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Electrooxidative Double C-H/C-H Coupling of Phenols with 3-Phenylbenzothiophenes: Facile Access to Benzothiophene Derivatives

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Contents

1. General information

All the solvents and other reagents were purchased from commercial suppliers without the purification. All reactions were performed in N_2 atmosphere unless otherwise. ^{1}H and ^{13}C NMR data were recorded with Bruker Advance III (400 MHz) or (600 MHz) spectrometers with tetramethylsilane as an internal standard. All chemical shifts (δ) are reported in ppm and coupling constants (J) in Hz. High resolution mass spectra (HRMS) of the products were obtained using a Bruker Daltonics micro TOF-Q spectrometer. Melting points were measured on a melting point apparatus equipped with a thermometer and were uncorrected. All reactions were monitored by thin-layer chromatography (TLC) through GF254 silica gel-coated plates. Flash chromatography was carried out on SiO_2 (silica gel 200-300 mesh). 3-Phenylbenzothiophenes were prepared by the reported literatures.[1]

2. Experimental procedure for the electrochemical oxidative C-H/C-H coupling of 3-phenylbenzothiophenes and *p*-methoxyphenol

a) In an oven-dried undivided three-necked bottle (25 mL) equipped with a stir bar, 3-phenylbenzothiophene (158 mg, 0.75 mmol), *p*-methoxyphenol (62 mg, 0.5 mmol), and *n*-Bu_4NPF_6 (290 mg, 0.75 mmol) were combined and added. The bottle was equipped with RVC anode (15 mm×15 mm×0.3 mm) as the anode and platinum plate (15 mm×15 mm×0.3 mm) as the cathode and then charged with nitrogen. Then HFIP (6 mL) and CH_2Cl_2 (4 mL) were injected into undivided three-necked bottle. The reaction mixture was stirred and electrolyzed at constant current of 10 mA under room temperature for 5 h. Upon the reaction completed, the reaction mixture was washed with water and extracted with CH_2Cl_2 (10 mL×3). The organic layers were combined, dried over Na_2SO_4, and concentrated under vacuum. The pure product was obtained by flash column chromatography on silica gel by petroleum ether and EtOAc (30:1) in 80% yield.

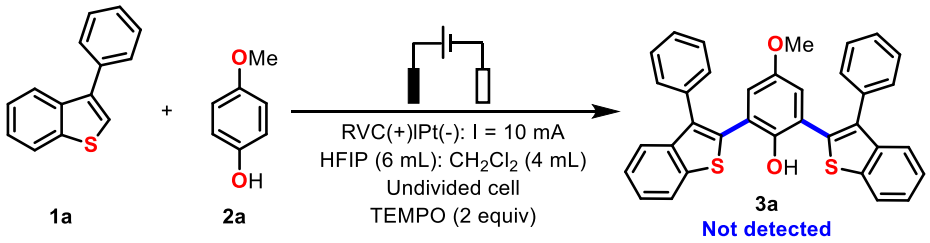
b) In an oven-dried undivided three-necked bottle (25 mL) equipped with a stir bar, 3-phenylbenzothiophene (63 mg, 0.3 mmol), *p*-methoxyphenol (124 mg, 1 mmol), and *n*-Bu_4NPF_6 (387 mg, 1 mmol) were combined and added. The bottle was equipped RVC anode (15 mm×15 mm×0.3 mm) as the anode and platinum plate (15 mm×15 mm×0.3 mm) as the cathode and then charged with nitrogen. Then HFIP (6 mL) and CH_2Cl_2 (4 mL) were injected into undivided three-necked bottle. The reaction mixture was stirred and electrolyzed at constant current of 10 mA under room temperature for 3 h. Upon the reaction completed, the reaction mixture was washed with water and extracted with CH_2Cl_2 (10 mL×3). The organic layers were combined, dried over Na_2SO_4, and concentrated. The pure product was obtained by flash column chromatography on silica gel by petroleum ether and EtOAc (30:1) in 66% yield.

c) In an oven-dried undivided three-necked bottle (25 mL) equipped with a stir bar, 3-phenylbenzothiophene (95 mg, 0.45 mmol), 4-methoxy-2-(3-(*p*-tolyl)benzo[*b*]thiophen-2-yl)phenol (104 mg, 0.3 mmol), and *n*-Bu_4NPF_6 (387 mg, 1 mmol) were combined and added. The bottle was equipped

RVC anode (15 mm×15 mm×0.3 mm) as the anode and platinum plate (15 mm×15 mm×0.3 mm) as the cathode and then charged with nitrogen. Then HFIP (6 mL) and CH_2Cl_2 (4 mL) were injected into undivided three-necked bottle. The reaction mixture was stirred and electrolyzed at constant current of 10 mA under room temperature for 3 h. Upon the reaction completed, the reaction mixture was washed with water and extracted with CH_2Cl_2 (10 mL×3). The organic layers were combined, dried over Na_2SO_4, and concentrated. The pure product was obtained by flash column chromatography on silica gel by petroleum ether and EtOAc (30:1) in 70% yield.

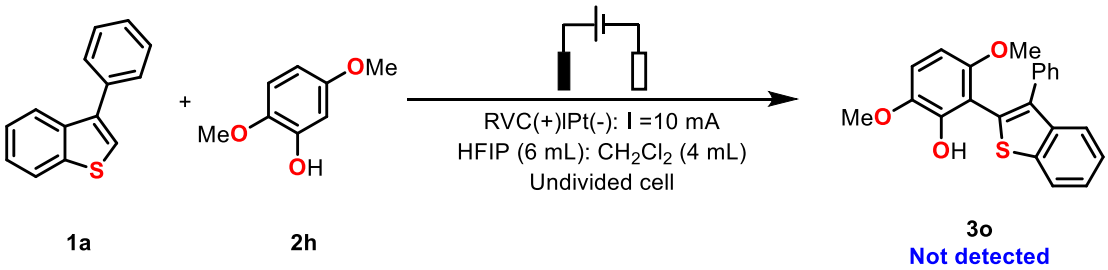
3. Experiments on investigation of mechanism

a) In an oven-dried undivided three-necked bottle (25 mL) equipped with a stir bar, 3-phenylbenzothiophene (158 mg, 0.75 mmol), *p*-methoxyphenol (62 mg, 0.5 mmol), TEMPO (156 mg, 1 mmol) and *n*-Bu_4NPF_6 (290 mg, 0.75 mmol) were combined and added. The bottle was equipped RVC anode (15 mm×15 mm×0.3 mm) as the anode and platinum plate (15 mm×15 mm×0.3 mm) as the cathode and then charged with nitrogen. Then HFIP (6 mL) and CH_2Cl_2 (4 mL) were injected into undivided three-necked bottle. Upon the reaction completed, the reaction mixture was stirred and electrolyzed at constant current of 10 mA under room temperature for 5 h. The reaction mixture was washed with water and extracted with CH_2Cl_2 (10 mL×3). The organic layers were combined, dried over Na_2SO_4, and concentrated. No desired product was observed in the reaction mixture (Scheme 1).



Scheme 1

b) In an oven-dried undivided three-necked bottle (25 mL) equipped with a stir bar, 3-phenylbenzothiophene (63 mg, 0.3 mmol), 2,5-methoxyphenol (154 mg, 1 mmol), and *n*-Bu_4NPF_6 (387 mg, 1 mmol) were combined and added. The bottle was equipped RVC anode (15 mm×15 mm×0.3 mm) as the anode and platinum plate (15 mm×15 mm×0.3 mm) as the cathode and then charged with nitrogen. Then HFIP (6 mL) and CH_2Cl_2 (4 mL) were injected into undivided three-necked bottle. The reaction mixture was stirred and electrolyzed at constant current of 10 mA under room temperature for 3 h. Upon the reaction completed, the reaction mixture was washed with water and extracted with CH_2Cl_2 (10 mL×3). The organic layers were combined, dried over Na_2SO_4, and concentrated. No desired product was observed (Scheme 2).



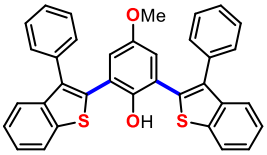
1a **2h** **3o**

Not detected

Scheme 2

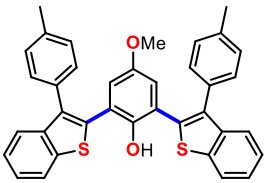
4. Characterization data for the products

4-Methoxy-2,6-bis(3-phenylbenzo[*b*]thiophen-2-yl)phenol (3a)



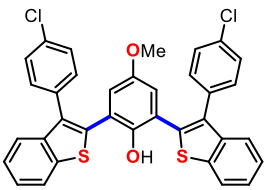
The title compound was prepared according to the general procedure and purified by flash column chromatography to give the white solid, 80% yield; m.p. 159-161 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87-7.85 (m, 2H), 7.72-7.70 (m, 2H), 7.39-7.33 (m, 10H), 7.27-7.24 (m, 4H), 6.72 (d, J = 1.2 Hz, 2H), 5.03 (br, 1H), 3.50 (s, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 152.3, 145.4, 139.9, 139.5, 135.8, 134.7, 134.4, 129.9, 128.6, 127.6, 124.9, 124.6, 123.4, 122.3, 121.8, 118.2, 55.6. HRMS, calculated for $C_{35}H_{24}NaO_2S_2$ ($M+Na^+$): 563.1110, found 563.1110.

4-Methoxy-2,6-bis(3-(*p*-tolyl)benzo[*b*]thiophen-2-yl)phenol (3b)



The title compound was prepared according to the general procedure and purified by flash column chromatography to give the white solid, 47% yield; m.p. 181-183 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88-7.86 (m, 2H), 7.75-7.72 (m, 2H), 7.38-7.36 (m, 4H), 7.21-7.16 (m, 8H), 6.74 (s, 2H), 5.04 (br, 1H), 3.53 (s, 3H), 2.41 (s, 6H); ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 152.3, 145.5, 139.9, 139.5, 137.2, 135.6, 134.2, 131.7, 129.7, 129.3, 124.8, 124.5, 123.4, 122.3, 122.0, 118.1, 55.6, 21.4. HRMS, calculated for $C_{37}H_{28}NaO_2S_2$ ($M+Na^+$): 591.1423, found 591.1426.

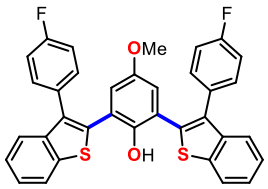
2,6-Bis(3-(4-chlorophenyl)benzo[*b*]thiophen-2-yl)-4-methoxyphenol (3c)



The title compound was prepared according to the general procedure and purified by

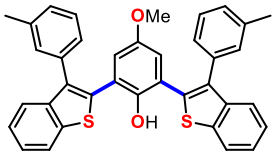
flash column chromatography to give the white solid, mg, 60% yield; m.p. 120-121 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90-7.87 (m, 2H), 7.68-7.65 (m, 2H), 7.41-7.36 (m, 8H), 7.20 (d, J = 8.0 Hz, 4H), 6.77 (s, 2H), 4.98 (br, 1H), 3.61 (s, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 152.5, 145.3, 139.9, 139.2, 134.7, 134.5, 133.6, 133.2, 131.1, 128.8, 125.1, 124.8, 123.2, 122.4, 121.5, 118.2, 55.7. HRMS, calculated for $C_{35}H_{22}Cl_2NaO_2S_2$ ($M+Na^+$): 631.0330, found 631.0334.

2,6-Bis(3-(4-fluorophenyl)benzo[*b*]thiophen-2-yl)-4-methoxyphenol (3d)



The title compound was prepared according to the general procedure and purified by flash column chromatography to give the white solid, 62% yield; m.p. 141-143 ^{o}C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.90 (d, J = 6.0 Hz, 2H), 7.69 (d, J = 6.0 Hz, 2H), 7.42-7.38 (m, 4H), 7.27-7.24 (m, 4H), 7.08 (t, J = 6.0 Hz, 4H), 6.80 (s, 2H), 4.99 (br, 1H), 3.62 (s, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 162.2 (d, J = 247.0 Hz), 152.5, 145.3, 139.8, 139.4, 134.8, 134.4, 131.5 (d, J = 10.1 Hz), 130.7 (d, J = 10.1 Hz), 125.0, 124.8, 123.2, 122.4, 121.6, 118.2, 115.6 (d, J = 20.2 Hz), 55.7. HRMS, calculated for $C_{36}H_{24}F_2NaO_2S_2$ ($M+Na^+$): 613.1078, found 613.1062.

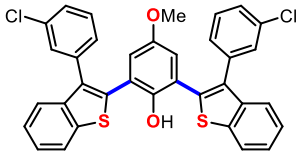
4-Methoxy-2,6-bis(3-(*m*-tolyl)benzo[*b*]thiophen-2-yl)phenol (3e)



The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red liquid, 36% yield. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.87 (d, J = 6.0 Hz, 2H), 7.73 (d, J = 6.0 Hz, 2H), 7.39-7.35 (m, 4H), 7.24 (t, J = 6.0 Hz, 2H), 7.20 (s, 2H), 7.16 (d, J = 6.0 Hz, 2H), 7.04 (d, J = 12.0 Hz, 2H), 6.76 (d, J = 1.2 Hz, 2H), 5.07 (br, 1H), 3.51 (s, 3H), 2.36 (s, 6H); ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 152.3, 145.4, 139.9, 139.6, 138.2, 135.7, 134.6, 134.4, 130.4, 128.6,

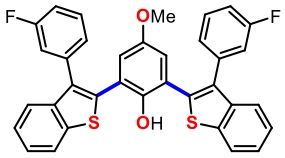
128.5, 127.0, 124.8, 124.5, 123.5, 122.2, 122.0, 118.2, 55.6, 21.6. HRMS, calculated for $C_{37}H_{29}O_2S_2$ ($M+H^+$): 569.1603, found 569.1603.

2,6-Bis(3-(3-chlorophenyl)benzo[*b*]thiophen-2-yl)-4-methoxyphenol (3f)



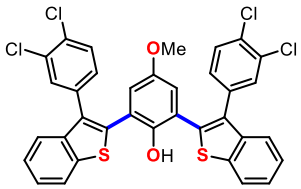
The title compound was prepared according to the general procedure and purified by flash column chromatography to give the white solid, 36% yield; m.p. 74-76 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.79-7.77 (m, 2H), 7.64-7.63 (m, 2H), 7.34-7.29 (m, 6H), 7.26 (d, J = 6.0 Hz, 2H), 7.23-7.20 (m, 2H), 7.07-7.05 (m, 2H), 6.73 (d, J = 4.8 Hz, 2H), 5.07 (br, 1H), 3.49 (s, 3H); ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 152.6, 145.4, 139.9, 139.2, 136.7, 136.7, 135.0, 135.0, 134.4, 134.4, 130.0, 129.7, 128.2, 127.9, 125.2, 124.9, 123.2, 122.4, 121.6, 121.5, 118.4, 66.0, 55.8. HRMS, calculated for $C_{35}H_{22}Cl_2NaO_2S_2$ ($M+Na^+$): 631.0330, found 631.0335.

2,6-Bis(3-(3-fluorophenyl)benzo[*b*]thiophen-2-yl)-4-methoxyphenol (3g)



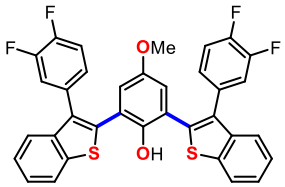
The title compound was prepared according to the general procedure and purified by flash column chromatography to give the white solid, 43% yield; m.p. 127-128 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91-7.88 (m, 2H), 7.74-7.72 (m, 2H), 7.43-7.39 (m, 4H), 7.38-7.32 (m, 2H), 7.08-7.02 (m, 6H), 6.78 (s, 2H), 5.04 (br, 1H), 3.58 (s, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 162.8 (d, J = 247.0 Hz), 152.5, 145.3, 139.9, 139.1, 136.9 (d, J = 10.1 Hz), 134.9, 134.5, 130.2 (d, J = 10.1 Hz), 125.7, 125.0 (d, J = 30.3 Hz), 123.2, 122.4, 121.5, 118.3, 116.7 (d, J = 22.2 Hz), 114.7 (d, J = 21.2 Hz), 55.7. HRMS, calculated for $C_{35}H_{22}F_2NaO_2S_2$ ($M+Na^+$): 599.0921, found 599.0925.

2,6-Bis(3-(3,4-dichlorophenyl)benzo[*b*]thiophen-2-yl)-4-methoxyphenol (3h)



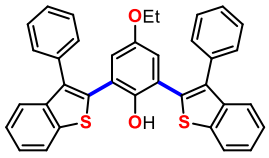
The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red solid, 33% yield; m.p. 86-87 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.90-7.89 (m, 2H), 7.67-7.66 (m, 2H), 7.48 (d, J = 6.0 Hz, 2H), 7.44-7.40 (m, 6H), 7.05 (dd, J = 8.4, 2.4 Hz, 2H), 6.76 (s, 2H), 5.02 (br, 1H), 3.62 (s, 3H); ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 152.6, 145.2, 139.8, 138.9, 135.0, 134.8, 133.5, 132.7, 131.8, 131.4, 130.7, 129.1, 125.3, 125.0, 123.0, 122.5, 121.2, 118.3, 55.7. HRMS, calculated for $C_{35}H_{20}Cl_4NaO_2S_2$ ($M+Na^+$): 698.9551, found 698.9553.

2,6-Bis(3-(3,4-difluorophenyl)benzo[*b*]thiophen-2-yl)-4-methoxyphenol (3i)



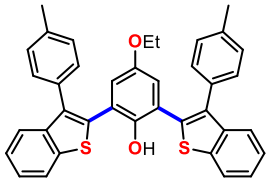
The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red solid, 33% yield; m.p. 125-127 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92-7.88 (m, 2H), 7.71-7.67 (m, 2H), 7.45-7.40 (m, 4H), 7.22-7.12 (m, 4H), 7.03-6.99 (m, 2H), 6.82 (s, 2H), 5.00 (br, 1H), 3.66 (s, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 152.7, 150.3 (dd, J = 252.5, 10.1 Hz), 149.9 (dd, J = 252.5, 10.1 Hz), 145.2, 139.8, 139.1, 134.8, 133.8, 131.7 (d, J = 5.1 Hz), 126.0 (q, J = 3.0 Hz), 125.1 (d, J = 20.2 Hz), 123.0, 122.4, 121.4, 118.7 (d, J = 20.2 Hz), 118.3, 117.6 (d, J = 10.1 Hz), 55.7. HRMS, calculated for $C_{35}H_{20}F_4NaO_2S_2$ ($M+Na^+$): 635.0733, found 635.0736.

4-Ethoxy-2,6-bis(3-phenylbenzo[*b*]thiophen-2-yl)phenol (3j)



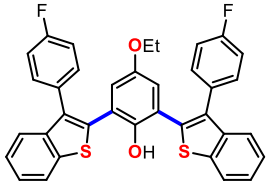
The title compound was prepared according to the general procedure and purified by flash column chromatography to give the white solid, 50% yield; m.p. 82-83 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87-7.85 (m, 2H), 7.72-7.69 (m, 2H), 7.38-7.32 (m, 10H), 7.26-7.23 (m, 4H), 6.73 (s, 2H), 5.01 (br, 1H), 3.69 (q, J = 6.7 Hz, 2H), 1.21 (t, J = 8.0 Hz, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 151.6, 145.4, 139.9, 139.5, 135.7, 134.7, 134.5, 129.8, 128.6, 127.6, 124.8, 124.5, 123.4, 122.3, 121.8, 119.0, 64.0, 14.7. HRMS, calculated for $C_{36}H_{26}NaO_2S_2$ ($M+Na^+$): 577.1226, found 577.1230.

4-Ethoxy-2,6-bis(3-(*p*-tolyl)benzo[*b*]thiophen-2-yl)phenol (3k)



The title compound was prepared according to the general procedure and purified by flash column chromatography to give the white solid, 41% yield; m.p. 213-215 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90-7.88 (m, 2H), 7.77-7.75 (m, 2H), 7.40-7.37 (m, 4H), 7.23-7.19 (m, 8H), 7.78 (t, J = 2.0 Hz,2H), 5.06 (br, 1H), 3.75 (q, J = 8.0 Hz, 2H), 2.43 (s, 6H), 1.28-1.24 (t, J = 8.0 Hz, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 151.6, 145.4, 140.0, 139.6, 137.2, 135.6, 134.3, 131.7, 129.7, 129.4, 124.8, 124.5, 123.4, 122.3, 122.0, 119.0, 64.0, 21.4, 14.7. HRMS, calculated for $C_{38}H_{30}NaO_2S_2$ ($M+Na^+$): 605.1579, found 605.1585.

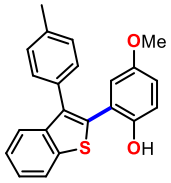
4-Ethoxy-2,6-bis(3-(4-fluorophenyl)benzo[*b*]thiophen-2-yl)phenol (3l)



The title compound was prepared according to the general procedure and purified by flash column chromatography to give the white solid, 40% yield; m.p. 171-173 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90-7.88 (m, 2H), 7.68-7.66 (m, 2H), 7.42-7.37 (m, 4H), 7.26-7.22 (m, 4H), 7.10-7.04 (m, 4H), 6.79 (s, 2H), 4.96 (br, 1H), 3.81 (q, J = 8.0 Hz, 2H), 1.31 (t, J = 8.0 Hz, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 162.2 (d, J = 248.0

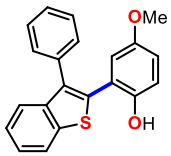
Hz), 151.7, 145.2, 139.8, 139.4, 134.8, 134.4, 131.5 (d, J = 10.1 Hz), 130.7 (d, J = 10.1 Hz), 124.9 (d, J = 30.3 Hz), 123.2, 122.4, 121.6, 118.9, 115.7, 115.6 (d, J = 20.2 Hz), 64.1, 14.7. HRMS, calculated for $C_{36}H_{24}F_2NaO_2S_2$ ($M+Na^+$): 613.1078, found 613. 1082.

4-Methoxy-2-(3-(*p*-tolyl)benzo[*b*]thiophen-2-yl)phenol (4a)



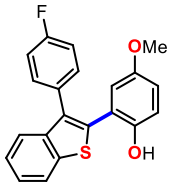
The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red liquid, 72% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89-7.87 (m, 1H), 7.77-7.75 (m, 1H), 7.39-7.37 (m, 2H), 7.25 (d, J = 8.0 Hz, 2H), 7.18 (d, J = 8.0 Hz, 2H), 6.88 (d, J = 4.0 Hz, 1H), 6.82-6.75 (m, 2H), 4.90 (br, 1H), 3.71 (s, 3H), 2.36 (s, 3H); ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 153.3, 147.1, 139.9, 139.7, 137.8, 135.3, 134.1, 131.2, 129.7, 129.5, 125.0, 124.8, 123.5, 122.3, 120.8, 117.2, 116.4, 116.4, 55.8, 21.4. HRMS, calculated for $C_{22}H_{18}NaO_2S$ ($M+Na^+$): 369.0920, found 369.0911.

4-Methoxy-2-(3-phenylbenzo[*b*]thiophen-2-yl)phenol (4b)



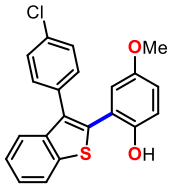
The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red liquid, 66% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93-7.90 (m, 1H), 7.79-7.77 (m, 1H), 7.43-7.36 (m, 7H), 6.87-6.84 (m, 1H), 6.81-6.77 (m, 2H), 4.89 (br, 1H), 3.71 (s, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 153.2, 147.1, 139.9, 139.6, 135.5, 134.4, 134.3, 129.7, 128.9, 128.0, 125.0, 124.8, 123.5, 122.3, 120.6, 117.1, 116.5, 116.4, 55.8. HRMS, calculated for$C_{21}H_{16}NaO_2S$ ($M+Na^+$): 355.0763, found 355.0769.

2-(3-(4-Fluorophenyl)benzo[*b*]thiophen-2-yl)-4-methoxyphenol (4c)



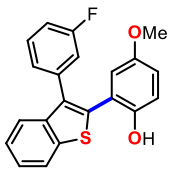
The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red liquid, 64% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92-7.90 (m, 1H), 7.73-7.71 (m, 1H), 7.43-7.41 (m, 2H), 7.35-7.31 (m, 2H), 7.08 (td, J = 8.0, 4.0 Hz, 2H), 6.84-6.77 (m, 3H), 4.82 (br, 1H), 3.72 (s, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 162.4 (d, J =248 Hz), 153.3, 147.1, 139.9, 139.5, 134.5, 134.4, 131.4 (d, J =10.1 Hz), 130.3 (d, J =10.1 Hz), 125.0 (d, J =20.2 Hz), 123.2, 122.4, 120.3, 117.0, 116.5 (d, J =10.1 Hz), 115.9 (d, J =20.2 Hz), 55.8. HRMS, calculated for $C_{21}H_{15}FNaO_2S$ ($M+Na^+$): 373.0670, found 373.0674.

2-(3-(4-Chlorophenyl)benzo[*b*]thiophen-2-yl)-4-methoxyphenol (4d)



The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red liquid, 50% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91-7.88 (m, 1H), 7.72-7.69 (m, 1H), 7.42-7.39 (m, 2H), 7.35-7.32 (m, 2H), 7.29-7.25 (m, 2H), 6.83-6.76 (m, 3H), 4.83 (br, 1H), 3.70 (s, 3H); ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 153.3, 147.1, 139.9, 139.3, 134.6, 134.4, 133.8, 132.8, 131.0, 129.1, 125.2, 125.0, 123.2, 122.4, 120.2, 117.0, 116.5, 116.4, 55.8. HRMS, calculated for $C_{21}H_{15}ClNaO_2S$ ($M+Na^+$): 389.0373, found 389.0375.

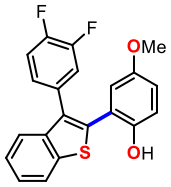
2-(3-(3-Fluorophenyl)benzo[*b*]thiophen-2-yl)-4-methoxyphenol (4e)



The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red liquid, 62% yield. ^{1}H NMR (400 MHz,

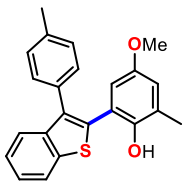
$CDCl_3$) δ 7.92-7.90 (m, 1H), 7.76-7.74 (m, 1H), 7.44-7.41 (m, 2H), 7.37-7.32 (m, 1H), 7.13 (d, J = 8.0 Hz, 1H), 7.09-7.02 (m, 2H), 6.84-6.78 (m, 3H), 4.85 (br, 1H), 3.71 (s, 3H); $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ 162.9 (d, J =246.0 Hz), 153.3, 147.2, 139.8, 139.2, 136.5 (d, J =7.6 Hz), 134.9, 134.3, 130.3 (d, J =9.1 Hz), 125.5 (d, J =3.0 Hz), 125.2, 125.0, 123.2, 122.4, 120.2, 117.0, 116.7 (d, J =10.6 Hz), 116.5, 116.3, 114.9 (d, J =15.1 Hz), 55.8. HRMS, calculated for $C_{21}H_{15}FNaO_2S$ ($M+Na^+$): 373.0670, found 373. 0675.

2-(3-(3,4-Difluorophenyl)benzo[*b*]thiophen-2-yl)-4-methoxyphenol (4f)



The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red liquid, 33% yield. ^1H NMR (400 MHz, $CDCl_3$) δ 7.92-7.90 (m, 1H), 7.72-7.70 (m, 1H), 7.44-7.42 (m, 2H), 7.20-7.13 (m, 2H), 7.09-7.05 (m, 1H), 6.86-6.78 (m, 3H), 4.84 (br, 1H), 3.72 (s, 3H); $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ 153.3, 150.4 (dd, J = 241.6, 15.1 Hz), 150.0 (dd, J = 241.6, 15.1 Hz), 147.1, 139.8, 139.1, 135.0, 133.5, 131.3 (d, J = 4.5 Hz), 126.0 (d, J = 3.0 Hz), 125.2 (d, J = 30.2 Hz), 123.0, 122.5, 120.0, 118.7 (d, J = 15.1 Hz), 117.6 (d, J = 15.1 Hz), 117.0, 116.5 (d, J = 28.7 Hz), 55.8. HRMS, calculated for $C_{21}H_{14}F_2NaO_2S$ ($M+Na^+$): 391.0575, found 391.0578.

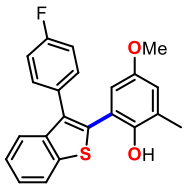
4-Methoxy-2-methyl-6-(3-(*p*-tolyl)benzo[*b*]thiophen-2-yl)phenol (4g)



The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red liquid, 87% yield. ^1H NMR (400 MHz, $CDCl_3$) δ 7.76-7.74 (m, 1H), 7.66-7.64 (m, 1H), 7.27-7.25 (m, 2H), 7.15-7.04 (m, 4H), 6.59 (s, 2H), 4.82 (br, 1H), 3.56 (s, 3H), 2.24 (s, 3H), 2.03 (s, 3H); $^{13}C\{^1H\}$ NMR

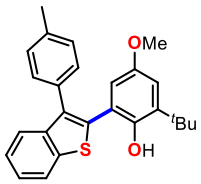
(151 MHz, $CDCl_3$) δ 152.6, 145.5, 139.9, 139.8, 137.7, 135.4, 134.5, 131.2, 129.7, 129.5, 126.4, 125.0, 124.7, 123.6, 122.3, 120.0, 118.0, 113.7, 55.7, 21.4, 16.6. HRMS, calculated for $C_{23}H_{20}NaO_2S$ ($M+Na^+$): 383.1076, found 383.1082.

2-(3-(4-Fluorophenyl)benzo[*b*]thiophen-2-yl)-4-methoxy-6-methylphenol (4h)



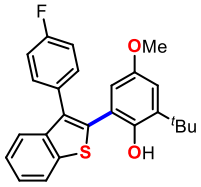
The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red liquid, 41% yield. ^1H NMR (600 MHz, $CDCl_3$) δ 7.92-7.90 (m, 1H), 7.74 (dd, J = 7.8, 1.8 Hz, 1H), 7.45-7.41 (m, 2H), 7.35-7.33 (m, 2H), 7.09-7.06 (m, 2H), 6.73 (d, J = 3.0 Hz, 1H), 6.67 (d, J = 3.0 Hz, 1H), 4.90 (br, 1H), 3.70 (s, 3H), 2.18 (s, 3H); $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ 162.3 (d, J = 248.0 Hz), 152.6, 145.5, 139.9, 139.5, 134.8, 134.5, 131.4 (d, J = 9.1 Hz), 130.3 (d, J = 3.0 Hz), 126.3, 125.0 (d, J = 30.2 Hz), 123.3, 122.4, 119.5, 118.1, 115.9, 115.8, 113.6, 55.7, 16.5. HRMS, calculated for $C_{22}H_{17}FNaO_2S$ ($M+Na^+$): 387.0826, found 387.0830.

2-(*tert*-Butyl)-4-methoxy-6-(3-(*p*-tolyl)benzo[*b*]thiophen-2-yl)phenol (4i)



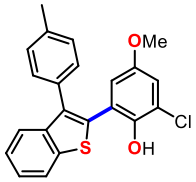
The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red liquid, 89% yield. ^1H NMR (400 MHz, $CDCl_3$) δ 7.92-7.90 (m, 1H), 7.84 (t, J = 4.0 Hz, 1H), 7.42-7.40 (m, 2H), 7.25 (d, J = 8.0 Hz, 2H), 7.18 (d, J = 8.0 Hz, 2H), 6.92 (d, J = 4.0 Hz, 1H), 6.78 (dd, J = 2.0, 1.0 Hz, 1H), 5.08 (br, 1H), 3.75 (s, 3H), 2.37 (s, 3H), 1.29 (s, 9H); $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 152.5, 145.9, 140.1, 139.5, 138.3, 137.7, 135.5, 134.8, 131.1, 129.5, 129.3, 125.0, 124.8, 123.5, 122.4, 120.8, 115.3, 112.6, 55.7, 35.0, 29.2, 21.3. HRMS, calculated for $C_{26}H_{26}NaO_2S$ ($M+Na^+$): 425.1546, found 425.1543.

2-(*tert*-Butyl)-6-(3-(4-fluorophenyl)benzo[*b*]thiophen-2-yl)-4-methoxyphenol (4j)



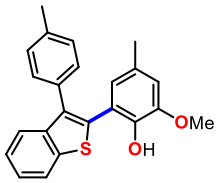
The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red liquid, 79% yield. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.93-7.92 (m, 1H), 7.77-7.76 (m, 1H), 7.44-7.42 (m, 2H), 7.32-7.30 (m, 2H), 7.06 (t, J = 6.0 Hz, 2H), 6.91 (d, J = 6.0 Hz, 1H), 6.73 (d, J = 3.0 Hz, 1H), 5.04 (br, 1H), 3.75 (s, 3H), 1.28 (s, 9H); ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 162.4 (d, J = 248.0 Hz), 152.5, 145.8, 140.0, 139.3, 138.3, 135.1, 134.6, 131.2 (d, J = 7.6 Hz), 130.1 (d, J = 4.5 Hz), 125.1, 124.9, 123.2, 122.5, 120.2, 115.8 (d, J = 21.1 Hz), 115.4, 112.5, 55.7, 35.0, 29.2. HRMS, calculated for $C_{25}H_{23}FNaO_2S$ ($M+Na^+$): 429.1295, found 429.1298.

2-Chloro-4-methoxy-6-(3-(*p*-tolyl)benzo[*b*]thiophen-2-yl)phenol (4k)



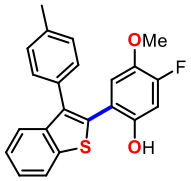
The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red liquid, 56% yield. ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.06 (br, 1H), 8.03 (d, J = 8.0 Hz, 1H), 7.57 (d, J = 8.0 Hz, 1H), 7.45-7.37 (m, 2H), 7.21 (s, 4H), 6.97 (d, J = 4.0 Hz, 1H), 6.59 (d, J = 4.0 Hz, 1H), 3.54 (s, 3H), 2.32 (s, 3H); ^{13}C{^{1}H} NMR (151 MHz, DMSO-d_6) δ 152.1, 145.4, 139.6, 139.5, 137.1, 132.2, 130.1, 129.6, 125.3, 125.0, 124.5, 123.3, 122.8, 122.6, 117.0, 115.8, 56.0, 21.3. HRMS, calculated for $C_{22}H_{17}ClNaO_2S$ ($M+Na^+$): 403.0530, found 403.0524.

2-Methoxy-4-methyl-6-(3-(*p*-tolyl)benzo[*b*]thiophen-2-yl)phenol (4l)



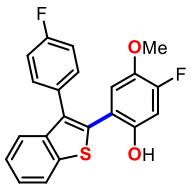
The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red liquid, 63% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85-7.83 (m, 1H), 7.71-7.69 (m, 1H), 7.33-7.30 (m, 2H), 7.25 (d, J = 8.0 Hz, 2H), 7.12 (d, J = 8.0 Hz, 2H), 6.62 (d, J = 12 Hz, 2H), 5.50 (br, 1H), 3.79 (s, 3H), 2.33 (s, 3H), 2.18 (s, 3H); ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 146.6, 141.5, 140.0, 139.9, 136.7, 135.1, 134.8, 132.6, 129.8, 129.1, 128.8, 124.4, 124.4, 124.2, 123.4, 122.1, 120.1, 111.8, 56.0, 21.4, 21.1. HRMS, calculated for $C_{23}H_{20}NaO_2S$ ($M+Na^+$): 383.1076, found 383.1067.

5-Fluoro-4-methoxy-2-(3-(*p*-tolyl)benzo[*b*]thiophen-2-yl)phenol (4m)



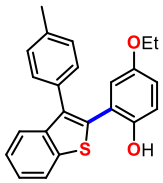
The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red liquid, 74% yield. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.90 (dd, J = 6.0, 3.0 Hz, 1H), 7.77 (d, J = 6.0, 3.0 Hz, 1H), 7.43-7.40 (m, 2H), 7.26-7.21 (m, 4H), 6.91 (d, J = 6.0 Hz, 1H), 6.64 (d, J = 12.0 Hz, 1H), 5.08 (br, 1H), 3.77 (s, 3H), 2.39 (s, 3H); ^{13}C{^{1}H} NMR (151 MHz, $CDCl_3$) δ 153.3 (d, J = 248.0 Hz), 147.4 (d, J = 15.1 Hz), 141.6 (d, J = 15.1 Hz), 139.7 (d, J = 30.2 Hz), 138.0, 135.7, 133.0, 131.0, 129.8, 129.4, 125.1, 124.9, 123.6, 122.3, 116.8, 115.3 (d, J = 4.5 Hz), 105.2 (d, J = 15.1 Hz), 57.0, 21.4. HRMS, calculated for $C_{22}H_{17}FNaO_2S$ ($M+Na^+$): 387.0826, found 387.0832.

5-Fluoro-2-(3-(4-fluorophenyl)benzo[*b*]thiophen-2-yl)-4-methoxyphenol (4n)



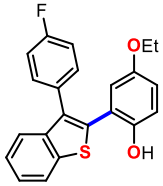
The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red liquid, 71% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93-7.89 (m, 1H), 7.73-7.71 (m, 1H), 7.44-7.41 (m, 2H), 7.33-7.29 (m, 2H), 7.09 (t, J = 10.0 Hz, 2H), 6.84 (d, J = 8.0 Hz, 1H), 6.66 (d, J = 12.0 Hz, 1H), 5.17 (br, 1H), 3.76 (s, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 162.4 (d, J = 247.0 Hz), 153.5 (d, J = 248.0 Hz), 147.6 (d, J = 10.1 Hz), 141.7 (d, J = 10.1 Hz), 139.6 (d, J = 20.2 Hz), 134.9, 133.4, 131.4 (d, J = 10.1 Hz), 130.2 (d, J = 10.1 Hz), 125.3, 125.0, 123.3, 122.4, 116.9 (d, J = 3.0 Hz), 116.0 (d, J = 20.2 Hz), 115.0 (d, J = 10.1 Hz), 105.1 (d, J = 20.2 Hz), 57.1. HRMS, calculated for $C_{21}H_{14}F_2NaO_2S$ ($M+Na^+$): 391.0575, found 391.0580.

4-Ethoxy-2-(3-(*p*-tolyl)benzo[*b*]thiophen-2-yl)phenol (4o)



The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red liquid, 51% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89-7.86 (m, 1H), 7.77-7.75 (m, 1H), 7.39-7.37 (m, 2H), 7.25 (d, J = 8.0 Hz, 2H), 7.18 (d, J = 8.0 Hz, 2H), 6.89 (d, J = 4.0 Hz, 1H), 6.82-6.73 (m, 2H), 4.89 (br, 1H), 3.92 (q, J = 8.0 Hz, 2H), 2.36 (s, 3H), 1.36 (t, J = 4.0 Hz, 3H); ^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$) δ 152.6, 147.1, 140.0, 139.7, 137.8, 135.3, 134.2, 131.2, 129.7, 129.5, 124.9, 124.7, 123.5, 122.3, 120.8, 117.3, 117.2, 117.1, 64.1, 21.4, 14.9. HRMS, calculated for $C_{23}H_{20}NaO_2S$ ($M+Na^+$): 383.1076, found 383.1079.

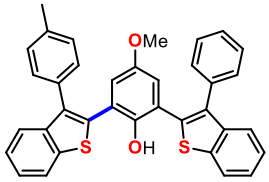
4-Ethoxy-2-(3-(4-fluorophenyl)benzo[*b*]thiophen-2-yl)phenol (4p)



The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red liquid, 65% yield. ^{1}H NMR (400 MHz,

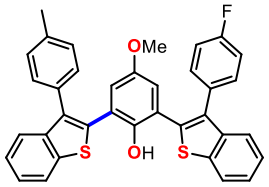
$CDCl_3$) δ 7.93-7.89 (m, 1H), 7.74-7.71 (m, 1H), 7.43-7.41 (m, 2H), 7.35-7.31 (m, 2H), 7.09-7.05 (m, 2H), 6.83-6.76 (m, 3H), 4.86 (br, 1H), 3.93 (q, J = 6.7 Hz, 2H), 1.37 (t, J = 8.0 Hz, 3H); $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 162.4 (d, J = 246.0 Hz), 152.6, 147.1, 139.9, 139.5, 134.5, 131.4, 131.3, 130.3 (d, J = 3.0 Hz), 125.1, 124.9, 123.2, 122.4, 120.3, 117.3 (d, J = 10.1 Hz), 117.0, 115.9 (d, J = 20.2 Hz), 64.1, 14.8. HRMS, calculated for $C_{22}H_{17}FNaO_2S$ ($M+Na^+$): 387.0826, found 387.0831.

4-Methoxy-2-(3-phenylbenzo[*b*]thiophen-2-yl)-6-(3-(*p*-tolyl)benzo[*b*]thiophen-2-yl)phenol (5a)



The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red solid, 70% yield; m.p. 147-148 °C. ^1H NMR (600 MHz, $CDCl_3$) δ 7.85-7.83 (m, 2H), 7.72-7.70 (m, 2H), 7.36-7.32 (m, 8H), 7.20-7.15 (m, 5H), 6.75-6.70 (m, 2H), 5.03 (br, 1H), 3.49 (s, 3H), 2.38 (s, 3H); $^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$) δ 152.4, 145.5, 140.0, 139.6, 139.5, 137.3, 135.8, 135.7, 134.8, 134.7, 134.1, 131.6, 129.9, 129.7, 129.5, 128.6, 127.6, 124.9, 124.6, 123.5, 123.4, 122.3, 122.0, 121.9, 118.2, 118.2, 55.6, 21.4. HRMS, calculated for $C_{36}H_{26}NaO_2S_2$ ($M+Na^+$): 577.1266, found 577.1257.

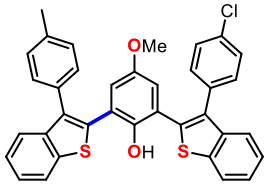
2-(3-(4-Fluorophenyl)benzo[*b*]thiophen-2-yl)-4-methoxy-6-(3-(*p*-tolyl)benzo[*b*]thiophen-2-yl)phenol (5b)



The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red solid, 74% yield; m.p. 127-129 °C. ^1H NMR (600 MHz, $CDCl_3$) δ 7.90-7.87 (m, 2H), 7.76-7.75 (m, 1H), 7.71-7.69 (m, 1H), 7.41-7.37 (m, 4H), 7.26-7.20 (m, 6H), 7.07 (t, J = 9.0 Hz, 2H), 6.85 (d, J = 3.6 Hz, 1H), 6.76 (d, J = 3.6 Hz, 1H), 5.07 (br, 1H), 3.59 (s, 3H), 2.44 (s, 3H); $^{13}C\{^1H\}$ NMR

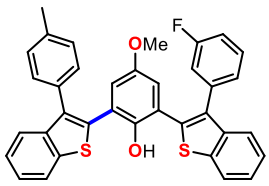
(151 MHz, $CDCl_3$) δ 162.1 (d, J = 248.0 Hz), 152.4, 145.5, 139.9 (d, J = 4.5 Hz), 139.7, 139.4, 137.5, 135.9, 134.9, 134.6, 133.8, 131.6, 131.5, 130.9 (d, J = 3.0 Hz), 129.7, 129.5, 125.0, 124.9, 124.7, 123.6, 123.2, 122.4, 122.3, 121.9, 121.8, 118.3, 118.2, 115.6 (d, J = 15.1 Hz), 55.7, 21.4. HRMS, calculated for $C_{36}H_{25}FNaO_2S_2$ ($M+Na^+$): 595.1172, found 595.1172.

2-(3-(4-Chlorophenyl)benzo[*b*]thiophen-2-yl)-4-methoxy-6-(3-(*p*-tolyl)benzo[*b*]thiophen-2-yl)phenol (5c)



The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red solid, 33% yield; m.p. 162-165 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 7.83-7.79 (m, 2H), 7.69-7.67 (m, 1H), 7.63-7.60 (m, 1H), 7.33-7.26 (m, 6H), 7.17-7.11 (m, 6H), 6.77 (d, J = 4.0 Hz, 1H), 6.66 (d, J = 4.0 Hz, 1H), 4.99 (br, 1H), 3.51 (s, 3H), 2.37 (s, 3H); $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 152.5, 145.5, 139.9, 139.7, 139.2, 137.5, 136.0, 135.2, 134.5, 133.7, 133.5, 133.4, 131.5, 131.2, 129.7, 129.5, 128.8, 125.0, 124.9, 124.7, 123.6, 123.1, 122.4, 122.3, 121.9, 121.7, 118.3, 118.2, 55.7, 21.4. HRMS, calculated for $C_{36}H_{25}ClNaO_2S_2$ ($M+Na^+$): 611.0877, found 611.0872.

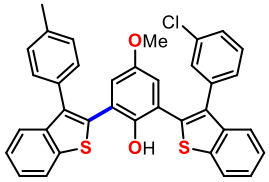
2-(3-(3-Fluorophenyl)benzo[*b*]thiophen-2-yl)-4-methoxy-6-(3-(*p*-tolyl)benzo[*b*]thiophen-2-yl)phenol (5d)



The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red solid, 67% yield; m.p. 122-123 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 7.83-7.80 (m, 2H), 7.73 (t, J = 4.0 Hz, 1H), 7.69-7.66 (m, 1H), 7.34-7.27 (m, 5H), 7.20-7.15 (m, 4H), 7.05-6.95 (m, 3H), 6.79 (d, J = 4.0 Hz, 1H), 6.69 (d, J = 3.2 Hz, 1H), 5.06 (br, 1H), 3.49 (s, 3H), 2.36 (s, 3H); $^{13}C\{^1H\}$ NMR

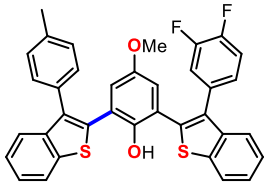
(101 MHz, $CDCl_3$) δ 162.9 (d, J = 247.0 Hz), 152.6, 145.5, 140.0 (d, J = 5.1 Hz), 139.6, 139.2, 137.6, 137.3, 137.3, 136.0, 135.6, 134.4, 134.3, 133.8, 131.5, 130.1 (d, J = 9.1 Hz), 129.7, 129.6, 125.8, 125.8, 125.0, 125.0, 124.7, 124.7, 123.6, 123.2, 122.4, 122.1, 121.7, 118.3 (d, J = 4.0 Hz), 116.9 (d, J = 22.2 Hz), 114.5 (d, J = 20.2 Hz), 55.7, 21.4. HRMS, calculated for $C_{36}H_{25}FNaO_2S_2$ ($M+Na^+$): 595.1172, found 595.1165.

2-(3-(3-Chlorophenyl)benzo[*b*]thiophen-2-yl)-4-methoxy-6-(3-(*p*-tolyl)benzo[*b*]thiophen-2-yl)phenol (5e)



The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red liquid, 28% yield. ^1H NMR (400 MHz, $CDCl_3$) δ 7.83-7.80 (m, 2H), 7.73-7.70 (m, 1H), 7.67-7.65 (m, 1H), 7.33-7.26 (m, 5H), 7.18-7.16 (m, 4H), 7.04-6.95 (m, 3H), 6.78 (d, J = 4.0 Hz, 1H), 6.99 (d, J = 4.0 Hz, 1H), 5.04 (br, 1H), 3.48 (s, 3H), 2.36 (s, 3H); $^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$) δ 164.1, 161.7, 152.6, 145.5, 140.0, 139.7, 139.2, 137.6, 137.3, 136.0, 135.6, 134.4, 134.3, 133.8, 131.5, 130.1, 130.0, 129.7, 129.6, 125.8, 125.0, 124.7, 123.6, 123.2, 122.4, 122.1, 121.7, 118.3, 117.0, 116.8, 114.6, 114.4, 55.7, 21.4. HRMS, calculated for $C_{36}H_{25}ClNaO_2S_2$ ($M+Na^+$): 611.0877, found 611.0878.

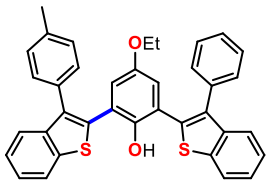
2-(3-(3,4-Difluorophenyl)benzo[*b*]thiophen-2-yl)-4-methoxy-6-(3-(*p*-tolyl)benzo[*b*]thiophen-2-yl)phenol (5f)



The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red liquid, 51% yield. ^1H NMR (400 MHz, $CDCl_3$) δ 7.92-7.87 (m, 2H), 7.79-7.76 (m, 1H), 7.70-7.67 (m, 1H), 7.43-7.39 (m, 4H), 7.26-7.21 (m, 4H), 7.17-7.09 (m, 2H), 7.03-7.00 (m, 1H), 6.89 (d, J = 4.0 Hz, 1H),

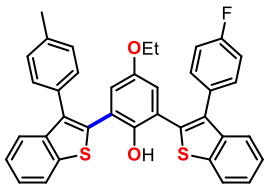
6.76 (d, J = 4.0 Hz, 1H), 5.07 (br, 1H), 3.63 (s, 3H), 2.44 (s, 3H); $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ 152.5, 150.3 (dd, J = 252.2, 10.1 Hz), 149.8 (dd, J = 242.2, 10.1 Hz), 145.4, 139.9 (d, J = 9.1 Hz), 139.7, 139.1, 137.7, 136.0, 135.7, 133.5, 133.4, 132.0 (q, J = 3.4 Hz), 131.3, 129.6 (d, J = 3.0 Hz), 126.1 (d, J = 3.4 Hz), 125.0 (d, J = 7.1 Hz), 124.7 (d, J = 3.0 Hz), 123.6, 122.9, 122.4 (d, J = 5.1 Hz), 122.0, 121.5, 118.7 (d, J = 17.2 Hz), 118.3 (d, J = 16.2 Hz), 117.3 (d, J = 17.2 Hz), 55.7, 21.3. HRMS, calculated for $C_{36}H_{24}F_2NaO_2S_2$ ($M+Na^+$): 613.1078, found 613.1067.

4-Ethoxy-2-(3-phenylbenzo[*b*]thiophen-2-yl)-6-(3-(*p*-tolyl)benzo[*b*]thiophen-2-yl) phenol (5g)



The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red liquid, 54% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82-7.80 (m, 2H), 7.72-7.69 (m, 2H), 7.36-7.30 (m, 7H), 7.25 (dd, J = 8.0, 4.0 Hz, 2H), 7.15-7.13 (m, 4H), 6.76 (d, J = 4.0 Hz, 1H), 6.71 (d, J = 3.2 Hz, 1H), 5.02 (br, 1H), 3.67 (q, J = 6.7 Hz, 2H), 2.36 (s, 3H), 1.19 (t, J = 8.0 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (101 MHz, $CDCl_3$) δ 151.7, 145.5, 140.0, 139.7, 139.6, 137.3, 135.7, 135.7, 134.9, 134.8, 134.2, 131.7, 130.0, 129.8, 129.5, 128.6, 127.6, 124.9, 124.6, 123.5, 123.4, 122.3, 122.0, 122.0, 119.1, 119.1, 64.1, 21.5, 14.8. HRMS, calculated for $C_{37}H_{28}NaO_2S_2$ ($M+Na^+$): 591.1423, found 591.1419.

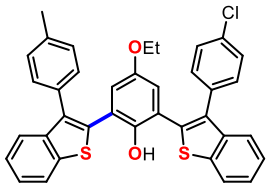
4-Ethoxy-2-(3-(4-fluorophenyl)benzo[*b*]thiophen-2-yl)-6-(3-(*p*-tolyl)benzo[*b*]thiop hen-2-yl)phenol (5h)



The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red liquid, 63% yield. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.90-7.87 (m, 2H), 7.75 (dd, J = 7.2, 1.8 Hz, 1H), 7.70 -7.68 (m, 1H),

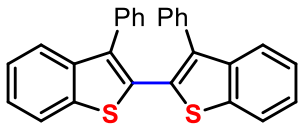
7.42-7.37 (m, 4H), 7.24-7.19 (m, 6H), 7.06 (t, *J* = 6.0 Hz, 2H), 6.84 (d, *J* = 3.0 Hz, 1H), 6.74 (d, *J* = 3.0 Hz, 1H), 5.02 (br, 1H), 3.79 (q, *J* = 6.0 Hz, 2H), 2.43 (s, 3H), 1.29 (t, *J* = 6.0 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (151 MHz, $CDCl_3$) δ 162.1 (d, *J* = 248.0 Hz), 151.7, 145.4, 139.9 (d, *J* = 6.0 Hz), 139.6, 139.4, 137.4, 135.8, 134.9, 134.5, 133.8, 131.5, 131.5, 130.8 (d, *J* = 4.5 Hz), 129.7, 129.4, 124.9, 124.8, 124.6, 123.5, 123.1, 122.3, 122.3, 121.9, 121.7, 119.0, 118.9, 115.5 (d, *J* = 21.1Hz), 64.0, 21.4, 14.7. HRMS, calculated for $C_{37}H_{27}FNaO_2S_2$ ($M+Na^+$): 609.1329, found 609.1330.

2-(3-(4-Chlorophenyl)benzo[*b*]thiophen-2-yl)-4-ethoxy-6-(3-(*p*-tolyl)benzo[*b*]thiophen-2-yl)phenol (5i)



The title compound was prepared according to the general procedure and purified by flash column chromatography to give the red solid, 39% yield; m.p. 174-175 ºC. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (t, *J* = 6.0 Hz, 2H), 7.75-7.67 (m, 2H), 7.41-7.32 (m, 6H), 7.23-7.17 (m, 6H), 6.83 (d, *J* = 2.8 Hz, 1H), 6.72 (d, *J* = 4.0 Hz, 1H), 5.02 (br, 1H), 3.78 (q, *J* = 6.7 Hz, 2H), 2.43 (s, 3H), 1.30-1.27 (t, *J* = 6.0 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (151 MHz, $CDCl_3$) δ 151.7, 145.3, 139.9, 139.6, 139.1, 137.5, 135.9, 135.2, 134.3, 133.7, 133.4, 133.3, 131.4, 131.2, 129.6, 129.4, 128.7, 124.9, 124.9, 124.6, 123.5, 123.0, 122.3, 122.3, 121.8, 121.6, 119.0, 118.9, 64.0, 21.4, 14.7. HRMS, calculated for $C_{37}H_{27}ClNaO_2S_2$ ($M+Na^+$): 625.1033, found 625.1026.

3,3'-Diphenyl-2,2'-bibenzo[*b*]thiophene



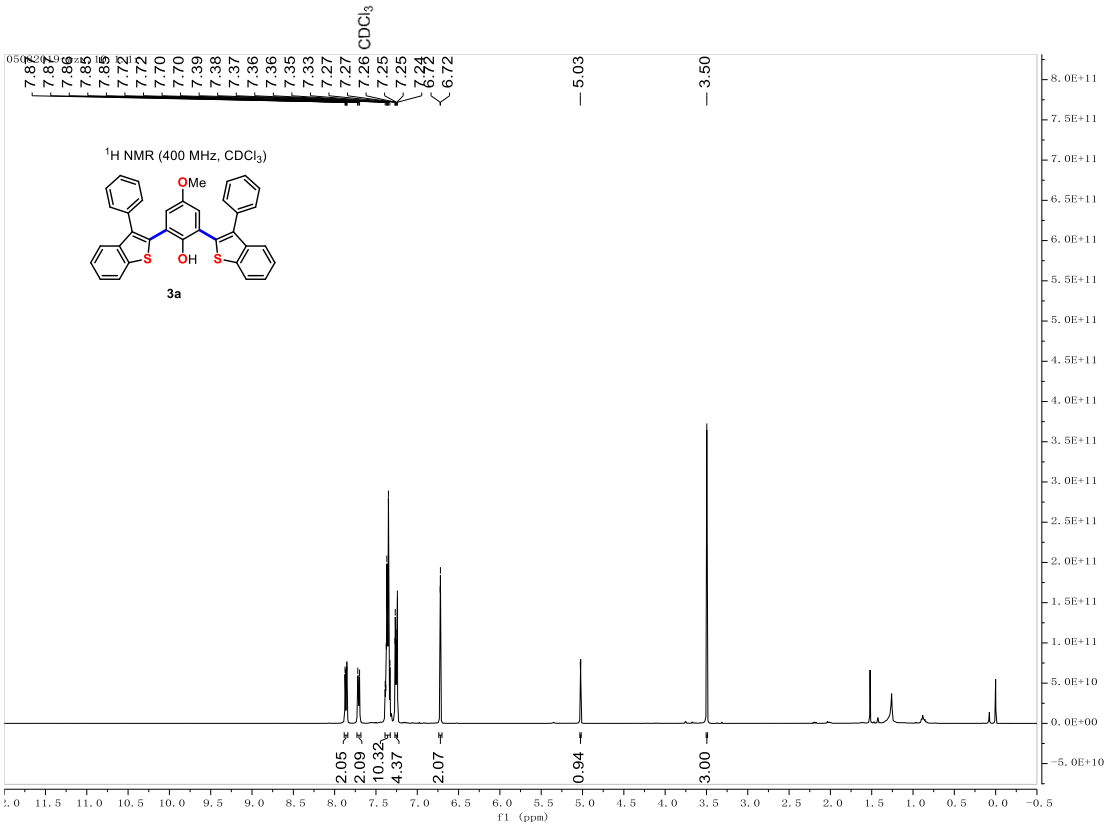
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.81 (d, *J* = 12.0 Hz, 2H), 7.56 (d, *J* = 6.0 Hz, 2H), 7.35 (td, *J* = 7.5, 1.2 Hz, 2H), 7.30 (td, *J* = 7.5, 1.2 Hz, 2H), 7.25-7.20 (m, 6H), 7.04-7.02 (m, 4H); $^{13}C\{^{1}H\}$ NMR (151 MHz, $CDCl_3$) δ 140.3, 139.6, 136.7, 134.7,

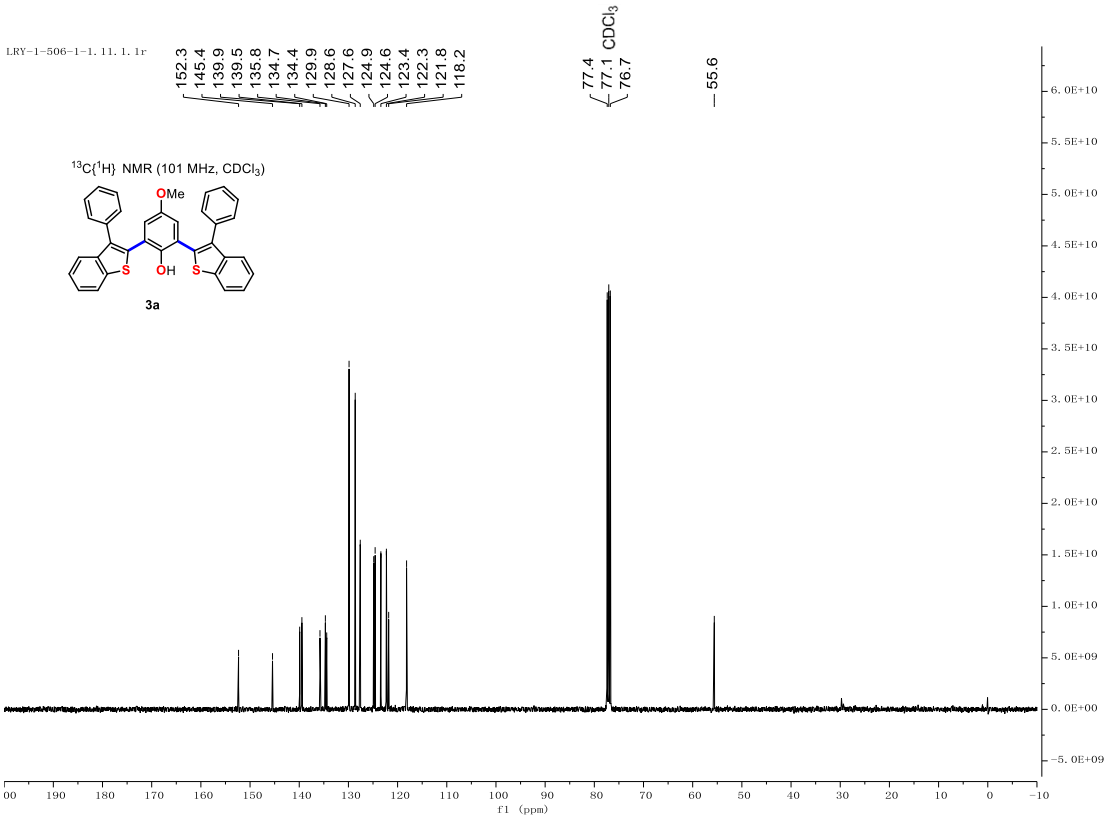
131.7, 129.9, 128.4, 127.1, 124.9, 124.5, 123.6, 122.1. $C_{28}H_{18}NaS_2$ ($M+Na^+$): 441.0742, found 441.0732.

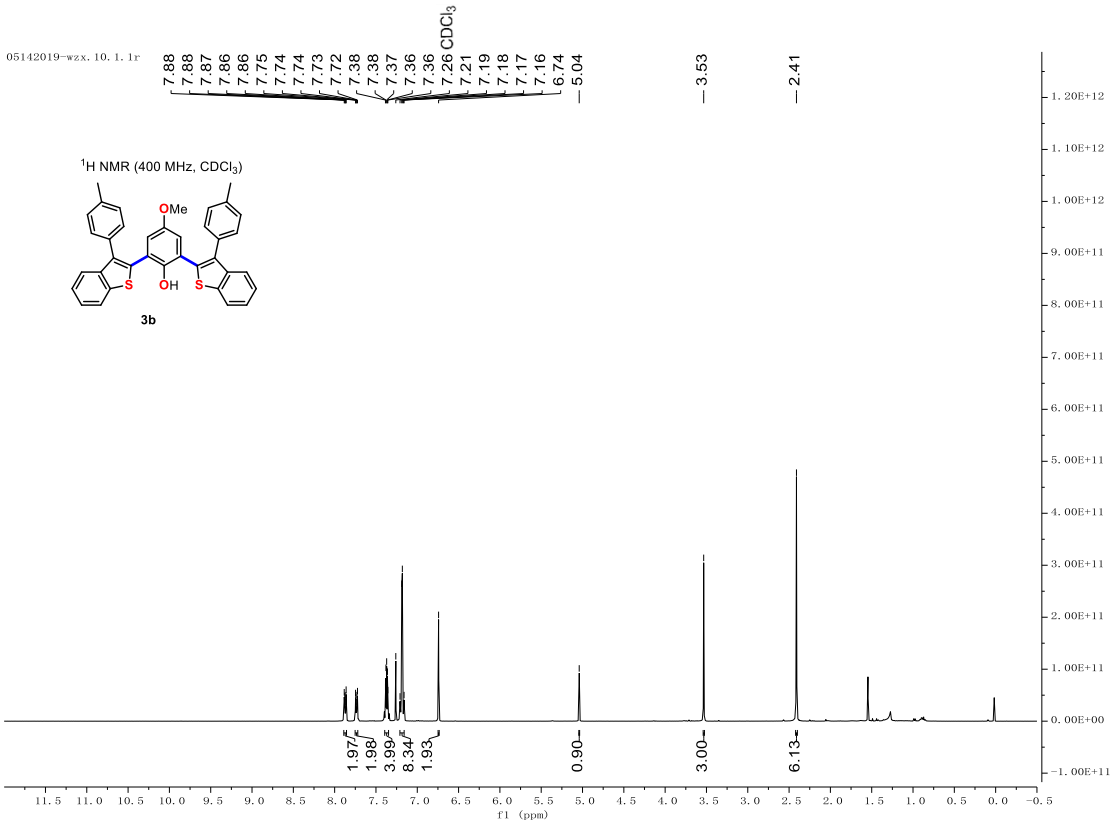
References:

1. Y. Zou, G. Yue, J. Xu, J. (Steve) Zhou, *Eur. J. Org. Chem.* **2014**, 5901-5905.

5. ^{1}H NMR and ^{13}C NMR copies of products






05142019-wzx. 10. 1. 1r
7.88
7.88
7.87
7.86
7.86
7.75
7.74
7.74
7.73
7.72
7.38
7.37
7.36
7.36
7.26 CDCl3
7.21
7.19
7.18
7.17
7.16
6.74
5.04
3.53
2.41
1H NMR (400 MHz, CDCl3)
OMe
OH
S
S
3b
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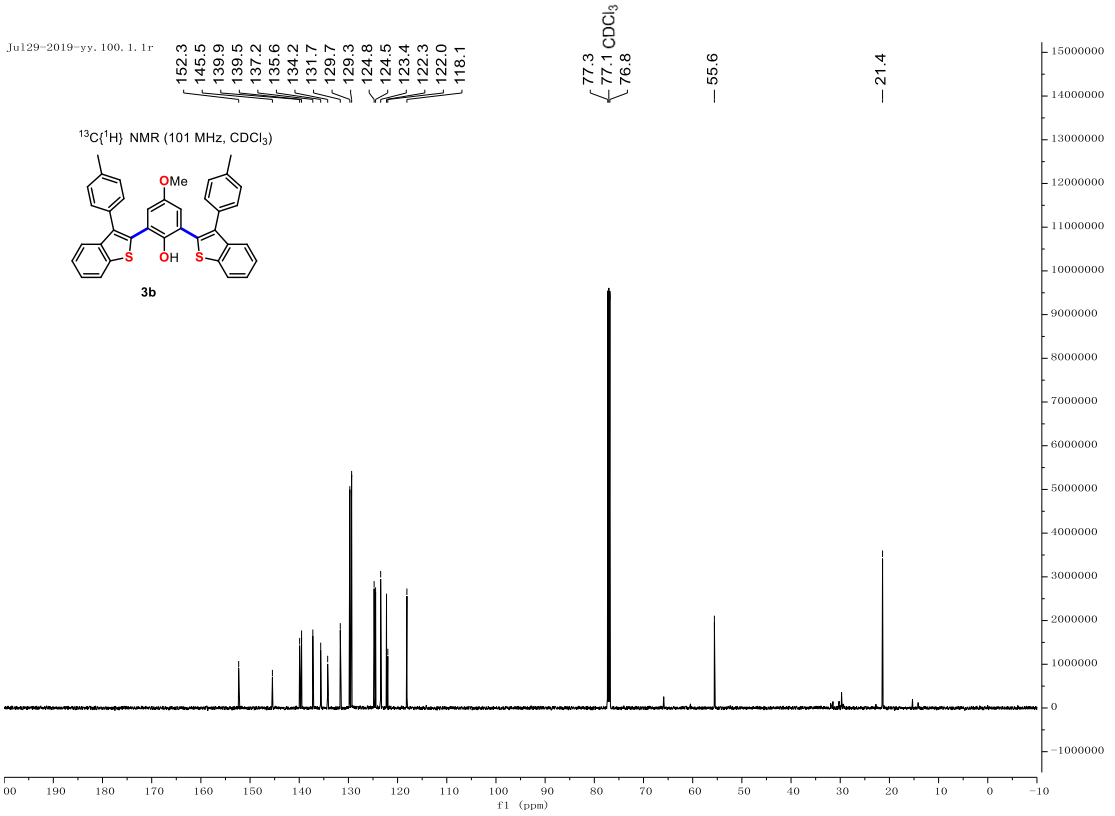
1.98
3.99
8.34
1.93
0.90
3.00
6.13
f1 (ppm)


Jul29-2019-yy. 100. 1. 1r
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139.9
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137.2
135.6
134.2
131.7
129.7
129.3
124.8
124.5
123.4
122.3
122.0
118.1
77.3
77.1 CDCl3
76.8
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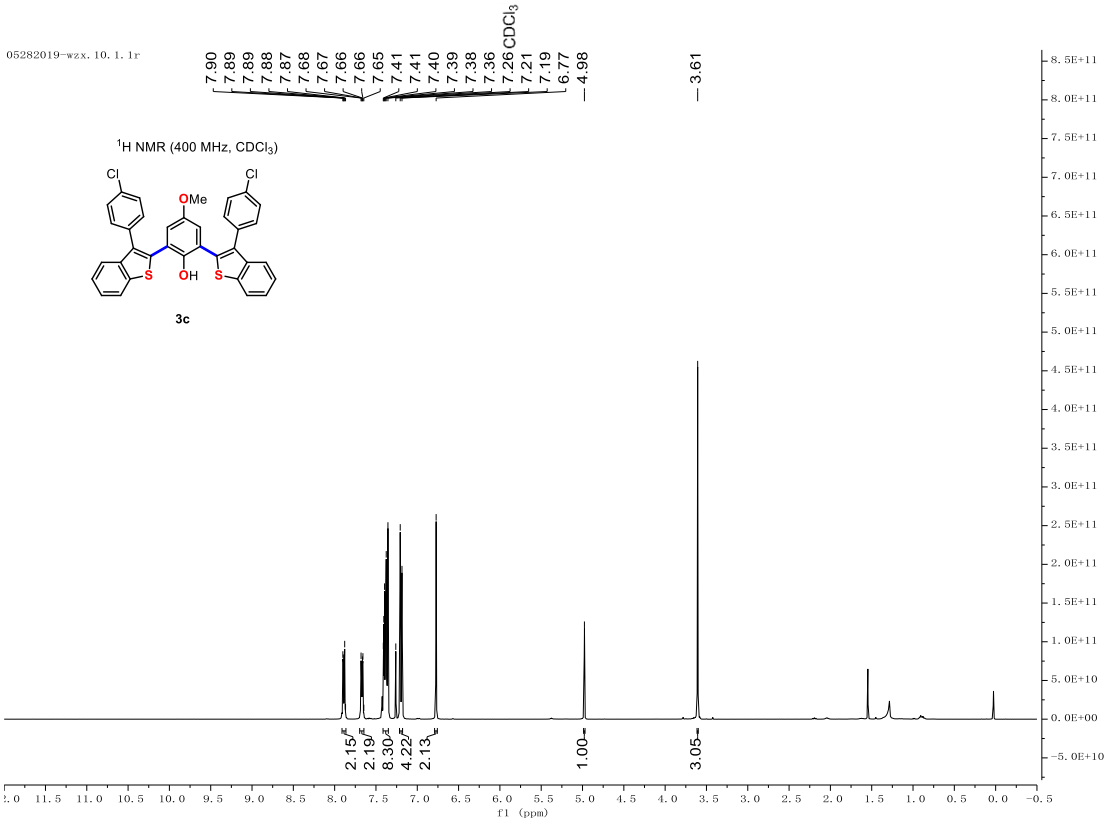
21.4
13C{1H} NMR (101 MHz, CDCl3)
OMe
OH
S
S
3b
f1 (ppm)


05282019-wzx. 10. 1. 1r
7.90
7.89
7.89
7.88
7.87
7.68
7.67
7.66
7.66
7.65
7.41
7.41
7.40
7.39
7.38
7.36
7.26 CDCl3
7.21
7.19
6.77
4.98
3.61
1H NMR (400 MHz, CDCl3)
Cl
Cl
OMe
S
OH
S
3c
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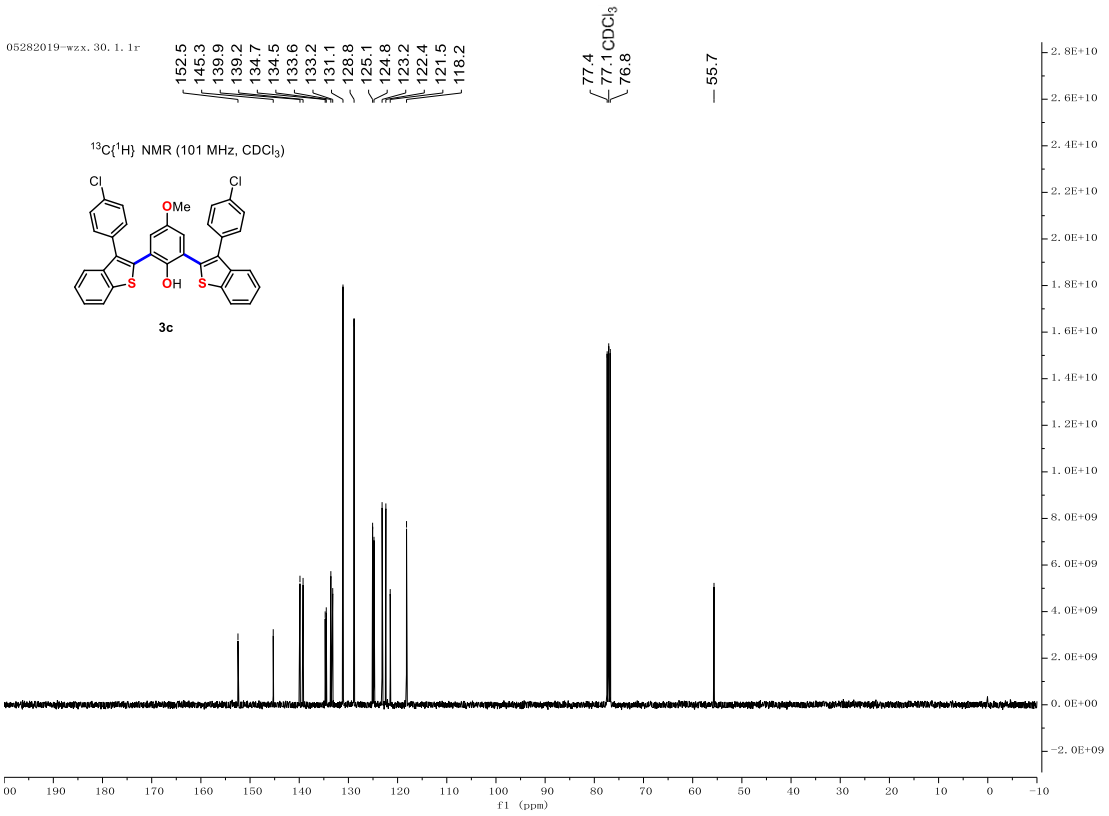
2.19
8.30
4.22
2.13
1.00
3.05
f1 (ppm)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145.3
139.9
139.2
134.7
134.5
133.6
133.2
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128.8
125.1
124.8
123.2
122.4
121.5
118.2
77.4
77.1 CDCl3
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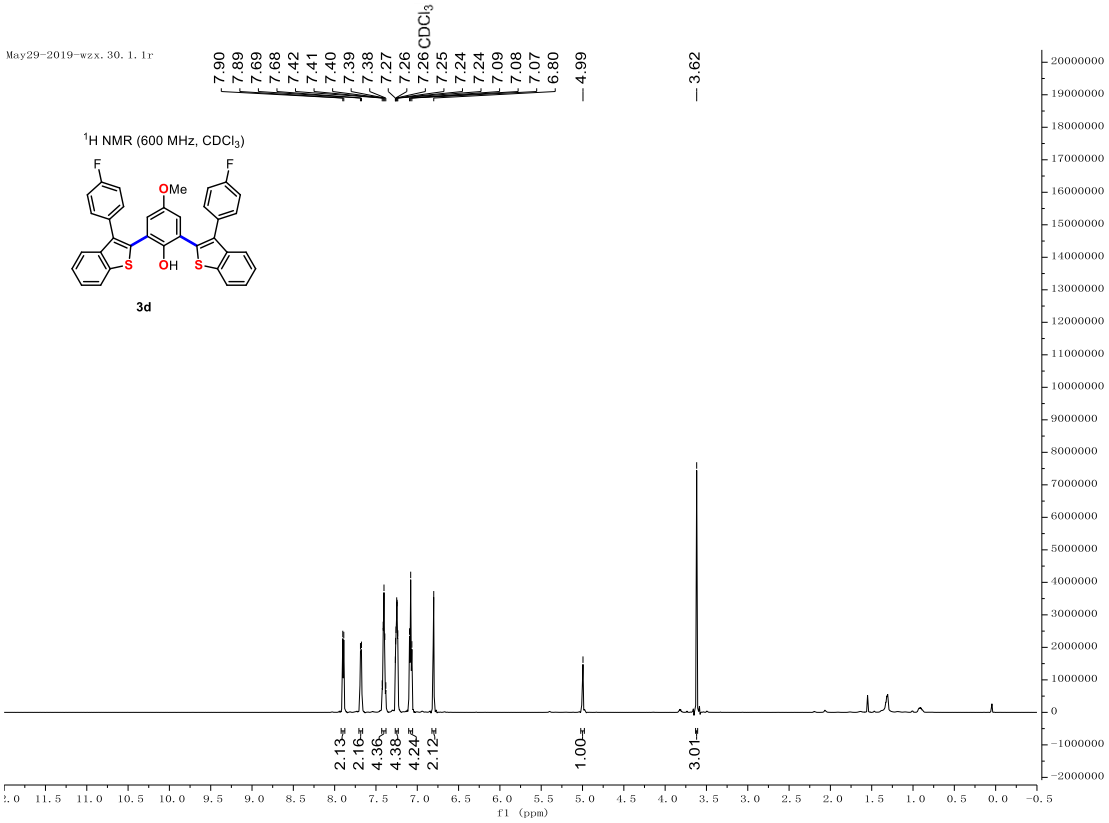
Cl
Cl
OMe
S
OH
S
3c
f1 (ppm)


May29-2019-wzx.30.1.1r
7.90
7.89
7.69
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7.42
7.41
7.40
7.39
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7.26
7.26 CDCl3
7.25
7.24
7.24
7.09
7.08
7.07
6.80
4.99
3.62
1H NMR (600 MHz, CDCl3)
F
OMe
F
S
OH
S
3d
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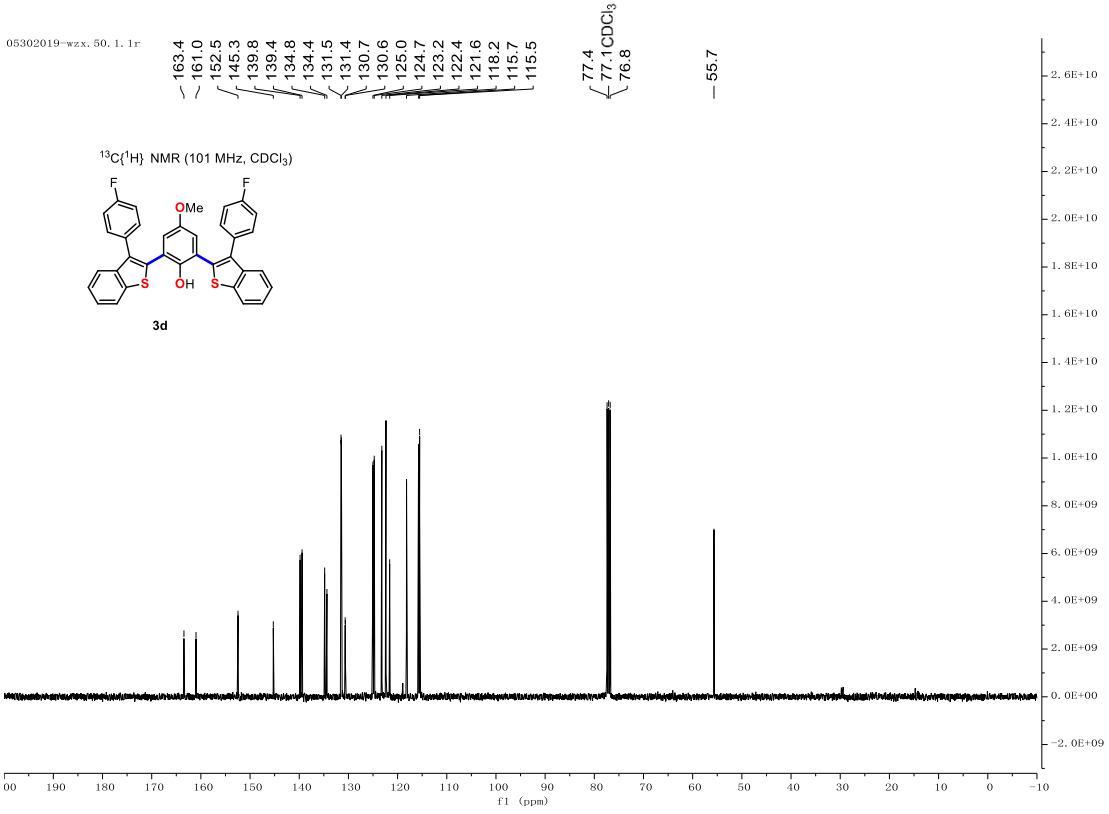
4.24
2.12
1.00
3.01
f1 (ppm)


05302019-wzx.50.1.1r
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152.5
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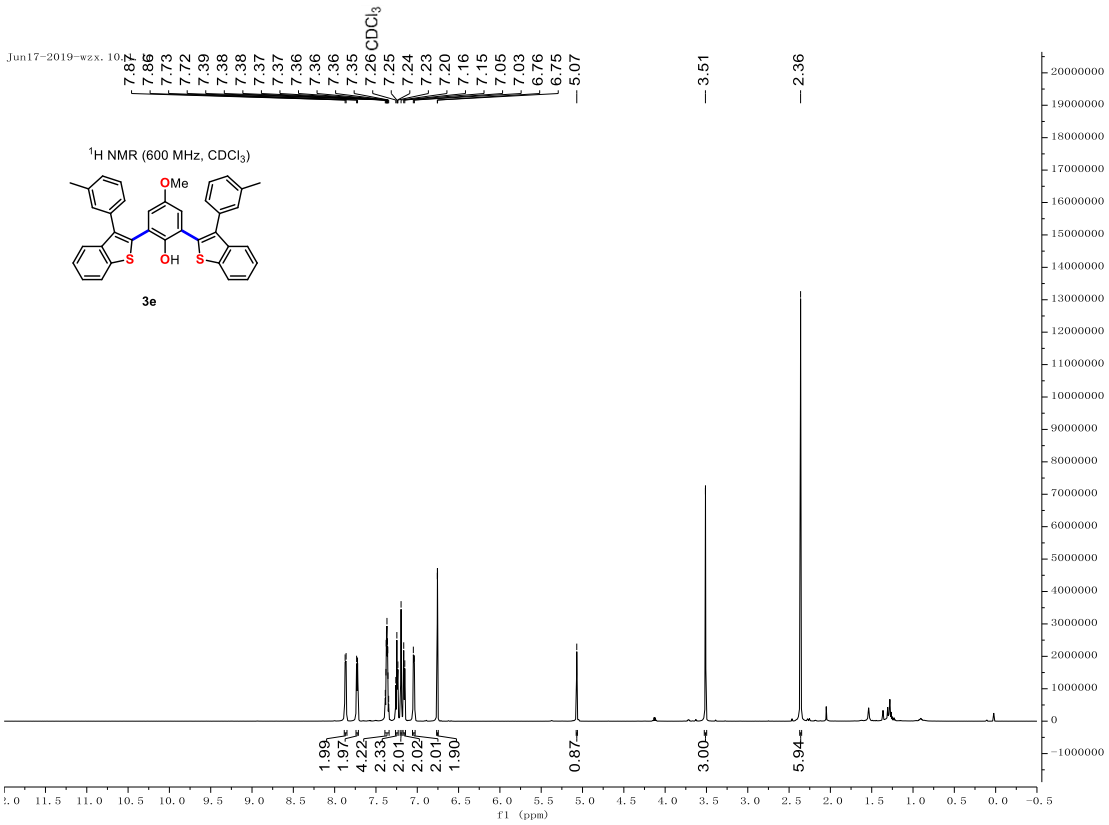
139.8
139.4
134.8
134.4
131.5
131.4
130.7
130.6
125.0
124.7
123.2
122.4
121.6
118.2
115.7
115.5
77.4
77.1 CDCl3
76.8
55.7
13C{1H} NMR (101 MHz, CDCl3)
F
OMe
F
S
OH
S
3d
f1 (ppm)


Jun17-2019-wzx. 10.1r
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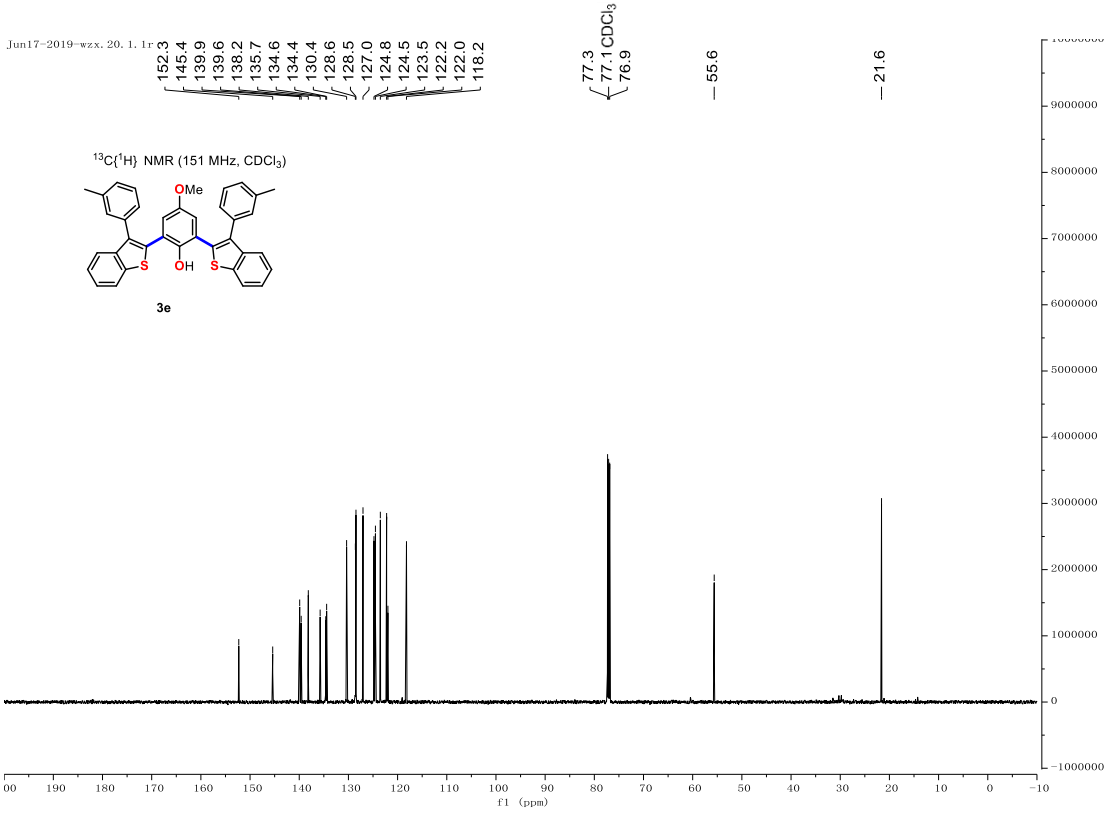
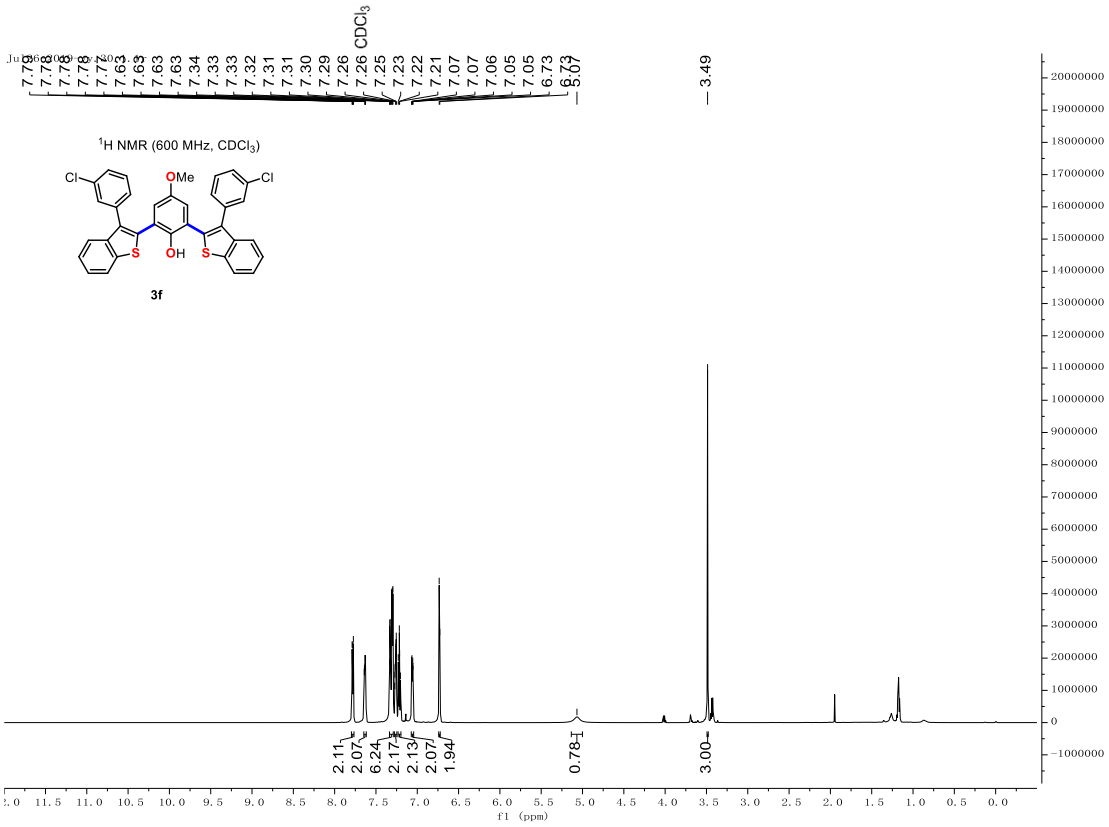
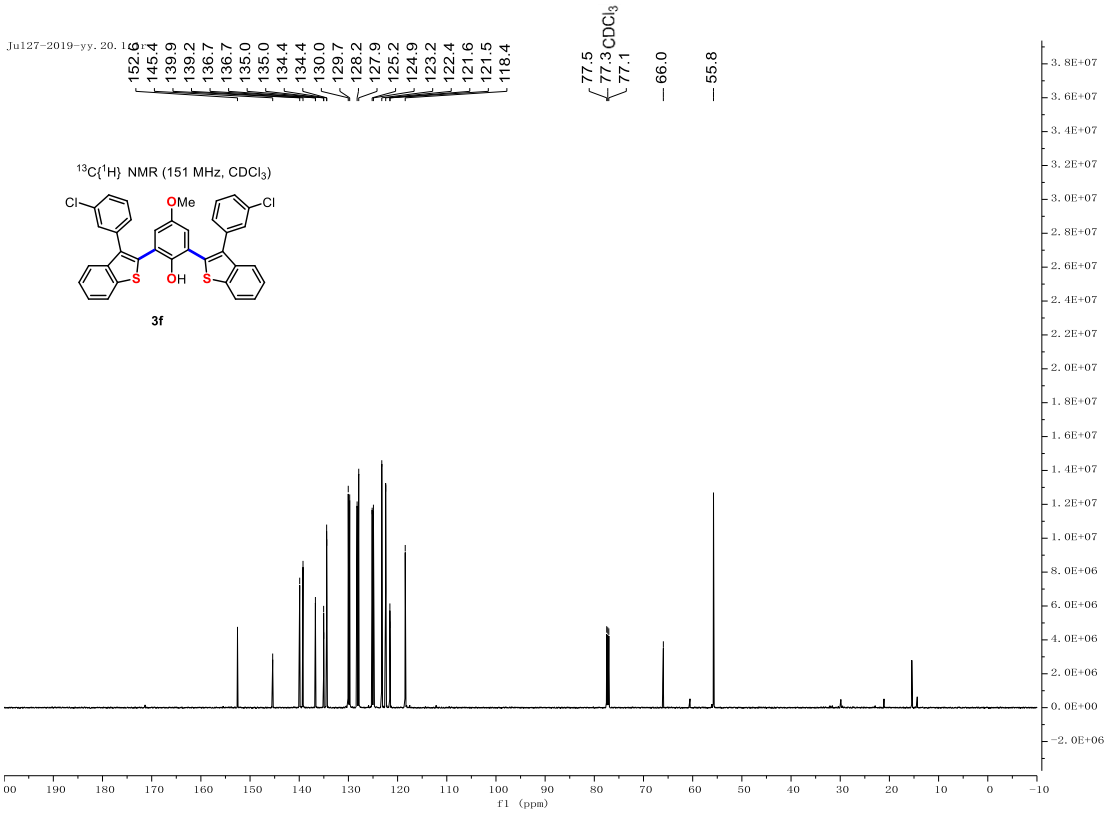
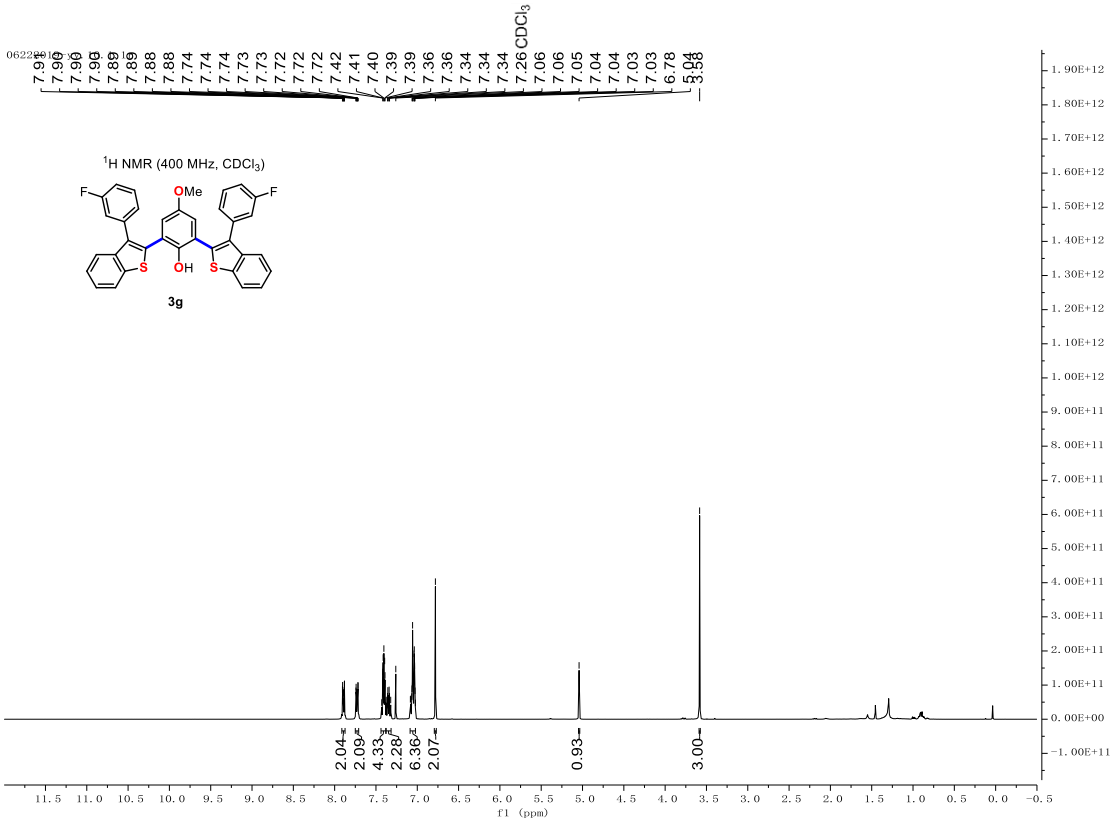
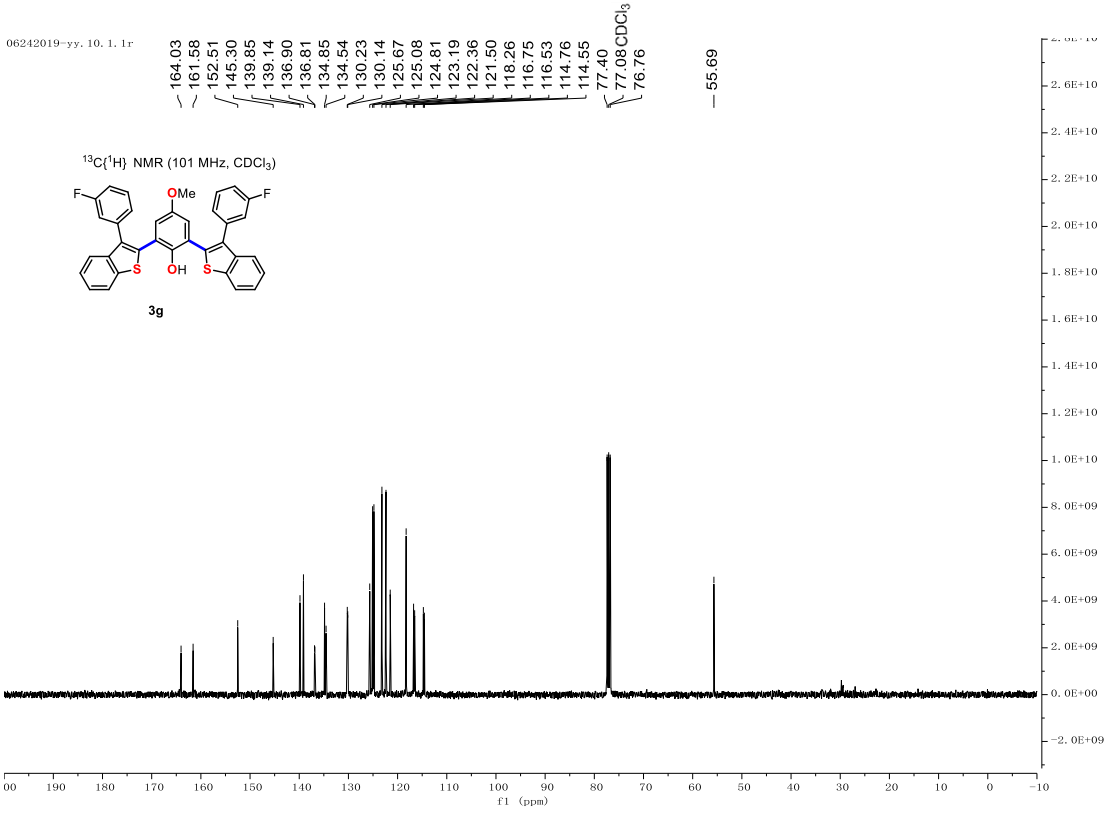
7.86
7.73
7.72
7.39
7.38
7.38
7.37
7.37
7.36
7.36
7.36
7.35
7.26 CDCl3
7.25
7.24
7.23
7.20
7.16
7.15
7.05
7.03
6.76
6.75
5.07
3.51
2.36
1H NMR (600 MHz, CDCl3)
OMe
OH
S
S
3e
1.99
1.97
4.22
2.33
2.01
2.02
2.01
1.90
0.87
3.00
5.94
f1 (ppm)

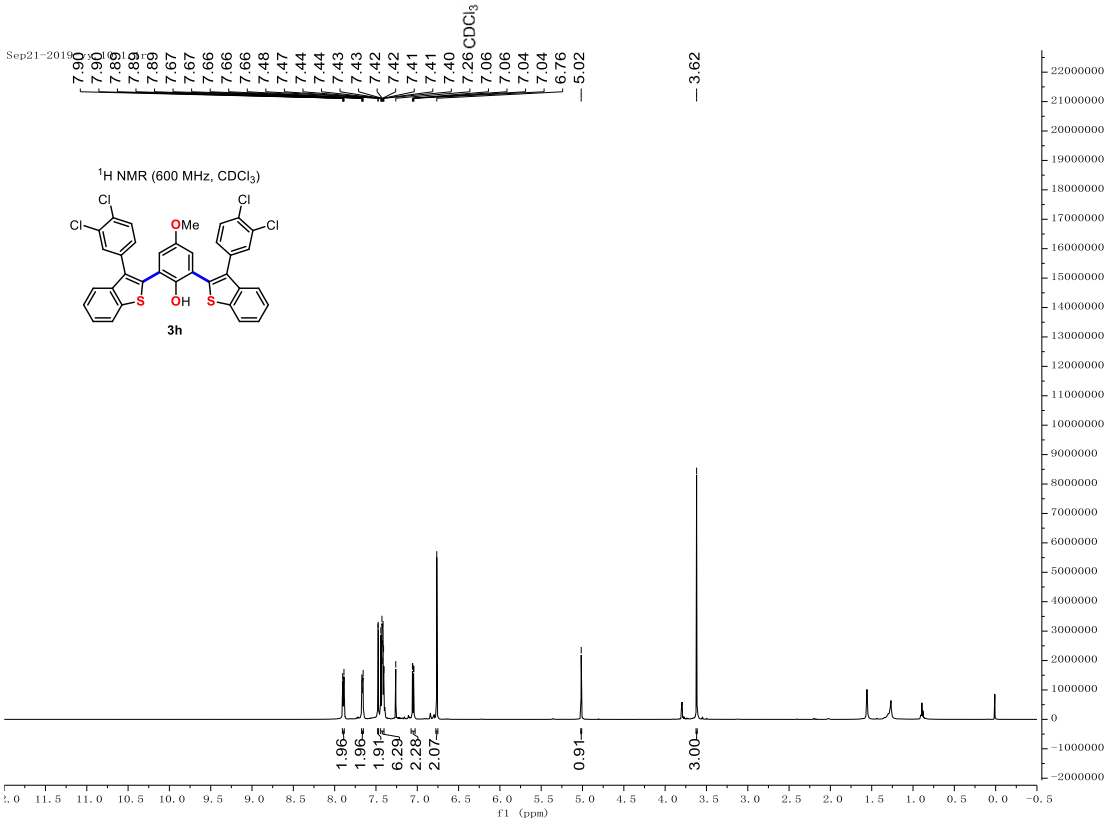

Jun17-2019-wzx. 20. 1. 1r
152.3
145.4
139.9
139.6
138.2
135.7
134.6
134.4
130.4
128.6
128.5
127.0
124.8
124.5
123.5
122.2
122.0
118.2
77.3
77.1 CDCl3
76.9
55.6
21.6
13C{1H} NMR (151 MHz, CDCl3)
OMe
OH
S
S
3e
f1 (ppm)

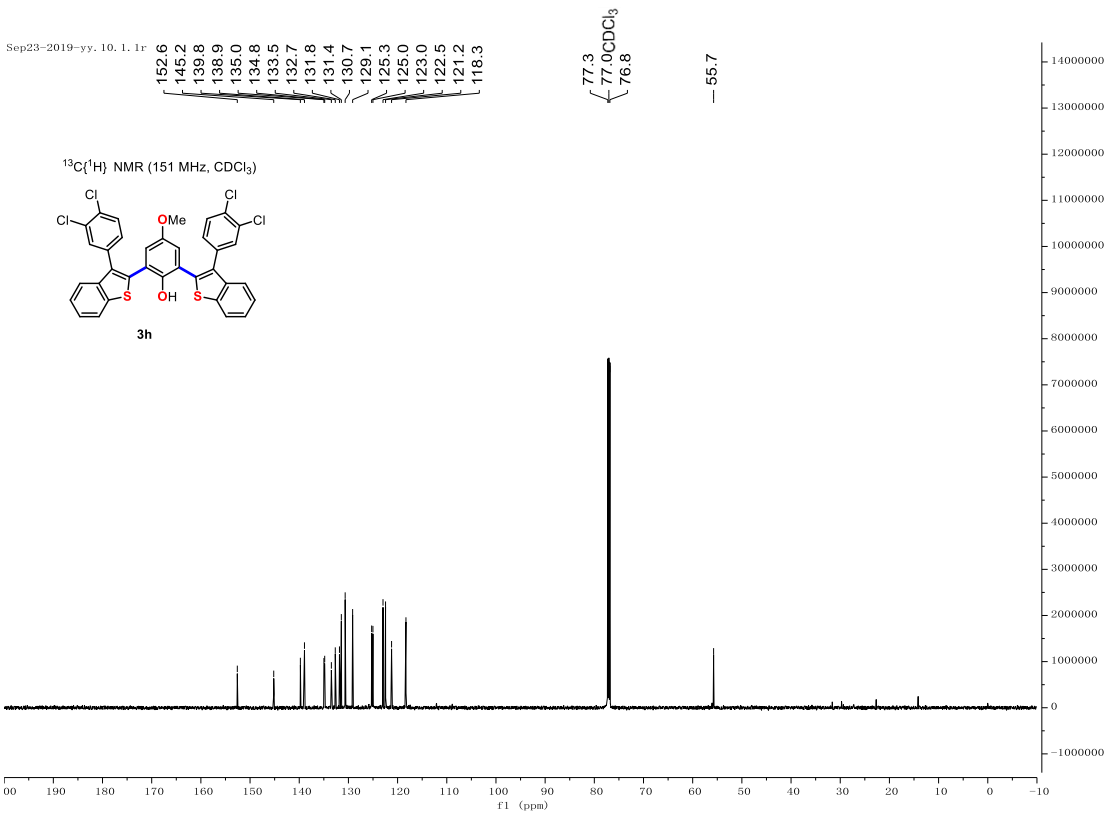

^{1}H NMR (600 MHz, CDCl$_3$)
Cl
OMe
Cl
S
OH
S
3f
CDCl$_3$
3.49
2.11
2.07
6.24
2.17
2.13
2.07
1.94
0.78
3.00
f1 (ppm)

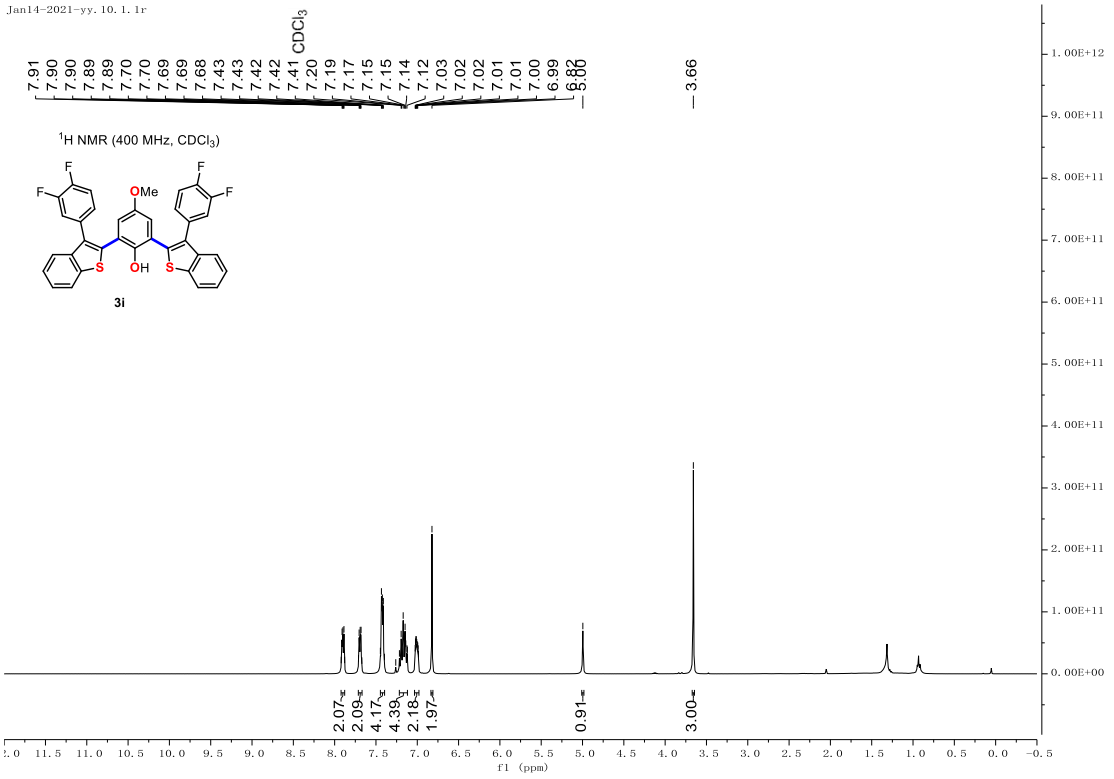

^{13}C{^{1}H} NMR (151 MHz, CDCl$_3$)
Cl
OMe
Cl
S
OH
S
3f
152.6
145.4
139.9
139.2
136.7
136.7
135.0
135.0
134.4
134.4
130.0
129.7
128.2
127.9
125.2
124.9
123.2
122.4
121.6
121.5
118.4
77.5
77.3
77.1
CDCl$_3$
66.0
55.8
f1 (ppm)

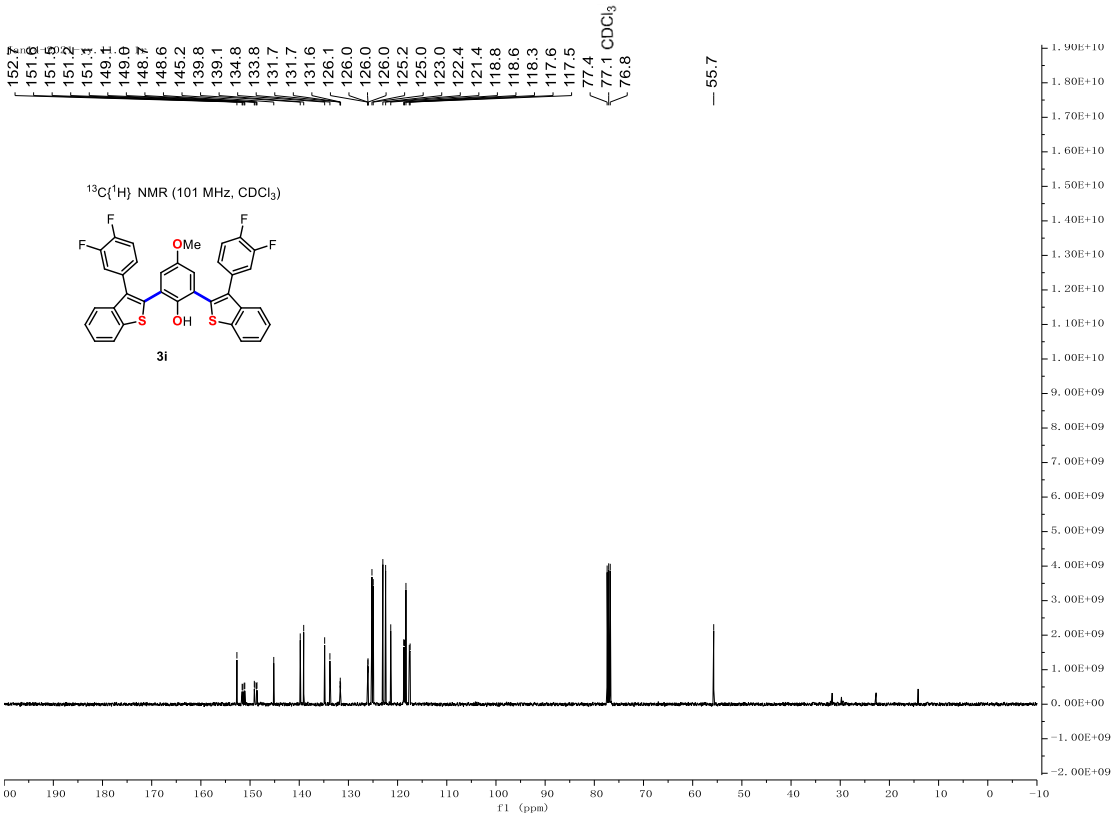

^{1}H NMR (400 MHz, CDCl$_3$)
OMe
F
F
S
OH
S
3g
CDCl$_3$
2.04
2.09
4.33
2.28
6.36
2.07
0.93
3.00
f1 (ppm)

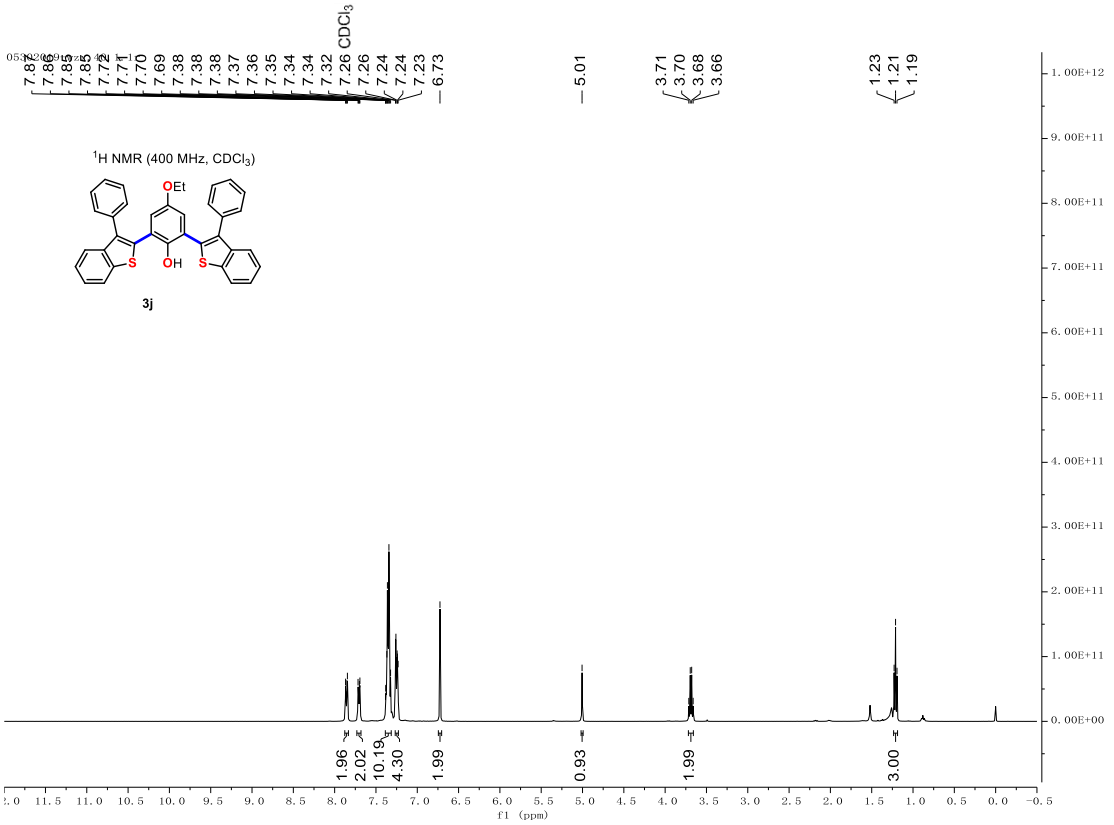

^{13}C{^{1}H} NMR (101 MHz, CDCl$_3$)
OMe
F
F
S
OH
S
3g
CDCl$_3$
55.69
f1 (ppm)

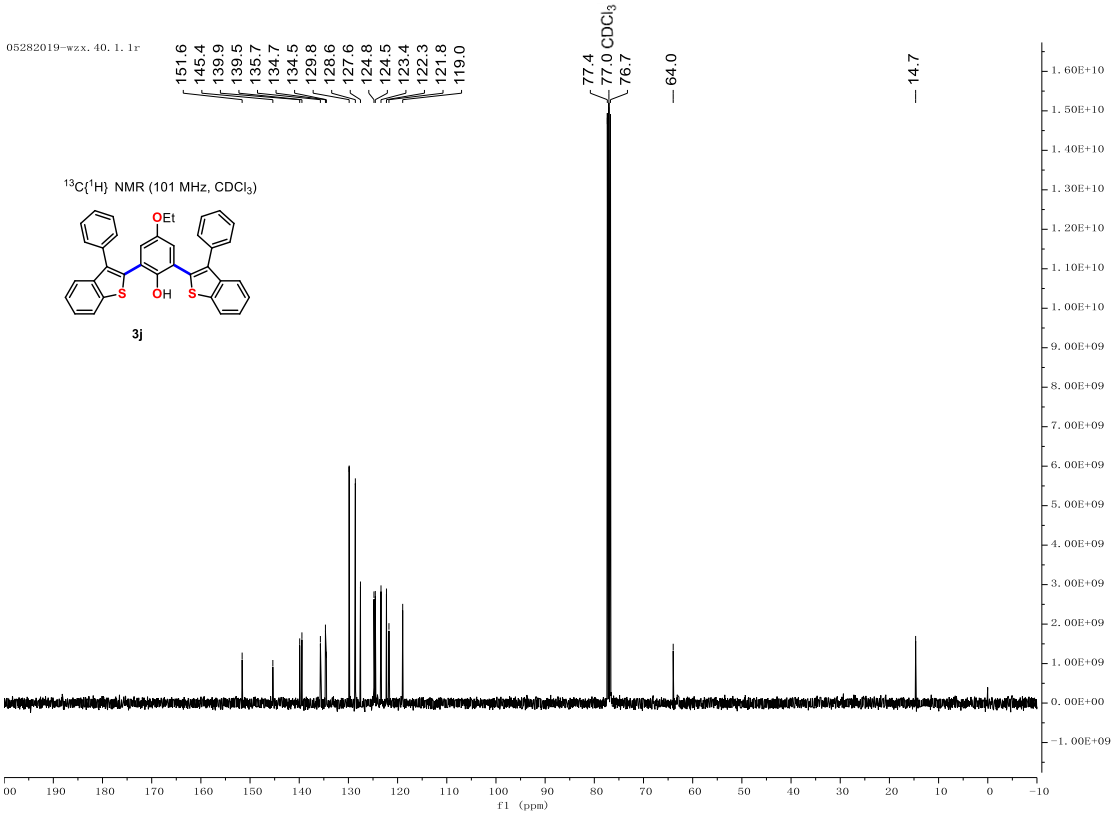

Sep21-2019
7.90
7.89
7.67
7.66
7.48
7.47
7.44
7.43
7.42
7.41
7.40
7.26 CDCl3
7.06
7.04
6.76
5.02
3.62
1H NMR (600 MHz, CDCl3)
Cl
Cl
OMe
Cl
Cl
S
OH
S
3h
1.96
1.96
1.91
6.29
2.28
2.07
0.91
3.00
f1 (ppm)

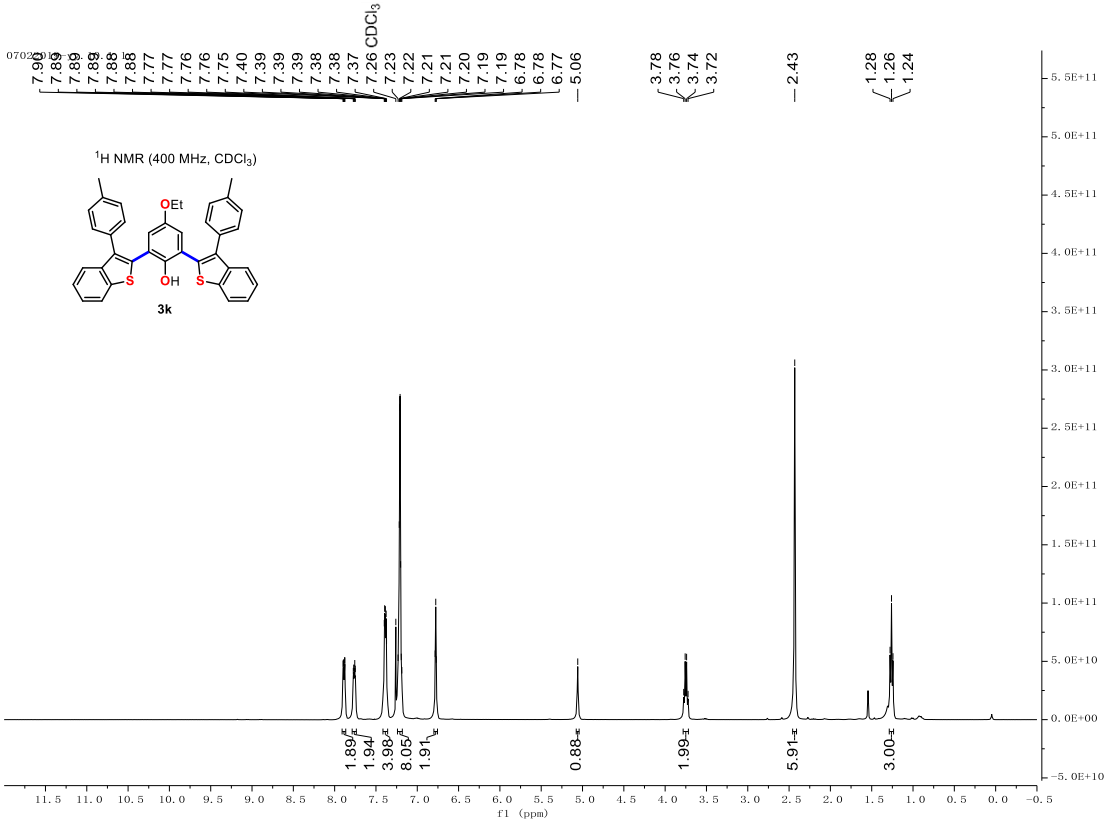

Sep23-2019-yy. 10. 1. 1r
152.6
145.2
139.8
138.9
135.0
134.8
133.5
132.7
131.8
131.4
130.7
129.1
125.3
125.0
123.0
122.5
121.2
118.3
77.3
77.0 CDCl3
76.8
55.7
13C{1H} NMR (151 MHz, CDCl3)
Cl
Cl
OMe
Cl
Cl
S
OH
S
3h
f1 (ppm)

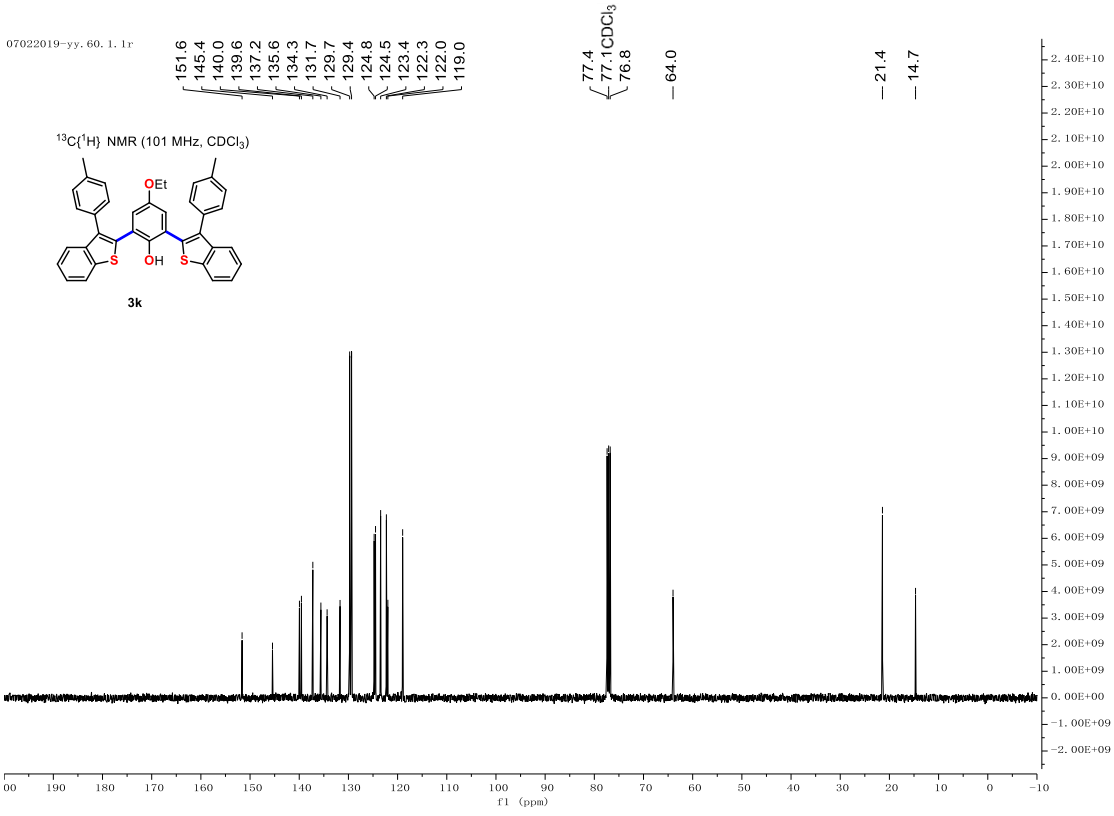

Jan14-2021-yy. 10. 1. 1r
CDCl3
7.91
7.90
7.90
7.89
7.89
7.70
7.70
7.69
7.69
7.68
7.43
7.43
7.42
7.42
7.41
7.20
7.19
7.17
7.15
7.15
7.14
7.12
7.03
7.02
7.02
7.01
7.01
7.00
6.99
6.82
5.00
3.66
1H NMR (400 MHz, CDCl3)
F
F
OMe
F
F
S
OH
S
3i
2.07
2.09
4.17
4.39
2.18
1.97
0.91
3.00
f1 (ppm)

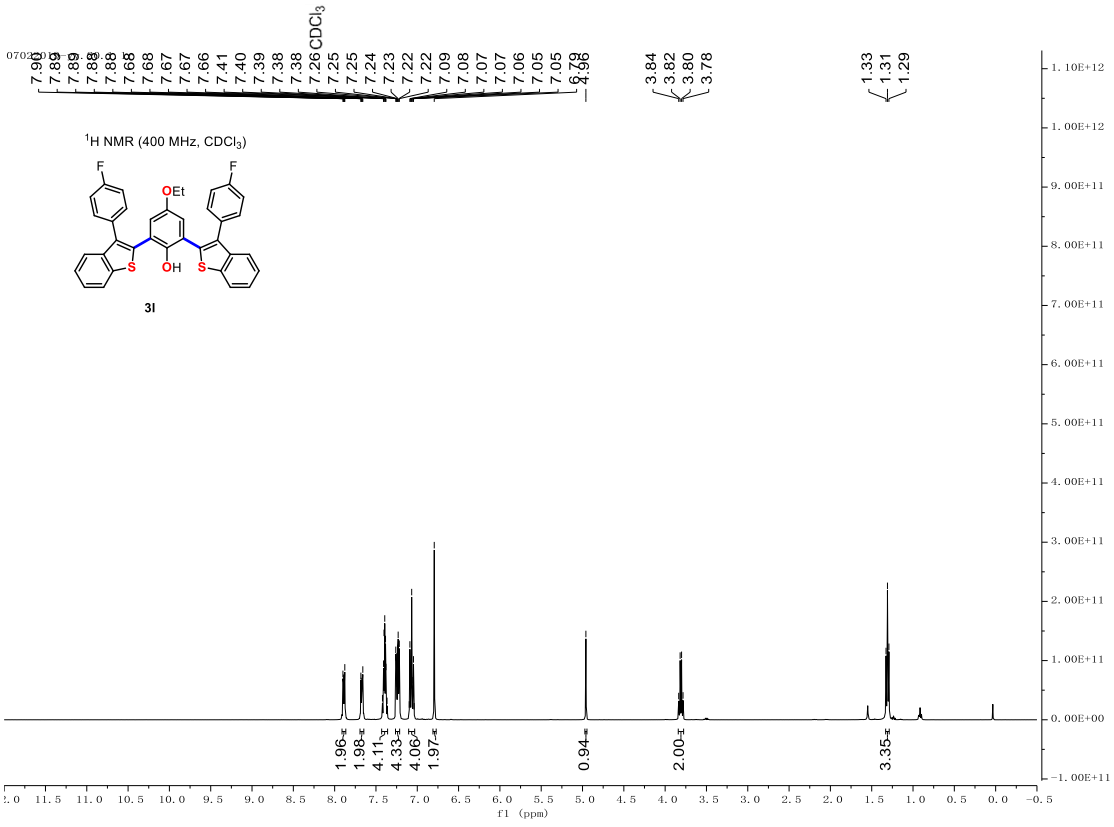

CDCl3
77.4
77.1
76.8
55.7
13C{1H} NMR (101 MHz, CDCl3)
F
F
OMe
F
F
S
OH
S
3i
f1 (ppm)

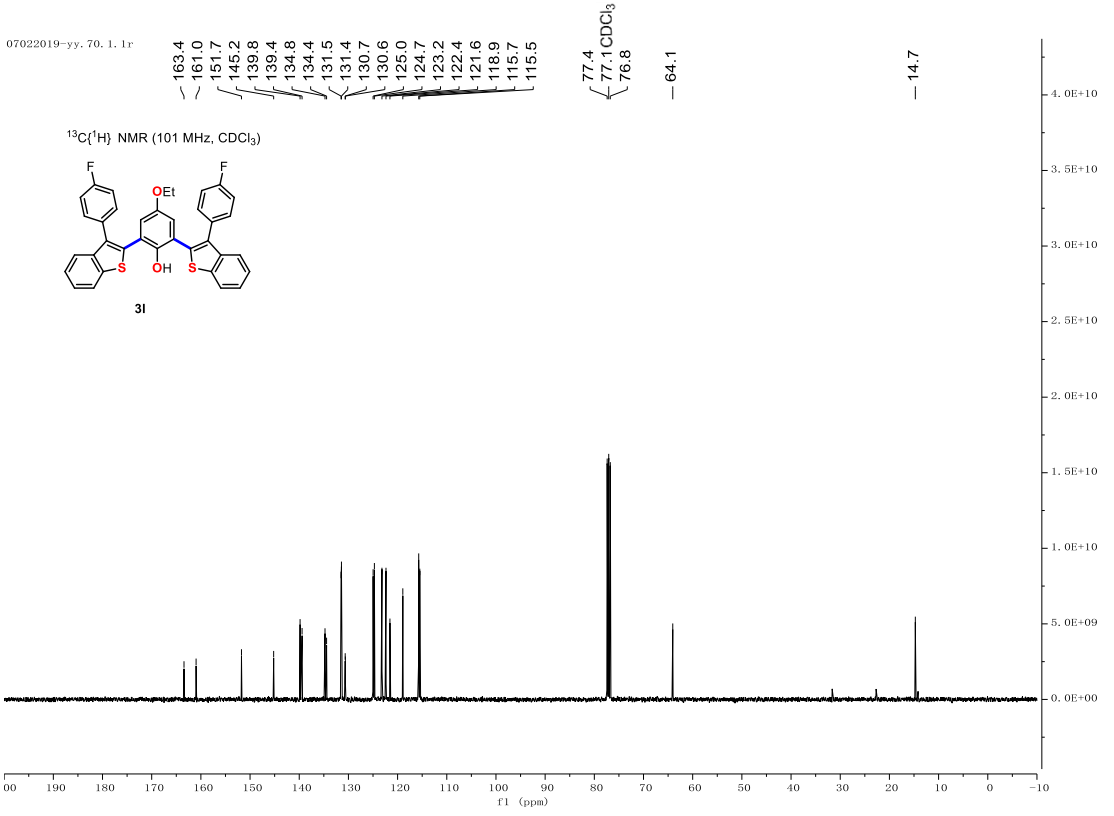

7.87
7.86
7.85
7.85
7.72
7.71
7.70
7.69
7.38
7.38
7.37
7.36
7.35
7.34
7.34
7.32
7.26 CDCl3
7.26
7.24
7.24
7.23
6.73
5.01
3.71
3.70
3.68
3.66
1.23
1.21
1.19
1H NMR (400 MHz, CDCl3)
OEt
OH
S
S
3j
1.96
2.02
10.19
4.30
1.99
0.93
1.99
3.00
f1 (ppm)

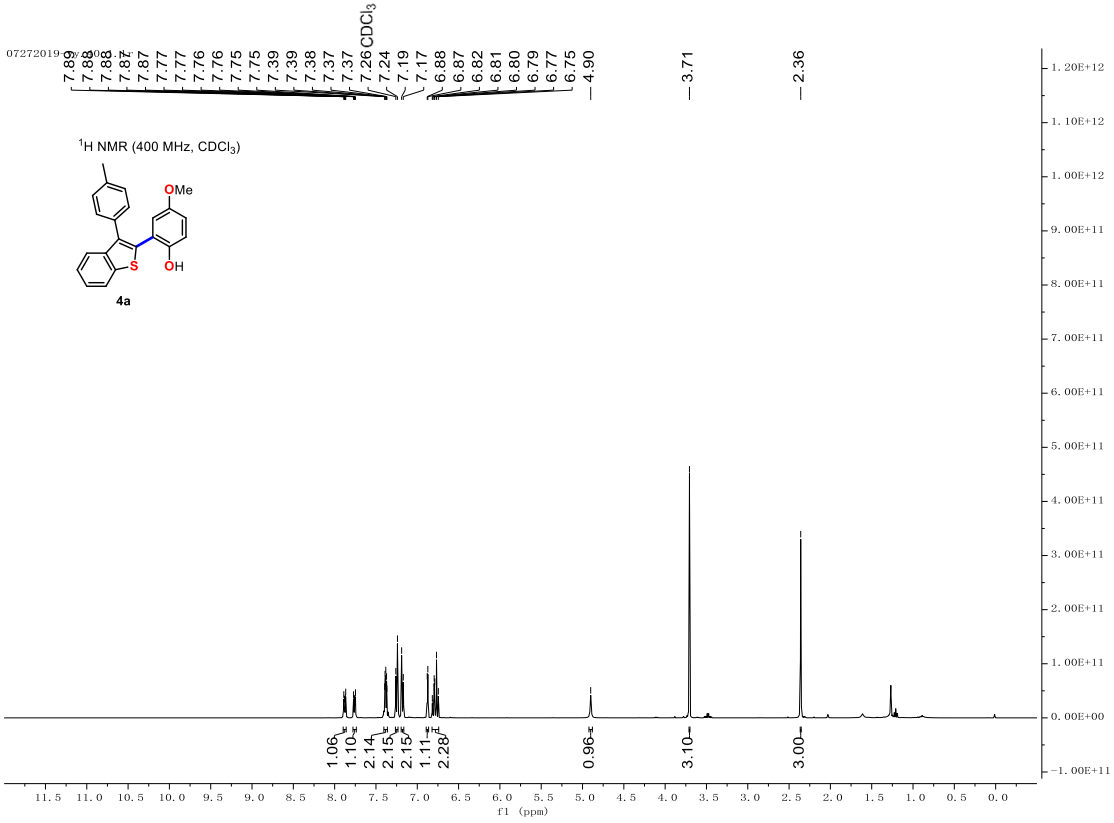

05282019-wzx. 40. 1. 1r
151.6
145.4
139.9
139.5
135.7
134.7
134.5
129.8
128.6
127.6
124.8
124.5
123.4
122.3
121.8
119.0
77.4
77.0 CDCl3
76.7
64.0
14.7
13C{1H} NMR (101 MHz, CDCl3)
OEt
OH
S
S
3j
f1 (ppm)

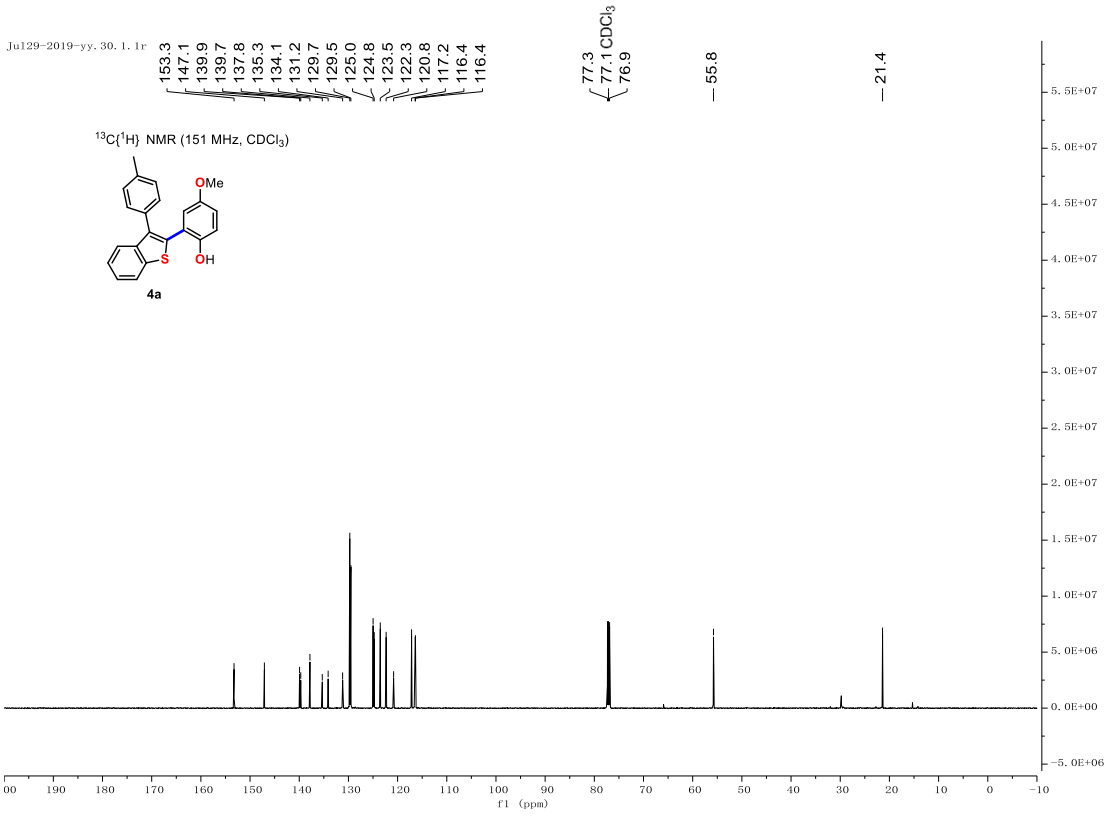

^{1}H NMR (400 MHz, $CDCl_3$)
OEt
OH
S
S
3k
7.90
7.89
7.89
7.89
7.88
7.88
7.77
7.77
7.76
7.76
7.75
7.40
7.39
7.39
7.38
7.38
7.37
7.26 $CDCl_3$
7.23
7.22
7.21
7.21
7.20
7.19
7.19
6.78
6.78
6.77
5.06
3.78
3.76
3.74
3.72
2.43
1.28
1.26
1.24
1.89
1.94
3.98
8.05
1.91
0.88
1.99
5.91
3.00
f1 (ppm)

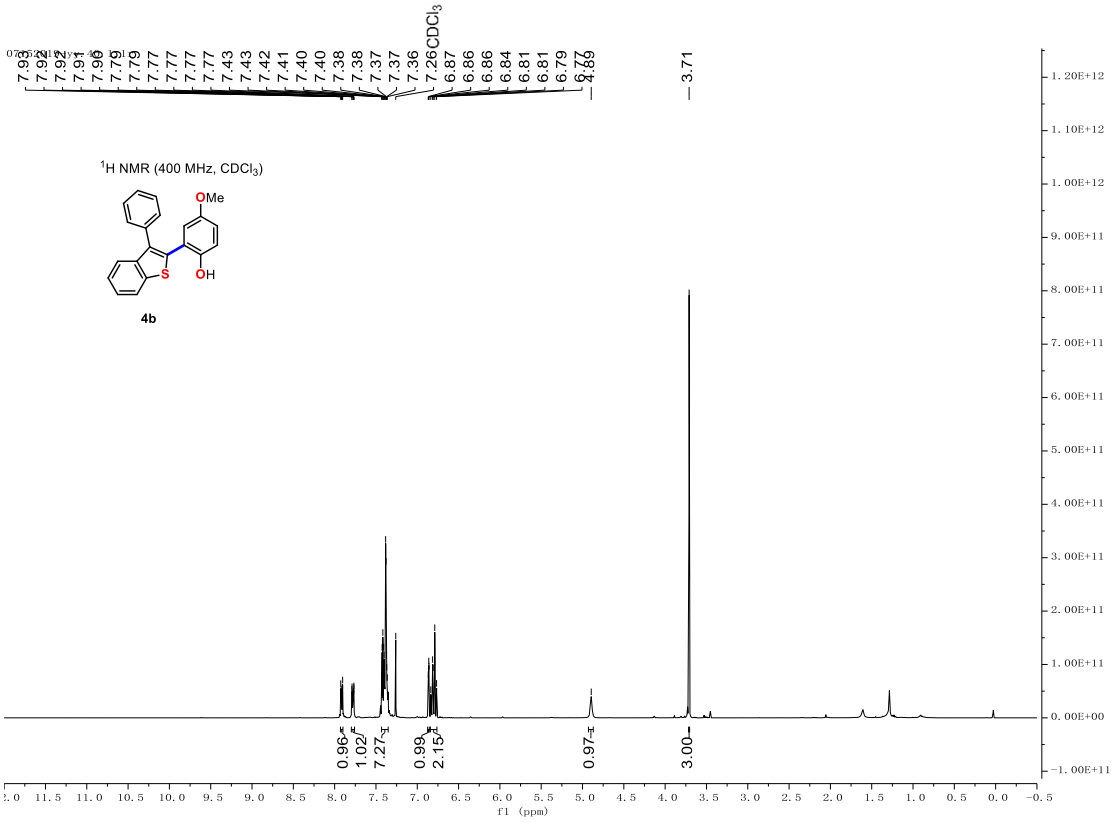

07022019-yy.60.1.1r
^{13}C{^{1}H} NMR (101 MHz, $CDCl_3$)
OEt
OH
S
S
3k
151.6
145.4
140.0
139.6
137.2
135.6
134.3
131.7
129.7
129.4
124.8
124.5
123.4
122.3
122.0
119.0
77.4
77.1 $CDCl_3$
76.8
64.0
21.4
14.7
f1 (ppm)

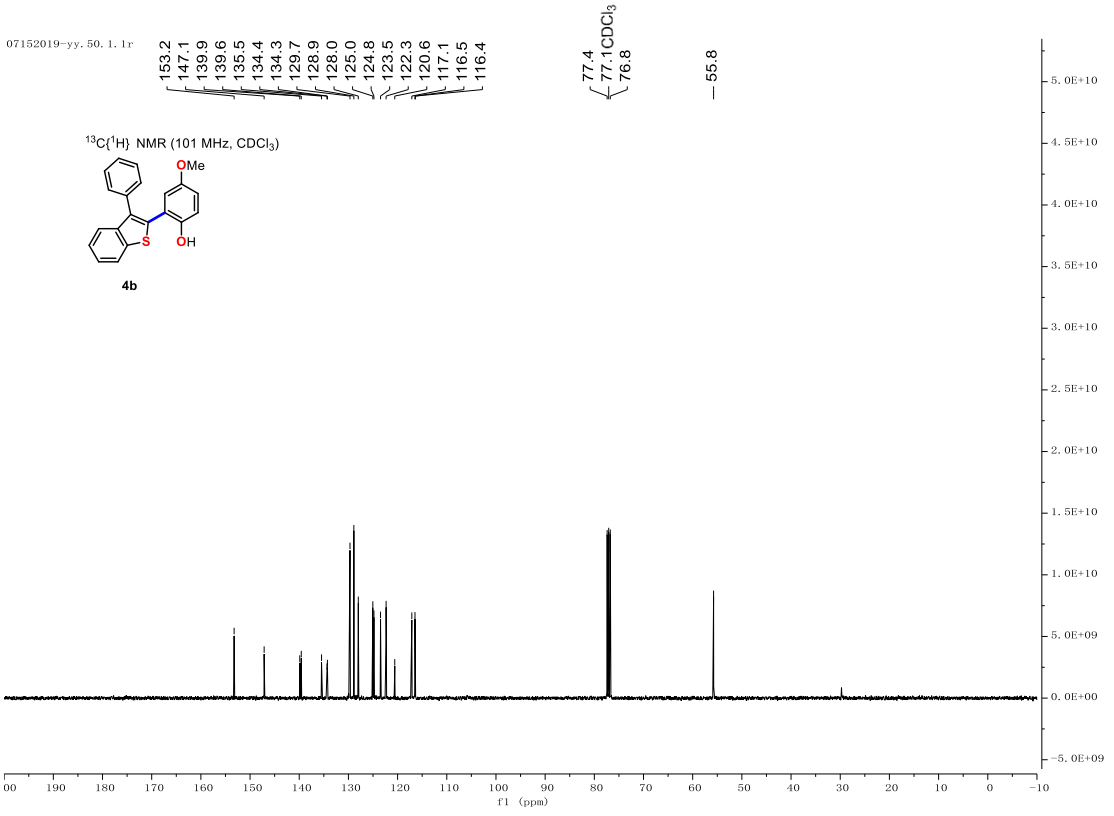

^{1}H NMR (400 MHz, $CDCl_3$)
3l
$CDCl_3$
f1 (ppm)

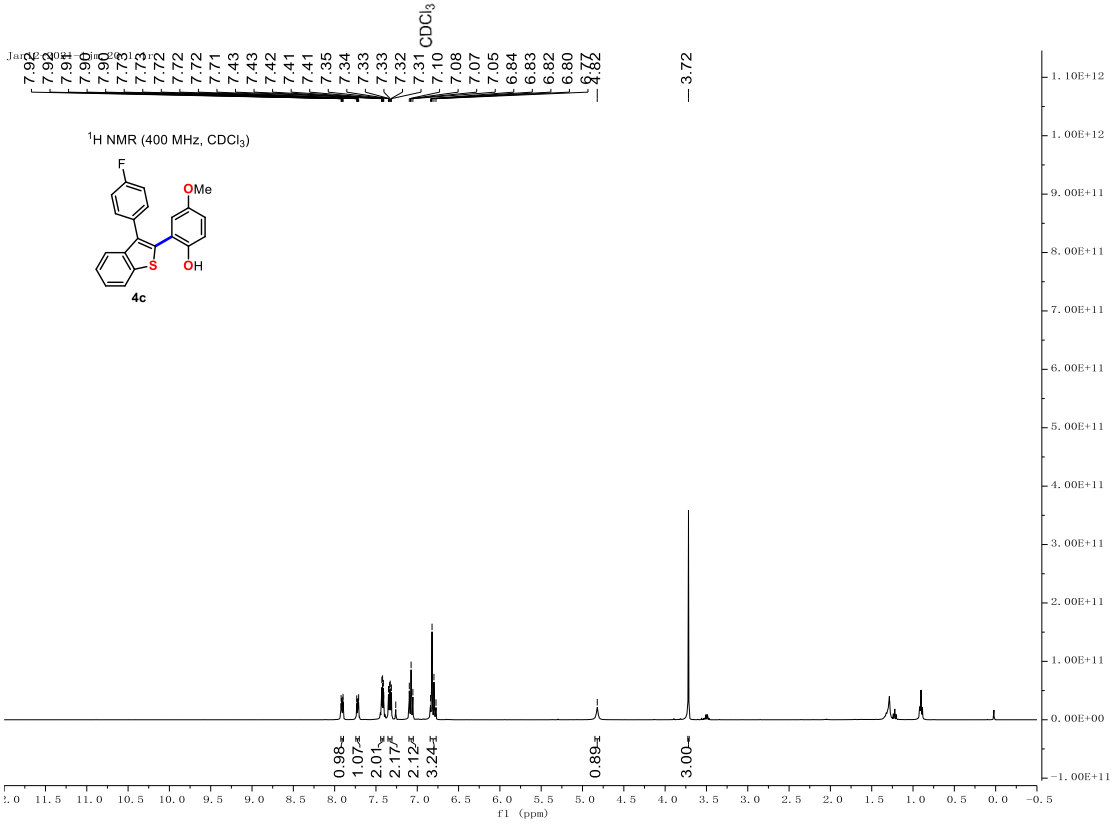

$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$)
3l
$CDCl_3$
f1 (ppm)

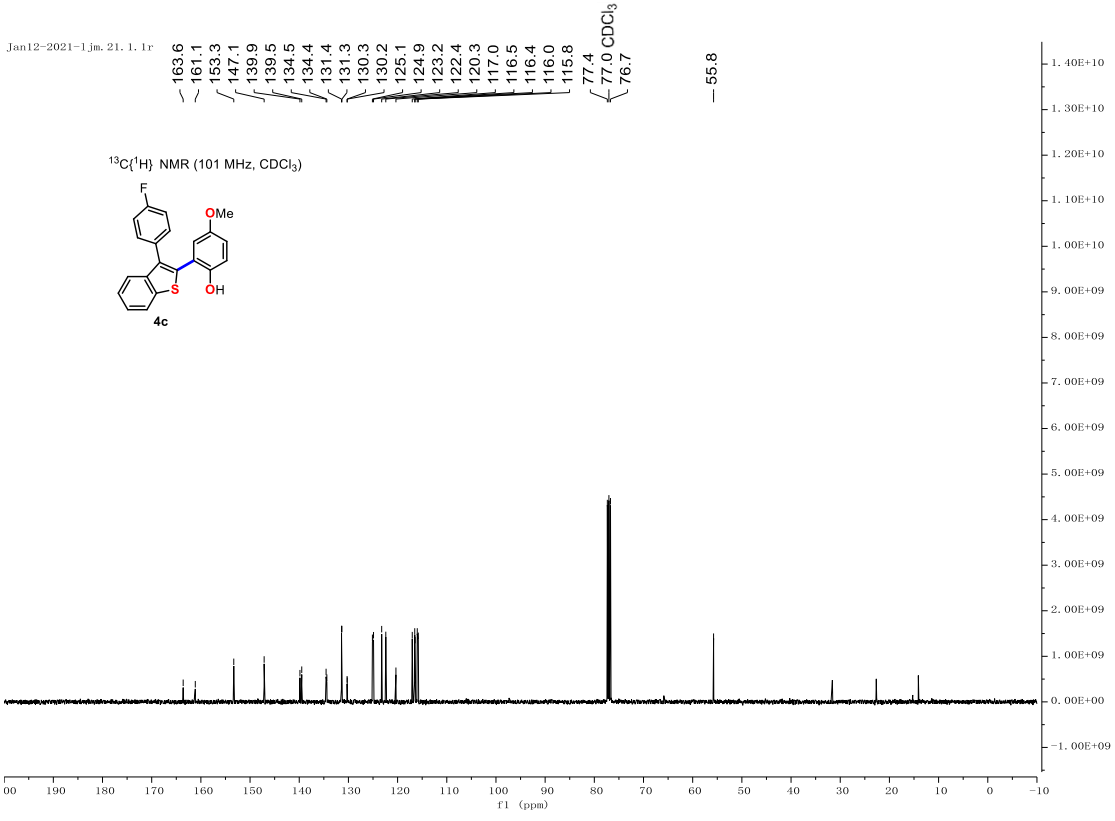

^{1}H NMR (400 MHz, CDCl$_3$)
OMe
OH
S
4a
CDCl$_3$
f1 (ppm)

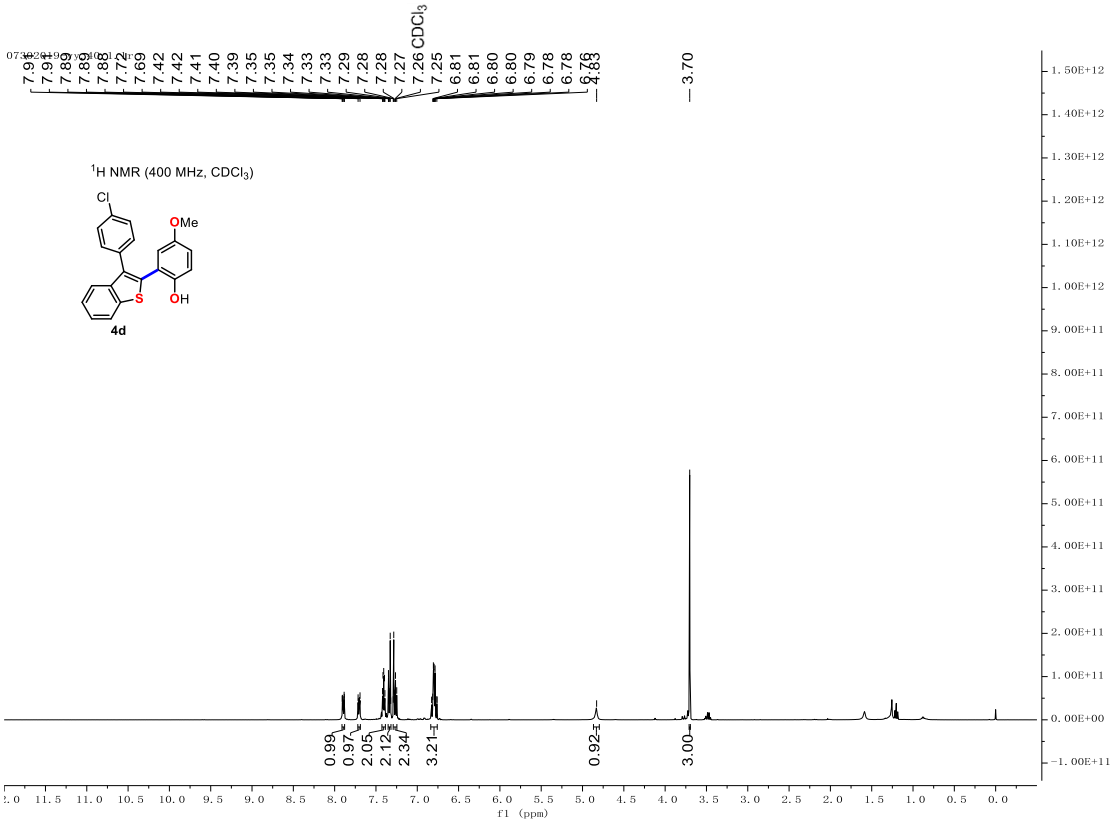

^{13}C{^{1}H} NMR (151 MHz, CDCl$_3$)
OMe
OH
S
4a
CDCl$_3$
f1 (ppm)

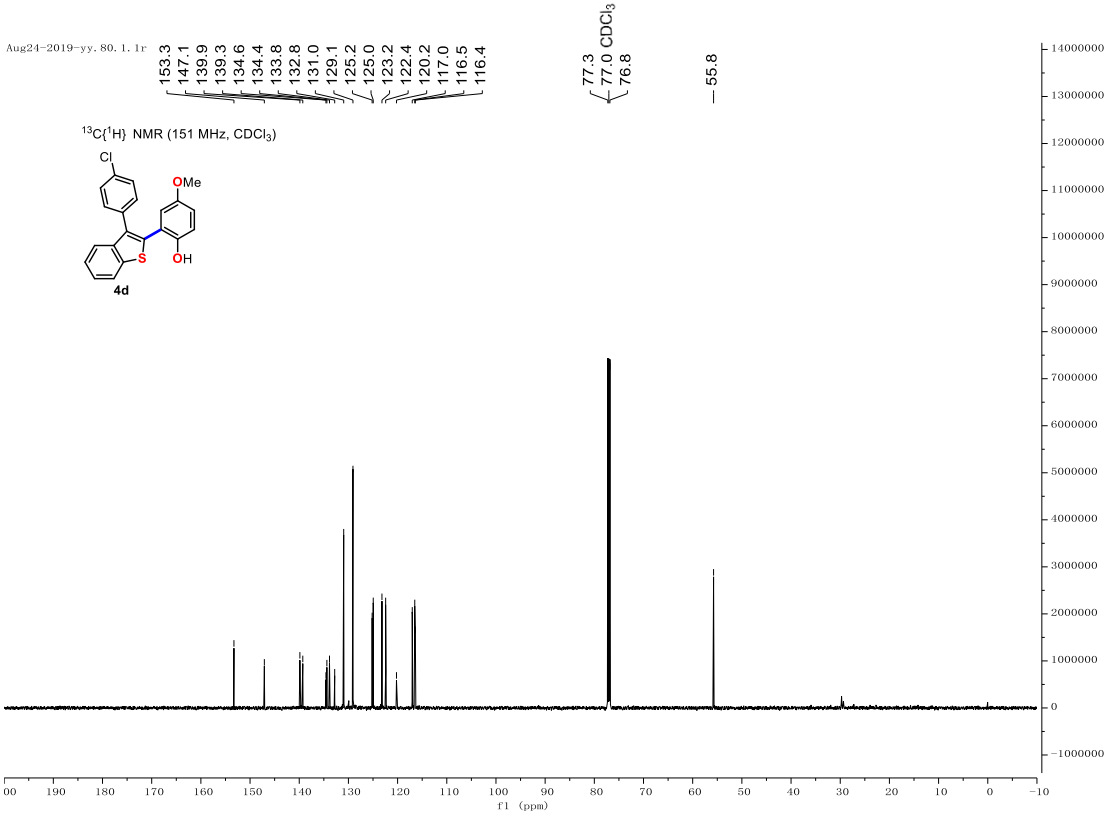

7.93
7.92
7.92
7.91
7.90
7.79
7.77
7.77
7.77
7.77
7.43
7.42
7.41
7.40
7.40
7.38
7.37
7.37
7.36
7.26CDCl3
6.87
6.86
6.86
6.84
6.81
6.81
6.79
6.77
4.89
3.71
1H NMR (400 MHz, CDCl3)
OMe
S
OH
4b
0.96
1.02
7.27
0.99
2.15
0.97
3.00
f1 (ppm)

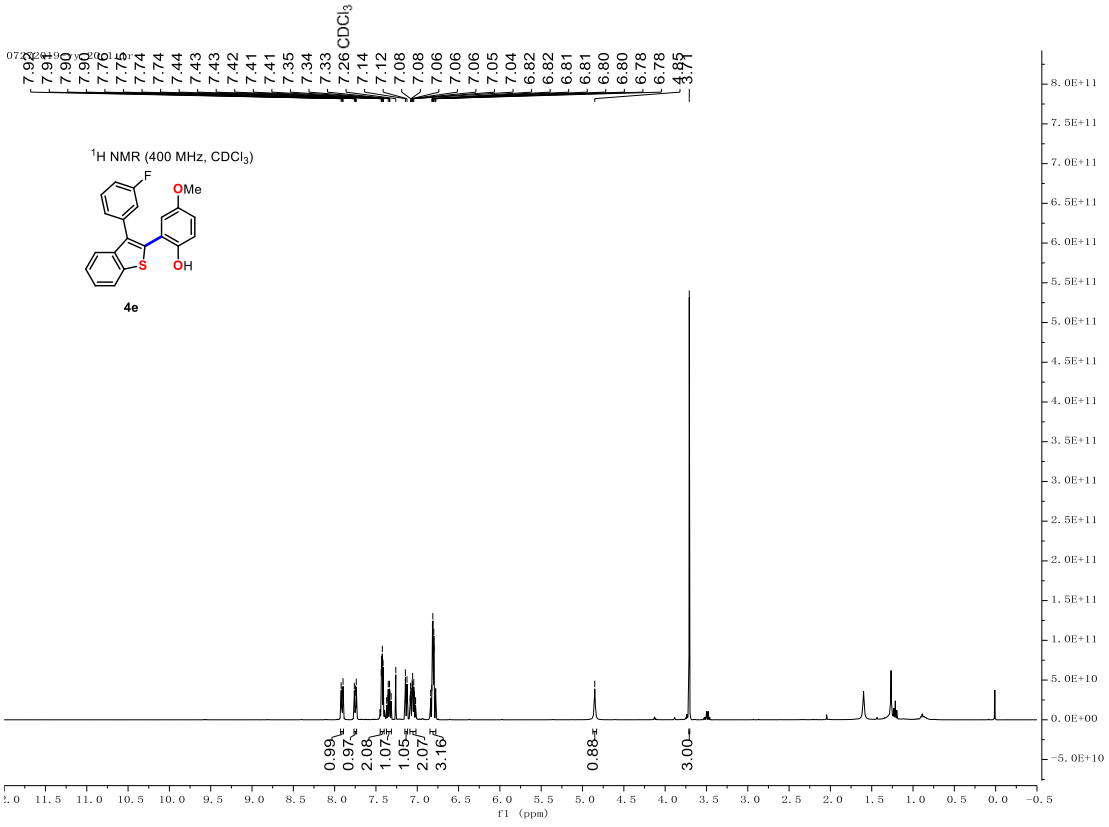

07152019-yy. 50. 1. 1r
153.2
147.1
139.9
139.6
135.5
134.4
134.3
129.7
128.9
128.0
125.0
124.8
123.5
122.3
120.6
117.1
116.5
116.4
77.4
77.1CDCl3
76.8
55.8
13C{1H} NMR (101 MHz, CDCl3)
OMe
S
OH
4b
f1 (ppm)

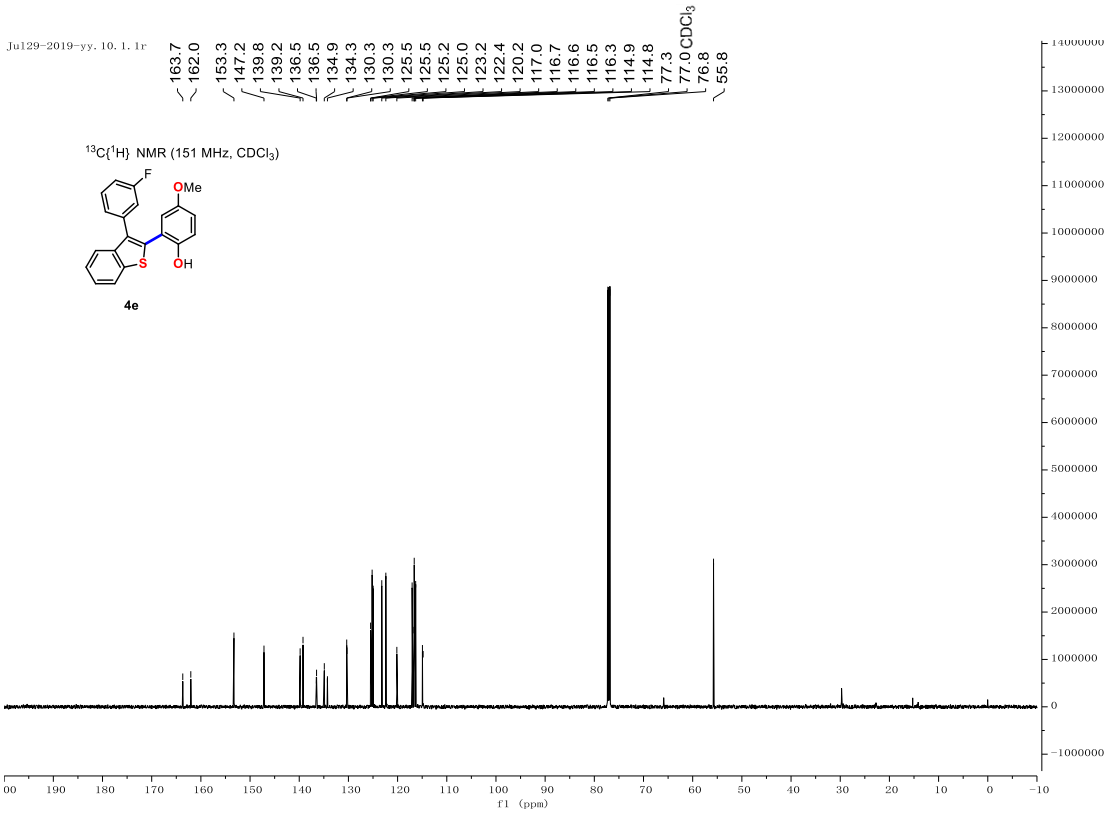

7.95
7.92
7.91
7.90
7.90
7.78
7.77
7.72
7.72
7.72
7.71
7.43
7.43
7.42
7.41
7.41
7.35
7.34
7.33
7.33
7.32
7.31 CDCl3
7.10
7.08
7.07
7.05
6.84
6.83
6.82
6.80
6.77
4.82
3.72
1H NMR (400 MHz, CDCl3)
F
OMe
S
OH
4c
0.98
1.07
2.01
2.17
2.12
3.24
0.89
3.00
f1 (ppm)

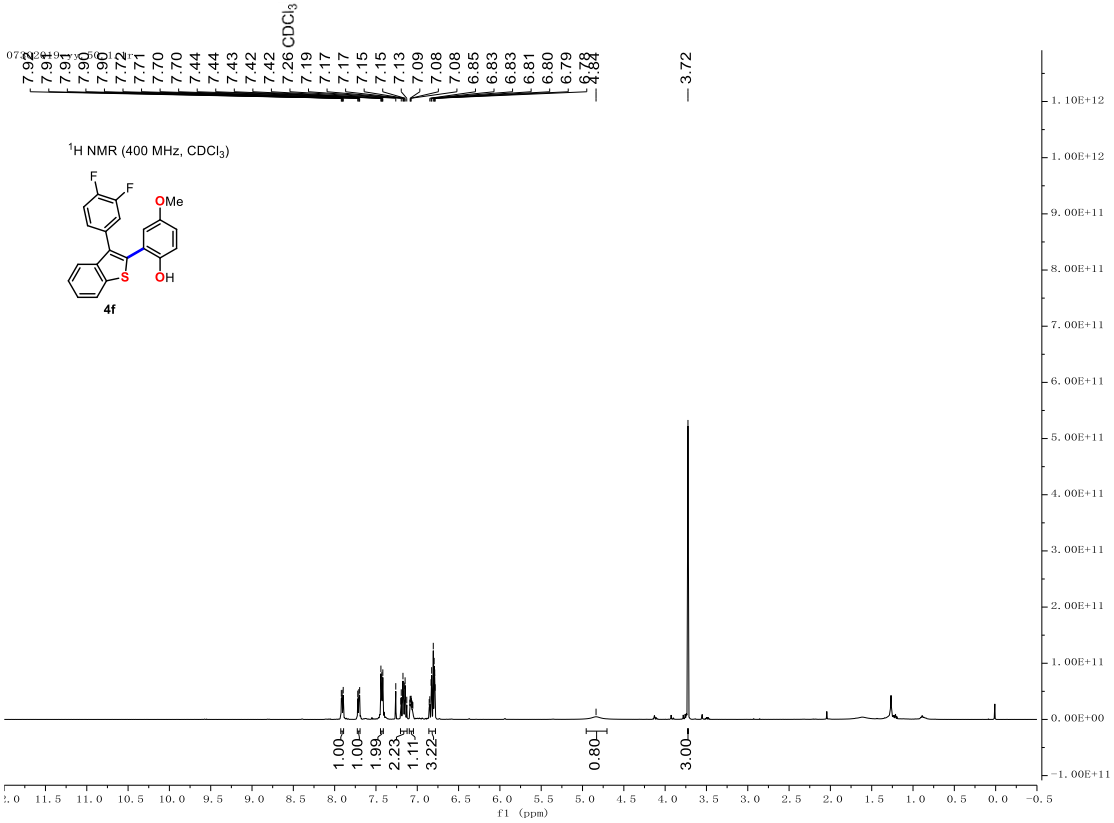

Jan12-2021-1jm.21.1.1r
163.6
161.1
153.3
147.1
139.9
139.5
134.5
134.4
131.4
131.3
130.3
130.2
125.1
124.9
123.2
122.4
120.3
117.0
116.5
116.4
116.0
115.8
77.4
77.0 CDCl3
76.7
55.8
13C{1H} NMR (101 MHz, CDCl3)
F
OMe
S
OH
4c
f1 (ppm)

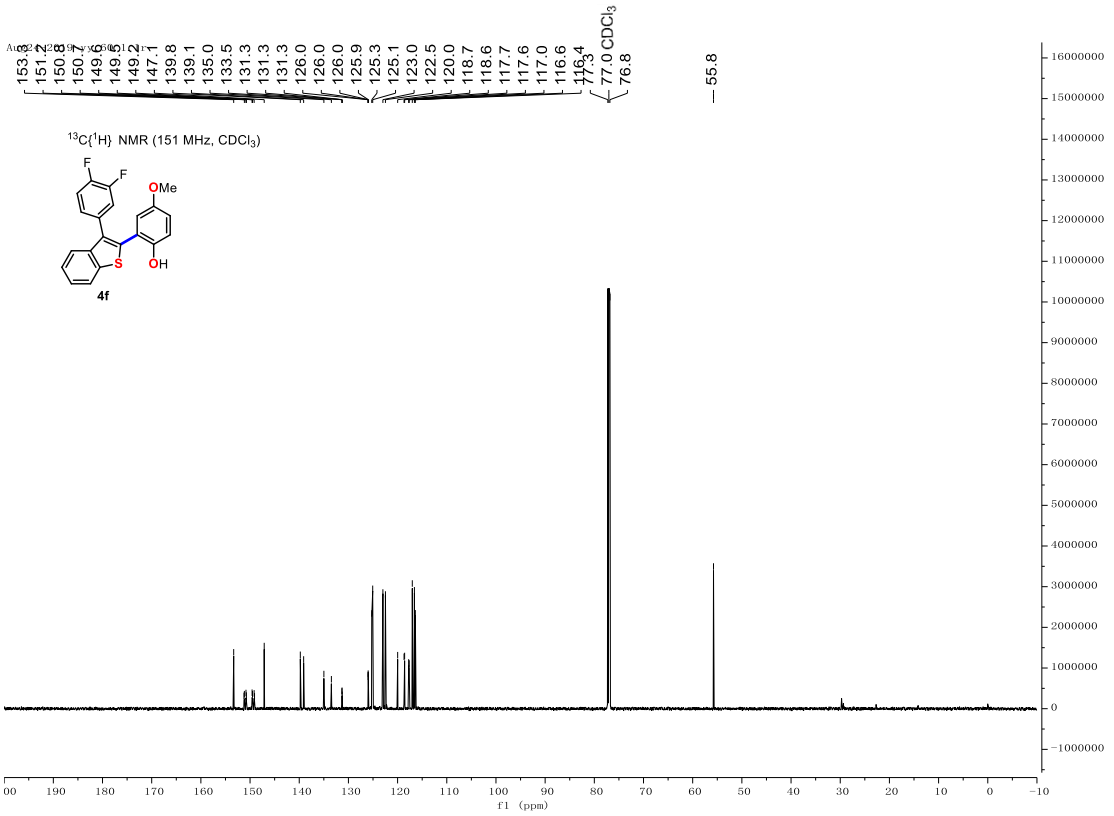

7.91
7.90
7.89
7.89
7.88
7.72
7.69
7.42
7.42
7.41
7.40
7.39
7.35
7.35
7.34
7.33
7.33
7.29
7.28
7.28
7.27
7.26 CDCl3
7.25
6.81
6.81
6.80
6.80
6.79
6.78
6.78
6.76
4.83
3.70
1H NMR (400 MHz, CDCl3)
Cl
OMe
S
OH
4d
0.99
0.97
2.05
2.12
2.34
3.21
0.92
3.00
f1 (ppm)

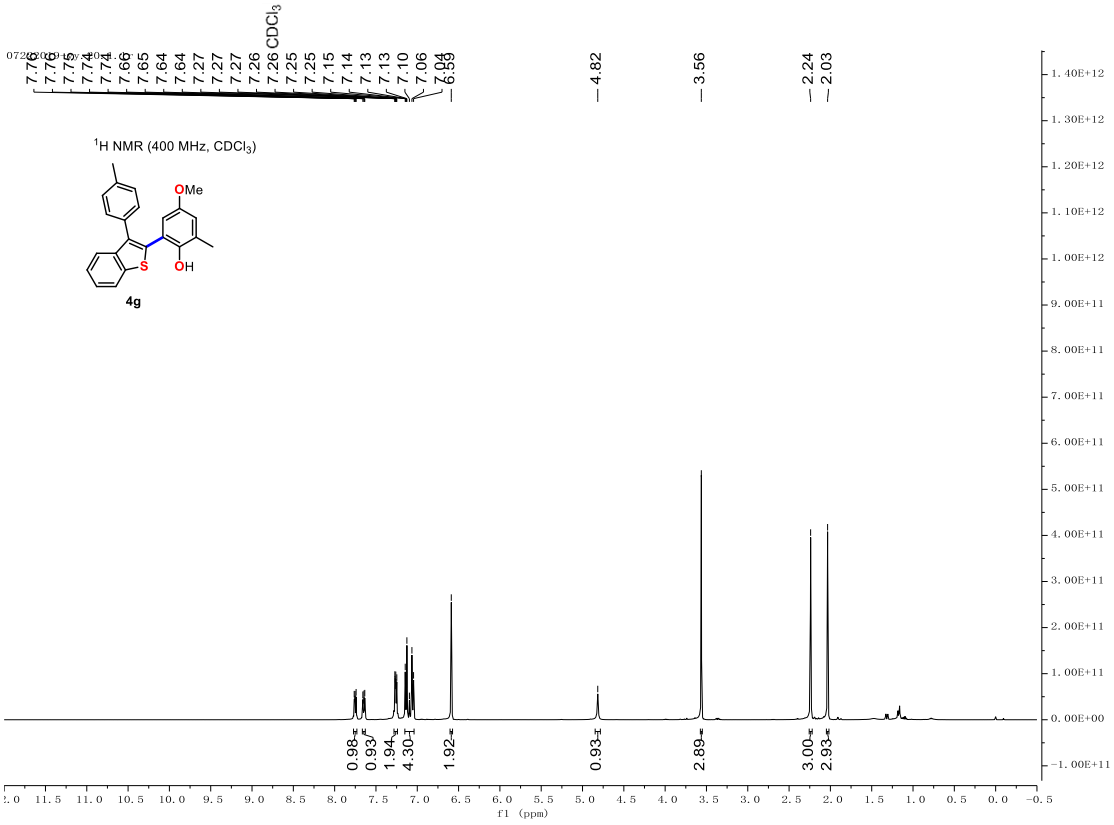

Aug24-2019-yy. 80. 1. 1r
153.3
147.1
139.9
139.3
134.6
134.4
133.8
132.8
131.0
129.1
125.2
125.0
123.2
122.4
120.2
117.0
116.5
116.4
77.3
77.0 CDCl3
76.8
55.8
13C{1H} NMR (151 MHz, CDCl3)
Cl
OMe
S
OH
4d
f1 (ppm)

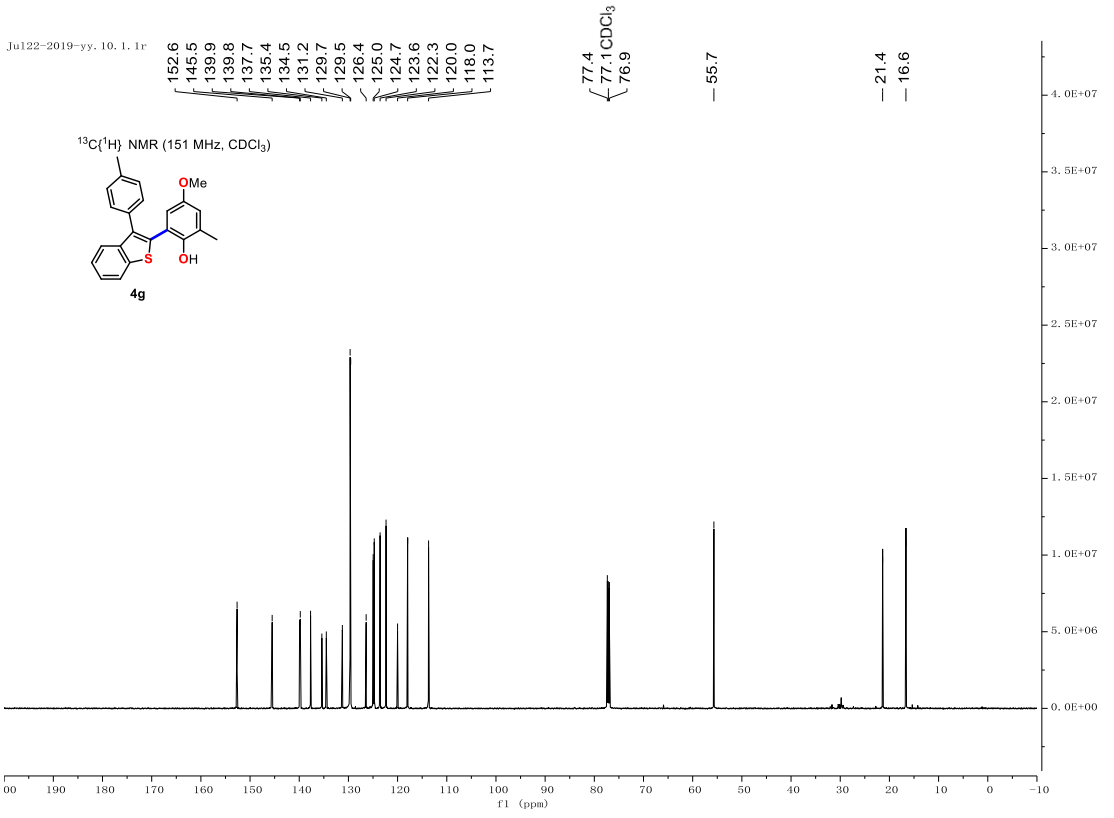

^{1}H NMR (400 MHz, CDCl$_3$)
4e
7.92 7.91 7.90 7.90 7.76 7.75 7.74 7.74 7.44 7.43 7.43 7.42 7.41 7.41 7.35 7.34 7.33 7.26 CDCl$_3$ 7.14 7.12 7.08 7.08 7.06 7.06 7.06 7.05 7.04 6.82 6.82 6.81 6.81 6.80 6.80 6.78 6.78 4.85 3.71
0.99 0.97 2.08 1.07 1.05 2.07 3.16 0.88 3.00
f1 (ppm)

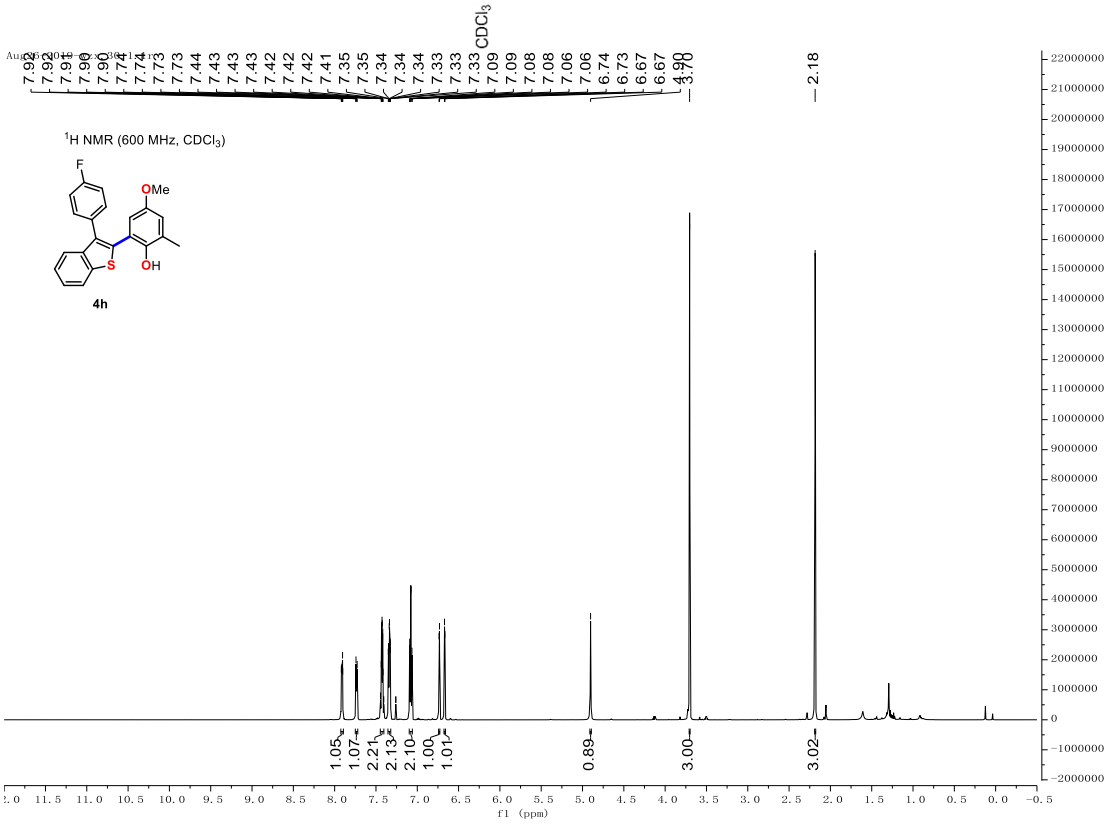

Ju129-2019-yy. 10. 1. 1r
^{13}C{^{1}H} NMR (151 MHz, CDCl$_3$)
4e
163.7 162.0 153.3 147.2 139.8 139.2 136.5 136.5 134.9 134.3 130.3 130.3 125.5 125.5 125.2 125.0 123.2 122.4 120.2 117.0 116.7 116.6 116.5 116.3 114.9 114.8 77.3 77.0 CDCl$_3$ 76.8 55.8
f1 (ppm)

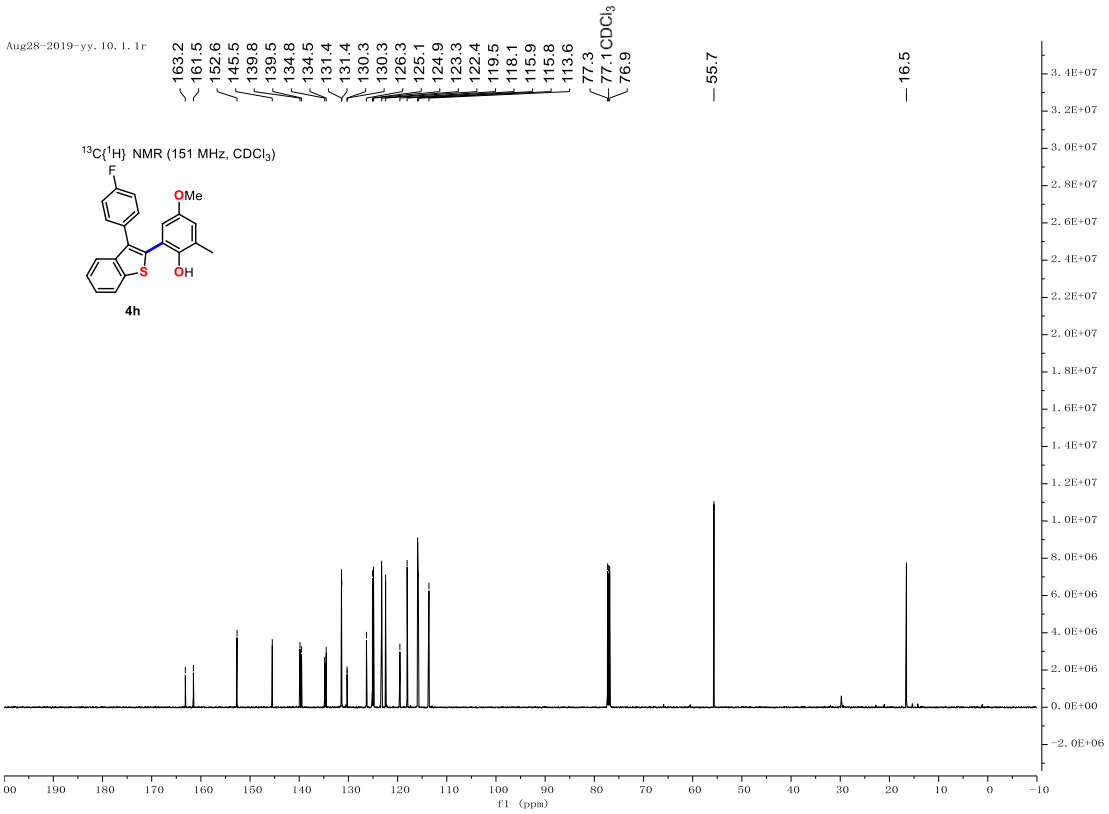

7.92
7.91
7.91
7.90
7.90
7.72
7.71
7.70
7.70
7.44
7.44
7.43
7.42
7.42
7.26 CDCl3
7.19
7.17
7.17
7.15
7.15
7.13
7.09
7.08
7.08
6.85
6.83
6.83
6.81
6.80
6.79
6.78
4.84
3.72
1H NMR (400 MHz, CDCl3)
F
F
OMe
S
OH
4f
1.00
1.00
1.99
2.23
1.11
3.22
0.80
3.00
f1 (ppm)

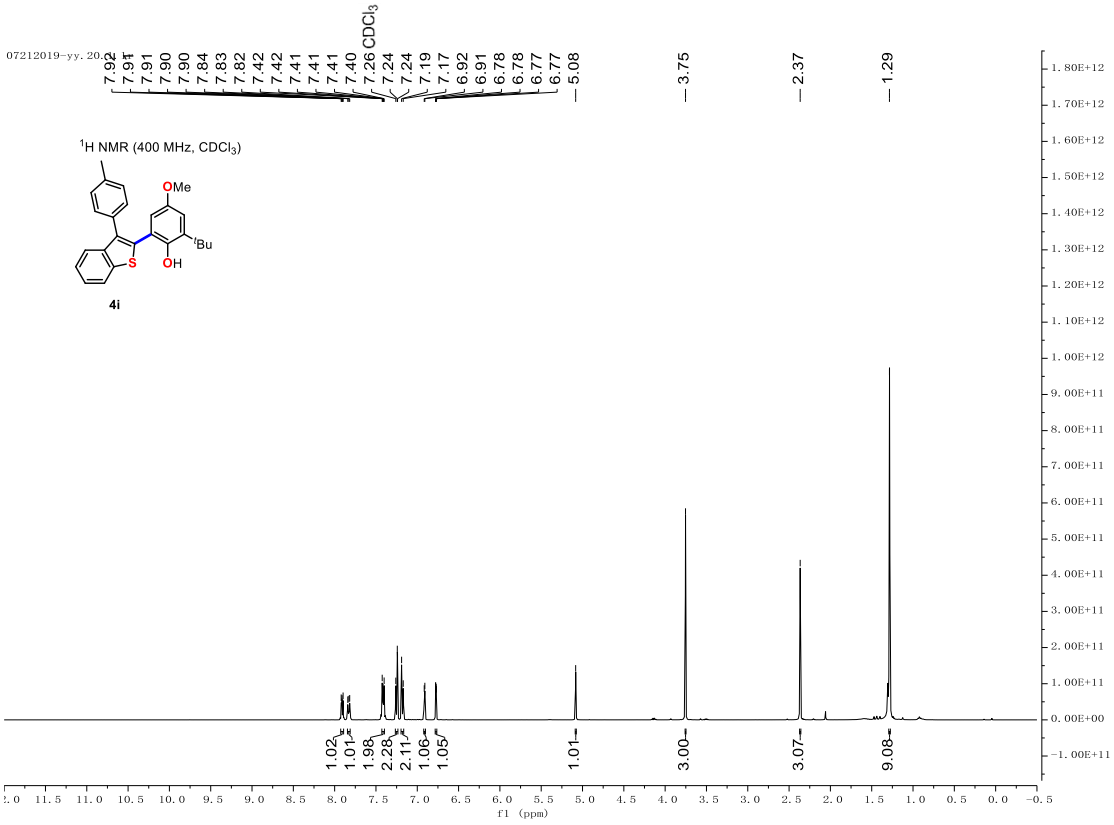

153.8
151.2
150.8
150.7
149.6
149.5
149.2
147.1
139.8
139.1
135.0
133.5
131.3
131.3
131.3
126.0
126.0
126.0
125.9
125.3
125.1
123.0
122.5
120.0
118.7
118.6
117.7
117.6
117.0
116.6
116.4
77.3
77.0 CDCl3
76.8
55.8
13C{1H} NMR (151 MHz, CDCl3)
F
F
OMe
S
OH
4f
f1 (ppm)

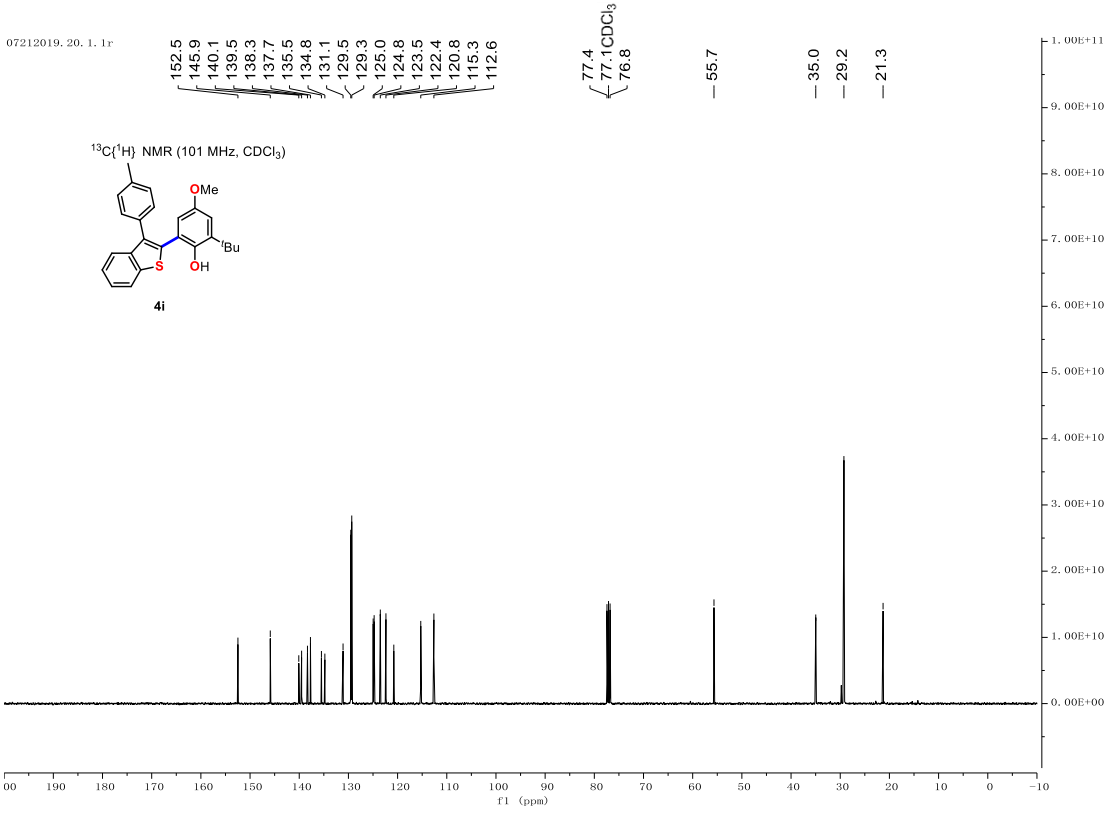

7.76
7.76
7.75
7.74
7.74
7.66
7.65
7.64
7.64
7.27
7.27
7.27
7.26 CDCl3
7.25
7.25
7.15
7.14
7.13
7.13
7.10
7.06
7.04
6.99
4.82
3.56
2.24
2.03
1H NMR (400 MHz, CDCl3)
OMe
S
OH
4g
0.98
0.93
1.94
4.30
1.92
0.93
2.89
3.00
2.93
f1 (ppm)

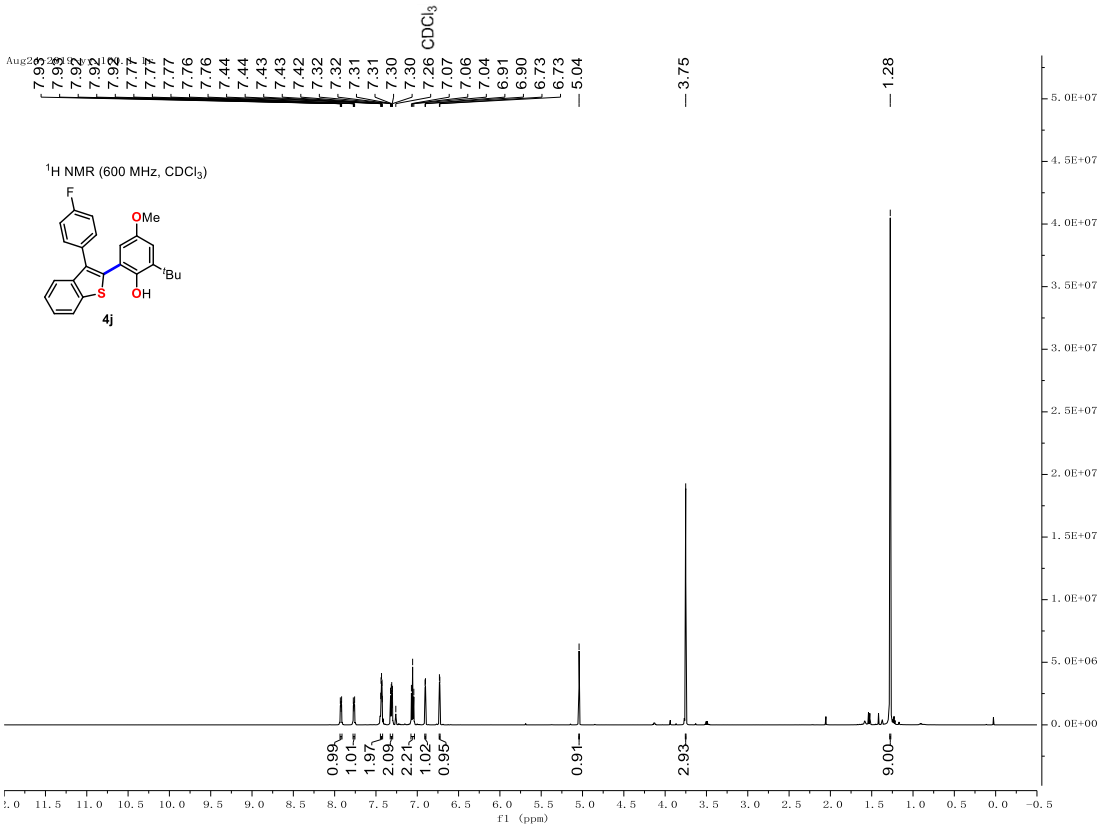

Ju122-2019-yy. 10. 1. 1r
152.6
145.5
139.9
139.8
137.7
135.4
134.5
131.2
129.7
129.5
126.4
125.0
124.7
123.6
122.3
120.0
118.0
113.7
77.4
77.1 CDCl3
76.9
55.7
21.4
16.6
13C{1H} NMR (151 MHz, CDCl3)
OMe
S
OH
4g
f1 (ppm)

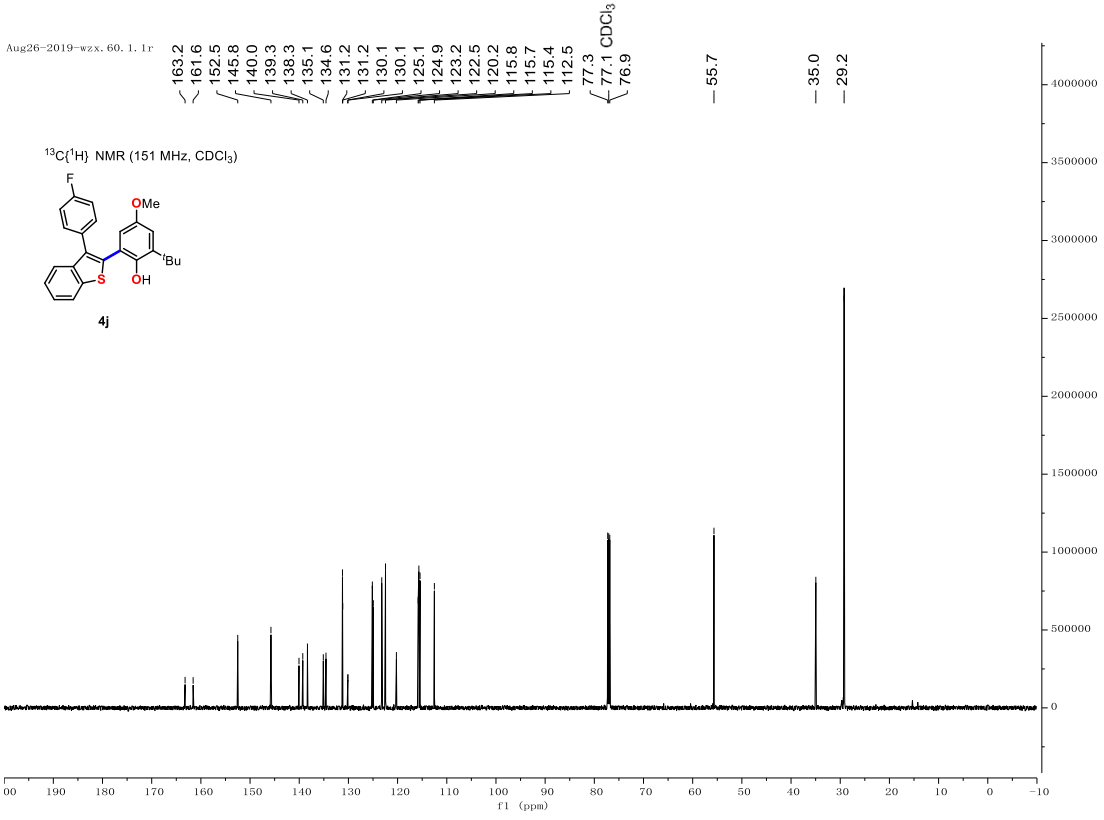

^{1}H NMR (600 MHz, CDCl$_3$)
4h
CDCl$_3$
2.18
1.05
1.07
2.21
2.13
2.10
1.00
1.01
0.89
3.00
3.02
f1 (ppm)

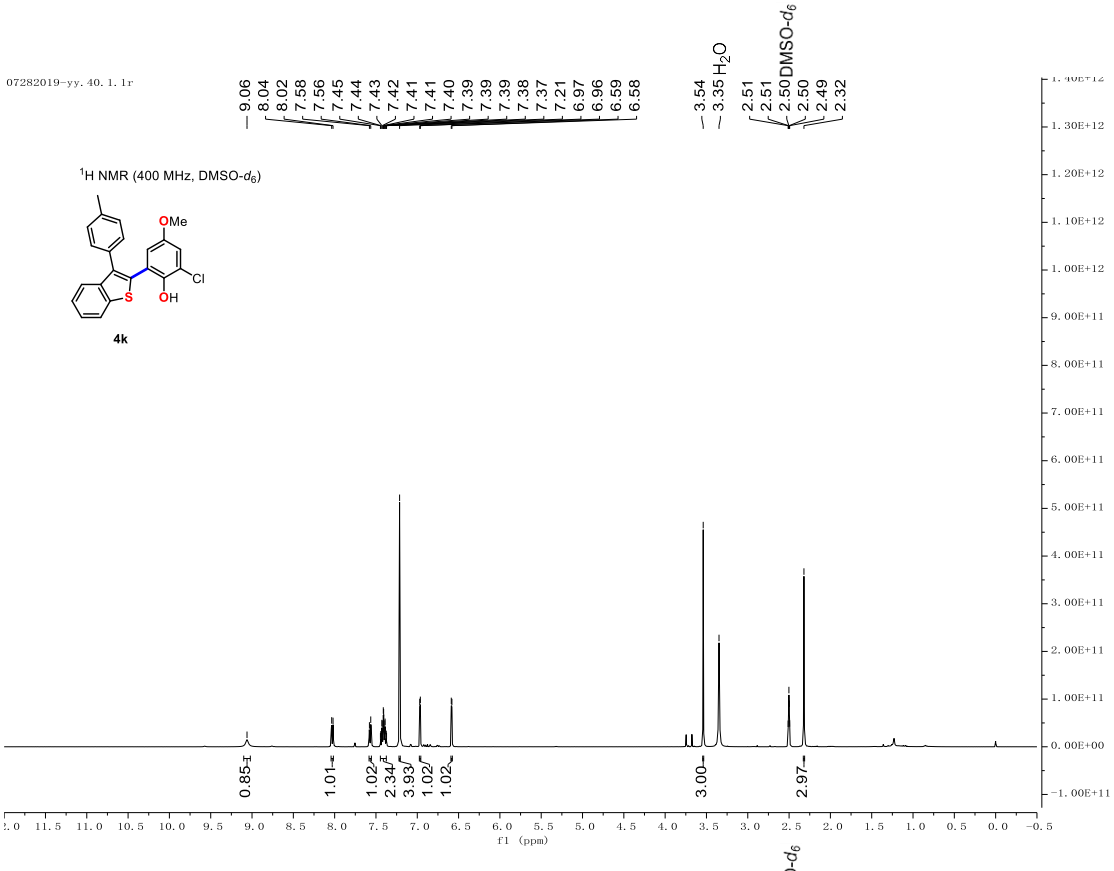

^{13}C{^{1}H} NMR (151 MHz, CDCl$_3$)
4h
CDCl$_3$
55.7
16.5
f1 (ppm)

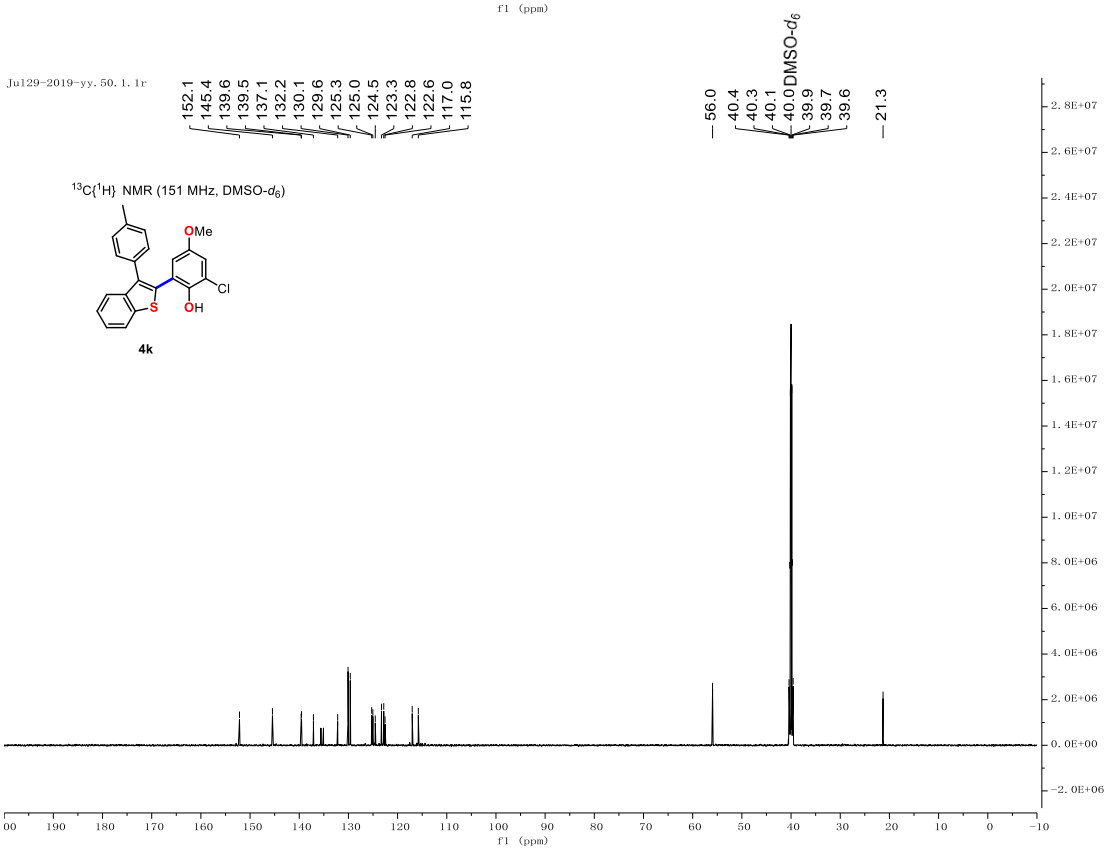

07212019-yy. 20.2
7.92
7.91
7.91
7.90
7.90
7.84
7.83
7.82
7.42
7.42
7.41
7.41
7.41
7.40
7.26 CDCl3
7.24
7.24
7.19
7.17
6.92
6.91
6.78
6.78
6.77
6.77
5.08
3.75
2.37
1.29
1H NMR (400 MHz, CDCl3)
OMe
tBu
S
OH
4i
1.02
1.01
1.98
2.28
2.11
1.06
1.05
1.01
3.00
3.07
9.08
f1 (ppm)

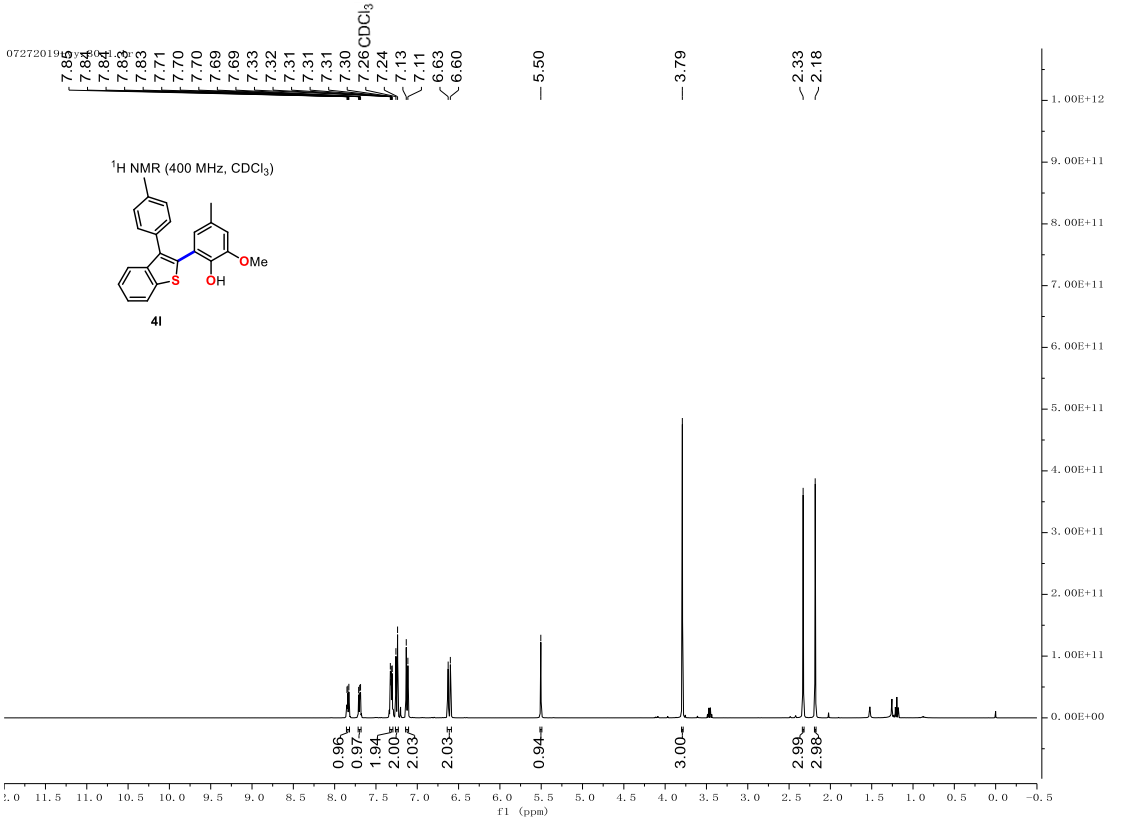

07212019. 20. 1. 1r
152.5
145.9
140.1
139.5
138.3
137.7
135.5
134.8
131.1
129.5
129.3
125.0
124.8
123.5
122.4
120.8
115.3
112.6
77.4
77.1 CDCl3
76.8
55.7
35.0
29.2
21.3
13C{1H} NMR (101 MHz, CDCl3)
OMe
tBu
S
OH
4i
f1 (ppm)

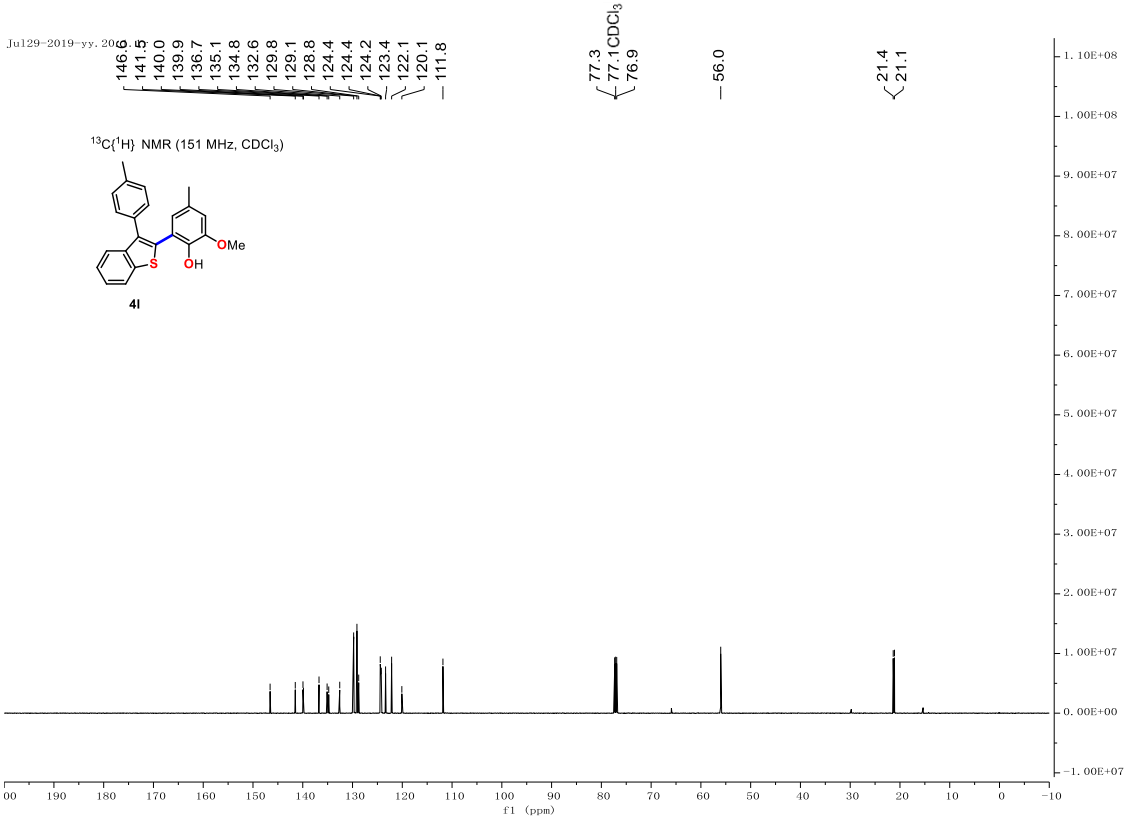

7.93
7.92
7.77
7.76
7.44
7.43
7.42
7.32
7.31
7.30
CDCl3
7.26
7.07
7.06
7.04
6.91
6.90
6.73
5.04
3.75
1.28
1H NMR (600 MHz, CDCl3)
F
OMe
tBu
S
OH
4j
0.99
1.01
1.97
2.09
2.21
1.02
0.95
0.91
2.93
9.00
f1 (ppm)

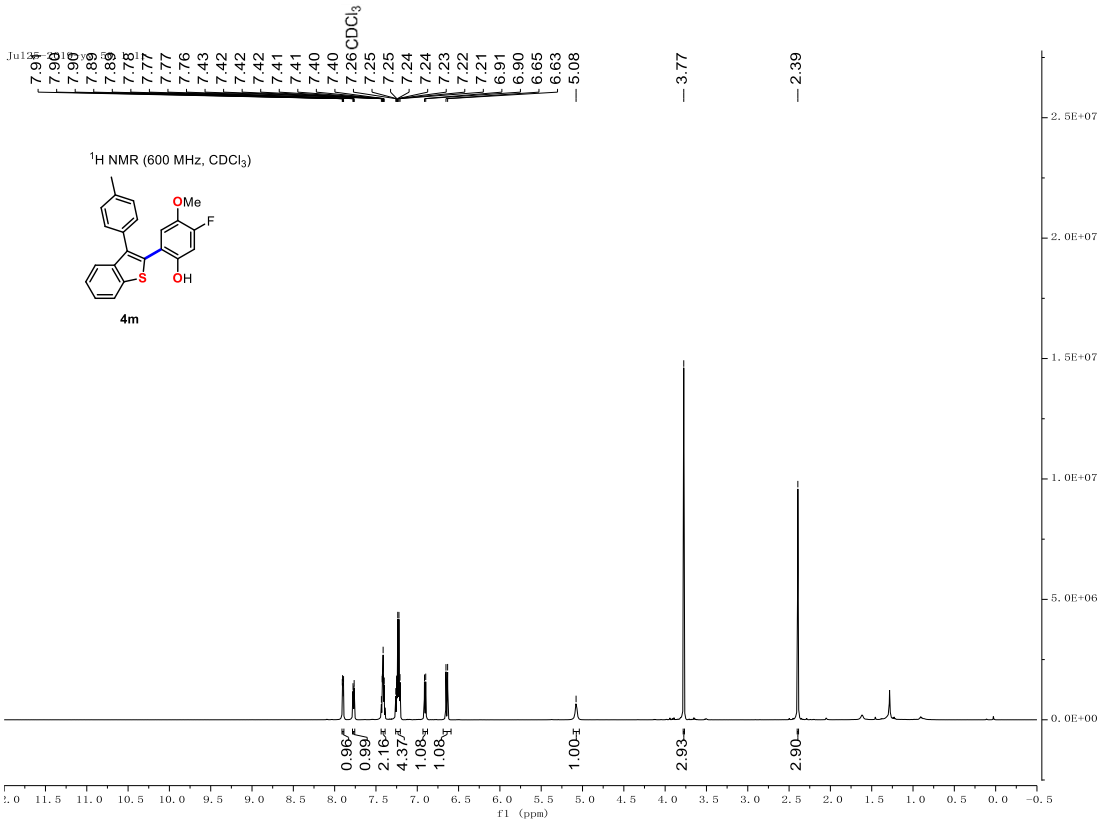

Aug26-2019-wzx.60.1.1r
163.2
161.6
152.5
145.8
140.0
139.3
138.3
135.1
134.6
131.2
131.2
130.1
130.1
125.1
124.9
123.2
122.5
120.2
115.8
115.7
115.4
112.5
77.3
77.1 CDCl3
76.9
55.7
35.0
29.2
13C{1H} NMR (151 MHz, CDCl3)
F
OMe
tBu
S
OH
4j
f1 (ppm)

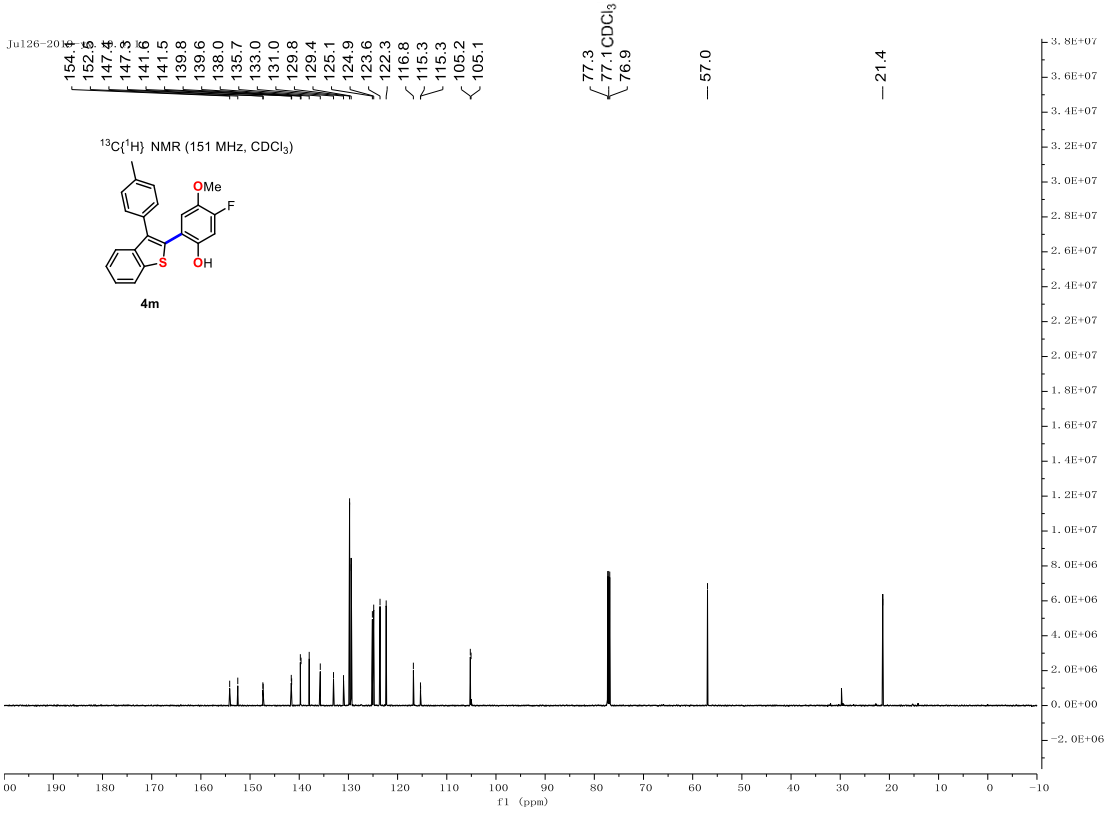

07282019-yy. 40. 1. 1r
9.06
8.04
8.02
7.58
7.56
7.45
7.44
7.43
7.42
7.41
7.41
7.40
7.39
7.39
7.39
7.38
7.37
7.21
6.97
6.96
6.59
6.58
3.54
3.35 H2O
2.51
2.51
2.50 DMSO-d6
2.50
2.49
2.32
1H NMR (400 MHz, DMSO-d6)
OMe
Cl
S
OH
4k
0.85
1.01
1.02
2.34
3.93
1.02
1.02
3.00
2.97
f1 (ppm)

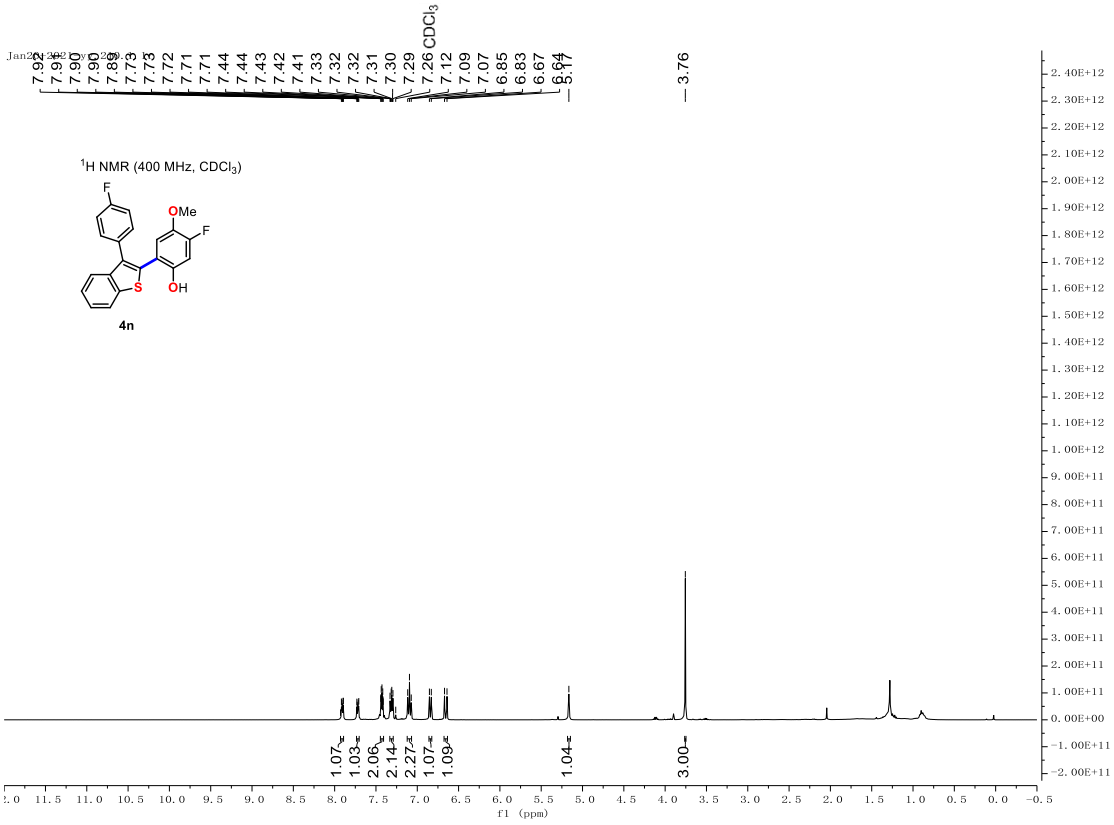

Ju129-2019-yy. 50. 1. 1r
152.1
145.4
139.6
139.5
137.1
132.2
130.1
129.6
125.3
125.0
124.5
123.3
122.8
122.6
117.0
115.8
56.0
40.4
40.3
40.1
40.0 DMSO-d6
39.9
39.7
39.6
21.3
13C{1H} NMR (151 MHz, DMSO-d6)
OMe
Cl
S
OH
4k
f1 (ppm)

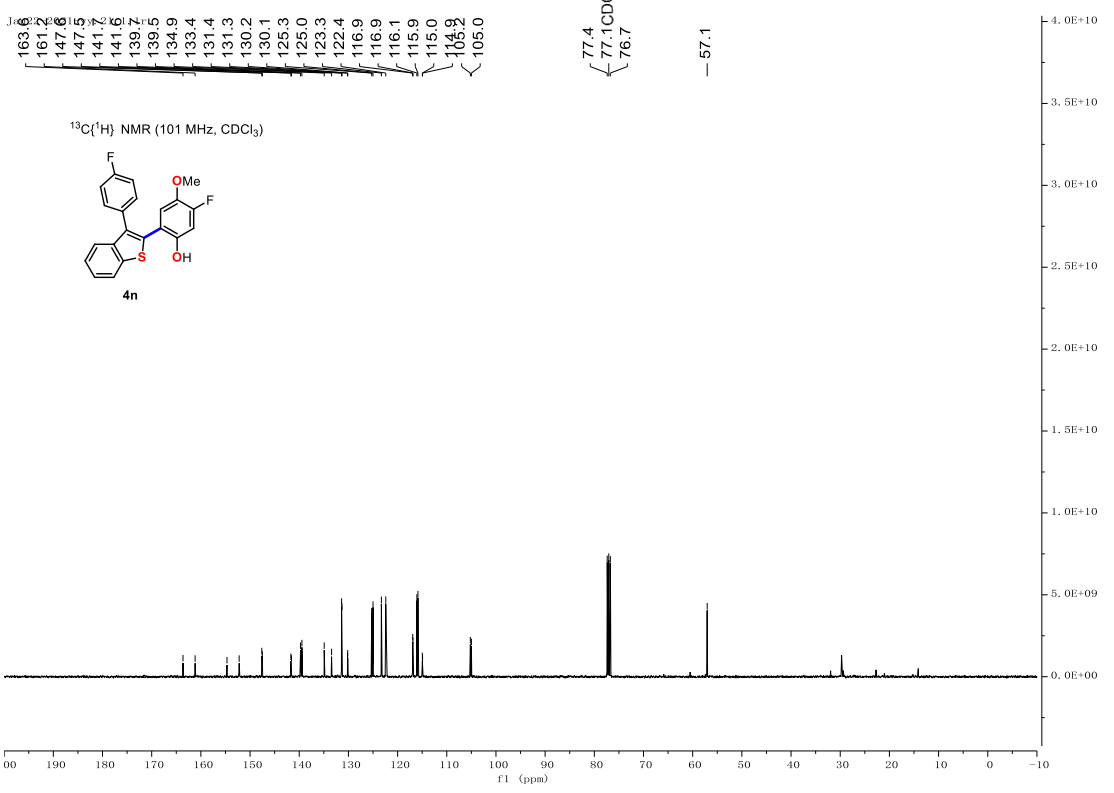

7.85
7.84
7.84
7.83
7.83
7.71
7.70
7.70
7.69
7.69
7.33
7.32
7.31
7.31
7.31
7.30
7.26 CDCl3
7.24
7.13
7.11
6.63
6.60
5.50
3.79
2.33
2.18
1H NMR (400 MHz, CDCl3)
OMe
OH
S
4l
0.96
0.97
1.94
2.00
2.03
2.03
0.94
3.00
2.99
2.98
f1 (ppm)

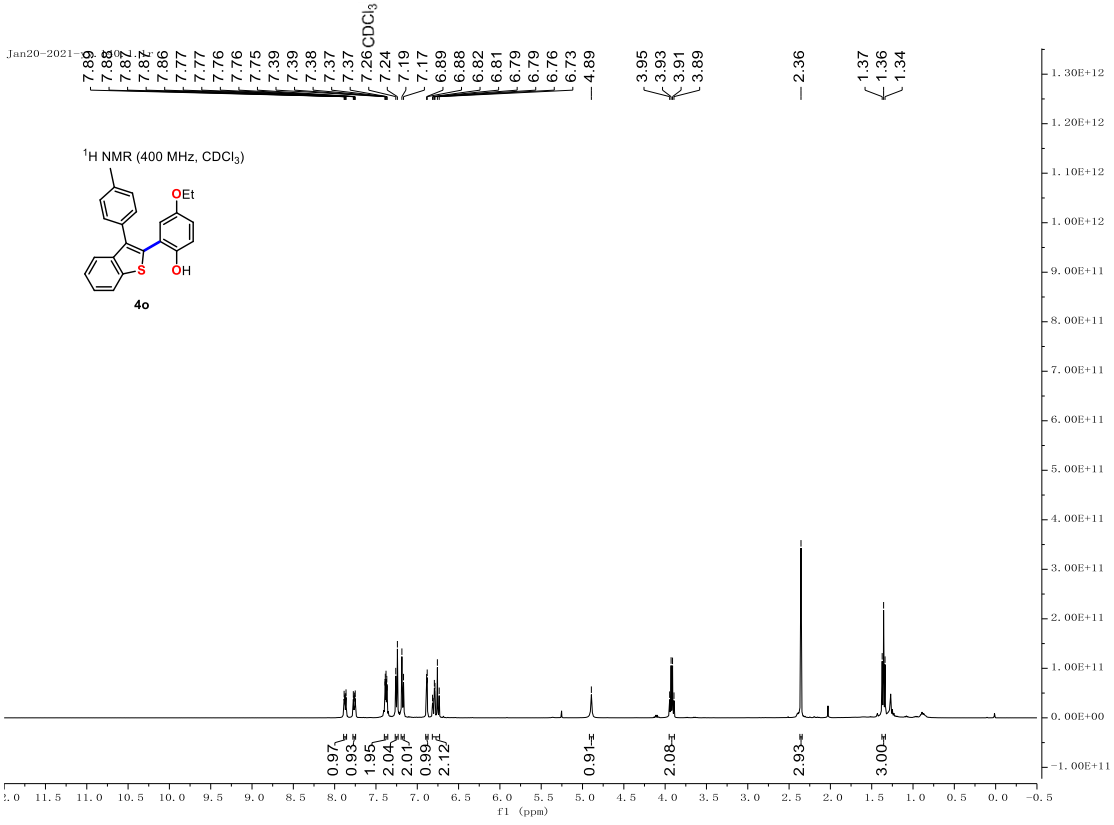

146.6
141.5
140.0
139.9
136.7
135.1
134.8
132.6
129.8
129.1
128.8
124.4
124.4
124.2
123.4
122.1
120.1
111.8
77.3
77.1 CDCl3
76.9
56.0
21.4
21.1
13C{1H} NMR (151 MHz, CDCl3)
OMe
OH
S
4l
f1 (ppm)

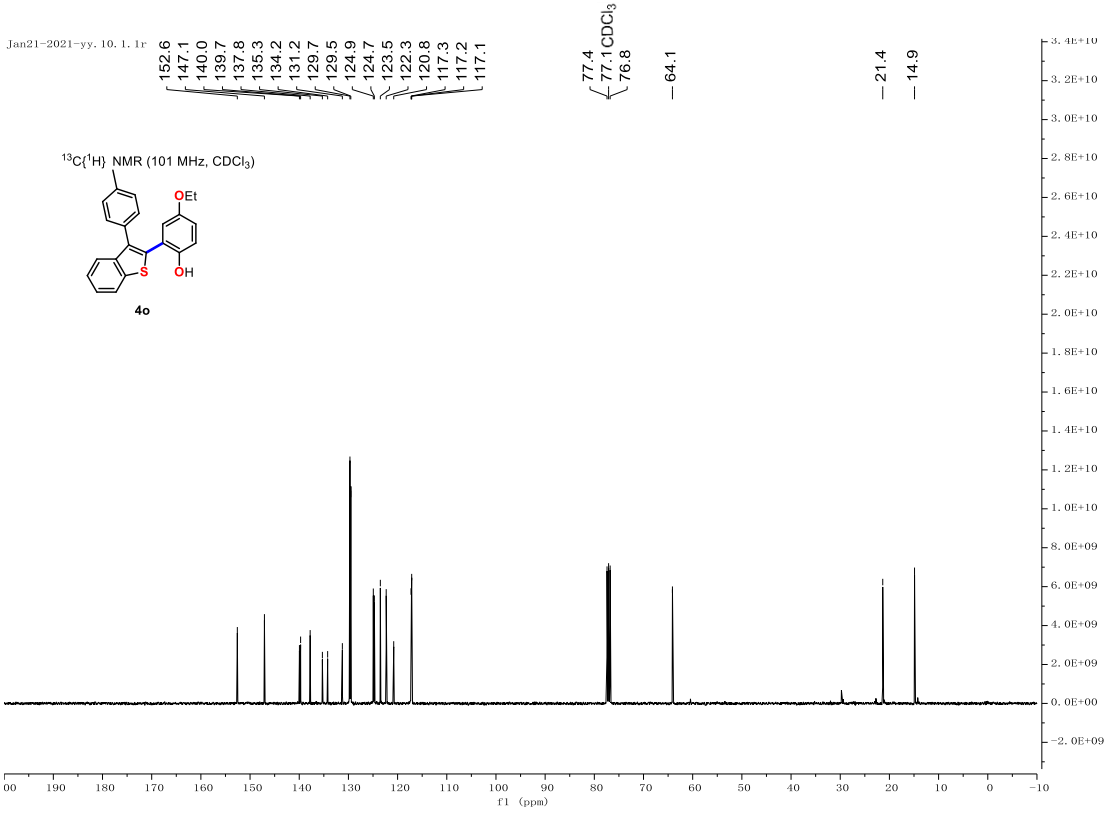

^{1}H NMR (600 MHz, CDCl$_3$)
OMe
F
S
OH
4m
CDCl$_3$
7.26
5.08
3.77
2.39
0.96
0.99
2.16
4.37
1.08
1.08
1.00
2.93
2.90
f1 (ppm)

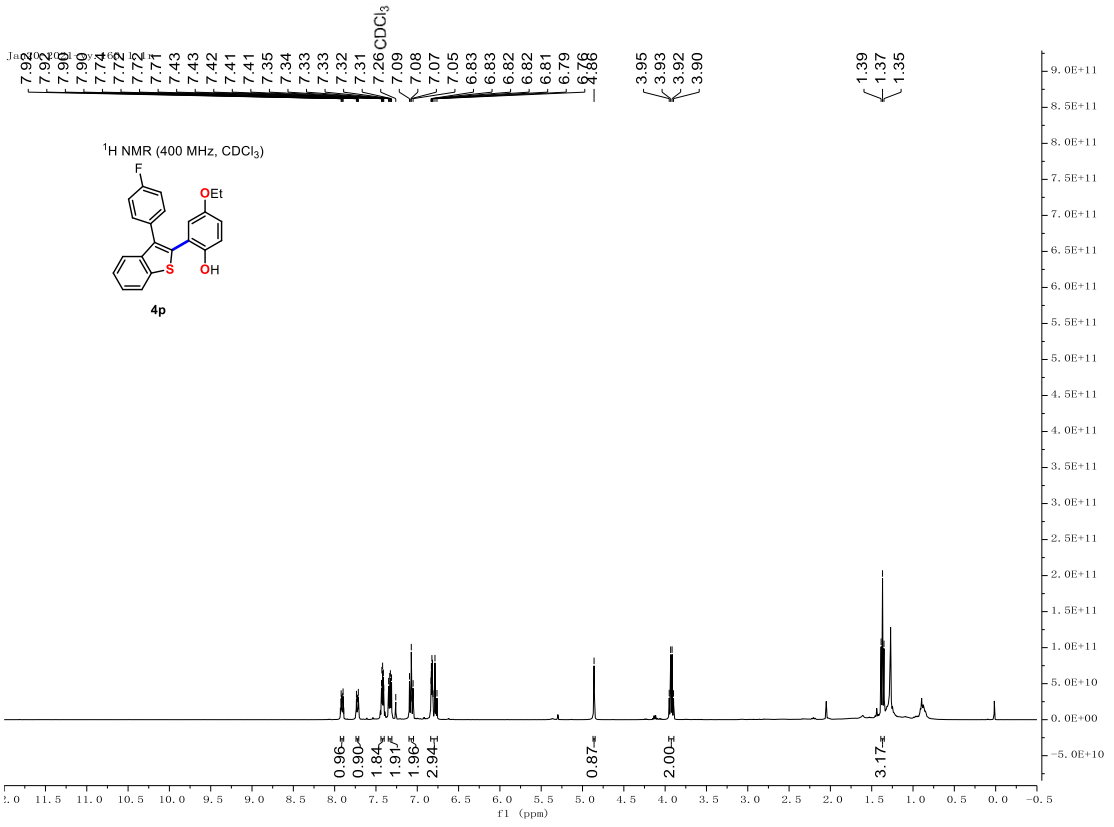

^{13}C{^{1}H} NMR (151 MHz, CDCl$_3$)
OMe
F
S
OH
4m
CDCl$_3$
77.3
77.1
76.9
57.0
21.4
f1 (ppm)

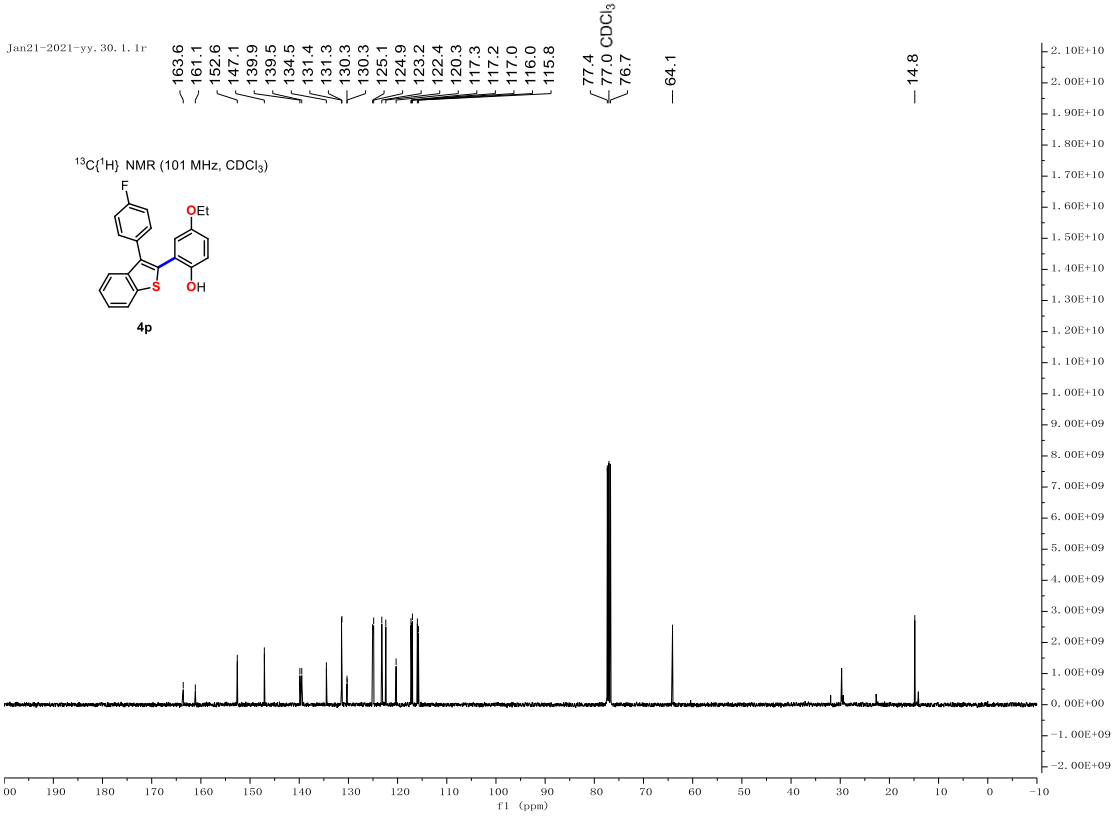

7.92
7.91
7.90
7.90
7.89
7.73
7.73
7.72
7.71
7.71
7.44
7.44
7.43
7.42
7.41
7.33
7.32
7.32
7.31
7.30
7.29
7.26 CDCl3
7.12
7.09
7.07
6.85
6.83
6.67
6.64
5.17
3.76
1H NMR (400 MHz, CDCl3)
F
OMe
F
S
OH
4n
1.07
1.03
2.06
2.14
2.27
1.07
1.09
1.04
3.00
f1 (ppm)

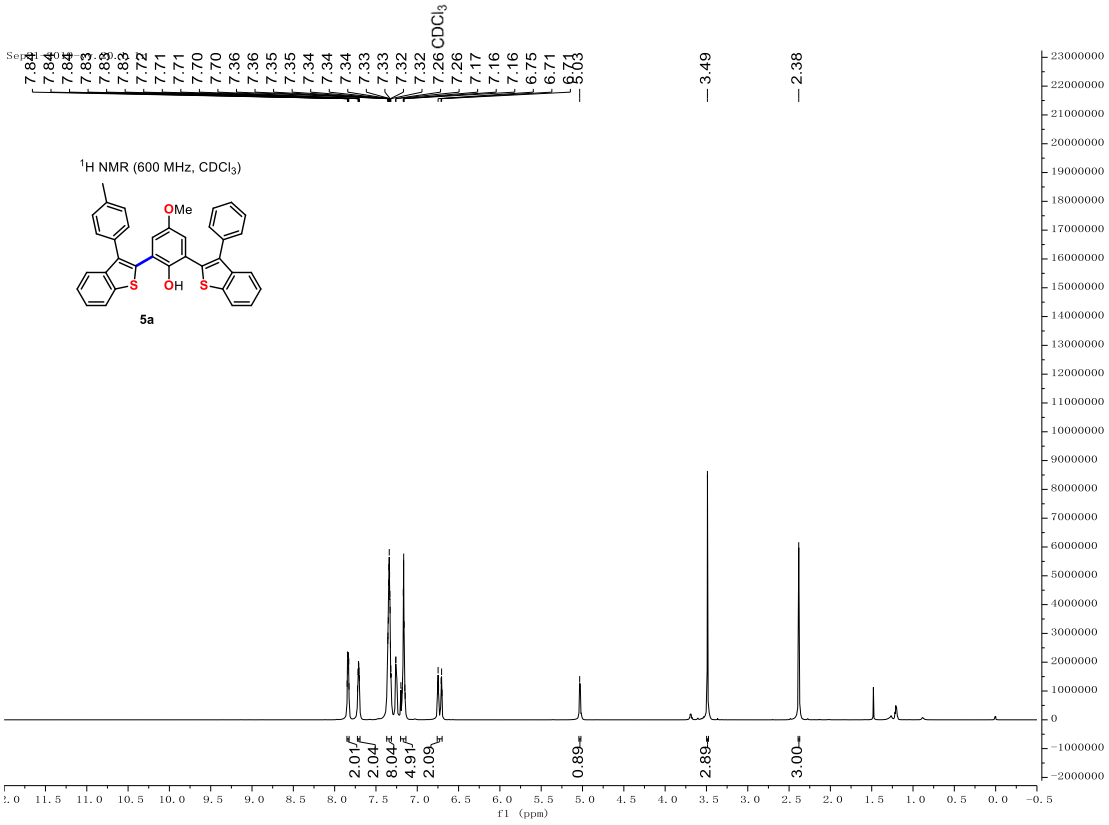

163.6
161.2
147.6
147.5
141.7
141.6
139.7
139.5
134.9
133.4
131.4
131.3
130.2
130.1
125.3
125.0
123.3
122.4
116.9
116.9
116.1
115.9
115.0
114.9
105.2
105.0
77.4
77.1CDCl3
76.7
57.1
13C{1H} NMR (101 MHz, CDCl3)
F
OMe
F
S
OH
4n
f1 (ppm)

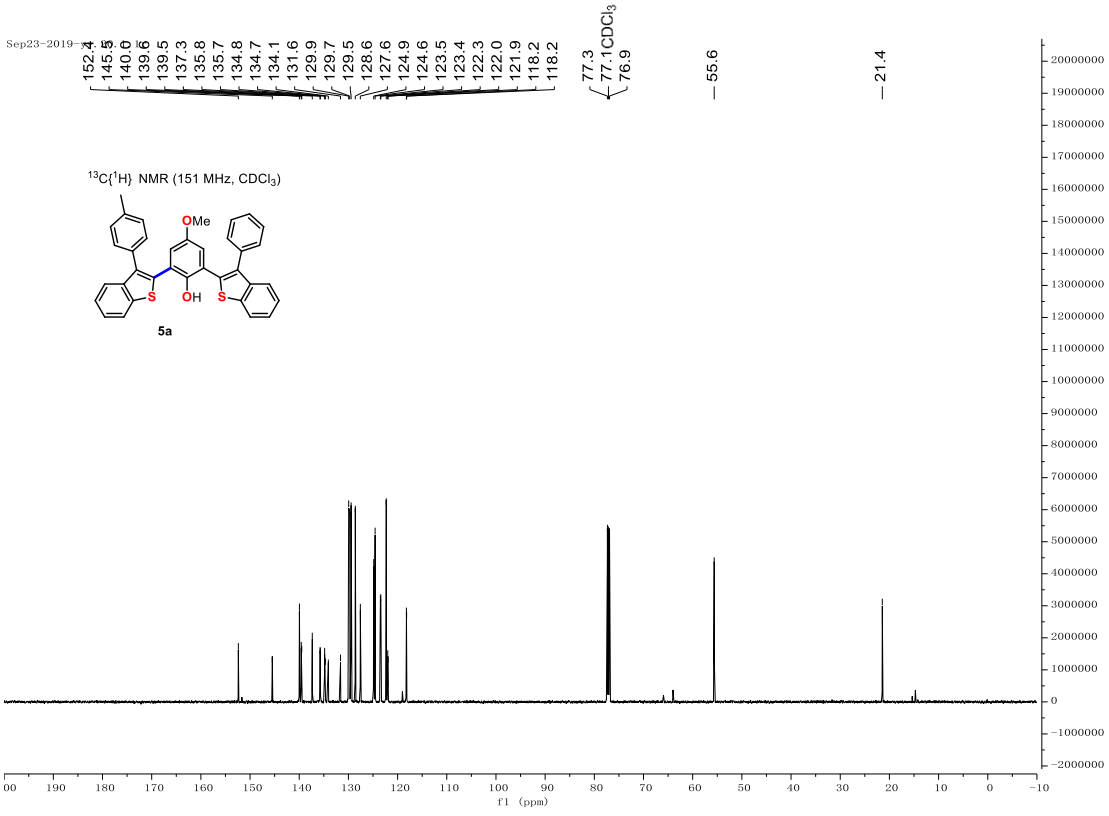

Jan20-2021-yy.10.1.1r
7.89
7.88
7.87
7.87
7.86
7.77
7.77
7.76
7.76
7.75
7.39
7.38
7.37
7.37
7.26 CDCl3
7.24
7.19
7.17
6.89
6.88
6.82
6.81
6.79
6.79
6.76
6.73
4.89
3.95
3.93
3.91
3.89
2.36
1.37
1.36
1.34
1H NMR (400 MHz, CDCl3)
OEt
S
OH
4o
0.97
0.93
1.95
2.04
2.01
0.99
2.12
0.91
2.08
2.93
3.00
f1 (ppm)

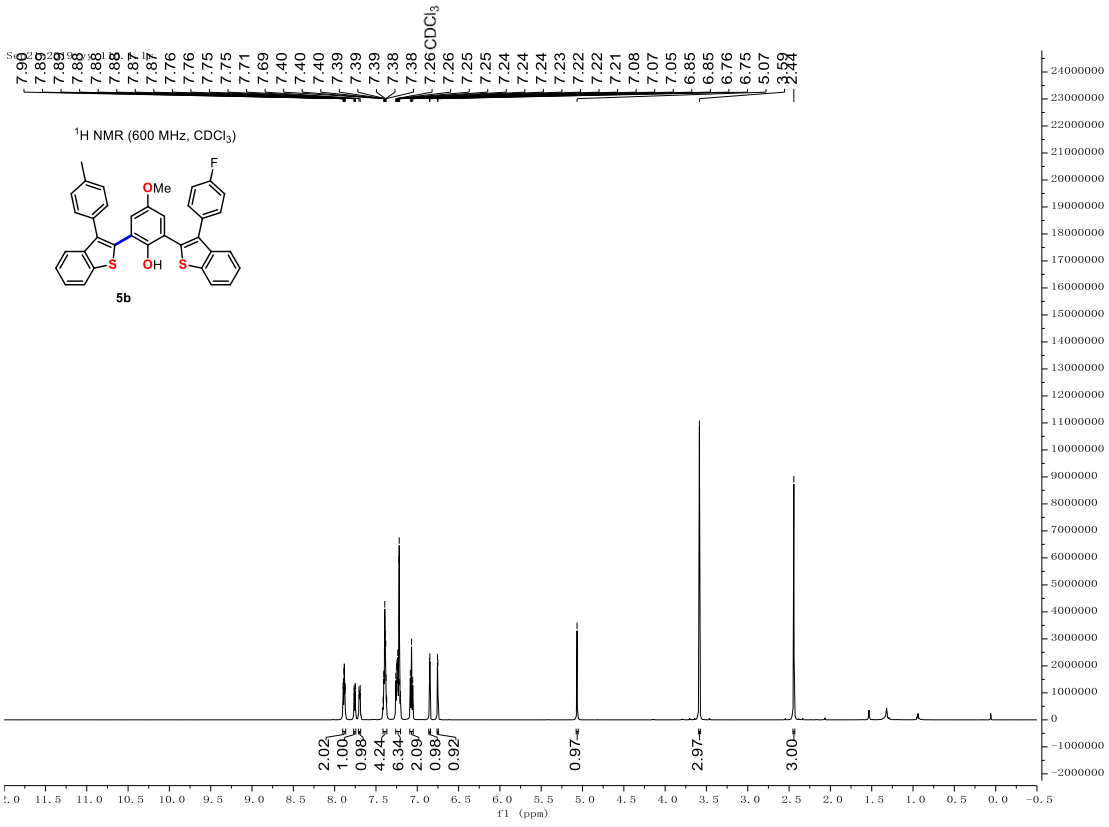

Jan21-2021-yy.10.1.1r
152.6
147.1
140.0
139.7
137.8
135.3
134.2
131.2
129.7
129.5
124.9
124.7
123.5
122.3
120.8
117.3
117.2
117.1
77.4
77.1 CDCl3
76.8
64.1
21.4
14.9
13C{1H} NMR (101 MHz, CDCl3)
OEt
S
OH
4o
f1 (ppm)

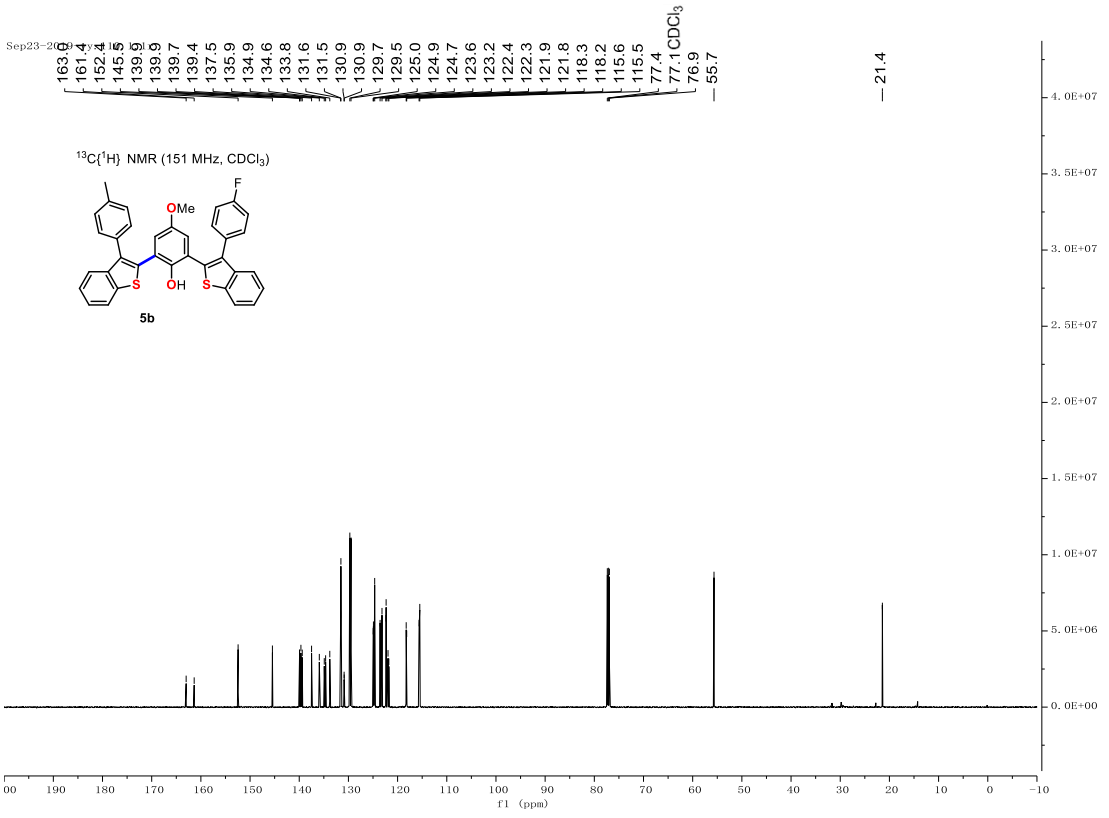

7.92
7.91
7.90
7.90
7.74
7.72
7.72
7.71
7.43
7.43
7.42
7.41
7.41
7.35
7.34
7.33
7.33
7.32
7.31
7.26 CDCl3
7.09
7.08
7.07
7.05
6.83
6.83
6.82
6.82
6.81
6.79
6.76
4.86
3.95
3.93
3.92
3.90
1.39
1.37
1.35
1H NMR (400 MHz, CDCl3)
F
OEt
S
OH
4p
0.96
0.90
1.84
1.91
1.96
2.94
0.87
2.00
3.17
f1 (ppm)

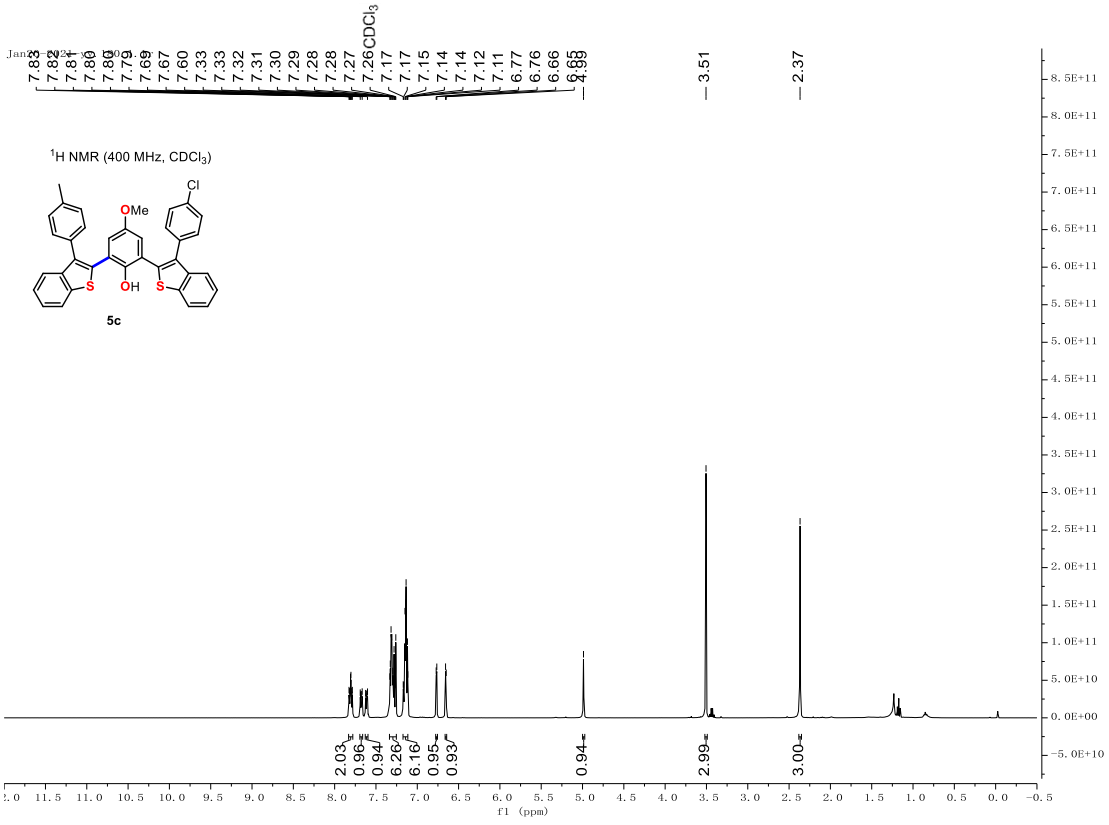

Jan21-2021-yy.30.1.1r
163.6
161.1
152.6
147.1
139.9
139.5
134.5
131.4
131.3
130.3
130.3
125.1
124.9
123.2
122.4
120.3
117.3
117.2
117.0
116.0
115.8
77.4
77.0 CDCl3
76.7
64.1
14.8
13C{1H} NMR (101 MHz, CDCl3)
F
OEt
S
OH
4p
f1 (ppm)

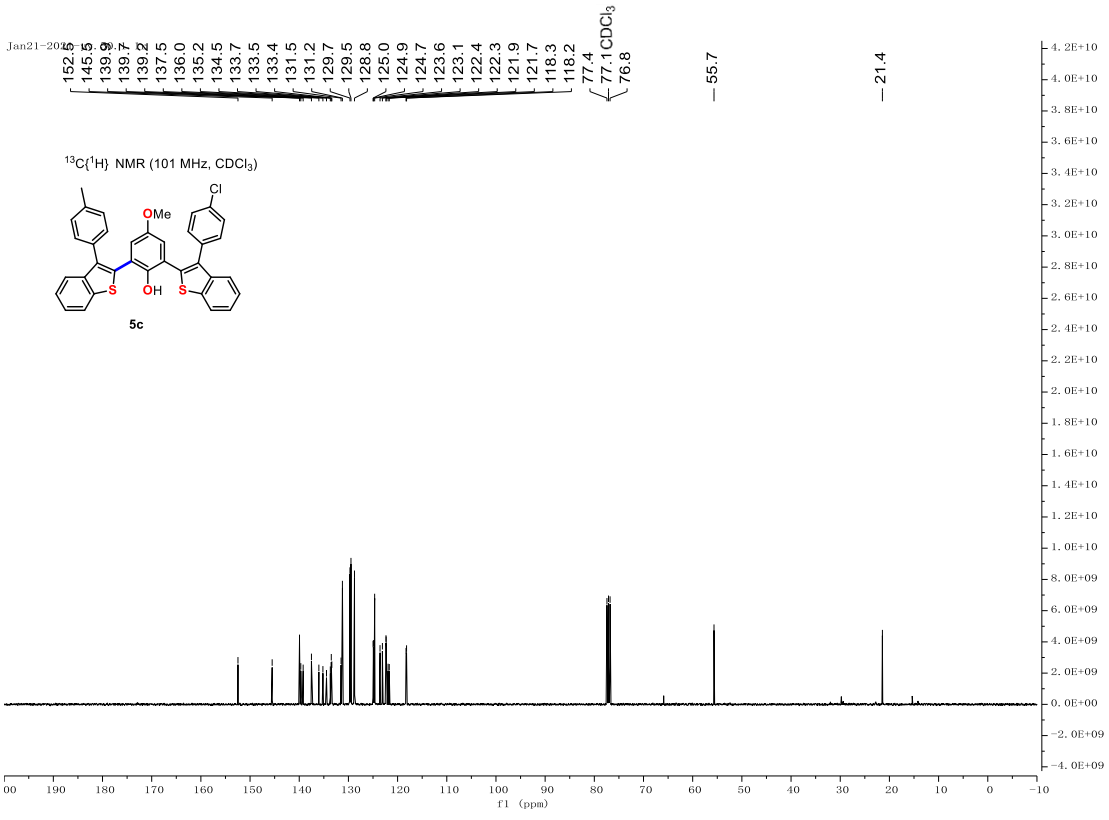

^{1}H NMR (600 MHz, CDCl$_3$)
OMe
OH
S
S
5a
CDCl$_3$
3.49
2.38
2.01
2.04
8.04
4.91
2.09
0.89
2.89
3.00
f1 (ppm)

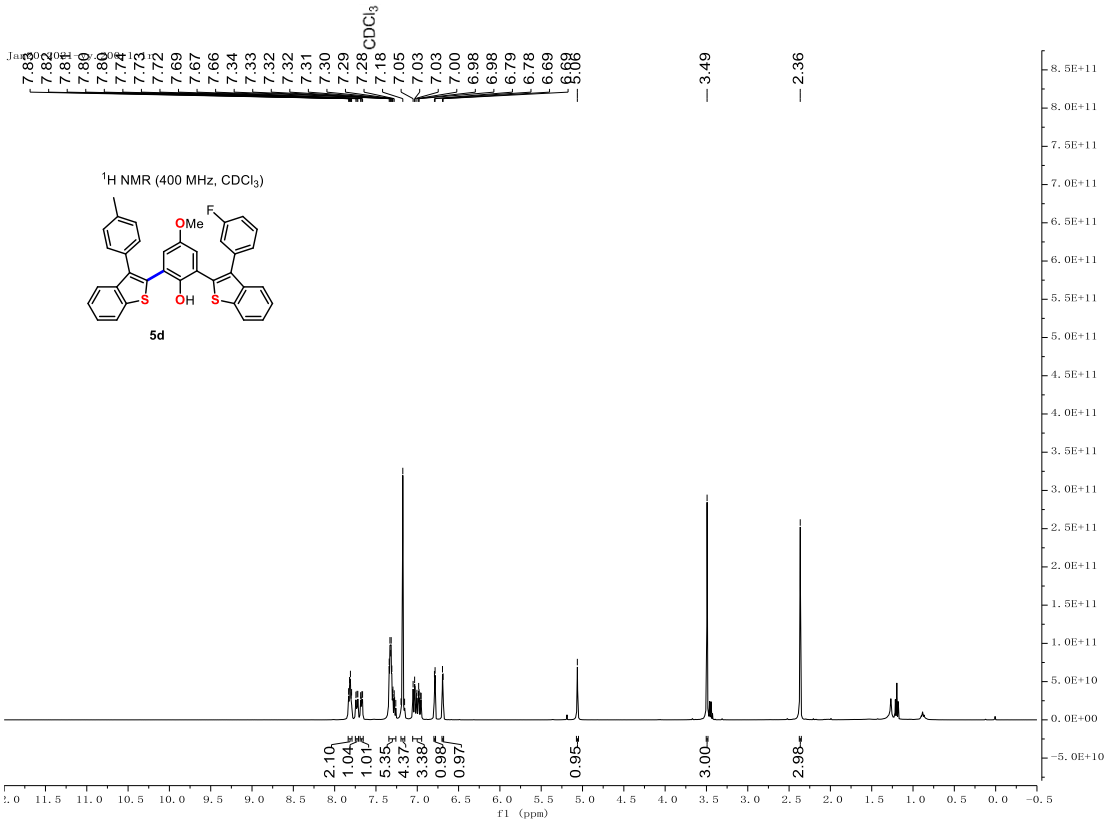

^{13}C{^{1}H} NMR (151 MHz, CDCl$_3$)
OMe
OH
S
S
5a
77.3
77.1
CDCl$_3$
76.9
55.6
21.4
f1 (ppm)

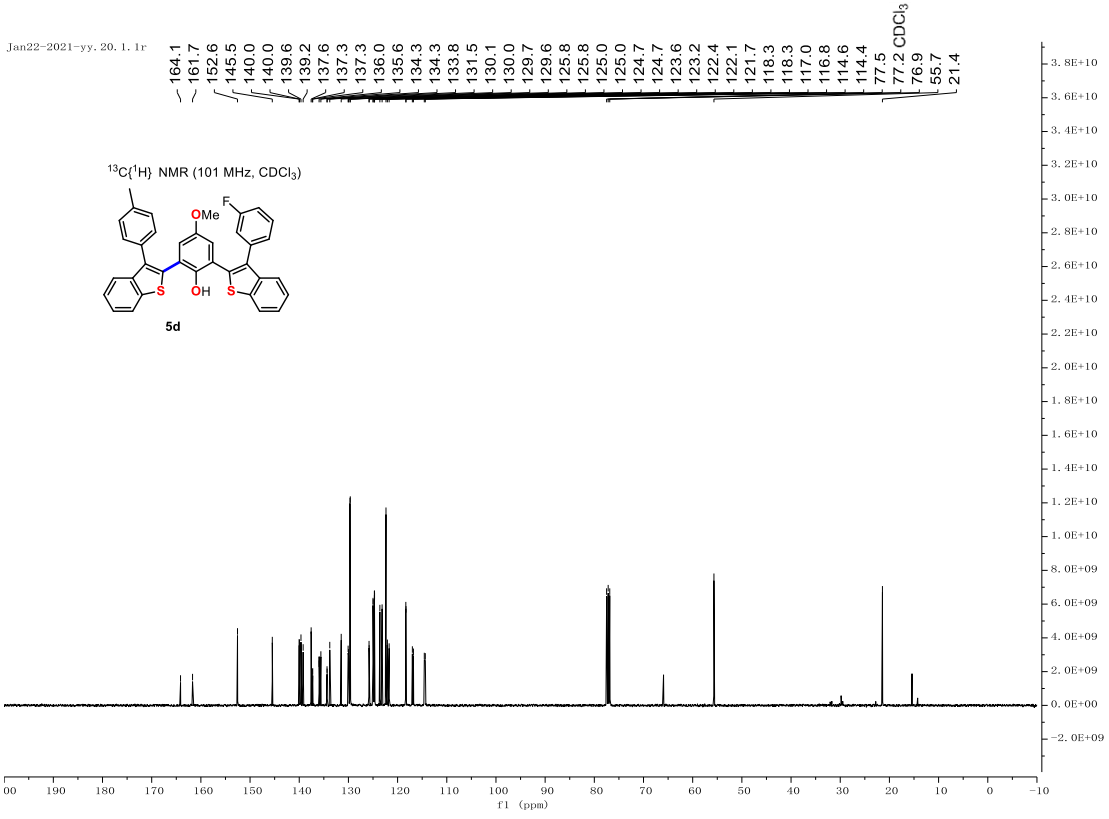

^{1}H NMR (600 MHz, CDCl$_3$)
OMe
F
OH
S
S
5b
CDCl$_3$
2.02
1.00
0.98
4.24
6.34
2.09
0.98
0.92
0.97
2.97
3.00
f1 (ppm)

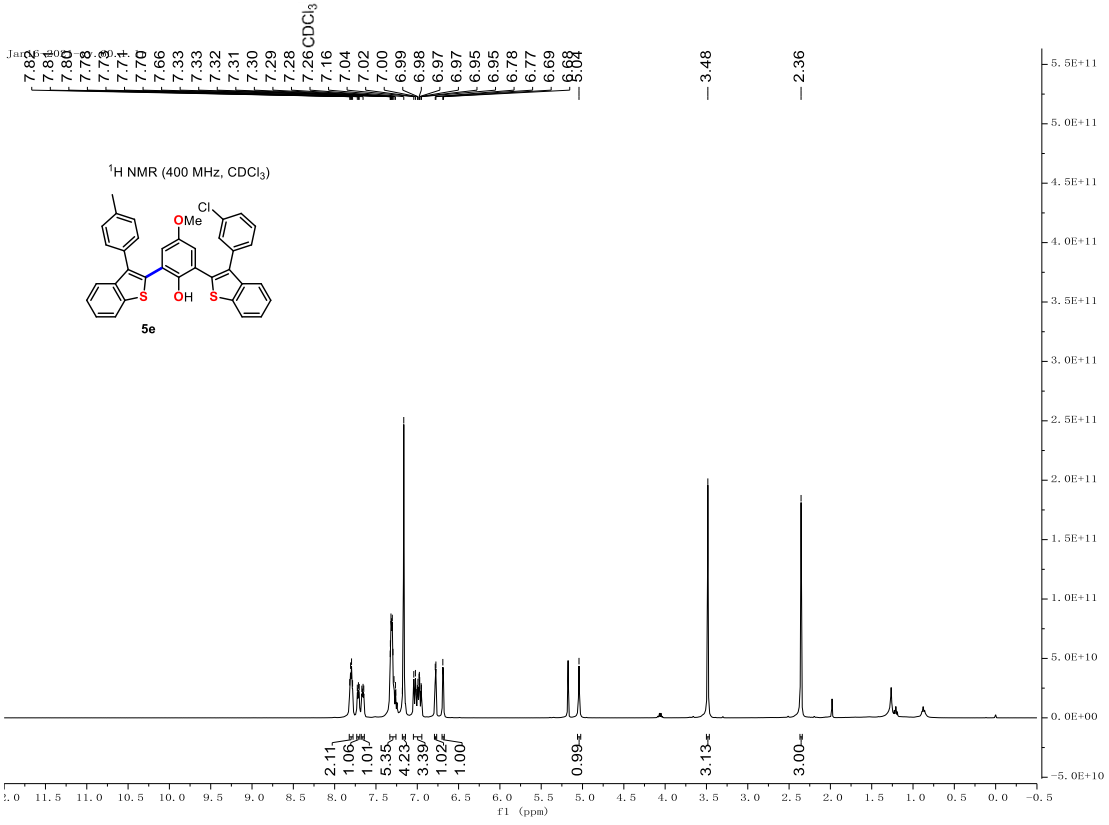

^{13}C{^{1}H} NMR (151 MHz, CDCl$_3$)
OMe
F
OH
S
S
5b
CDCl$_3$
21.4
55.7
f1 (ppm)

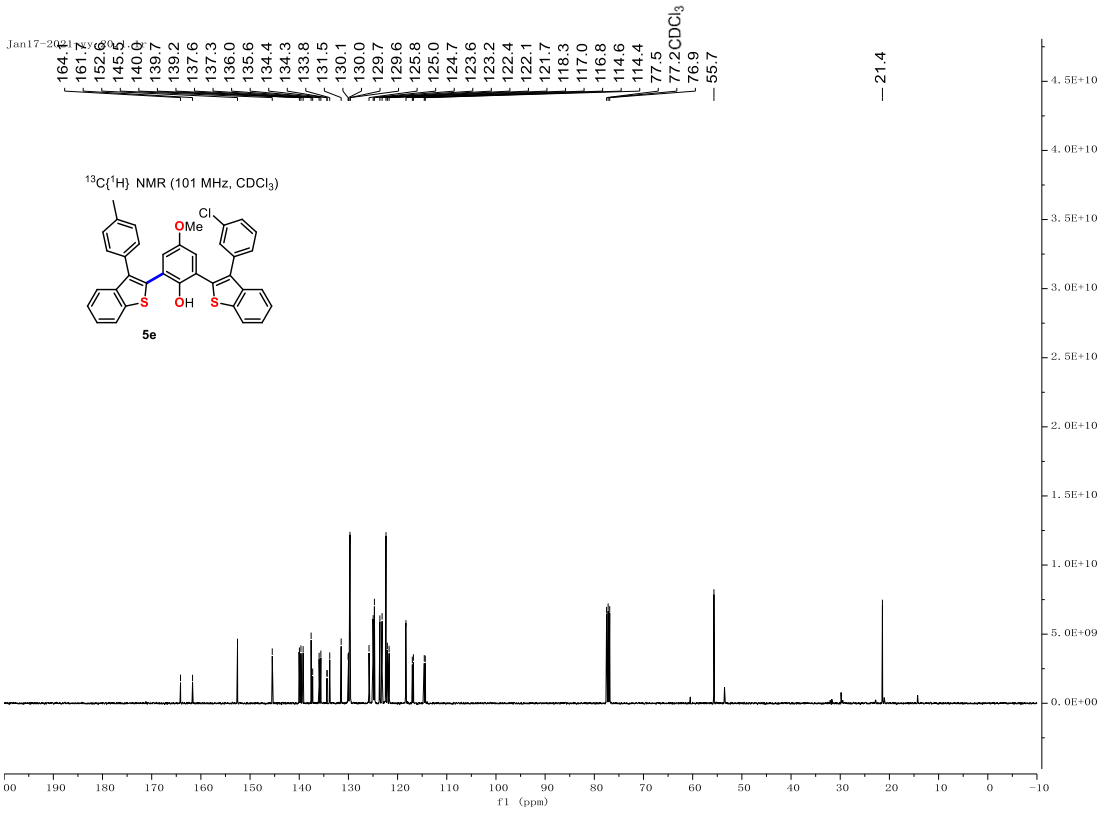

^{1}H NMR (400 MHz, $CDCl_3$)
Cl
OMe
OH
S
S
5c
$CDCl_3$
3.51
2.37
2.03
0.96
0.94
6.26
6.16
0.95
0.93
0.94
2.99
3.00
f1 (ppm)

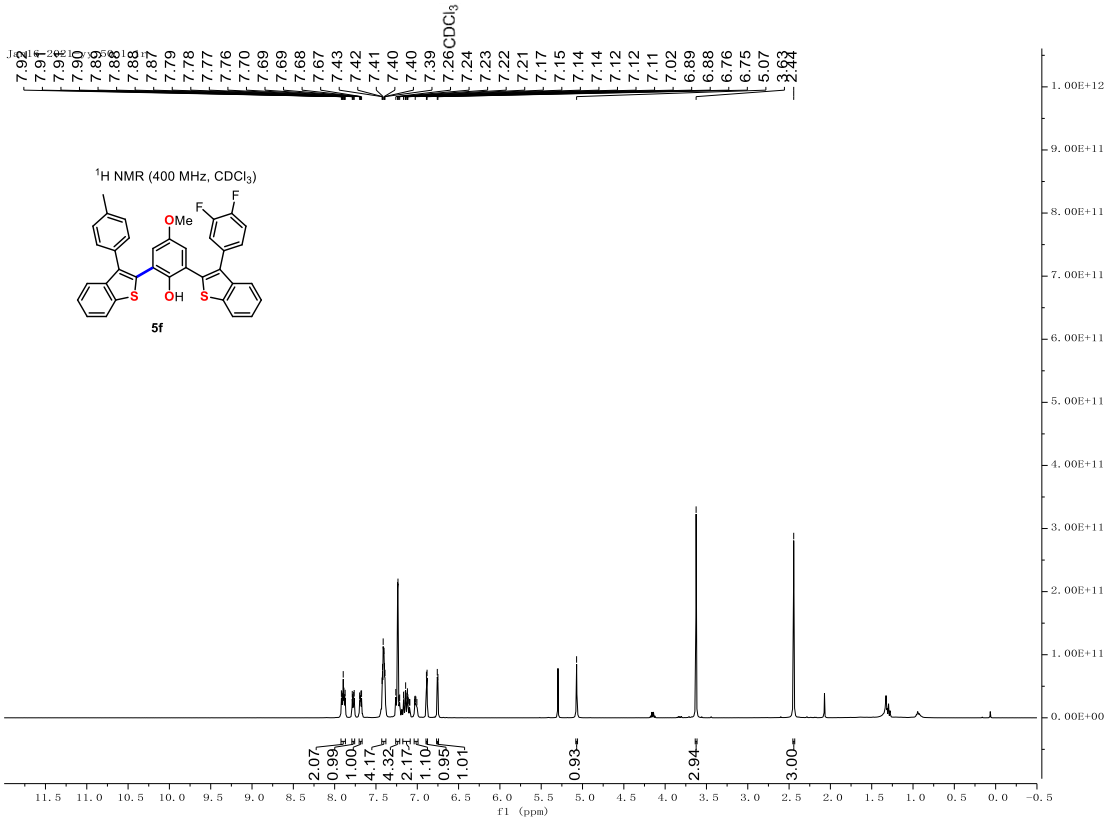

$^{13}C\{^1H\}$ NMR (101 MHz, $CDCl_3$)
Cl
OMe
OH
S
S
5c
$CDCl_3$
55.7
21.4
f1 (ppm)

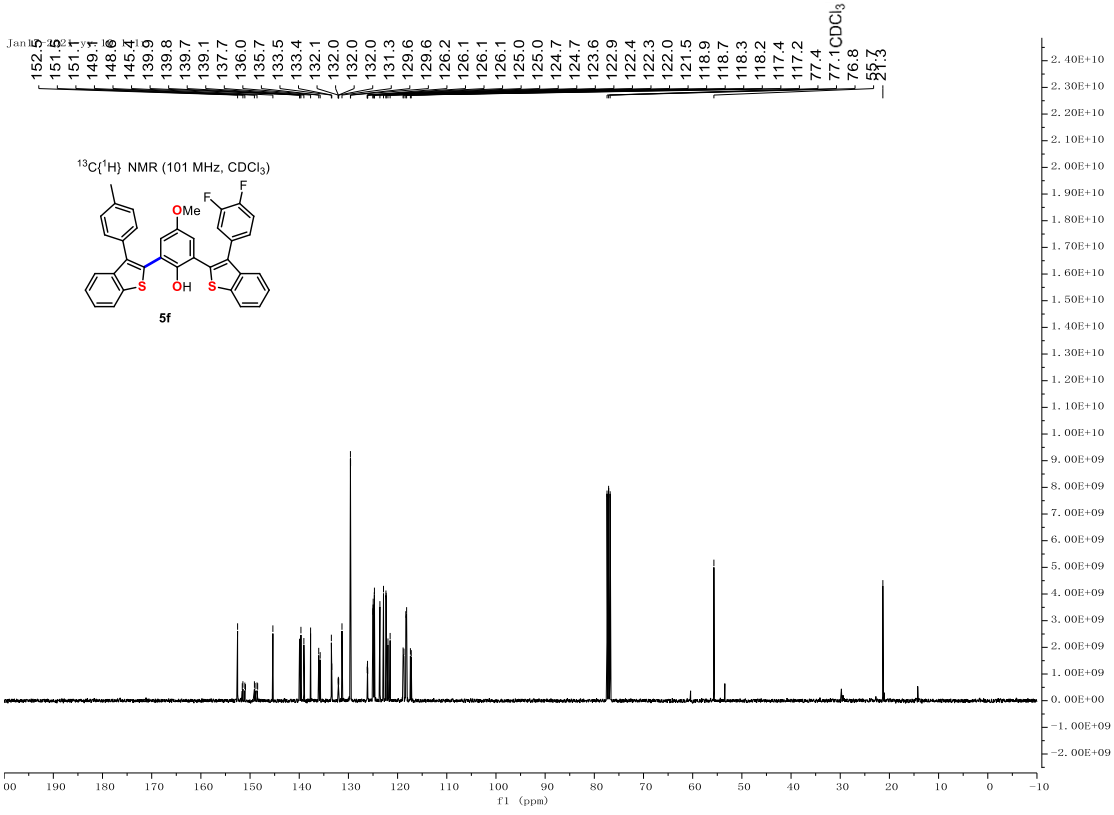

^{1}H NMR (400 MHz, CDCl$_3$)
OMe
F
OH
S
S
5d
CDCl$_3$
3.49
2.36
2.10
1.04
1.01
5.35
4.37
3.38
0.98
0.97
0.95
3.00
2.98
f1 (ppm)

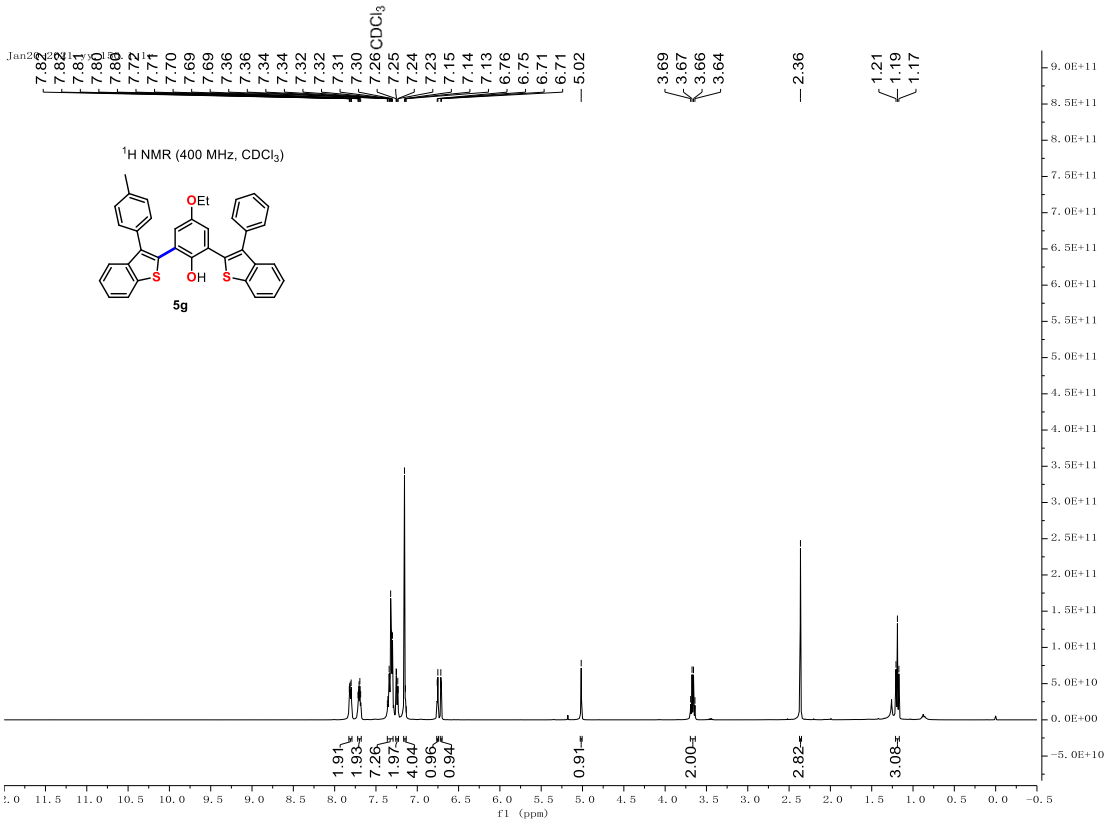

^{13}C{^{1}H} NMR (101 MHz, CDCl$_3$)
OMe
F
OH
S
S
5d
CDCl$_3$
f1 (ppm)

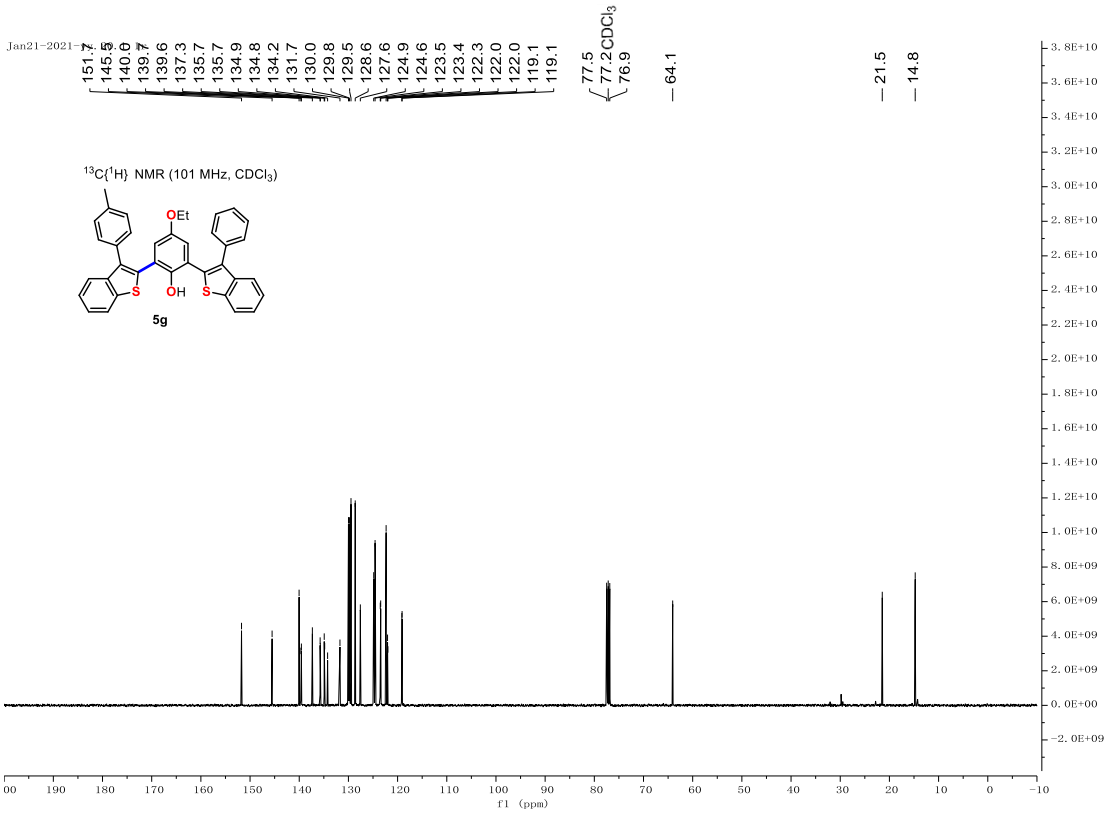

^{1}H NMR (400 MHz, CDCl$_3$)
Cl
OMe
OH
S
S
5e
CDCl$_3$
3.48
2.36
2.11
1.06
1.01
5.35
4.23
3.39
1.02
1.00
0.99
3.13
3.00
f1 (ppm)

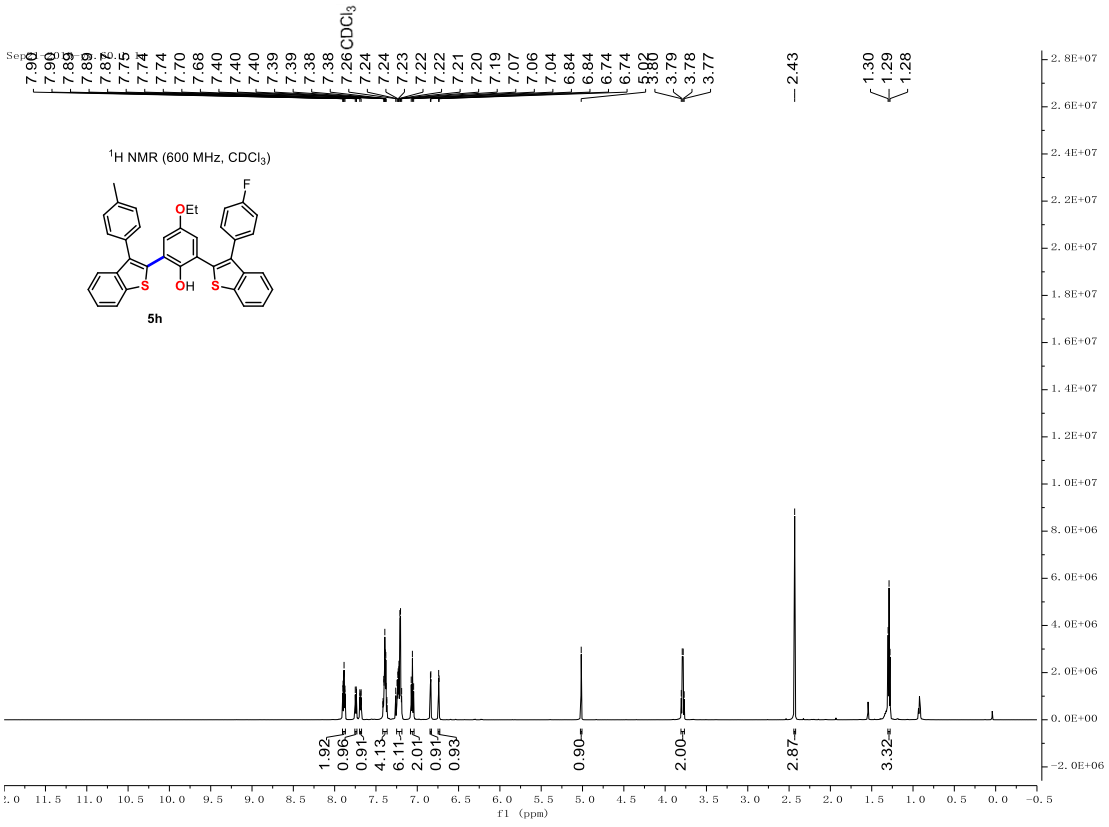

^{13}C{^{1}H} NMR (101 MHz, CDCl$_3$)
Cl
OMe
OH
S
S
5e
CDCl$_3$
55.7
21.4
f1 (ppm)

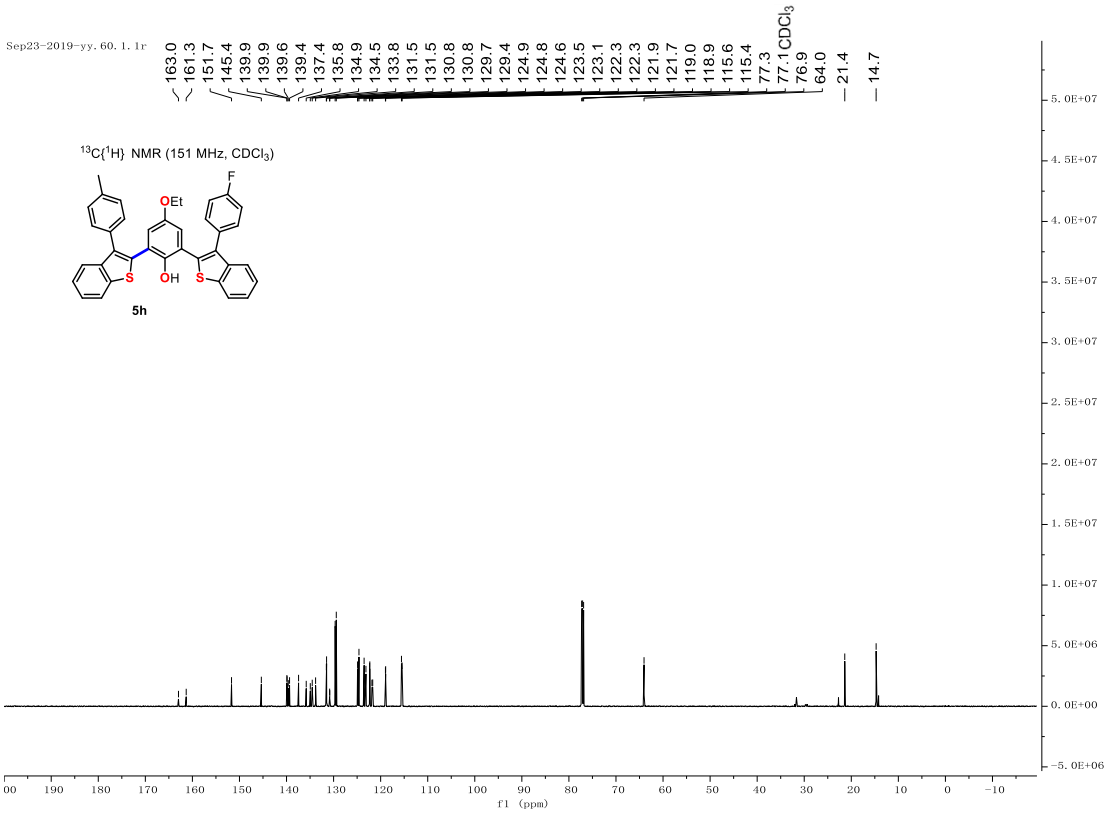

7.92
7.91
7.90
7.89
7.88
7.87
7.79
7.78
7.77
7.76
7.70
7.69
7.69
7.68
7.67
7.43
7.42
7.41
7.40
7.40
7.39
7.26CDCl3
7.24
7.23
7.22
7.21
7.17
7.15
7.14
7.14
7.12
7.12
7.11
7.02
6.89
6.88
6.76
6.75
5.07
3.63
2.44
1H NMR (400 MHz, CDCl3)
OMe
F
F
S
OH
S
5f
2.07
0.99
1.00
4.17
4.32
2.17
1.10
0.95
1.01
0.93
2.94
3.00
f1 (ppm)

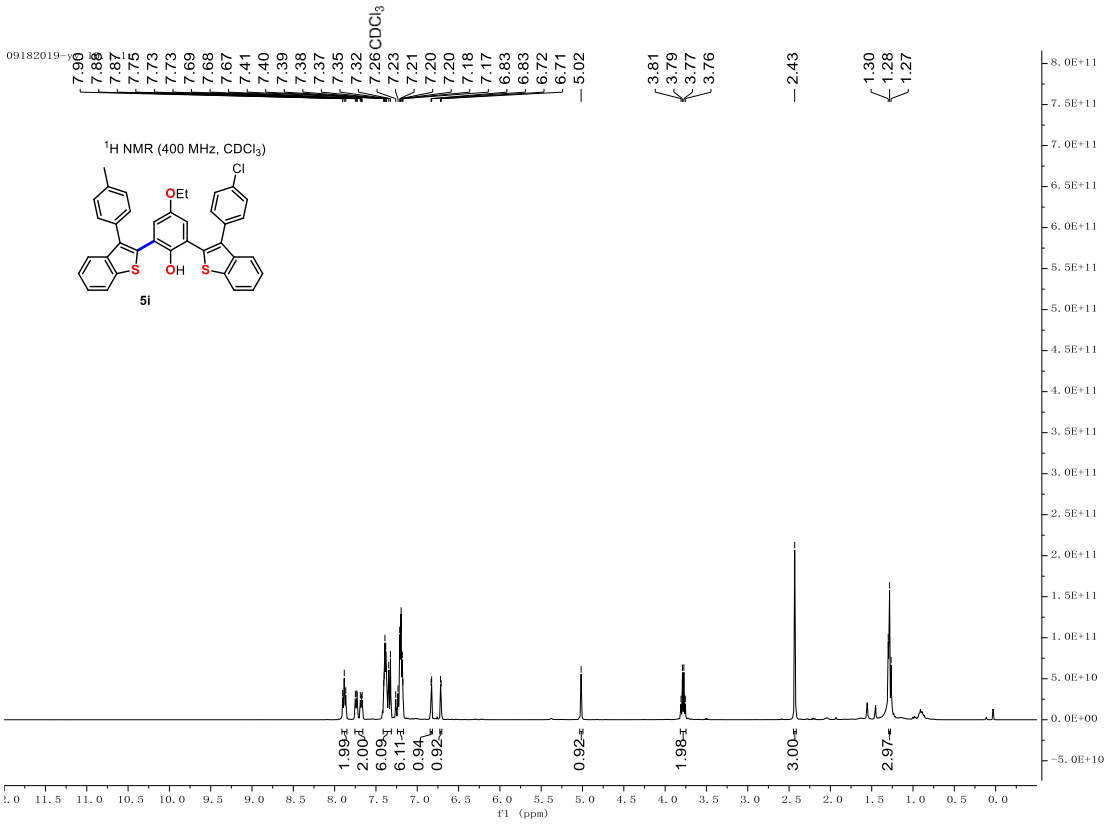

152.5
151.5
151.1
149.1
148.6
145.4
139.9
139.8
139.7
139.1
137.7
136.0
135.7
133.5
133.4
132.1
132.0
132.0
132.0
131.3
129.6
129.6
126.2
126.1
126.1
125.0
125.0
124.7
124.7
123.6
122.9
122.4
122.3
122.0
121.5
118.9
118.7
118.3
118.2
117.4
117.2
77.4
77.1CDCl3
76.8
55.7
21.3
13C{1H} NMR (101 MHz, CDCl3)
OMe
F
F
S
OH
S
5f
f1 (ppm)

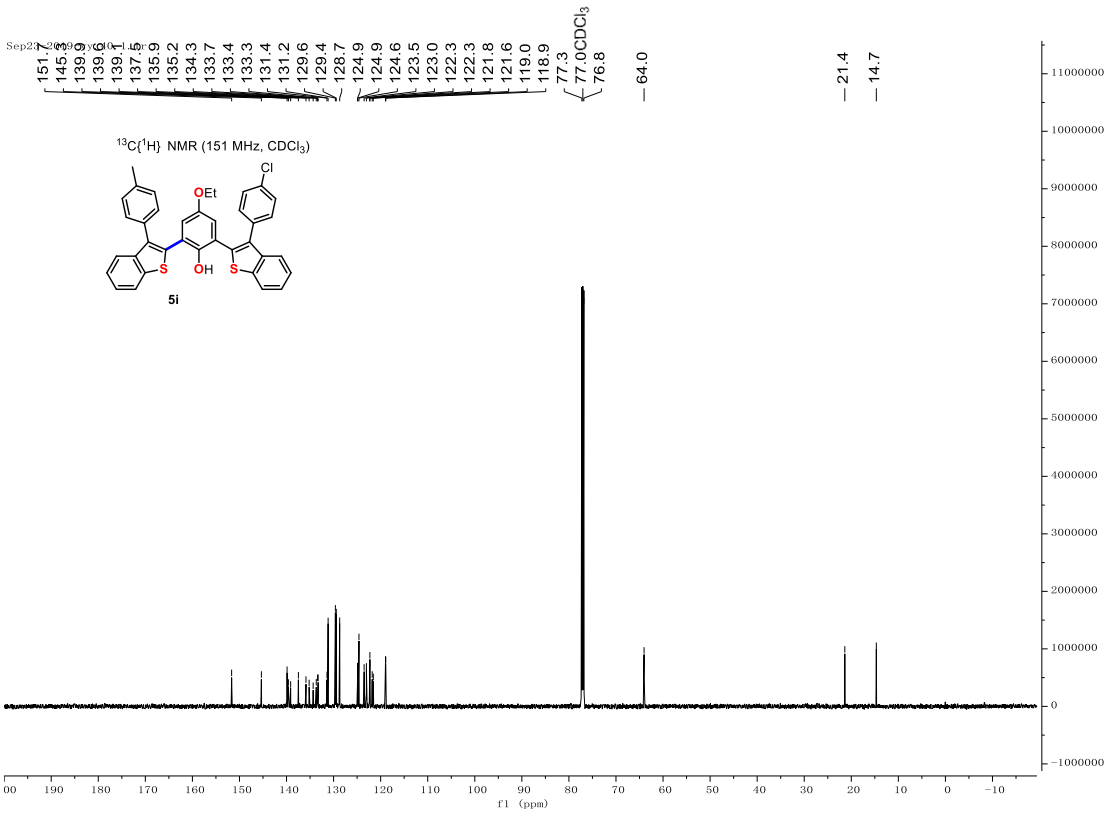

7.82
7.82
7.81
7.80
7.80
7.72
7.71
7.70
7.69
7.69
7.36
7.34
7.34
7.32
7.32
7.31
7.30
7.26 CDCl3
7.25
7.24
7.23
7.15
7.14
7.13
6.76
6.75
6.71
6.71
5.02
3.69
3.67
3.66
3.64
2.36
1.21
1.19
1.17
1H NMR (400 MHz, CDCl3)
OEt
OH
S
S
5g
1.91
1.93
7.26
1.97
4.04
0.96
0.94
0.91
2.00
2.82
3.08
f1 (ppm)

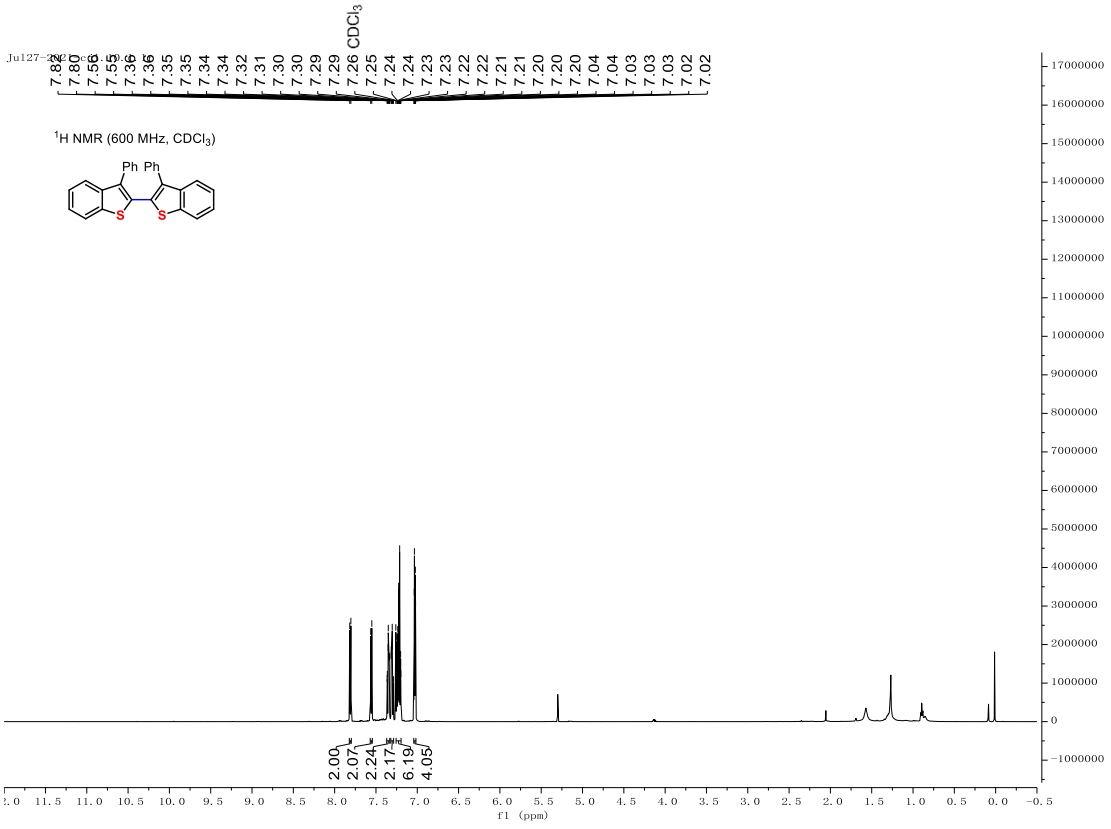

151.7
145.5
140.6
139.7
139.6
137.3
135.7
135.7
134.9
134.8
134.2
131.7
130.0
129.8
129.5
128.6
127.6
124.9
124.6
123.5
123.4
122.3
122.0
122.0
119.1
119.1
77.5
77.2 CDCl3
76.9
64.1
21.5
14.8
13C{1H} NMR (101 MHz, CDCl3)
OEt
OH
S
S
5g
f1 (ppm)

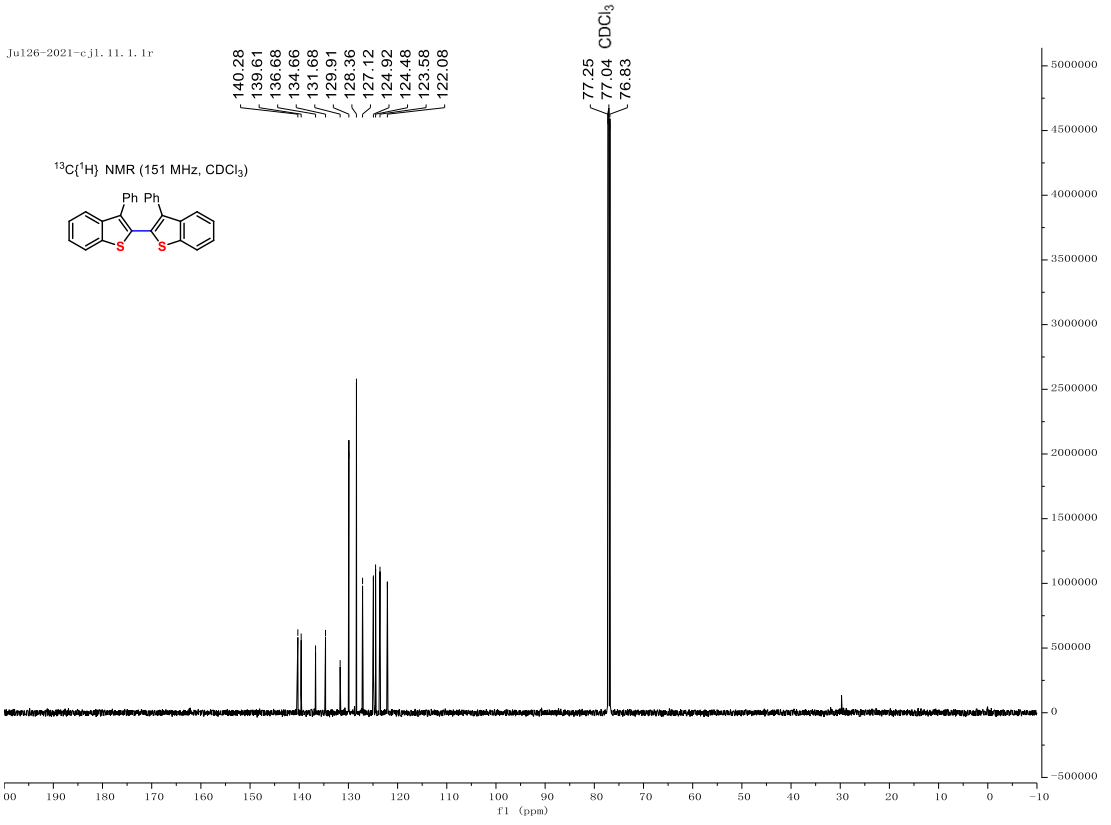

^{1}H NMR (600 MHz, $CDCl_3$)
OEt
F
OH
S
S
5h
CDCl3
2.43
1.30
1.29
1.28
1.92
0.96
0.91
4.13
6.11
2.01
0.91
0.93
0.90
2.00
2.87
3.32
f1 (ppm)


Sep23-2019-yy.60.1.1r
$^{13}C\{^1H\}$ NMR (151 MHz, $CDCl_3$)
OEt
F
OH
S
S
5h
CDCl3
21.4
14.7
f1 (ppm)


^{1}H NMR (400 MHz, CDCl$_3$)
OEt
Cl
OH
S
S
5i
7.90
7.85
7.87
7.75
7.73
7.73
7.69
7.68
7.67
7.41
7.40
7.39
7.38
7.37
7.35
7.32
7.26 CDCl$_3$
7.23
7.21
7.20
7.20
7.18
7.17
6.83
6.72
6.71
5.02
3.81
3.79
3.77
3.76
2.43
1.30
1.28
1.27
1.99
2.00
6.09
6.11
0.94
0.92
0.92
1.98
3.00
2.97
f1 (ppm)


^{13}C{^{1}H} NMR (151 MHz, CDCl$_3$)
OEt
Cl
OH
S
S
5i
139.9
139.0
139.1
137.5
135.9
135.2
134.3
133.7
133.4
133.3
131.4
131.2
129.6
129.4
128.7
124.9
124.9
124.6
123.5
123.0
122.3
121.8
121.6
119.0
118.9
77.3
77.0 CDCl$_3$
76.8
64.0
21.4
14.7
f1 (ppm)


7.82
7.80
7.56
7.55
7.36
7.36
7.35
7.35
7.34
7.34
7.32
7.31
7.30
7.30
7.29
7.29
7.26 CDCl3
7.25
7.24
7.24
7.23
7.23
7.22
7.22
7.21
7.21
7.20
7.20
7.20
7.04
7.04
7.03
7.03
7.03
7.02
7.02
1H NMR (600 MHz, CDCl3)
Ph Ph
S
S
2.00
2.07
2.24
2.17
6.19
4.05
f1 (ppm)


Jul26-2021-cjl.11.1.1r
140.28
139.61
136.68
134.66
131.68
129.91
128.36
127.12
124.92
124.48
123.58
122.08
77.25
77.04 CDCl3
76.83
13C{1H} NMR (151 MHz, CDCl3)
Ph Ph
S
S
f1 (ppm)