H_2O-Controlled Selective Disulfide Etherification and Thiocyanation of Pyrroles via Linear Paired Electrolysis

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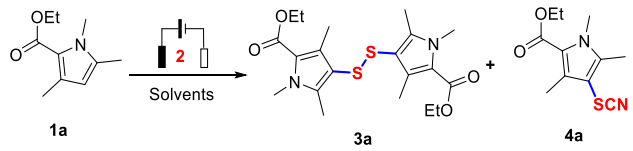
content

1. General information

All reactions that required anhydrous conditions were carried with standard procedures under nitrogen atmosphere. The solvents and chemicals were obtained from commercial sources, and were used without further purification. Column chromatography was generally performed on silica gel (300-400 mesh) and reactions were monitored by thin-layer chromatography (TLC) using 254 nm UV light. ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) were measured on Bruker Avance III 400 spectrometer. Chemical shifts are expressed in parts per million (ppm) with respect to tetramethylsilane. Coupling constants were reported as Hertz (Hz), signal shapes and splitting patterns were indicated as follows: s = singlet; d = doublet; t = triplet; q = quartet; m = multiplet. High-resolution mass spectra (HRMS) were recorded on Agilent mass spectrometer equipped with the APCI source and a Q-TOF detector. The X-ray analysis of crystal structure was carried out on Bruker SMART 1000 CCD.

2. Optimization of electrochemical reaction conditions

Table S1. Additional optimization of electrochemical reactions



Entry	**2**	Solvents		Yield%	
		MeCN	H_2O	**3a**	**4a**
1	NH_4SCN	4.0 mL	0	0	88
2	KSCN	4.0 mL	0	trace	trace.
3	NaSCN	4.0 mL	0	trace	trace
4	$^{n}Bu_4NSCN$	4.0 mL	0	38	18
5	$^{n}Bu_4NSCN$	4.0 mL	0.036 mL	60	0
6	$^{n}Bu_4NSCN$	4.0 mL	0.072 mL	64	0
7	$^{n}Bu_4NSCN$	3.5 mL	0.500 mL	68	0
8	$^{n}Bu_4NSCN$	3.0 mL	1.000 mL	64	trace
9	$^{n}Bu_4NSCN$	2.0 mL	2.000 mL	53	trace

[a]Reaction conditions: **1a** (0.2 mmol), **2** (0.3 mmol), in Solvents (4.0 mL) under air at room temperature for 1.5 hours, graphite felt anode, graphite felt cathode, 20 mA

3. A study on the synthesis of selenoethers and selenocyanation using pyrrole derivatives with KSeCN

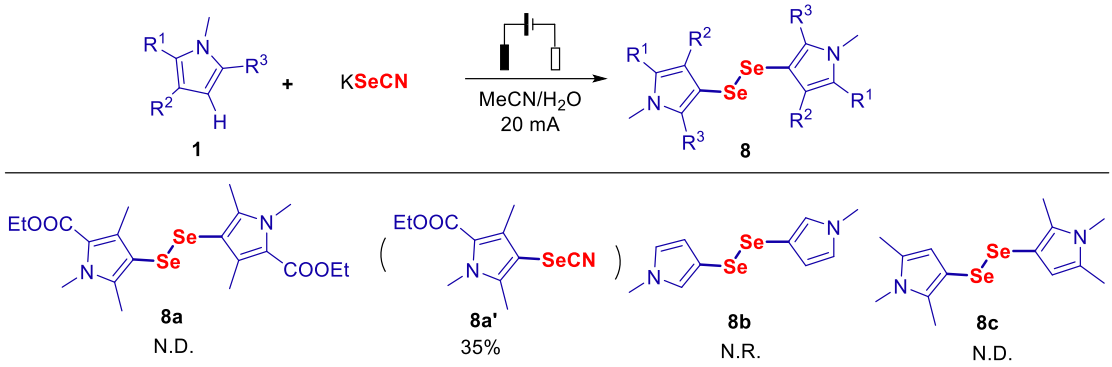


Figure S1. A study on the synthesis of selenoethers using pyrrole derivatives with KSeCN

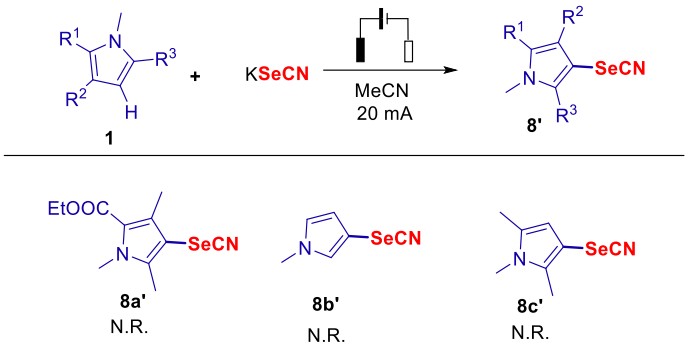
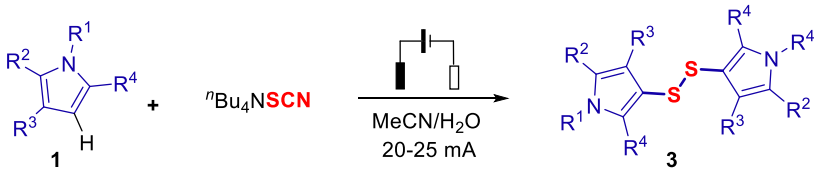


Figure S2. A study on the synthesis of selenocyanation using pyrrole derivatives with KSeCN

Electrochemical reactions of pyrrole derivatives **1a**, **1ae** and **1aj** with KSeCN using general procedure A did not afford the expected selenoethers **8a-8c**. Notably, the reaction stopped at the selenocyanation intermediate, giving the corresponding selenocyanate **8a'** in 35% yield, while only a trace amount of **8c'** was detected (**Figure S1**). When water was removed from the reaction system, no electrochemical reaction occurred due to the low solubility of KSeCN (**Figure S2**).

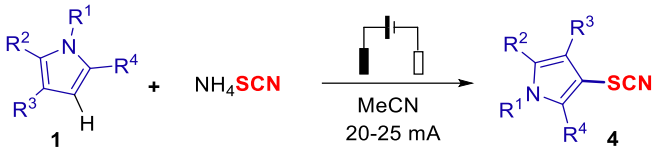
4. General procedures for electrochemical reactions

4.1 General procedure A:



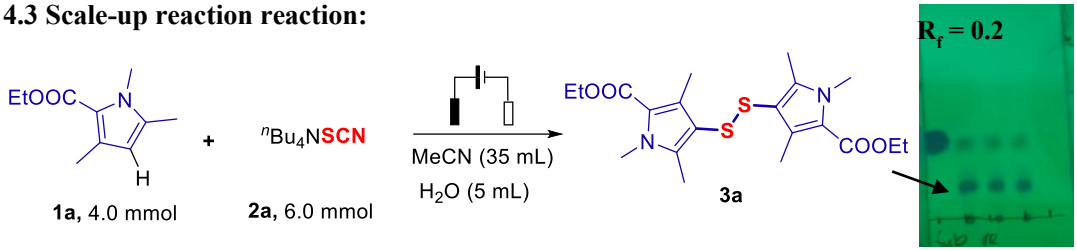
A 10 mL three-necked heart-shaped flask was charged with the substrate **1** (0.2 mmol), $^{n}Bu_4NSCN$ (0.3 mmol), MeCN (3.5 mL), H_2O (0.5 mL) and a magnetic stirrer. The graphite felt (1.0 cm x 1.0 cm x 0.3 cm, produced by Inner Mongolia Wanxing Carbon Company) as both the anode and cathode. The electrochemical reaction unit was installed according to our previous work (*Org. Biomol. Chem.* **2024**, *22*, 2549-2553). Electrolysis was carried out under a constant current of 20-25 mA at room temperature. After the reaction completed as monitored with TLC, the mixture was extracted with ethyl acetate and water. The combined organic layers were dried $MgSO_4$, filtered, and concentrated. The residue was purified by flash column chromatography to afford the target products **3**.

4.2 General procedure B:



A 10 mL three-necked heart-shaped flask was charged with the substrate **1** (0.2 mmol), NH_4SCN (0.3 mmol), MeCN (4.0 mL) and a magnetic stirrer. The graphite felt (1.0 cm x 1.0 cm x 0.3 cm, produced by Inner Mongolia Wanxing Carbon Company) as both the anode and cathode. The electrochemical reaction unit was installed according to our previous work (*Org. Biomol. Chem.* **2024**, *22*, 2549-2553). Electrolysis was carried out under a constant current of 20-25 mA at room temperature. After the reaction completed as monitored with TLC, the mixture was concentrated under reduced pressure. The residue was purified by chromatography on silica gel to afford the desired product **4**.

4.3 Scale-up reaction reaction:

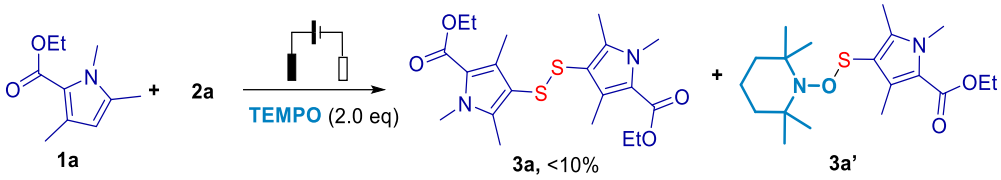


A 60 mL reaction bottle was charged with the substrate **1a** (4.0 mmol, 0.72 g), **2a** (6.0 mmol, 1.8 g), MeCN (35 mL), H_2O (5.0 mL) and a magnetic stirrer. The flask was equipped with two graphite felts (3.0 cm x 1.8 cm x 0.5 cm, produced by Inner Mongolia Wanxing Carbon Company), which were used as both the anode and cathode. The electrochemical reaction unit was installed according to **Figure S1**. Electrolysis was carried out under a constant current of 350 mA at room temperature. After 3.5 hours, the completion of the reaction was confirmed by thin-layer chromatography (TLC) using ethyl acetate and petroleum ether (1:9 v/v) as the eluent (R_f = 0.2). The reaction mixture was extracted with ethyl acetate and water. The combined organic layers were dried $MgSO_4$, filtered, and concentrated. The residue was purified by flash column chromatography to afford the desired product **3a** in 56% yield (0.475 g). *Caution: The reaction time of the scale-up reaction should not be excessively prolonged and*

must be monitored promptly by TLC.

Figure S3. Electrolysis setup

5. Detection and analysis of pyrrole- substituted *S*-centred radical adduct 3a'



A 10 mL three-necked heart-shaped flask was charged with the substrate **1a** (0.2 mmol, 0.036 g), $^{n}Bu_4NSCN$ (0.3 mmol, 0.090 g), TEMPO (0.4 mmol, 0.063 g), MeCN (3.5 mL), H_2O (0.5 mL) and a magnetic stirrer. The graphite felt (1.0 cm x 1.0 cm x 0.3 cm, produced by Inner Mongolia Wanxing Carbon Company) as both the anode and cathode. The electrochemical reaction unit was installed according to **General procedure A**. Electrolysis was carried out under a constant current of 25 mA at room temperature. After 1.5 hours, the reaction mixture was concentrated under reduced pressure and analyzed by high-resolution mass spectrometry (HRMS). The analysis results for *S*-centred radical adduct 3a' are presented in **Figure S4**.

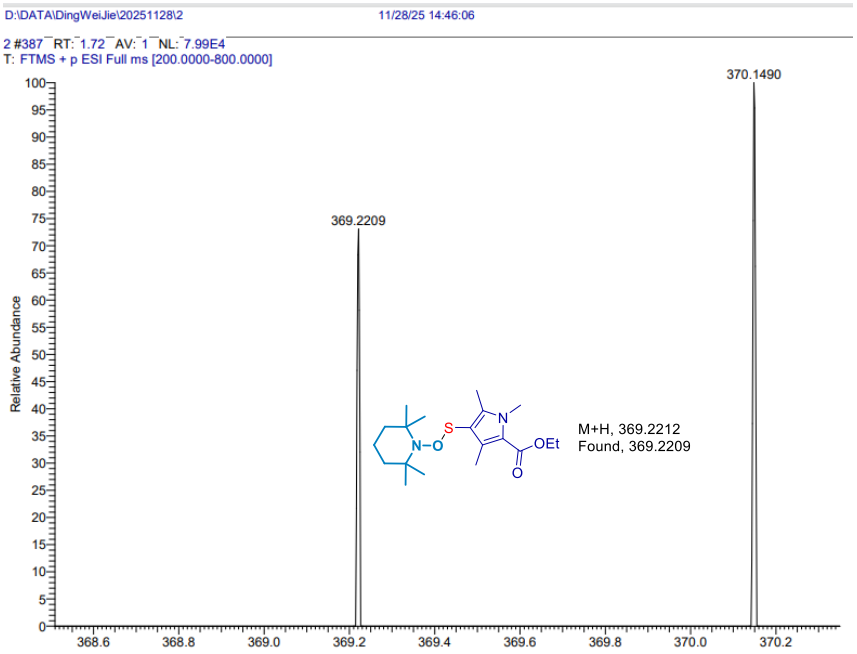


Figure S4. The HMRS analysis of radical adduct **3a'**

6. Cyclic voltammetry experiments of reactants

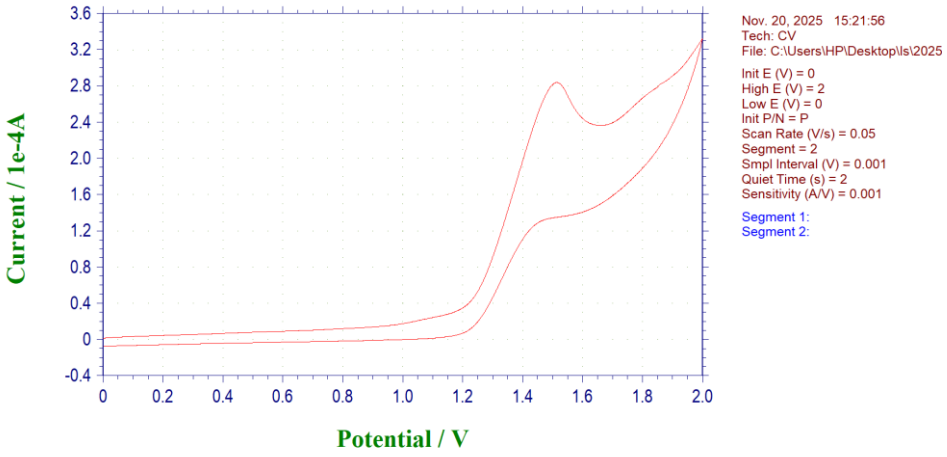


Figure S5. Cyclic voltammograms of **1a**

A solution of $^{n}Bu_4NBF_4$ (0.1 mmol) and **1a** (0.05 mmol) in a mixture of 3.5 mL MeCN and 0.5 mL H_2O was subjected to the cyclic voltammetry experiment. Electrodes included a 4.0 mm glassy carbon working electrode, a gauze platinum counter electrode and a saturated calomel reference electrode (SCE) via a salt bridge charged with a solution of $^{n}Bu_4NBF_4$ (0.025 M in MeCN/H_2O). Potential sweep rate was 50 mV/s.

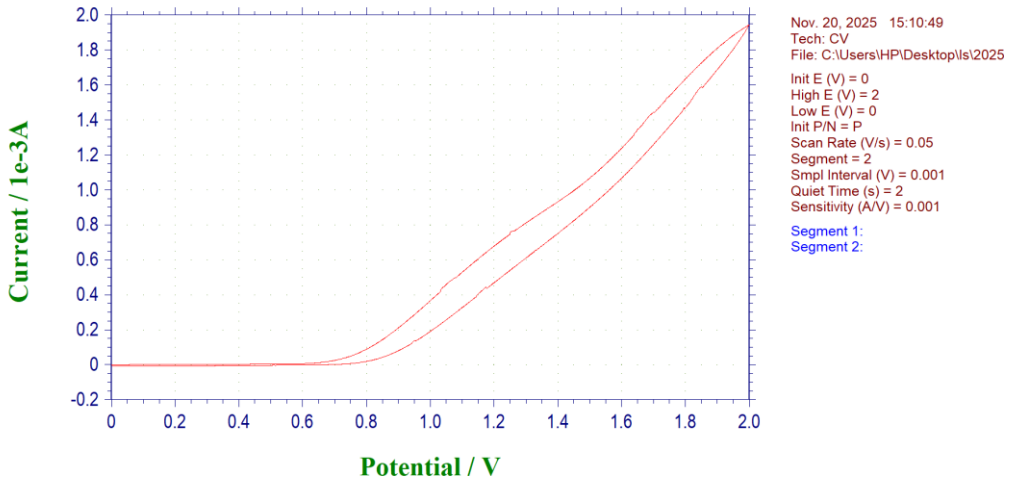


Figure S6. Cyclic voltammograms of ^{n}**Bu$_4$NSCN**

A solution of ^{n}Bu$_4$NBF$_4$ (0.1 mmol) and **^{n}Bu$_4$NSCN** (0.05 mmol) in a mixture of 3.5 mL MeCN and 0.5 mL H_2O was subjected to the cyclic voltammetry experiment. Electrodes included a 4.0 mm glassy carbon working electrode, a gauze platinum counter electrode and a saturated calomel reference electrode (SCE) via a salt bridge charged with a solution of ^{n}Bu$_4$NBF$_4$ (0.025 M in MeCN/H_2O). Potential sweep rate was 50 mV/s.

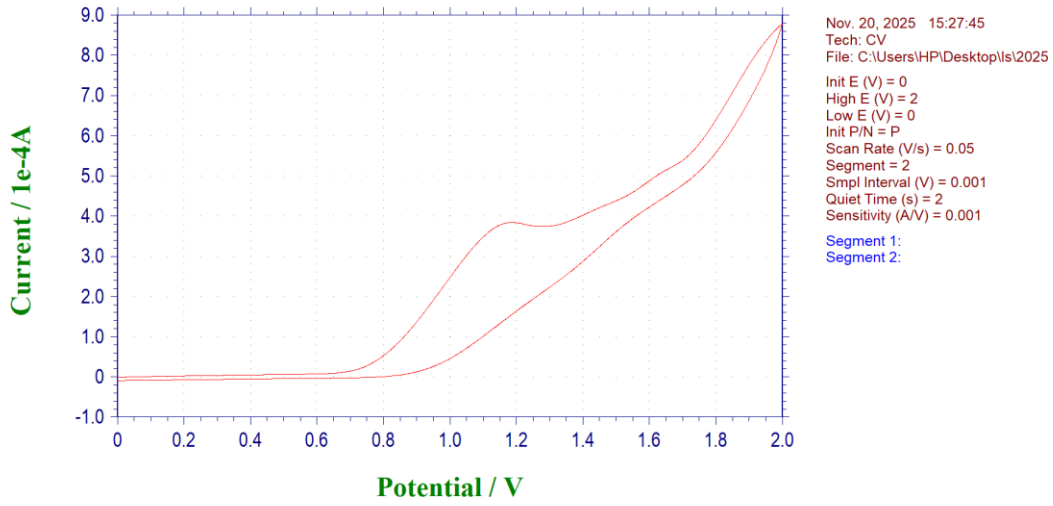


Figure S7. Cyclic voltammograms of **^{n}Bu$_4$NSCN** and **1a**

A solution of ^{n}Bu$_4$NBF$_4$ (0.1 mmol), **1a** (0.05 mmol) and **^{n}Bu$_4$NSCN** (0.075 mmol) in a mixture of 3.5 mL MeCN and 0.5 mL H_2O was subjected to the cyclic voltammetry experiment. Electrodes included a 4.0 mm glassy carbon working electrode, a gauze platinum counter electrode and a saturated calomel reference electrode (SCE) via a salt bridge charged with a solution of ^{n}Bu$_4$NBF$_4$ (0.025 M in MeCN/H_2O). Potential sweep rate was 50 mV/s.

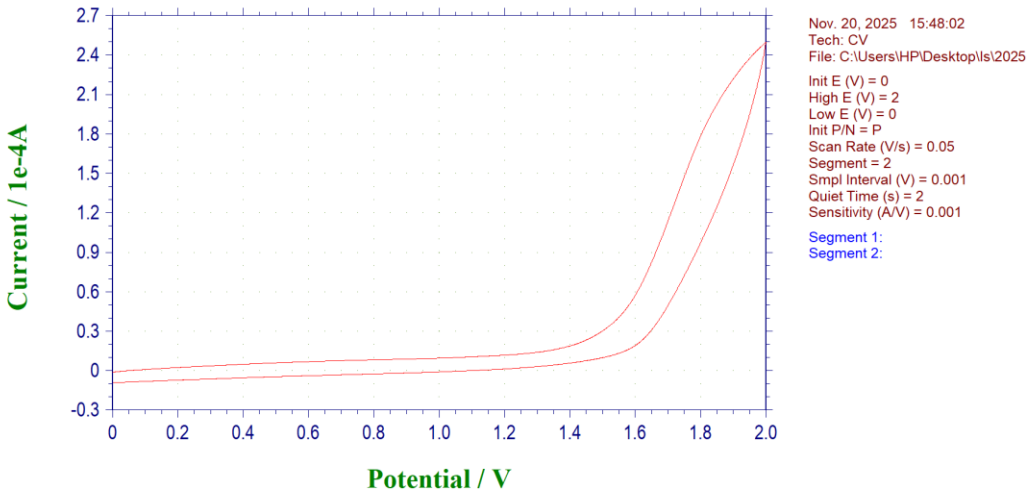


Figure S8. Cyclic voltammograms of **4a**

A solution of $^{n}Bu_4NBF_4$ (0.1 mmol) and **4a** (0.05 mmol) in a mixture of 3.5 mL MeCN and 0.5 mL H_2O was subjected to the cyclic voltammetry experiment. Electrodes included a 4.0 mm glassy carbon working electrode, a gauze platinum counter electrode and a saturated calomel reference electrode (SCE) via a salt bridge charged with a solution of $^{n}Bu_4NBF_4$ (0.025 M in MeCN/H_2O). Potential sweep rate was 50 mV/s.

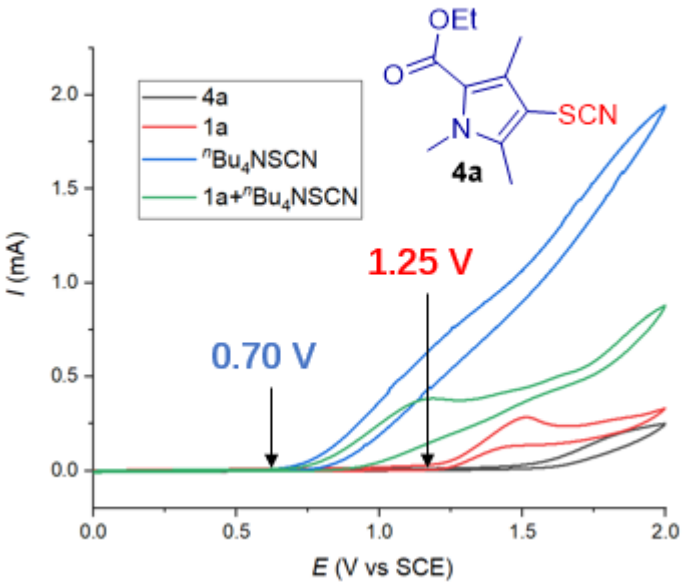


Figure S9. Overlapped CV results

7. The thermal ellipsoid plot of 3z

Add 100 mg of **3z** to a 25 mL beaker, completely dissolve it with EtOH. A single crystal **3z** was obtained by slowly evaporating at room temperature under the air conditions.

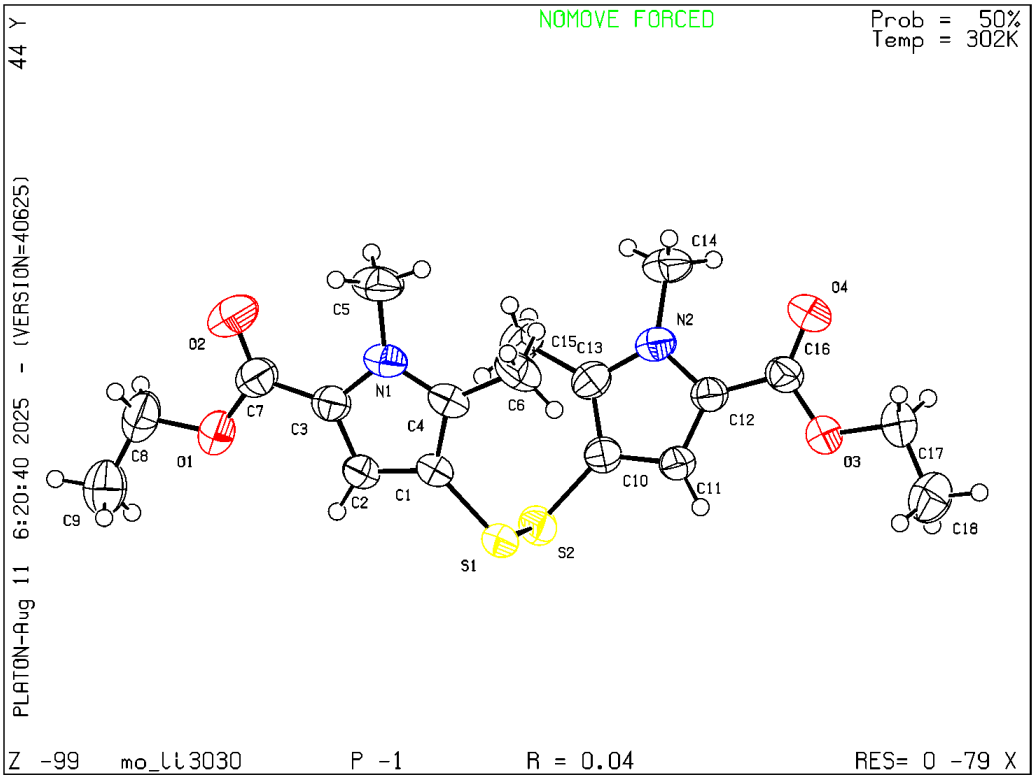


Table S2 Crystal data and structure refinement for 3z

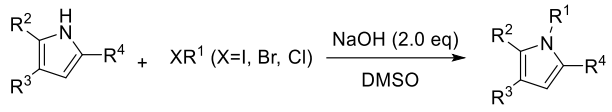
Identification code	3z
CCDC	2505118
Empirical formula	$C_{18}H_{24}N_2O_4S_2$
Formula weight	396.51
Temperature/K	302.00
Crystal system	triclinic
Space group	P-1
a/Å	8.5875(10)

b/Å	10.6673(10)
c/Å	11.4787(14)
α/°	78.905(4)
β/°	85.544(4)
γ/°	85.215(4)
Volume/Å^3	1026.2(2)
Z	2
ρ_{calc}g/cm^3	1.283
μ/mm^{-1}	0.284
F(000)	420.0
Crystal size/mm^3	0.33 × 0.23 × 0.21
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	3.9 to 56.58
Index ranges	-11≤h≤11, -13≤k≤14, -15≤l≤15
Reflections collected	42967
Independent reflections	5090 [R_{int} = 0.0553, R_{sigma} = 0.0289]
Data/restraints/parameters	5090/0/241
Goodness-of-fit on F^2	1.074
Final R indexes [I>=2σ (I)]	R_1 = 0.0400, wR_2 = 0.1145
Final R indexes [all data]	R_1 = 0.0480, wR_2 = 0.1223
Largest diff. peak/hole / e Å^{-3}	0.27/-0.20

8. Synthesis of pyrrole derivatives

1b-1q, 1aa, 1ac, 1ad and **1w** were prepared according to the following C. **1r-1v** were prepared according to the following procedure D. **1a, 1x, 1y, 1z, 1ab** and **4r** were purchased from company of Innochem.

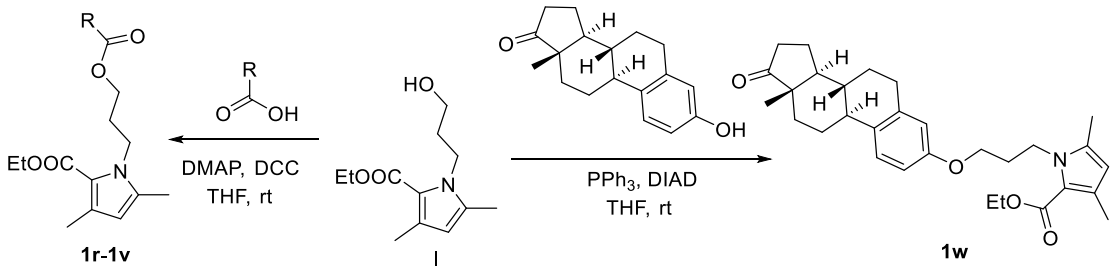
8.1 General procedure C



1b-1q, 1aa, 1ac, 1ad and **1w**

Following our previous work (*Chem. Commun.*, 2025, **61**, 11045-11048), 1*H*-pyrrole derivatives (5.0 mmol), NaOH (10.0 mmol, 0.40 g) and halogenated hydrocarbon (6.0 mmol) were dissolved in DMSO (20 ml). The reaction mixture was stirred overnight at room temperature. The mixture was concentrated, and extracted with ethyl acetate and water for three times. The combined organic layers were dried $MgSO_4$, filtered, and concentrated. The residue was purified by flash column chromatography to afford the target products.

8.2 General procedure D

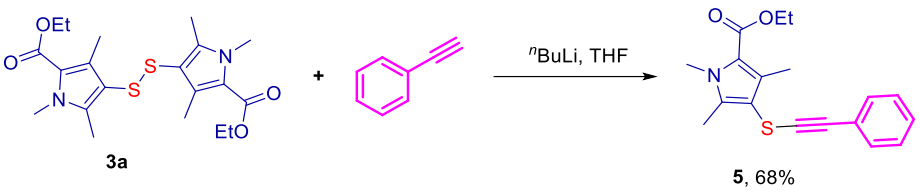


Following our previous work (*Nat. Commun.,* 2022, **13***,* 425), ethyl 1-(3-hydroxypropyl)-3,5-dimethyl-1*H*-pyrrole-2-carboxylate (0.45 g, 2.0 mmol), RCOOH (2.0 mmol, 1.0 eq), DMAP (0.024 g, 0.2 mmol, 0.01 eq) and DCC (0.618 g, 3.0 mmol, 1.5 eq) were dissolved in THF (10 ml) under argon. The solution was stirred overnight at room temperature. The mixture was concentrated, and purified with flash chromatography to afford the desired products **1r**-**1v**.

Following our previous work (*Nat. Commun.,* 2022, **13***,* 425), ethyl 1-(3-hydroxypropyl)-3,5-dimethyl-1*H*-pyrrole-2-carboxylate (0.225 g, 1.0 mmol) and Ph_3P (0.393 g, 1.5 mmol, 1.5 eq.) were dissolved in THF (5.0 ml). The solution was stirred for 0.5 h under argon. To this solution, DIAD (0.303 g, 1.5 mmol, 1.5 eq.) was added gradually at 0 °C. The reaction mixture was stirred overnight at room temperature. The mixture was concentrated, and purified with flash chromatography to afford the desired product **1w**.

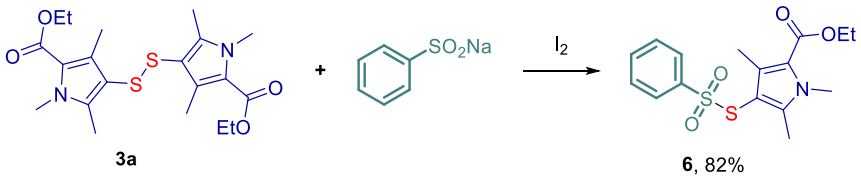
9. Further transformations of 3a

9.1 General procedure E



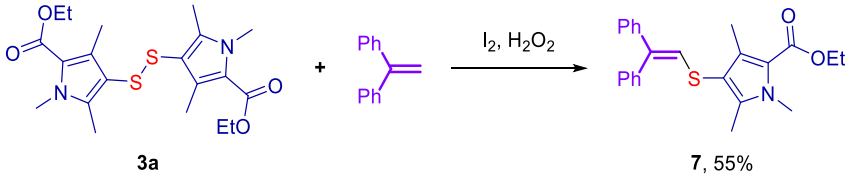
To a flask containing ethynylbenzene (10.2 mg, 0.1 mmol) in 10 mL of anhydrous THF was added ^{n}BuLi (0.048 mL, 2.5 M in hexanes, 0.12 mmol) at –78 °C under an argon atmosphere. The reaction mixture was stirred for 10 min at this temperature, followed by the addition of compound **3a** (63.6 mg, 0.15 mmol) at –78 °C. After the addition was completed, the reaction was allowed to warm to room temperature and stirred for 1 h. Upon completion, the reaction was quenched with saturated aqueous NH_4Cl solution, and the mixture was extracted three times with ethyl acetate. The combined organic layers were washed with water, dried over $MgSO_4$, and concentrated under reduced pressure. The residue was purified by flash chromatography to afford the desired product **5**.

9.2 General procedure F



Compound **3a** (42.4 mg, 0.1 mmol), sodium benzenesulfinate (52.5 mg, 0.32 mmol) and d I_2 (50.6 mg, 0.2 mmol) were dissolved in DCM (10 mL) at room temperature. The reaction mixture was stirred and monitored by TLC until complete consumption of **3a** was observed. The reaction was then quenched with aqueous $Na_2S_2O_3$ (1 M). The aqueous layer was extracted with DCM, and the combined organic layers were washed with water, dried over Na_2SO_4, and concentrated under reduced pressure. The residue was purified by flash chromatography to afford the desired product **6**.

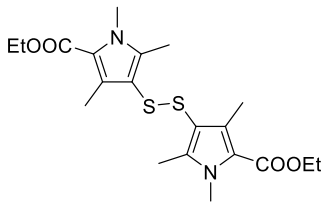
9.3 General procedure G



Compound **3a** (84.8 mg, 0.2 mmol), ethene-1,1-diyldibenzene (36.0 mg, 0.2 mmol), 30% aqueous H_2O_2 (v/v) (0.006 mL, 0.06 mmol) and I_2 (5.06 mg, 0.02 mmol) were dissolved in MeCN (2 mL) under air atmosphere. The reaction mixture was then stirred at 80 °C for 18 h. Upon completion, the reaction was quenched with water, followed by extraction with ethyl acetate (3 × 3 mL). The combined organic layers were washed with water, dried over Na_2SO_4, and concentrated under reduced pressure. The residue was purified by flash chromatography to afford the desired product **7**.

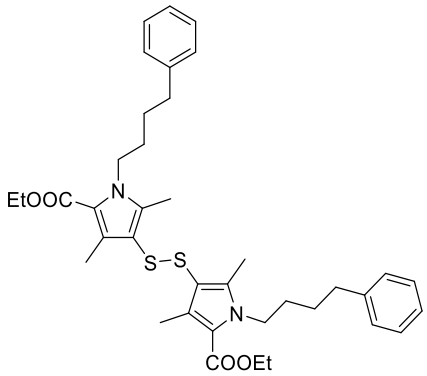
10. Experimental data

Diethyl 4,4'-disulfanediylbis(1,3,5-trimethyl-1H-pyrrole-2-carboxylate) (3a)



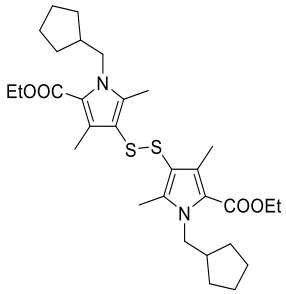
Following the general procedure A on 0.2 mmol scale, the substrate **3a** was obtained as a white solid in 68% yield (28.8 mg). mp 115.8-116.8 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 4.28 (q, J = 7.2 Hz, 4H), 3.76 (s, 6H), 2.18 (s, 6H), 1.95 (s, 6H), 1.35 (t, J = 7.2 Hz, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 162.2, 141.8, 133.3, 119.6, 113.3, 59.8, 33.7, 14.5, 12.2, 10.6. **HRMS m/z (ESI)** calcd. for $C_{20}H_{29}N_2O_4S_2^+$ (M + H)$^+$ 425.1569, found 425.1565.

Diethyl 4,4'-disulfanediylbis(3,5-dimethyl-1-(4-phenylbutyl)-1H-pyrrole-2-carboxylate) (3b)



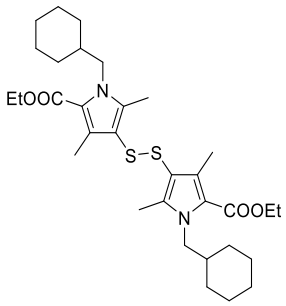
Following the general procedure A on 0.2 mmol scale, the substrate **3a** was obtained as a colorless oil in 72% yield (47.5 mg). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.26 (dd, J = 8.0, 2.4 Hz, 4H), 7.16 (dd, J = 8.8, 7.2 Hz, 6H), 4.27 (q, J = 7.2 Hz, 4H), 4.20 (t, J = 6.8 Hz, 4H), 2.62 (t, J = 6.8 Hz, 4H), 2.18 (s, 6H), 1.94 (s, 6H), 1.74-1.63 (m, 8H), 1.34 (t, J = 7.2 Hz, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.9, 142.0, 141.1, 133.6, 128.4(2), 128.4(1), 125.9, 118.7, 113.5, 59.8, 46.1, 35.6, 31.0, 28.6, 14.5, 12.3, 10.5. **HRMS m/z (ESI)** calcd. for $C_{38}H_{49}N_2O_4S_2^+$ (M + H)$^+$ 661.3134, found 661.3130.

Diethyl 4,4'-disulfanediylbis(1-(cyclopentylmethyl)-3,5-dimethyl-1H-pyrrole-2-carboxylate) (3c)



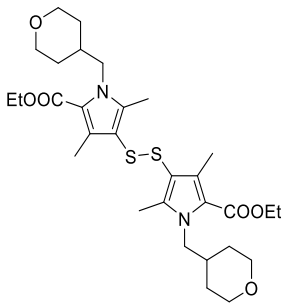
Following the general procedure A on 0.2 mmol scale, the substrate **3c** was obtained as a white solid in 81% yield (45.4 mg). mp 173.1-174.1 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 4.33 (q, J = 7.2 Hz, 4H), 4.25 (d, J = 7.2 Hz, 4H), 2.25 (s, 6H), 2.11 (s, 6H), 1.70-1.64 (m, 10H), 1.60-1.52 (m, 4H), 1.41 (t, J = 7.2 Hz, 6H), 1.29-1.20 (m, 4H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 162.0, 141.3, 133.6, 119.0, 113.5, 59.7, 50.2, 42.0, 30.1, 24.8, 14.5, 12.4, 11.1. **HRMS m/z (ESI)** calcd. for $C_{30}H_{45}N_2O_4S_2^+$ (M + H)$^+$ 561.2821, found 561.2821.

Diethyl 4,4'-disulfanediylbis(1-(cyclohexylmethyl)-3,5-dimethyl-1H-pyrrole-2-carboxylate) (3d)



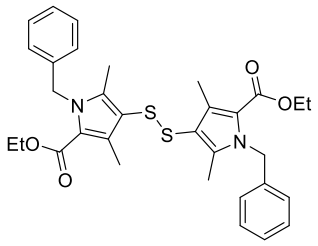
Following the general procedure A on 0.2 mmol scale, the substrate **3d** was obtained as a white solid in 71% yield (41.7 mg). 175.4-176.4 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 4.33 (q, J = 6.8 Hz, 4H), 4.27-3.99 (m, 4H), 2.26 (s, 6H), 2.09 (s, 6H), 1.76-1.67 (m, 8H), 1.58 (d, J = 12.0 Hz, 4H), 1.41 (t, J = 7.2 Hz, 6H), 1.19 (d, J = 7.2 Hz, 6H), 0.98 (d, J = 11.2 Hz, 4H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 162.0, 141.5, 133.6, 120.0, 113.6, 59.7, 52.0, 39.8, 30.7, 26.4, 26.0, 14.5, 12.5, 11.2. **HRMS m/z (ESI)** calcd. for $C_{32}H_{48}N_2NaO_4S_2^+$ (M + Na)$^+$ 611.2953, found 611.2944.

Diethyl 4,4'-disulfanediylbis(3,5-dimethyl-1-((tetrahydro-2H-pyran-4-yl)methyl)-1H-pyrrole-2-carboxylate) (3e)



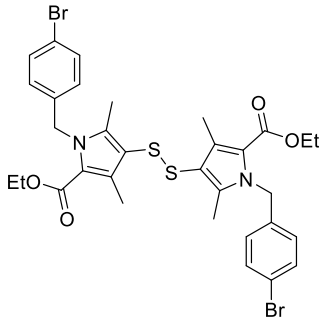
Following the general procedure A on 0.2 mmol scale, the substrate **3e** was obtained as a white solid in 70% yield (41.4 mg). mp 112.6-113.6 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 4.34 (q, J = 7.2 Hz, 4H), 4.31-4.06 (m, 4H), 3.99 (d, J = 10.0 Hz, 4H), 3.35 (t, J = 11.6 Hz, 4H), 2.23 (s, 6H), 2.14 (s, 6H), 2.23-1.94 (m, 2H), 1.50-1.37 (m, 14H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.9, 141.4, 133.8, 119.0, 114.0, 67.7, 59.8, 51.4, 37.3, 30.5, 14.5, 12.4, 11.3. **HRMS m/z (ESI)** calcd. for $C_{30}H_{45}N_2O_6S_2^+$ $(M + H)^+$ 593.2719, found 593.2717.

Diethyl 4,4'-disulfanediylbis(1-benzyl-3,5-dimethyl-1H-pyrrole-2-carboxylate) (3f)



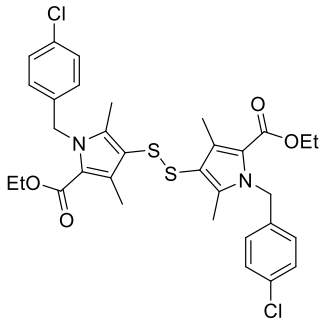
Following the general procedure A on 0.2 mmol scale, the substrate **3f** was obtained as a white solid in 75% yield (43.2 mg). mp 168.7-169.7 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.26 (dd, J = 2.4, 1.2Hz, 2H), 7.24-7.17 (m, 4H), 6.90 (d, J = 6.8 Hz, 4H), 5.46 (s, 4H), 4.21 (q, J = 7.2 Hz, 4H), 2.30 (s, 6H), 1.92 (s, 6H), 1.27 (t, J = 7.2 Hz, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.8, 141.8, 138.0, 133.6, 128.6, 127.1, 126.0, 119.3, 114.5, 59.9, 49.1, 14.4, 12.5, 10.7. **HRMS m/z (ESI)** calcd. for $C_{32}H_{36}N_2NaO_4S_2^+$ $(M + Na)^+$ 599.2014, found 599.2004.

Diethyl 4,4'-disulfanediylbis(1-(4-bromobenzyl)-3,5-dimethyl-1H-pyrrole-2-carboxylate) (3g)



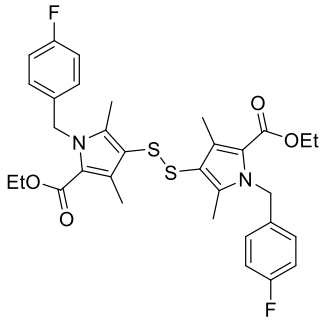
Following the general procedure A on 0.2 mmol scale, the substrate **3g** was obtained as a white solid in 66% yield (48.3 mg). mp 81.7-82.7 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.36 (d, J = 8.4 Hz, 4H), 6.78 (d, J = 8.4 Hz, 4H), 5.41 (s, 4H), 4.22 (q, J = 7.2 Hz, 4H), 2.29 (s, 6H), 1.90 (s, 6H), 1.28 (t, J = 7.2 Hz, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.8, 141.6, 137.1, 133.6, 131.8, 127.9, 121.0, 119.3, 114.8, 60.0, 48.7, 14.4, 12.5, 10.7. **HRMS m/z (ESI)** calcd. for $C_{32}H_{34}Br_2N_2NaO_4S_2^+$ $(M + Na)^+$ 757.0204, found 757.0193.

Diethyl 4,4'-disulfanediylbis(1-(4-chlorobenzyl)-3,5-dimethyl-1H-pyrrole-2-carboxylate) (3h)



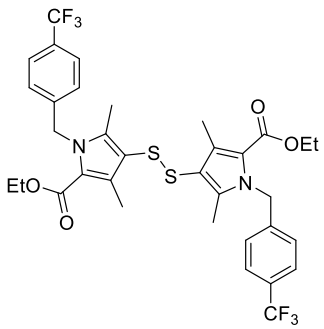
Following the general procedure A on 0.2 mmol scale, the substrate **3h** was obtained as a white solid in 78% yield (50.2 mg). mp 138.5-139.5 ºC. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.20 (d, J = 8.8 Hz, 4H), 6.84 (d, J = 8.8 Hz, 4H), 5.42 (s, 4H), 4.21 (q, J = 7.2 Hz, 4H), 2.29 (s, 6H), 1.91 (s, 6H), 1.28 (t, J = 7.2 Hz, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** 161.8, 141.6, 136.5, 133.6, 133.0, 128.8, 127.5, 119.3, 114.8, 60.0, 48.6, 14.4, 12.5, 10.7. **HRMS m/z (ESI)** calcd. for $C_{32}H_{34}Cl_2N_2NaO_4S_2^+$ $(M + Na)^+$ 667.1235, found 667.1225.

Diethyl 4,4'-disulfanediylbis(1-(4-fluorobenzyl)-3,5-dimethyl-1H-pyrrole-2-carboxylate) (3i)



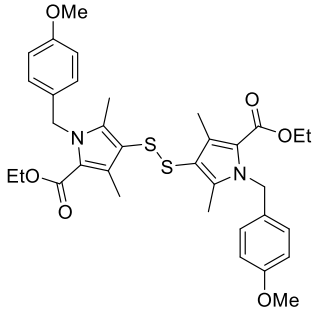
Following the general procedure A on 0.2 mmol scale, the substrate **3i** was obtained as a colorless oil in 68% yield (41.6 mg). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 6.95-6.85 (m, 8H), 5.43 (s, 4H), 4.22 (q, J = 7.2 Hz, 4H), 2.27 (s, 6H), 1.92 (s, 6H), 1.28 (t, J = 7.2 Hz, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.9 (d, J = 244.3 Hz), 161.8, 141.6, 133.7 (d, J = 3.2 Hz), 133.6, 127.7 (d, J = 7.9 Hz), 119.3, 115.5 (d, J = 22.7 Hz), 114.7, 60.0, 48.5, 14.4, 12.5, 10.7. **HRMS m/z (ESI)** calcd. for $C_{32}H_{35}F_2N_2O_4S_2^+$ $(M + H)^+$ 613.2006, found 613.1988.

Diethyl 4,4'-disulfanediylbis(3,5-dimethyl-1-(4-(trifluoromethyl)benzyl)-1H-pyrrole-2-carboxylate) (3j)



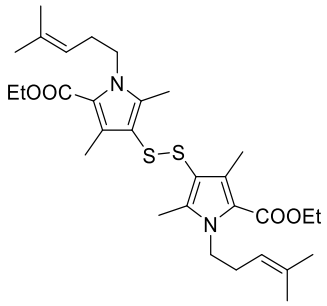
Following the general procedure A on 0.2 mmol scale, the substrate **3j** was obtained as a white solid in 52% yield (37.0 mg). mp 167.2-168.2 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.55 (d, J = 8.0 Hz, 4H), 7.05 (d, J = 8.0 Hz, 4H), 5.61 (s, 4H), 4.27 (q, J = 7.2 Hz, 4H), 2.37 (s, 6H), 2.03 (s, 6H), 1.33 (t, J = 7.2 Hz, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.7, 142.1, 141.4, 133.7, 129.5 (q, J = 32.4 Hz), 126.2, 125.9 (q, J = 3.7 Hz), 124.0 (q, J = 270.3 Hz), 119.3, 115.1, 60.0, 48.8, 14.3, 12.5, 10.7. **HRMS m/z (ESI)** calcd. for $C_{34}H_{34}F_6N_2NaO_4S_2^+$ $(M + Na)^+$ 735.1762, found 735.1749.

Diethyl 4,4'-disulfanediylbis(1-(4-methoxybenzyl)-3,5-dimethyl-1H-pyrrole-2-carboxylate) (3k)



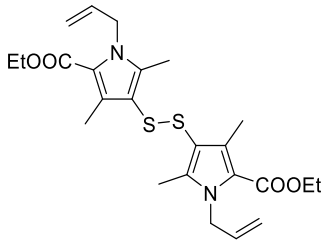
Following the general procedure A on 0.2 mmol scale, the substrate **3k** was obtained as a white solid in 55% yield (35.0 mg). mp 162.2-163.2 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 6.88 (dd, J = 34.0, 8.0 Hz, 8H), 5.43 (s, 4H), 4.29 (q, J = 7.2 Hz, 4H), 3.81 (s, 6H), 2.36 (s, 6H), 1.93 (s, 6H), 1.36 (t, J = 7.2 Hz, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.9, 158.6, 141.8, 133.5, 130.0, 127.4, 119.2, 114.2, 114.0, 59.8, 55.3, 48.6, 14.4, 12.5, 10.7. **HRMS m/z (ESI)** calcd. for $C_{34}H_{41}N_2O_6S_2^+$ $(M + H)^+$ 637.2406, found 637.2393.

Diethyl 4,4'-disulfanediylbis(3,5-dimethyl-1-(4-methylpent-3-en-1-yl)-1H-pyrrole-2-carboxylate) (3l)



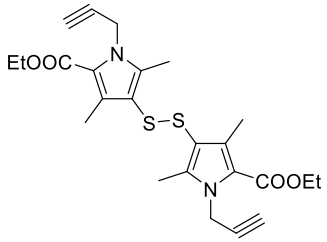
Following the general procedure A on 0.2 mmol scale, the substrate **3l** was obtained as a colorless oil in 66% yield (37.0 mg). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 5.15 (t, J = 6.8 Hz, 2H), 4.33 (q, J = 7.2 Hz, 4H), 4.22 (t, J = 7.6 Hz, 4H), 2.34 (dd, J = 14.8, 7.6 Hz, 4H), 2.24 (s, 6H), 2.05 (s, 6H), 1.73 (s, 6H), 1.62 (s, 6H), 1.40 (t, J = 7.2 Hz, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.9, 141.2, 134.7, 133.6, 119.8, 118.7, 113.2, 59.8, 45.9, 30.1, 25.8, 17.7, 14.5, 12.3, 10.4. **HRMS m/z (ESI)** calcd. for $C_{30}H_{45}N_2O_4S_2^+$ $(M + H)^+$ 561.2821, found 561.2808.

Diethyl 4,4'-disulfanediylbis(1-allyl-3,5-dimethyl-1H-pyrrole-2-carboxylate) (3m)



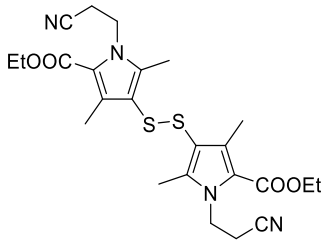
Following the general procedure A on 0.2 mmol scale, the substrate **3m** was obtained as a white solid in 60% yield (28.6 mg). mp 128.7-129.7 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 6.04-5.87 (m, 2H), 5.07 (dd, J = 10.4, 1.6 Hz, 2H), 5.03-4.82 (m, 4H), 4.79 (ddd, J = 17.2, 3.2, 1.6 Hz, 2H), 4.26 (q, J = 7.2 Hz, 4H), 2.22 (s, 6H), 1.97 (s, 6H), 1.33 (t, J = 7.2 Hz, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.9, 141.7, 134.2, 133.5, 118.9, 115.8, 113.8, 59.8, 48.2, 14.5, 12.4, 10.4. **HRMS m/z (ESI)** calcd. for $C_{24}H_{33}N_2O_4S_2^+$ $(M + H)^+$ 477.1882, found 477.1880.

Diethyl 4,4'-disulfanediylbis(3,5-dimethyl-1-(prop-2-yn-1-yl)-1H-pyrrole-2-carboxylate) (3n)



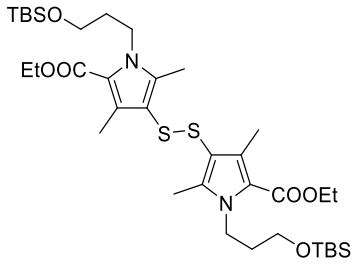
Following the general procedure A on 0.2 mmol scale, the substrate **3n** was obtained as a white solid in 51% yield (24.0 mg). mp 118.1-119.1 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 5.17 (s, 4H), 4.38 (q, J = 6.8 Hz, 4H), 2.33 (s, 8H), 2.07 (s, 6H), 1.43 (t, J = 6.8 Hz, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.8, 141.9, 133.9, 118.6, 114.1, 78.9, 72.0, 60.1, 35.3, 14.4, 12.2, 10.2. **HRMS m/z (ESI)** calcd. for $C_{24}H_{28}N_2NaO_4S_2^+$ $(M + Na)^+$ 495.1388, found 495.1382.

Diethyl 4,4'-disulfanediylbis(1-(2-cyanoethyl)-3,5-dimethyl-1H-pyrrole-2-carboxylate) (3o)



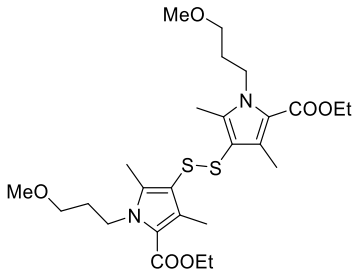
Following the general procedure A on 0.2 mmol scale, the substrate **3p** was obtained as a white solid in 54% yield (27.1 mg). mp 147.3-148.3 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 4.47 (t, J = 6.4 Hz, 4H), 4.28 (q, J = 7.2 Hz, 4H), 2.82 (t, J = 6.4 Hz, 4H), 2.23 (s, 6H), 2.14 (s, 6H), 1.36 (t, J = 7.2 Hz, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** 161.9, 141.6, 134.4, 118.7, 117.7, 114.9, 60.2, 42.0, 19.5, 14.5, 12.4, 10.8. **HRMS m/z (ESI)** calcd. for $C_{24}H_{30}N_4NaO_4S_2^+$ $(M + Na)^+$ 525.1606, found 525.1600.

Diethyl 4,4'-disulfanediylbis(1-(3-((tert-butyldimethylsilyl)oxy)propyl)-3,5-dimethyl-1H-pyrrole-2-carboxylate) (3p)



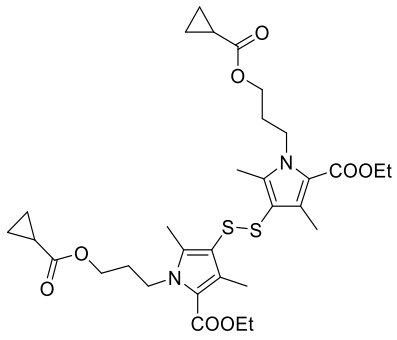
Following the general procedure A on 0.2 mmol scale, the substrate **3p** was obtained as a colorless oil in 64% yield (47.4 mg). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 4.39-4.29 (m, 8H), 3.67 (t, J = 5.2 Hz, 4H), 2.21 (s, 6H), 2.09 (s, 6H), 1.92-1.84 (m, 4H), 1.40 (t, J = 7.2 Hz, 6H), 0.95 (s, 18H), 0.10 (s, 12H). **^{13}C NMR (100 MHz, Chloroform-*d*)** 161.7, 141.4, 133.8, 118.5, 113.2, 60.1, 59.6, 43.5, 34.3, 25.9, 18.2, 14.5, 12.3, 10.3, -5.4. **HRMS m/z (ESI)** calcd. for $C_{36}H_{65}N_2O_6S_2Si_2^+$ (M + H)$^+$ 741.3823, found 741.3809.

Diethyl 4,4'-disulfanediylbis(1-(3-methoxypropyl)-3,5-dimethyl-1H-pyrrole-2-carboxylate) (3q)



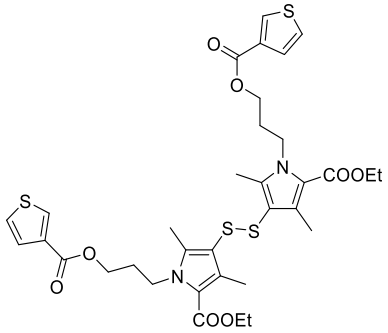
Following the general procedure A on 0.2 mmol scale, the substrate **3q**was obtained as a white solid in 68% yield (36.7 mg). mp 106.7-107.7 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 4.31-4.24 (m, 8H), 3.33-3.29 (m, 10H), 2.16 (s, 6H), 2.01 (s, 6H), 1.88 (td, J = 12.0, 6.0 Hz, 4H), 1.34 (t, J = 7.2 Hz, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.8, 141.6, 133.9, 118.6, 113.3, 69.6, 59.8, 58.7, 43.6, 31.3, 14.5, 12.3, 10.3. **HRMS m/z (ESI)** calcd. for $C_{26}H_{40}N_2NaO_6S_2^+$ (M + Na)$^+$ 563.2225, found 563.2219.

Diethyl 4,4'-disulfanediylbis(1-(3-((cyclopropanecarbonyl)oxy)propyl)-3,5-dimethyl-1H-pyrrole-2-carboxylate) (3r)



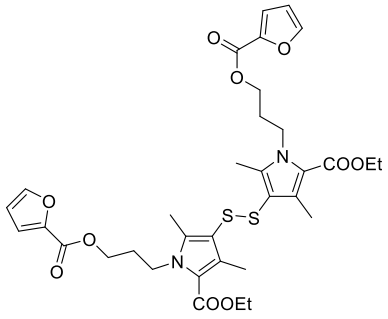
Following the general procedure A on 0.2 mmol scale, the substrate **3r** was obtained as a colorless oil in 68% yield (44.0 mg). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 4.31 (dt, J = 20.4, 7.2 Hz, 8H), 4.10 (t, J = 5.8 Hz, 4H), 2.18 (s, 6H), 2.06-1.97 (m, 10H), 1.62 (dd, J = 8.0, 4.4 Hz, 2H), 1.37 (t, J = 7.2 Hz, 6H), 1.01 (s, 4H), 0.89 (d, J = 6.2 Hz, 4H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 174.8, 161.7, 141.1, 133.8, 118.7, 113.6, 61.8, 59.8, 43.3, 30.4, 14.5, 12.8, 12.2, 10.3, 8.5. **HRMS m/z (ESI)** calcd. for $C_{32}H_{44}N_2NaO_8S_2^+$ (M + Na)$^+$ 671.2437, found 671.2429.

Diethyl 4,4'-disulfanediylbis(3,5-dimethyl-1-(3-((thiophene-3-carbonyl)oxy) propyl)-1H-pyrrole-2-carboxylate) (3s)



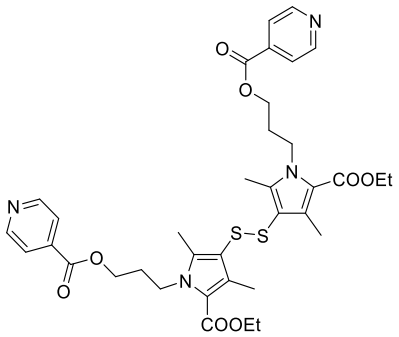
Following the general procedure A on 0.2 mmol scale, the substrate **3s** was obtained as a colorless oil in 51% yield (37.3 mg). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.17 (s, 2H), 7.56 (d, J = 3.6 Hz, 2H), 7.37 (d, J = 1.2 Hz, 2H), 4.44 (d, J = 6.8 Hz, 4H), 4.31 (dd, J = 12.8, 6.0 Hz, 8H), 2.22-2.07 (m, 16H), 1.39 (t, J = 6.4 Hz, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 162.6, 161.8, 141.1, 133.8, 133.4, 132.9, 127.9, 126.2, 118.7, 113.7, 62.00, 59.8, 43.3, 30.4, 14.5, 12.3, 10.4. **HRMS m/z (ESI)** calcd. for $C_{34}H_{40}N_2NaO_8S_4^+$ (M + Na)$^+$ 755.1565, found 755.1557.

Diethyl 4,4'-disulfanediylbis(1-(3-((furan-2-carbonyl)oxy)propyl)-3,5-dimethyl-1H-pyrrole-2-carboxylate) (3t)



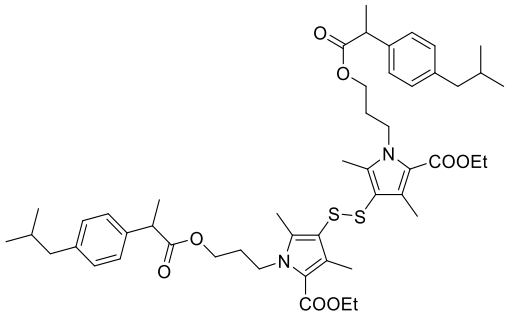
Following the general procedure A on 0.2 mmol scale, the substrate **3t** was obtained as a colorless oil in 41% yield (28.7 mg). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.64 (s, 2H), 7.23 (s, 2H), 6.57 (s, 2H), 4.44 (t, J = 6.8 Hz, 4H), 4.38-4.25 (m, 8H), 2.22 (s, 6H), 2.18-1.92 (m, 10H), 1.40 (t, J = 7.2 Hz, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.8, 158.5, 146.5, 144.5, 141.1, 133.8, 118.7, 118.1, 113.8, 111.9, 62.2, 59.8, 43.2, 30.4, 14.4, 12.3, 10.4. **HRMS m/z (ESI)** calcd. for $C_{34}H_{40}N_2NaO_{10}S_2^+$ (M + Na)$^+$ 723.2022, found 723.2010.

(Disulfanediylbis(5-(ethoxycarbonyl)-2,4-dimethyl-1H-pyrrole-3,1-diyl))bis (propane-3,1-diyl) diisonicotinate (3u)



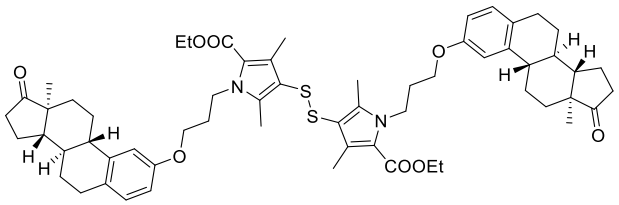
Following the general procedure A on 0.2 mmol scale, the substrate **3u** was obtained as a colorless oil in 44% yield (31.7 mg). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.83 (d, J = 4.4 Hz, 4H), 7.88 (d, J = 4.4 Hz, 4H), 4.45 (dt, J = 10.2, 6.0 Hz, 8H), 4.30 (dd, J = 14.0, 7.2 Hz, 4H), 2.26-2.21 (m, 16H), 1.39 (dd, J = 13.6, 7.2 Hz, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 165.0, 161.8, 150.7, 141.0, 137.2, 133.8, 122.9, 118.7, 113.9, 63.2, 59.9, 43.2, 30.2, 14.5, 12.3, 10.4. **HRMS m/z (ESI)** calcd. for $C_{36}H_{43}N_4O_8S_2^+$ $(M + H)^+$ 723.2522, found 723.2513.

Diethyl 4,4'-disulfanediylbis(1-(3-((2-(4-isobutylphenyl)propanoyl)oxy)propyl)-3,5-dimethyl-1H-pyrrole-2-carboxylate) (3v)



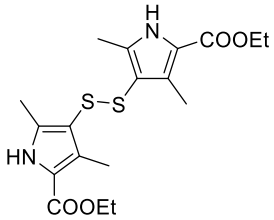
Following the general procedure A on 0.2 mmol scale, the substrate **3v** was obtained as a white solid in 63% yield (55.9 mg). mp 100.3-101.3 ºC. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.19 (dd, J = 45.2, 7.6 Hz, 8H), 4.32 (q, J = 6.8 Hz, 4H), 4.22 (t, J = 7.2 Hz, 4H), 4.13 (d, J = 5.2 Hz, 4H), 3.75 (q, J = 6.8 Hz, 2H), 2.46 (t, J = 15.6 Hz, 4H), 2.22 (s, 6H), 2.04-1.80 (m, 12H), 1.55 (d, J = 7.2 Hz, 6H), 1.40 (t, J = 7.2 Hz, 6H), 0.94 (d, J = 6.4 Hz, 12H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 174.6, 161.7, 141.1, 140.7, 137.7, 133.7, 129.4, 127.2, 118.5, 113.6, 61.9, 59.8, 45.2, 45.0, 43.2, 30.3, 30.2, 22.4, 18.5, 14.5, 12.2, 10.2. **HRMS m/z (ESI)** calcd. for $C_{50}H_{68}N_2NaO_8S_2^+$ $(M + Na)^+$ 911.4315, found 911.4305.

Diethyl 4,4'-disulfanediylbis(3,5-dimethyl-1-(3-(((8R,9S,13S,14S)-13-methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-2-yl)oxy)propyl)-1H-pyrrole-2-carboxylate) (3w)



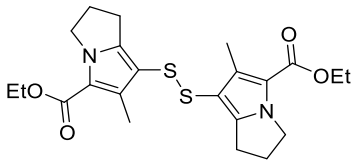
Following the general procedure A on 0.2 mmol scale, the substrate **3w** was obtained as a colorless oil in 55% yield (55.9 mg). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.23 (d, J = 8.4 Hz, 2H), 6.72 (d, J = 8.4 Hz, 2H), 6.65 (s, 2H), 4.46 (t, J = 6.8 Hz, 4H), 4.33 (q, J = 7.2 Hz, 4H), 3.94 (t, J = 5.2 Hz, 4H), 2.93 (d, J = 4.8 Hz, 4H), 2.56 (dd, J = 18.8, 8.4 Hz, 2H), 2.42 (t, J = 11.6 Hz, 2H), 2.3-1.98 (m, 24H), 1.70-1.48 (m, 14H), 1.41 (t, J = 7.2 Hz, 6H), 0.96 (s, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 200.9, 153.5, 149.4, 137.3, 134.3, 131.2, 129.9, 125.2, 118.9, 115.6, 114.8, 113.7, 75.8, 71.9, 64.4, 62.5, 59.3, 58.8, 54.7, 52.8, 49.3, 48.9, 47.8, 45.3, 44.8, 41.3, 35.6, 35.2, 33.9, 32.3. **HRMS m/z (ESI)** calcd. for $C_{60}H_{76}N_2NaO_8S_2^+$ (M + Na)$^+$ 1039.4941, found 1039.4944.

Diethyl 4,4'-disulfanediylbis(3,5-dimethyl-1H-pyrrole-2-carboxylate) (3x)



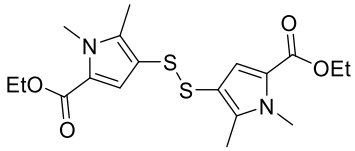
Following the general procedure A on 0.2 mmol scale, the substrate **3x** was obtained as a white solid in 50% yield (19.8 mg). mp 217.9-218.9 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 8.90 (s, 2H), 4.42-4.29 (m, 4H), 2.28 (s, 6H), 2.04 (s, 6H), 1.42 (t, J = 6.6 Hz, 6H). **^{13}C NMR (100 MHz, Acetone-*d*$_6$)** δ 160.6, 139.0, 131.3, 118.1, 113.7, 59.4, 13.9, 10.3, 10.2. **HRMS m/z (ESI)** calcd. for $C_{18}H_{24}N_2NaO_4S_2^+$ (M + Na)$^+$ 419.1075, found 419.1066

Diethyl 7,7'-disulfanediylbis(6-methyl-2,3-dihydro-1H-pyrrolizine-5-carboxylate) (3y)



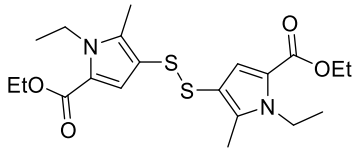
Following the general procedure A on 0.2 mmol scale, the substrate **3y** was obtained as a white solid in 68% yield (30.5 mg). mp 119.1-120.1 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 4.32 (dt, J = 14.4, 7.2 Hz, 8H), 2.54 (t, J = 7.2 Hz, 4H), 2.44-2.35 (m, 4H), 2.26 (s, 6H), 1.41 (t, J = 7.2 Hz, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.6, 148.30, 136.8, 116.3, 107.7, 59.7, 49.5, 25.9, 23.8, 14.6, 11.7. **HRMS m/z (ESI)** calcd. for $C_{22}H_{29}N_2O_4S_2^+$ (M + H)$^+$ 449.1569, found 449.1565.

Diethyl 4,4'-disulfanediylbis(1,5-dimethyl-1H-pyrrole-2-carboxylate) (3z)



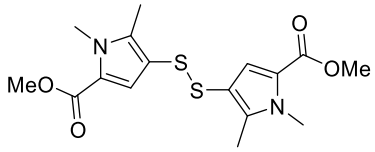
Following the general procedure A on 0.2 mmol scale, the substrate **3z** was obtained as a white solid in 54% yield (21.4 mg). mp 172.9-173.9 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.01 (s, 2H), 4.31 (q, J = 6.8 Hz, 4H), 3.90 (s, 6H), 2.16 (s, 6H), 1.39 (t, J = 6.4 Hz, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.1, 141.8, 122.9, 122.1, 113.0, 59.9, 33.3, 14.5, 10.6. **HRMS m/z (ESI)** calcd. for $C_{18}H_{25}N_2O_4S_2^+$ $(M + H)^+$ 397.1256, found 397.1248.

Diethyl 4,4'-disulfanediylbis(1-ethyl-5-methyl-1H-pyrrole-2-carboxylate) (3aa)



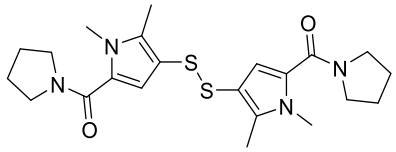
Following the general procedure A on 0.2 mmol scale, the substrate **3aa** was obtained as a white solid in 55% yield (23.3 mg). mp 100.7-101.7 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.01 (s, 2H), 4.38 (q, J = 6.8 Hz, 4H), 4.30 (q, J = 6.8 Hz, 4H), 2.13 (s, 6H), 1.35 (dt, J = 22.0, 7.6 Hz, 12H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 160.7, 141.0, 123.4, 121.2, 112.8, 59.9, 41.1, 16.1, 14.4, 10.0. **HRMS m/z (ESI)** calcd. for $C_{20}H_{29}N_2O_4S_2^+$ $(M + H)^+$ 425.1569, found 425.1559.

Dimethyl 4,4'-disulfanediylbis(1,5-dimethyl-1H-pyrrole-2-carboxylate) (3ab)



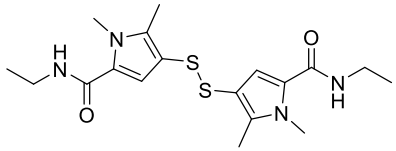
Following the general procedure A on 0.2 mmol scale, the substrate **3ab** was obtained as a white solid in 48% yield (17.7 mg). mp 215.6-216.6 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 6.92 (s, 2H), 3.83 (s, 6H), 3.78 (s, 6H), 2.09 (s, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.5, 141.9, 123.0, 121.7, 113.0, 51.2, 33.4, 10.6. **HRMS m/z (ESI)** calcd. for $C_{16}H_{21}N_2O_4S_2^+$ $(M + H)^+$ 369.0943, found 369.0935.

(Disulfanediylbis(1,5-dimethyl-1H-pyrrole-4,2-diyl))bis(pyrrolidin-1-ylmethanone) (3ac)



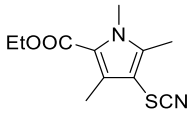
Following the general procedure A on 0.2 mmol scale, the substrate **3ac** was obtained as a colorless oil in 64% yield (28.5 mg). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 6.52 (s, 2H), 3.79 (s, 6H), 3.71-3.55 (s, 8H), 2.14 (s, 6H), 2.02-1.91 (m, 8H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.6, 139.3, 125.8, 118.5, 111.1, 49.6, 46.4, 33.5, 26.6, 24.2, 10.3. **HRMS m/z (ESI)** calcd. for $C_{22}H_{31}N_4O_2S_2^+$ $(M + H)^+$ 447.1888, found 447.1886.

4,4'-disulfanediylbis(N-ethyl-1,5-dimethyl-1H-pyrrole-2-carboxamide) (3ad)



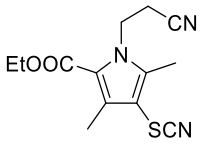
Following the general procedure A on 0.2 mmol scale, the substrate **3ad** was obtained as a white solid in 68% yield (26.8 mg). mp 122.3-123.3 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 6.46 (s, 2H), 5.81 (s, 2H), 3.81 (s, 6H), 3.37 (dt, J = 13.8, 7.2 Hz, 4H), 2.02 (s, 6H), 1.19 (t, J = 7.2 Hz, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.7, 140.4, 125.7, 116.5, 111.6, 34.3, 33.3, 15.1, 10.3. **HRMS m/z (ESI)** calcd. for $C_{18}H_{27}N_4O_2S_2^+$ (M + H)$^+$ 395.1575, found 395.1571.

Ethyl 1,3,5-trimethyl-4-thiocyanato-1H-pyrrole-2-carboxylate (4a)



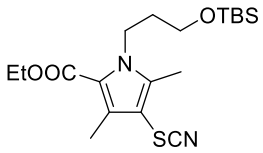
Following the general procedure B on 0.2 mmol scale, the substrate **4a** was obtained as a white solid in 88% yield (41.8 mg). mp 107.5-108.5 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 4.30 (qd, J = 7.2, 1.0 Hz, 2H), 3.81 (s, 3H), 2.42 (s, 3H), 2.39 (s, 3H), 1.36 (td, J = 7.2, 1.2 Hz, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.6, 140.6, 132.0, 120.8, 111.7, 98.7, 60.2, 34.0, 14.5, 12.4, 11.3. **HRMS m/z (ESI)** calcd. for $C_{11}H_{15}N_2O_2S^+$ (M + H)$^+$ 239.0854, found 239.0849.

Ethyl 1-(2-cyanoethyl)-3,5-dimethyl-4-thiocyanato-1H-pyrrole-2-carboxylate (4b)



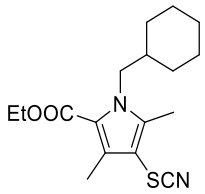
Following the general procedure B on 0.2 mmol scale, the substrate **4b** was obtained as a colorless oil in 74% yield (41.0 mg). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 4.60 (t, J = 6.4 Hz, 2H), 4.37 (q, J = 7.2 Hz, 2H), 2.93 (t, J = 6.4 Hz, 2H), 2.58 (s, 3H), 2.50 (s, 3H), 1.43 (t, J = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, Chloroform-d)** δ 161.4, 140.9, 133.3, 119.8, 117.4, 111.1, 100.7, 60.6, 42.2, 19.4, 14.4, 12.6, 11.4. **HRMS m/z (ESI)** calcd. for $C_{13}H_{16}N_3O_2S^+$ (M + H)$^+$ 278.0963, found 278.0958.

Ethyl 1-(3-((tert-butyldimethylsilyl)oxy)propyl)-3,5-dimethyl-4-thiocyanato-1H-pyrrole-2-carboxylate (4c)



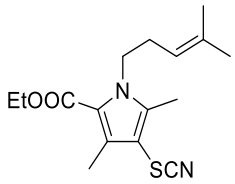
Following the general procedure B on 0.2 mmol scale, the substrate **4c** was obtained as a colorless oil in 80% yield (63.4 mg). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 4.42 (t, J = 7.2 Hz, 2H), 4.35 (q, J = 7.2 Hz, 2H), 3.66 (t, J = 5.4 Hz, 2H), 2.49 (s, 6H), 1.96-1.86 (m, 2H), 1.42 (t, J = 7.2 Hz, 3H), 0.95 (s, 9H), 0.11 (s, 6H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.2, 140.5, 132.5, 119.9, 111.7, 98.6, 60.1, 59.9, 43.9, 34.1, 25.9, 18.3, 14.4, 12.5, 11.0, -5.4. **HRMS m/z (ESI)** calcd. for $C_{19}H_{33}N_2O_3SSi^+$ (M + H)$^+$ 397.1981, found 397.1977.

Ethyl 1-(cyclohexylmethyl)-3,5-dimethyl-4-thiocyanato-1H-pyrrole-2-carboxylate (4d)



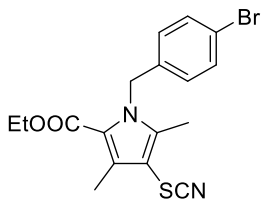
Following the general procedure B on 0.2 mmol scale, the substrate **4d** was obtained as a colorless oil in 84% yield (53.8 mg) . **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 4.29 (qd, *J* = 7.2, 0.4 Hz, 2H), 4.20-4.03 (m, 2H), 2.42 (s, 3H), 2.38 (s, 3H), 1.70-1.60 (m, 4H), 1.51 (d, *J* = 12.0 Hz, 2H), 1.35 (td, *J* = 7.2, 0.4 Hz, 3H), 1.14 (dt, *J* = 17.2, 6.0 Hz, 3H), 0.98-0.89 (m, 2H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.4, 140.6, 132.5, 120.4, 111.7, 98.8, 60.1, 52.4, 39.7, 30.7, 26.4, 25.9, 14.5, 12.6, 11.8. **HRMS m/z (ESI)** calcd. for $C_{17}H_{25}N_2O_2S^+$ (M + H) $^+$ 321.1637, found 321.1632.

Ethyl 3,5-dimethyl-1-(4-methylpent-3-en-1-yl)-4-thiocyanato-1H-pyrrole-2-carboxylate (4e)



Following the general procedure B on 0.2 mmol scale, the substrate **4e** was obtained as a white solid in 76% yield (46.5 mg). mp 111.5-112.5 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 5.13 (t, *J* = 7.3 Hz, 1H), 4.31 (m, 4H), 2.49 (s, 3H), 2.45 (s, 3H), 2.39 (q, *J* = 7.4 Hz, 2H), 1.73 (s, 3H), 1.53 (s, 3H), 1.43 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.3, 140.2, 135.3, 132.2, 120.0, 119.4, 111.7, 98.8, 60.1, 46.2, 29.8, 25.8, 17.5, 14.4, 12.5, 11.2. **HRMS m/z (ESI)** calcd. for $C_{16}H_{23}N_2O_2S^+$ (M + H) $^+$ 307.1480, found 307.1477.

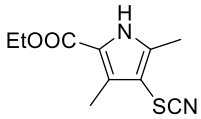
Ethyl 1-(4-bromobenzyl)-3,5-dimethyl-4-thiocyanato-1H-pyrrole-2-carboxylate (4f)



Following the general procedure B on 0.2 mmol scale, the substrate **4f** was obtained as a white solid in 80% yield (62.7 mg). mp 103.1-104.1 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.40 (d, *J* = 8.4 Hz, 2H), 6.79 (d, *J* = 8.4 Hz, 2H), 5.54 (s, 2H), 4.23 (q, *J* = 7.2 Hz, 2H), 2.47 (s, 3H), 2.32 (s, 3H), 1.29 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.3, 140.9, 136.3, 132.7, 132.0, 127.6, 121.3,

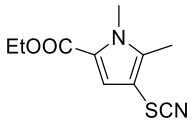
120.5, 111.4, 100.1, 60.4, 49.1, 14.4, 12.6, 11.2. **HRMS m/z (ESI)** calcd. for $C_{17}H_{18}BrN_2O_2S^+$ (M + H) $^+$ 393.0272, found 393.0270.

Ethyl 3,5-dimethyl-4-thiocyanato-1H-pyrrole-2-carboxylate (4g)



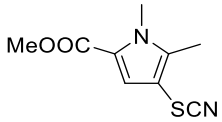
Following the general procedure B on 0.2 mmol scale, the substrate **4g** was obtained as a white solid in 82% yield (36.7 mg). mp 207.8-208.8 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 9.41 (s, 1H), 4.40 (q, J = 7.2 Hz, 2H), 2.49 (s, 6H), 1.43 (t, J = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.2, 138.0, 131.1, 119.0, 111.4, 100.1, 60.7, 14.5, 12.0, 11.2. **HRMS m/z (ESI)** calcd. for $C_{10}H_{13}N_2O_2S^+$ (M + H) $^+$ 225.0698, found 225.0692.

Ethyl 1,5-dimethyl-4-thiocyanato-1H-pyrrole-2-carboxylate (4h)



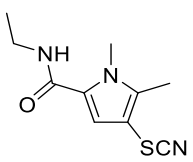
Following the general procedure B on 0.2 mmol scale, the substrate **4h** was obtained as a white solid in 84% yield (37.6 mg). mp 95.5-96.5 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.10 (s, 1H), 4.26 (q, J = 7.3 Hz, 2H), 3.86 (s, 3H), 2.38 (s, 3H), 1.32 (t, J = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 160.5, 141.4, 123.4, 121.9, 111.7, 97.0, 60.3, 33.6, 14.4, 10.8. **HRMS m/z (ESI)** calcd. for $C_{10}H_{13}N_2O_2S^+$ (M + H) $^+$ 225.0698, found 225.0692.

Methyl 1,5-dimethyl-4-thiocyanato-1H-pyrrole-2-carboxylate (4i)



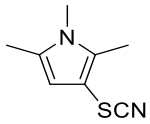
Following the general procedure B on 0.2 mmol scale, the substrate **4i** was obtained as a white solid in 78% yield (32.8 mg). mp 98.4-98.5 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.16 (s, 1H), 3.93 (s, 3H), 3.87 (s, 3H), 2.45 (s, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 160.9, 141.5, 122.9, 122.0, 111.7, 97.1, 51.4, 33.6, 10.9. **HRMS m/z (ESI)** calcd. for $C_9H_{11}N_2O_2S^+$ (M + H) $^+$ 211.0541, found 211.0536.

N-ethyl-1,5-dimethyl-4-thiocyanato-1H-pyrrole-2-carboxamide (4j)



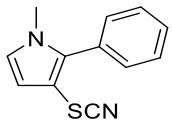
Following the general procedure B on 0.2 mmol scale, the substrate **4j** was obtained as a white solid in 88% yield (39.2 mg). mp 67.3-68.3 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 6.73 (s, 1H), 6.07 (s, 1H), 3.90 (s, 3H), 3.43 (p, J = 7.2 Hz, 2H), 2.41 (s, 3H), 1.25 (t, J = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.1, 139.9, 127.1, 115.4, 112.1, 95.6, 34.4, 33.5, 14.9, 10.7. **HRMS m/z (ESI)** calcd. for $C_{10}H_{14}N_3OS^+$ (M + H)$^+$ 224.0858, found 224.0853.

1,2,5-trimethyl-3-thiocyanato-1H-pyrrole (4ae)



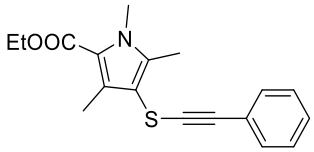
Following the general procedure B on 0.2 mmol scale, the substrate **4ae** was obtained as a colorless liquid in 86% yield (28.6 mg). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 5.94 (s, 1H), 3.34 (s, 3H), 2.25 (s, 3H), 2.11 (s, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ134.3, 129.4, 113.1, 110.2, 93.2, 31.15, 12.3, 10.7. **HRMS m/z (ESI)** calcd. for $C_8H_{11}N_2S^+$ (M + H)$^+$ 167.0643, found 167.0646.

1-methyl-2-phenyl-3-thiocyanato-1H-pyrrole (4af)



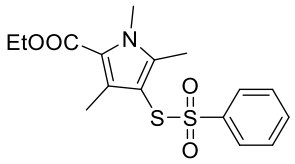
Following the general procedure B on 0.2 mmol scale, the substrate **4af** was obtained as a white oil in 58% yield (24.8 mg). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.49-7.36 (m, 5H), 6.72 (d, J = 3.9 Hz, 1H), 6.27 (d, J = 3.9 Hz, 1H), 3.77 (s, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 141.2, 132.3, 129.0, 128.7, 128.2, 120.7, 110.3, 110.1, 106.1, 32.7. **HRMS m/z (ESI)** calcd. for $C_{12}H_{11}N_2S^+$ (M + H)$^+$ 215.0643, found 215.0640.

Ethyl 1,3,5-trimethyl-4-((phenylethynyl)thio)-1H-pyrrole-2-carboxylate (5)



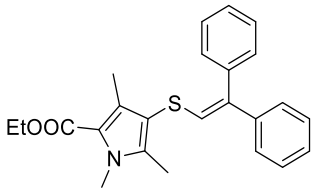
Following the general procedure E on 0.1 mmol scale, the substrate **5** was obtained as a colorless oil in 68% yield (21.4 mg). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.41 (d, J = 2.4 Hz, 2H), 7.31 (d, J = 1.2 Hz, 3H), 4.36 (q, J = 7.2 Hz, 2H), 3.88 (s, 3H), 2.51 (d, J = 27.6 Hz, 6H), 1.43 (t, J = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 162.0, 139.3, 131.8, 131.6, 128.2, 128.0, 123.4, 119.8, 107.0, 88.4, 80.1, 59.8, 33.7, 14.5, 12.5, 11.3. **HRMS m/z (ESI)** calcd. for $C_{18}H_{20}N_2OS^+$ (M + H)$^+$ 314.1215, found 314.1210.

Ethyl 1,3,5-trimethyl-4-((phenylsulfonyl)thio)-1H-pyrrole-2-carboxylate (6)



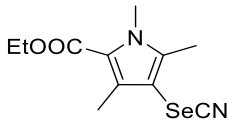
Following the general procedure F on 0.1 mmol scale, the substrate **6** was obtained as a white solid in 82% yield (28.9 mg). mp 158.6-159.6 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.59 (ddd, J = 12.2, 7.8, 1.2 Hz, 3H), 7.44 (dd, J = 11.2, 4.4 Hz, 2H), 4.26 (q, J = 7.2 Hz, 2H), 3.78 (s, 3H), 2.01 (s, 3H), 1.91 (s, 3H), 1.32 (t, J = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.8, 143.7, 143.4, 133.8, 133.6, 129.1, 127.6, 120.6, 105.4, 60.1, 34.1, 14.5, 11.9, 11.0. **HRMS m/z (ESI)** calcd. for $C_{16}H_{20}NO_4S^+$ $(M + H)^+$ 354.0834, found 354.0829.

Ethyl 4-((2,2-diphenylvinyl)thio)-1,3,5-trimethyl-1H-pyrrole-2-carboxylate (7)



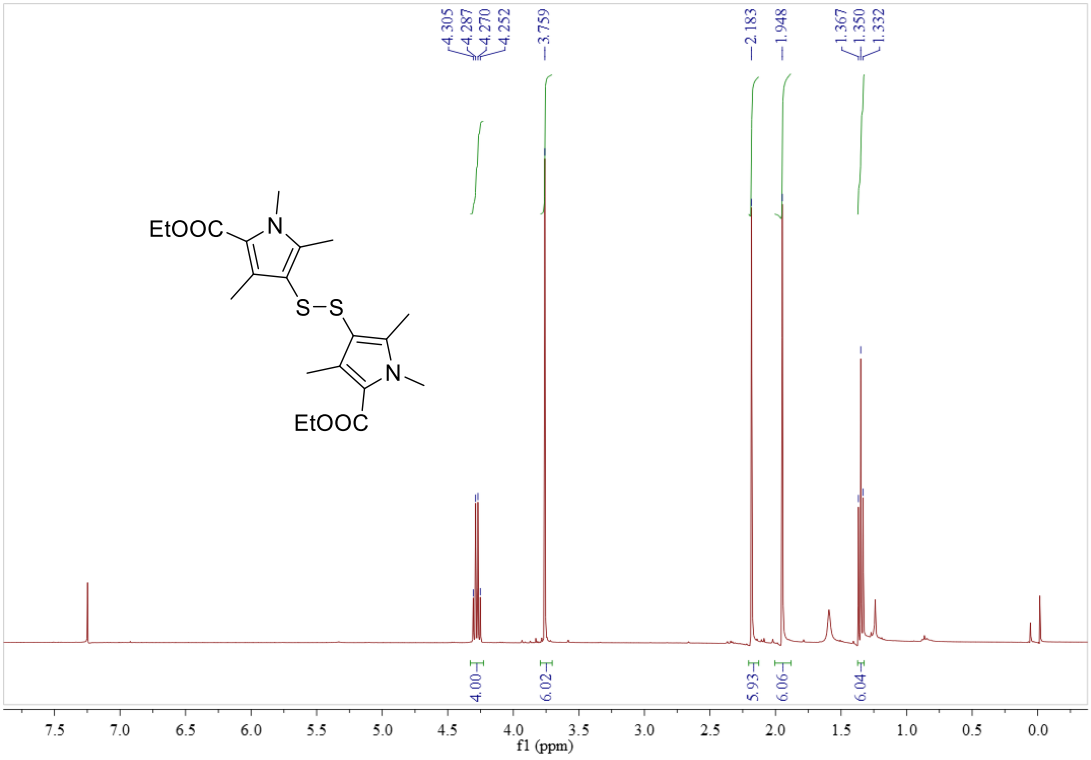
Following the general procedure C on 0.2 mmol scale, the substrate **7** was obtained as a colorless oil in 55% yield (43.0 mg). **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 7.54-7.41 (m, 5H), 7.31-7.20 (m, 5H), 6.40 (s, 1H), 4.37 (q, J = 7.2 Hz, 2H), 3.88 (s, 3H), 2.45 (s, 3H), 2.38 (s, 3H), 1.43 (t, J = 7.2 Hz, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 162.1, 141.7, 139.7, 139.4, 137.7, 132.3, 129.9, 129.5, 128.5, 128.3, 127.6, 127.0, 126.9, 119.6, 111.6, 59.8, 33.7, 14.6, 12.8, 11.4. **HRMS m/z (ESI)** calcd. for $C_{24}H_{26}NO_2S^+$ $(M + H)^+$ 392.1684, found 392.1679.

Ethyl 1,3,5-trimethyl-4-selenocyanato-1H-pyrrole-2-carboxylate (8a')

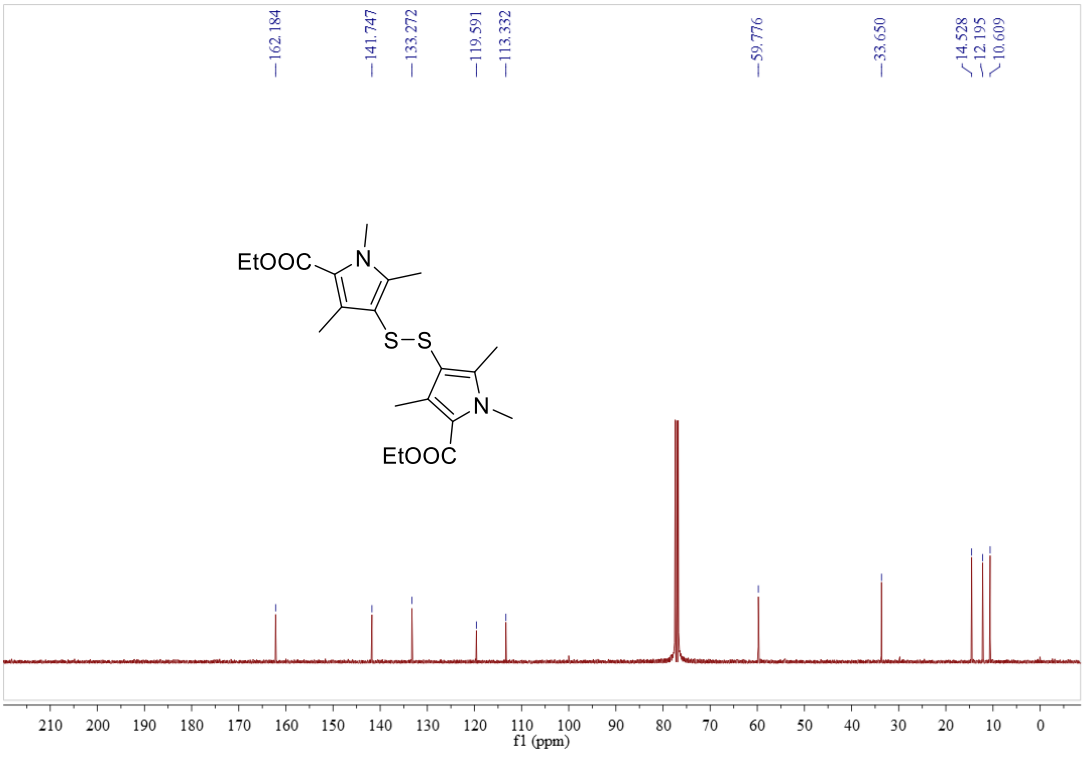


Following the general procedure B on 0.2 mmol scale, the substrate **8a'**was obtained as a white solid in 35% yield (20.0 mg). mp 91.3-92.3 °C. **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 4.32 (q, J = 7.1 Hz, 2H), 3.85 (s,3H), 2.44 (d, J = 5.8 Hz, 6H), 1.38 (t, J = 7.1 Hz, 3H). **^{13}C NMR (100 MHz, Chloroform-*d*)** δ 161.6, 140.7, 132.4, 120.8, 101.7, 98.2, 60.1, 34.2, 14.4, 13.8, 12.4. **HRMS m/z (ESI)** calcd. for $C_{11}H_{15}N_2O_2Se^+$ $(M + H)^+$ 287.0299, found 287.0302.

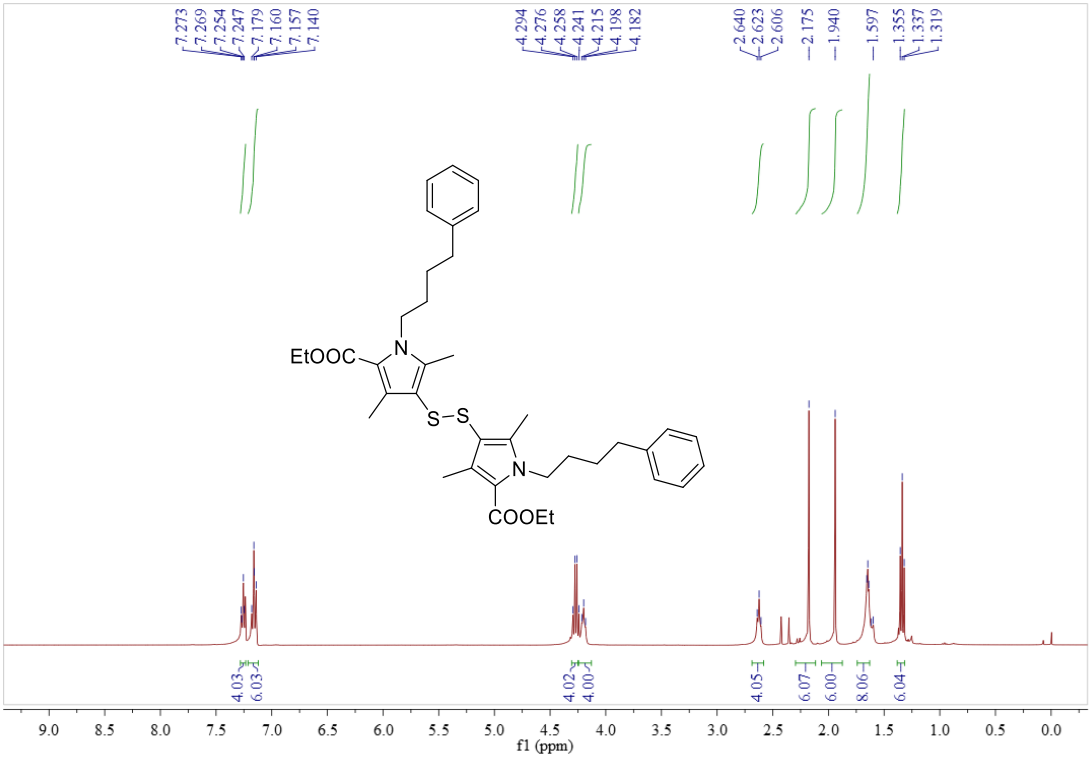
11. NMR spectra



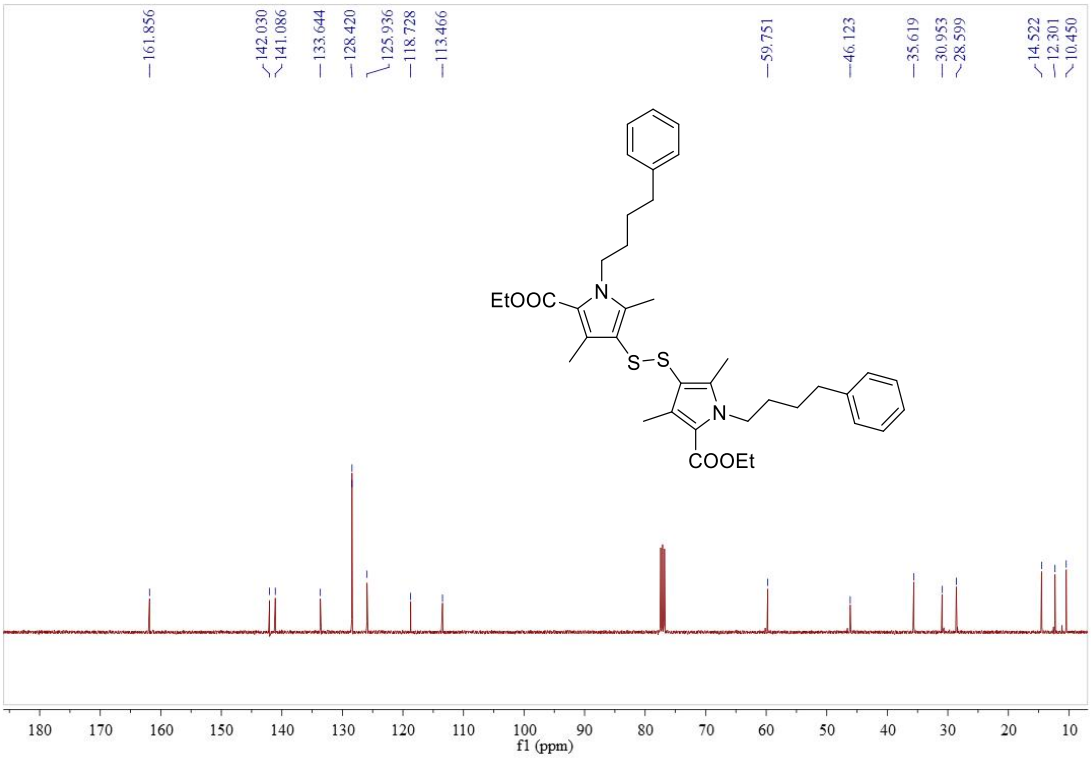
^{1}H NMR (400 MHz, Chloroform-*d*) of **3a**



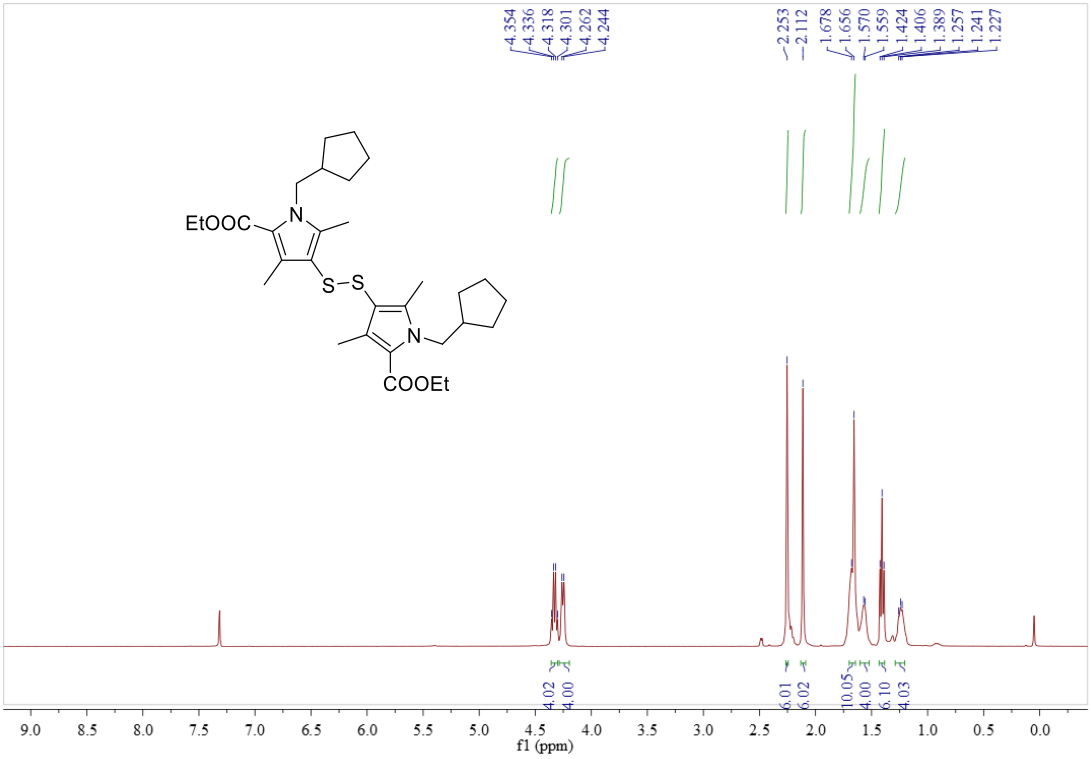
^{13}C NMR (100 MHz, Chloroform-*d*) of **3a**



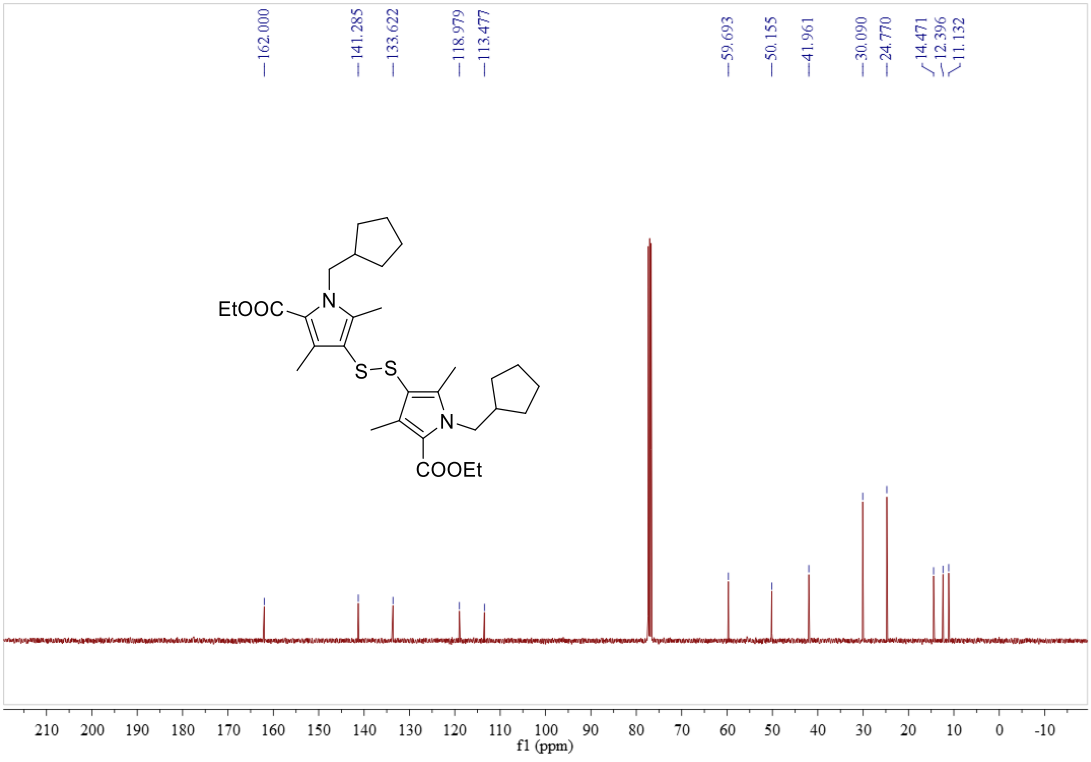
^{1}H NMR (400 MHz, Chloroform-*d*) of **3b**



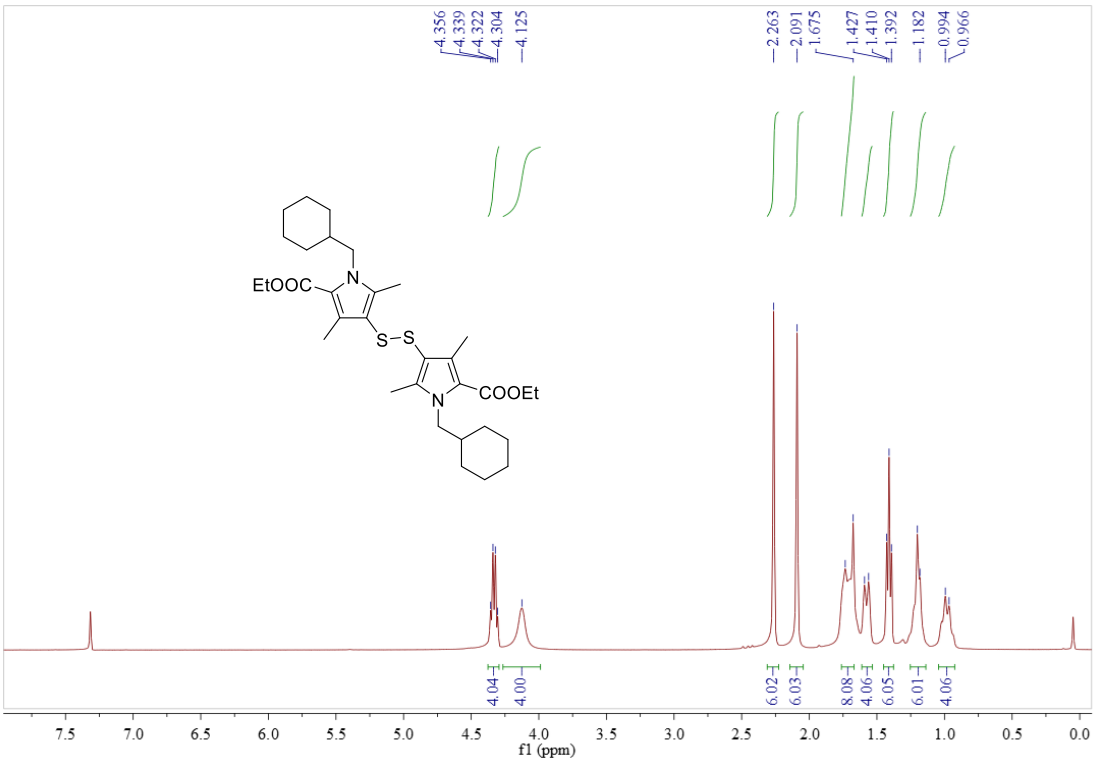
^{13}C NMR (100 MHz, Chloroform-*d*) of **3b**



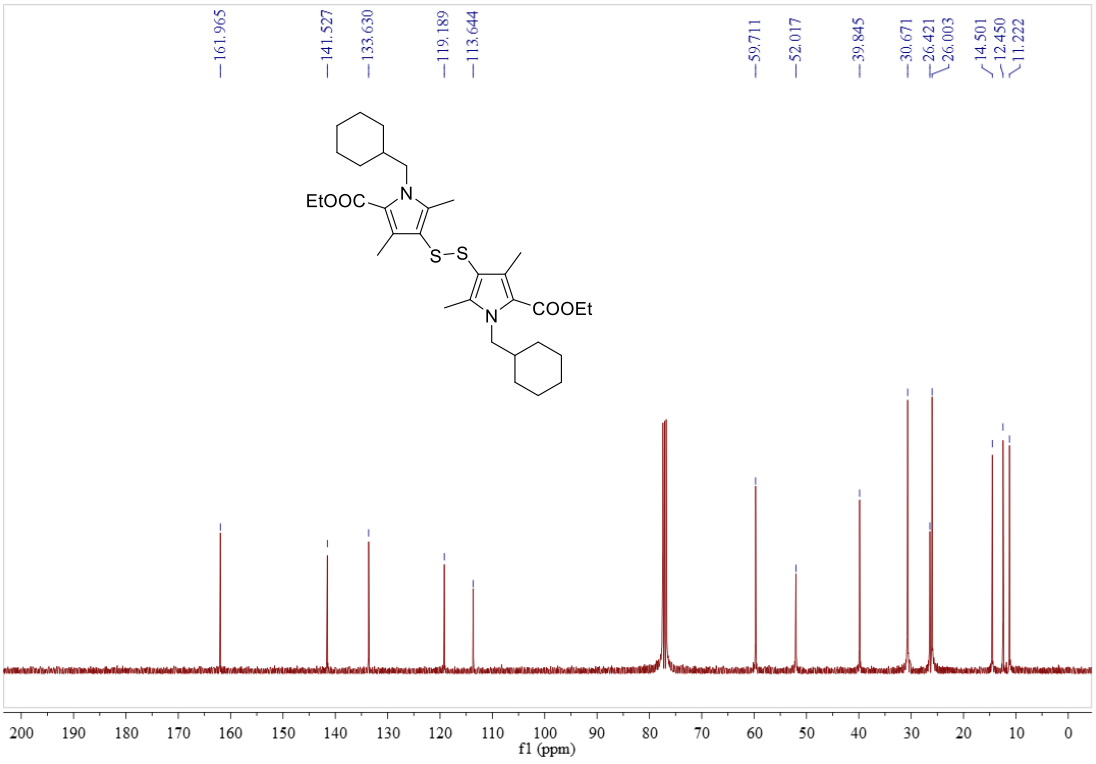
^{1}H NMR (400 MHz, Chloroform-*d*) of **3c**



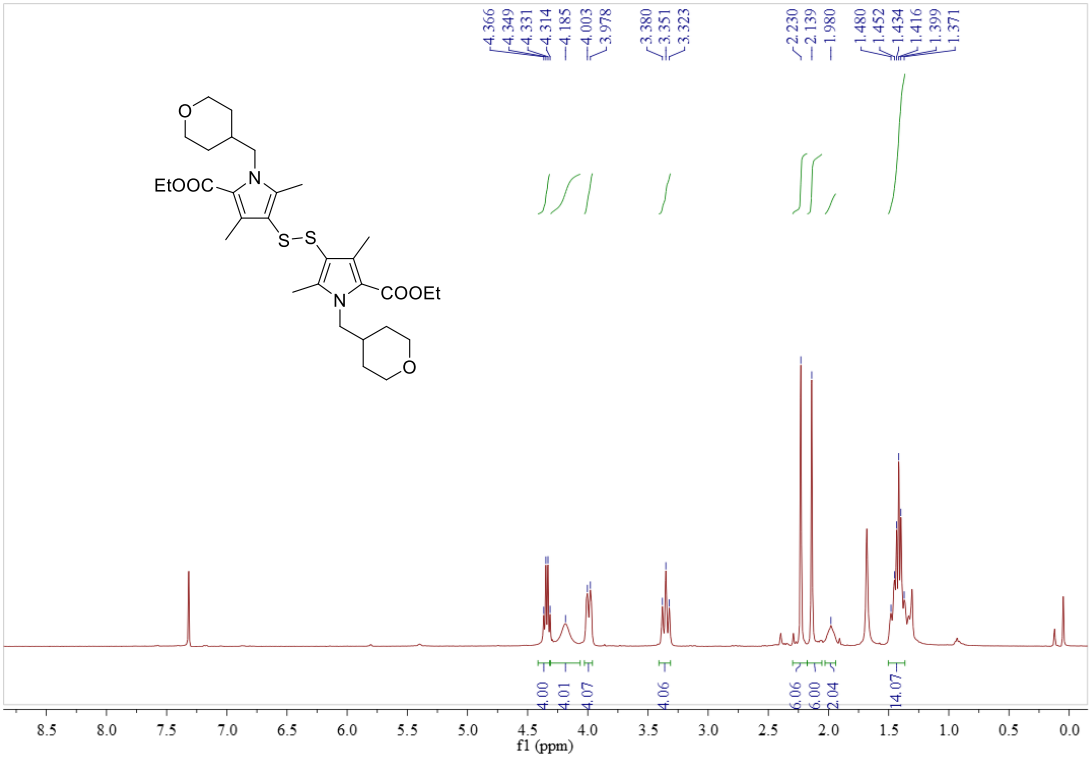
^{13}C NMR (100 MHz, Chloroform-*d*) of **3c**



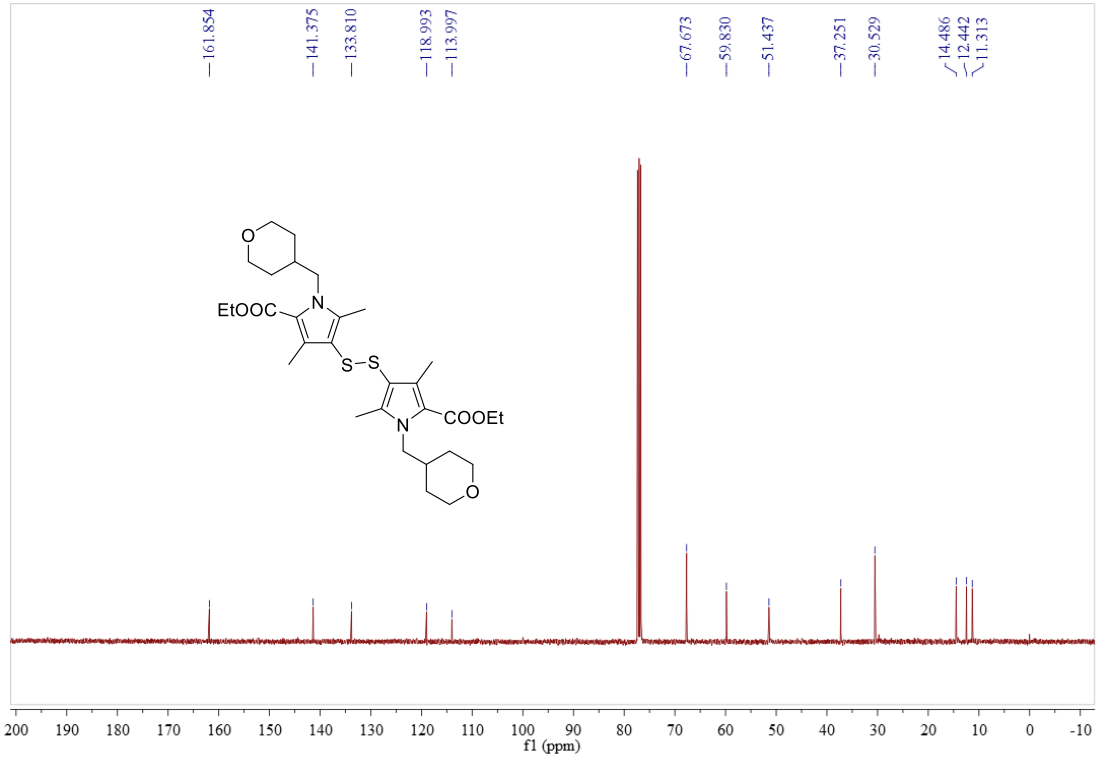
^{1}H NMR (400 MHz, Chloroform-*d*) of **3d**



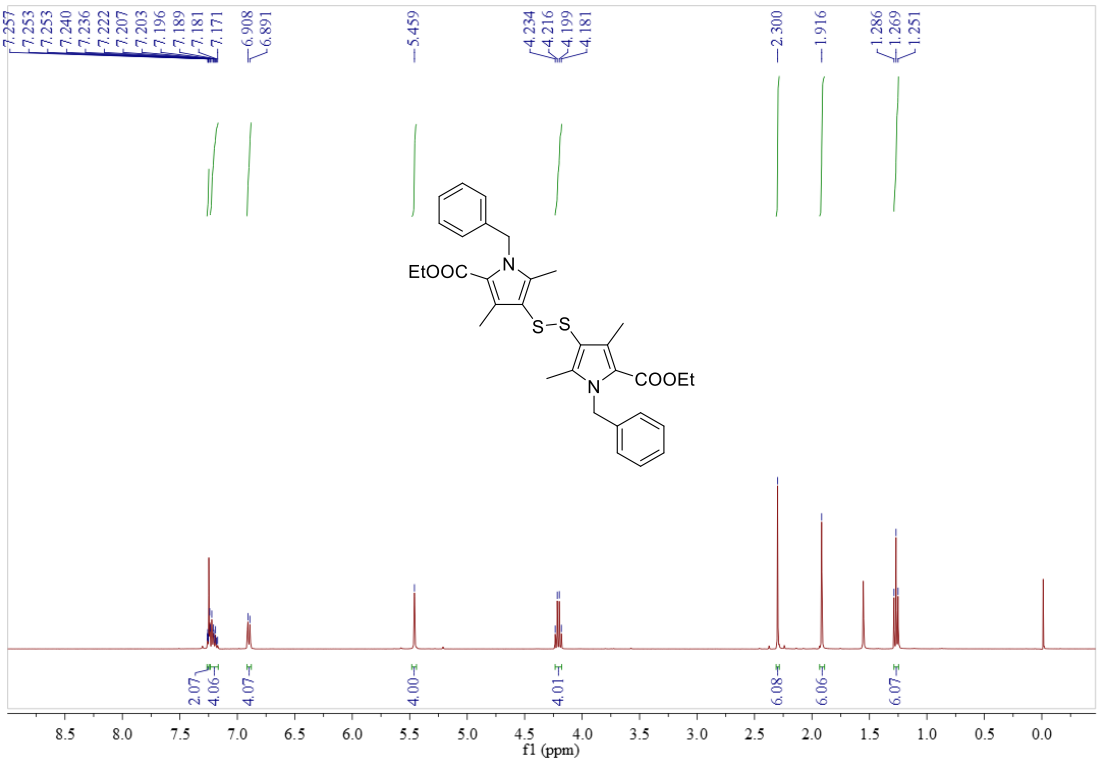
^{13}C NMR (100 MHz, Chloroform-*d*) of **3d**



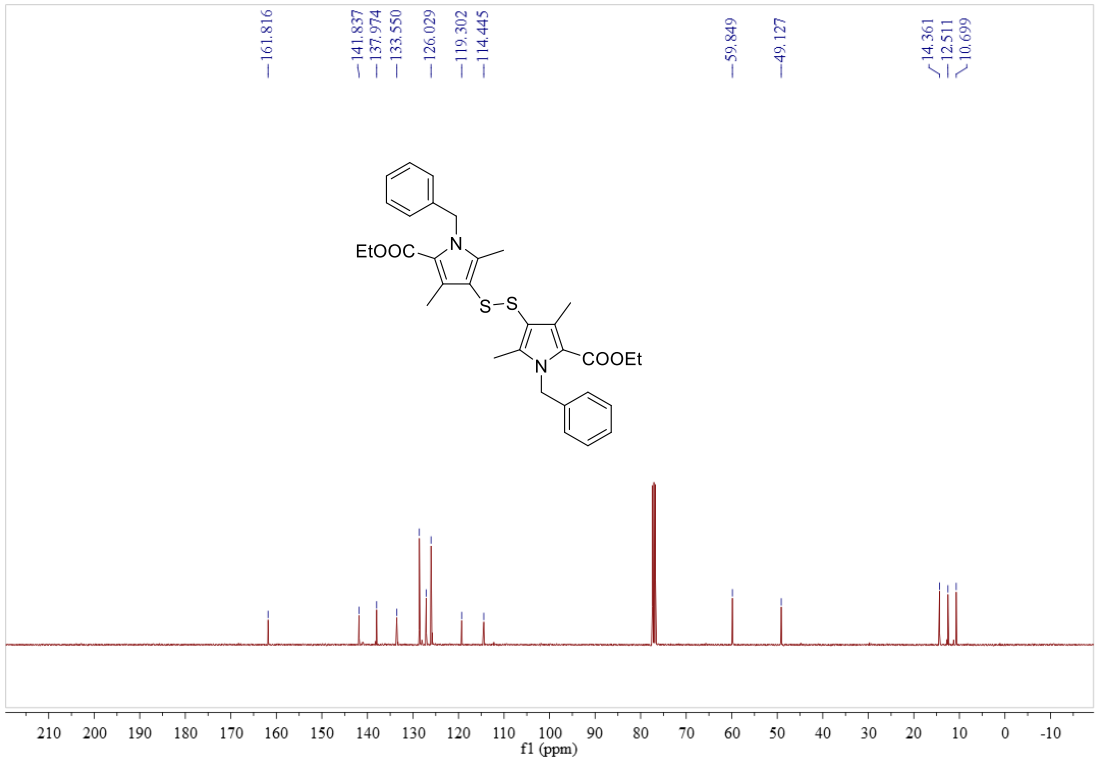
^{1}H NMR (400 MHz, Chloroform-*d*) of **3e**



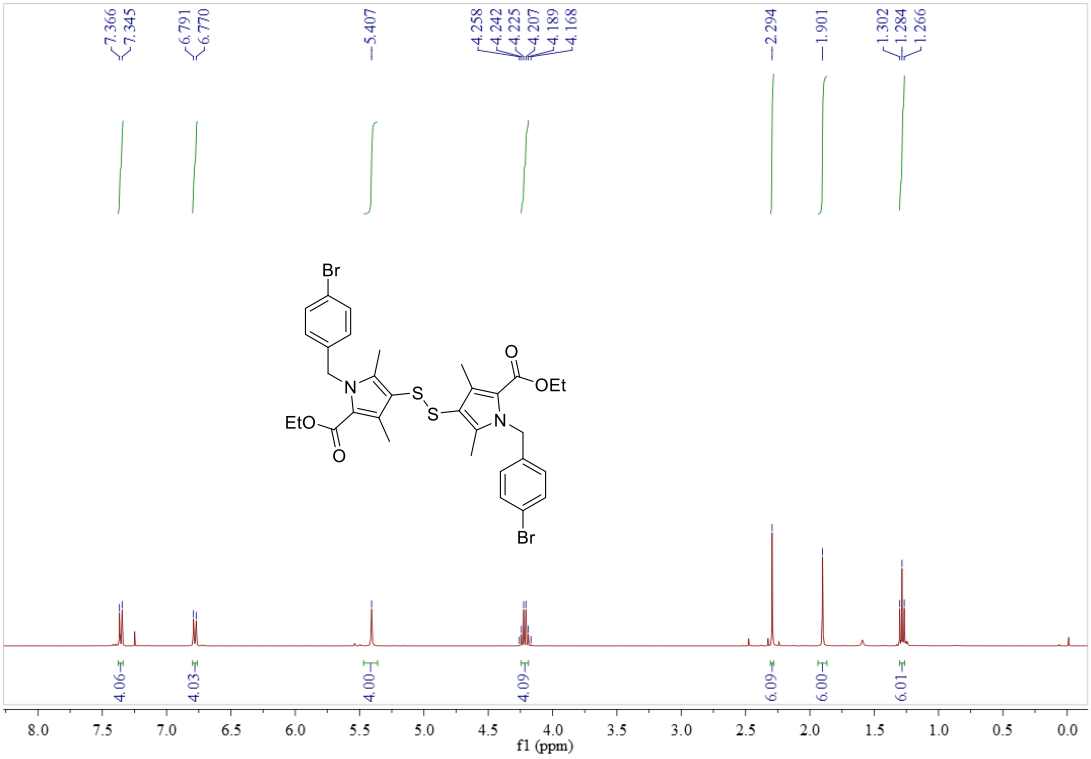
^{13}C NMR (100 MHz, Chloroform-*d*) of **3e**



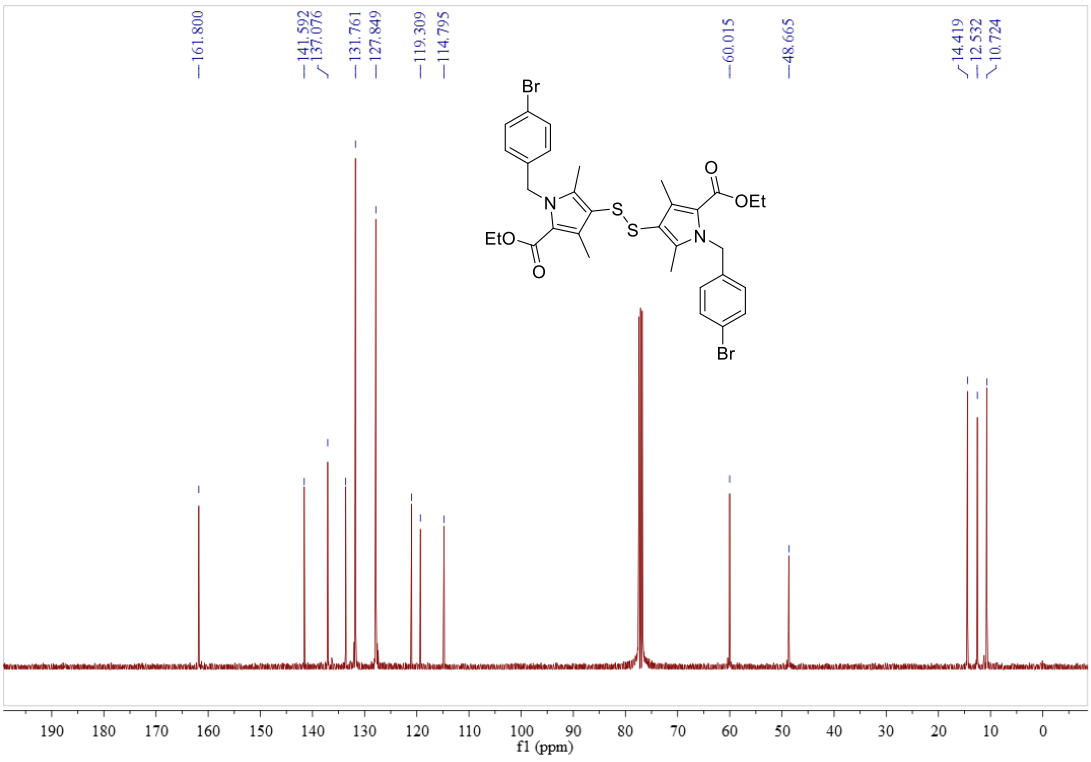
^{1}H NMR (400 MHz, Chloroform-*d*) of **3f**



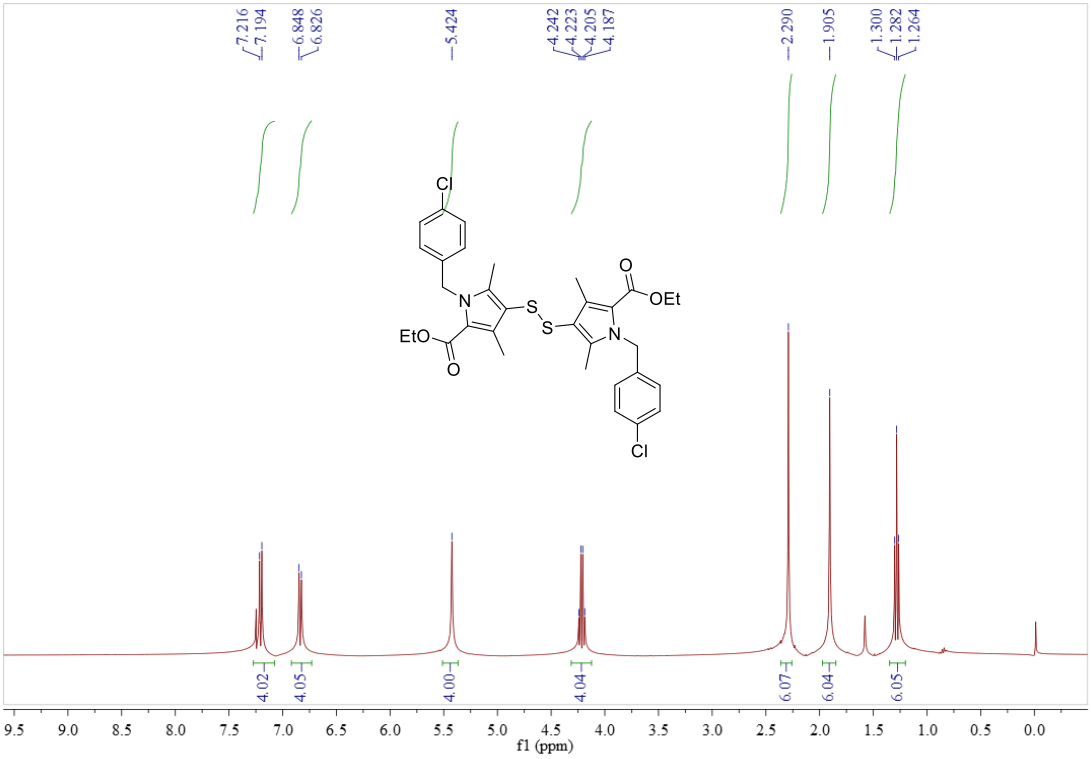
^{13}C NMR (100 MHz, Chloroform-*d*) of **3f**



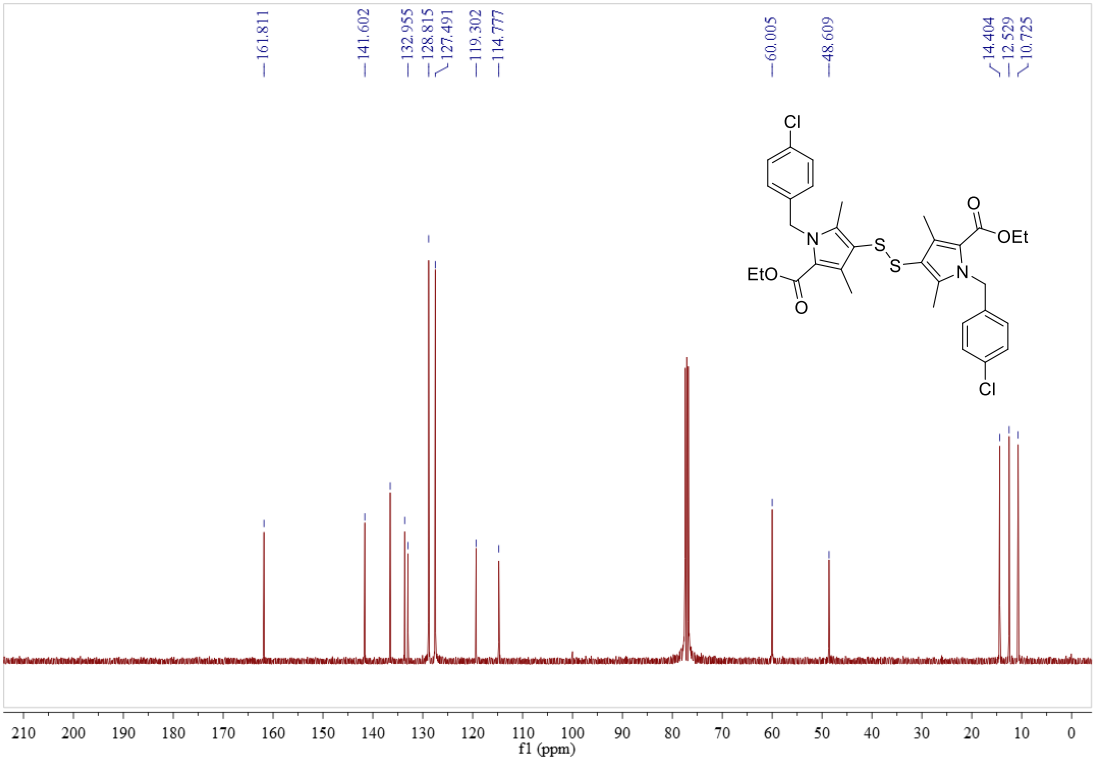
^{1}H NMR (400 MHz, Chloroform-*d*) of **3g**



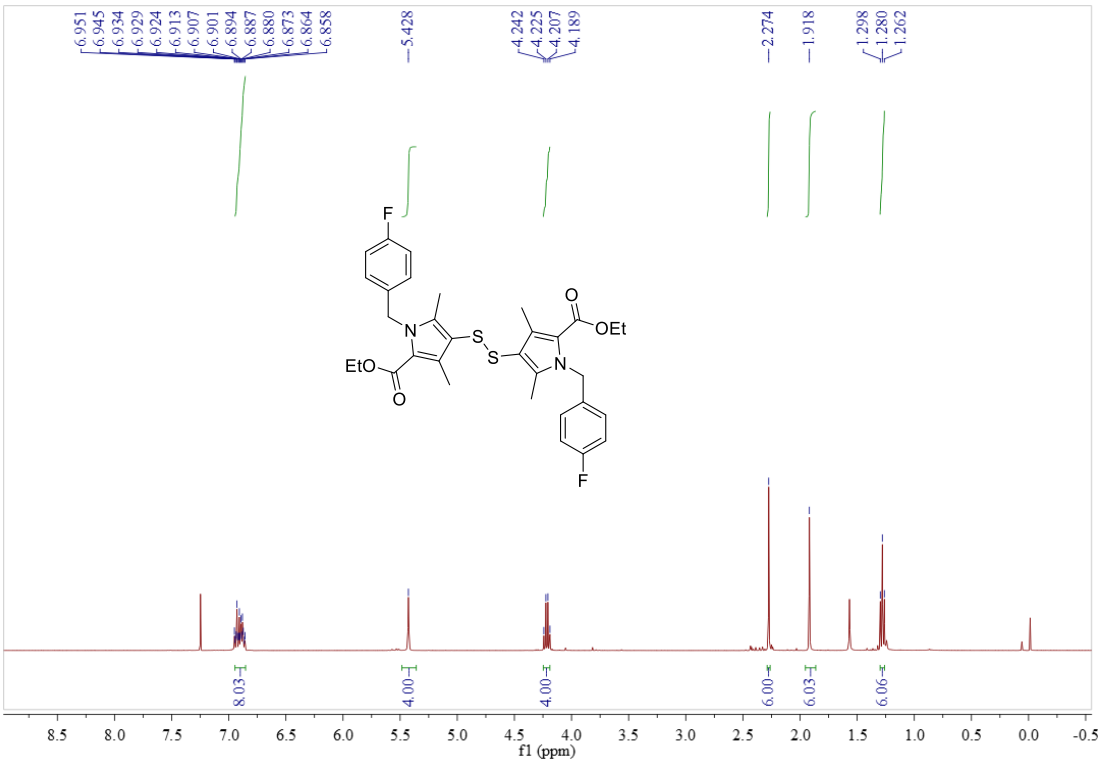
^{13}C NMR (100 MHz, Chloroform-*d*) of **3g**



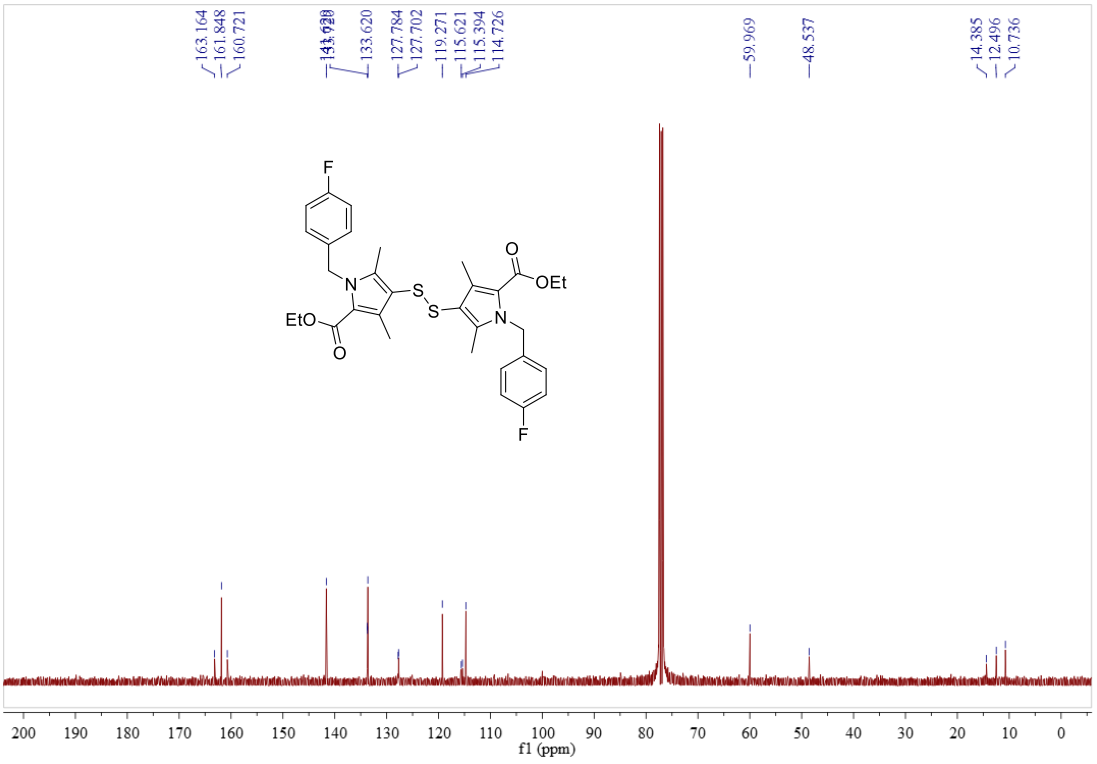
^{1}H NMR (400 MHz, Chloroform-*d*) of **3h**



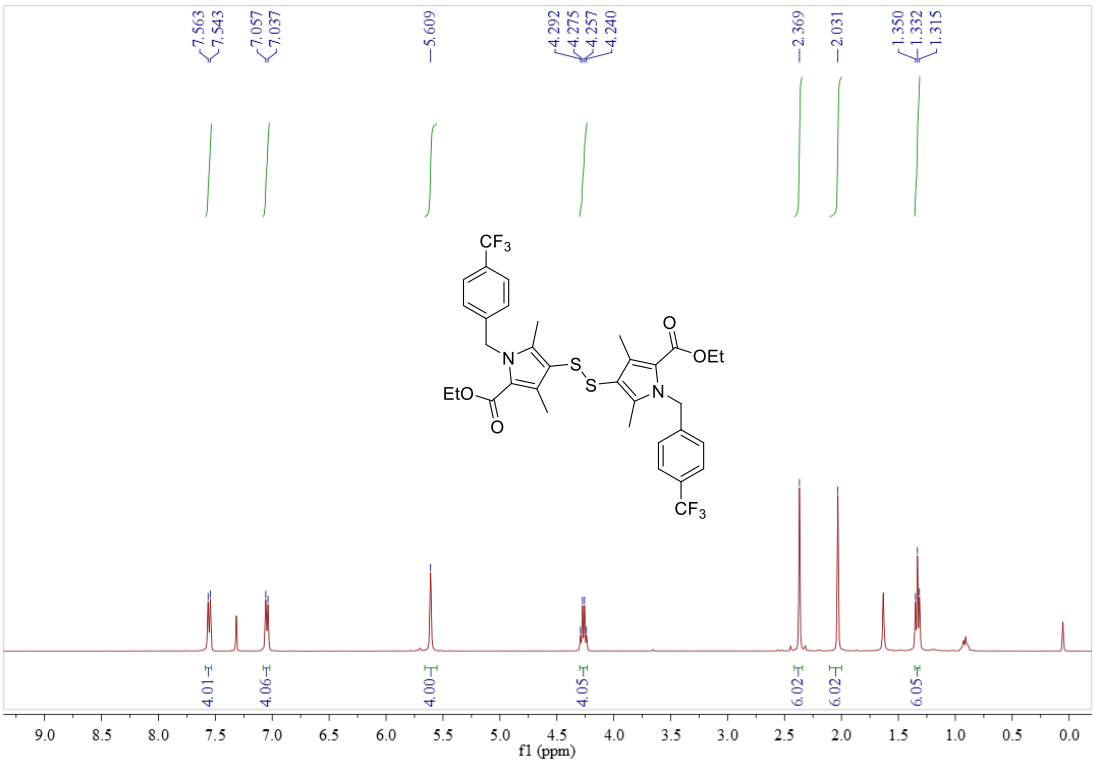
^{13}C NMR (100 MHz, Chloroform-*d*) of **3h**



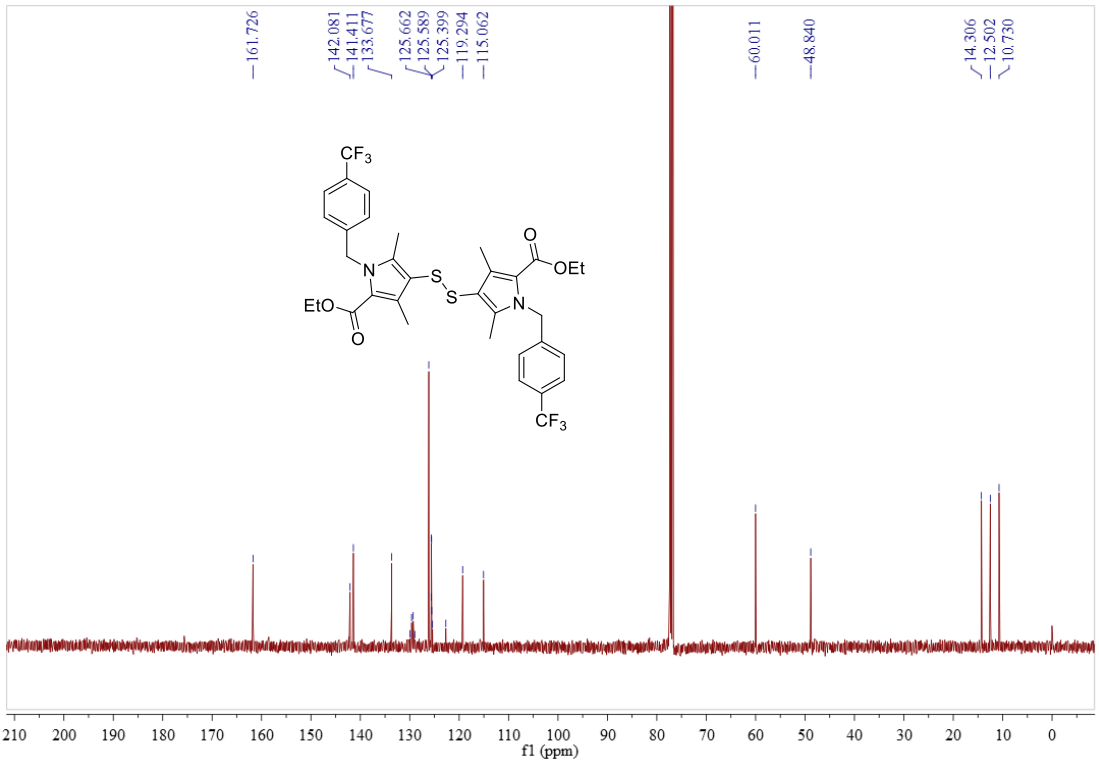
^{1}H NMR (400 MHz, Chloroform-*d*) of **3i**



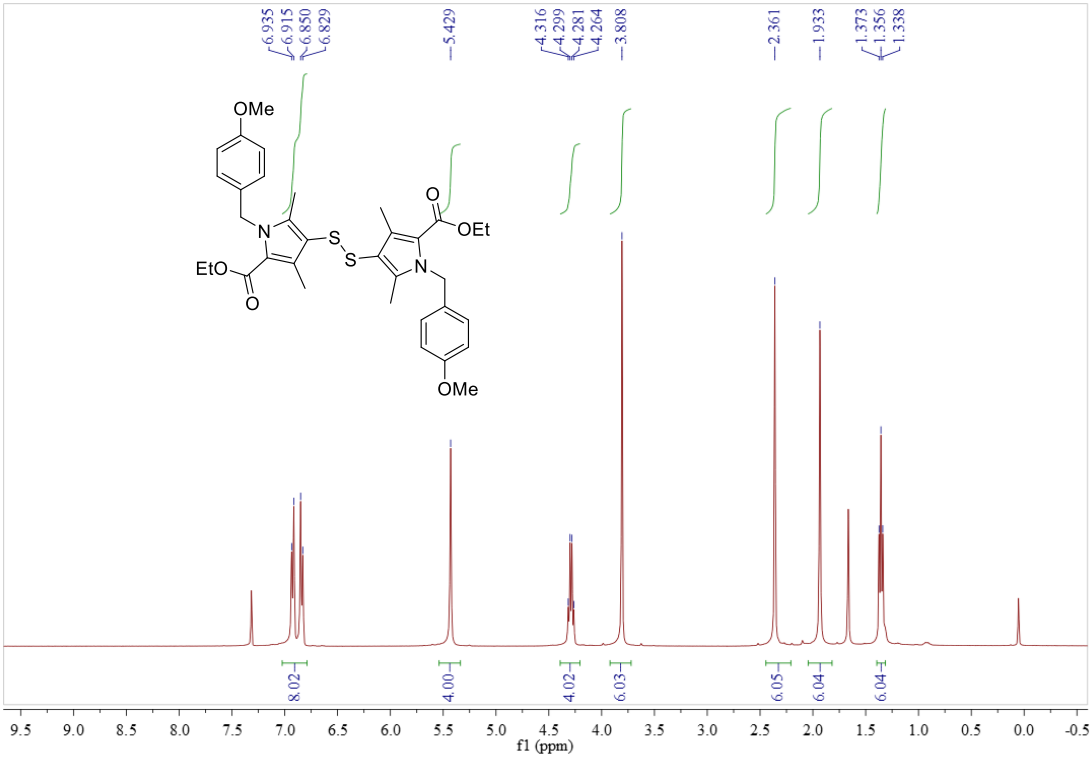
^{13}C NMR (100 MHz, Chloroform-*d*) of **3i**



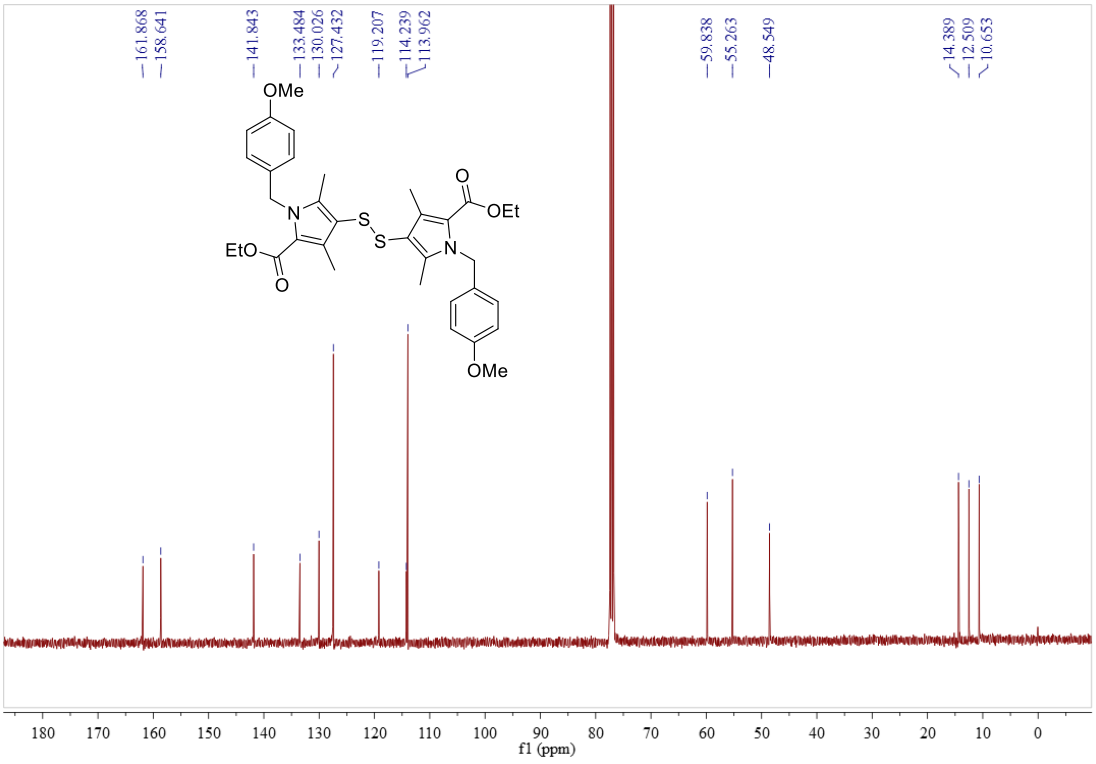
^{1}H NMR (400 MHz, Chloroform-*d*) of **3j**



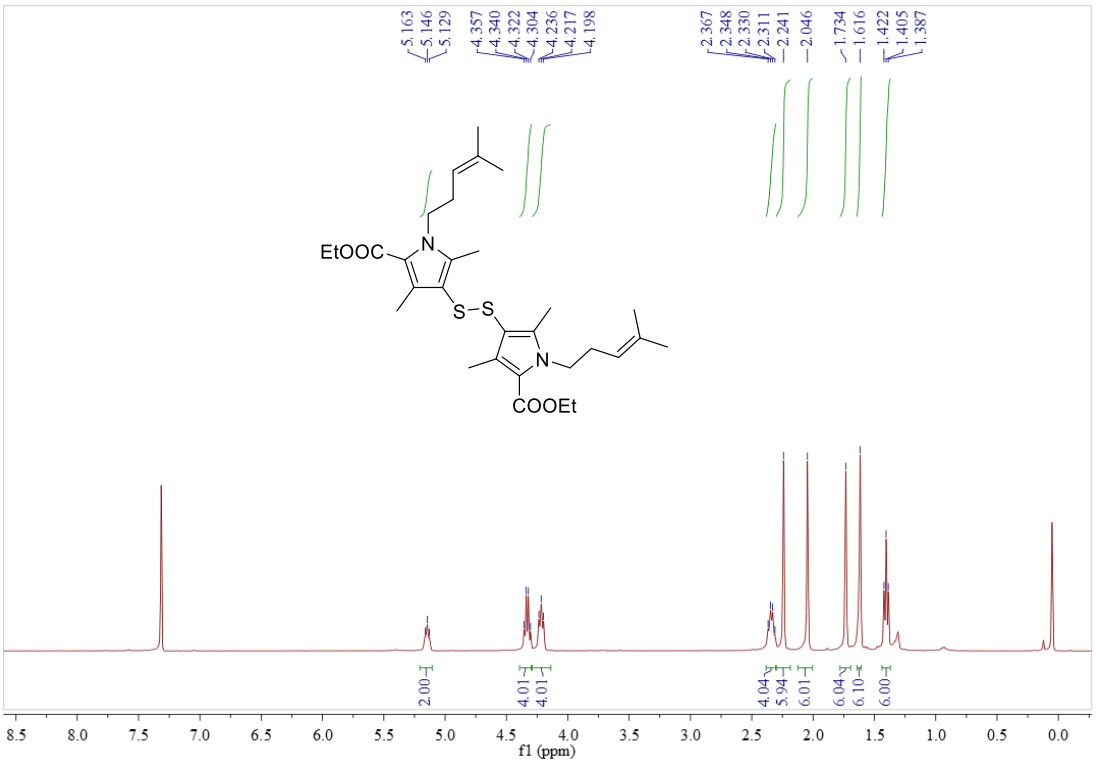
^{13}C NMR (100 MHz, Chloroform-*d*) of **3j**



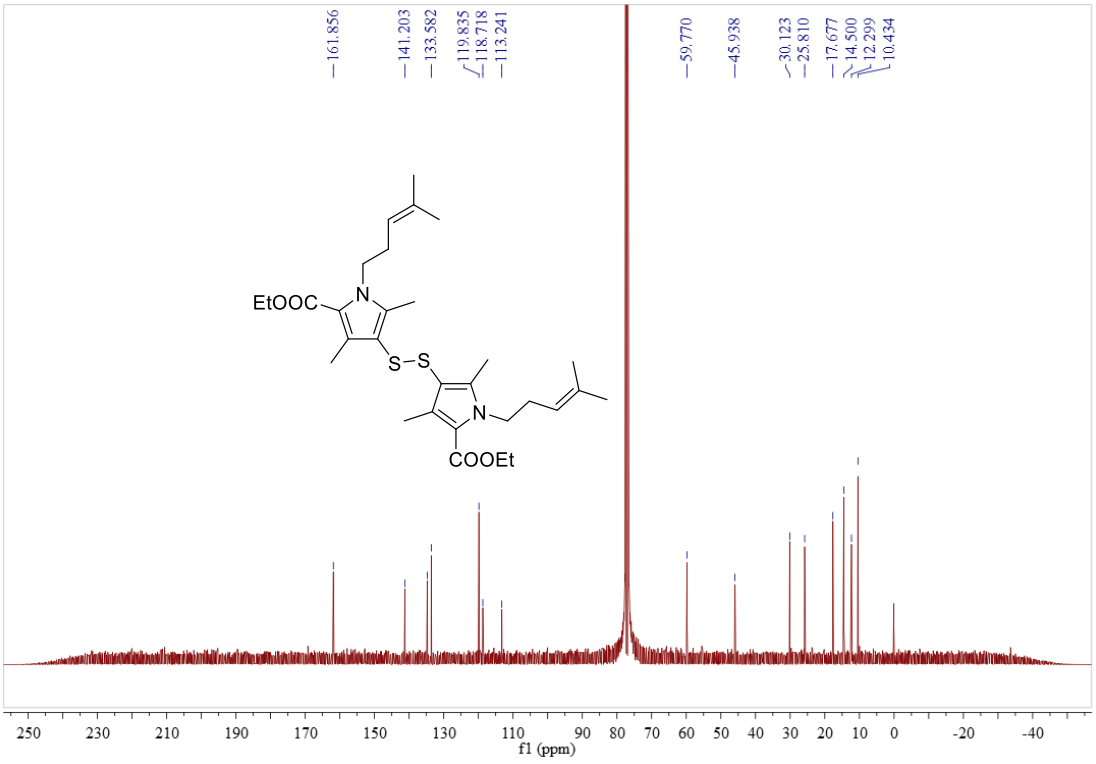
^{1}H NMR (400 MHz, Chloroform-*d*) of **3k**



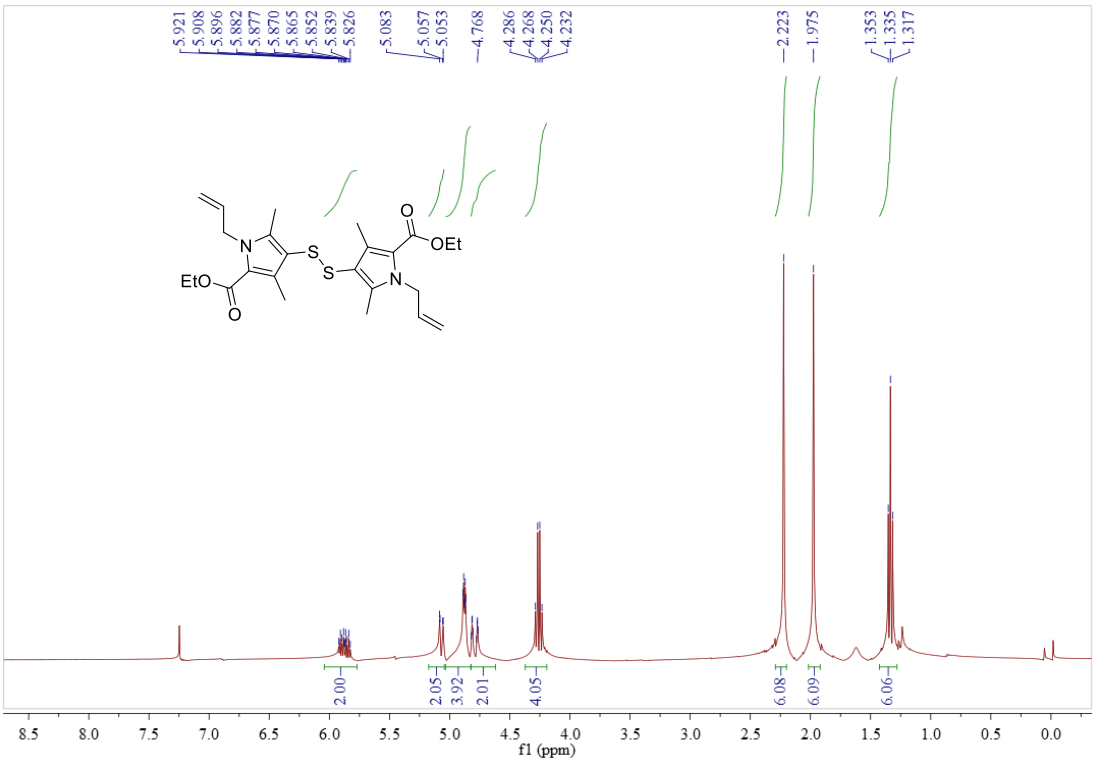
^{13}C NMR (100 MHz, Chloroform-*d*) of **3k**



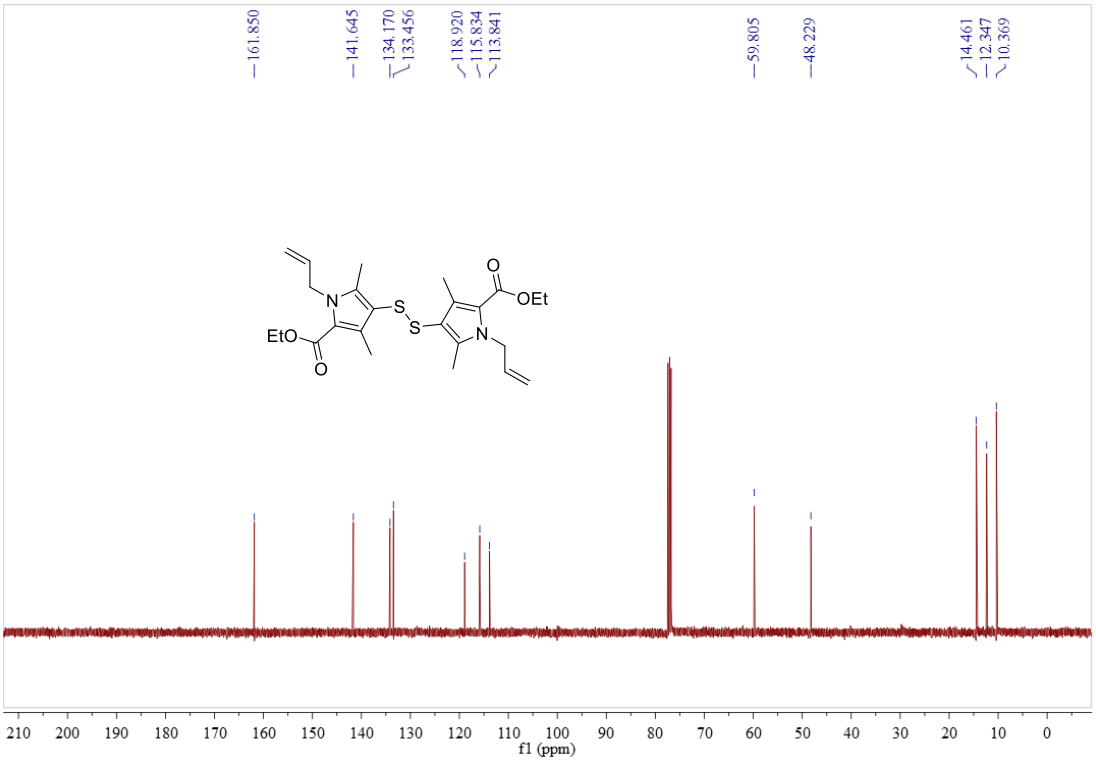
^{1}H NMR (400 MHz, Chloroform-*d*) of **3l**



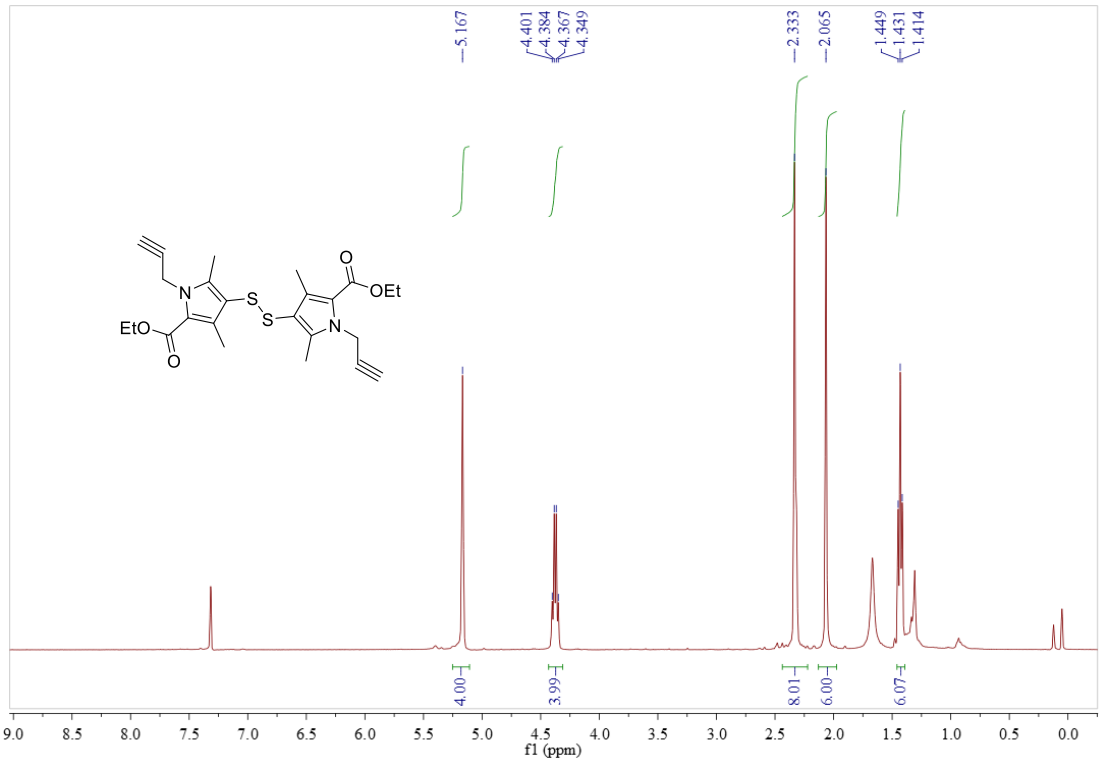
^{13}C NMR (100 MHz, Chloroform-*d*) of **3l**



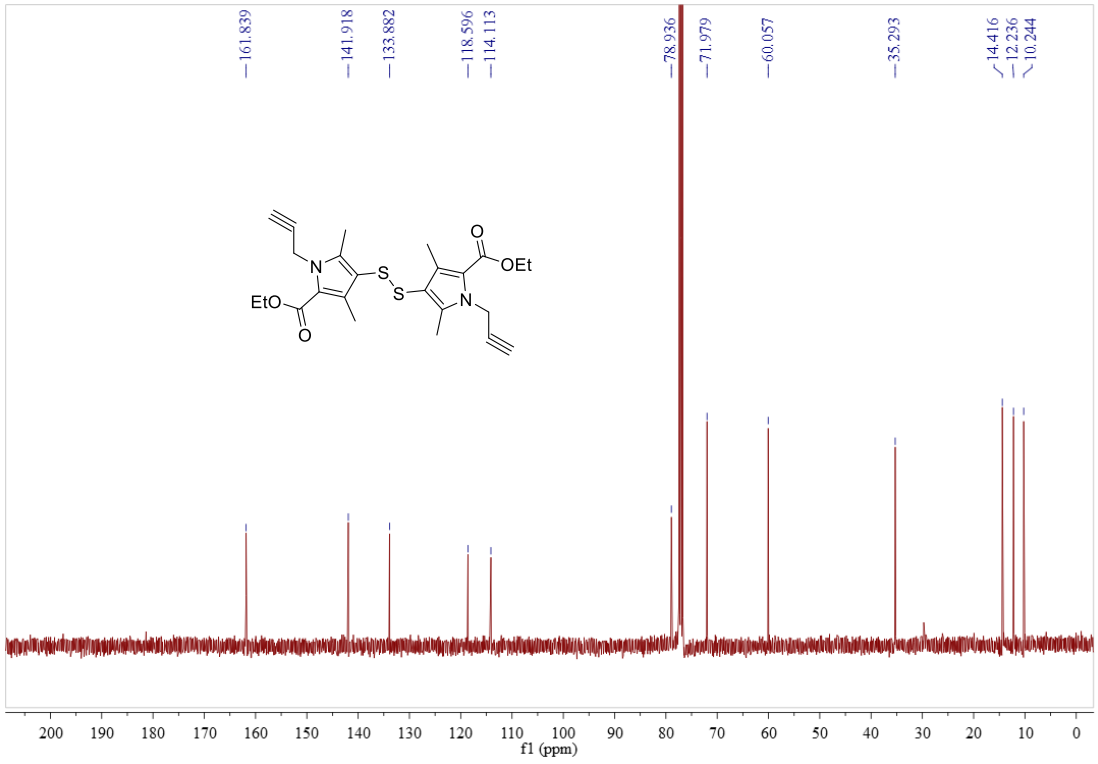
^{1}H NMR (400 MHz, Chloroform-*d*) of **3m**



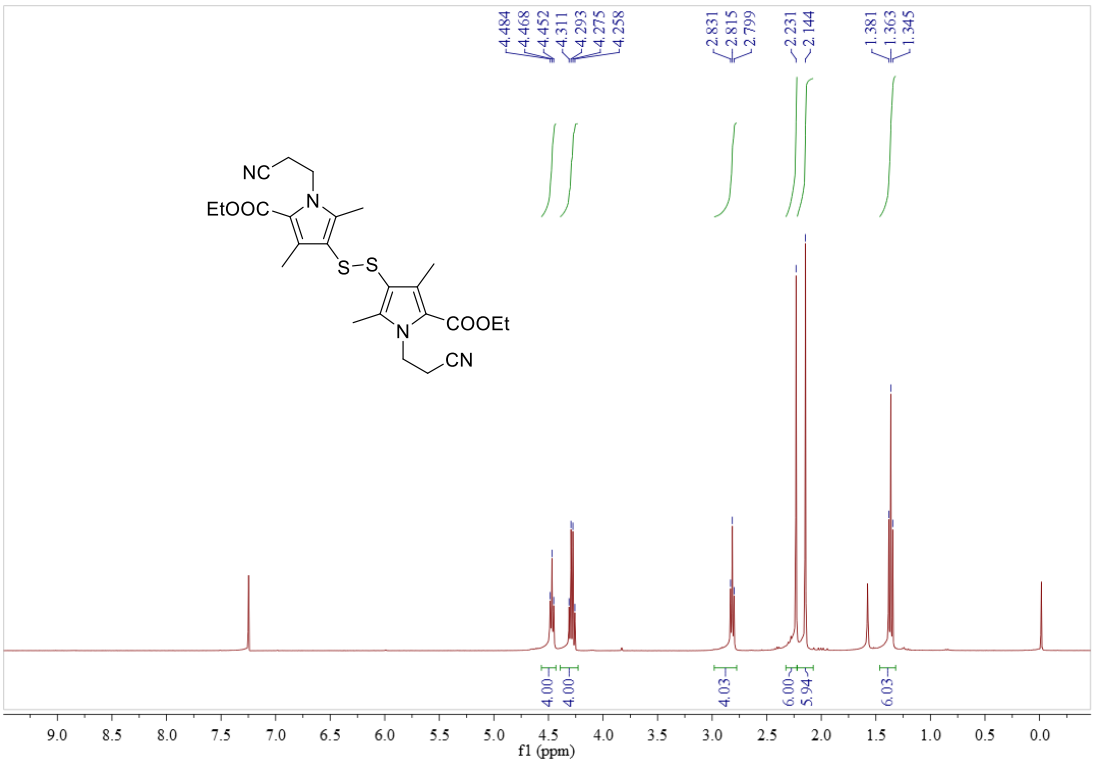
^{13}C NMR (100 MHz, Chloroform-*d*) of **3m**



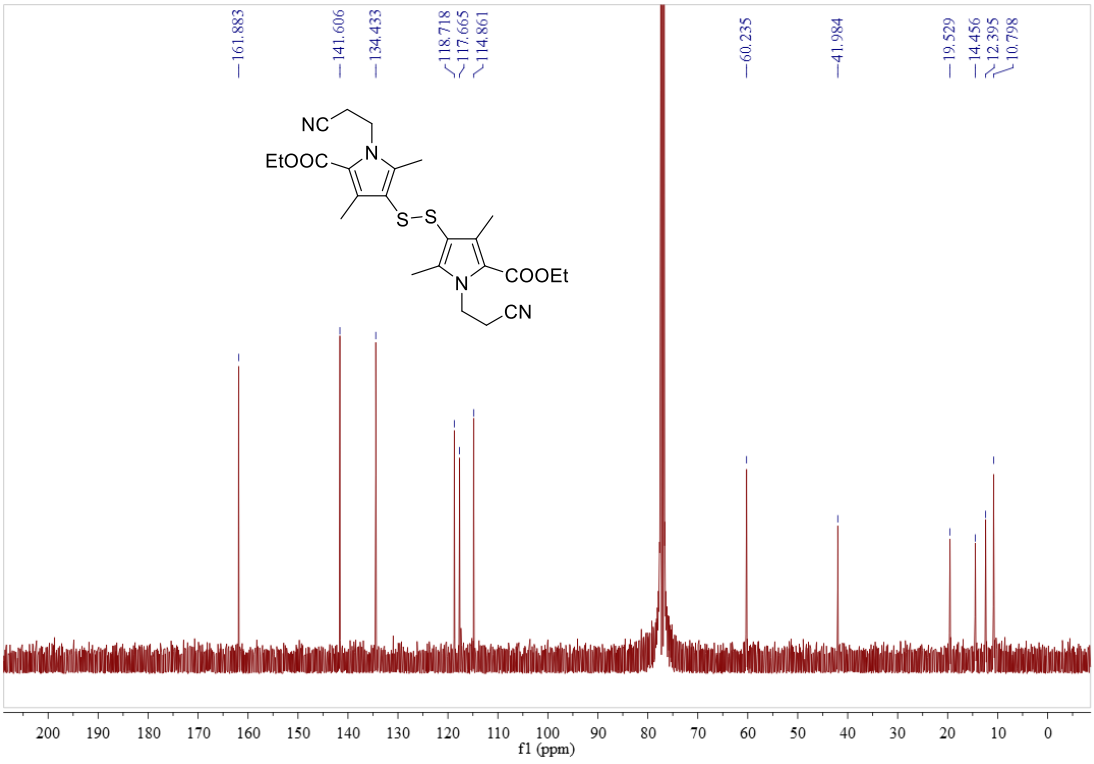
^{1}H NMR (400 MHz, Chloroform-*d*) of **3n**



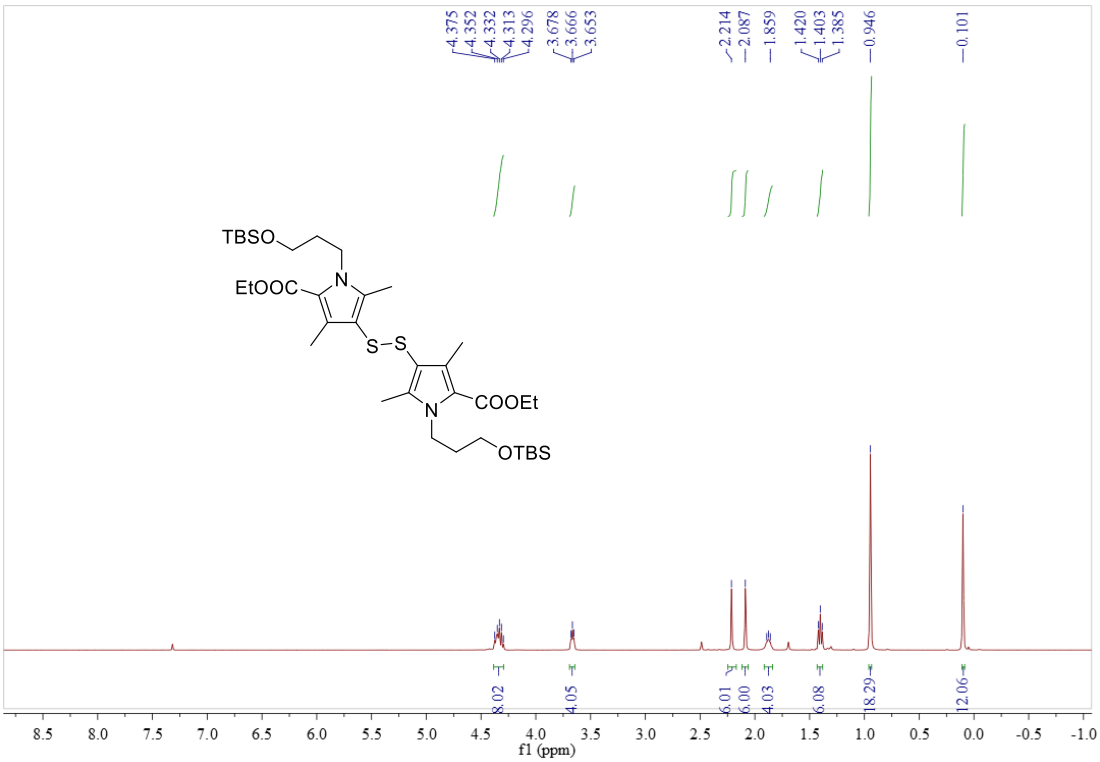
^{13}C NMR (100 MHz, Chloroform-*d*) of **3n**



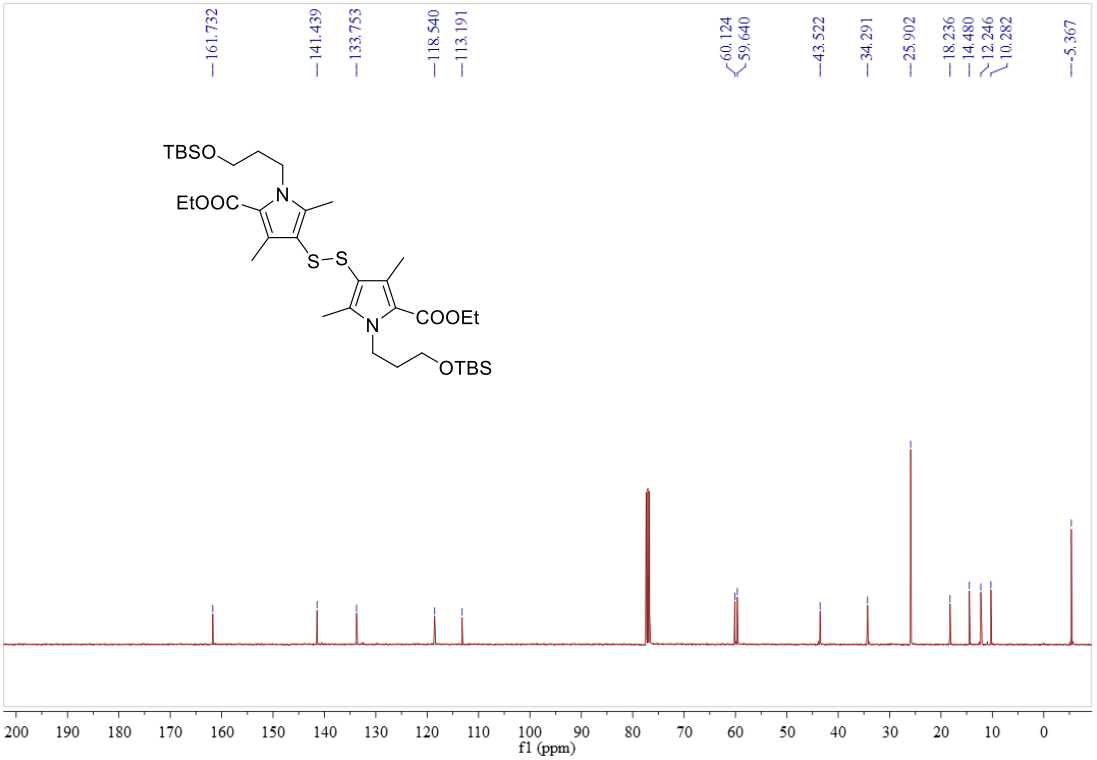
^{1}H NMR (400 MHz, Chloroform-*d*) of **3o**



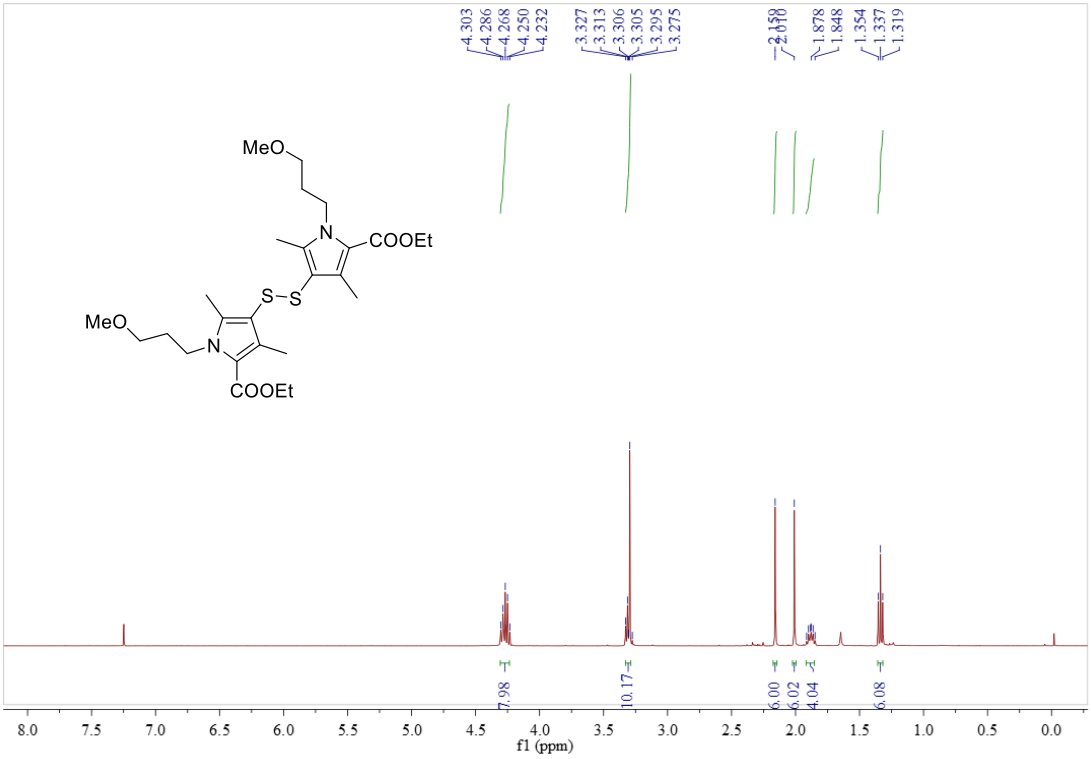
^{13}C NMR (100 MHz, Chloroform-*d*) of **3o**



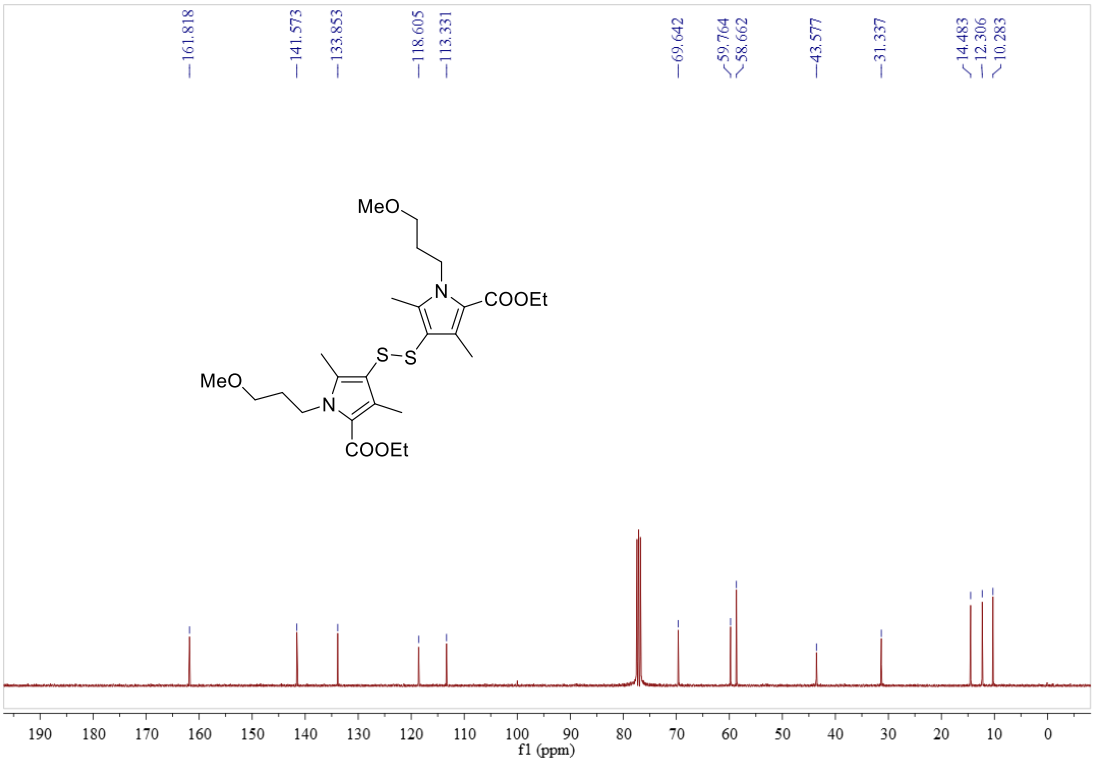
^{1}H NMR (400 MHz, Chloroform-*d*) of **3p**



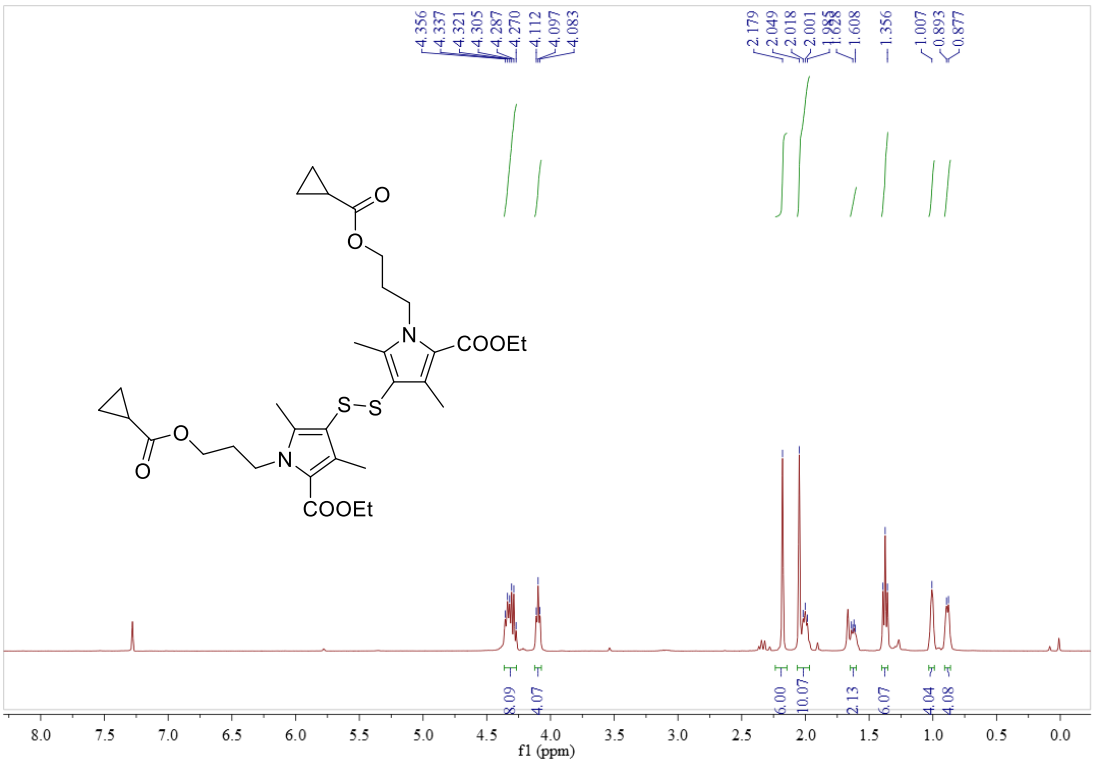
^{13}C NMR (100 MHz, Chloroform-*d*) of **3p**



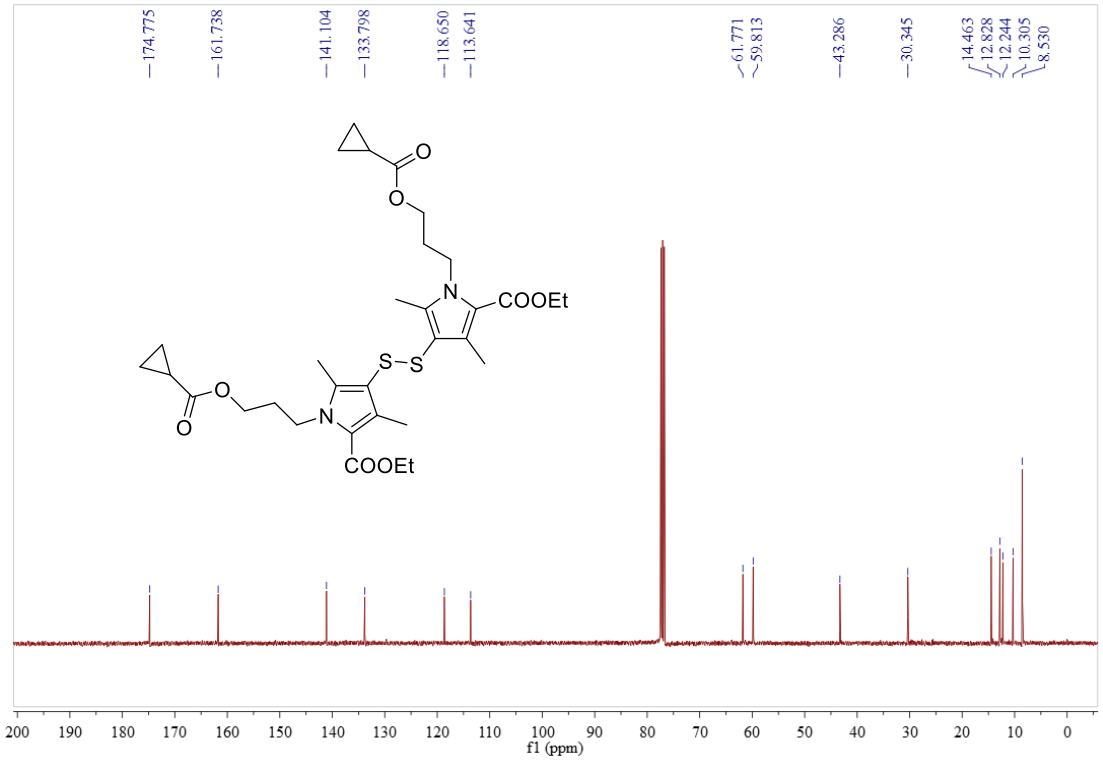
^{1}H NMR (400 MHz, Chloroform-*d*) of **3q**



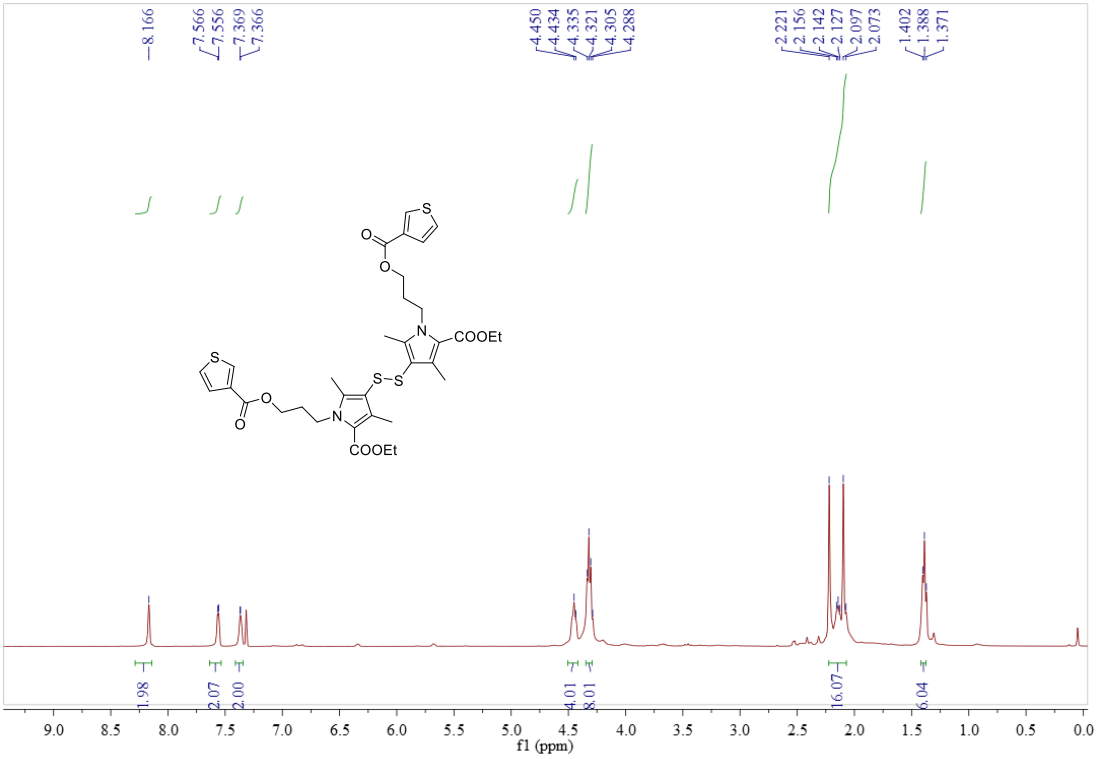
^{13}C NMR (100 MHz, Chloroform-*d*) of **3q**



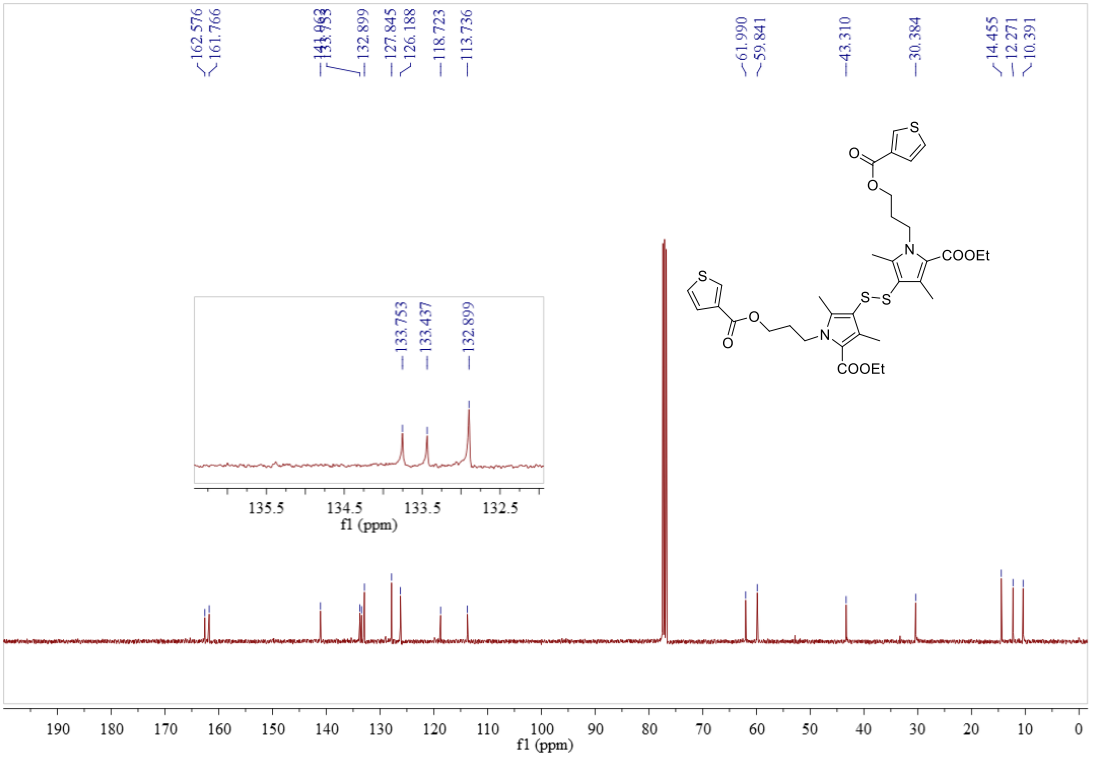
^{1}H NMR (400 MHz, Chloroform-*d*) of **3r**



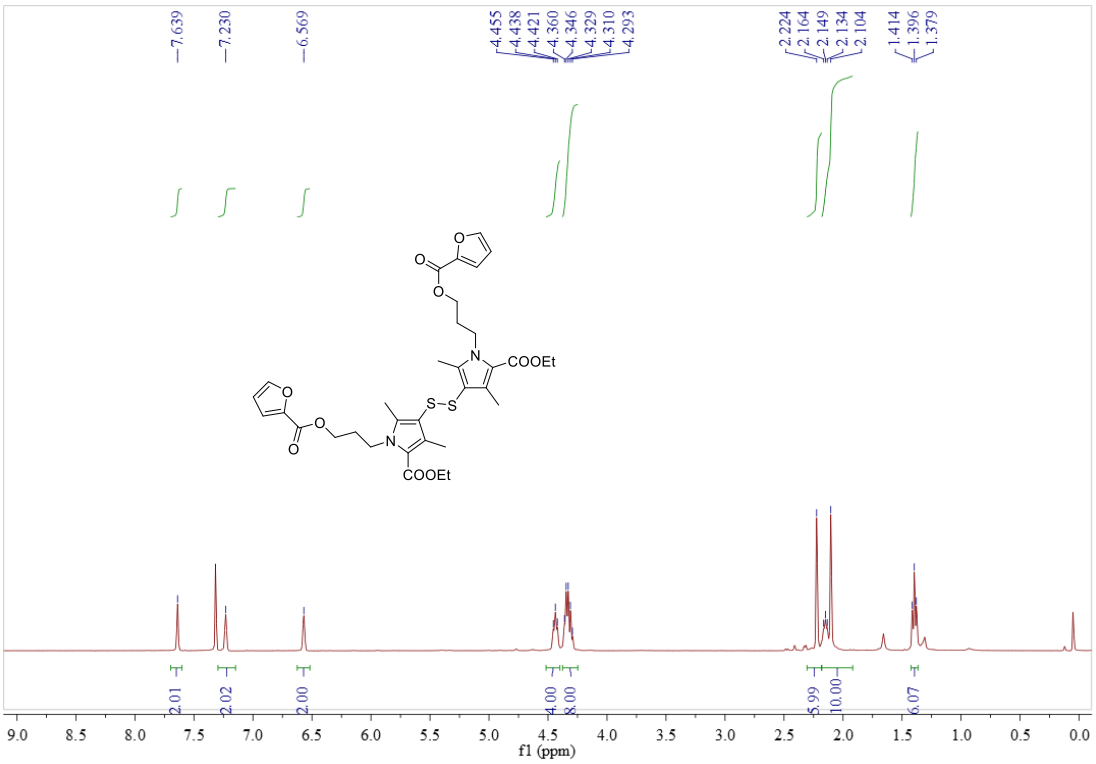
^{13}C NMR (100 MHz, Chloroform-*d*) of **3r**



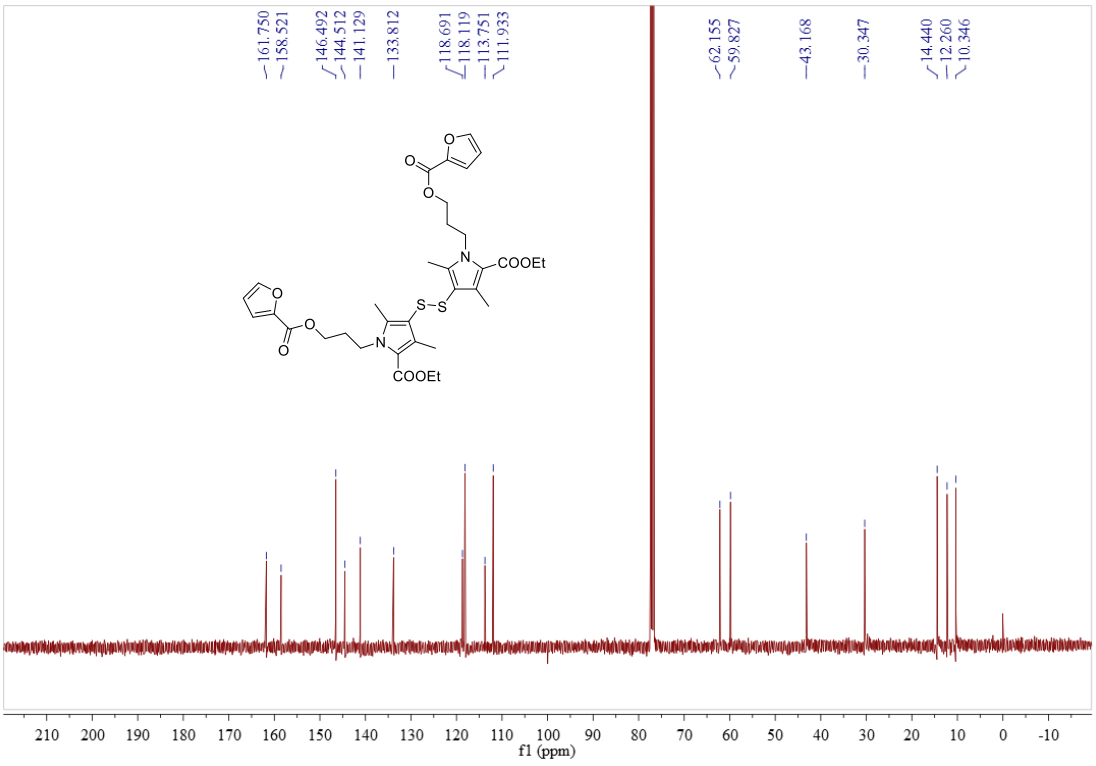
^{1}H NMR (400 MHz, Chloroform-*d*) of **3s**



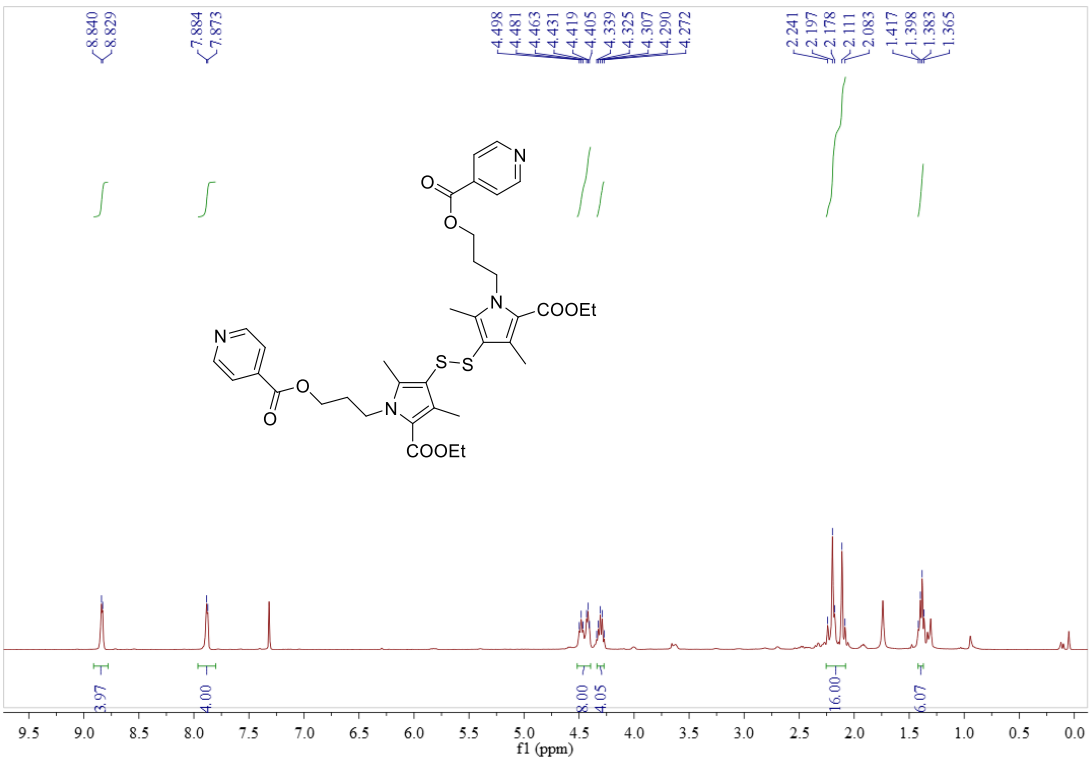
^{13}C NMR (100 MHz, Chloroform-*d*) of **3s**



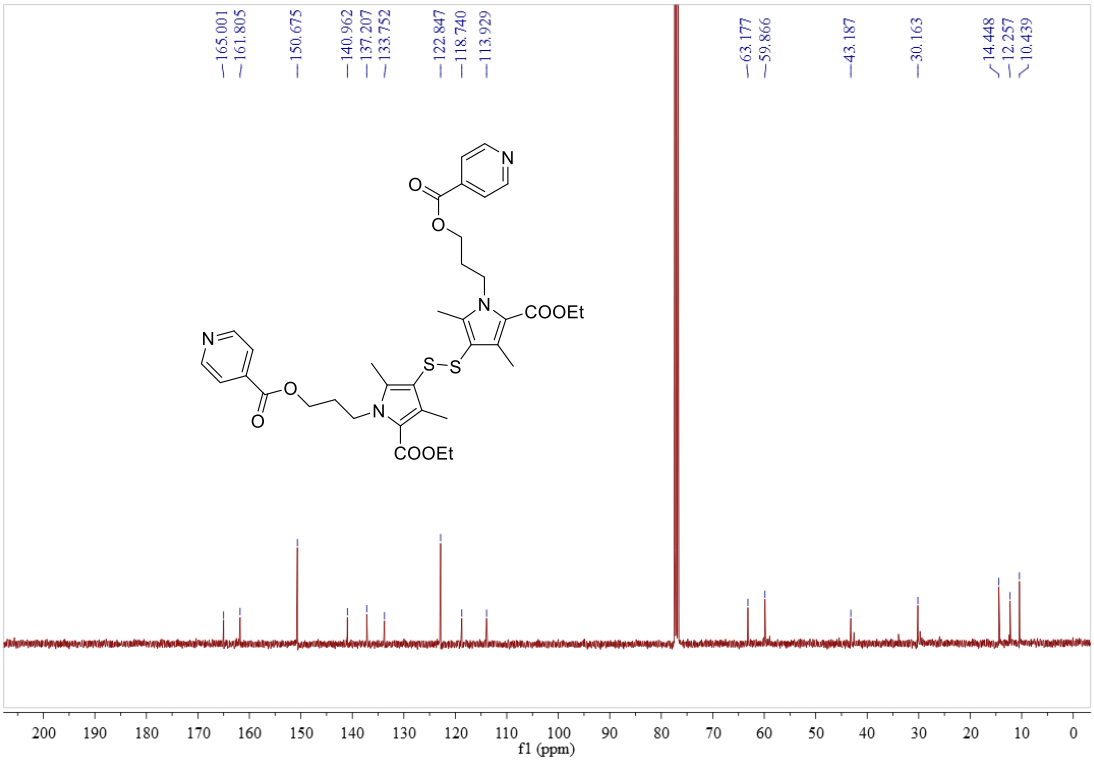
^{1}H NMR (400 MHz, Chloroform-*d*) of **3t**



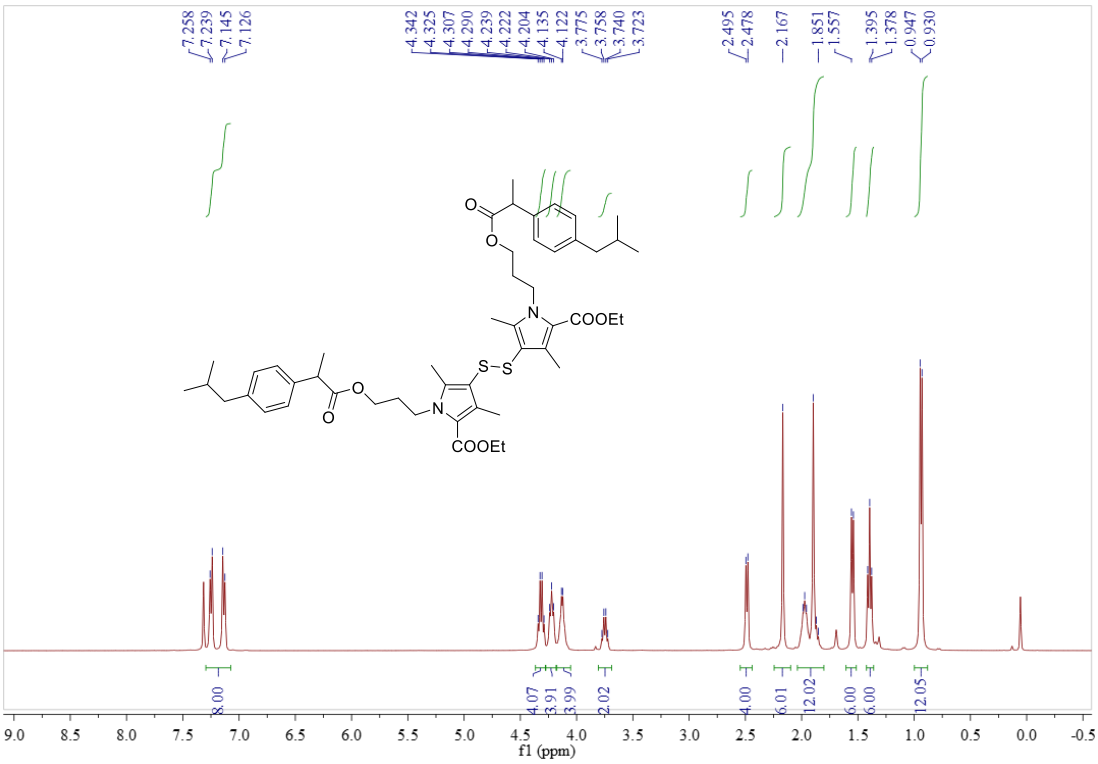
^{13}C NMR (100 MHz, Chloroform-*d*) of **3t**



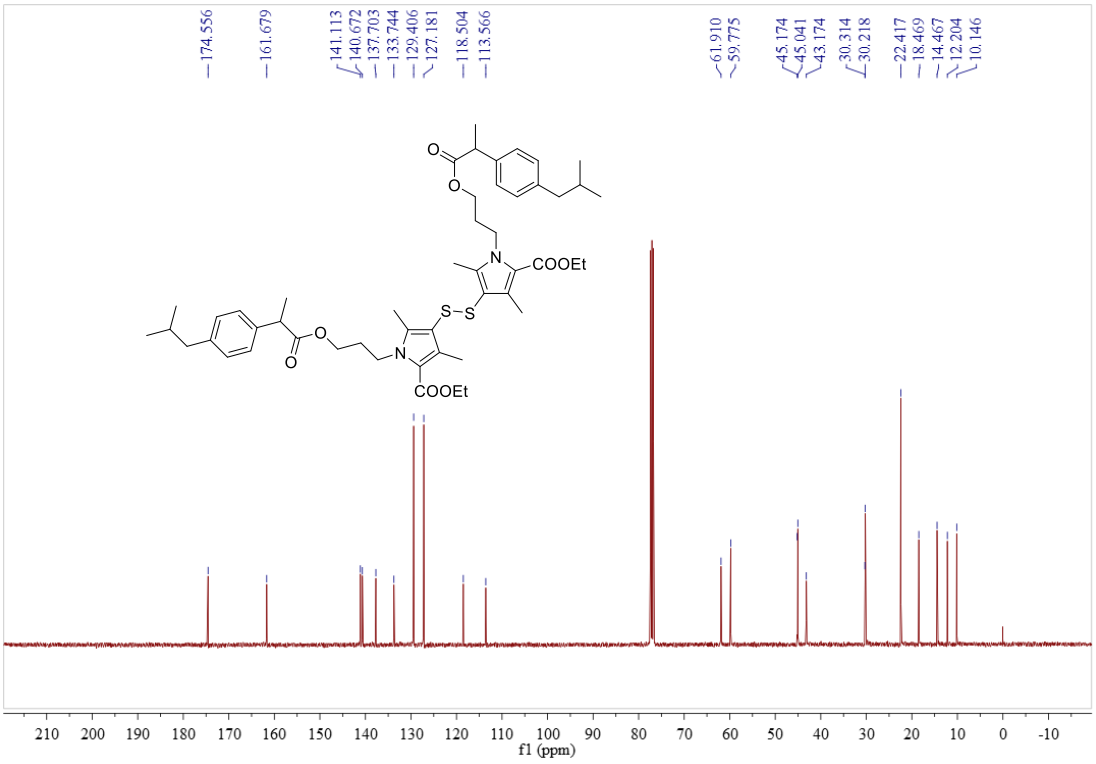
^{1}H NMR (400 MHz, Chloroform-*d*) of **3u**



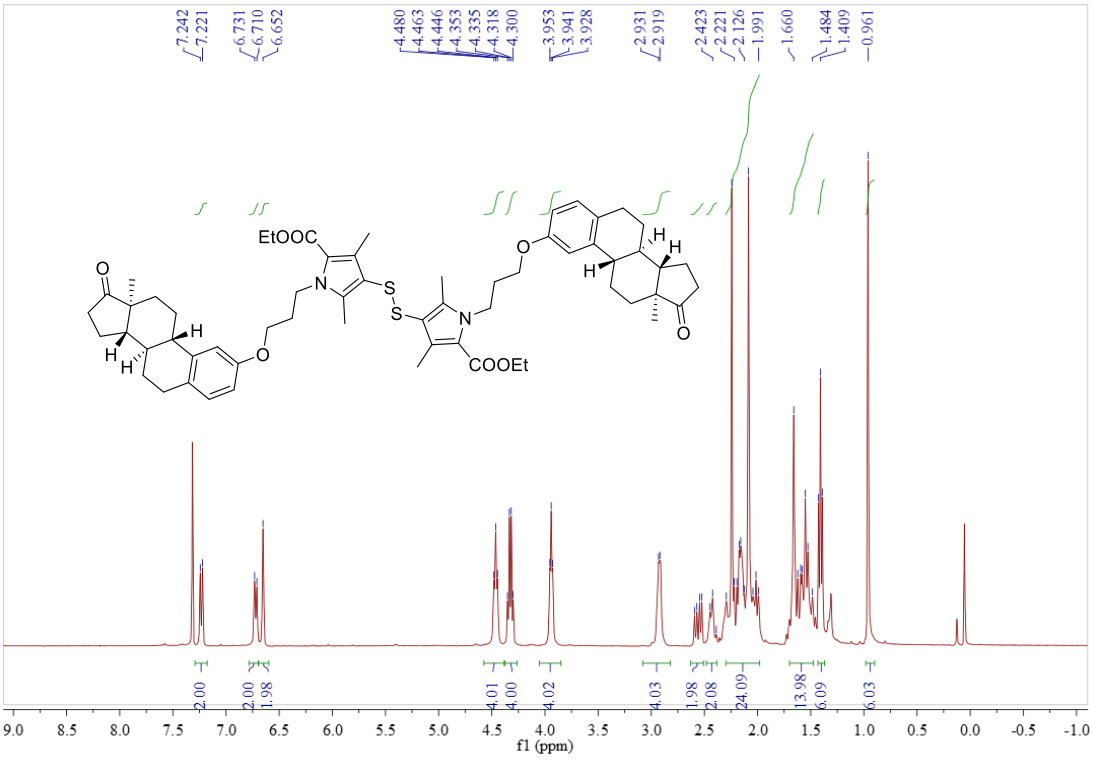
^{13}C NMR (100 MHz, Chloroform-*d*) of **3u**



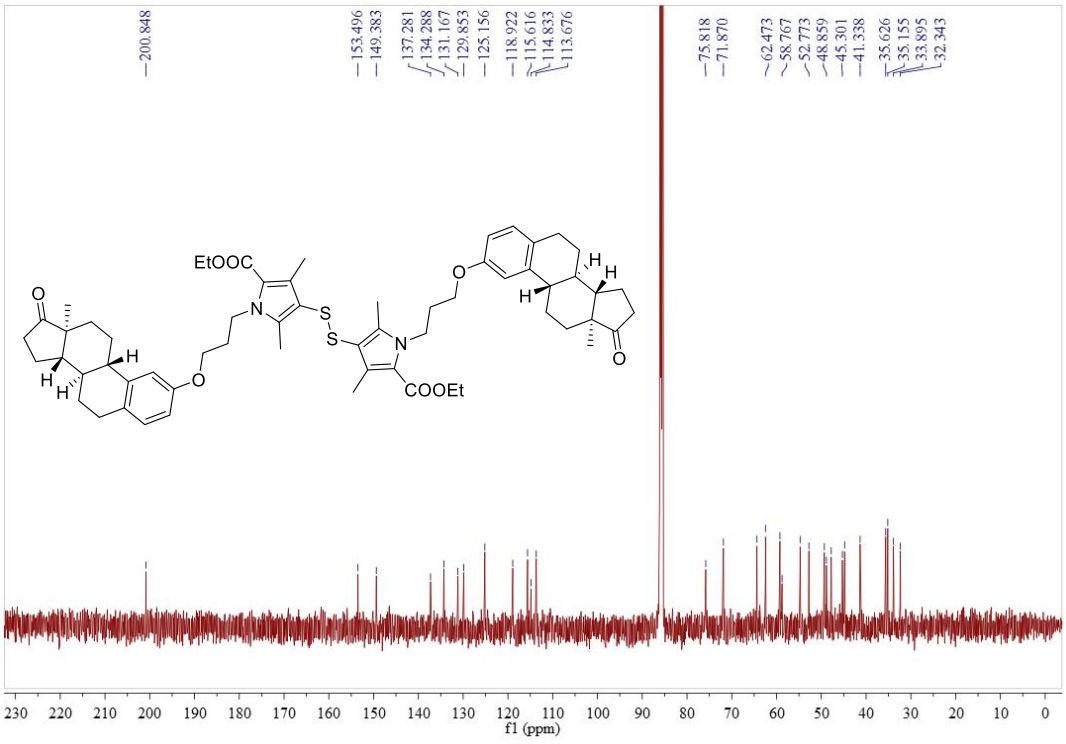
^{1}H NMR (400 MHz, Chloroform-*d*) of 3**v**



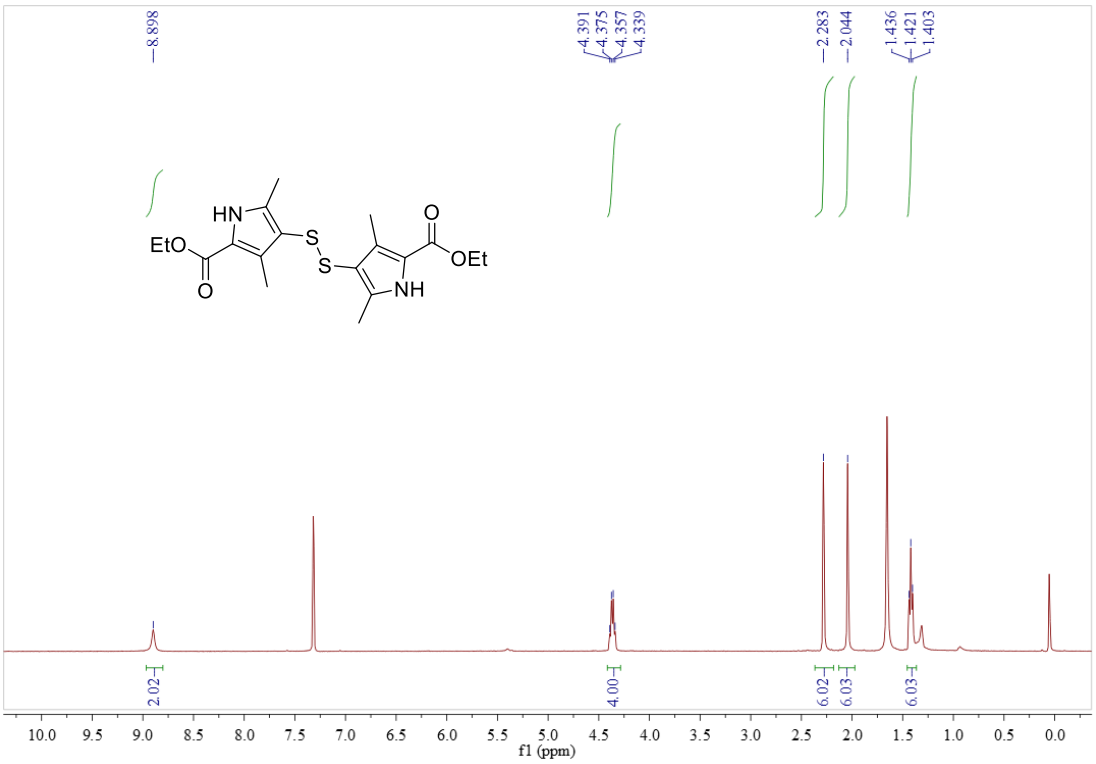
^{13}C NMR (100 MHz, Chloroform-*d*) of **3v**



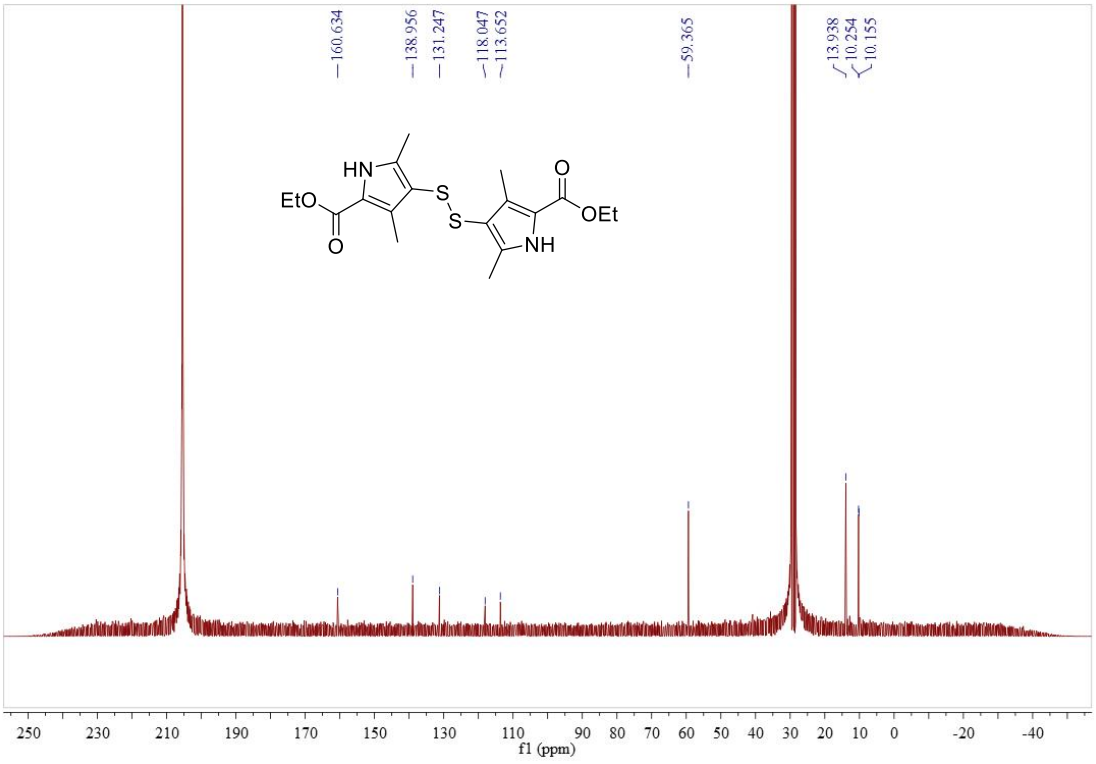
^{1}H NMR (400 MHz, Chloroform-*d*) of **3w**



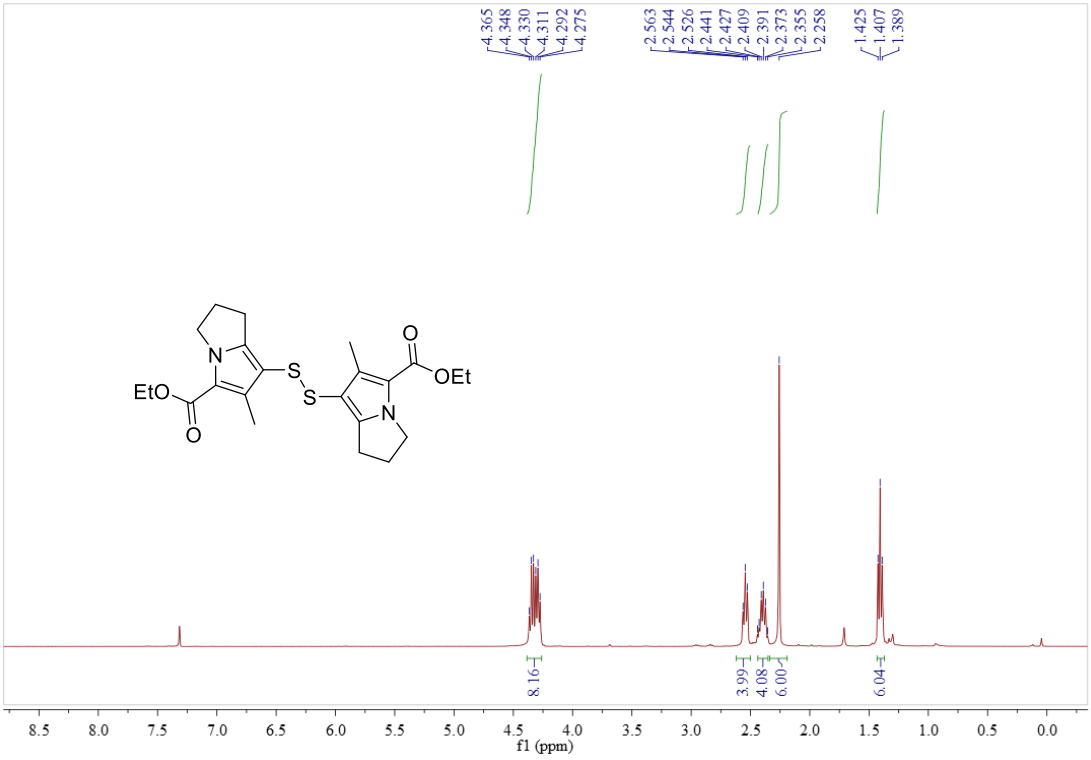
^{13}C NMR (100 MHz, Chloroform-*d*) of **3w**



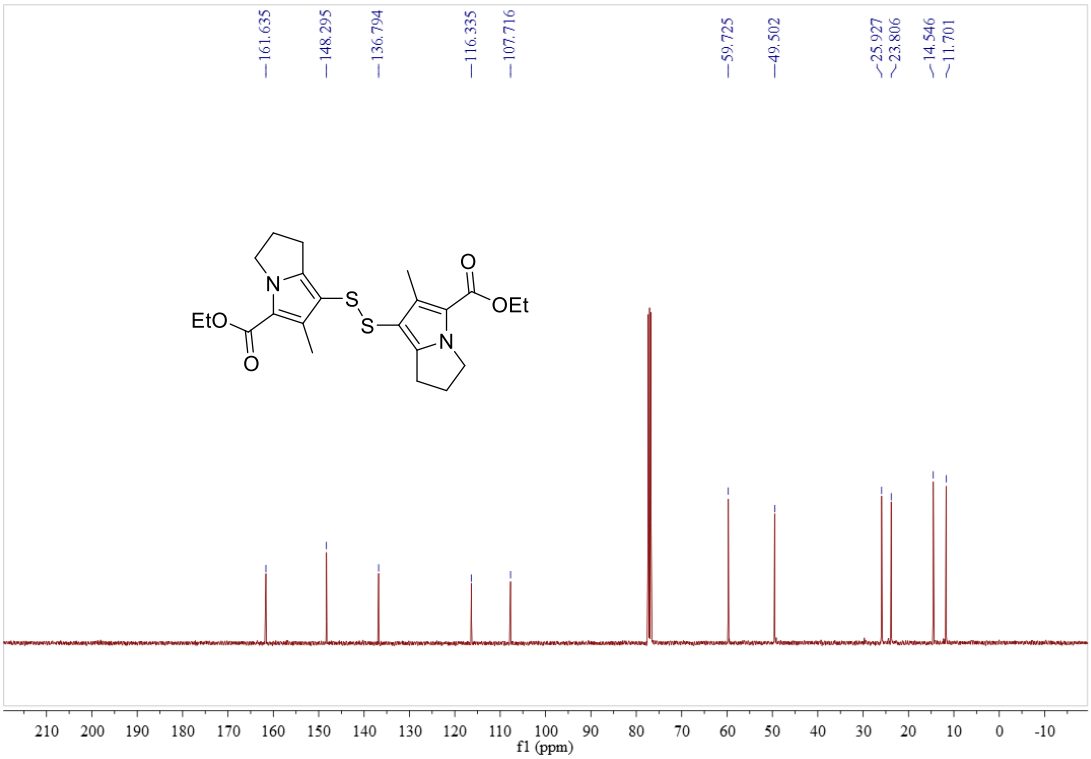
^{1}H NMR (400 MHz, Chloroform-*d*) of **3x**



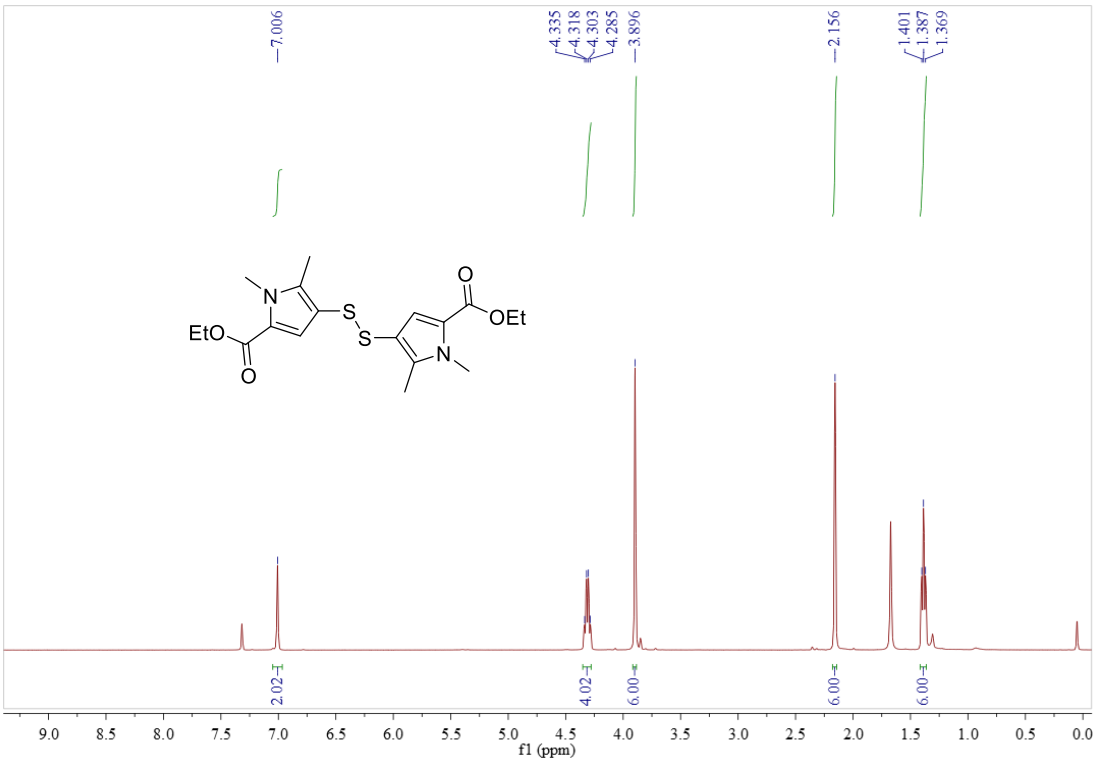
^{13}C NMR (100 MHz, Acetone-d_6) of **3x**



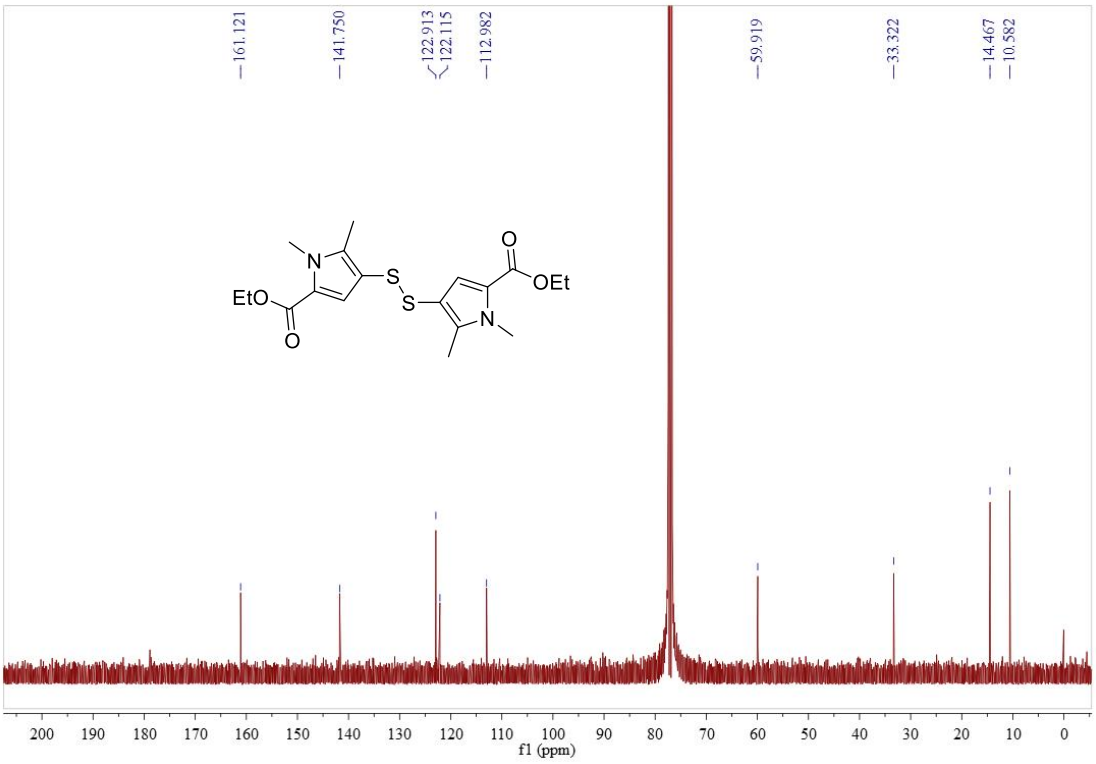
^{1}H NMR (400 MHz, Chloroform-*d*) of **3y**



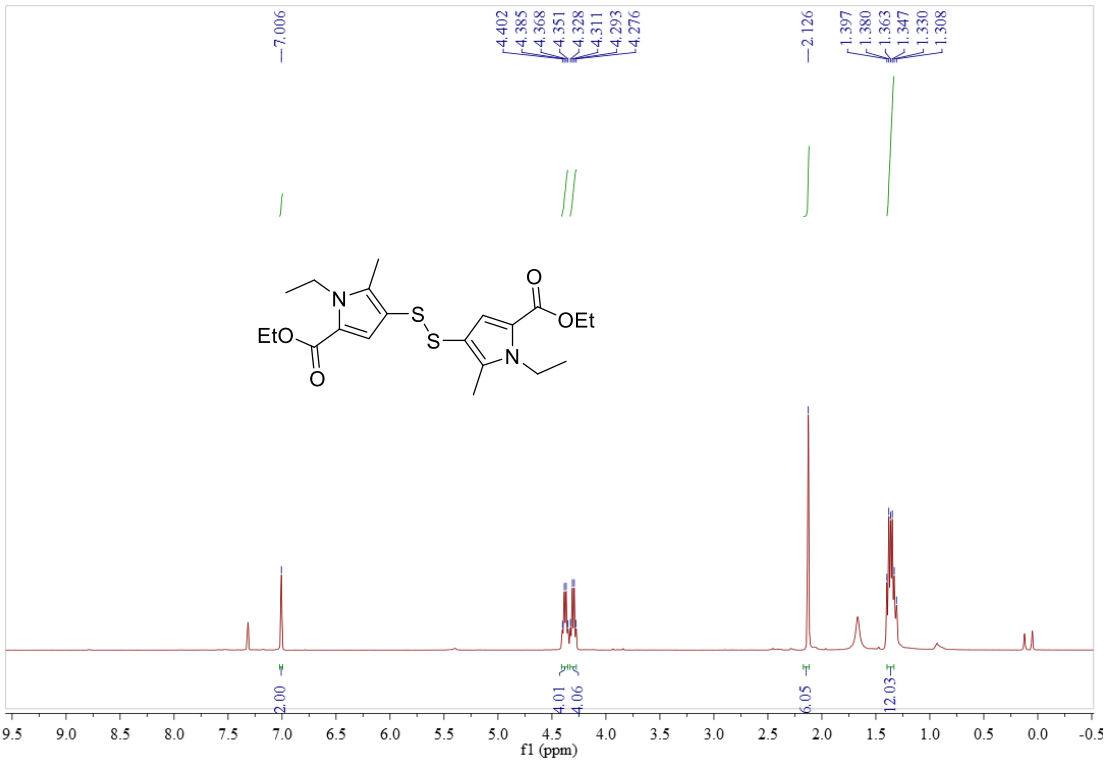
^{13}C NMR (100 MHz, Chloroform-*d*) of **3y**



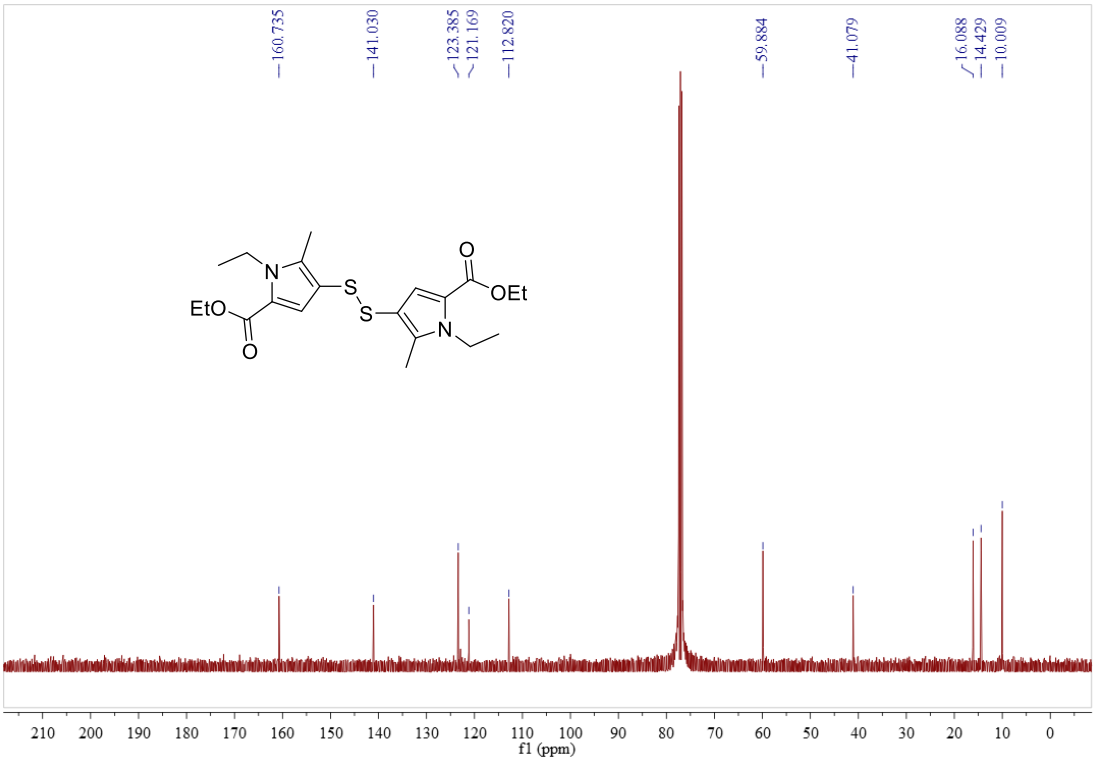
^{1}H NMR (400 MHz, Chloroform-*d*) of **3z**



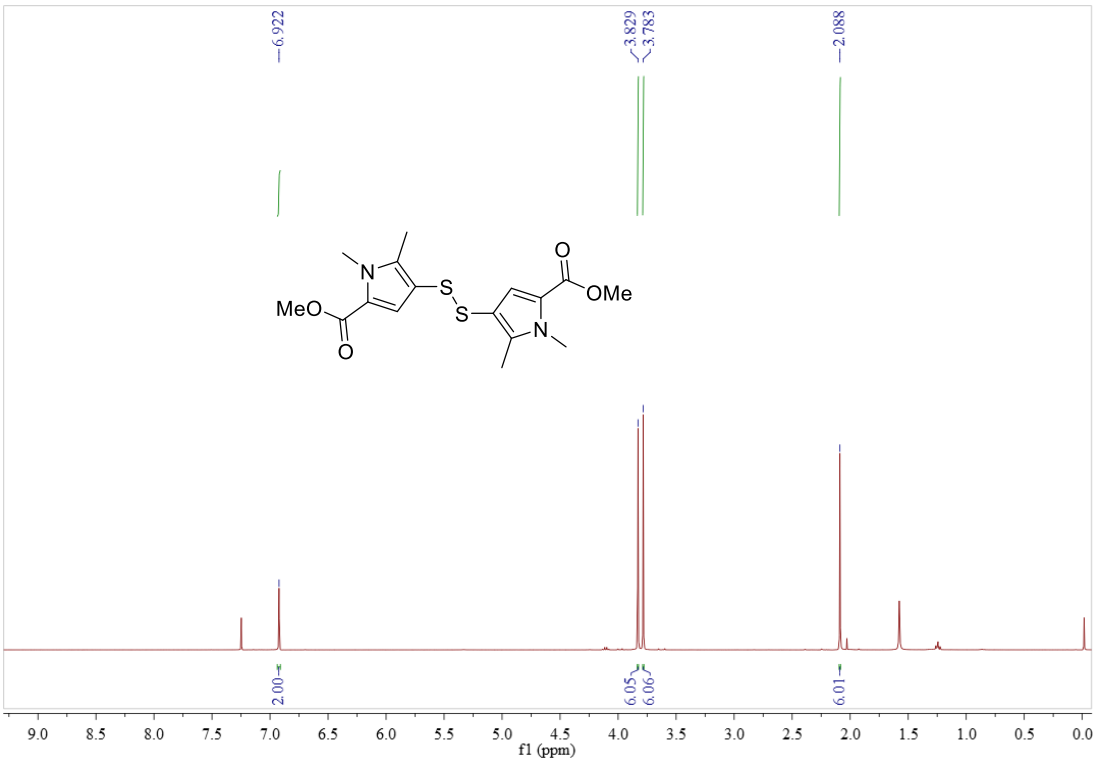
^{13}C NMR (100 MHz, Chloroform-*d*) of **3z**



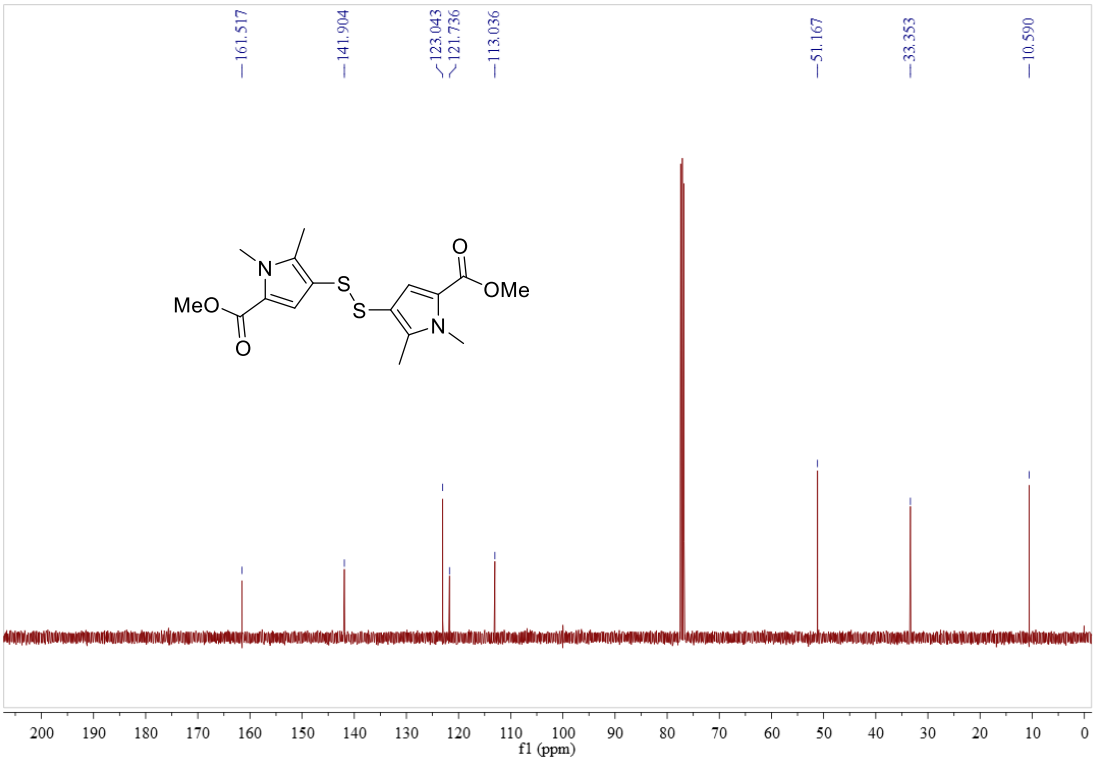
^{1}H NMR (400 MHz, Chloroform-*d*) of **3aa**



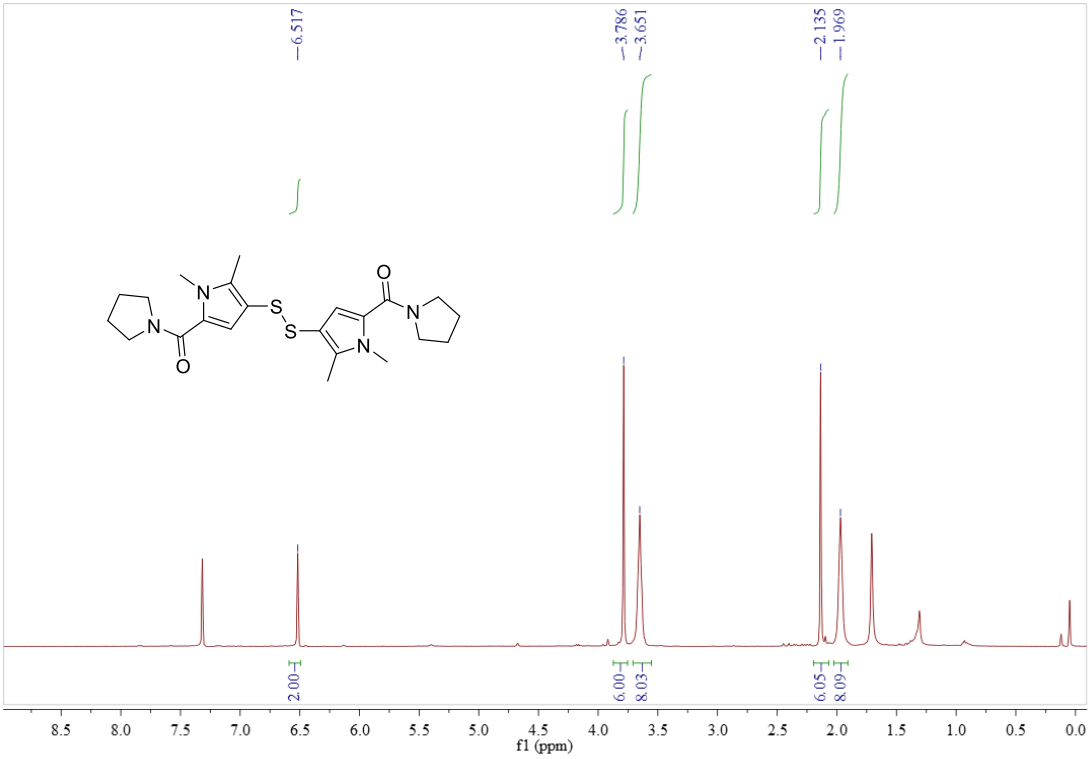
^{13}C NMR (100 MHz, Chloroform-*d*) of **3aa**



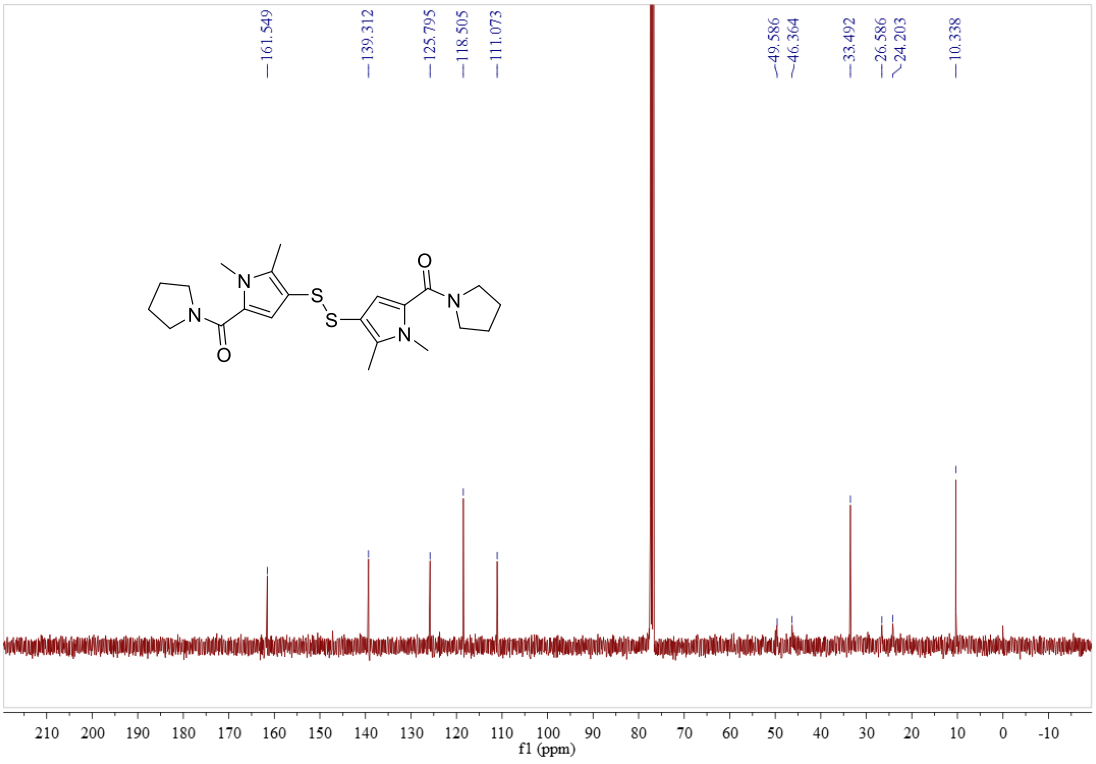
^{1}H NMR (400 MHz, Chloroform-*d*) of **3ab**



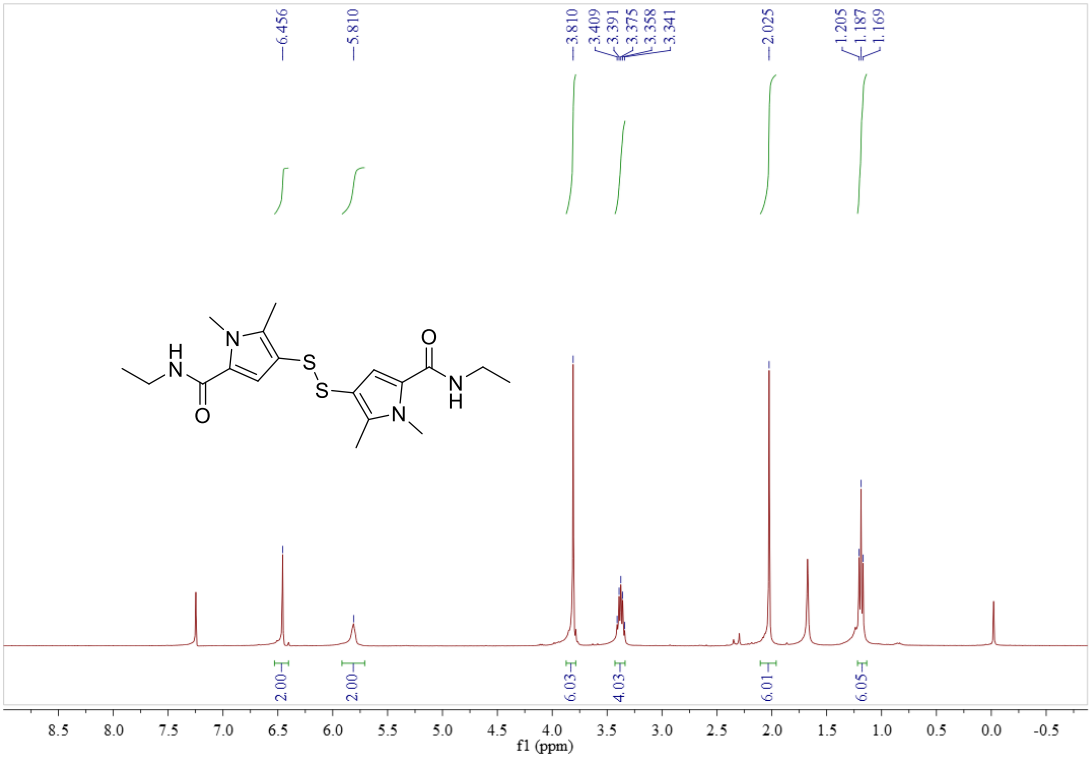
^{13}C NMR (100 MHz, Chloroform-*d*) of **3ab**



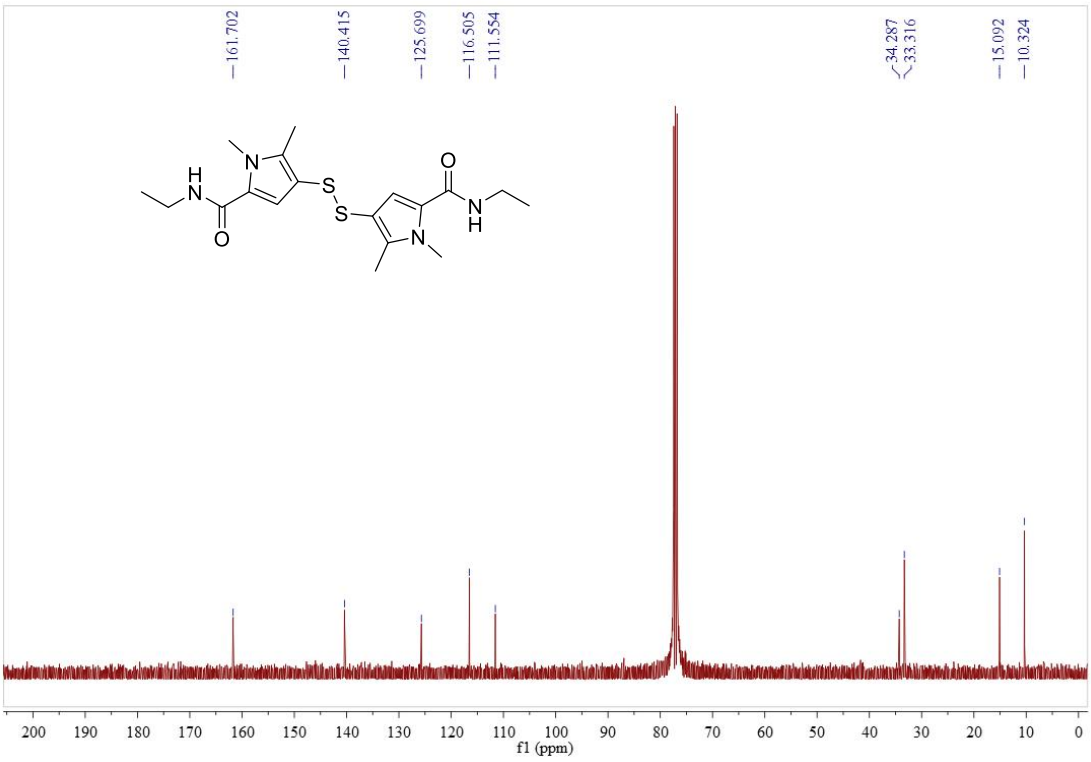
^{1}H NMR (400 MHz, Chloroform-*d*) of **3ac**



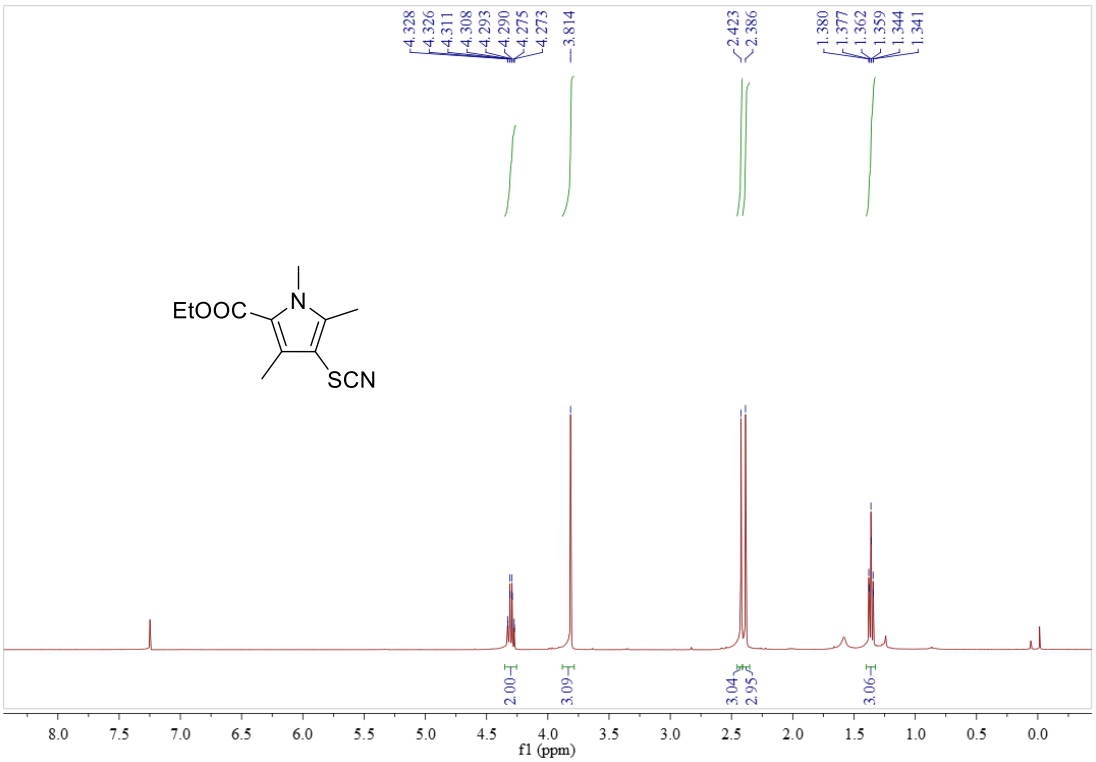
^{13}C NMR (100 MHz, Chloroform-*d*) of **3ac**



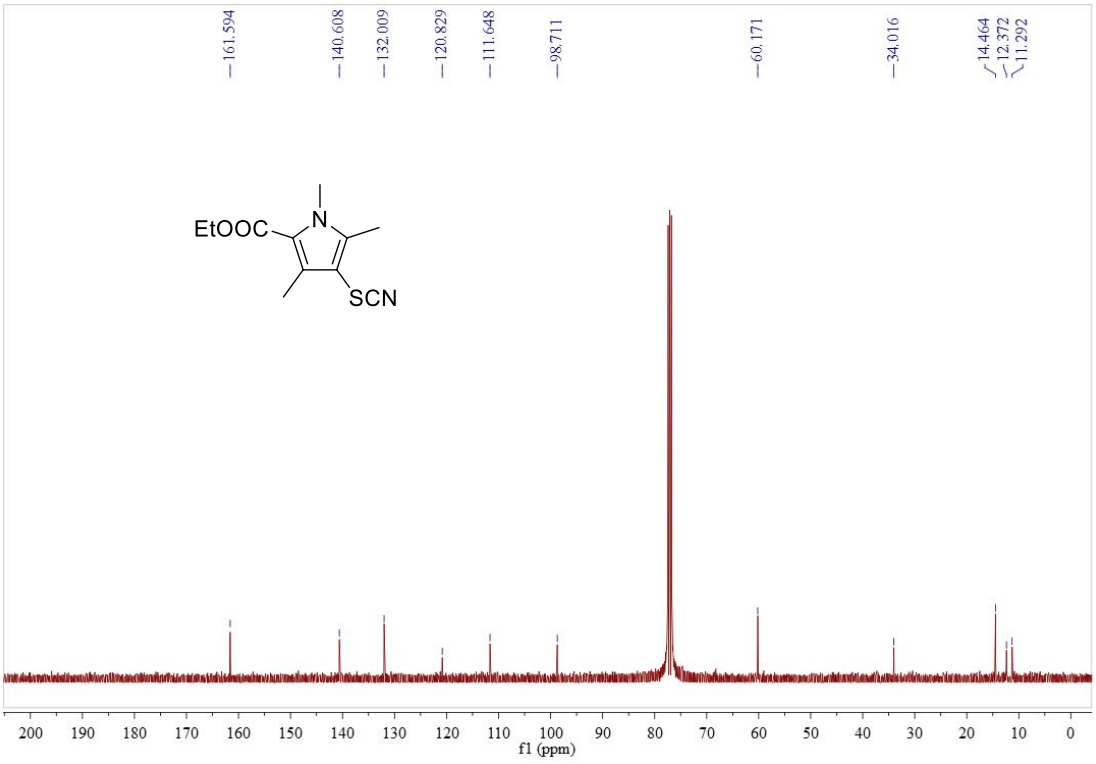
^{1}H NMR (400 MHz, Chloroform-*d*) of **3ad**



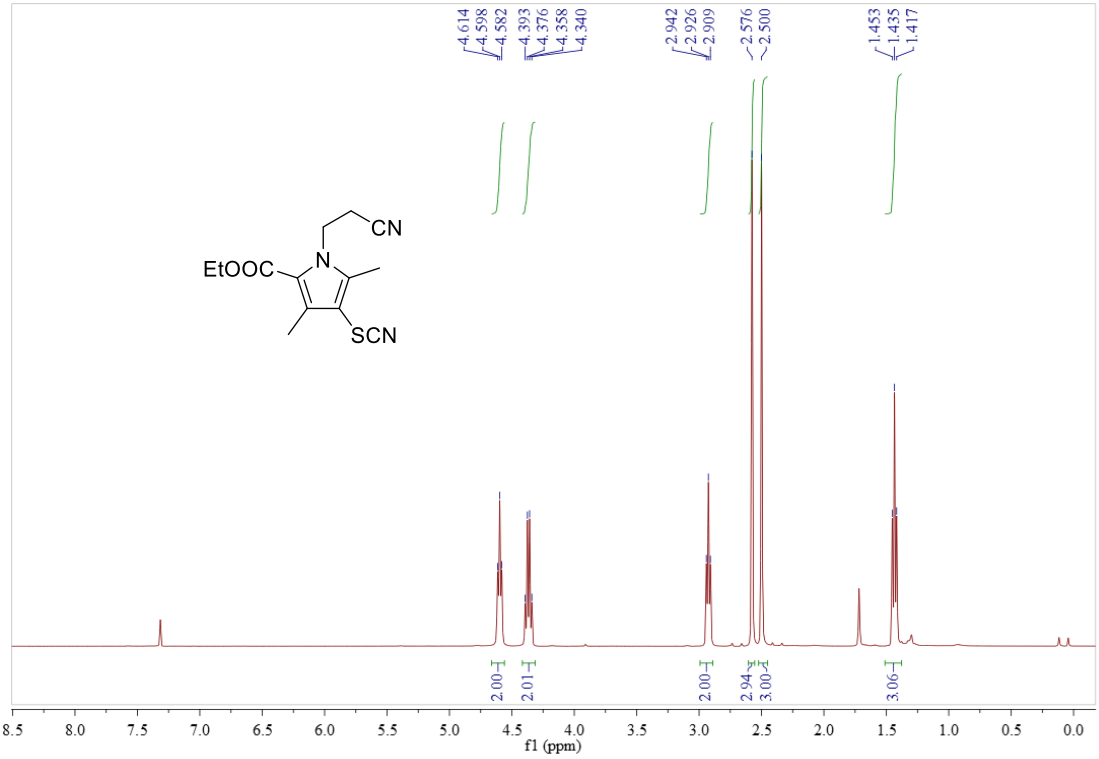
^{13}C NMR (100 MHz, Chloroform-*d*) of **3ad**



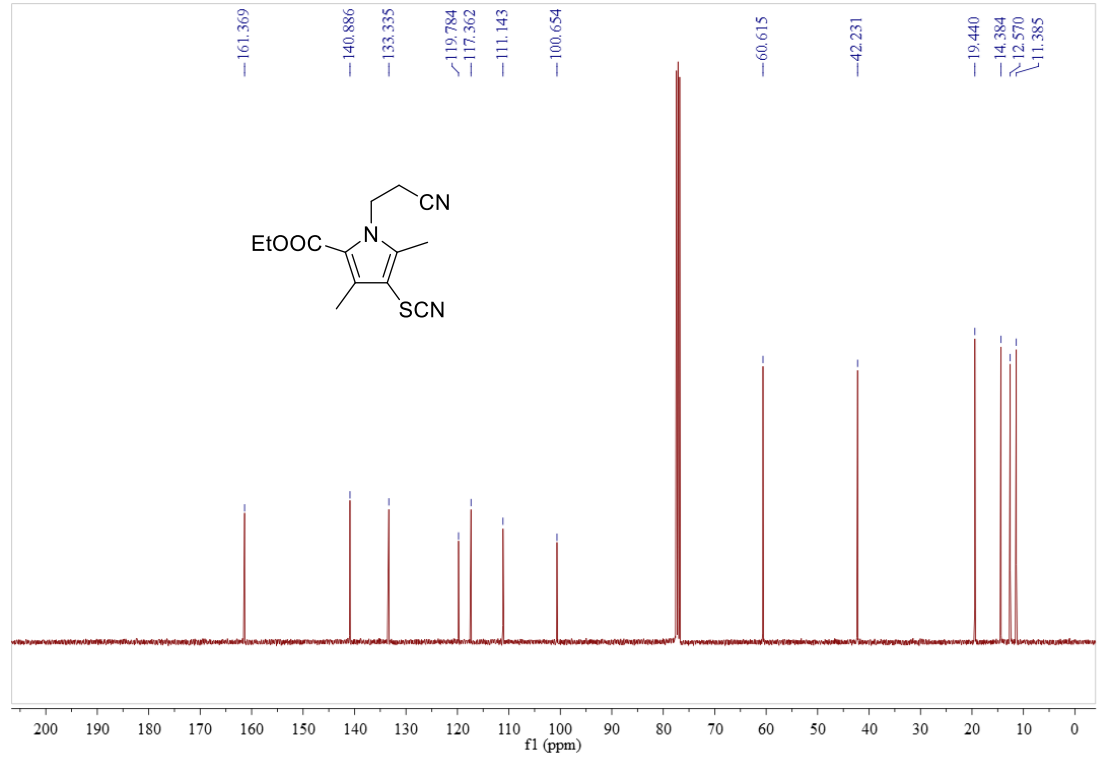
^{1}H NMR (400 MHz, Chloroform-*d*) of **4a**



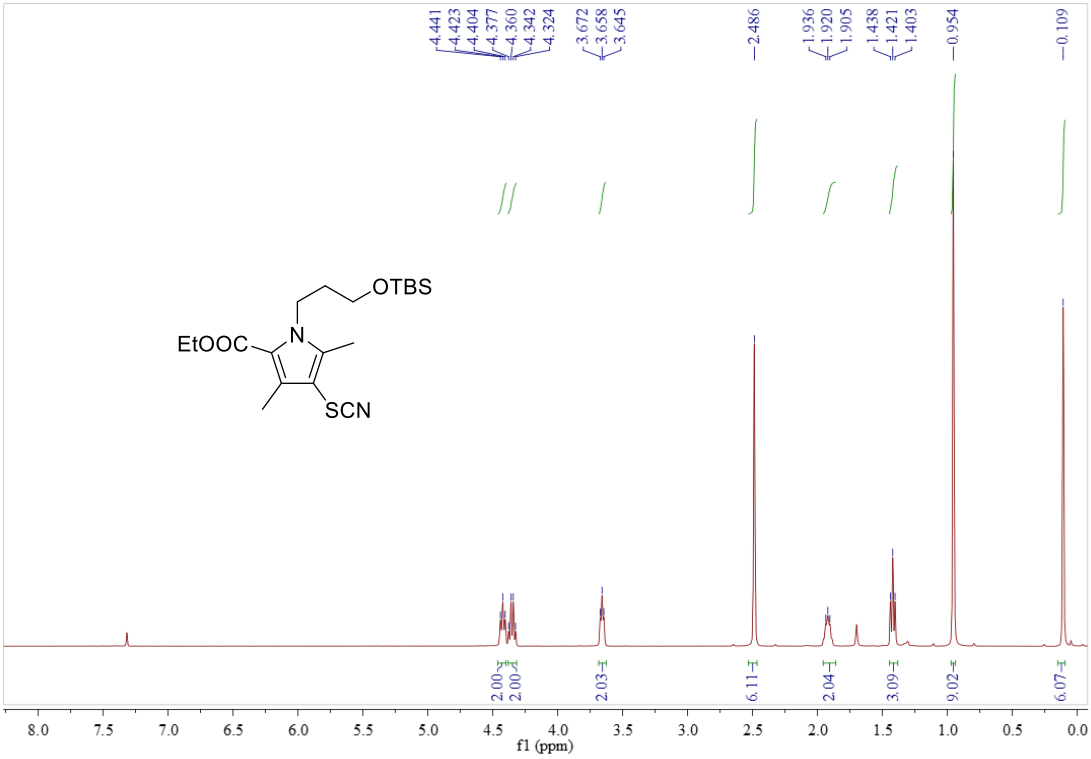
^{13}C NMR (100 MHz, Chloroform-*d*) of **4a**



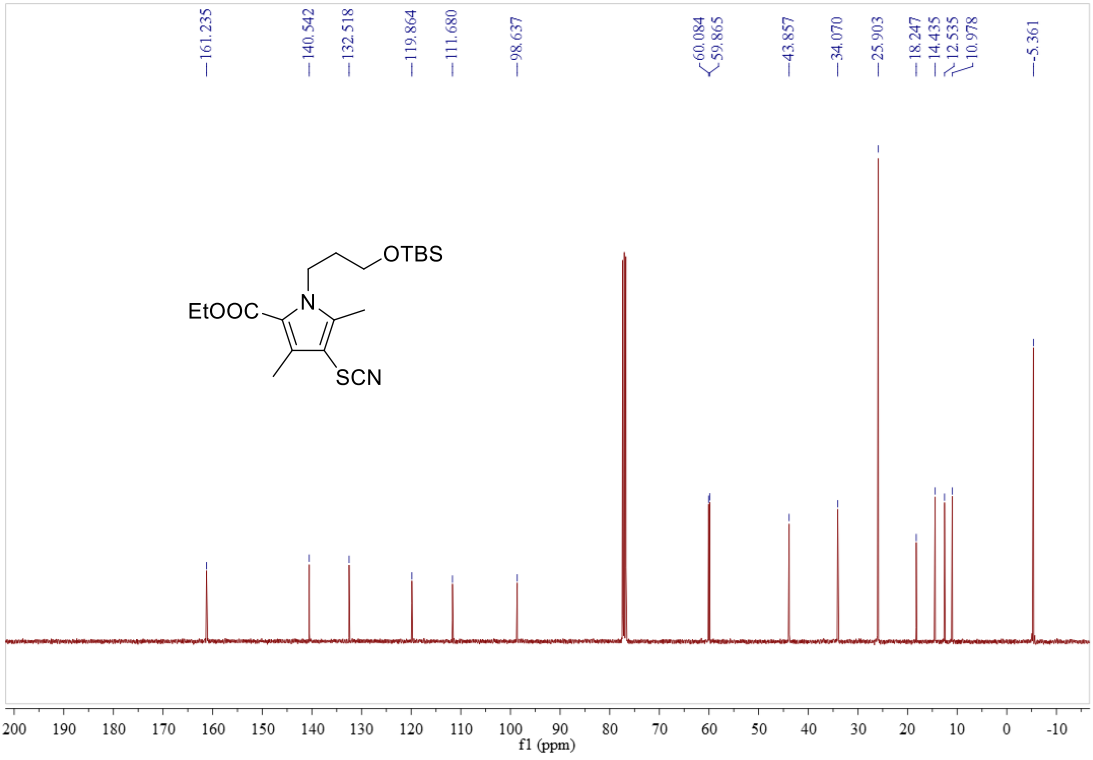
^{1}H NMR (400 MHz, Chloroform-*d*) of **4b**



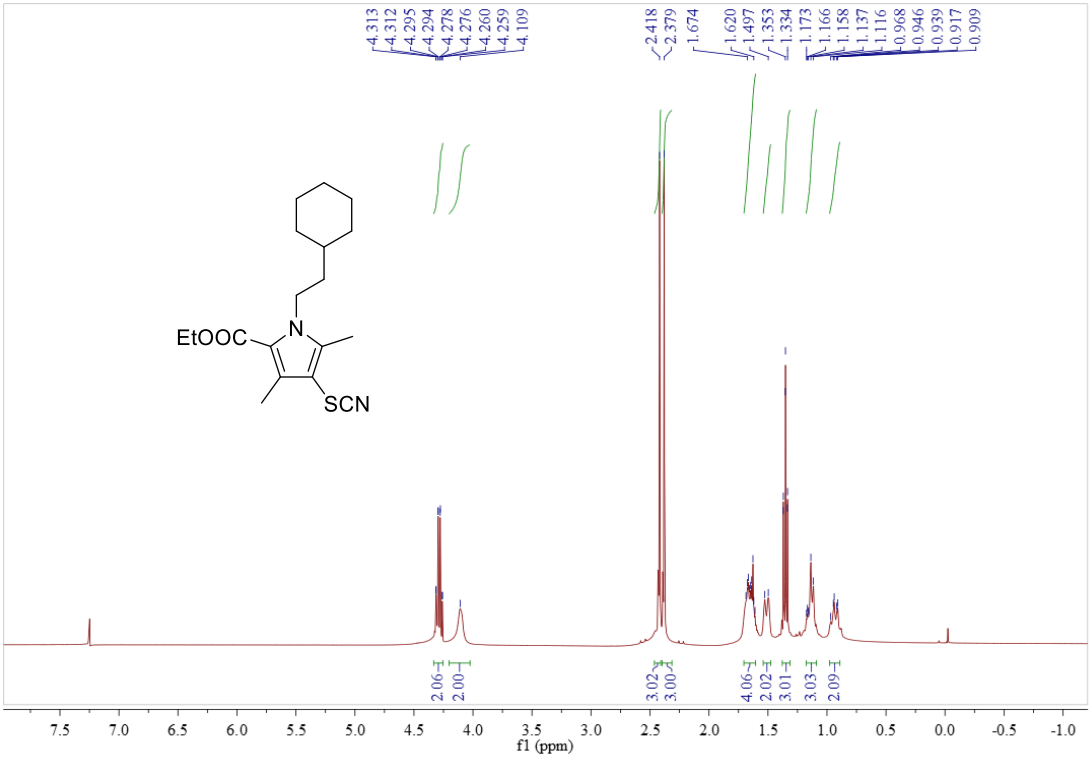
^{13}C NMR (100 MHz, Chloroform-*d*) of **4b**



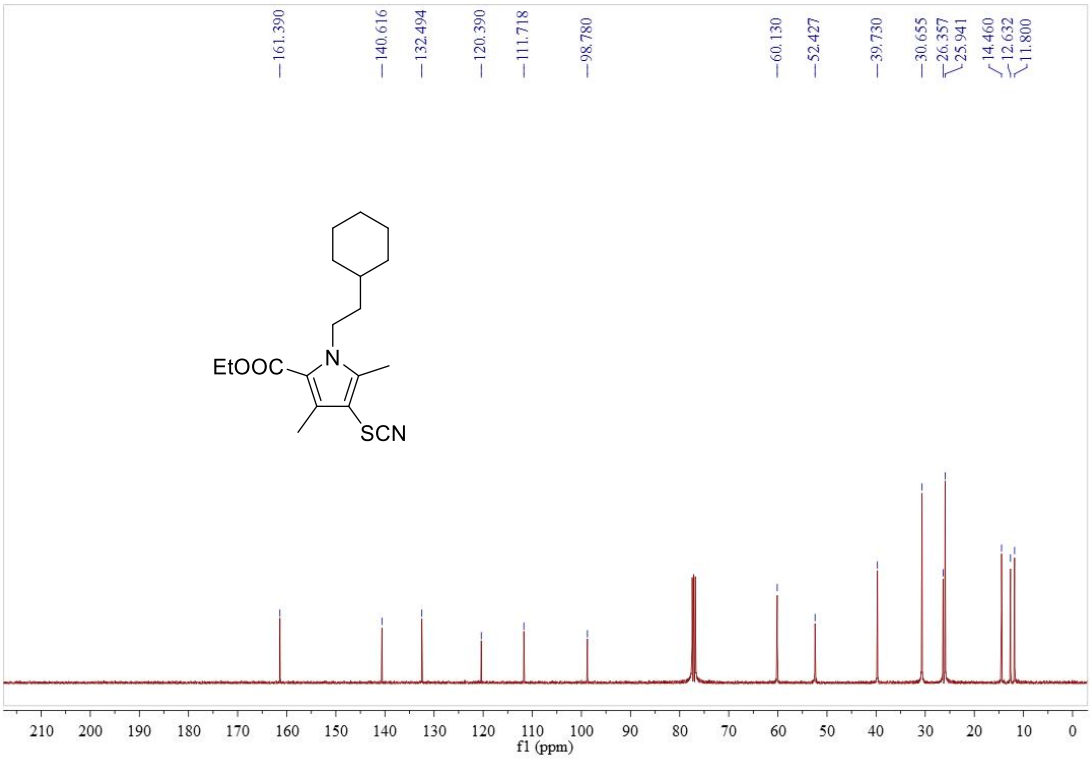
^{1}H NMR (400 MHz, Chloroform-*d*) of **4c**



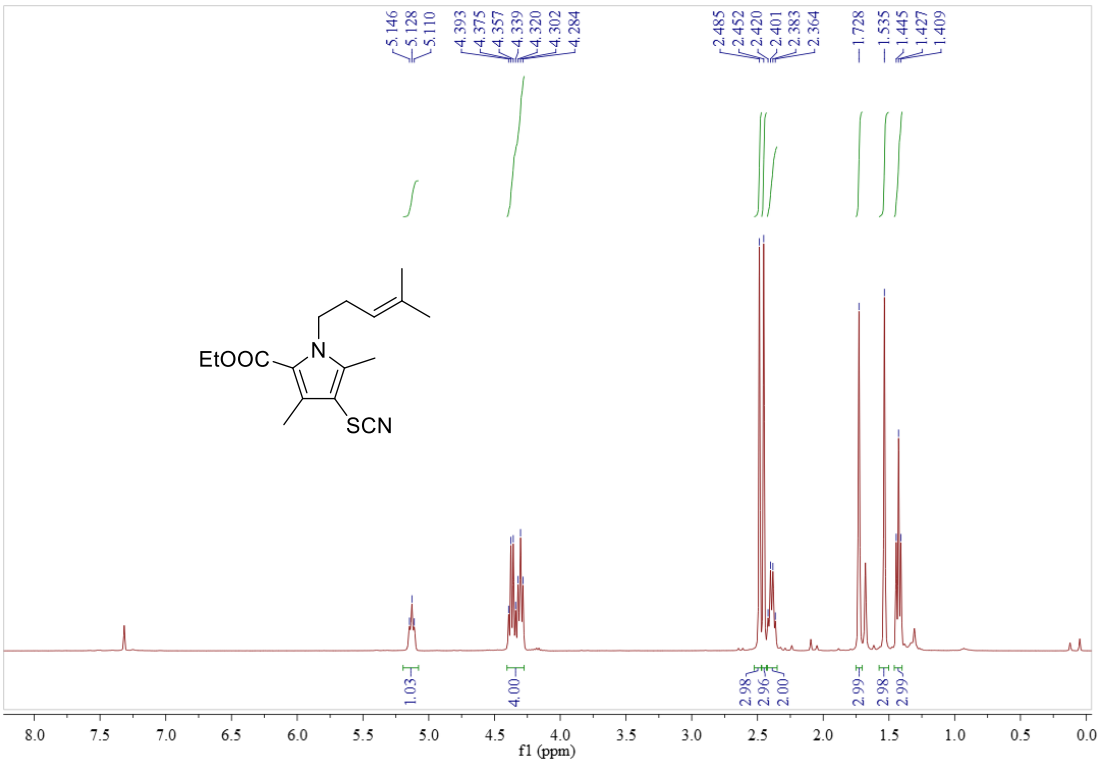
^{13}C NMR (100 MHz, Chloroform-*d*) of **4c**



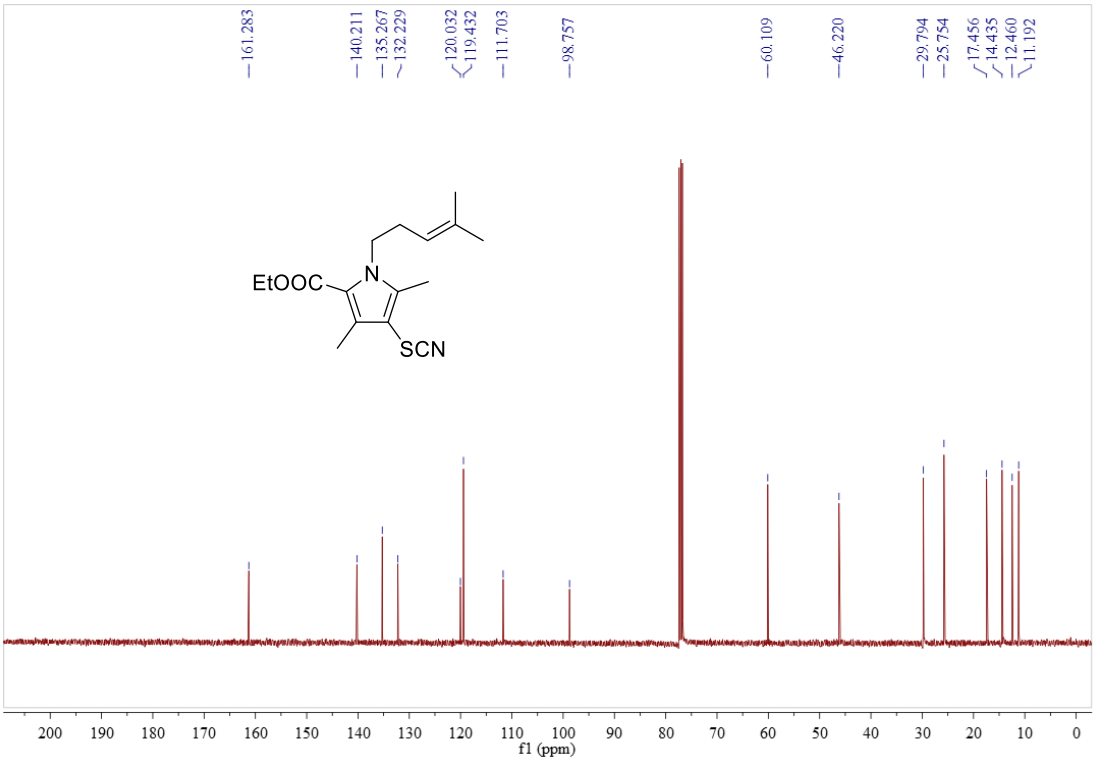
^{1}H NMR (400 MHz, Chloroform-*d*) of **4d**



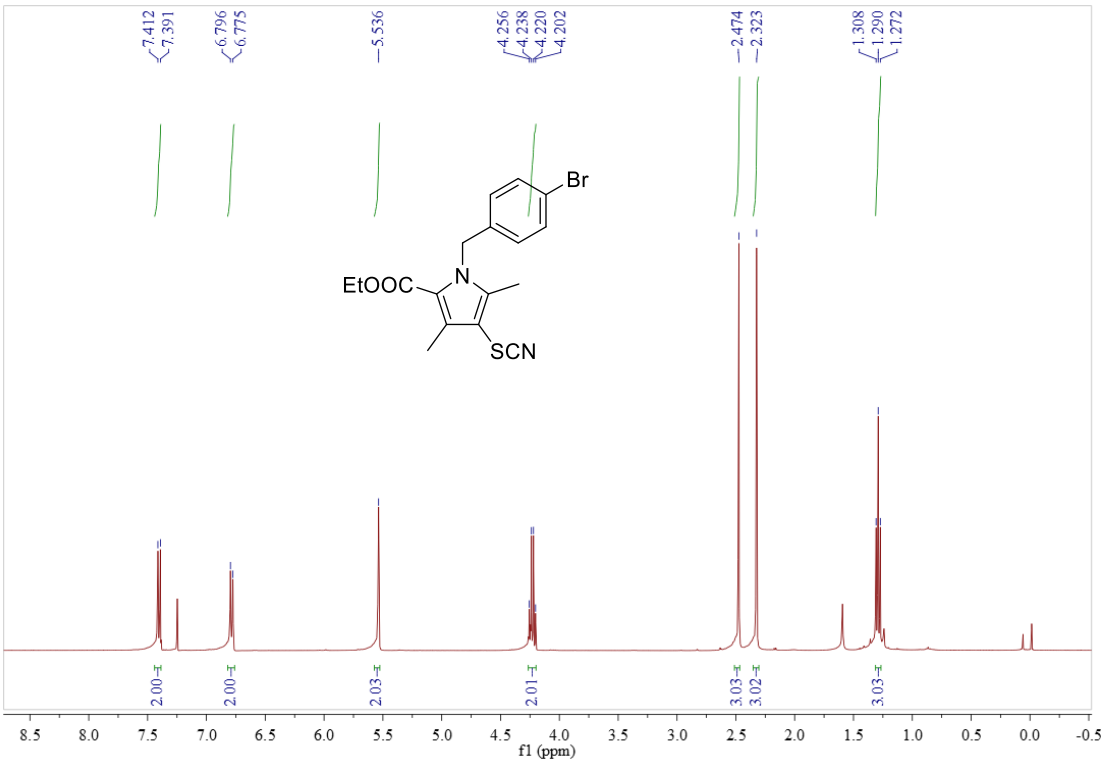
^{13}C NMR (100 MHz, Chloroform-*d*) of **4d**



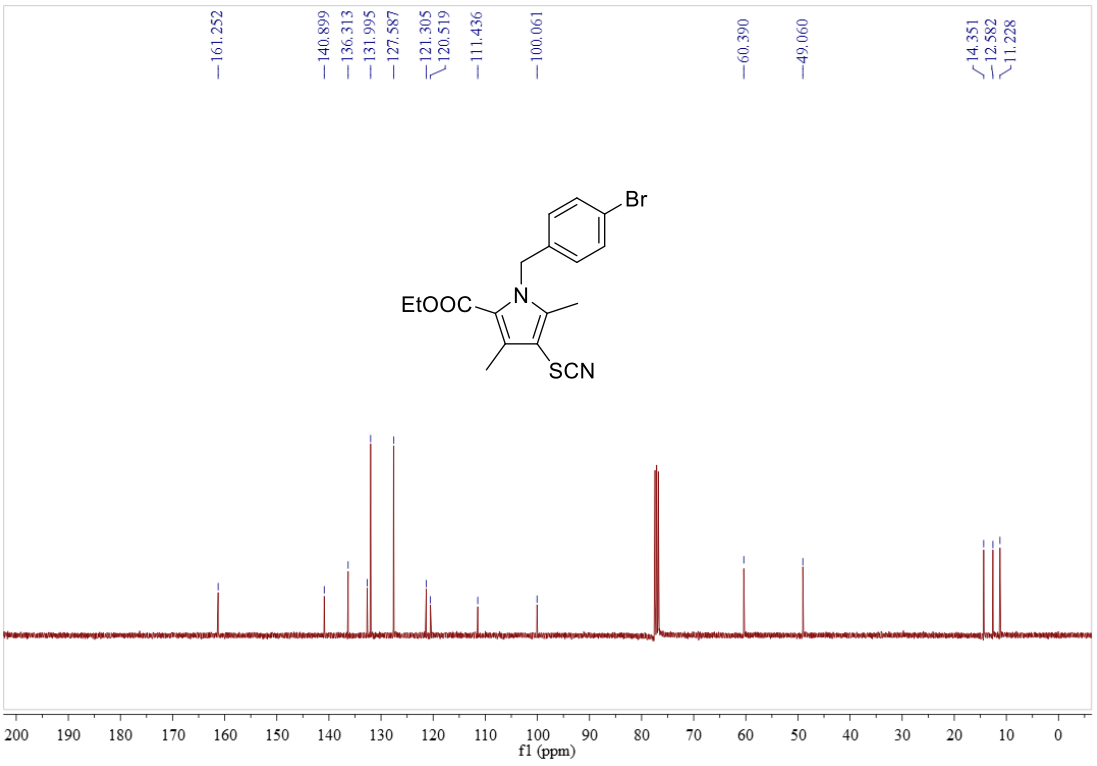
^{1}H NMR (400 MHz, Chloroform-*d*) of **4e**



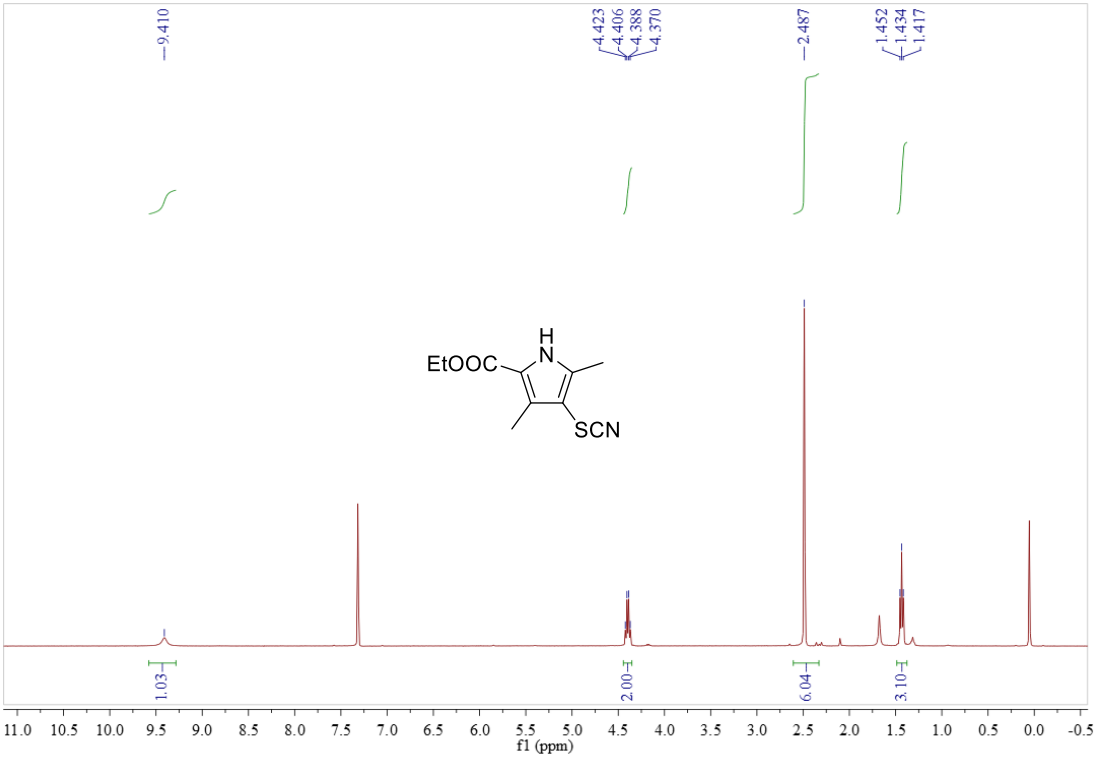
^{13}C NMR (100 MHz, Chloroform-*d*) of **4e**



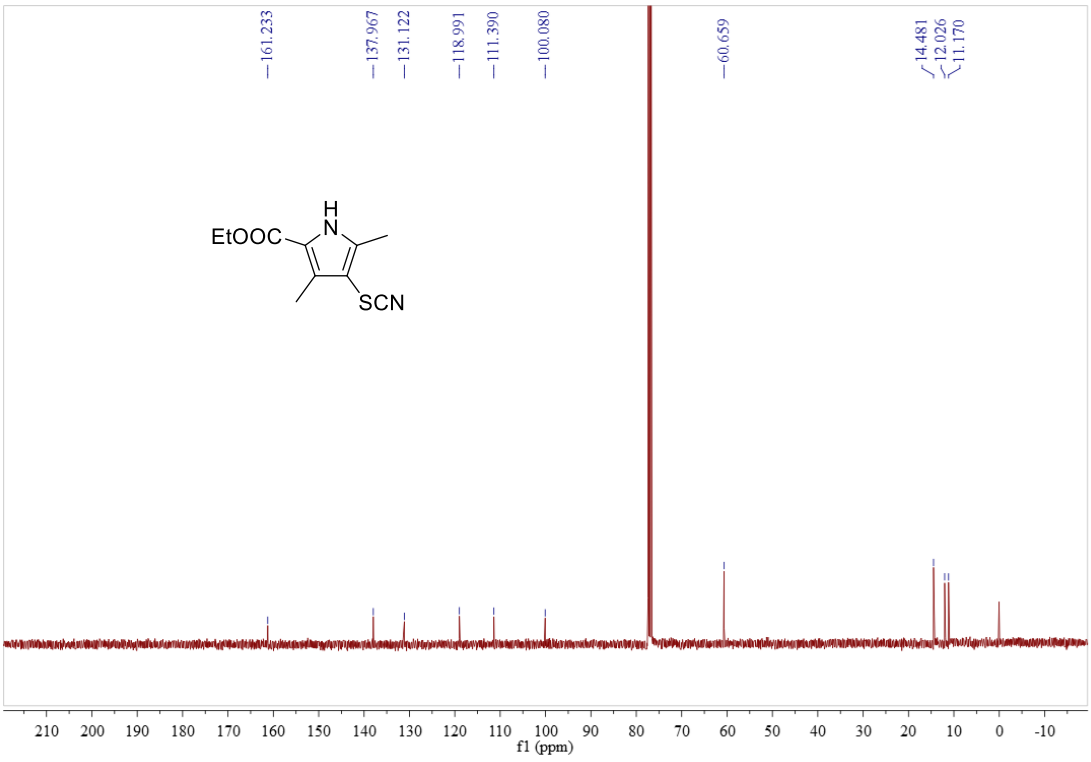
^{1}H NMR (400 MHz, Chloroform-*d*) of **4f**



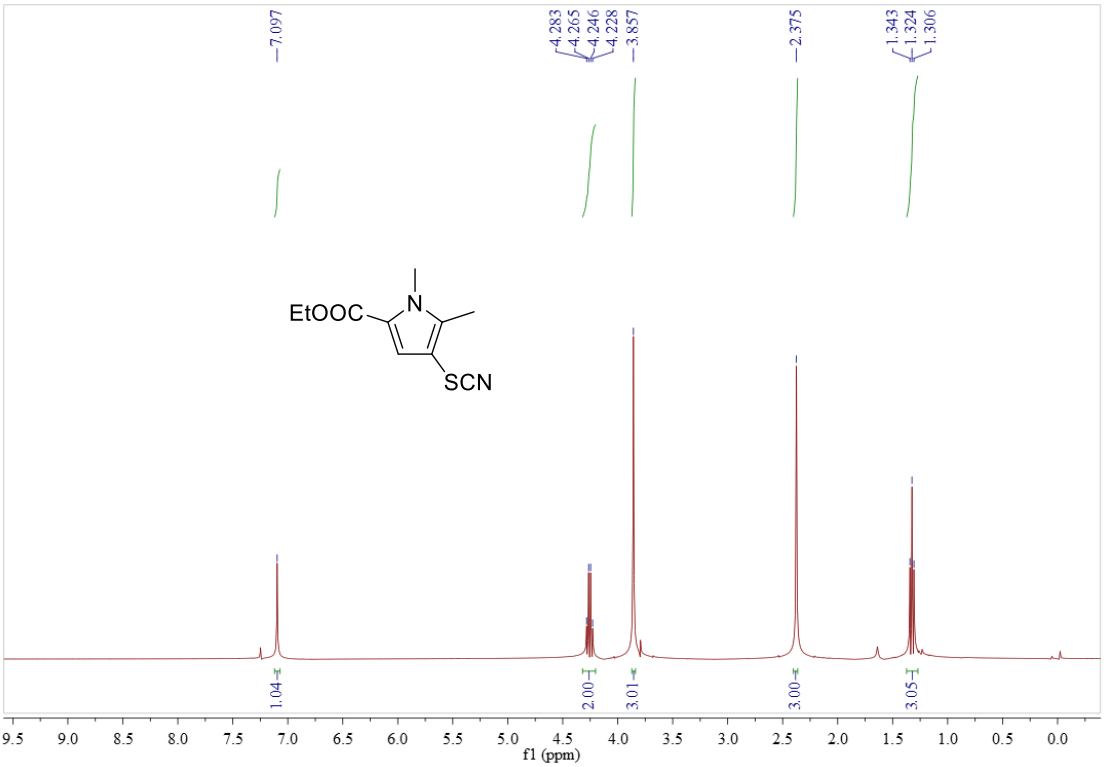
^{13}C NMR (100 MHz, Chloroform-*d*) of **4f**



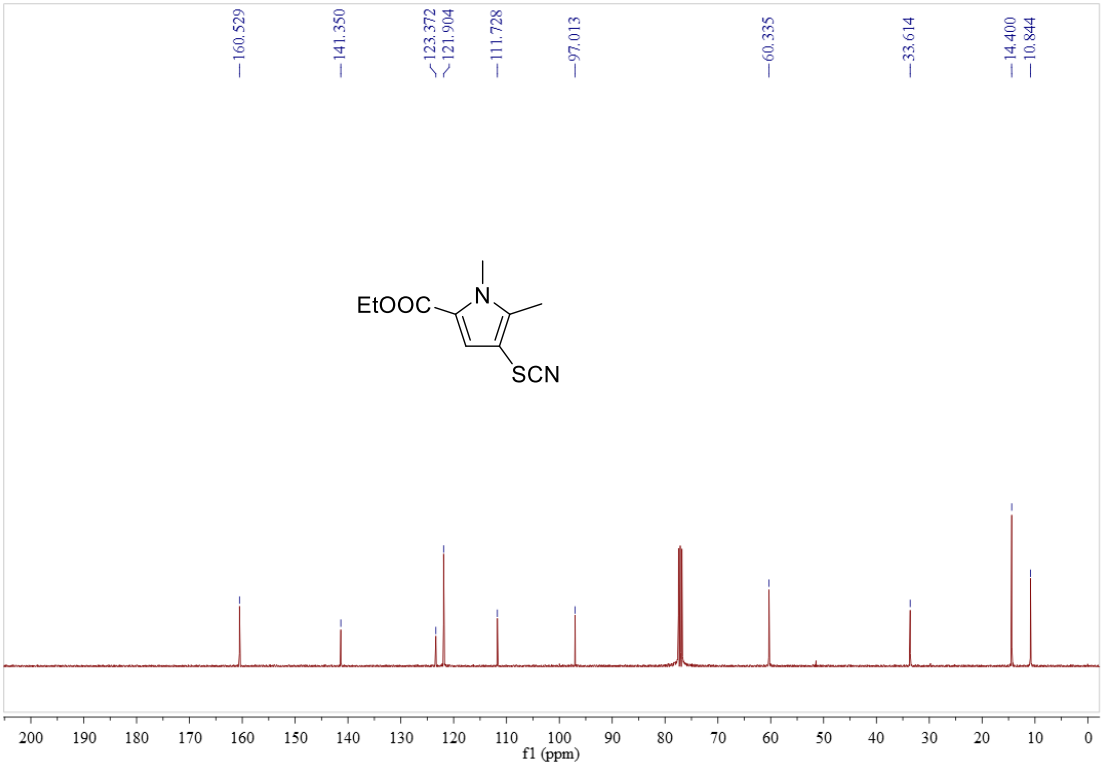
^{1}H NMR (400 MHz, Chloroform-*d*) of **4g**



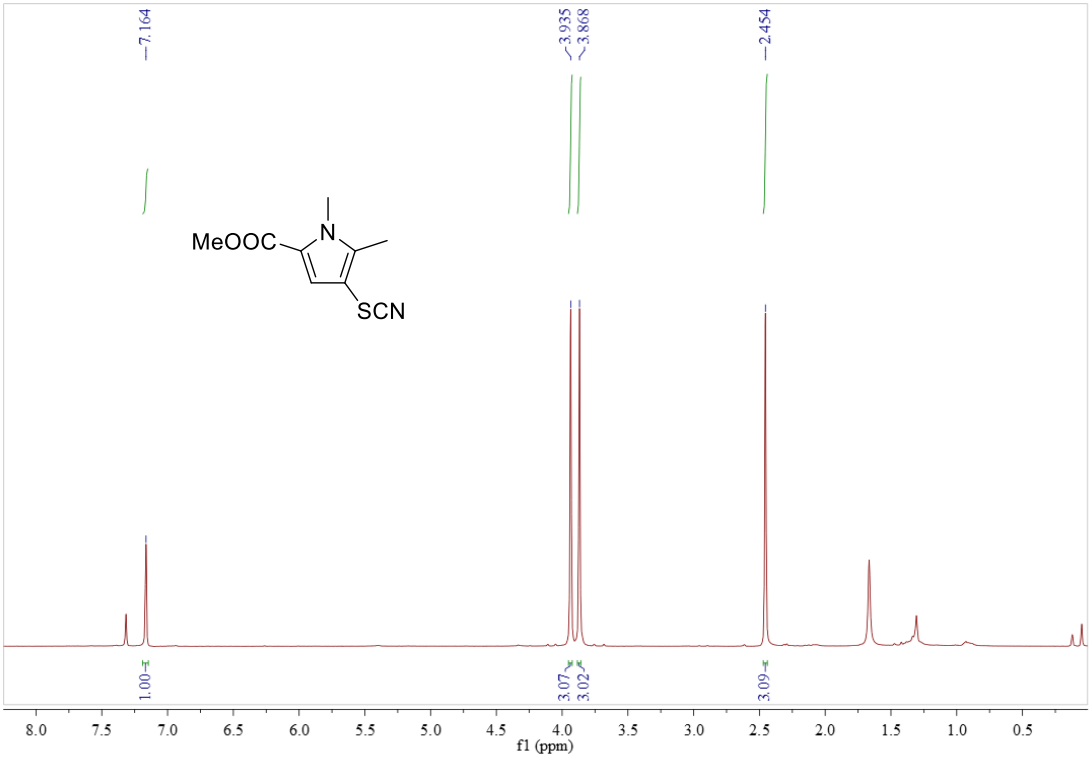
^{13}C NMR (100 MHz, Chloroform-*d*) of **4g**



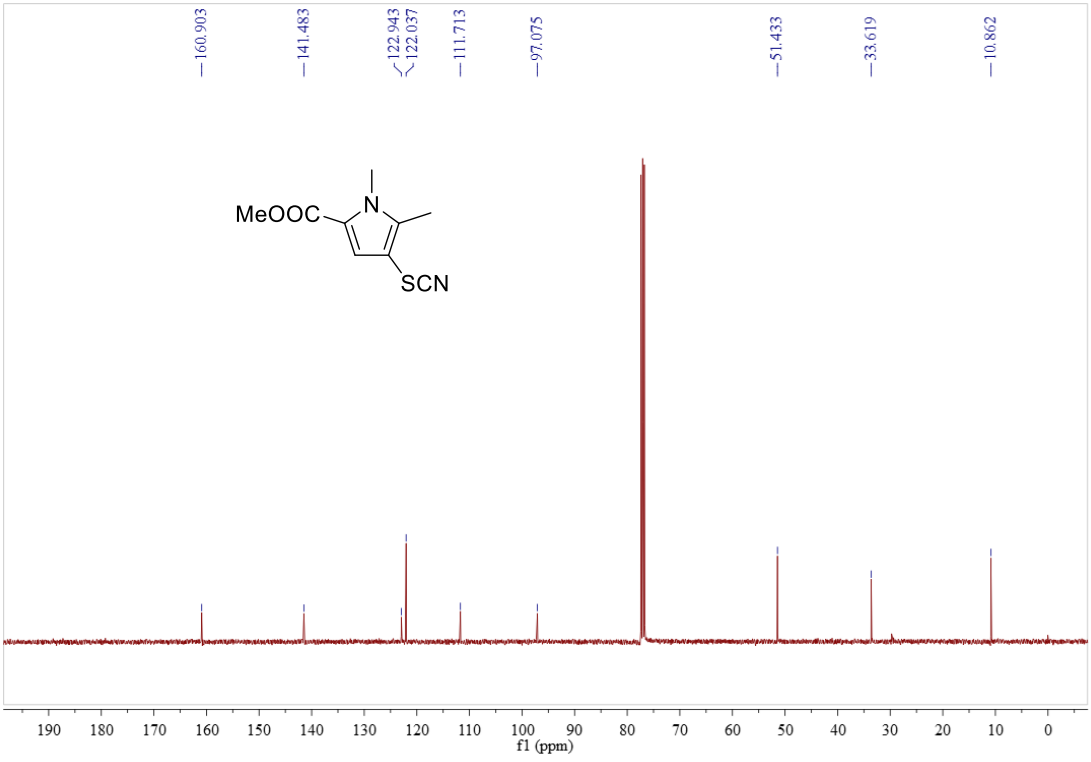
^{1}H NMR (400 MHz, Chloroform-*d*) of **4h**



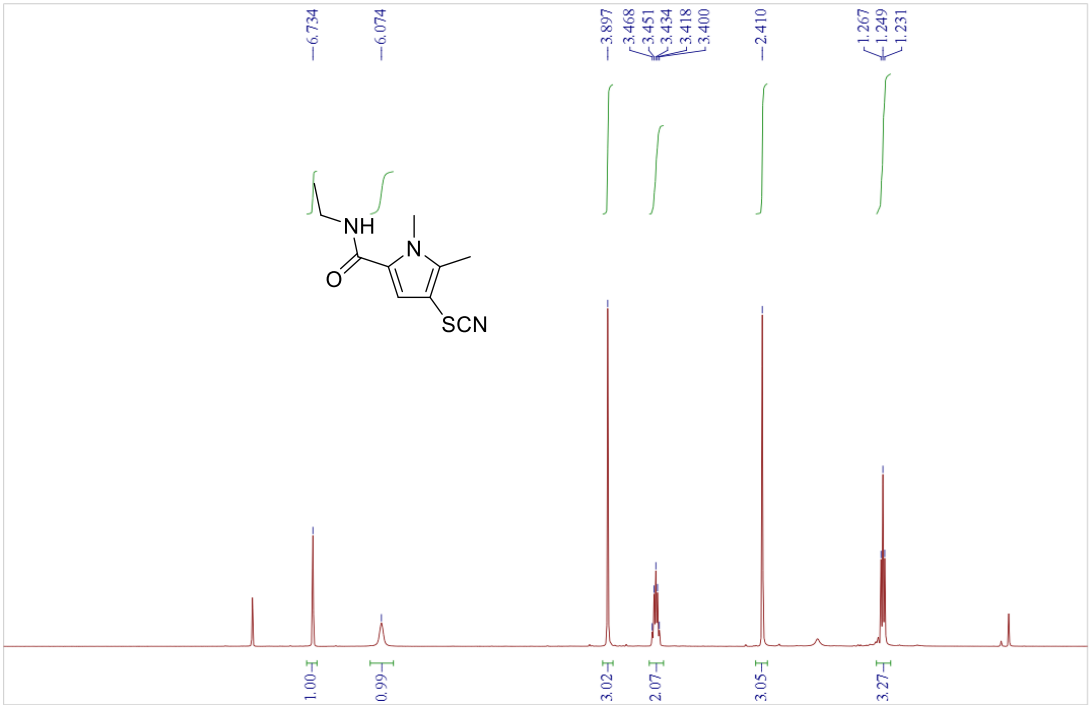
^{13}C NMR (100 MHz, Chloroform-*d*) of **4h**



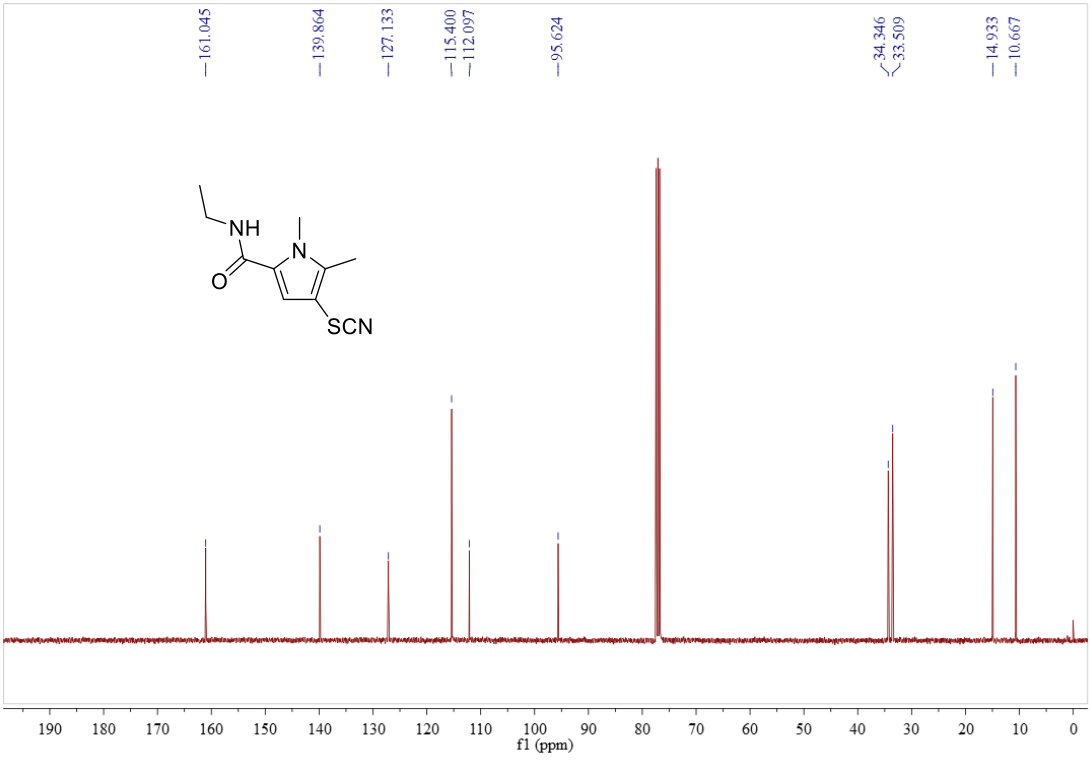
^{1}H NMR (400 MHz, Chloroform-*d*) of **4i**



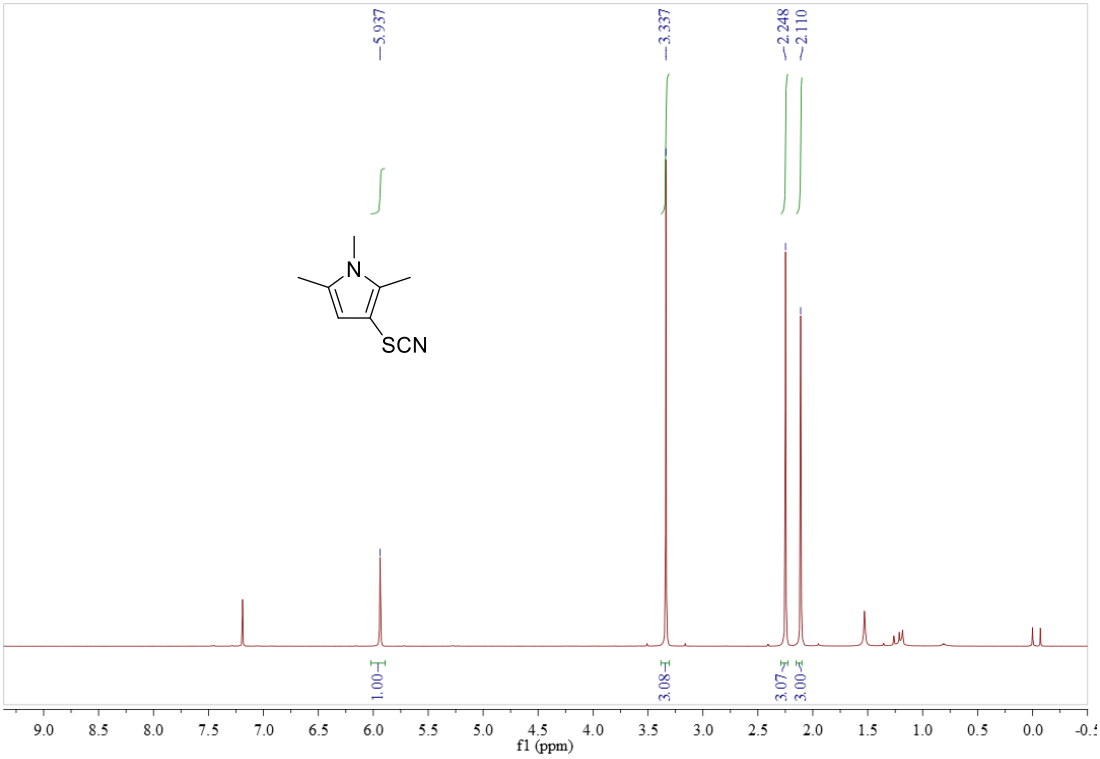
^{13}C NMR (100 MHz, Chloroform-*d*) of **4i**



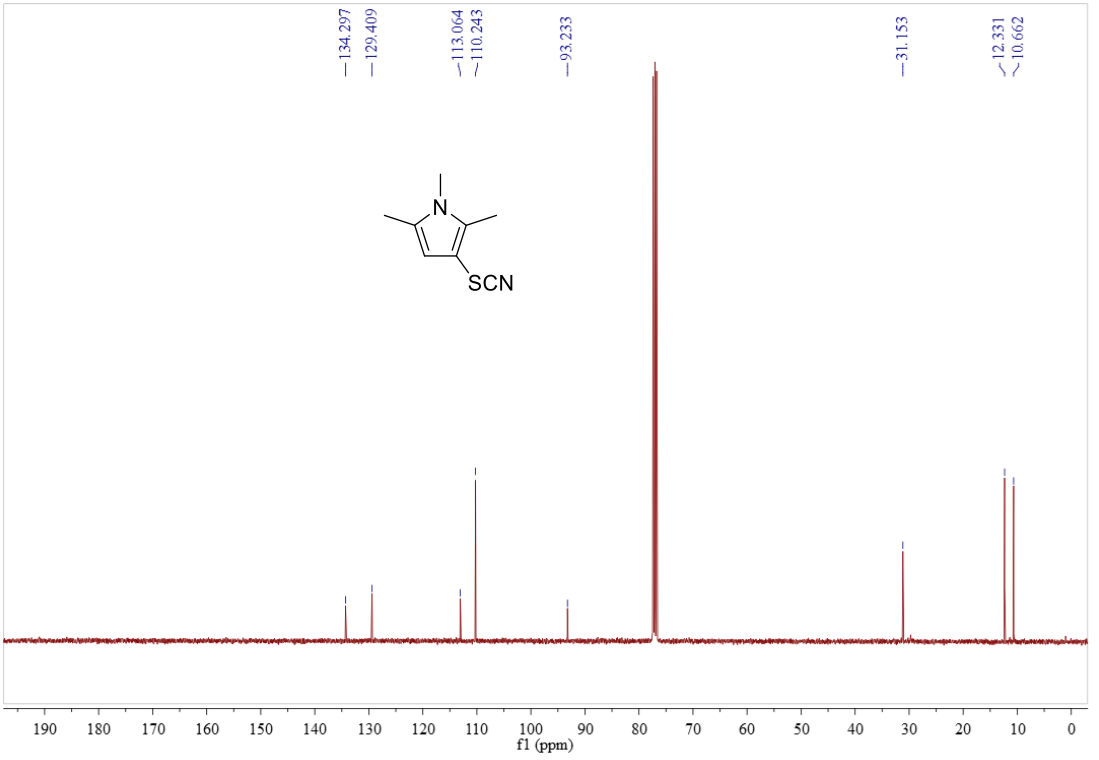
^{1}H NMR (400 MHz, Chloroform-*d*) of **4j**



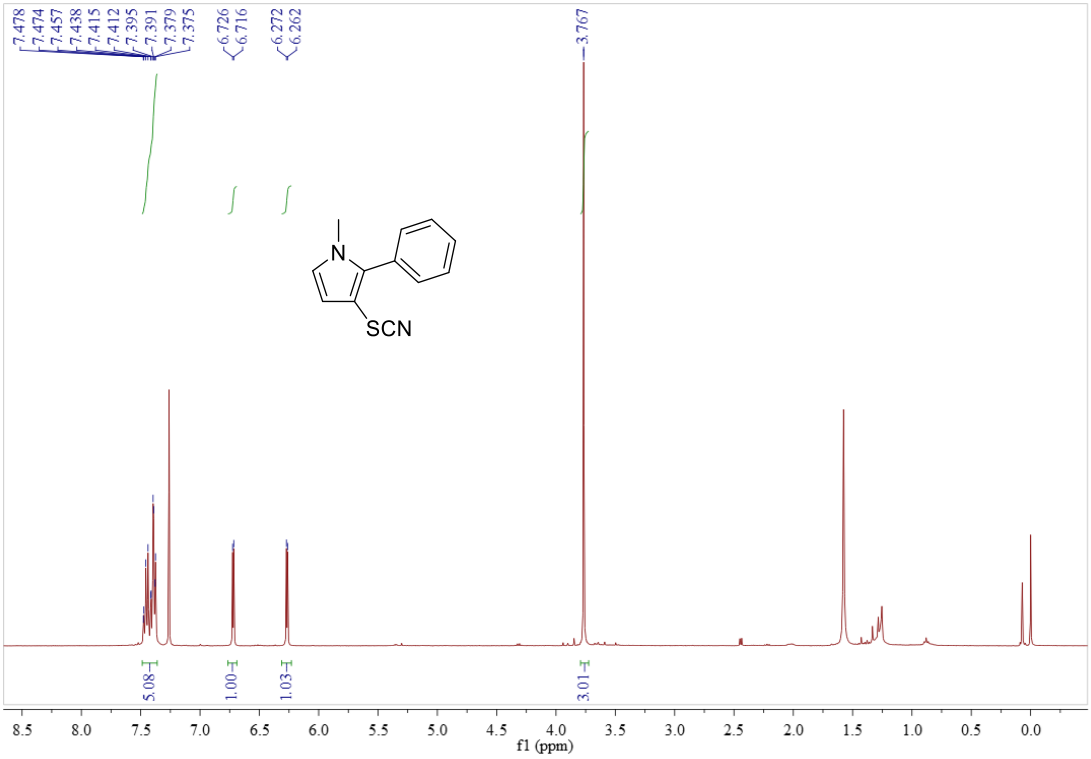
^{13}C NMR (100 MHz, Chloroform-*d*) of **4j**



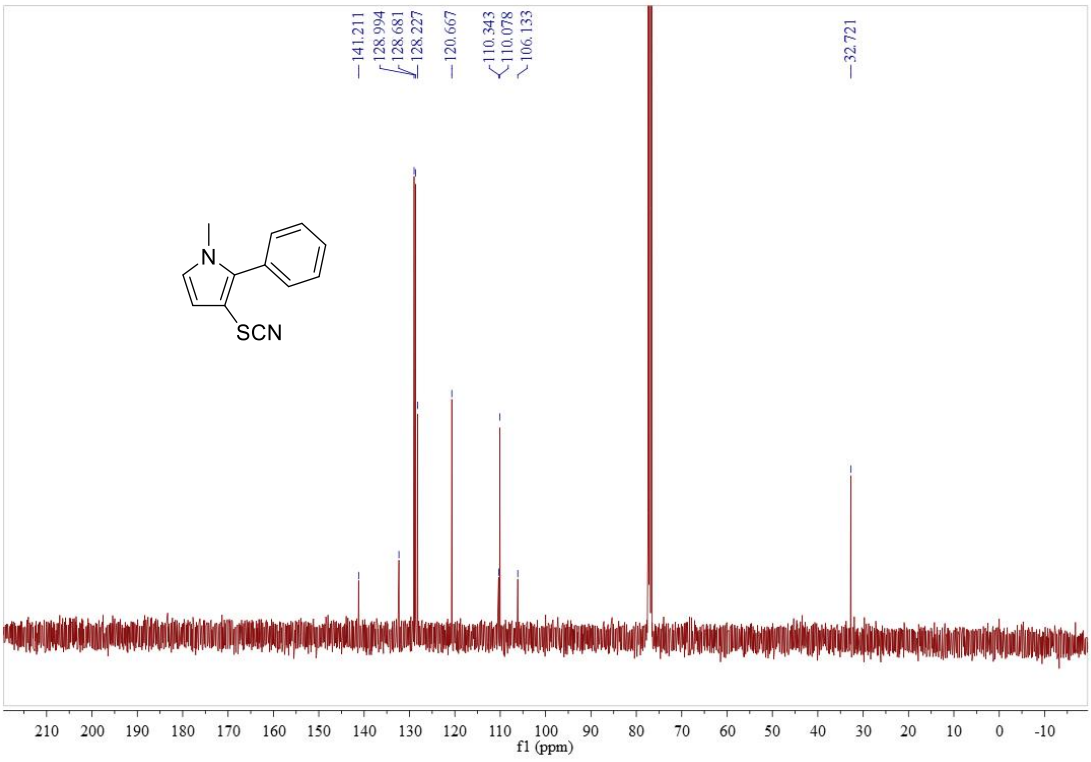
^{1}H NMR (400 MHz, Chloroform-*d*) of **4ae**



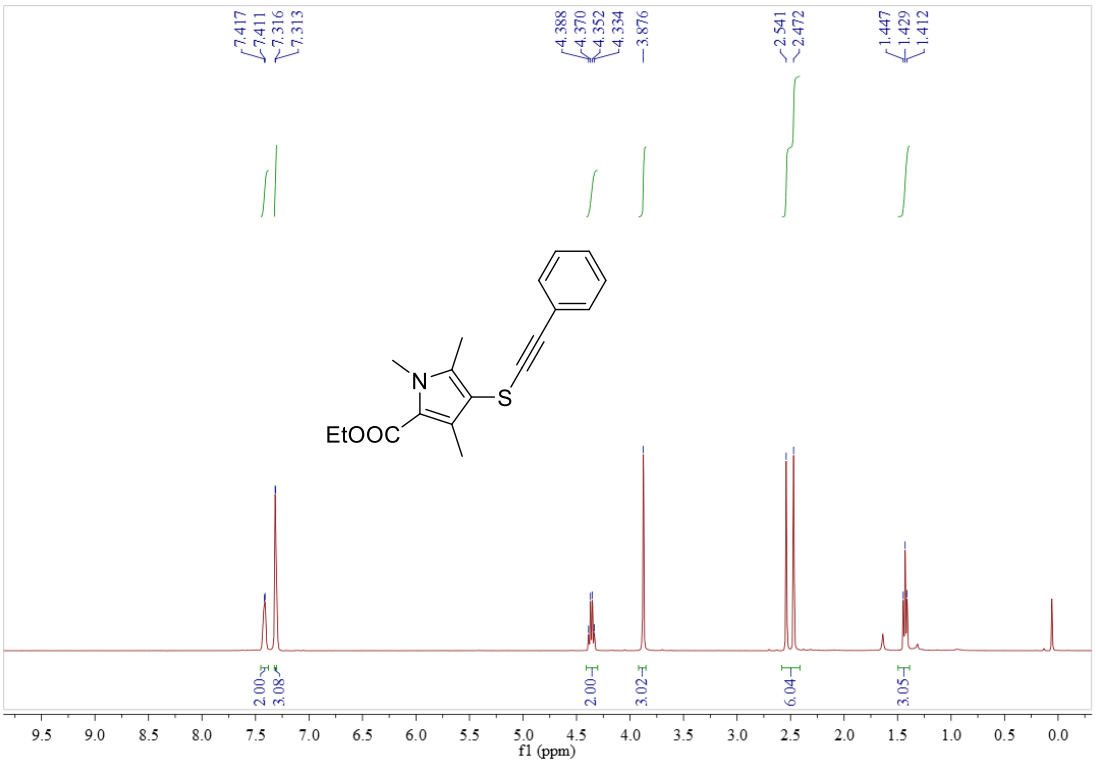
^{13}C NMR (100 MHz, Chloroform-*d*) of **4ae**



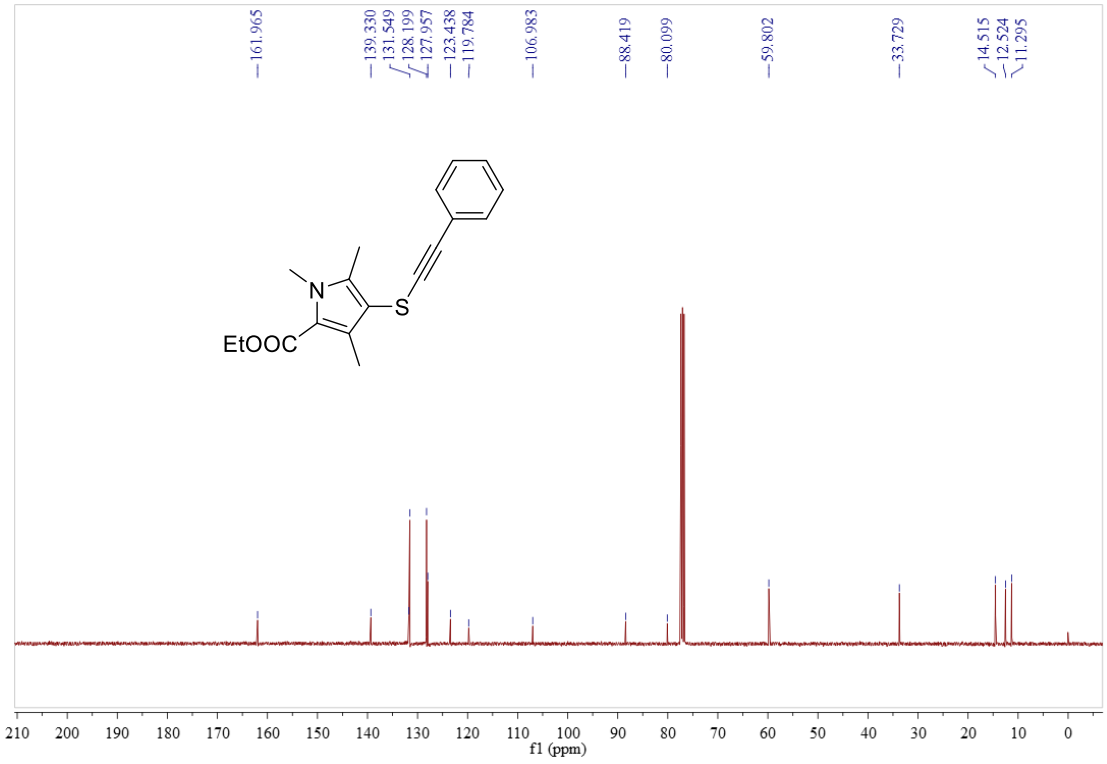
^{1}H NMR (400 MHz, Chloroform-*d*) of **4af**



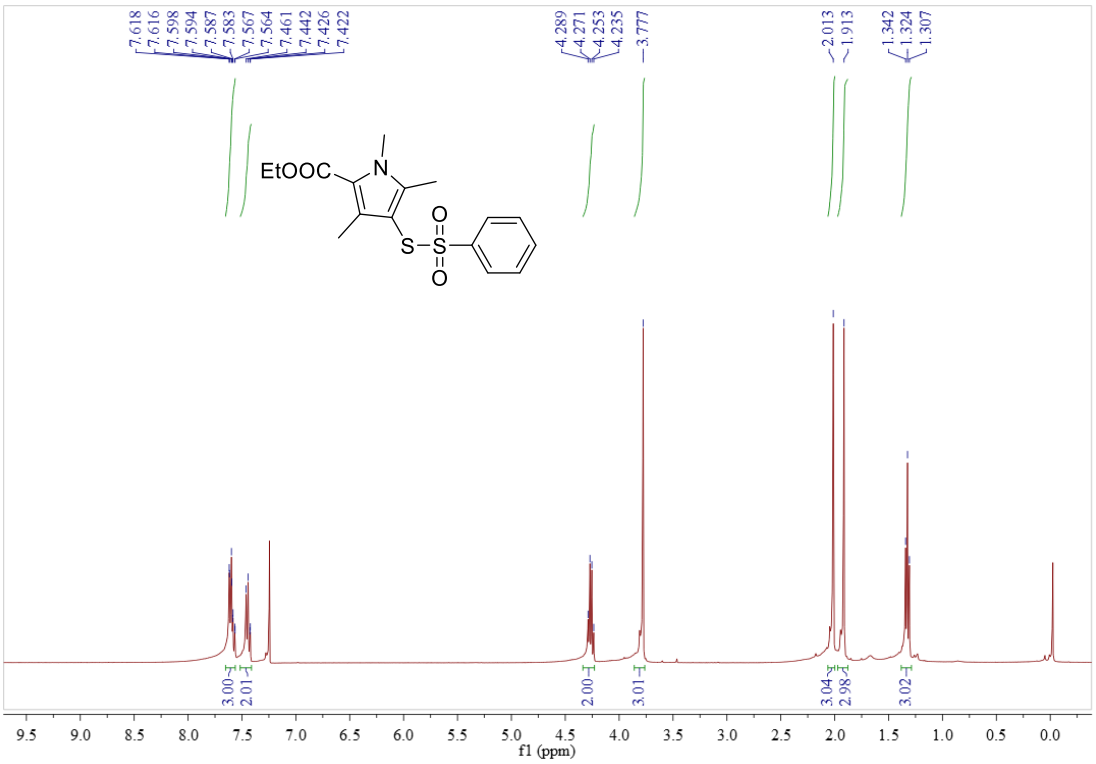
^{13}C NMR (100 MHz, Chloroform-*d*) of **4af**



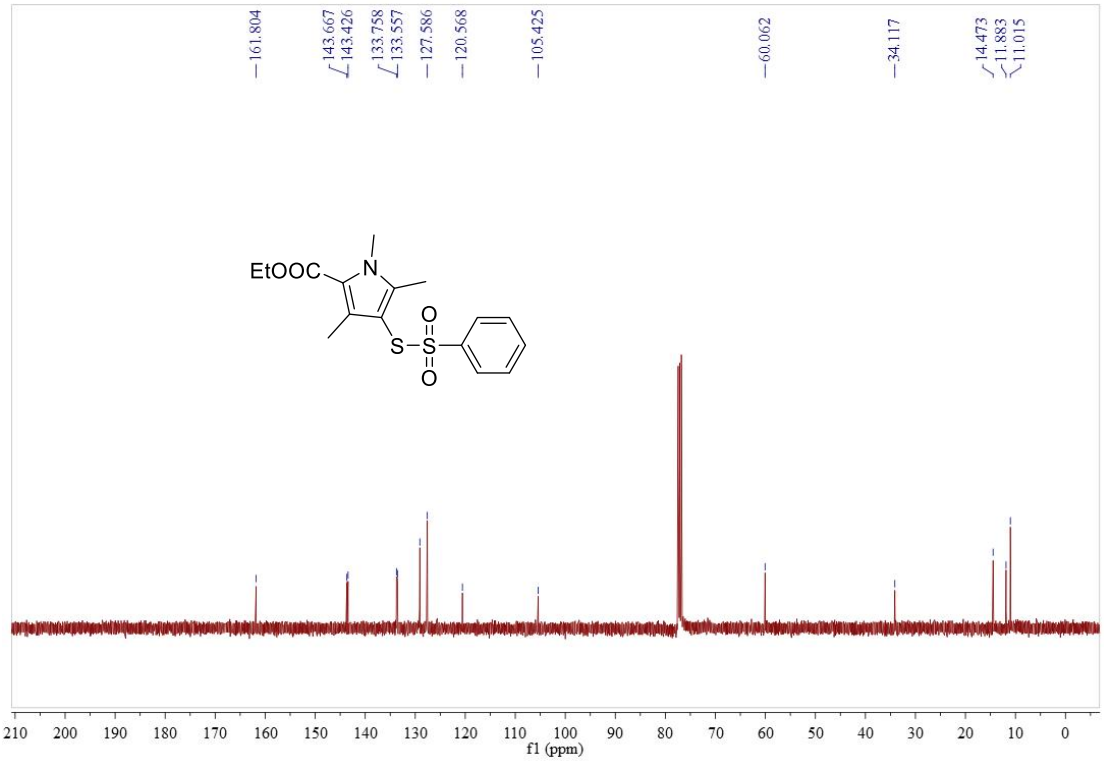
^{1}H NMR (400 MHz, Chloroform-*d*) of **5**



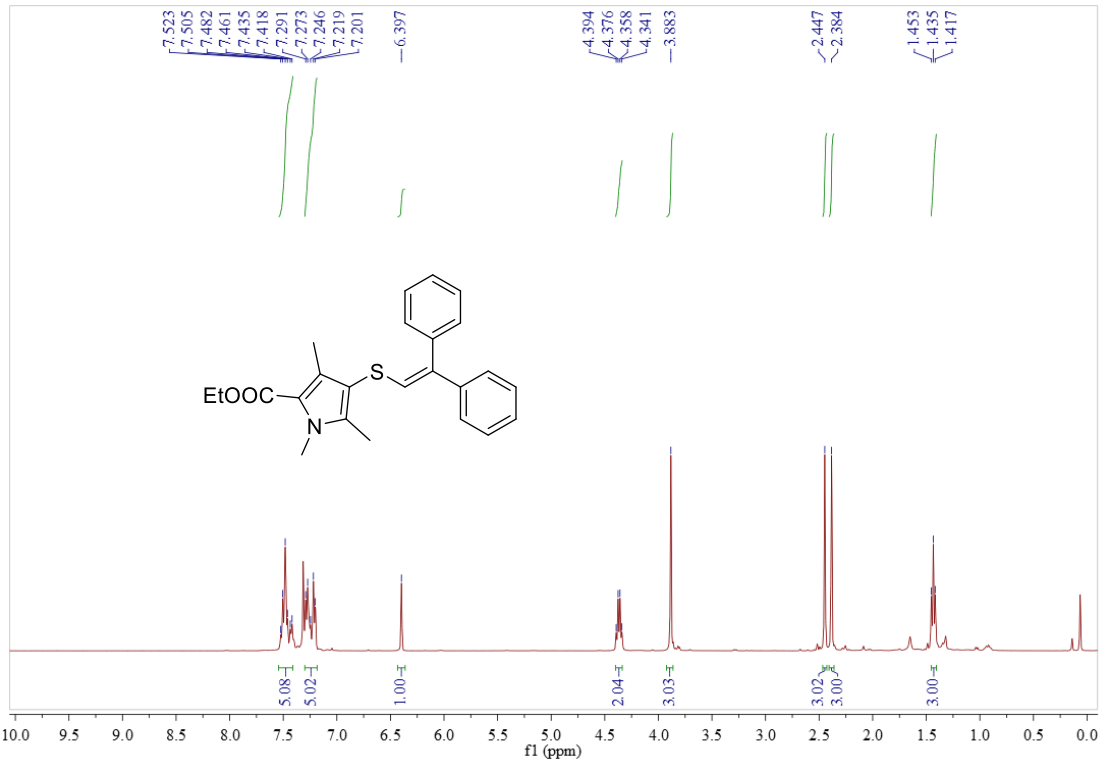
^{13}C NMR (100 MHz, Chloroform-*d*) of **5**



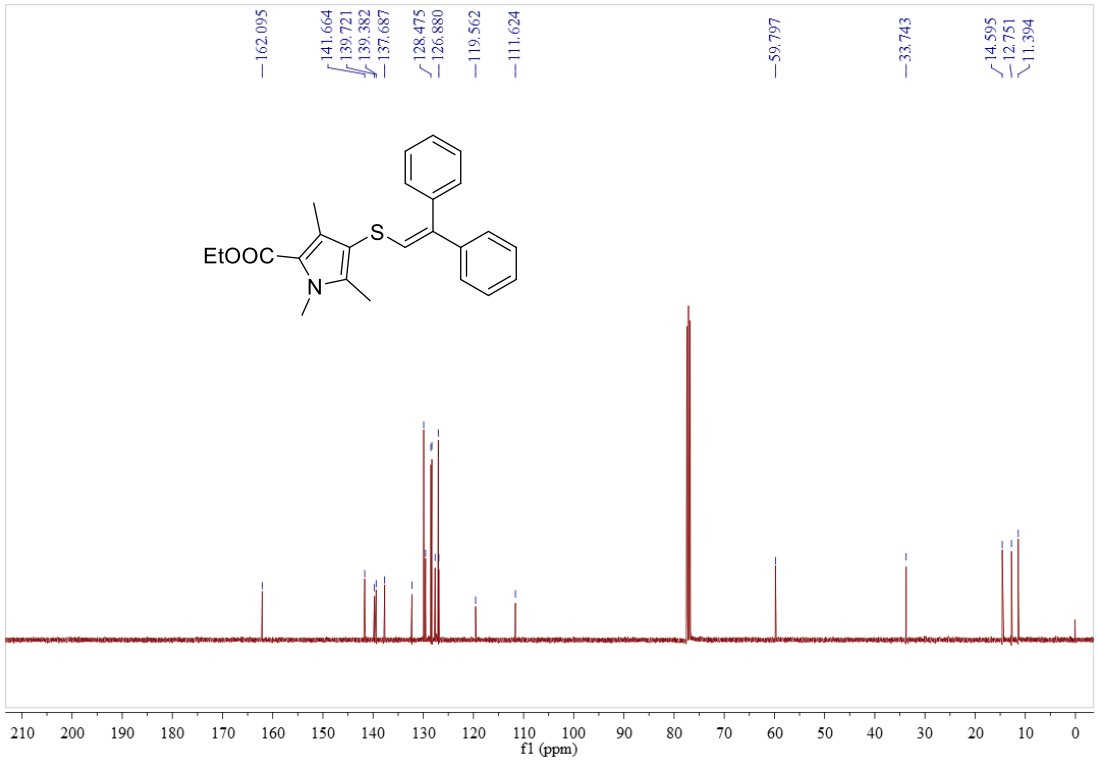
^{1}H NMR (400 MHz, Chloroform-*d*) of **6**



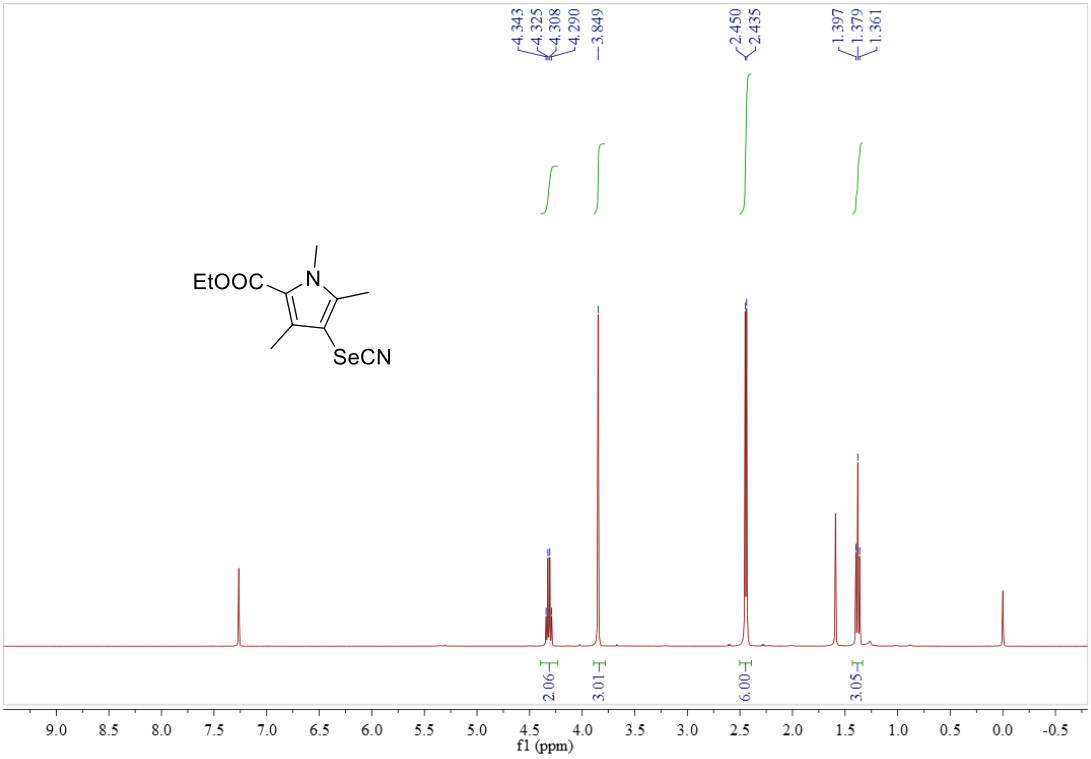
^{13}C NMR (100 MHz, Chloroform-*d*) of **6**



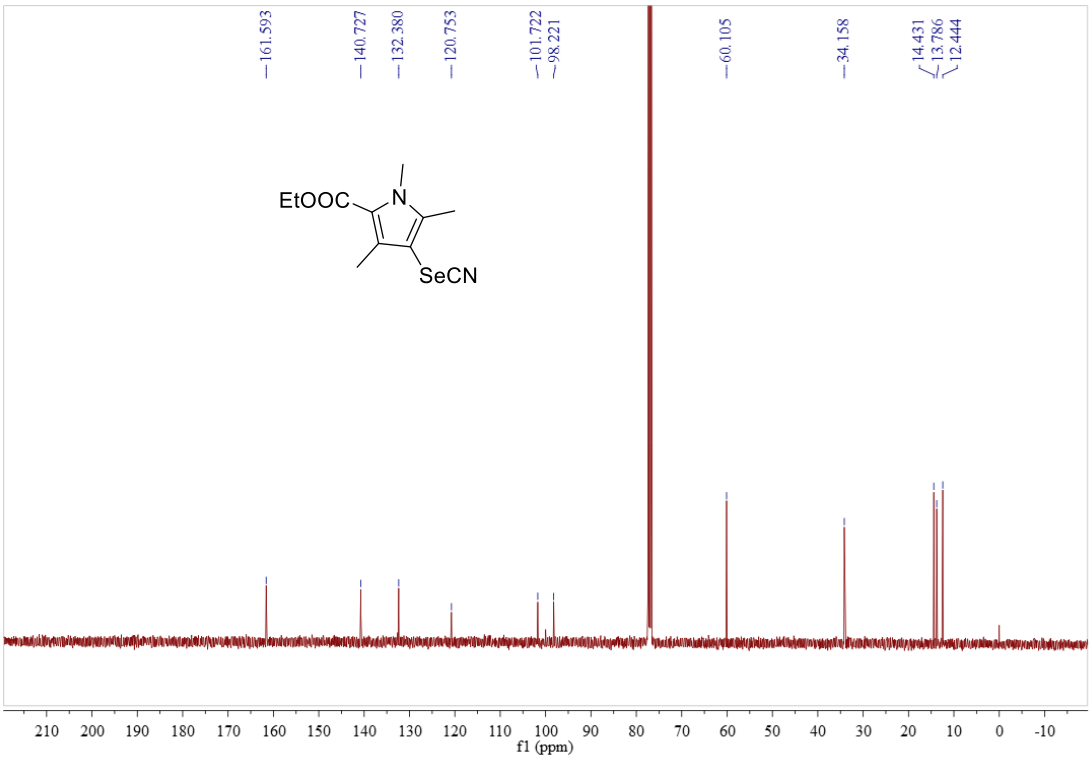
^{1}H NMR (400 MHz, Chloroform-*d*) of **7**



^{13}C NMR (100 MHz, Chloroform-*d*) of **7**



^{1}H NMR (400 MHz, Chloroform-*d*) of **8a'**



^{13}C NMR (100 MHz, Chloroform-*d*) of **8a'**