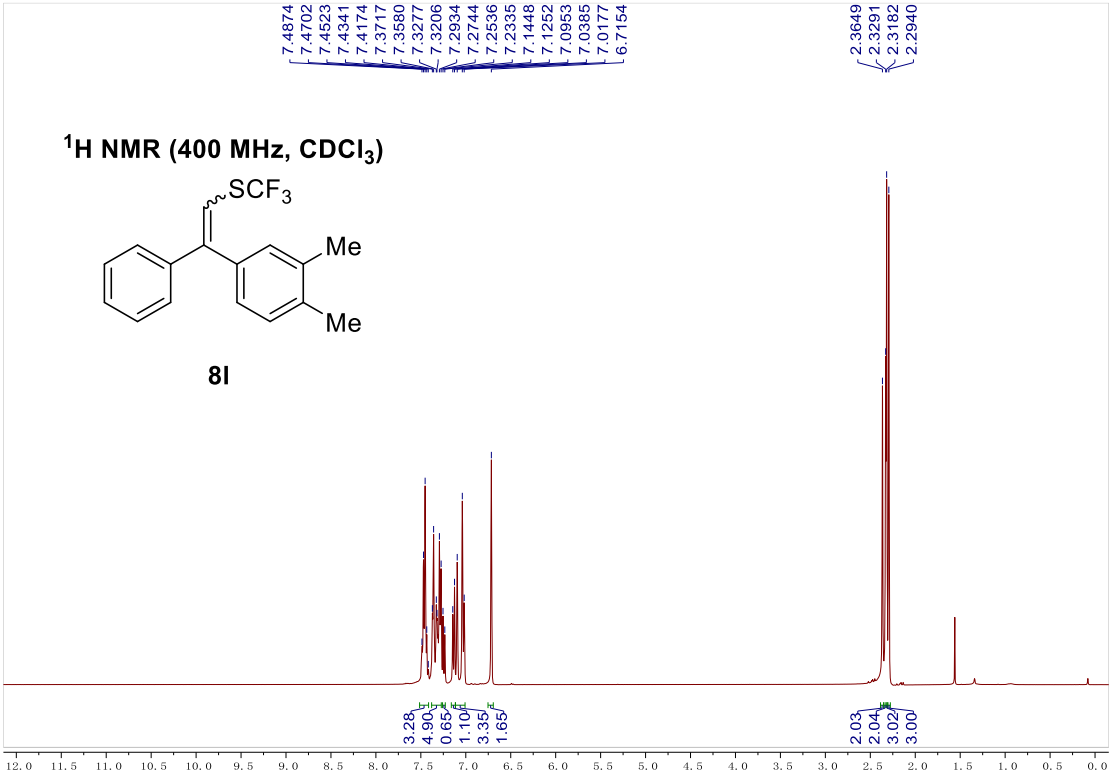
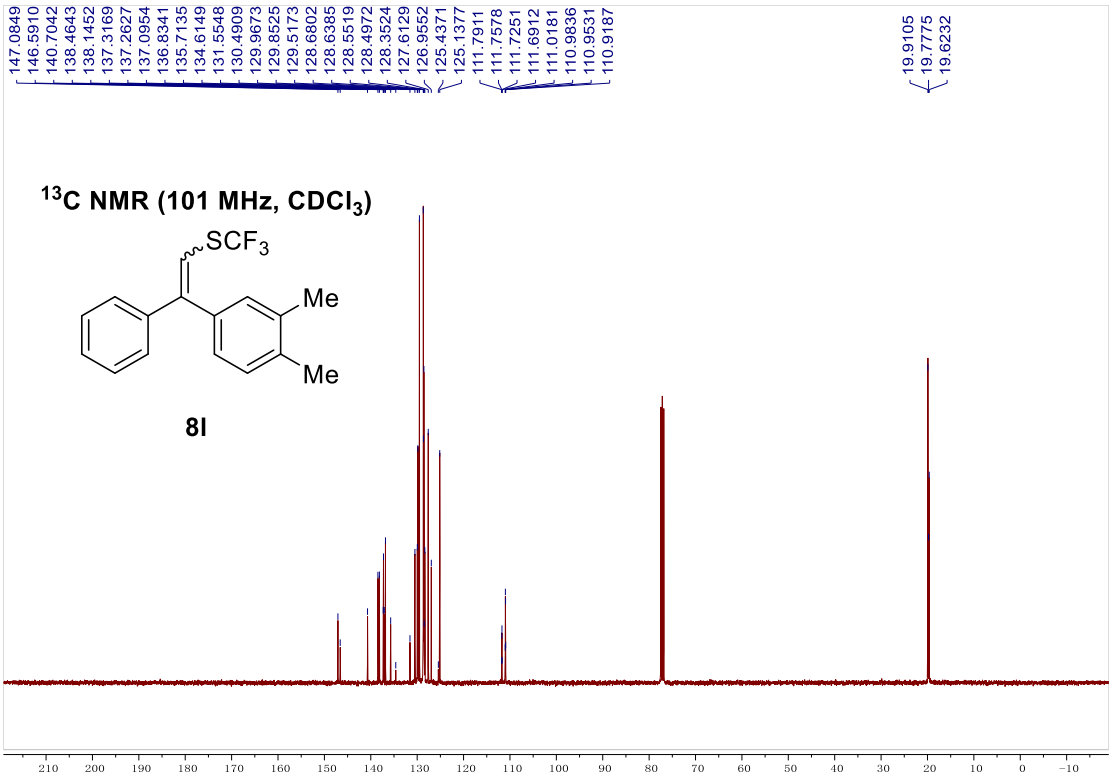
145.2739
141.3018
138.6602
138.3344
137.3440
136.4142
136.3050
131.5781
131.4685
130.8208
130.7619
130.1208
129.5855
128.8269
128.7938
128.6435
128.5310
128.4522
128.4006
126.4191
126.2465
126.0416
113.6906
113.6549
113.6242
113.5897
113.0945
113.0615
113.0287
112.9956
20.4044
19.4030
13C NMR (101 MHz, CDCl3)
SCF3
Me
8k
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0


-42.1566
-42.9583
19F NMR (471 MHz, CDCl3)
SCF3
Me
8k
1.00
0.42


7.4874
7.4702
7.4523
7.4341
7.4174
7.3717
7.3580
7.3277
7.3206
7.2934
7.2744
7.2536
7.2335
7.1448
7.1252
7.0953
7.0385
7.0177
6.7154
2.3649
2.3291
2.3182
2.2940
1H NMR (400 MHz, CDCl3)
SCF3
Me
Me
8l
3.28
4.90
0.65
1.10
3.35
1.65
2.03
2.04
3.02
3.00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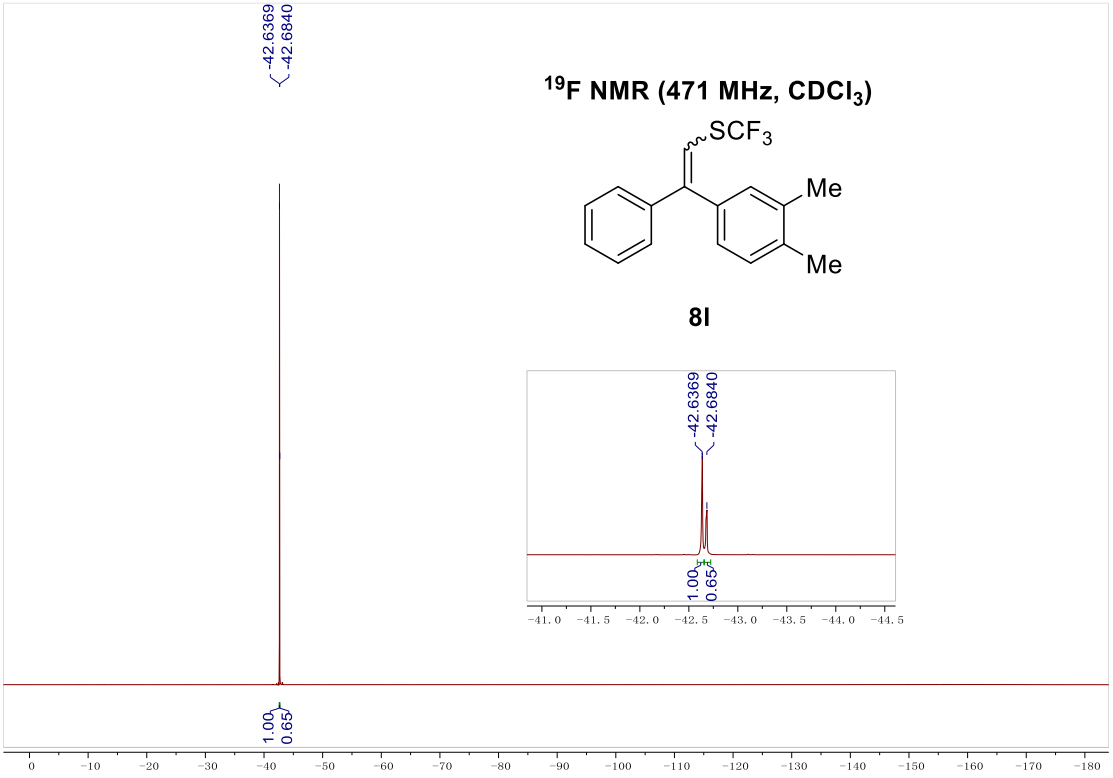
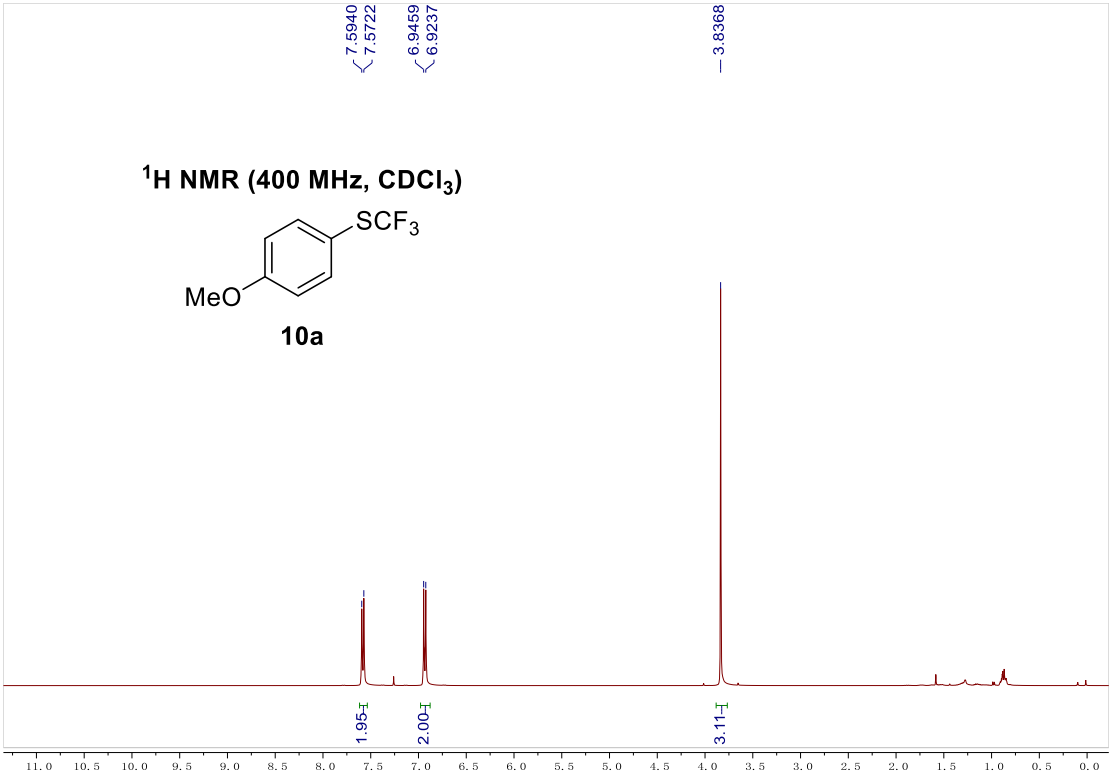
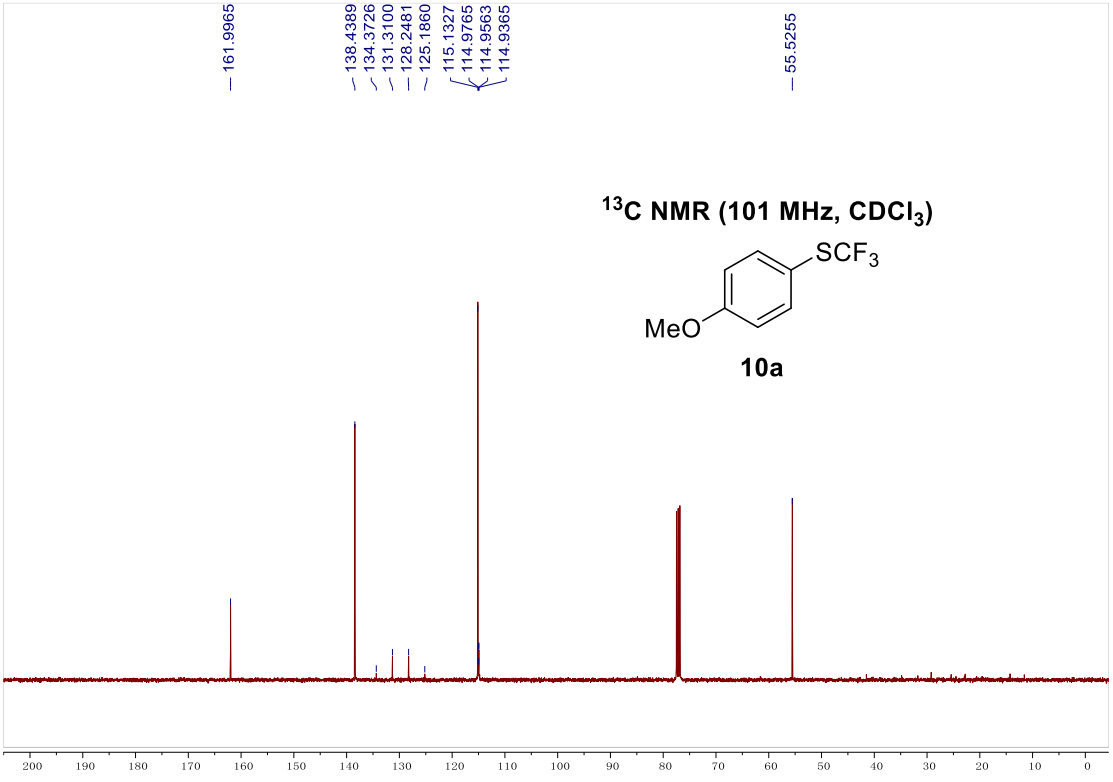
140.7042
138.4643
138.1452
137.3169
137.2627
137.0954
136.8341
135.7135
134.6149
131.5548
130.4909
129.9673
129.8525
129.5173
128.6802
128.6385
128.5519
128.4972
128.3524
127.6129
126.9552
125.4371
125.1377
111.7911
111.7578
111.7251
111.6912
111.0181
110.9836
110.9531
110.9187
19.9105
19.7775
19.6232
13C NMR (101 MHz, CDCl3)
SCF3
Me
Me
8l
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10


-42.6369
-42.6840
19F NMR (471 MHz, CDCl3)
SCF3
Me
Me
8l
-42.6369
-42.6840
1.00
0.65
−41.0 −41.5 −42.0 −42.5 −43.0 −43.5 −44.0 −44.5
1.00
0.65
0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180


7.5940
7.5722
6.9459
6.9237
3.8368
1H NMR (400 MHz, CDCl3)
SCF3
MeO
10a
1.95
2.00
3.11
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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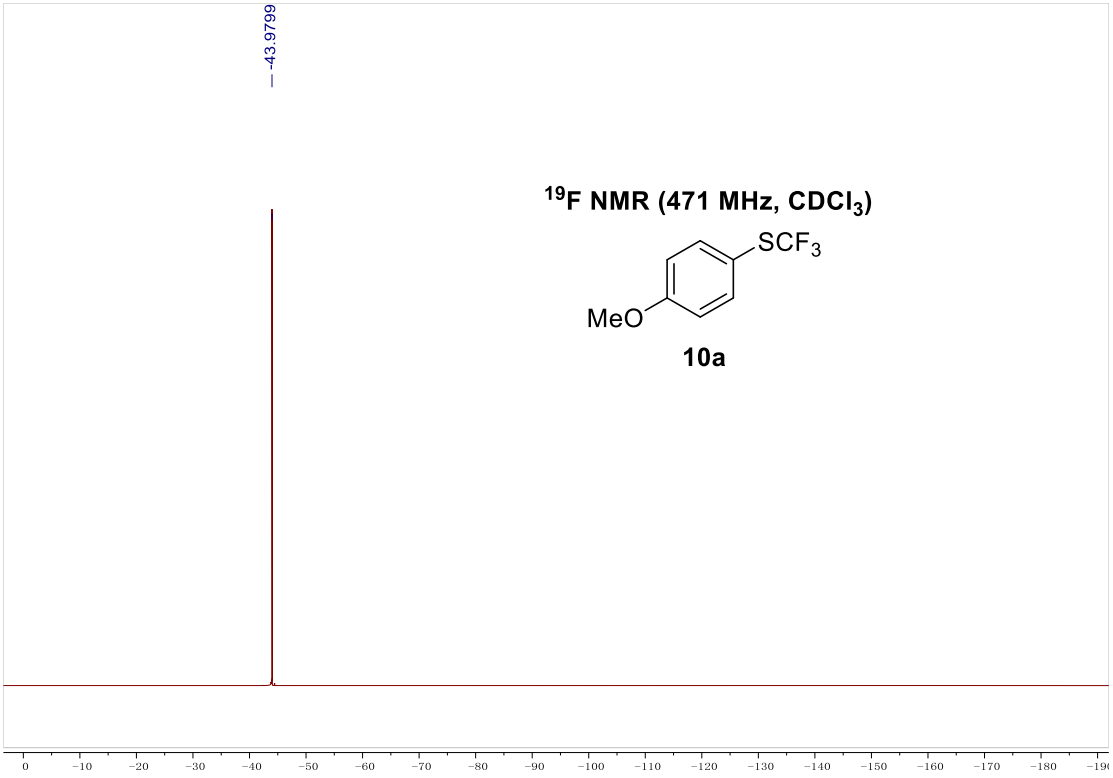
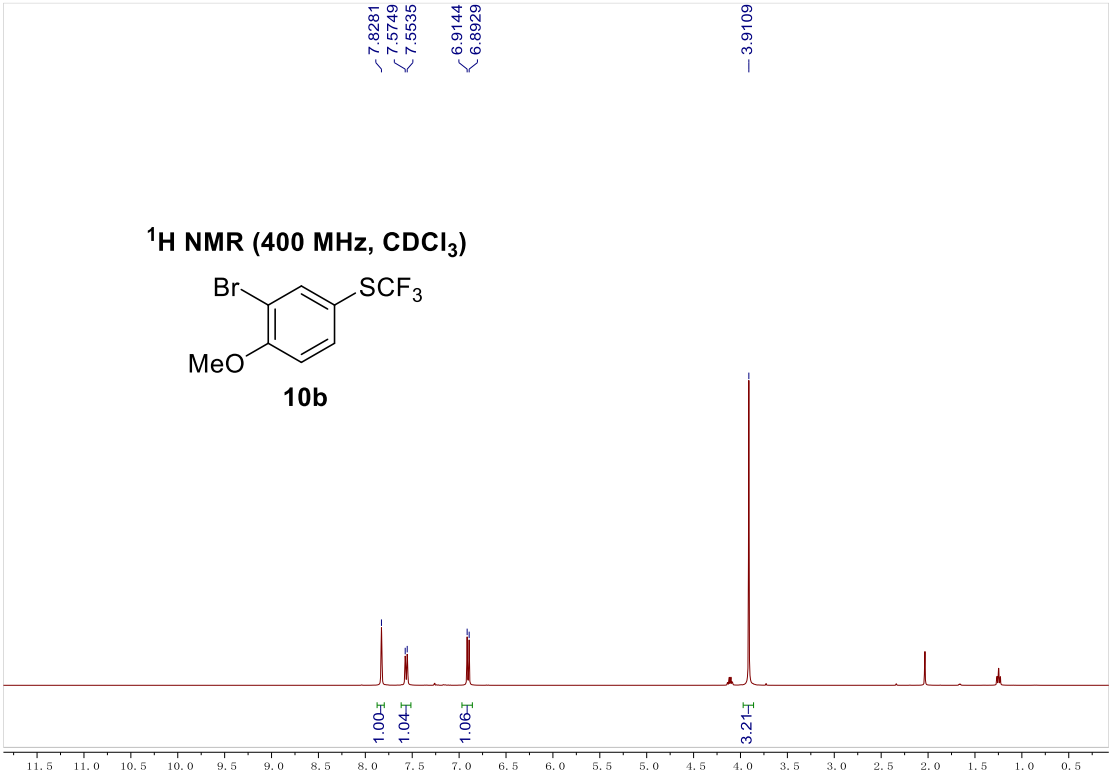
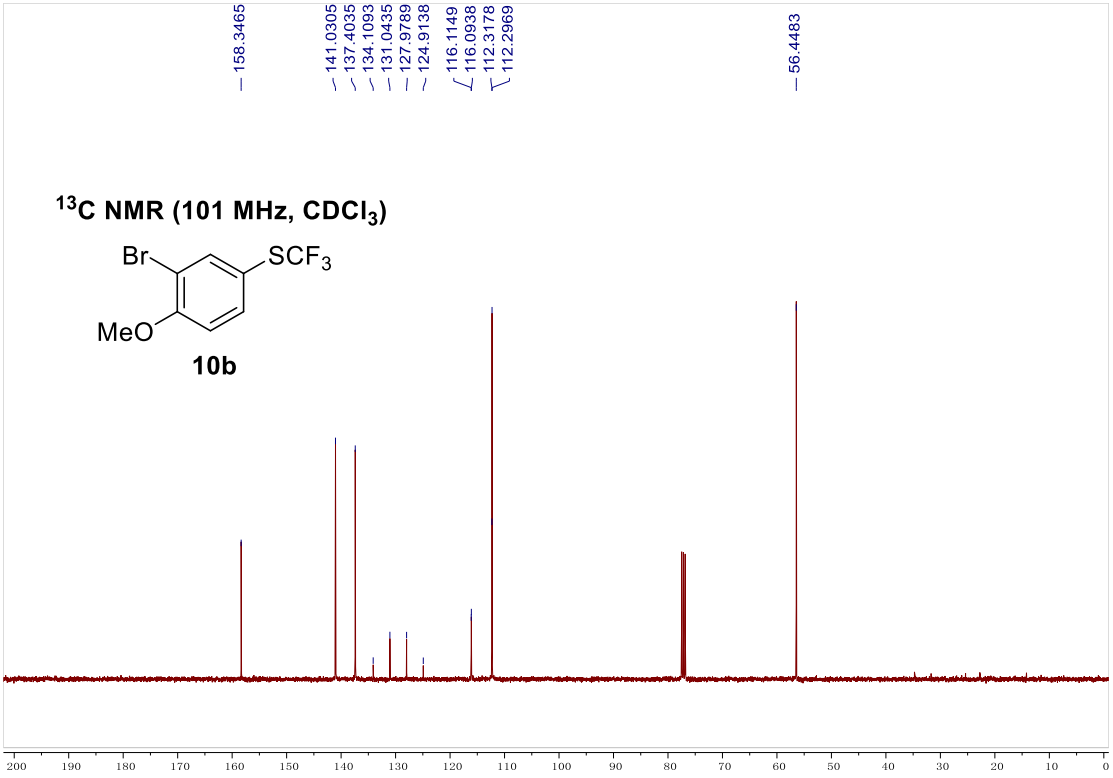
138.4389
134.3726
131.3100
128.2481
125.1860
115.1327
114.9765
114.9563
114.9365
55.5255
13C NMR (101 MHz, CDCl3)
SCF3
MeO
10a
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0


-43.9799
19F NMR (471 MHz, CDCl3)
SCF3
MeO
10a


7.8281
7.5749
7.5535
6.9144
6.8929
3.9109
1H NMR (400 MHz, CDCl3)
Br
SCF3
MeO
10b
1.00
1.04
1.06
3.21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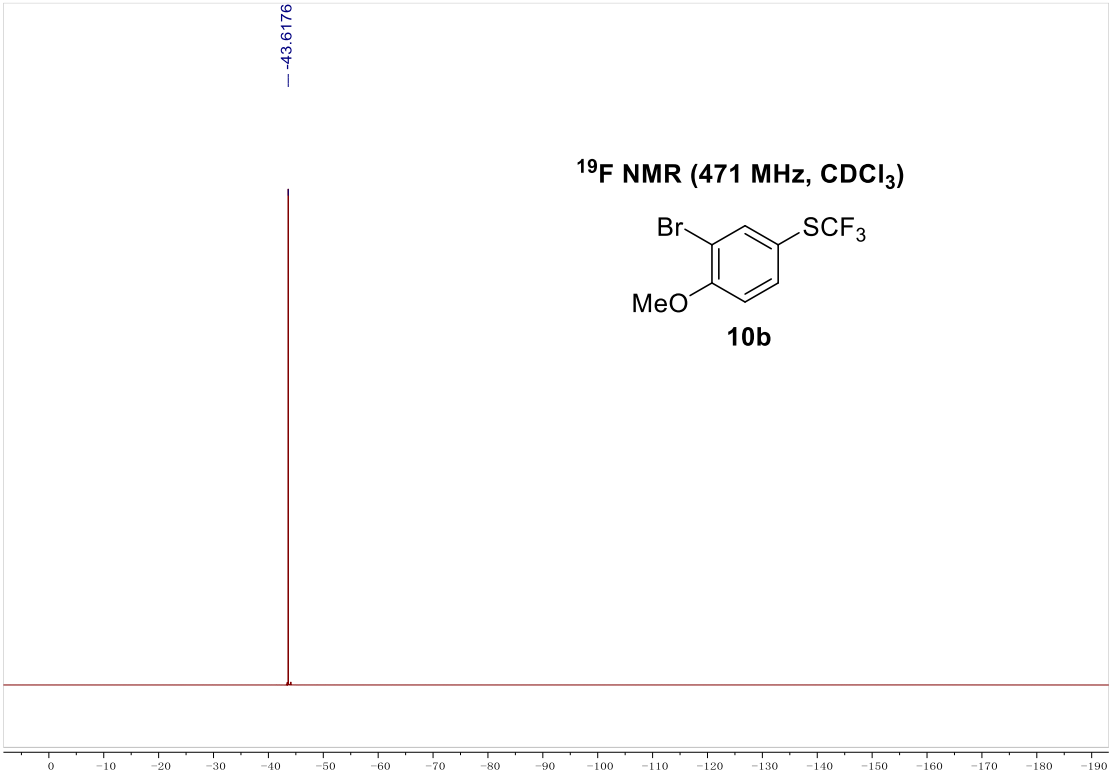
141.0305
137.4035
134.1093
131.0435
127.9789
124.9138
116.1149
116.0938
112.3178
112.2969
56.4483
13C NMR (101 MHz, CDCl3)
Br
SCF3
MeO
10b
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0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Br
SCF3
MeO
10b
0
-10
-20
-30
-40
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-60
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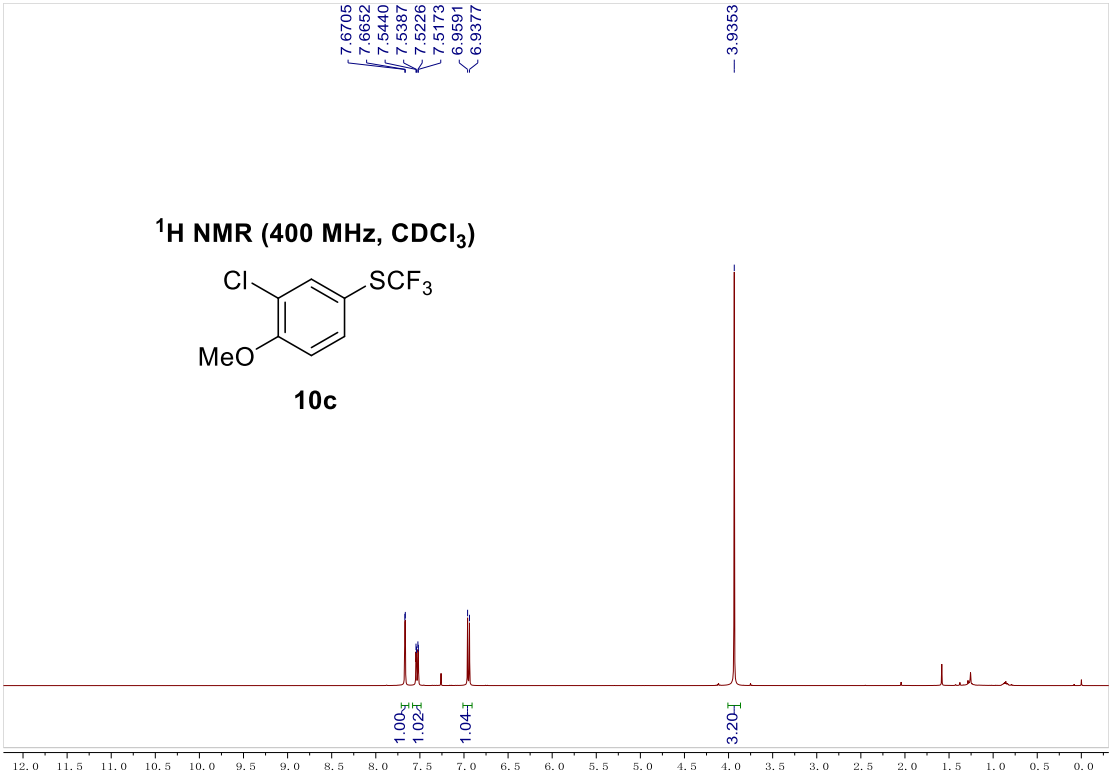
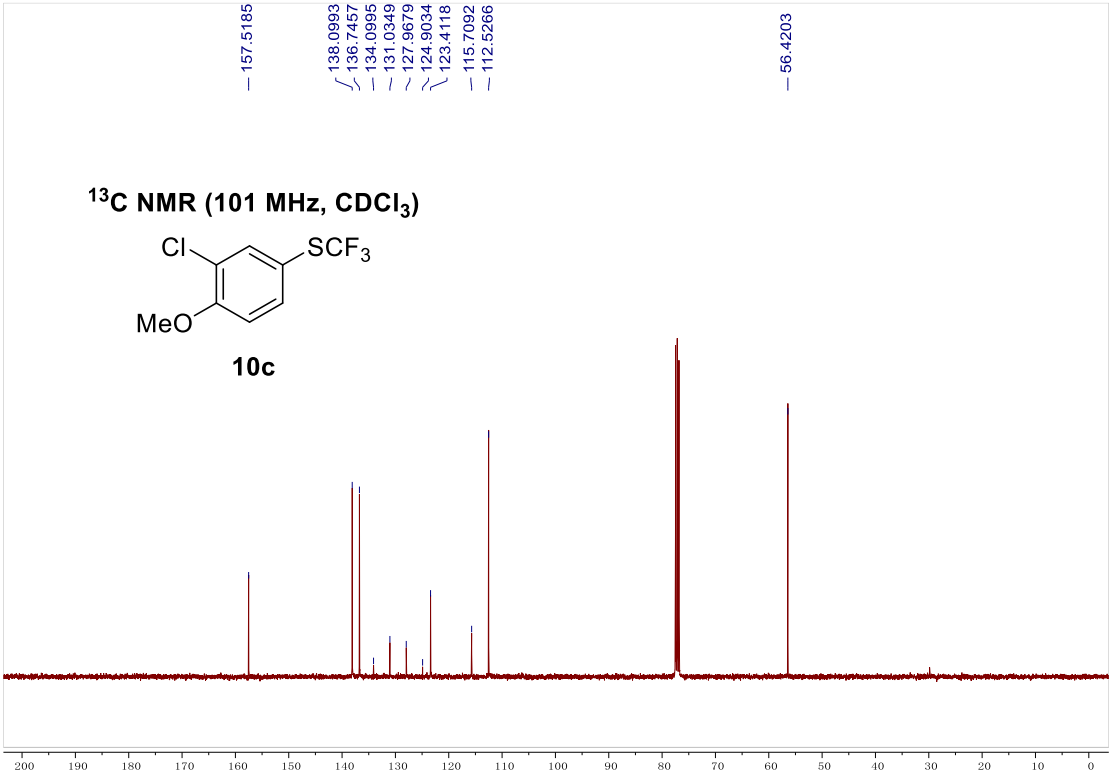
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190


7.6705
7.6652
7.5440
7.5387
7.5226
7.5173
6.9591
6.9377
3.9353
1H NMR (400 MHz, CDCl3)
Cl
SCF3
MeO
10c
1.00
1.02
1.04
3.20
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0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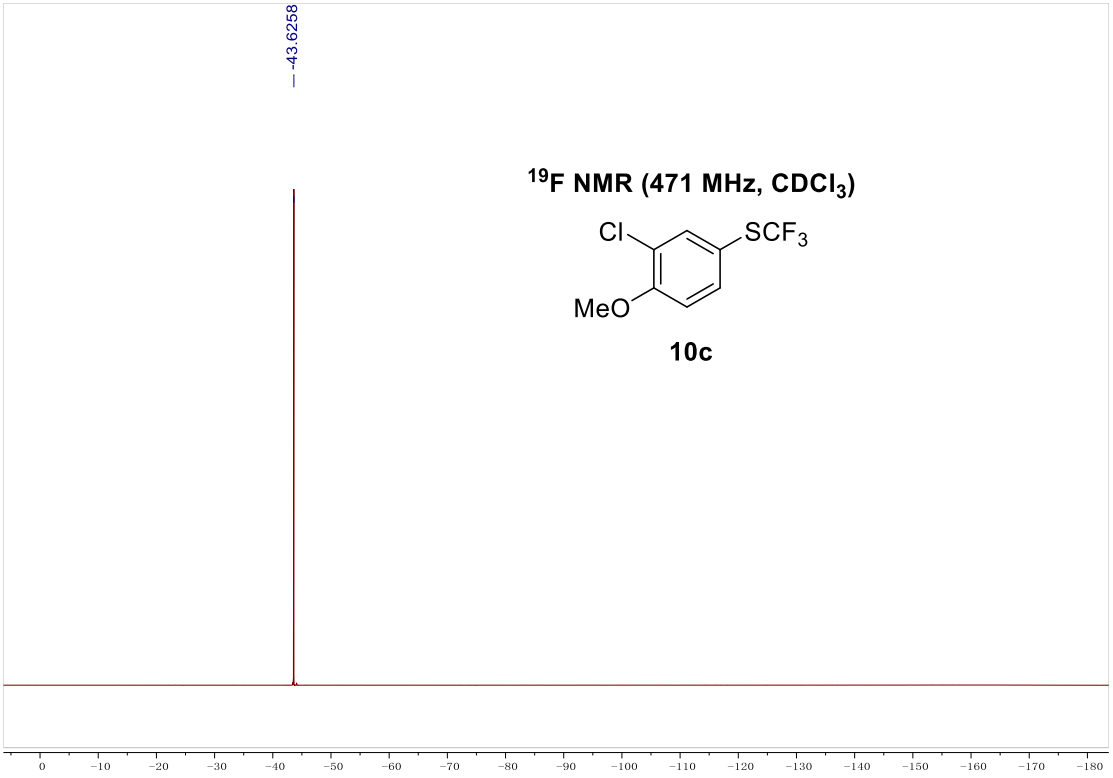
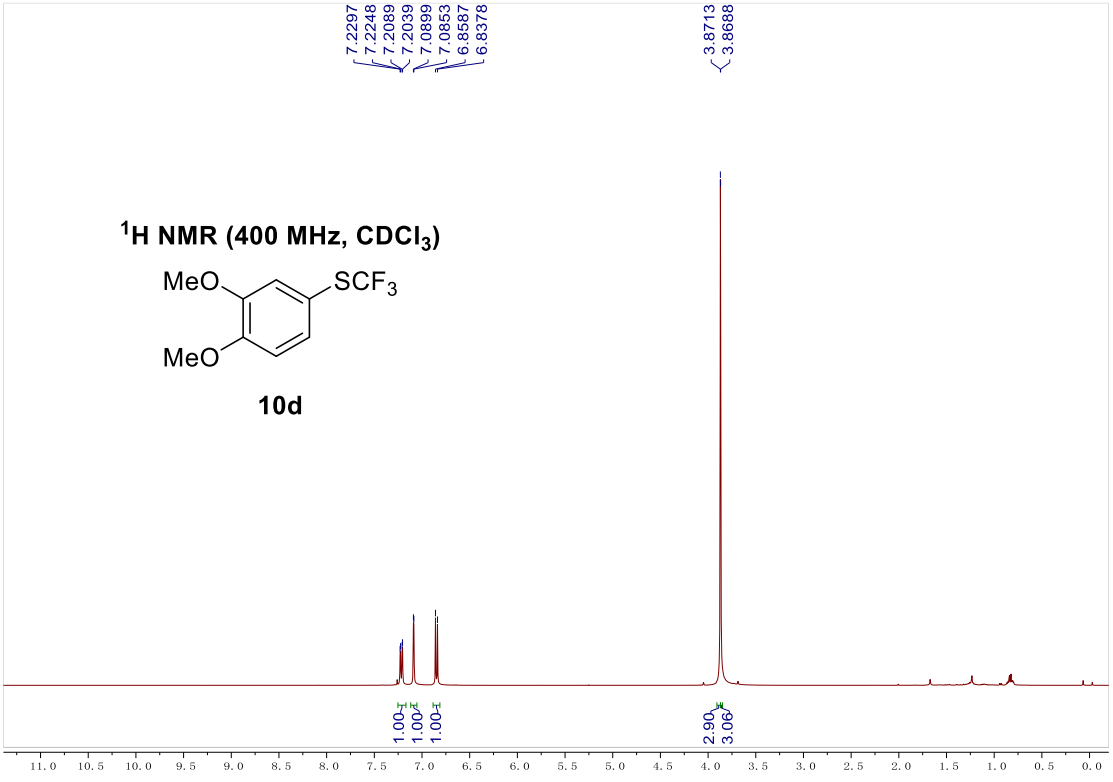
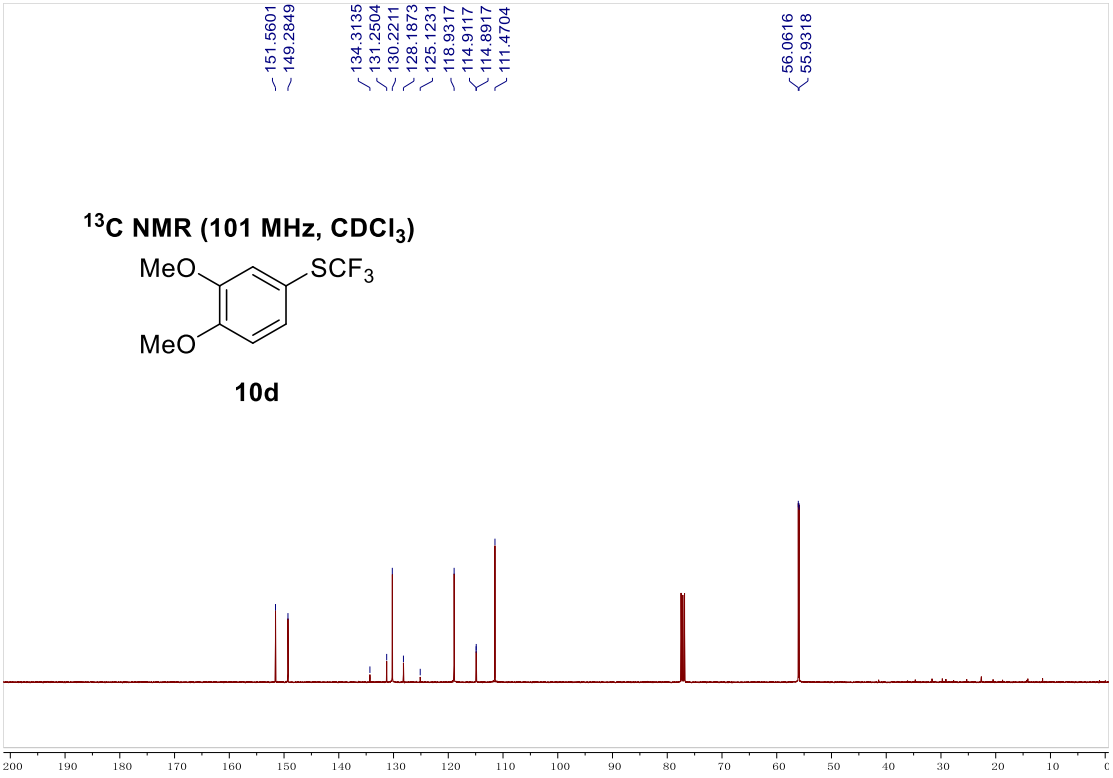
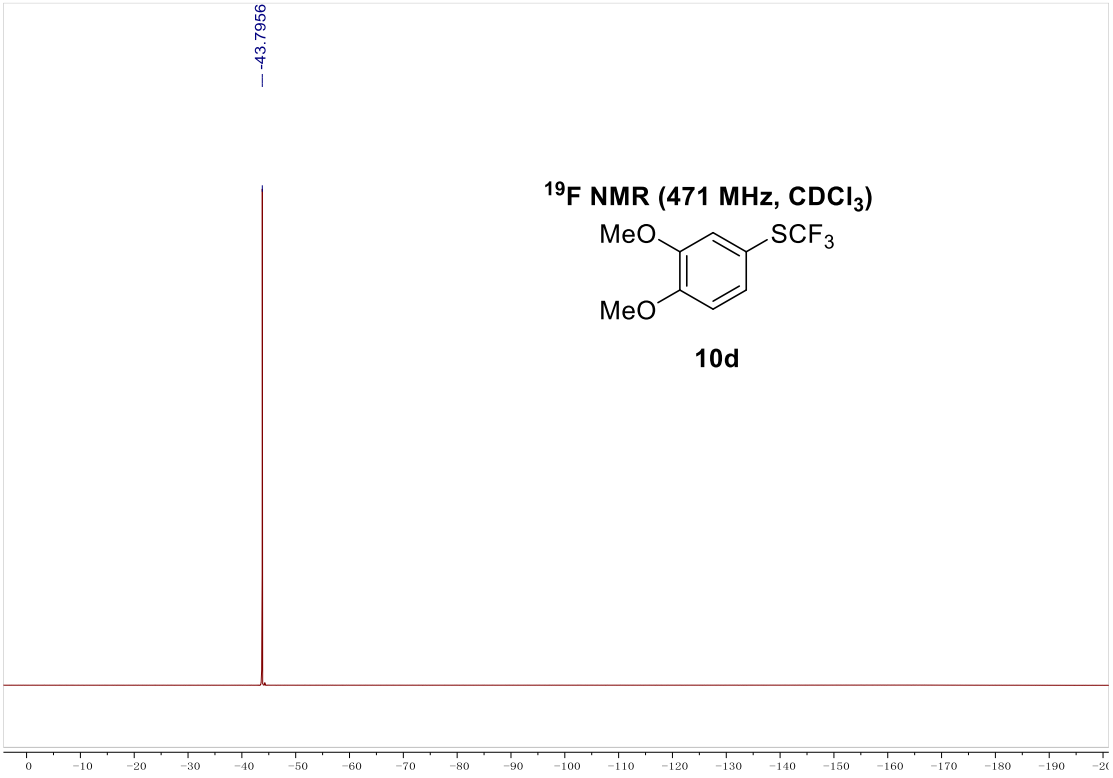
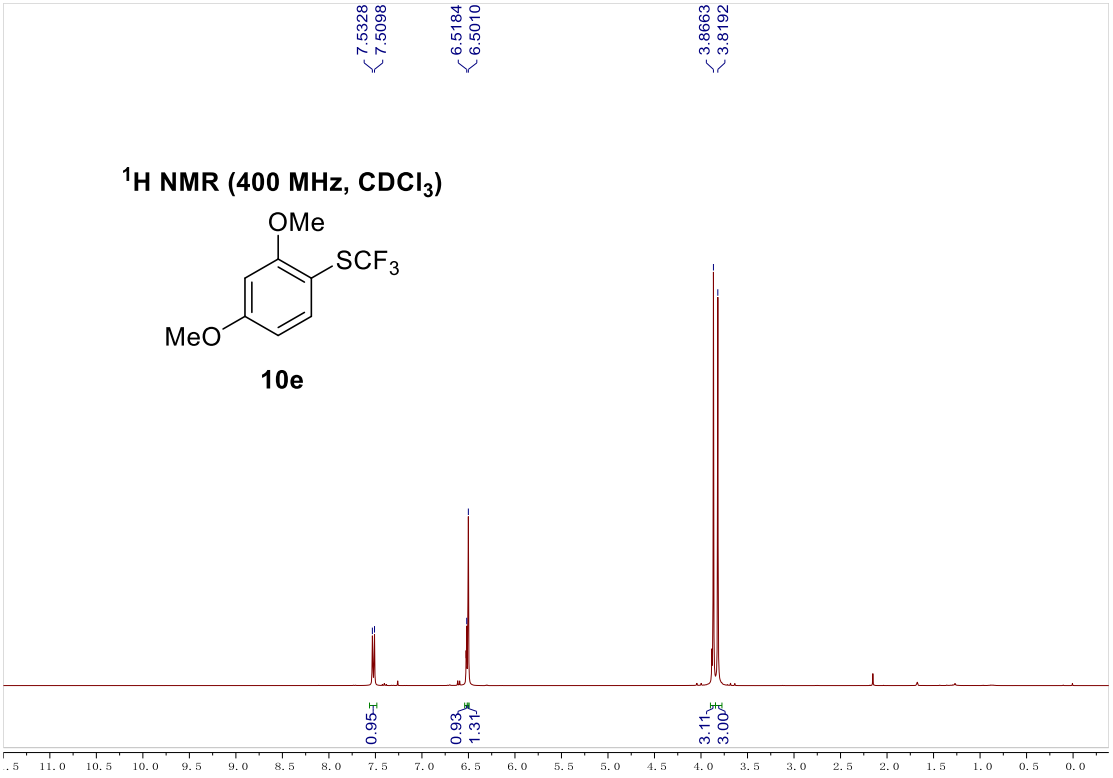
138.0993
136.7457
134.0995
131.0349
127.9679
124.9034
123.4118
115.7092
112.5266
56.4203
13C NMR (101 MHz, CDCl3)
Cl
SCF3
MeO
10c
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0


-43.6258
^{19}F NMR (471 MHz, CDCl$_3$)
Cl
SCF$_3$
MeO
10c
0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180


7.2297
7.2248
7.2089
7.2039
7.0899
7.0853
6.8587
6.8378
3.8713
3.8688
^{1}H NMR (400 MHz, CDCl$_3$)
MeO
SCF$_3$
MeO
10d
1.00
1.00
1.00
2.90
3.06
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

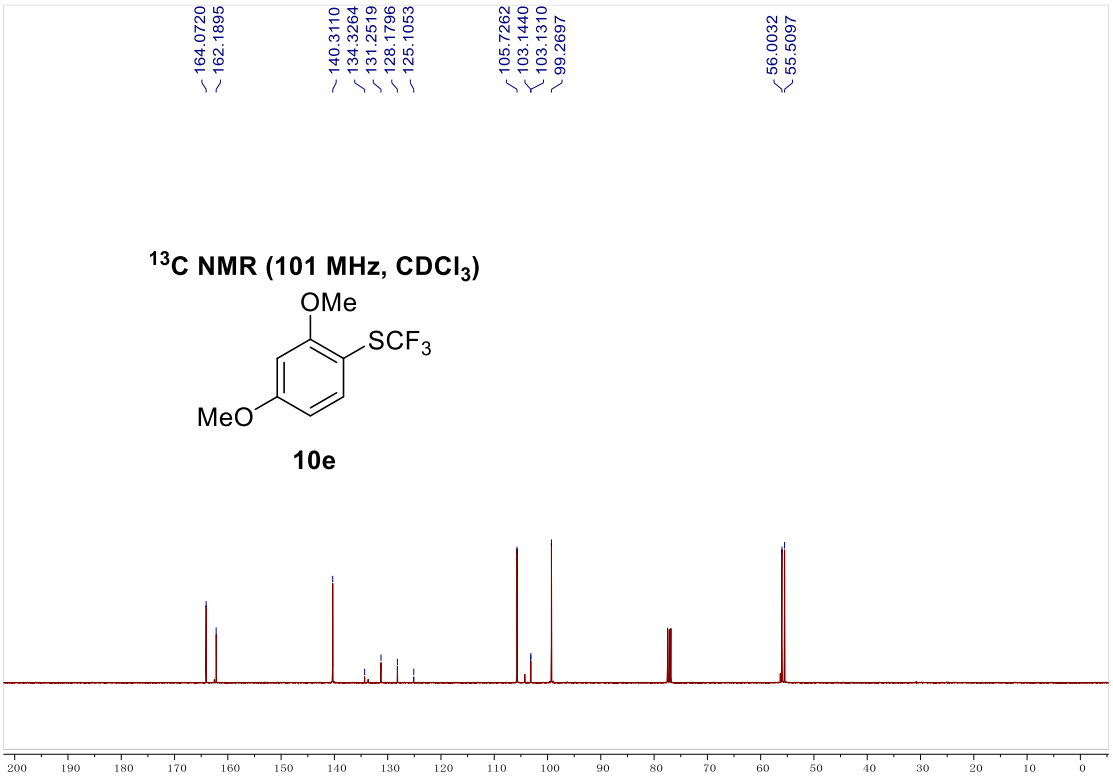

151.5601
149.2849
134.3135
131.2504
130.2211
128.1873
125.1231
118.9317
114.9117
114.8917
111.4704
56.0616
55.9318
13C NMR (101 MHz, CDCl3)
MeO
SCF3
MeO
10d


-43.7956
19F NMR (471 MHz, CDCl3)
MeO
SCF3
MeO
10d



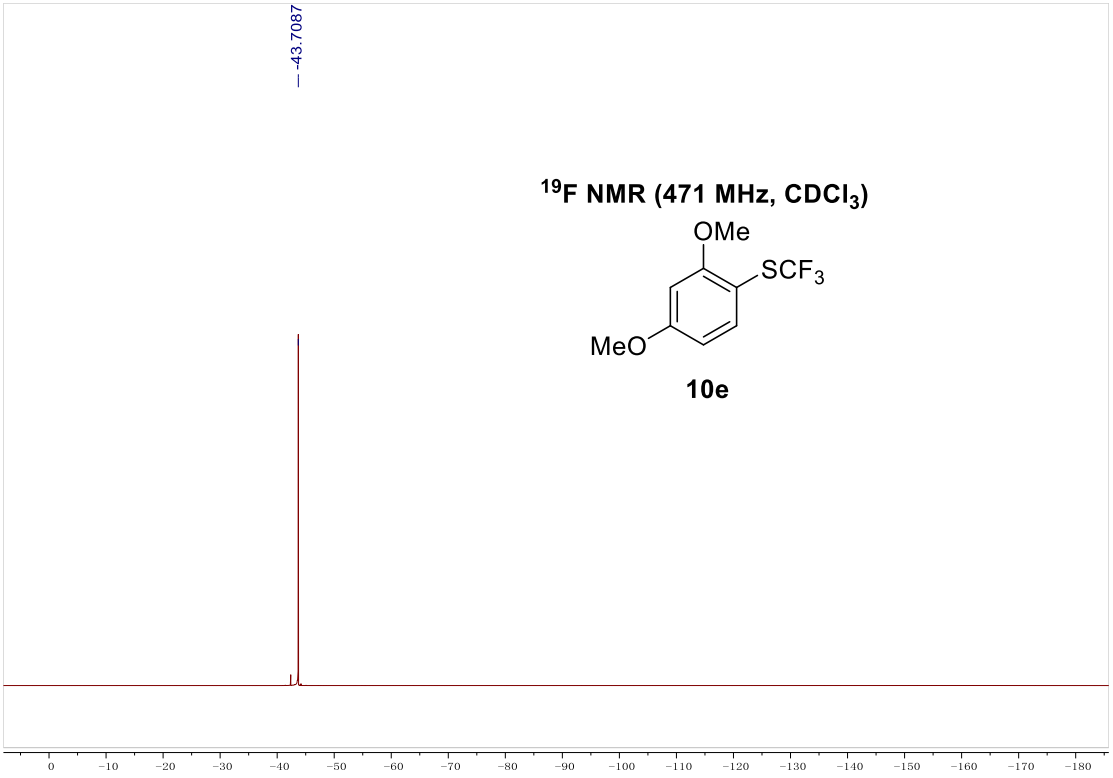
^{1}H NMR (400 MHz, $CDCl_3$)

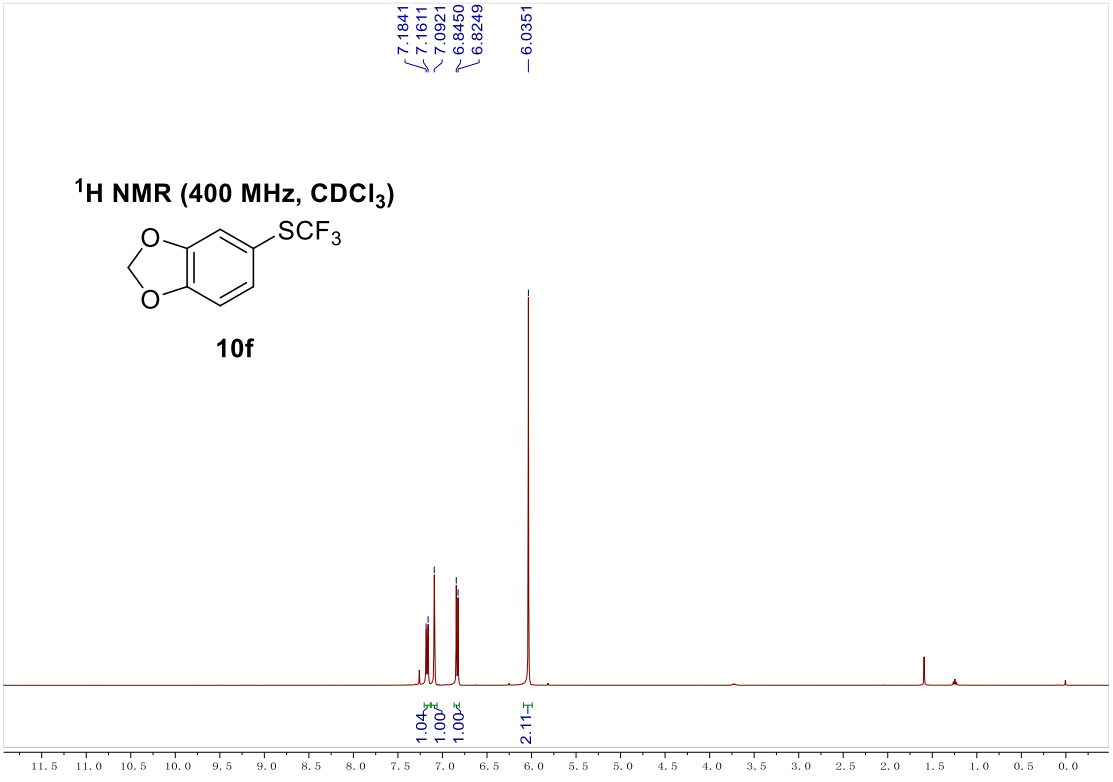
10e

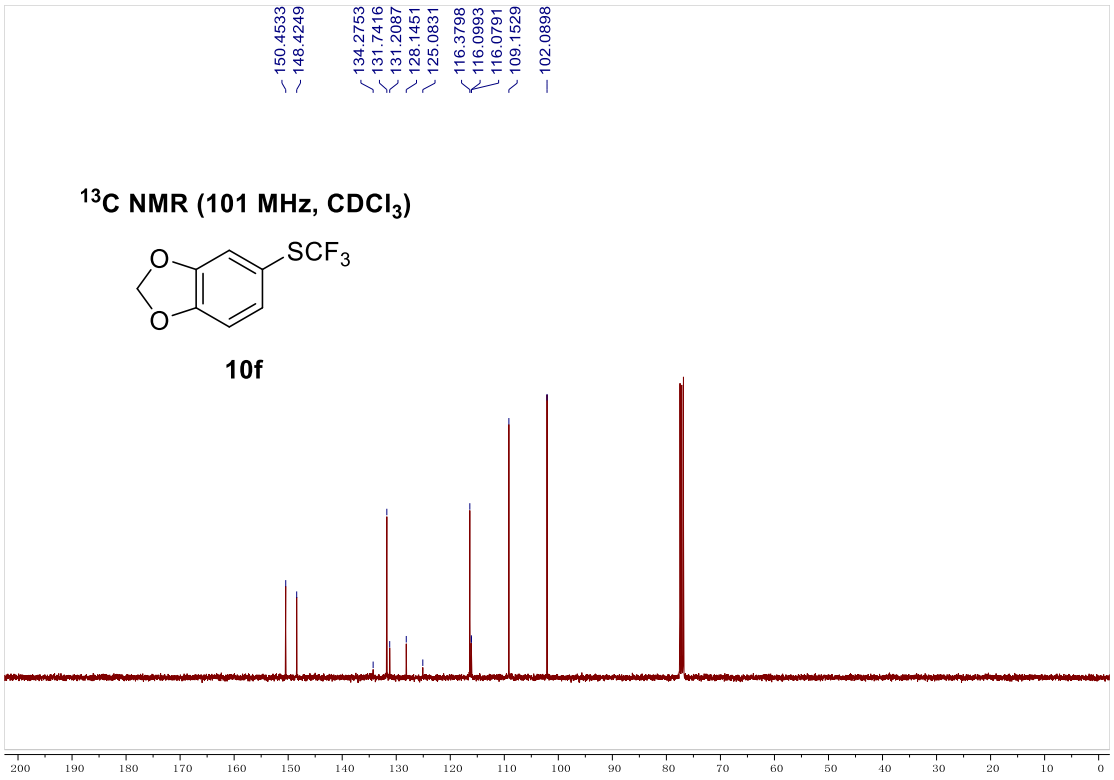


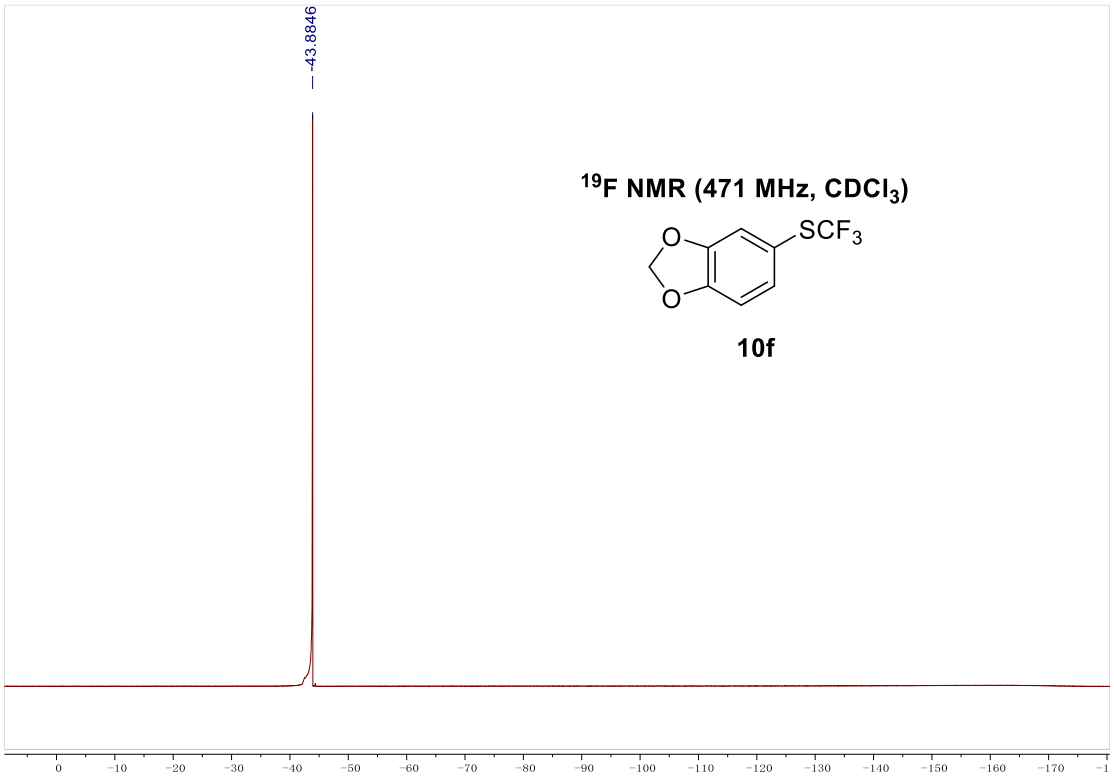
^{13}C NMR (101 MHz, $CDCl_3$)

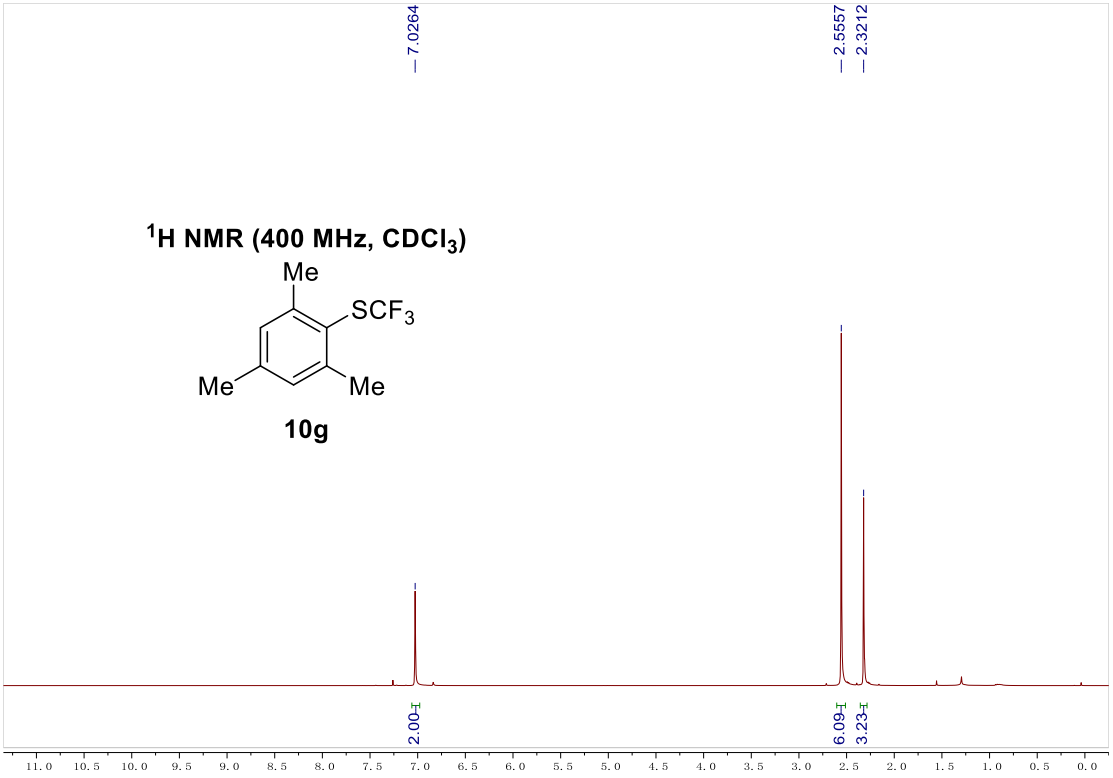
10e

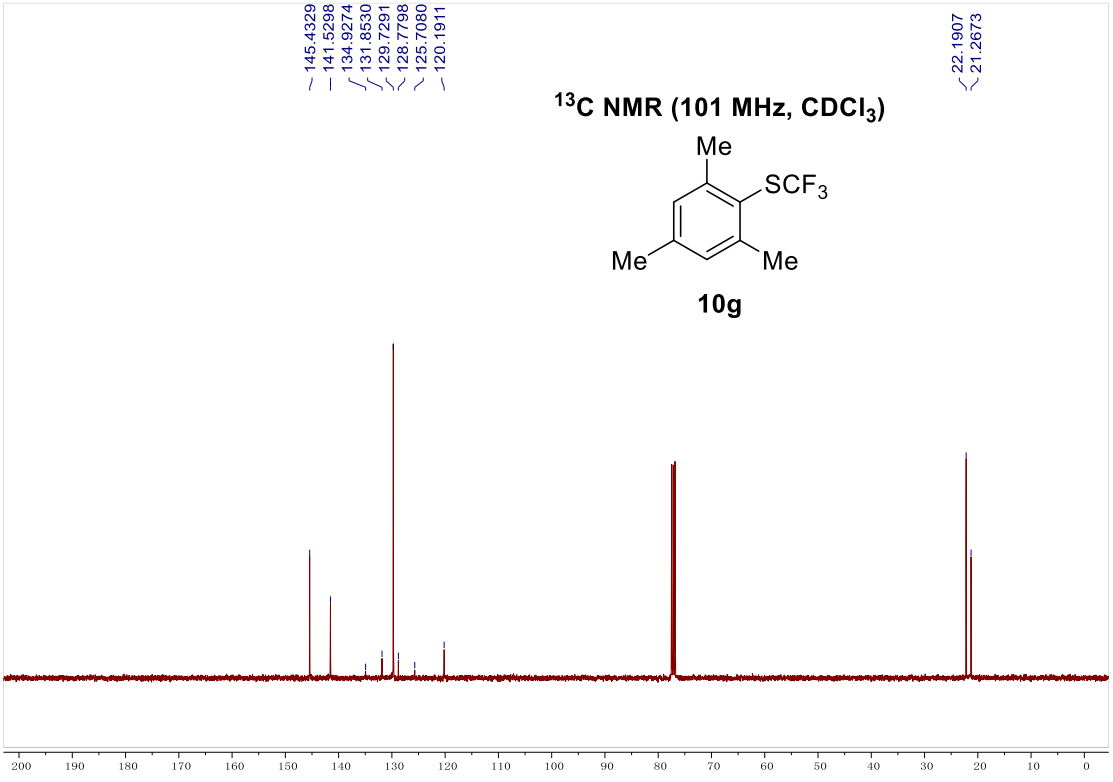

-43.7087
19F NMR (471 MHz, CDCl3)
OMe
SCF3
MeO
10e

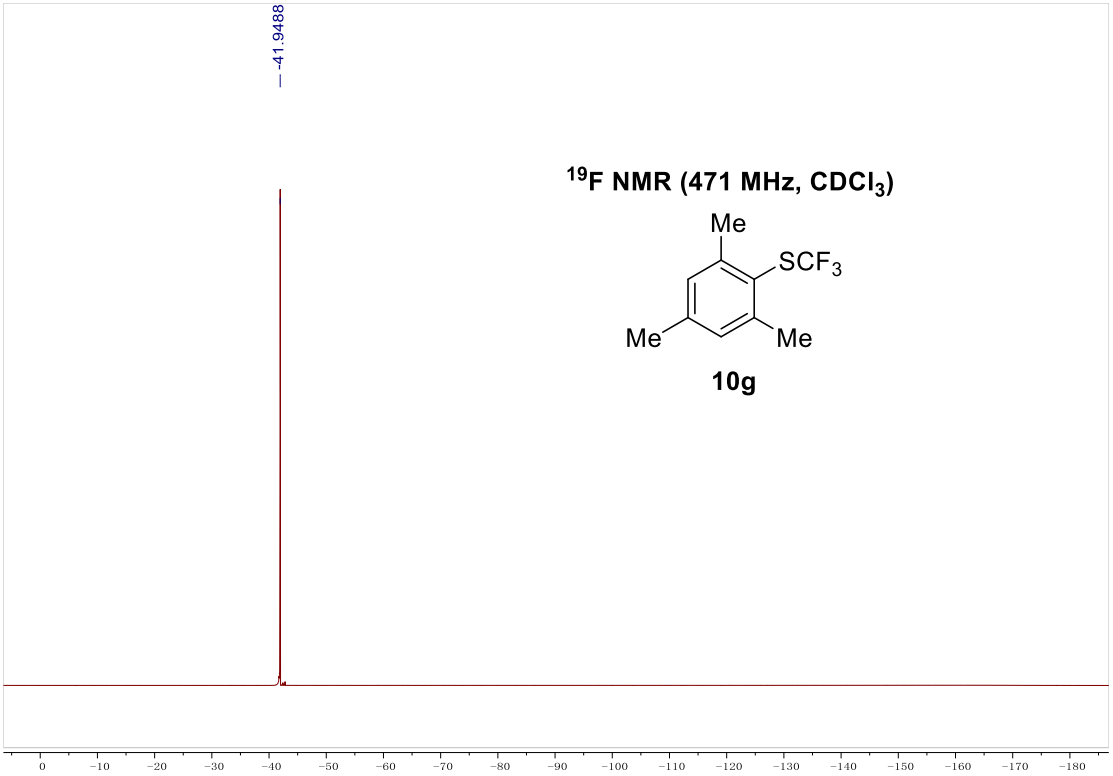

7.1841
7.1611
7.0921
6.8450
6.8249
6.0351
1H NMR (400 MHz, CDCl3)
SCF3
10f
1.04
1.00
1.00
2.11

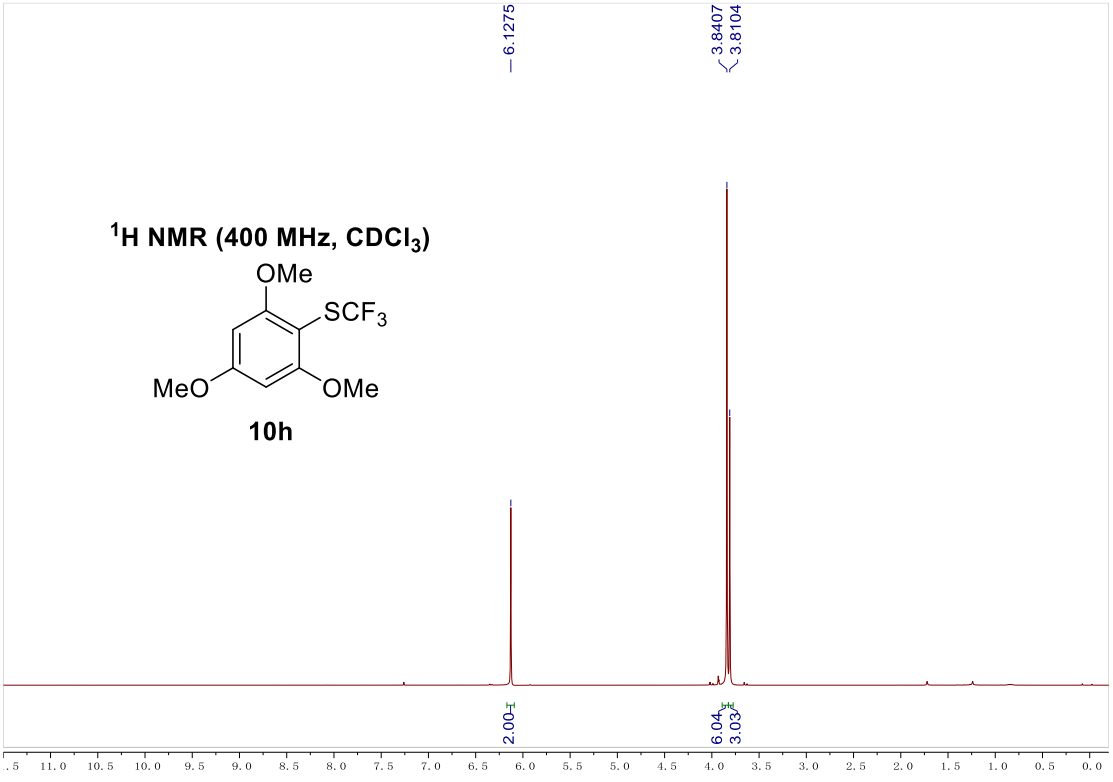

150.4533
148.4249
134.2753
131.7416
131.2087
128.1451
125.0831
116.3798
116.0993
116.0791
109.1529
102.0898
13C NMR (101 MHz, CDCl3)
SCF3
O
O
10f
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

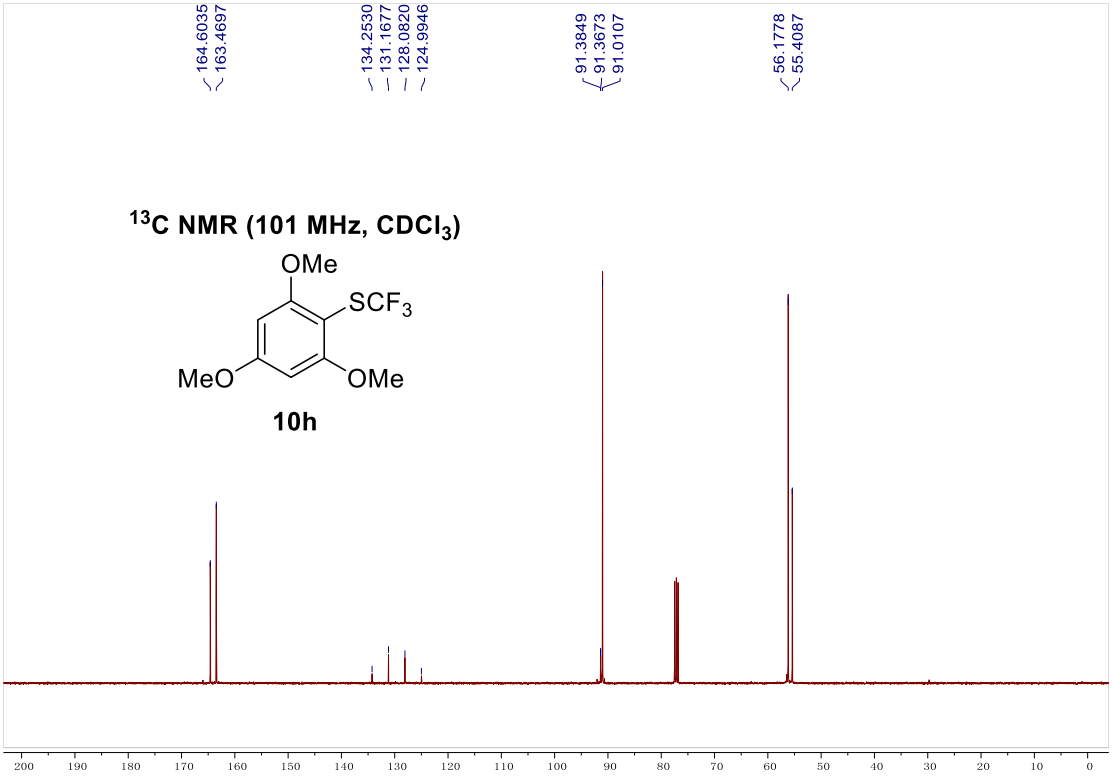

-43.8846
19F NMR (471 MHz, CDCl3)
SCF3
O
O
10f
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170

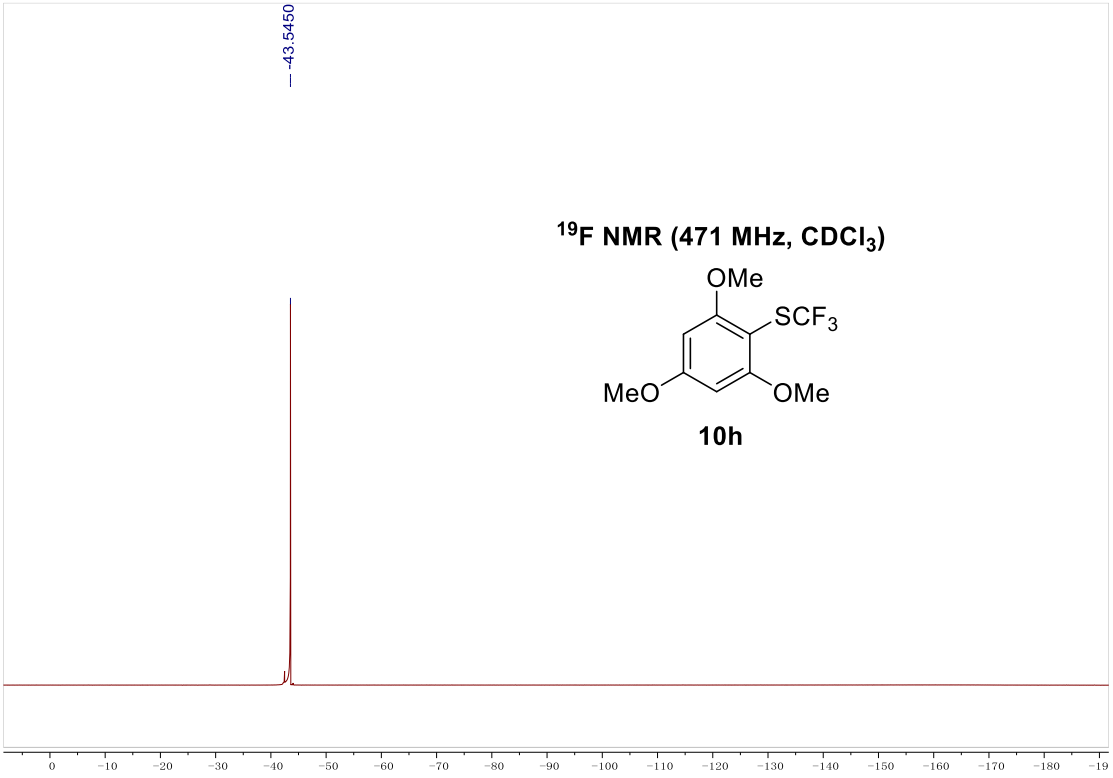

7.0264
2.5557
2.3212
1H NMR (400 MHz, CDCl3)
Me
SCF3
Me
Me
10g
2.00
6.09
3.23
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

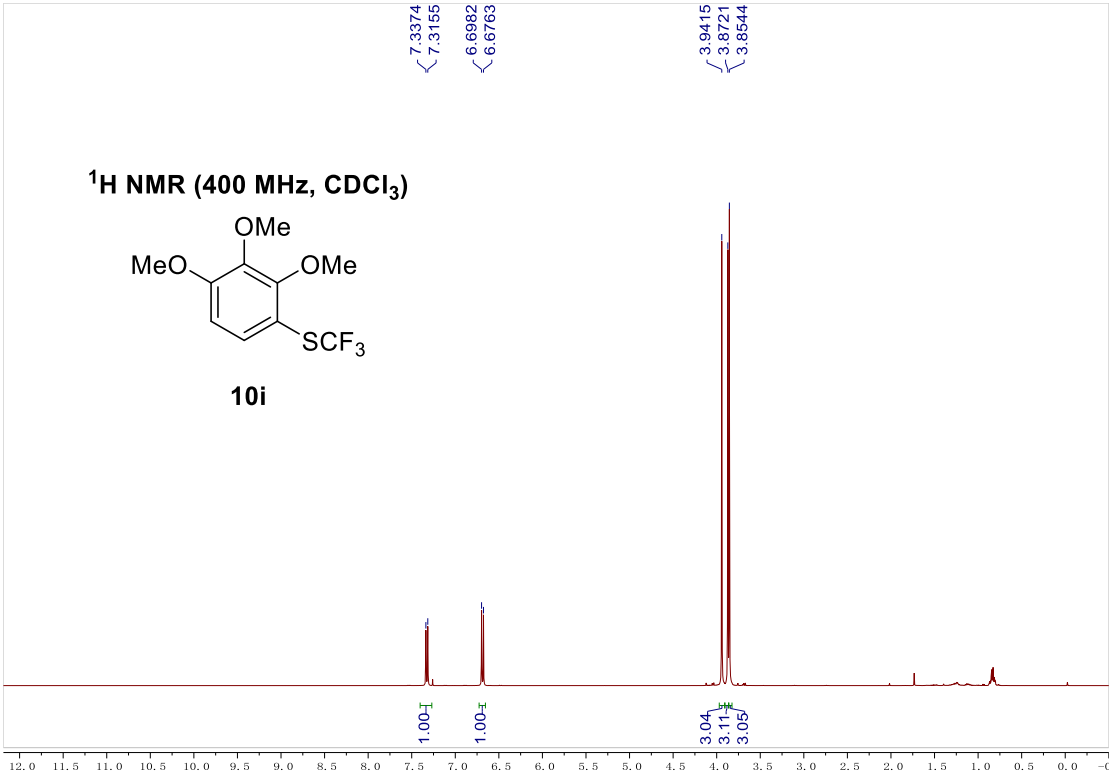

145.4329
141.5298
134.9274
131.8530
129.7291
128.7798
125.7080
120.1911
22.1907
21.2673
13C NMR (101 MHz, CDCl3)
Me
SCF3
Me
Me
10g
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0


-41.9488
19F NMR (471 MHz, CDCl3)
Me
SCF3
Me
Me
10g


6.1275
3.8407
3.8104
1H NMR (400 MHz, CDCl3)
OMe
SCF3
MeO
OMe
10h
2.00
6.04
3.03

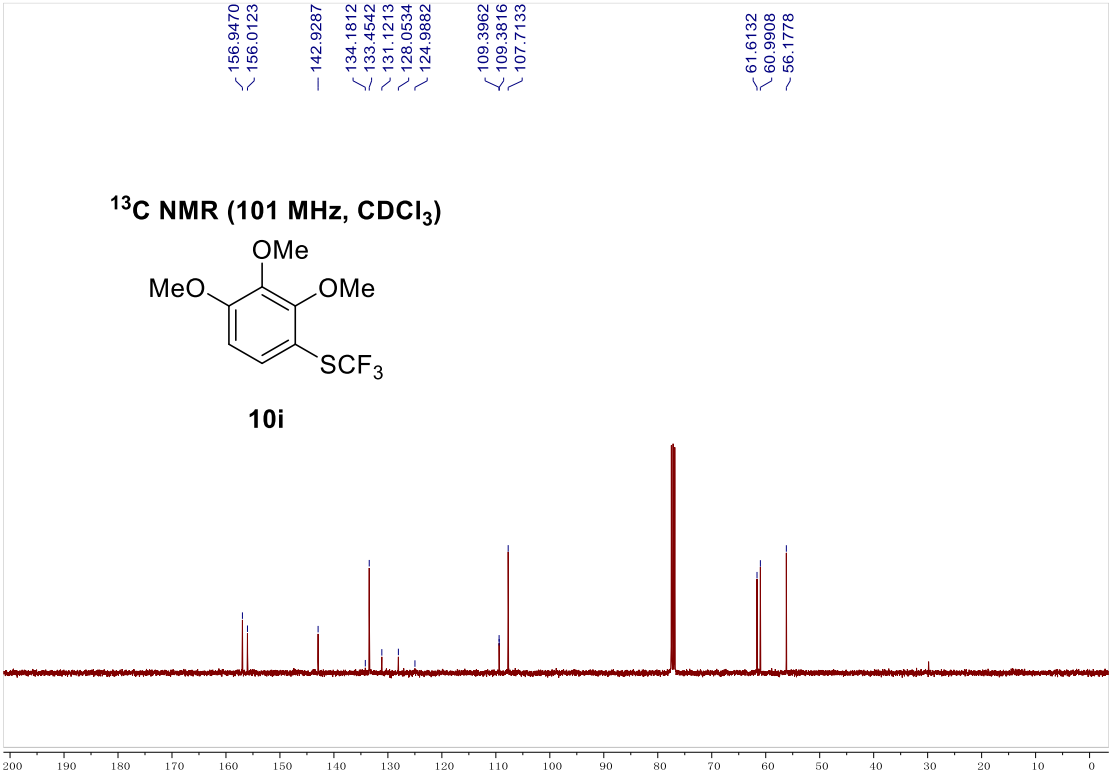

164.6035
163.4697
134.2530
131.1677
128.0820
124.9946
91.3849
91.3673
91.0107
56.1778
55.4087
13C NMR (101 MHz, CDCl3)
OMe
SCF3
MeO
OMe
10h
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


-43.5450
19F NMR (471 MHz, CDCl3)
OMe
SCF3
MeO
OMe
10h
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-19



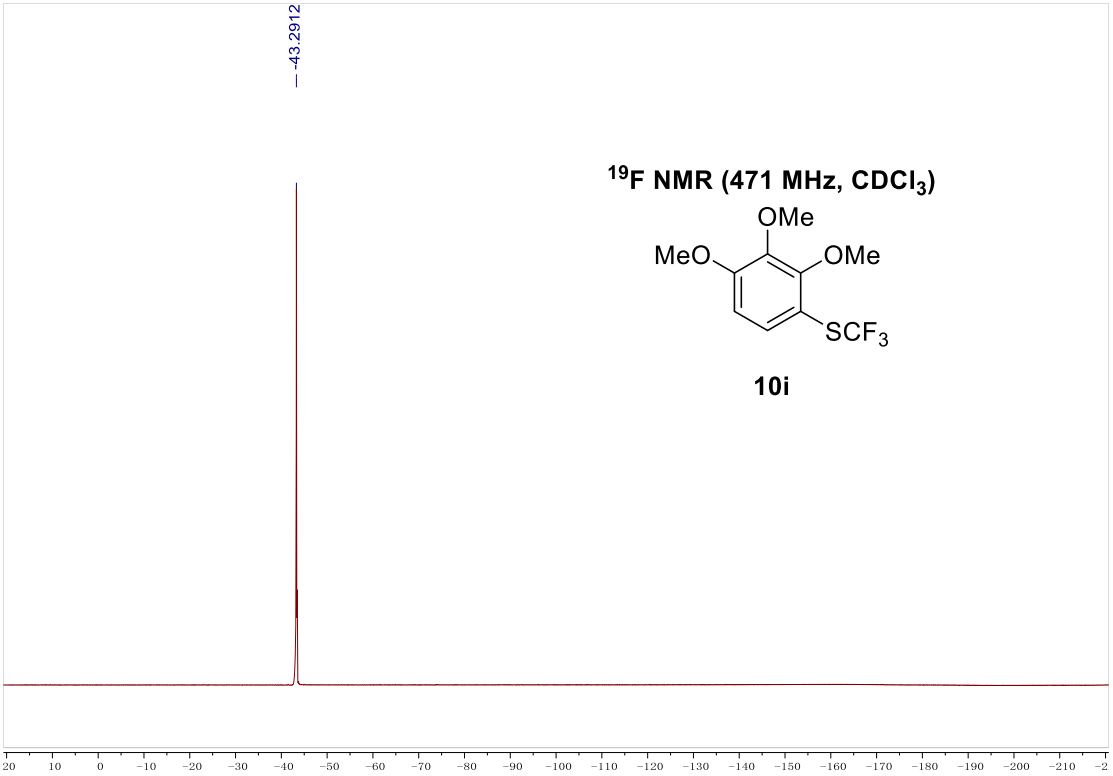
^{1}H NMR (400 MHz, $CDCl_3$)

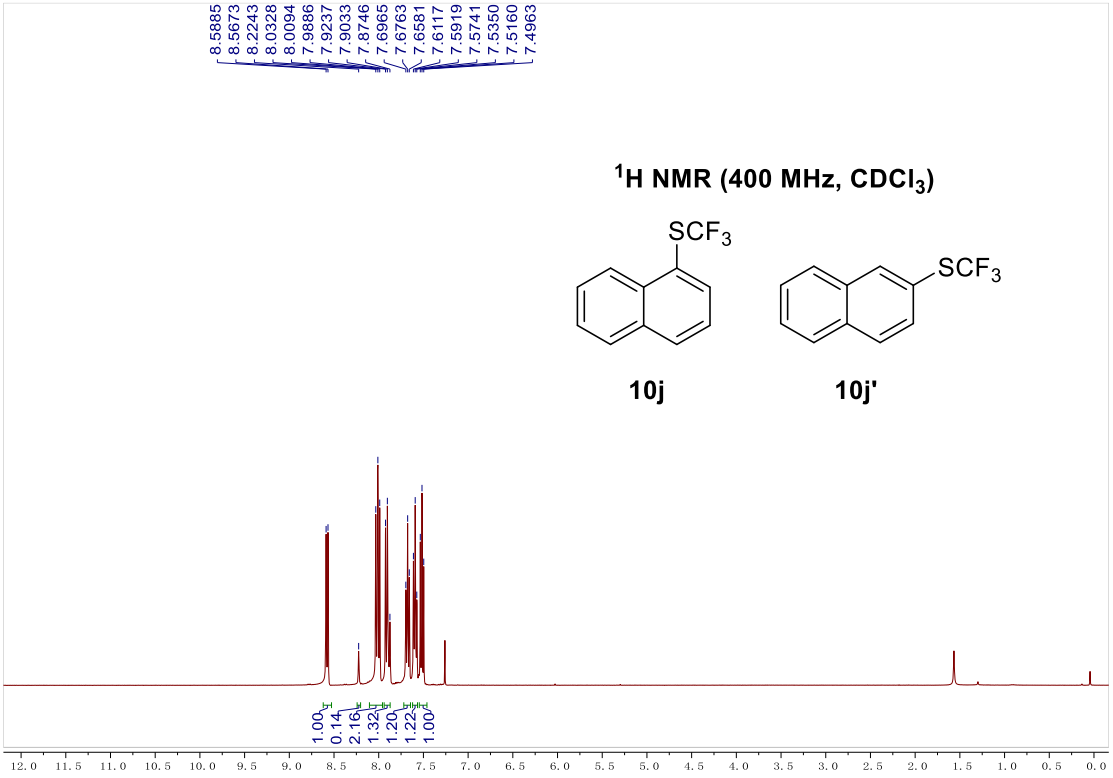
10i

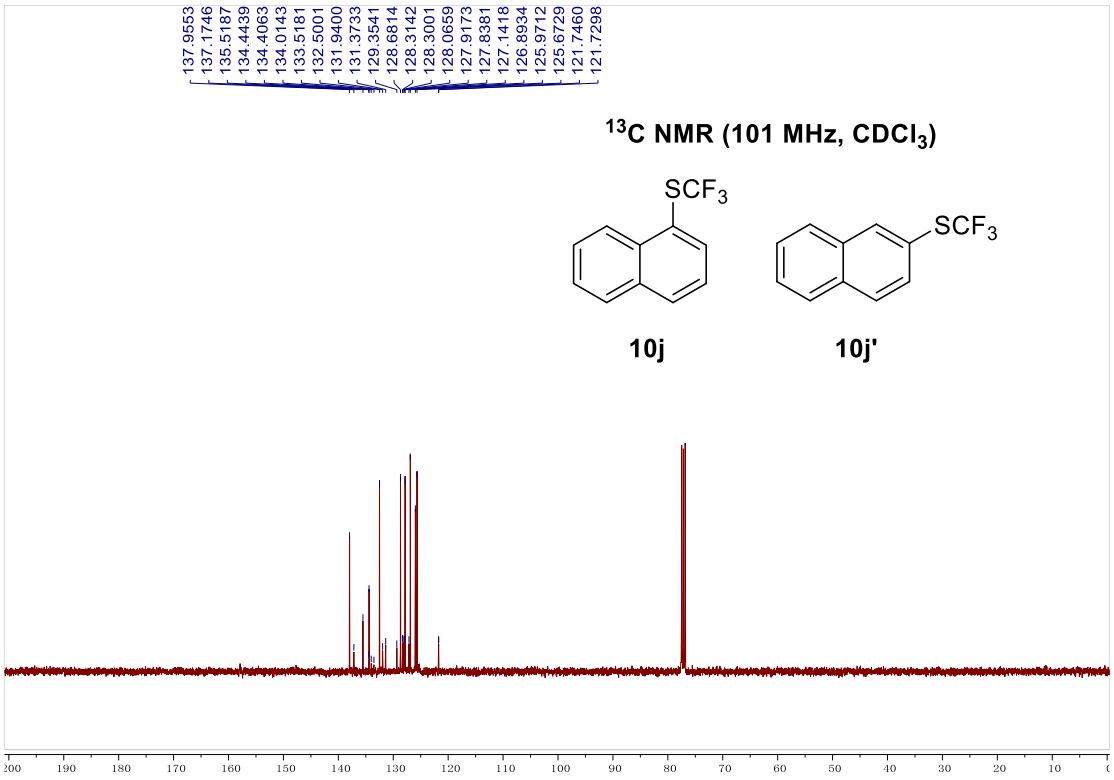


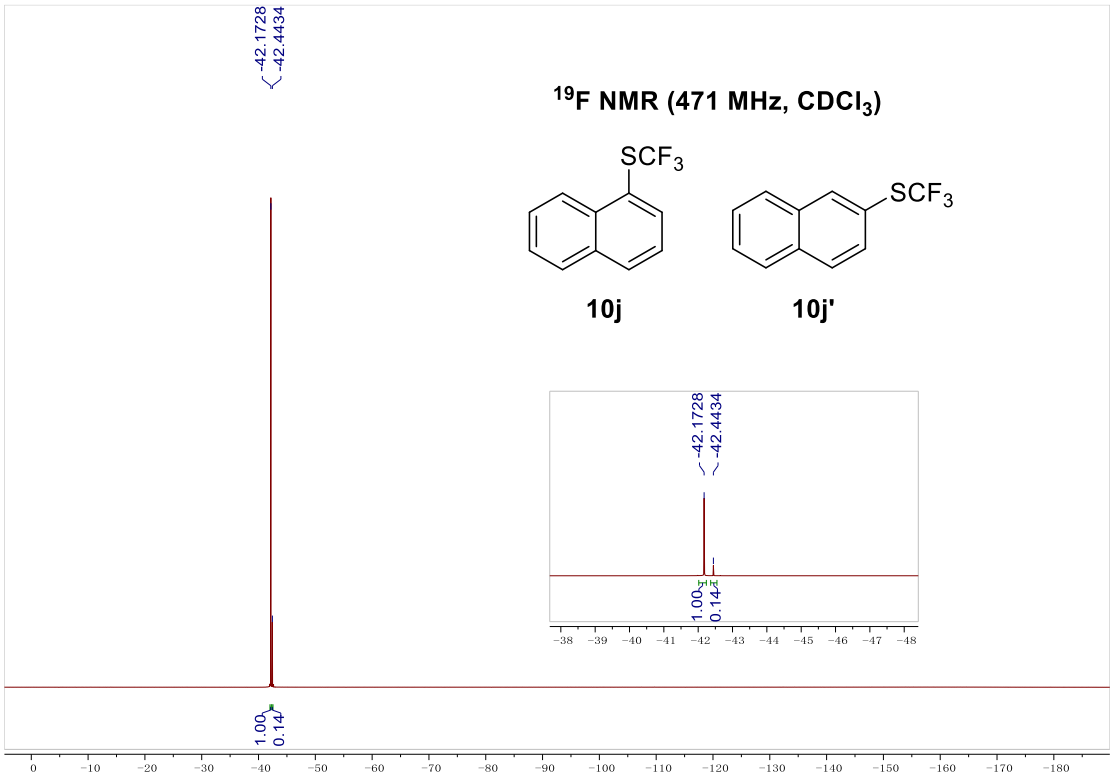
^{13}C NMR (101 MHz, $CDCl_3$)

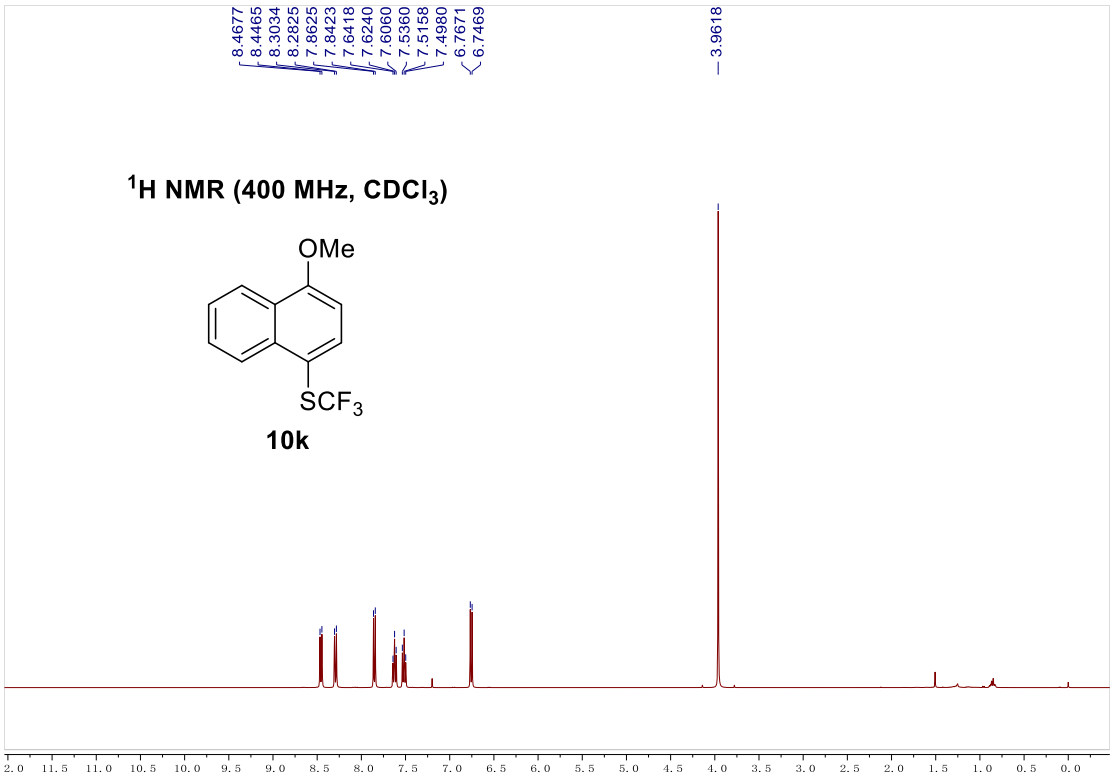
10i

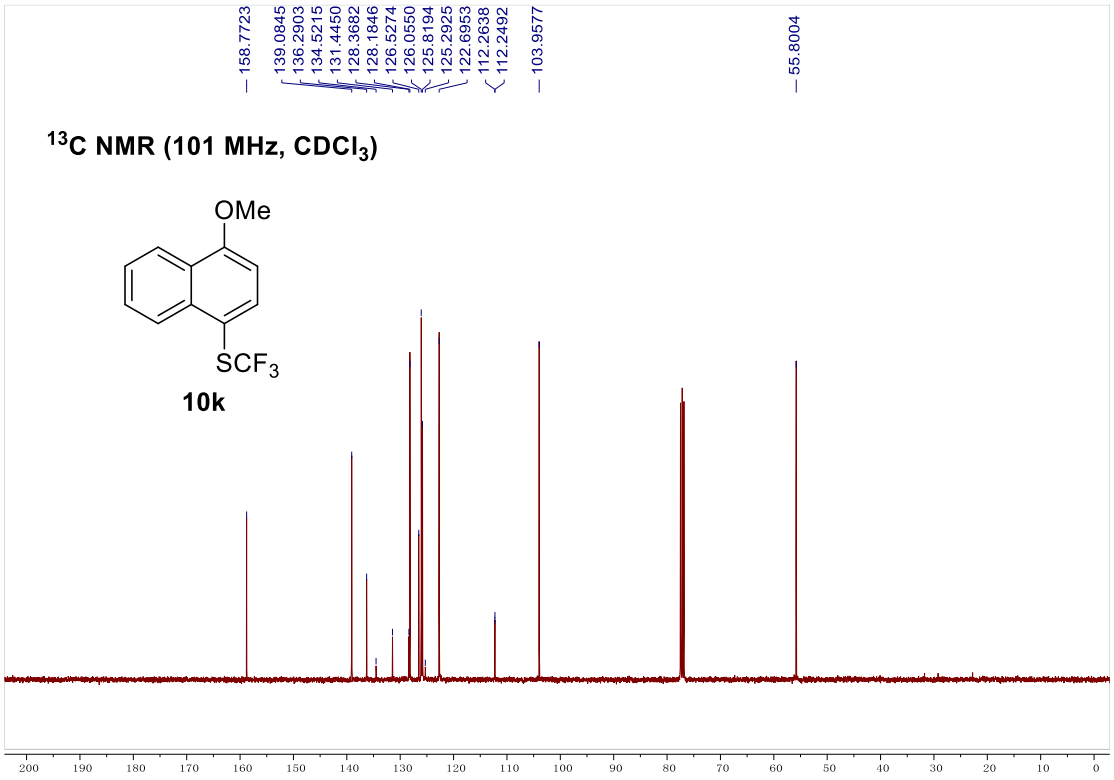

-43.2912
19F NMR (471 MHz, CDCl3)
OMe
MeO
OMe
SCF3
10i

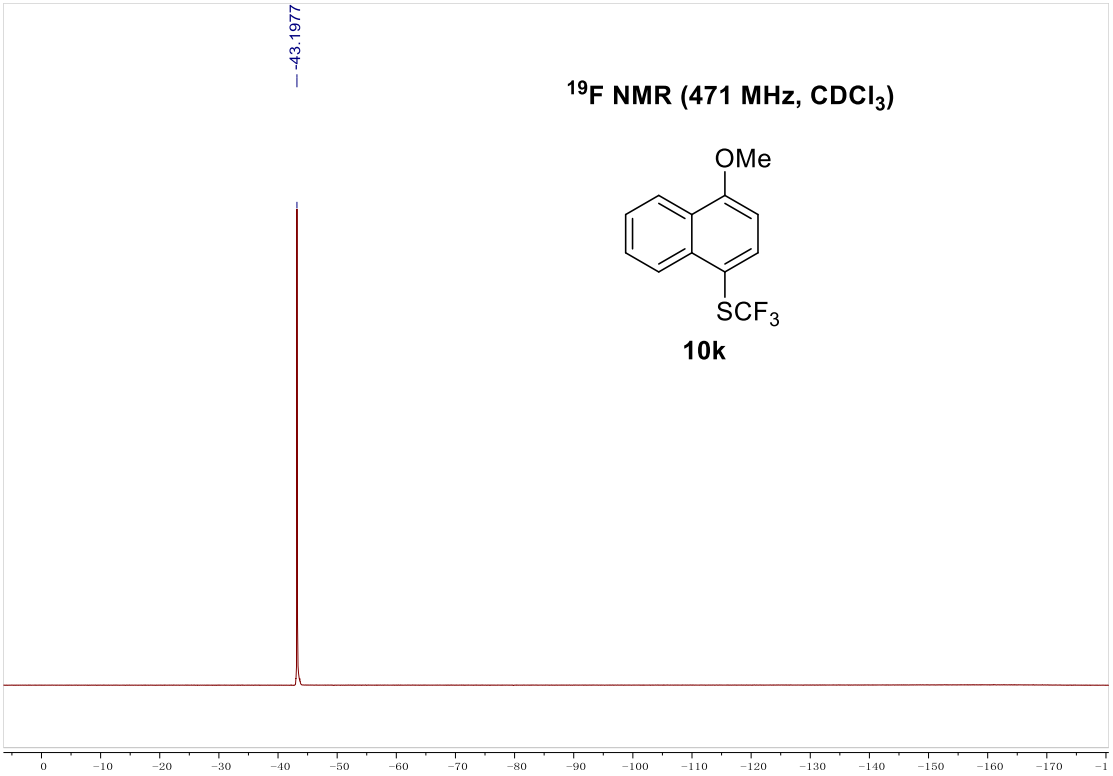

8.5885
8.5673
8.2243
8.0328
8.0094
7.9886
7.9237
7.9033
7.8746
7.6965
7.6763
7.6581
7.6117
7.5919
7.5741
7.5350
7.5160
7.4963
1H NMR (400 MHz, CDCl3)
SCF3
SCF3
10j
10j'
1.00
0.14
2.16
1.32
1.20
1.22
1.00

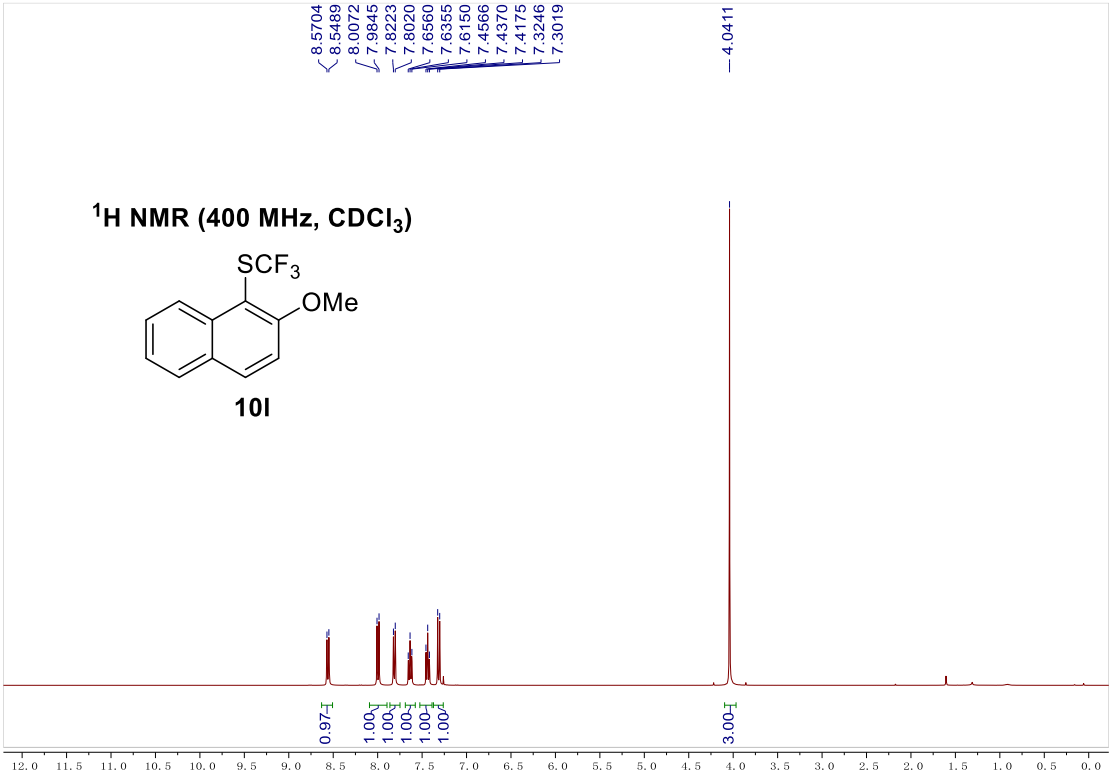

137.9553
137.1746
135.5187
134.4439
134.4063
134.0143
133.5181
132.5001
131.9400
131.3733
129.3541
128.6814
128.3142
128.3001
128.0659
127.9173
127.8381
127.1418
126.8934
125.9712
125.6729
121.7460
121.7298
13C NMR (101 MHz, CDCl3)
SCF3
SCF3
10j
10j'

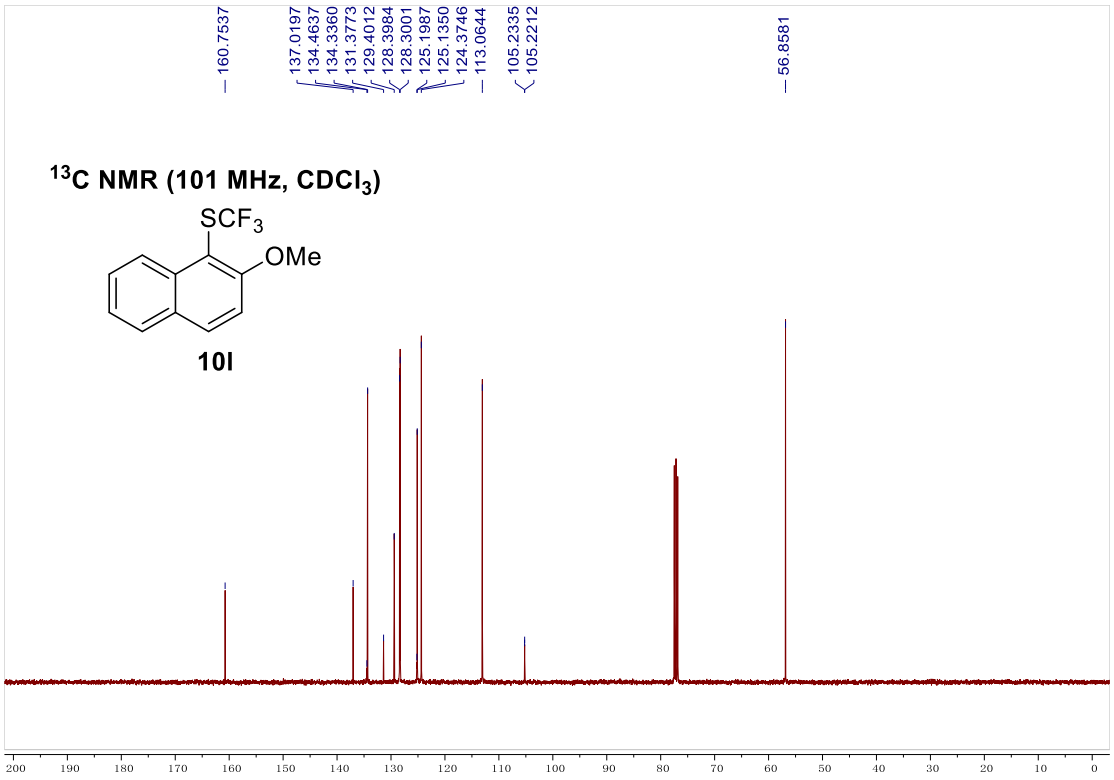

-42.1728
-42.4434
19F NMR (471 MHz, CDCl3)
SCF3
SCF3
10j
10j'
1.00
0.14

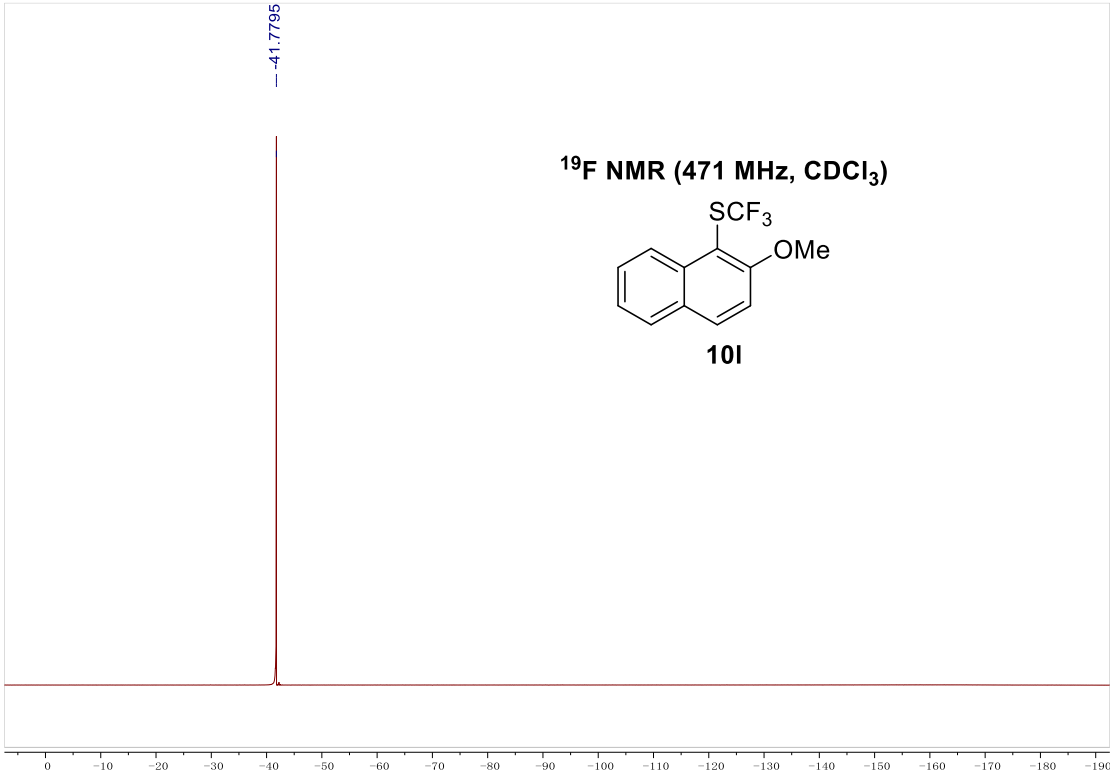

8.4677
8.4465
8.3034
8.2825
7.8625
7.8423
7.6418
7.6240
7.6060
7.5360
7.5158
7.4980
6.7671
6.7469
3.9618
1H NMR (400 MHz, CDCl3)
OMe
SCF3
10k

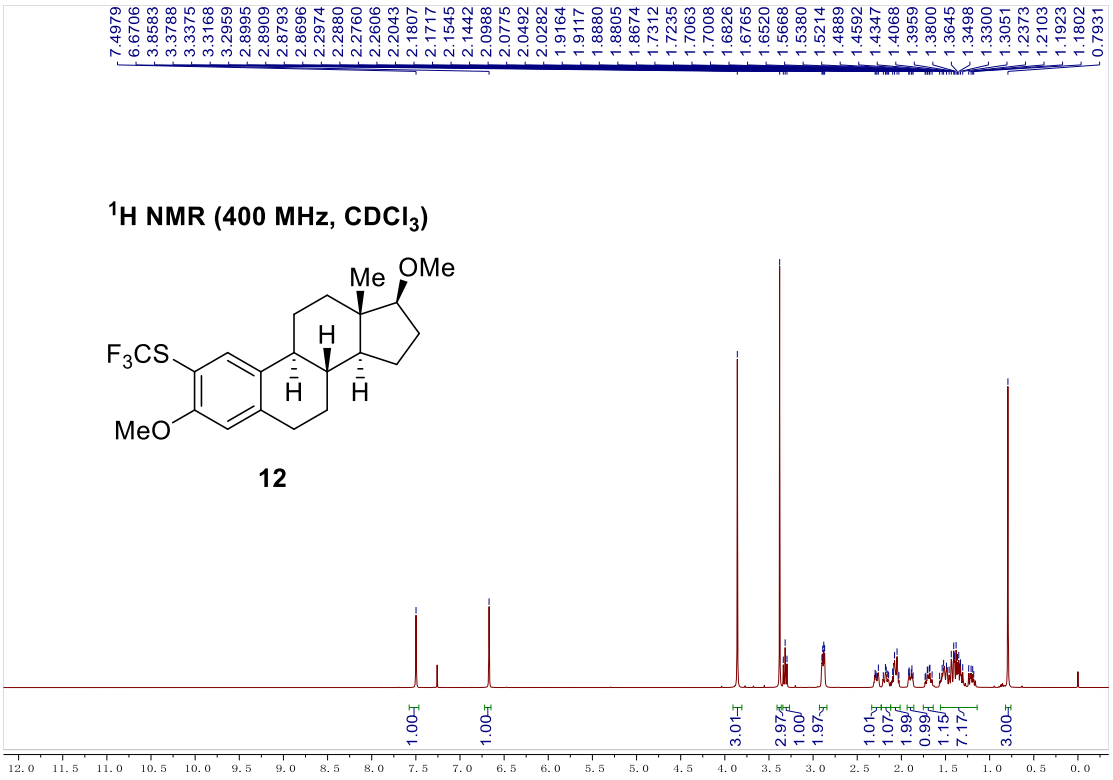

158.7723
139.0845
136.2903
134.5215
131.4450
128.3682
128.1846
126.5274
126.0550
125.8194
125.2925
122.6953
112.2638
112.2492
103.9577
55.8004
13C NMR (101 MHz, CDCl3)
OMe
SCF3
10k

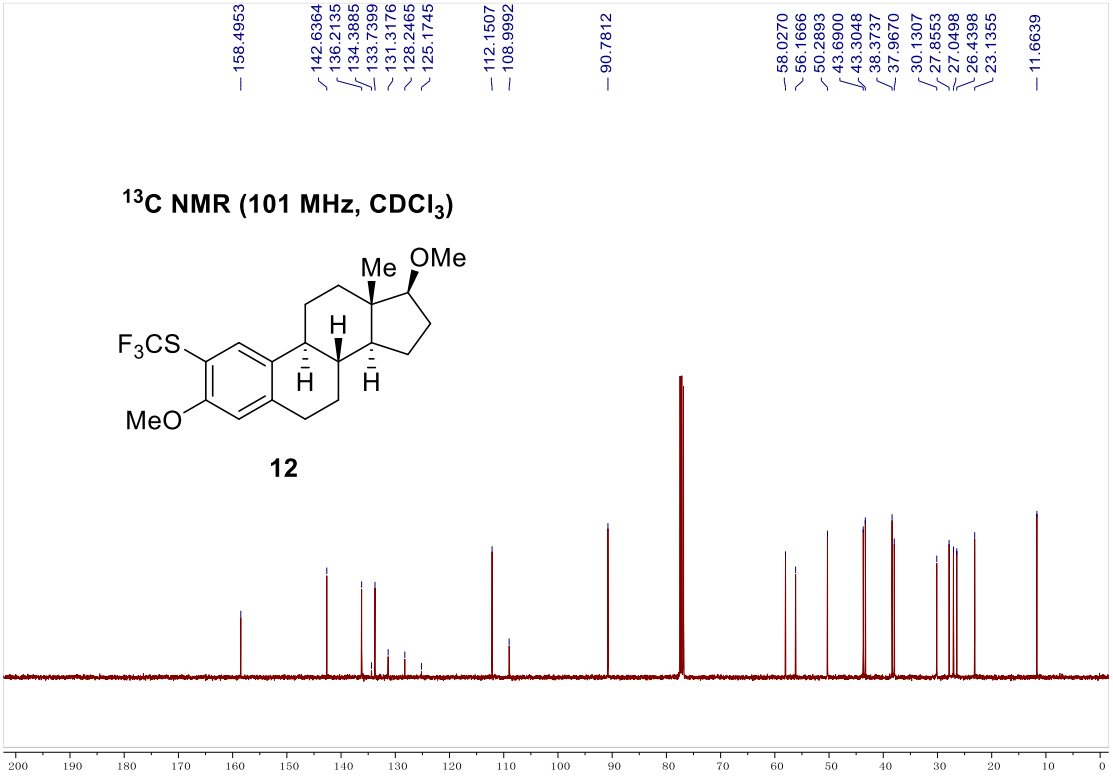

-43.1977
19F NMR (471 MHz, CDCl3)
OMe
SCF3
10k

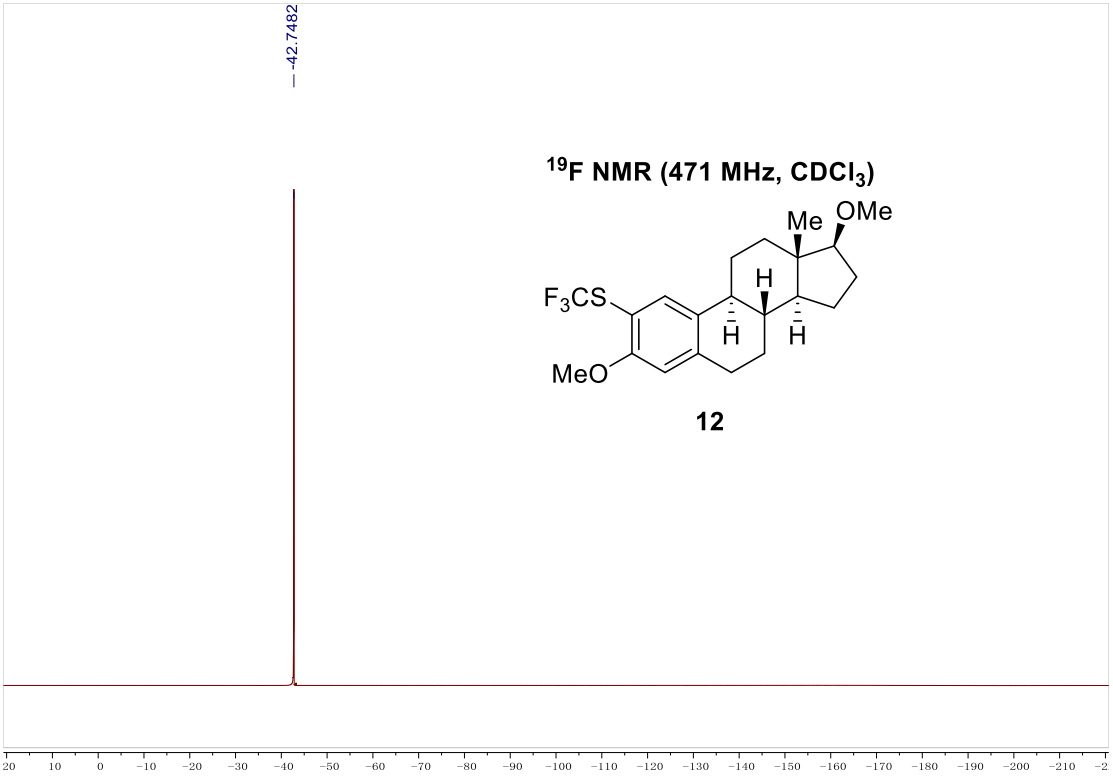

8.5704
8.5489
8.0072
7.9845
7.8223
7.8020
7.6560
7.6355
7.6150
7.4566
7.4370
7.4175
7.3246
7.3019
4.0411
1H NMR (400 MHz, CDCl3)
SCF3
OMe
10l
0.97
1.00
1.00
1.00
1.00
1.00
3.00


160.7537
137.0197
134.4637
134.3360
131.3773
129.4012
128.3984
128.3001
125.1987
125.1350
124.3746
113.0644
105.2335
105.2212
56.8581
13C NMR (101 MHz, CDCl3)
SCF3
OMe
10l
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0


-41.7795
19F NMR (471 MHz, CDCl3)
SCF3
OMe
10l
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190


7.4979
6.6706
3.8583
3.3788
3.3375
3.3168
3.2959
2.8995
2.8909
2.8793
2.8696
2.2974
2.2880
2.2760
2.2606
2.2043
2.1807
2.1717
2.1545
2.1442
2.0988
2.0775
2.0492
2.0282
1.9164
1.9117
1.8880
1.8805
1.8674
1.7312
1.7235
1.7063
1.7008
1.6826
1.6765
1.6520
1.5668
1.5380
1.5214
1.4889
1.4592
1.4347
1.4068
1.3959
1.3800
1.3645
1.3498
1.3300
1.3051
1.2373
1.2103
1.1923
1.1802
0.7931
1H NMR (400 MHz, CDCl3)
Me
OMe
H
F3CS
H
H
MeO
12
1.00
1.00
3.01
2.97
1.00
1.97
1.01
1.07
1.99
0.99
1.15
7.17
3.00
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0


158.4953
142.6364
136.2135
134.3885
133.7399
131.3176
128.2465
125.1745
112.1507
108.9992
90.7812
58.0270
56.1666
50.2893
43.6900
43.3048
38.3737
37.9670
30.1307
27.8553
27.0498
26.4398
23.1355
11.6639
13C NMR (101 MHz, CDCl3)
Me
OMe
H
F3CS
H
H
MeO
12
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0


-42.7482
19F NMR (471 MHz, CDCl3)
Me
OMe
H
F3CS
H
H
MeO
12
20 10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180 −190 −200 −210