Supporting Information

Synthesis of Sulfur- and Oxygen-Bridged Cationic [4]-Helicenes Mediated by Friedel–Crafts-S_NAr Tandem Reactions for Red-light-driven Organophotoredox Catalysis

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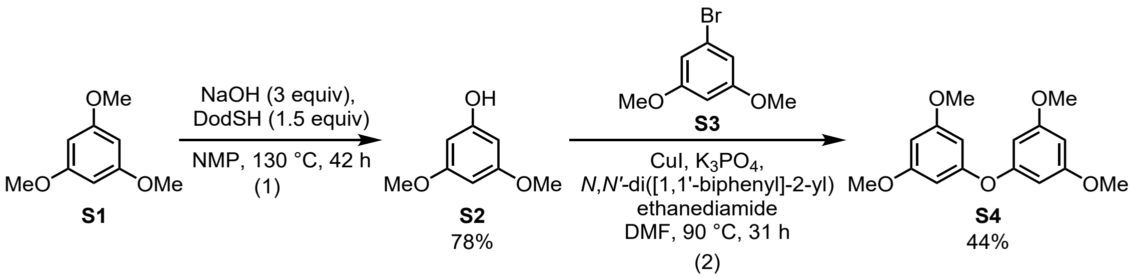
1. General information

Infrared (IR) spectra were recorded on a JASCO FT/IR-4100. ^{1}H NMR spectra were recorded on Bruker AVANCE NEO 600 (600 MHz), JEOL NM-ECZ600R (600 MHz), JNM ECA-500 (500 MHz), or JEOL JNM ECX-400 (400 MHz) with tetramethylsilane (TMS) as internal standard. Chemical shifts are reported in ppm relative to TMS (δ 0). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants, integration. ^{13}C NMR spectra were recorded on JEOL JNM ECA-500 (126 MHz) or a JEOL JNM ECX-400 (101 MHz) spectrometer with complete proton decoupling. High-resolution mass spectrometry were recorded on a Hitachi High-Technologies Nanofrontier LD Spectrometer in TOF mode and Bruker micrOTOF II (ESI–TOF–MS). Column chromatography was carried out with Cicareagent silica gel 60 N (spherical, particle size 63-210 mm). Thin layer chromatography (TLC) was carried out using Merck TLC plates with silica gel 60 F254. Unless otherwise noted, commercially available reagents were used without purification. UV absorption spectra were measured on a JASCO V-630 and JASCO V-770 spectrometer. The fluorescence spectra were obtained on a JASCO FP-8500 spectrofluorometer. Cyclic voltammetry measurements were carried out using an Admiral instruments Squidstat™ Solo and a ECstat-101. The photochemical reaction was carried out with a photoreactor (EvoluChem™ PhotoRedOx Box) and Red LED (λ_{ex} = 640 nm, 40W Kessil-PR160L).

2. Experimental Section

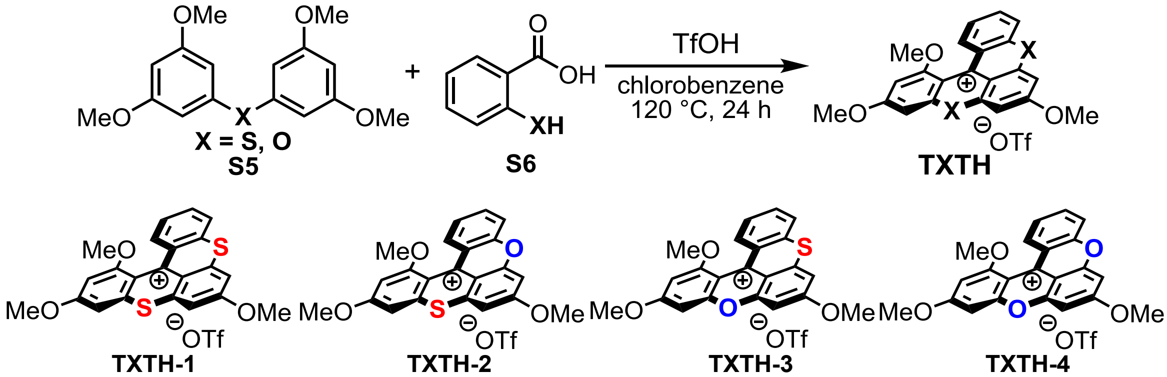
Procedure for the synthesis of bis(3,5-dimethoxyphenyl)ether[1-3]



1) A mixture of 1,3,5-trimethoxybenzene **S1** (3.34 g, 20 mmol), NaOH (2.40 g, 60 mmol), and 1-dodecanethiol (7.2 mL, 30 mmol) in anhydrous NMP (20 mL) was stirred under N_2 at 130 °C for 42 h. After the reaction mixture was allowed to cool to room temperature, it was acidified with 1 M HCl and diluted with EtOAc. The solution was extracted with EtOAc and the organic layer was washed with brine, dried over Na_2SO_4, and concentrated in vacuo. The crude was purified by column chromatography on silica gel (hexane/ethyl acetate = 10/1) to afford 3,5-dimethoxyphenol **S2** (2.39 g, 78% yield) as yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.08 (t, J = 2.4 Hz, 1H), 6.03 (d, J = 1.8 Hz, 2H), 3.76 (s, 6H).

2) A mixture of 3,5-dimethoxyphenol **S2** (585 mg, 3.8 mmol), CuI (10.0 mg, 0.05 mmol), *N,N'*-di([1,1'-biphenyl]-2-yl)ethanediamide (22.5 mg, 0.06 mmol), 1-bromo-3,5-dimethoxybenzene **S3** (577 mg, 2.7 mmol), and potassium phosphate (1.03 g, 4.9 mmol) in DMF (1.0 mL) was stirred under N_2 at 90 °C for 31 h. The reaction mixture was diluted with EtOAc (5 mL) and filtered. The crude was purified by column chromatography on silica gel (hexane/ethyl acetate = 10/1) to provide bis(3,5-dimethoxyphenyl)ether **S4** (343 mg, 44% yield) as yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.23 (t, J = 2.0 Hz, 1H), 6.20 (d, J = 2.3 Hz, 2H), 3.76 (s, 6H).

Procedure for the synthesis of helicenium salt TXTH[4]



To a solution of Ar-X-Ar **S5** (0.20 mmol) and salicylic acid **S6** (0.60 mmol) in chlorobenzene (5.0 mL) was placed in a 50 mL recovery flask under N_2. Trifluoromethanesulfonic acid (0.60 mmol) was slowly added to the solution, which was heated to 120 °C for 24 h. It was cooled to room temperature and excess Et_2O was added to precipitate a solid. After stirred for 18 h, the mixture was filtered. The solid was washed with Et_2O and dried in vacuo, affording the desired helicenium salt **TXTH**.

1,3,7-Trimethoxythiochromeno[2,3,4-*kl*]thioxanthen-5-ium trifluoromethanesulfonate (**TXTH-1**)
Black solid (75.8 mg, 71% yield). ^{1}H NMR (400 MHz, $(CD_3)_2CO$) δ 8.18 (dd, *J* = 0.9, 8.2 Hz, 1H), 8.06 (d, *J* = 2.7 Hz, 1H), 8.03 (d, *J* = 2.7 Hz, 1H), 8.02 - 7.93 (m, 2H), 7.73 (ddd, *J* = 0.9, 6.9, 8.3 Hz, 1H), 7.52 (d, *J* = 2.3 Hz, 1H), 6.99 (d, *J* = 2.3 Hz, 1H), 4.23 (s, 3H), 4.20 (s, 3H), 3.76 (s, 3H); ^{13}C NMR (101 MHz, $(CD_3)_2CO$) δ 169.07, 164.58, 163.77, 158.30, 147.45, 144.33, 143.14, 139.25, 137.03, 134.96, 131.07, 128.55, 127.23, 122.06, 115.47, 112.20, 111.56, 103.04, 101.51, 57.97, 57.84, 56.60; IR (ATR): 1574, 1457, 1241, 1221, 1152, 1031, 835, 756, 637 cm^{-1}; HRMS (ESI+) m/z calcd for $C_{22}H_{17}O_3S_2$ ([M]$^+$): 393.06137, found: 393.06270.

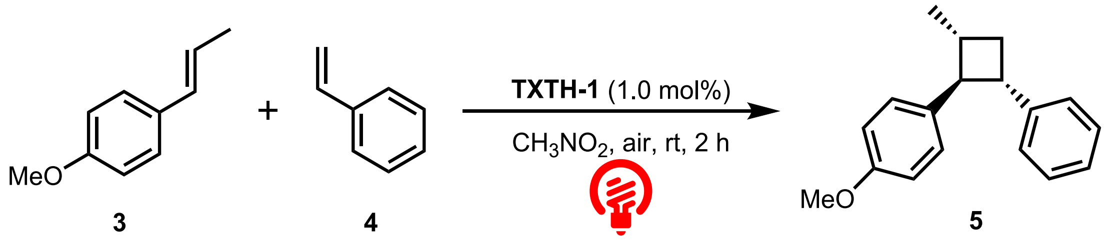
7,11,13-Trimethoxythiochromeno[2,3,4-*kl*]xanthen-9-ium trifluoromethanesulfonate (**TXTH-2**)
Brown solid (75.4 mg, 72% yield). ^{1}H NMR (500 MHz, CD_3CN) δ 7.97 (ddd, *J* = 1.7, 6.8, 8.6 Hz, 1H), 7.82 (dd, *J* = 1.4, 8.3 Hz, 1H), 7.72 (d, *J* = 8.4 Hz, 1H), 7.60 (d, *J* = 2.3 Hz, 1H), 7.51 (ddd, *J* = 1.2, 6.9, 8.0 Hz, 1H), 7.36 (d, *J* = 2.3 Hz, 1H), 7.24 (d, *J* = 2.3 Hz, 1H), 6.84 (d, *J* = 2.3 Hz, 1H), 4.10 (s, 3H), 4.09 (s, 3H), 3.77 (s, 3H); ^{13}C NMR (126 MHz, CD_3CN) δ 169.26, 167.99, 164.57, 157.73, 156.10, 154.82, 147.93, 139.81, 138.95, 134.02, 125.67, 123.51, 120.96, 120.74, 116.15, 111.62, 109.76, 103.72, 101.72, 101.37, 58.51, 58.28, 56.87; IR (ATR): 1581, 1467, 1377, 1333, 1253, 1222, 1150, 1027, 997, 836, 777, 635 cm^{-1}; HRMS (ESI+) m/z calcd for $C_{22}H_{17}O_4S$ ([M]$^+$): 377.08421, found: 377.08451.

1,3,7-Trimethoxythiochromeno[2,3,4-*kl*]xanthen-5-ium trifluoromethanesulfonate (**TXTH-3**)
Brown solid (68.5 mg, 69% yield). ^{1}H NMR (400 MHz, CD_3CN) δ 8.05 (dd, *J* = 1.1, 8.5 Hz, 1H), 8.00 (d, *J* = 7.3 Hz, 1H), 7.86 (t, *J* = 7.3 Hz, 1H), 7.68 (d, *J* = 2.3 Hz, 1H), 7.62 (t, *J* = 7.7 Hz, 1H), 7.37 (d, *J* = 2.3 Hz, 1H), 6.97 (d, *J* = 2.3 Hz, 1H), 6.77 (d, *J* = 2.3 Hz, 1H), 4.11 (s, 3H), 4.10 (s, 3H), 3.79 (s, 3H); ^{13}C NMR (101 MHz, CD_3CN) δ 172.14, 167.65, 161.88, 161.28, 157.28, 155.74, 140.80, 140.59, 137.11, 135.79, 127.86, 127.56, 126.45, 116.18, 109.39, 108.32, 101.48, 99.73, 94.87, 58.46, 58.41, 57.02; IR (ATR): 1602, 1483, 1255, 1222, 1153, 1029, 834, 637, 571 cm^{-1}. HRMS (ESI+) m/z calcd for C22H17O4S ([M]+): 377.08421, found: 377.0832.

1,3,7-Trimethoxychromeno[2,3,4-*kl*]xanthen-5-ium trifluoromethanesulfonate (**TXTH-4**)

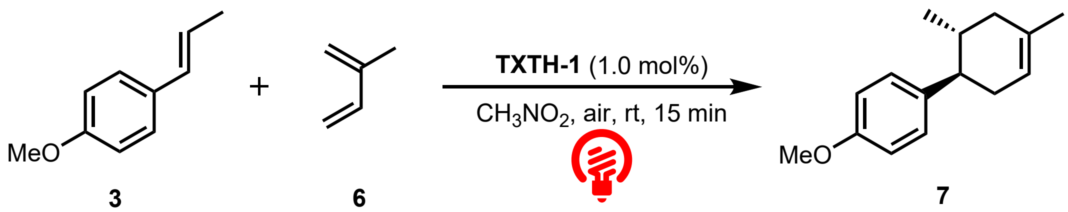
Orange solid (73.0 mg, 72% yield). ^{1}H NMR (500 MHz, $(CD_3)_2CO$) δ 8.47 (dd, *J* = 1.7, 8.6 Hz, 1H), 8.15 (ddd, *J* = 1.4, 7.2, 8.6 Hz, 1H), 7.88 (dd, *J* = 1.1, 8.6 Hz, 1H), 7.68 (ddd, *J* = 1.1, 7.2, 8.3 Hz, 1H), 7.50 (d, *J* = 1.7 Hz, 1H), 7.44 (d, *J* = 2.3 Hz, 1H), 7.14 (d, *J* = 2.9 Hz, 1H), 6.98 (d, *J* = 2.3 Hz, 1H), 4.26 (s, 3H), 4.23 (s, 3H), 4.11 (s, 3H); ^{13}C NMR (101 MHz, $(CD_3)_2CO$) δ 172.44, 170.93, 162.24, 161.59, 157.27, 154.78, 154.40, 152.80, 139.76, 133.71, 125.54, 120.84, 118.92, 117.81, 110.04, 105.46, 99.34, 99.10, 98.75, 95.48, 58.32, 58.08, 56.99; IR (ATR): 1601, 1475, 1254, 1152, 1028, 845, 767, 637, 567, 556 cm^{-1}; HRMS (ESI+) m/z calcd for $C_{22}H_{17}O_5$ ($[M]^+$): 361.10705, found: 361.10912.

Procedure for cross-[2+2] cycloaddition of styrenes under red LED irradiation[4]



The reaction was conducted with *trans*-anethole **3** (76.0 μL, 0.50 mmol), styrene **4** (575 μL, 5.0 mmol), **TXTH-1** (2.7 mg, 0.005 mmol), and CH_3NO_2 (8.0 mL). The resulting solution was stirred at room temperature under air and Red LED irradiation (λ_{ex} = 640 nm, 40W). After being stirred for 2 h, the reaction mixture was concentrated in vacuo. The resulting residue was purified by column chromatography on silica gel (hexane/EtOAc = 20:1) to give the desired product **5**. Colorless oil (108.8 mg, 86% yield). ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.32-7.30 (m, 2H), 7.25-7.21 (m, 5H), 6.89 (d, *J* = 8.4 Hz, 2H), 3.82 (s, 3H), 3.43 (q, *J* = 9.6 Hz, 1H), 2.99 (t, *J* = 9.0 Hz, 1H), 2.56 (q, *J* = 7.2 Hz, 1H), 2.41-2.35 (m, 1H), 1.74 (q, *J* = 10.2 Hz, 1H), 1.23 (d, *J* = 6.6 Hz, 3H).

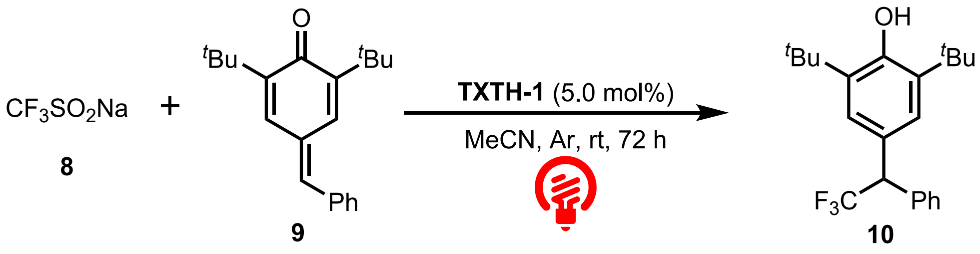
Procedure for radical-cation Diels–Alder reaction under red LED irradiation[5]



The reaction was conducted with *trans*-Anethole **3** (76.0 μL, 0.50 mmol), isoprene **6** (150 μL, 1.5 mmol), **TXTH-1** (2.7 mg, 0.005 mmol) and CH_3NO_2 (8.0 mL). The resulting solution was stirred at room temperature under air and Red LED irradiation (λ_{ex} = 640 nm, 40W) for 15 min. The desired product **7** was isolated by column chromatography on silica gel (hexane/EtOAc = 20:1). Colorless oil (110.1 mg, >99% yield).

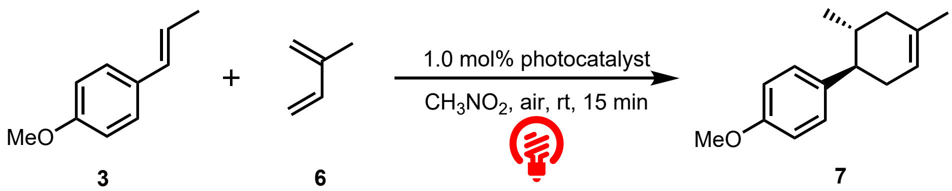
^1H NMR (600 MHz, $CDCl_3$) δ 7.07 (d, J = 8.4 Hz, 2H), 6.83 (d, J = 9.0 Hz, 2H), 5.43 (br s, 1H), 3.78 (s, 3H), 2.29 (dt, J = 10.8, 5.4 Hz, 1H), 2.21-2.05 (m, 3H), 1.91-1.76 (m, 2H), 1.68 (s, 3H), 0.70 (d, J = 6.6 Hz, 3H).

Procedure for trifluoromethylation of *p*-quinone methide[6]



The reaction was conducted with **8** (46.9 mg, 0.300 mmol), **9** (45.5 mg, 0.154 mmol), **TXTH-1** (4.4 mg, 0.008 mmol), and CH_3CN (3.0 mL). The vial was sealed with a PTFE septum and bubbled with argon gas for 5 min. The resulting solution was stirred at room temperature under Ar and Red LED irradiation (λ_{ex} = 640 nm, 40W). After being stirred for 72 h, the reaction mixture was added with water, followed by extracting with EtOAc. The combined organic phases were washed with brine, dried over anhydrous Na_2SO_4 and concentrated in vacuo. The resulting residue was purified by PTLC (hexane : ethyl acetate = 10 : 1) to give the desired product **10**. Yellow oil (48.1 mg, 85%). ^1H NMR (600 MHz, $CDCl_3$) δ 7.42 (d, J = 7.8 Hz, 2H), 7.37 (t, J = 7.2 Hz, 2H), 7.32-7.30 (m, 1H), 7.17 (s, 2H), 5.22 (s, 1H), 4.59 (q, J = 10.2 Hz, 1H), 1.43 (s, 18H).

Radical-cation Diels–Alder reaction in the presence of photocatalyst under red LED irradiation



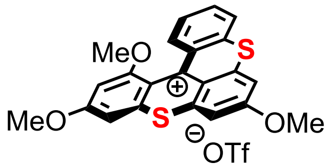
Catalyst	Yield (%)
Methylene blue	0
Bridged eosin Y	91
TXTH-1	>99%

3. Photophysical and redox properties of TXTH catalysts

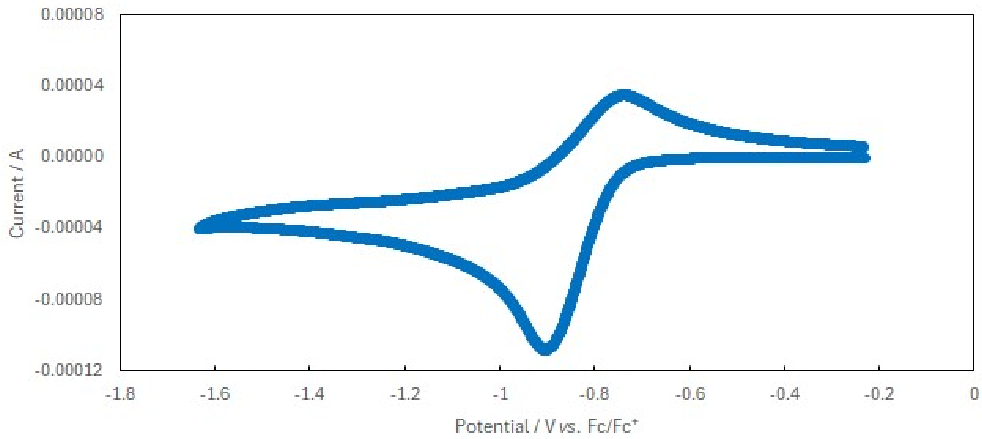
The samples for electrochemical measurements were prepared with 10 mL of a 0.1 M *tetra*-n-butylammonium tetrafluoroborate (**TXTH-2, TXTH-3, TXTH-4**) or *tetra*-n-butylammonium perchlorate (**TXTH-1**) solution in CH_3CN and 0.01 mmol of substrate. Cyclic voltammetry measurements were carried out with Squidstat Solo. Cyclic Voltammetry was recorded using an undivided cell equipped with a working electrode (Pt disk electrode, φ 3mm), a counter electrode (Pt wire), and a reference electrode (Ag wire). The ferrocene/ferrocenium couple (Fc/Fc^+) was also measured in the same electrochemical system, and the electrode potential was reported as values referred to the apparent standard potential of the system. The referenced value was converted to SCE by adding 0.38 V (in CH_3CN). A scan rate of 0.5 V/s was used for **TXTH**, and 0.3 V/s for **TXT**.

Samples for photophysical measurements were prepared using high purity acetonitrile. Solutions of the substrates were diluted to a concentration of 10 μM and a total volume of 3.0 mL before being transferred to a 3.5 mL quartz cell. The solvent absorbance background was subtracted from the reported spectra.

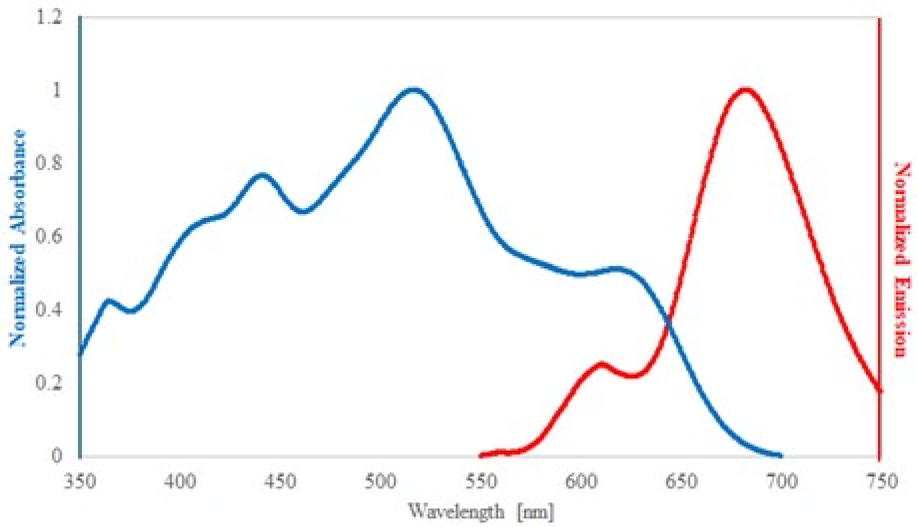
Excited-state reduction potentials ($E_{1/2}$*) were calculated by subtracting the ground-state reduction potential ($E_{1/2}$), obtained by cyclic voltammetry, from the excitation energy ($E_{0,0}$); $E_{0,0}$ is determined by calculating the energy of the wavelength at which the substrate's UV-Vis absorption and emission spectra overlap.



TXTH-1

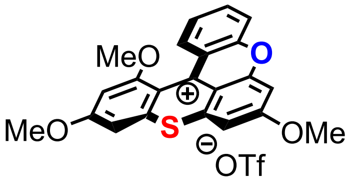


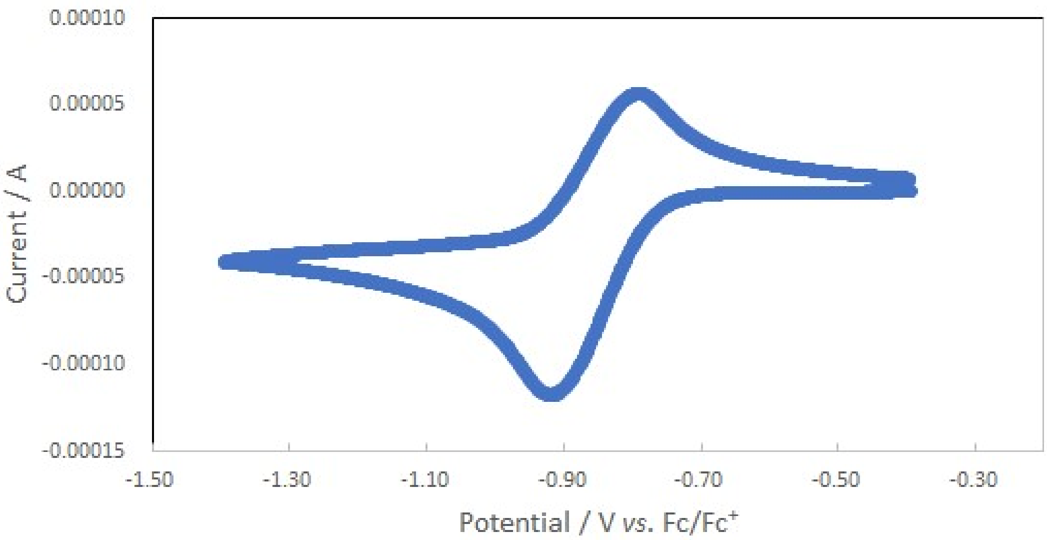
$E_{1/2}$ = – 0.82 V vs Fc/Fc$^+$ (– 0.42 V vs SCE) in CH_3CN



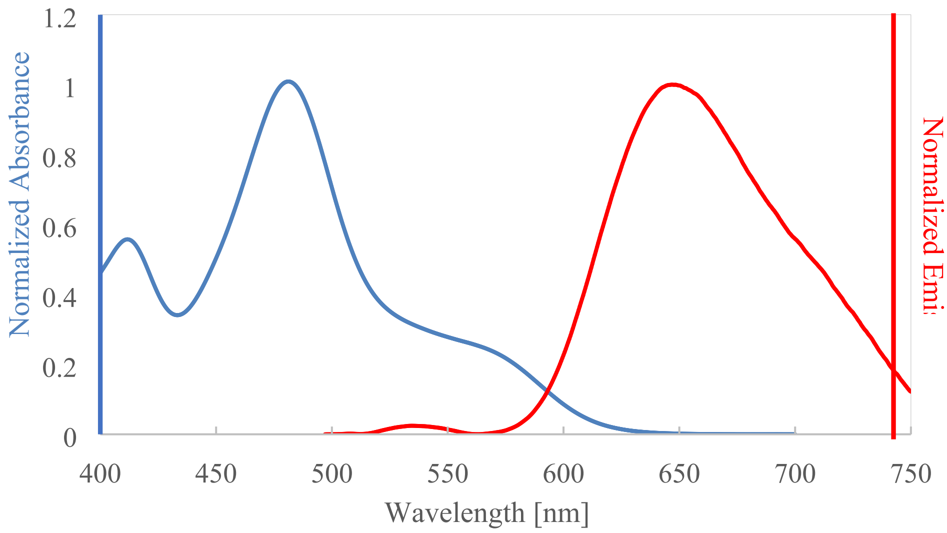
$$E_{0,0} = \frac{1240\ eV \cdot nm}{644\ nm} = 1.93\ eV$$

$$E_{1/2}{}^{*} = E_{1/2} + E_{0,0}$$
$$= -\ 0.42 + 1.93\ \mathrm{V}$$
$$= +\ 1.51\ \mathrm{V\ vs\ SCE\ in\ CH_3CN}$$





$E_{1/2}$ = – 0.86 V vs Fc/Fc^{+} (– 0.46 V vs SCE) in CH_3CN

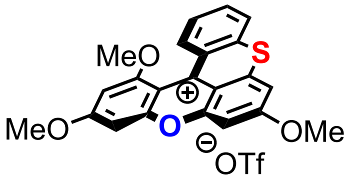


$$E_{0,0} = \frac{1240\ eV \cdot nm}{594\ nm} = 2.09\ eV$$

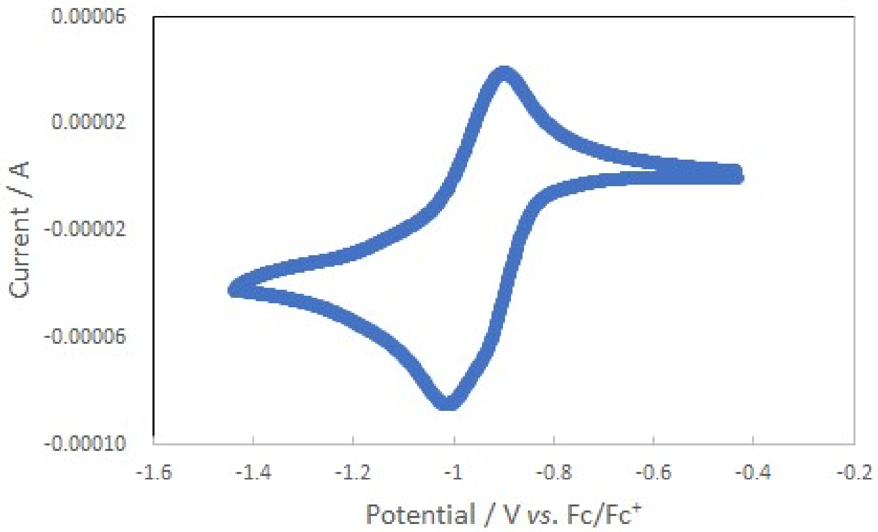
$$E_{1/2}^{*} = E_{1/2} + E_{0,0}$$

$$= -0.46 + 2.09\ \text{V}$$

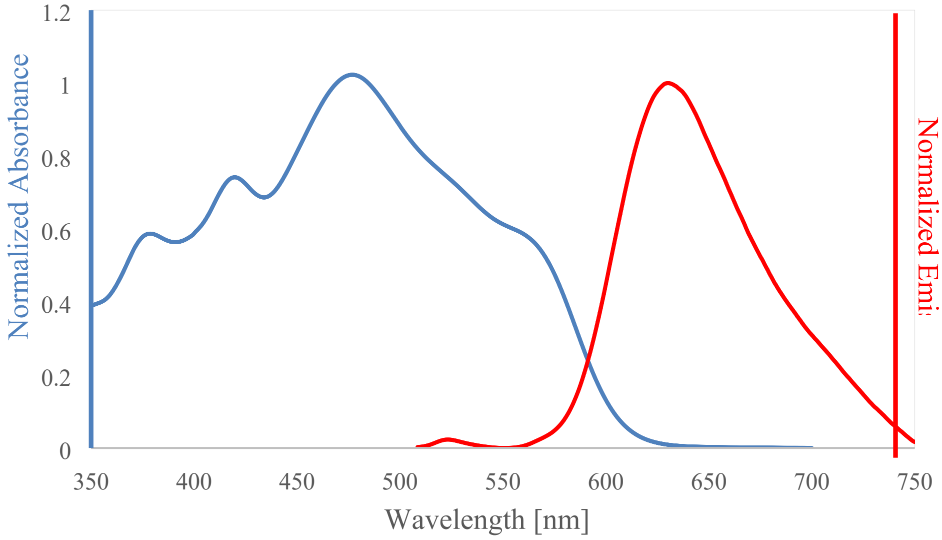
$$= +1.63\ \text{V vs SCE in } CH_3CN$$



TXTH-3

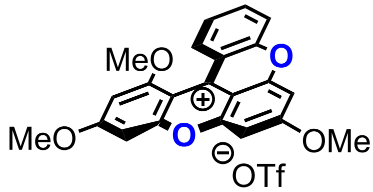


$E_{1/2}$ = – 0.96 V vs Fc/Fc^{+} (– 0.56 V vs SCE) in CH_3CN

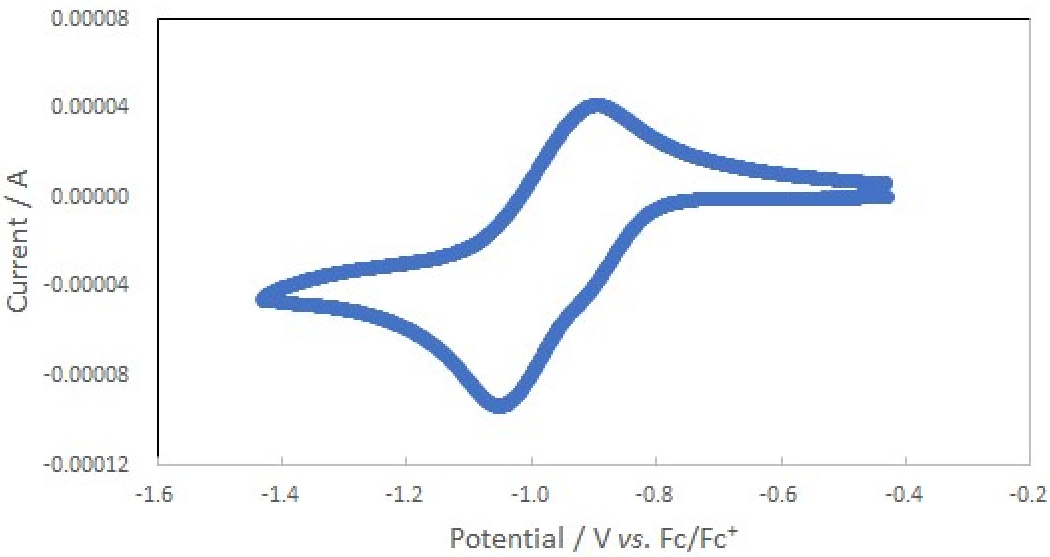


$$E_{0,0} = \frac{1240\ eV \cdot nm}{590\ nm} = 2.10\ eV$$

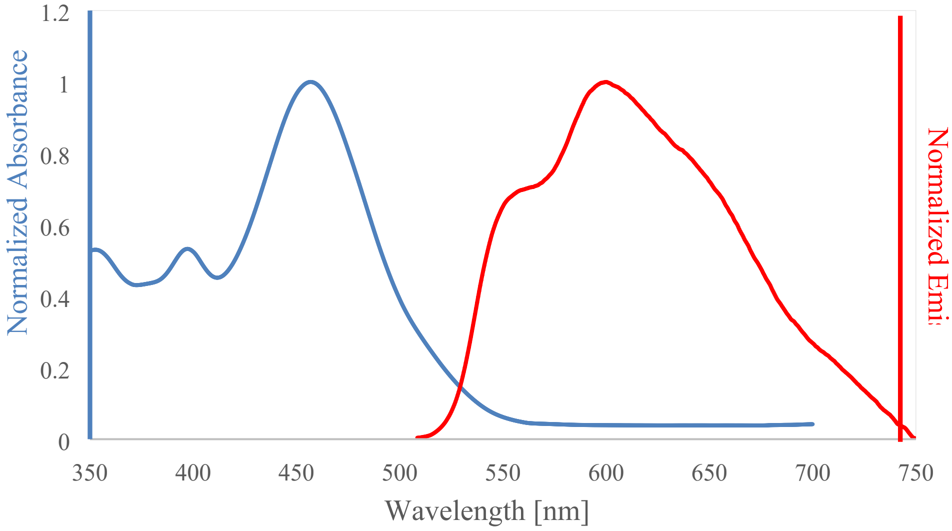
$$E_{1/2}{}^{*} = E_{1/2} + E_{0,0}$$
$$= -\ 0.56 + 2.10\ \mathrm{V}$$
$$= +\ 1.54\ \mathrm{V\ vs\ SCE\ in\ CH_3CN}$$



TXTH-4



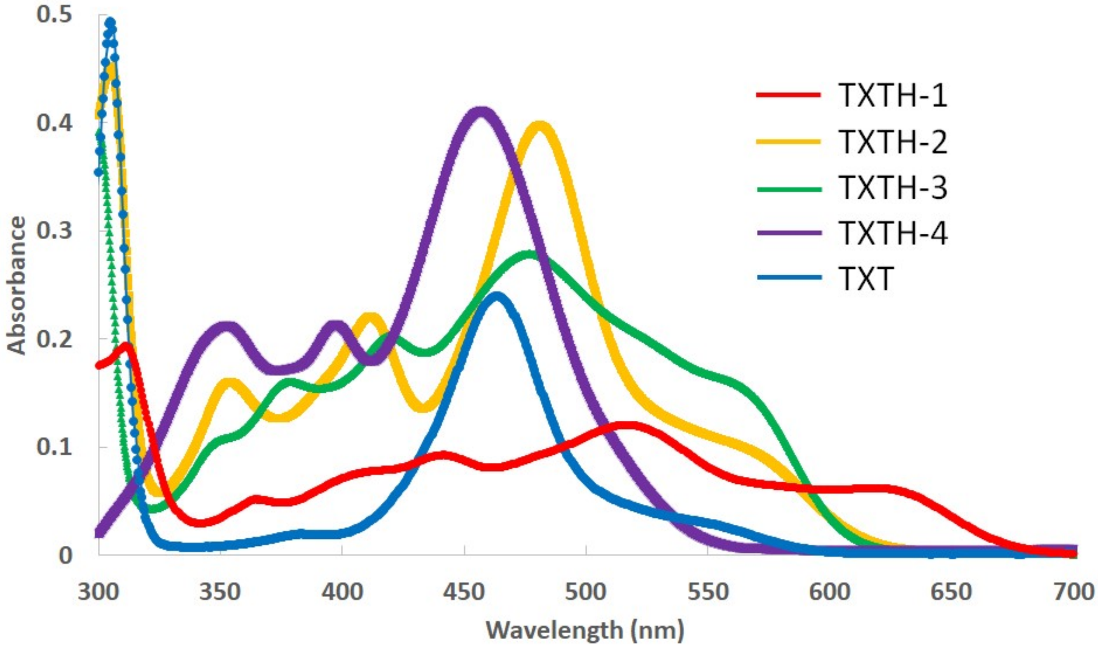
$E_{1/2}$ = – 0.98 V vs Fc/Fc^+ (– 0.58 V vs SCE) in CH_3CN



$$E_{0,0} = \frac{1240\ eV \cdot nm}{529\ nm} = 2.34\ eV$$

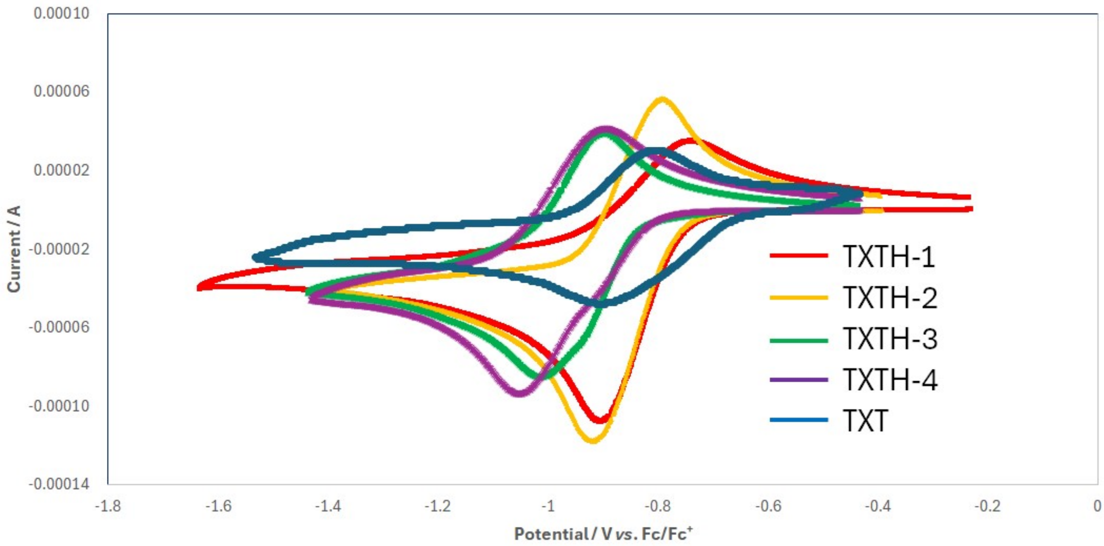
$$E_{1/2}{*} = E_{1/2} + E_{0,0}$$
$$= -\ 0.58 + 2.34\ \text{V}$$
$$= +\ 1.76\ \text{V vs SCE in } CH_3CN$$

UV-vis spectra of TXTH and TXT

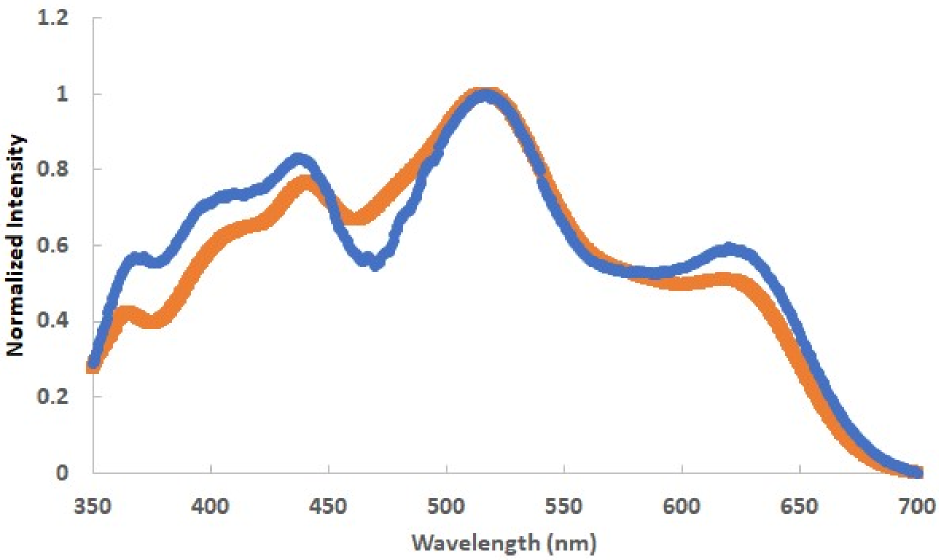


Catalyst	λ_{max} [nm] / ε [mol^{-1} dm$_3$ cm^{-1}]
TXTH1	517 / 1.80 × 10^4
TXTH2	481 / 3.98 × 10^4
TXTH3	477 / 2.79 × 10^4
TXTH4	457 / 4.11 × 10^4

Cyclic voltammograms of TXTH and TXT



The excitation and absorption spectra of TXTH-1



Blue line: excitation spectrum, Orange line: absorption spectrum

4. References

(1) Y. Pauvert and A. B. Charette, *Org. Lett.*, **2024**, *26*, 6081–6085.

(2) J. Chae, *Arch. Pharm. Res.*, **2008**, *31*, 305–309.

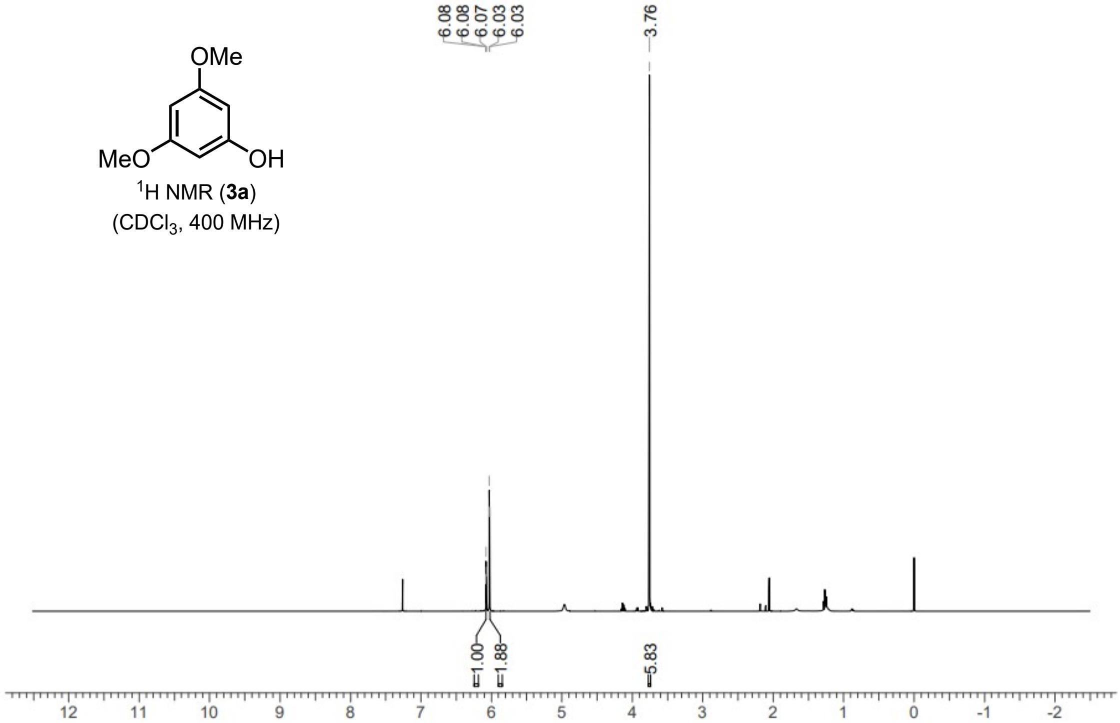
(3) S. Hor, K. Oyama, N. Koga and M. Tsukamoto, *Polymer*, **2022**, *238*, 124326.

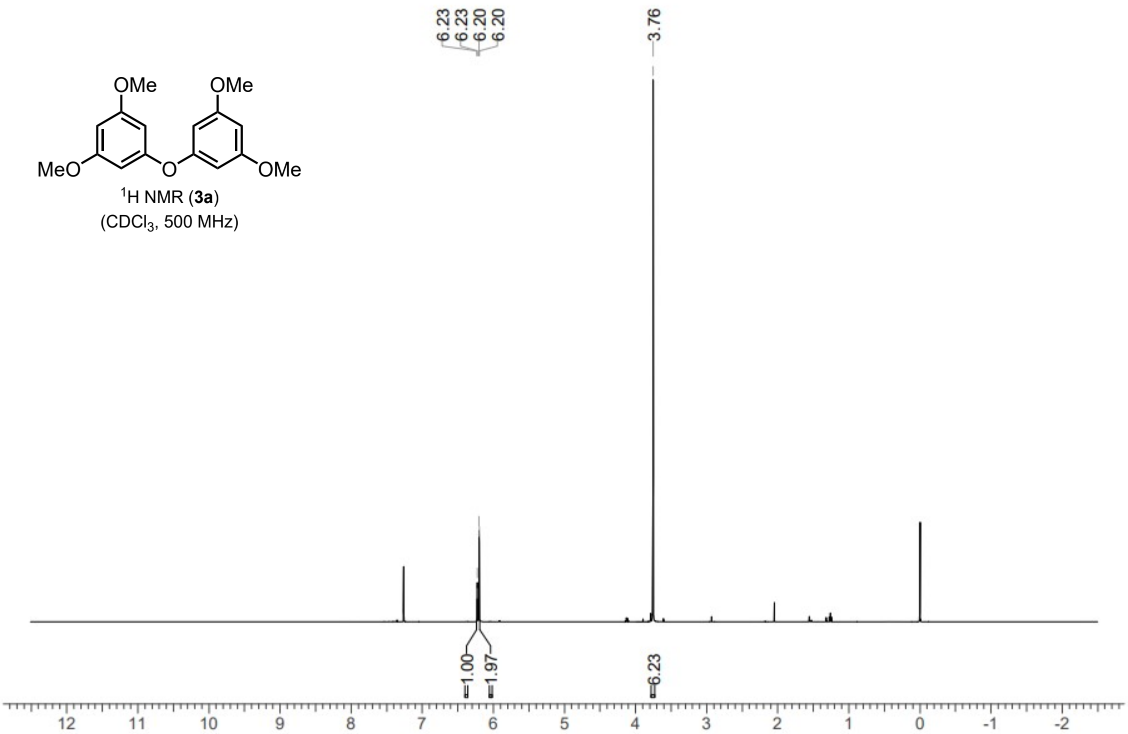
(4) K. Tanaka, Y. Iwama, M. Kishimoto, N. Ohtsuka, Y. Hoshino and K. Honda, *Org. Lett.*, **2020**, *22*, 5207–5211.

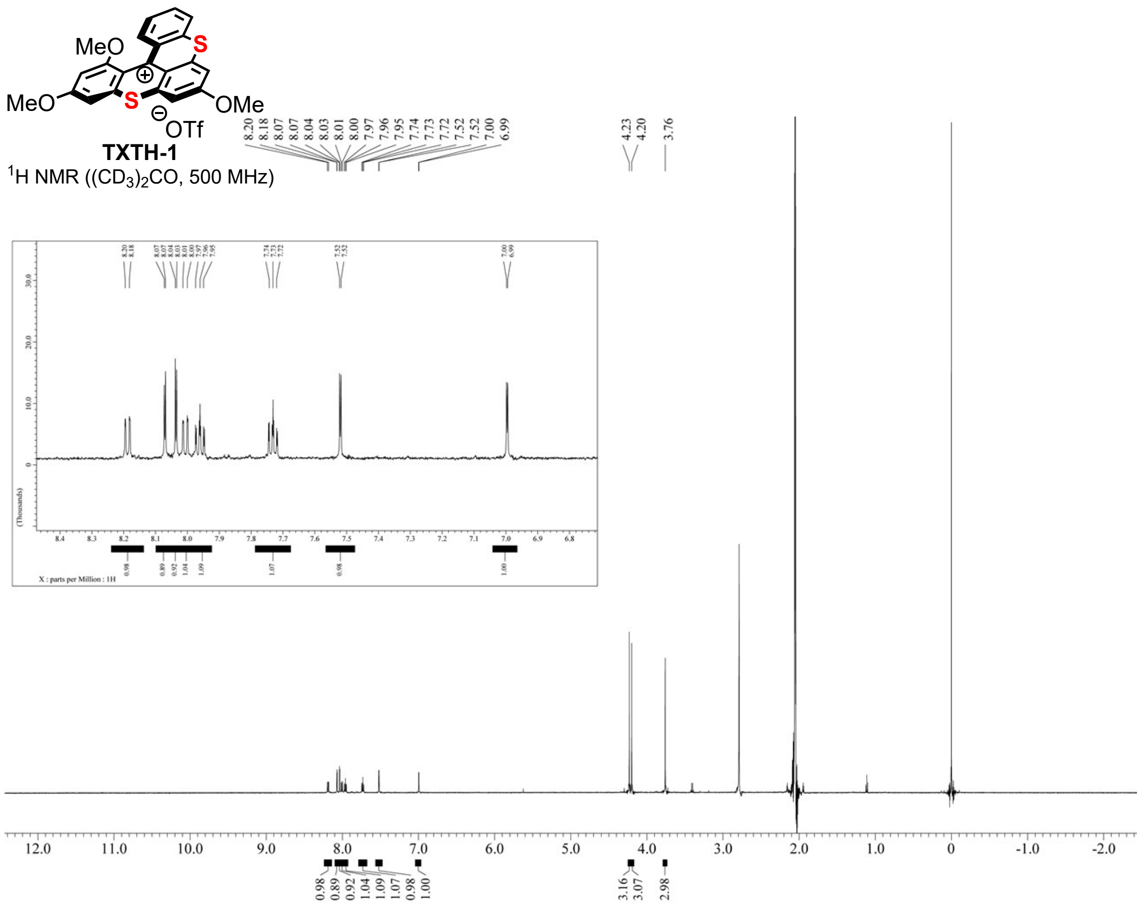
(5) K. Tanaka, M. Kishimoto, Y. Tanaka, Y. Kamiyama, Y. Asada, M. Sukekawa, N. Ohtsuka, T. Suzuki, N. Momiyama, K. Honda and Y. Hoshino, *J. Org. Chem.*, **2022**, *87*, 3319–3328.

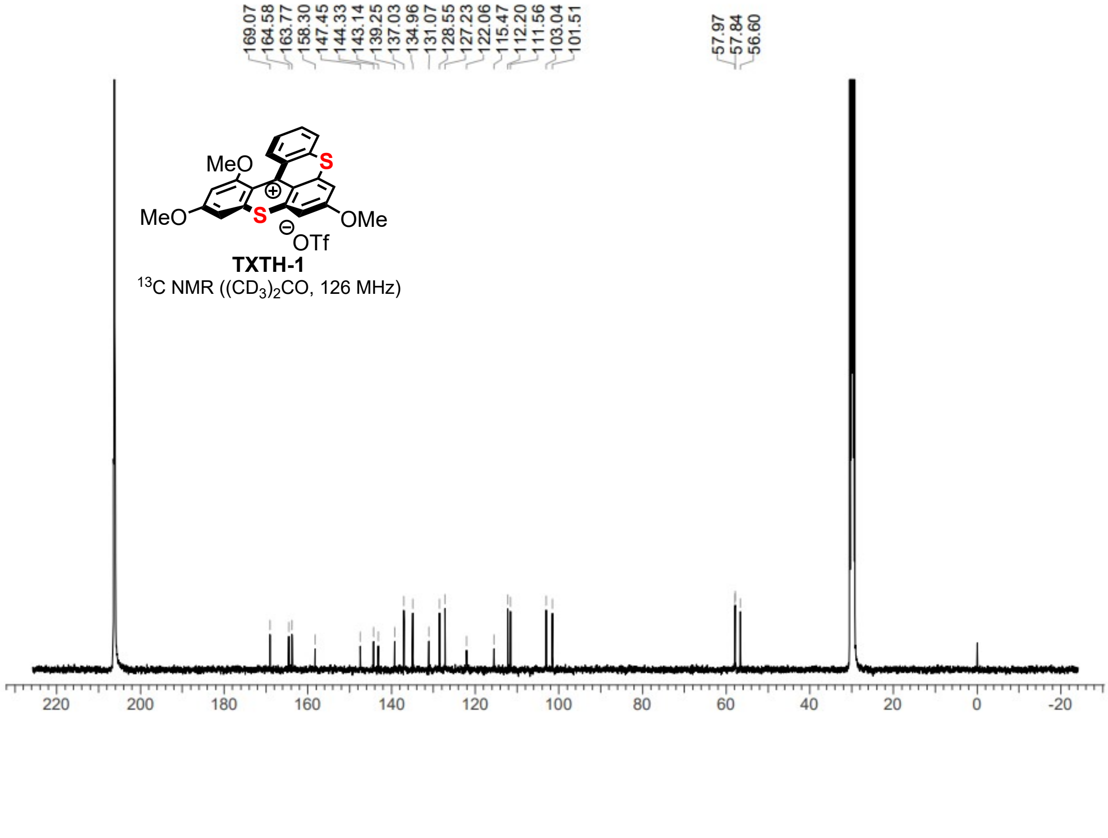
(6) K. G. Ghosh, P. Chandu, S. Mondal and D. Sureshkumar, *Tetrahedron*, **2019**, *75*, 4471–4478.

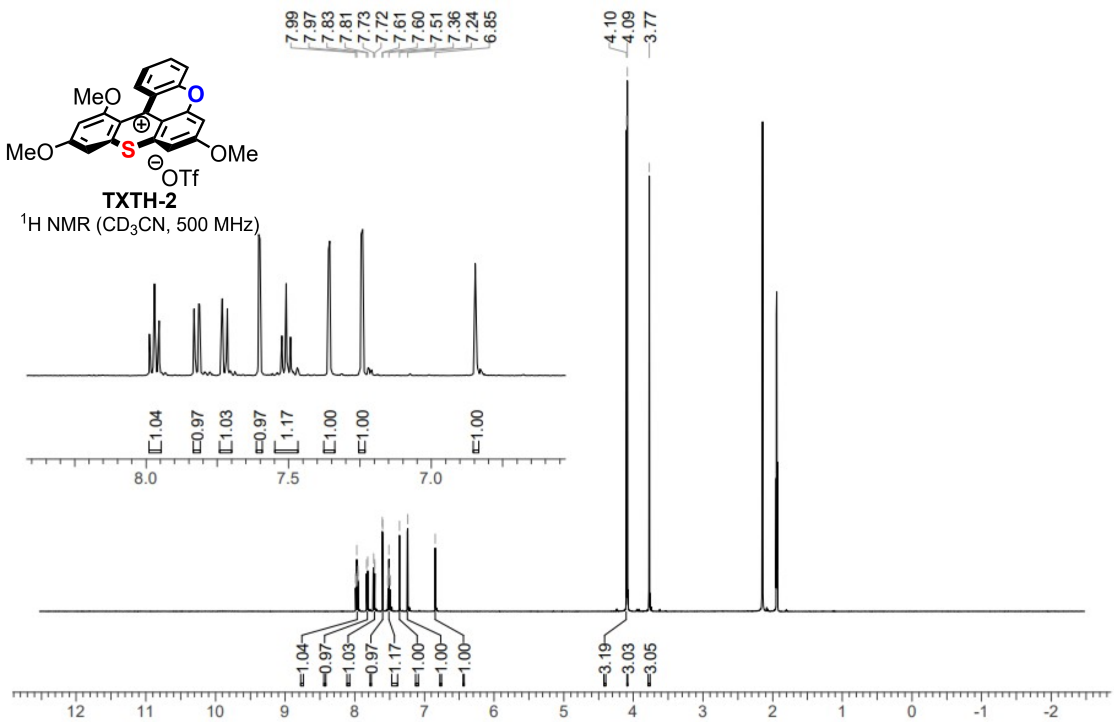
5. ^{1}H NMR and ^{13}C NMR spectral data

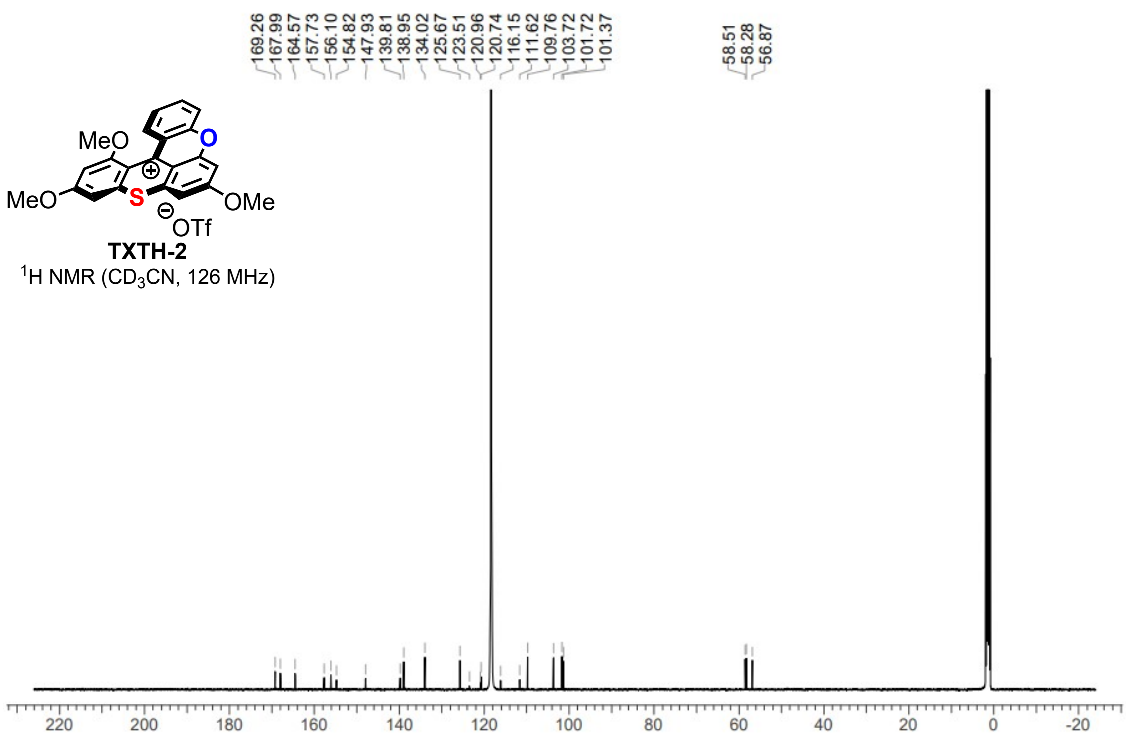


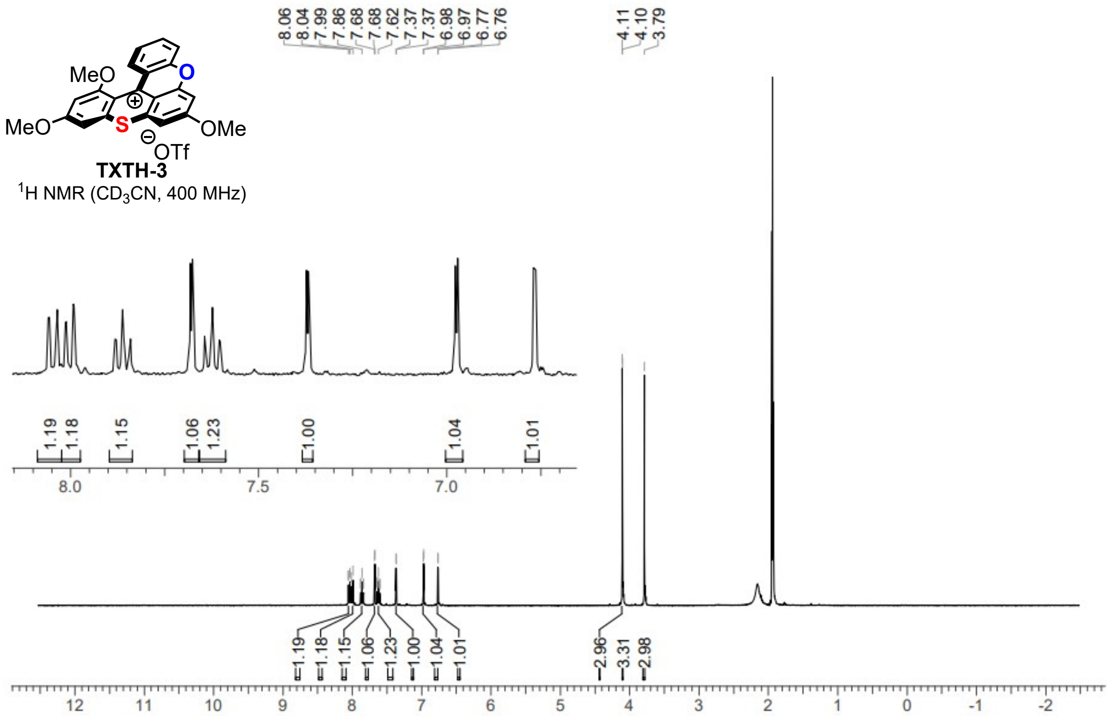


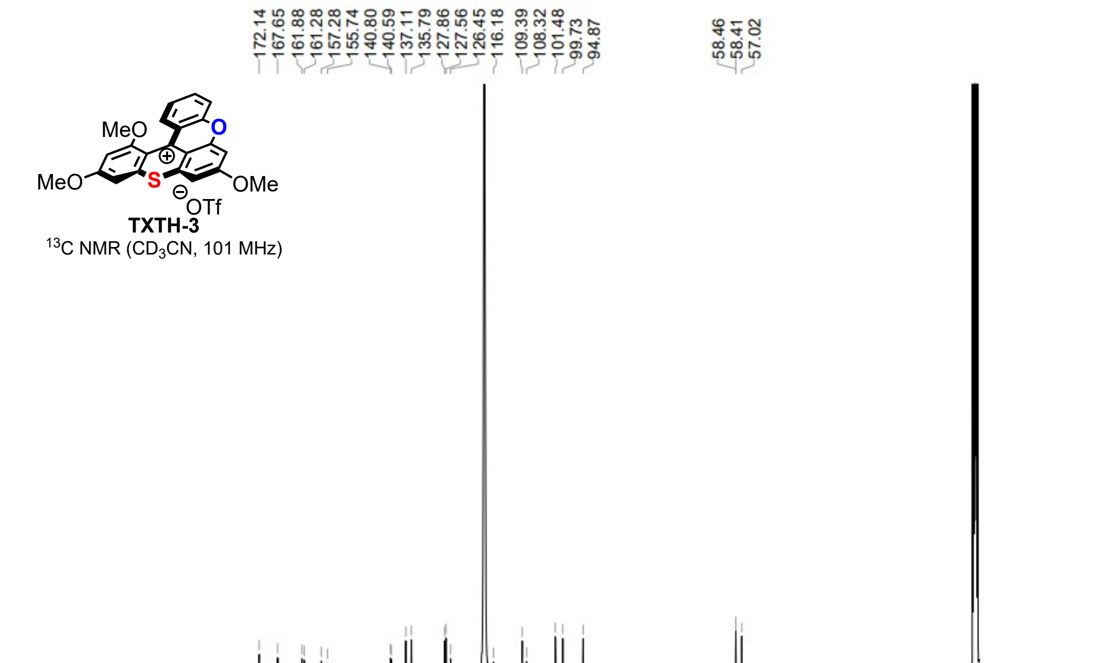

MeO
MeO
OMe
S
S
OTf
TXTH-1
1H NMR ((CD3)2CO, 500 MHz)
8.20 8.18 8.07 8.07 8.04 8.03 8.01 8.00 7.97 7.96 7.95 7.74 7.73 7.72 7.52 7.52 7.00 6.99
4.23 4.20 3.76
0.98 0.89 0.92 1.04 1.09 1.07 0.98 1.00
3.16 3.07 2.98
X : parts per Million : 1H

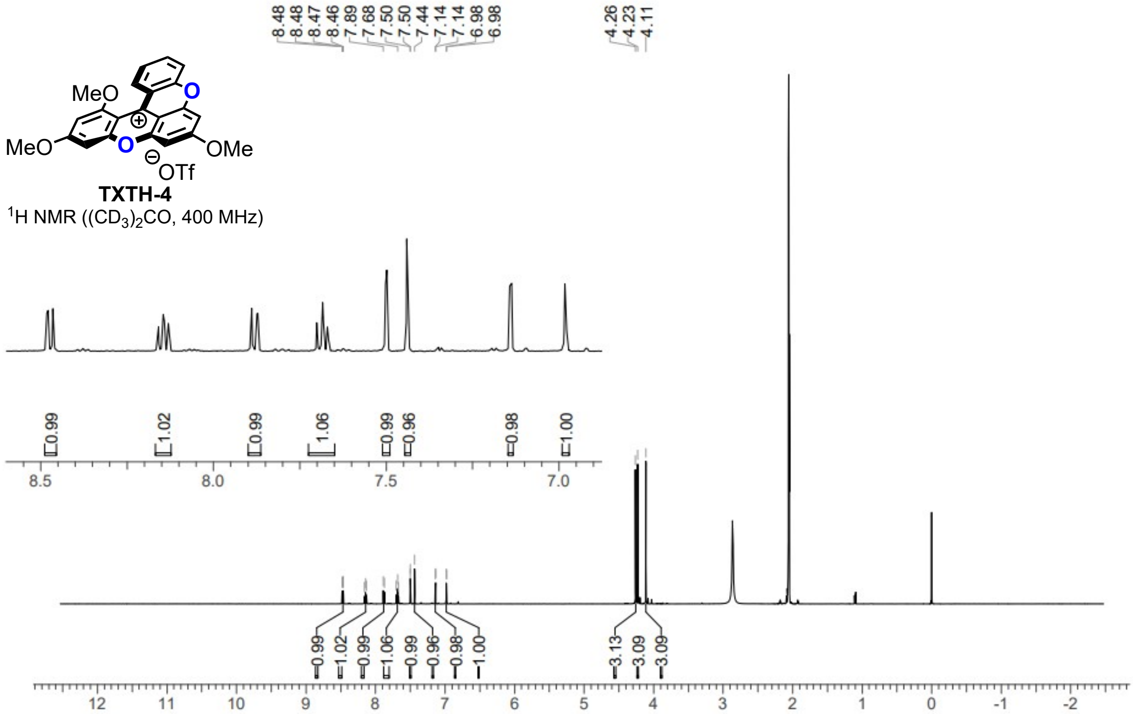

169.07 164.58 163.77 158.30 147.45 144.33 143.14 139.25 137.03 134.96 131.07 128.55 127.23 122.06 115.47 112.20 111.56 103.04 101.51
57.97 57.84 56.60
MeO
MeO
OMe
S
S
OTf
TXTH-1
13C NMR ((CD3)2CO, 126 MHz)

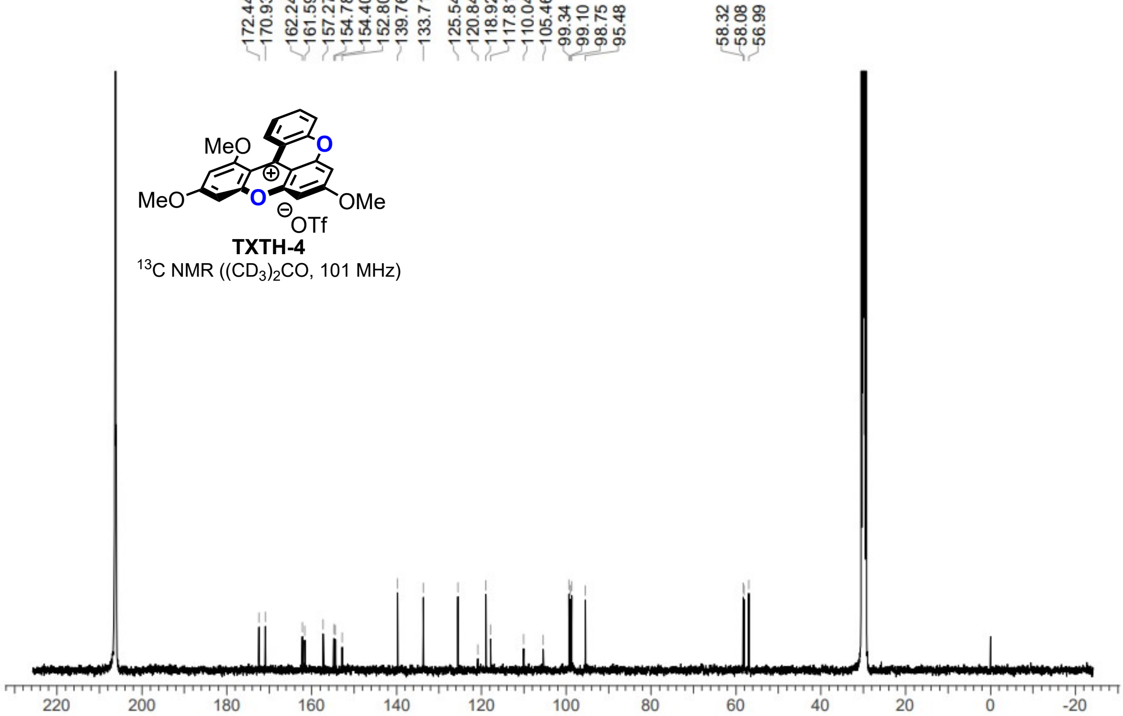

7.99 7.97 7.83 7.81 7.73 7.72 7.61 7.60 7.51 7.36 7.24 6.85
4.10 4.09 3.77
MeO
O
MeO
S
OMe
OTf
TXTH-2
1H NMR (CD3CN, 500 MHz)
1.04 0.97 1.03 0.97 1.17 1.00 1.00 1.00
3.19 3.03 3.05

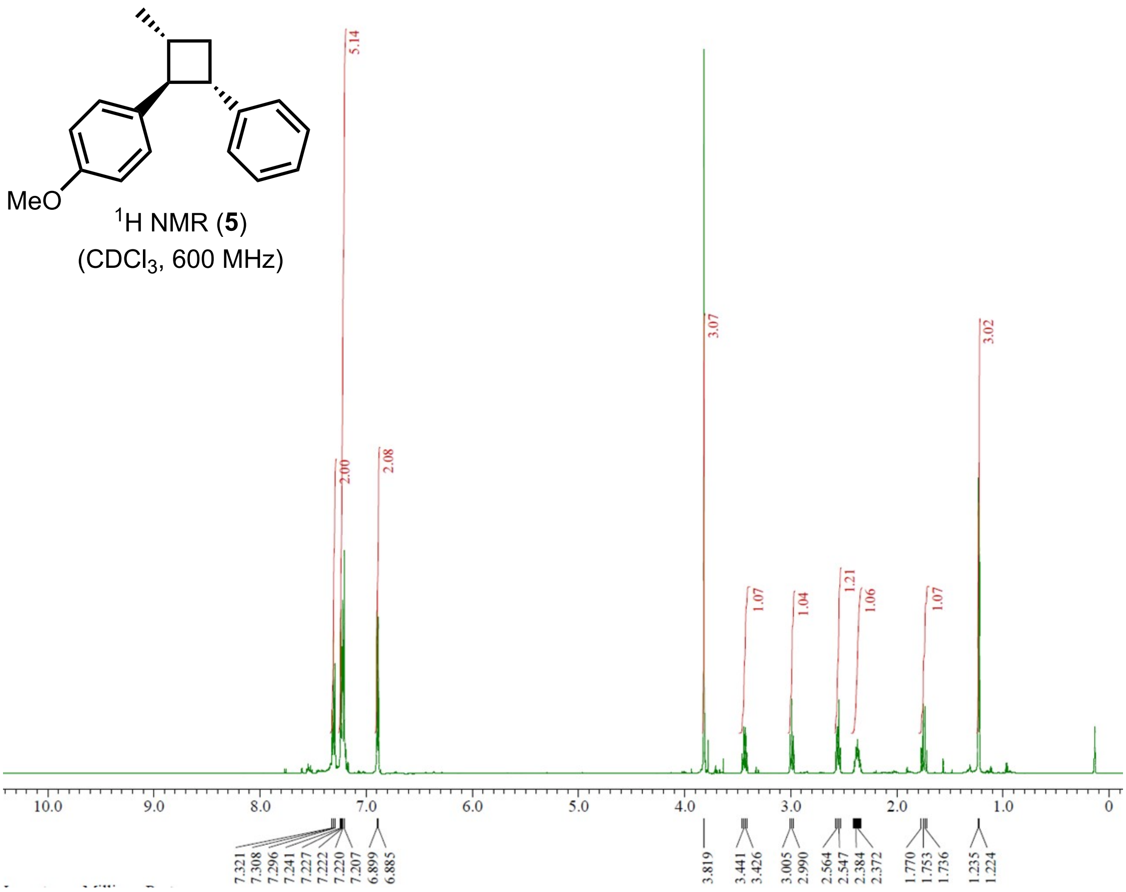

169.26 167.99 164.57 157.73 156.10 154.82 147.93 139.81 138.95 134.02 125.67 123.51 120.96 120.74 116.15 111.62 109.76 103.72 101.72 101.37
58.51 58.28 56.87
MeO
O
MeO
S
OMe
OTf
TXTH-2
1H NMR (CD3CN, 126 MHz)

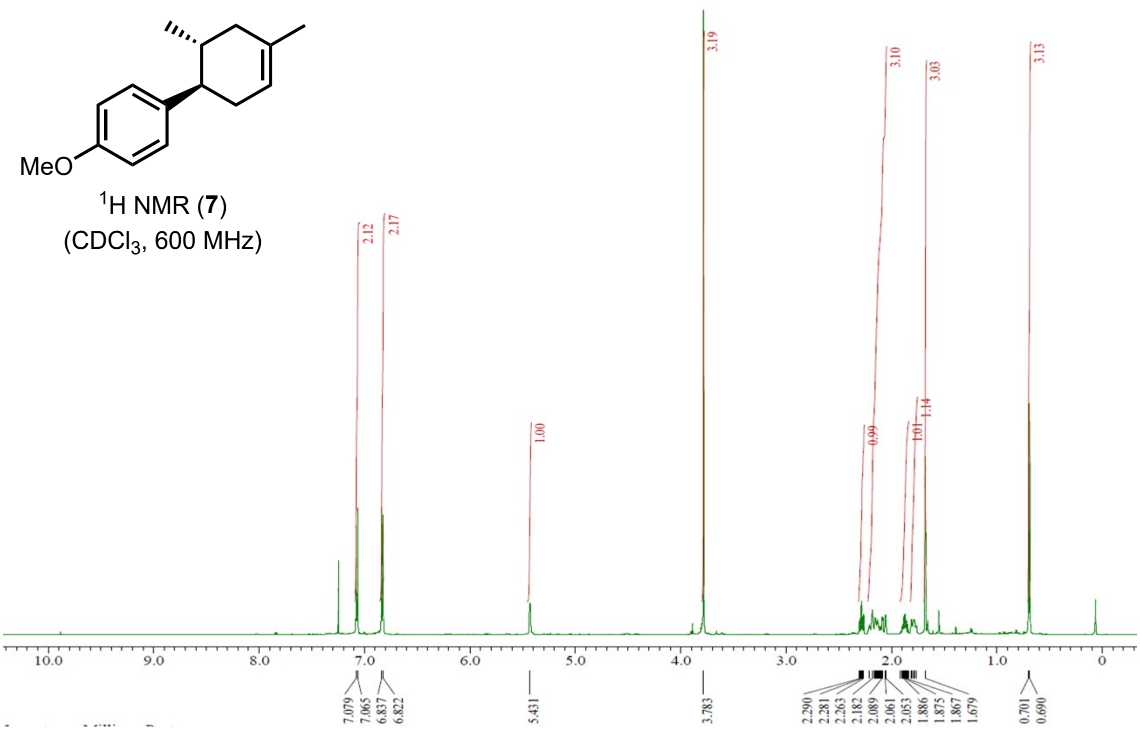

8.06 8.04 7.99 7.86 7.68 7.68 7.62 7.37 7.37 6.98 6.97 6.77 6.76
4.11 4.10 3.79
MeO
O
MeO
S
OMe
OTf
TXTH-3
^{1}H NMR (CD_3CN, 400 MHz)
1.19 1.18 1.15 1.06 1.23 1.00 1.04 1.01
8.0 7.5 7.0
1.19 1.18 1.15 1.06 1.23 1.00 1.04 1.01
2.96 3.31 2.98
12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2

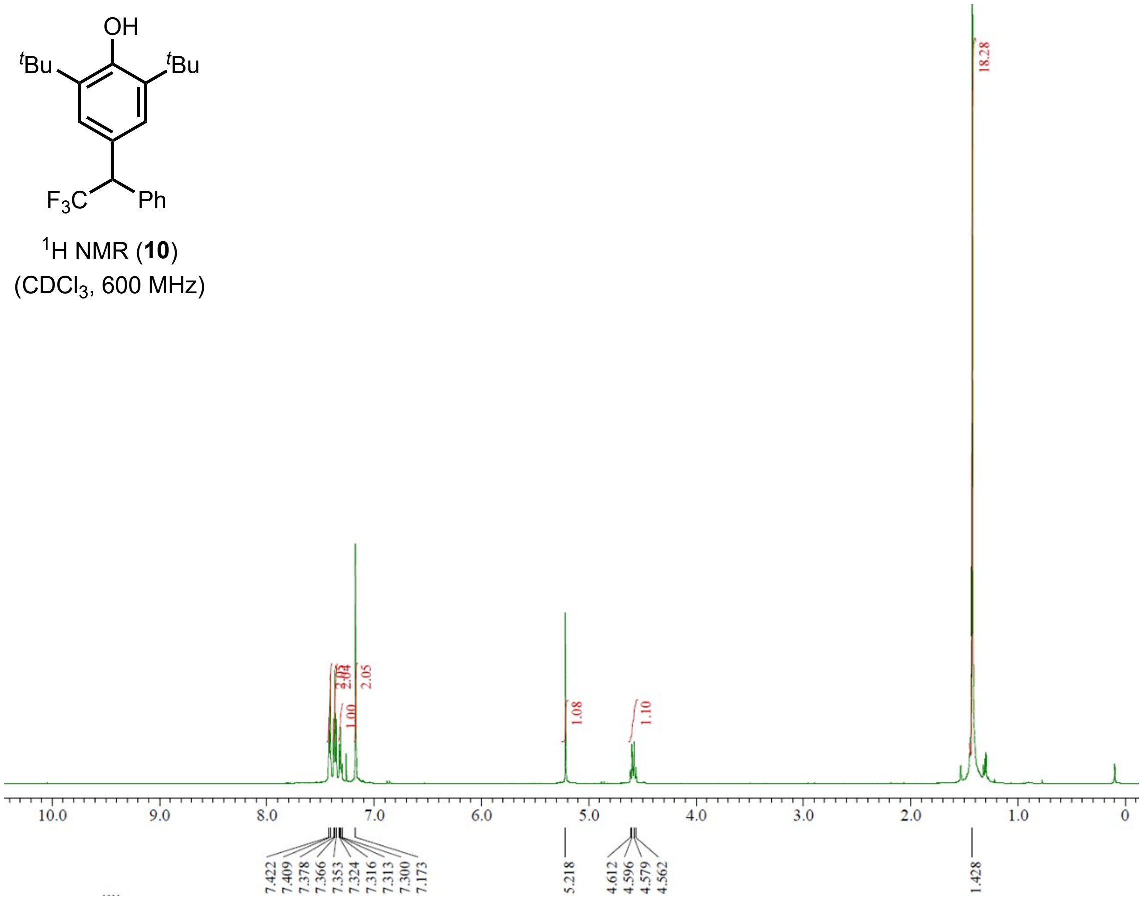

172.14 167.65 161.88 161.28 157.28 155.74 140.80 140.59 137.11 135.79 127.86 127.56 126.45 116.18 109.39 108.32 101.48 99.73 94.87
58.46 58.41 57.02
MeO
O
MeO
S
OMe
OTf
TXTH-3
^{13}C NMR (CD_3CN, 101 MHz)
220 200 180 160 140 120 100 80 60 40 20 0 -20


8.48 8.48 8.47 8.46 7.89 7.68 7.50 7.50 7.44 7.14 7.14 6.98 6.98
4.26 4.23 4.11
MeO
O
MeO
O
OTf
OMe
TXTH-4
1H NMR ((CD3)2CO, 400 MHz)
0.99 1.02 0.99 1.06 0.99 0.96 0.98 1.00
8.5 8.0 7.5 7.0
0.99 1.02 0.99 1.06 0.99 0.96 0.98 1.00 3.13 3.09 3.09
12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2


172.44 170.93 162.24 161.59 157.27 154.78 154.40 152.80 139.76 133.71 125.54 120.84 118.92 117.81 110.04 105.46 99.34 99.10 98.75 95.48
58.32 58.08 56.99
MeO
O
MeO
O
OTf
OMe
TXTH-4
13C NMR ((CD3)2CO, 101 MHz)
220 200 180 160 140 120 100 80 60 40 20 0 -20


MeO
1H NMR (5)
(CDCl3, 600 MHz)


MeO
1H NMR (7)
(CDCl3, 600 MHz)



OH
tBu
tBu
F3C
Ph
1H NMR (10)
(CDCl3, 600 MHz)
18.28
2.05
1.00
1.08
1.10
10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0
7.422 7.409 7.378 7.366 7.353 7.324 7.316 7.313 7.300 7.173
5.218
4.612 4.596 4.579 4.562
1.428