

*Electronic Supplementary Information for*

**TADF-based purely organic heterogeneous photocatalyst with  
hydrophobic domains for efficient oxidation of sulfide into sulfoxide  
in water**

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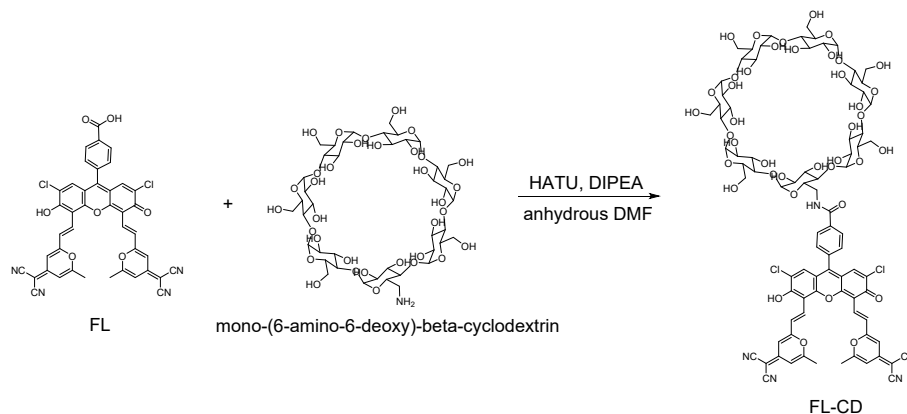
## 1 General

All chemicals were commercially available and used as received without further purification. Mass spectrometric data were obtained by a LTQ Orbitrap XL mass spectrometer of Thermo Scientific. Powder X-ray diffraction (PXRD) profiles were recorded on a Bruker D8 Advance diffractometer (40 KV, 40 mA, Cu-K $\alpha$ ,  $\lambda = 1.5418$  Å) from 5 - 40° with a step of 0.01° at a scan speed of 6° min<sup>-1</sup>. Fourier Transform Infrared (FT-IR) spectra were recorded with a Nicolet IS50 FT-IR spectrophotometer of Thermo Scientific in the range of 400 to 4000 cm<sup>-1</sup>. Scanning electron microscopy (SEM) images were recorded on a Nova NanoSEM 450 field emission scanning electron microscope of FEI and a gold layer was sputtered on the surface of each sample before the measurement. Thermogravimetric analysis (TGA) profiles were recorded on a SDT-Q600 simultaneous DSC-TGA instrument of TA from 25 °C to 800 °C with a temperature increasing rate of 10 °C min<sup>-1</sup>. Water droplet contact angle (WCA) values were obtained by an OCA50 optical contact angle measuring and contour analysis system of DATAPHYSICS. Electron spin resonance (ESR) spectra were recorded on a Bruker E500 spectrometer in MeOH at room temperature. Absorption spectra were recorded on a Carry 60 UV-vis spectrophotometer of Agilent with a sample concentration of 5  $\mu$ M in a quartz cuvette (1 cm $\times$ 1 cm) at room temperature. Fluorescence spectra were recorded on a Cary Eclipse fluorescence spectrophotometer of Agilent with a sample concentration of 5  $\mu$ M and an excitation wavelength of 460 nm at room temperature. Both the excitation and emission slits are 5 nm and PMT detector voltage is 600 V. <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were recorded on Bruker Avance II 400 and Vaian DLG400 spectrometer using DMSO-*d*<sub>6</sub>, CDCl<sub>3</sub>, CD<sub>3</sub>CN and 90% H<sub>2</sub>O+10% D<sub>2</sub>O as solvent. The chemical shift was internally referenced to tetramethylsilane signal (TMS:  $\delta$  <sup>1</sup>H = 0) or residual solvent signals (DMSO-*d*<sub>6</sub>:  $\delta$  <sup>1</sup>H = 2.50,  $\delta$  <sup>13</sup>C = 39.53. CDCl<sub>3</sub>:  $\delta$  <sup>1</sup>H = 7.26,  $\delta$  <sup>13</sup>C = 77.06. CD<sub>3</sub>CN:  $\delta$  <sup>1</sup>H = 1.94,  $\delta$  <sup>13</sup>C = 1.32. D<sub>2</sub>O:  $\delta$  <sup>1</sup>H = 4.79). <sup>1</sup>H-NMR are recorded as follows: chemical shift ( $\delta$ , ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet or unresolved, br = broad singlet, coupling constant(s) in Hz, integration). <sup>13</sup>C-NMR are reported in terms of

chemical shift ( $\delta$ , ppm).

## 2 Synthesis

### 2.1 Synthesis of FL-CD



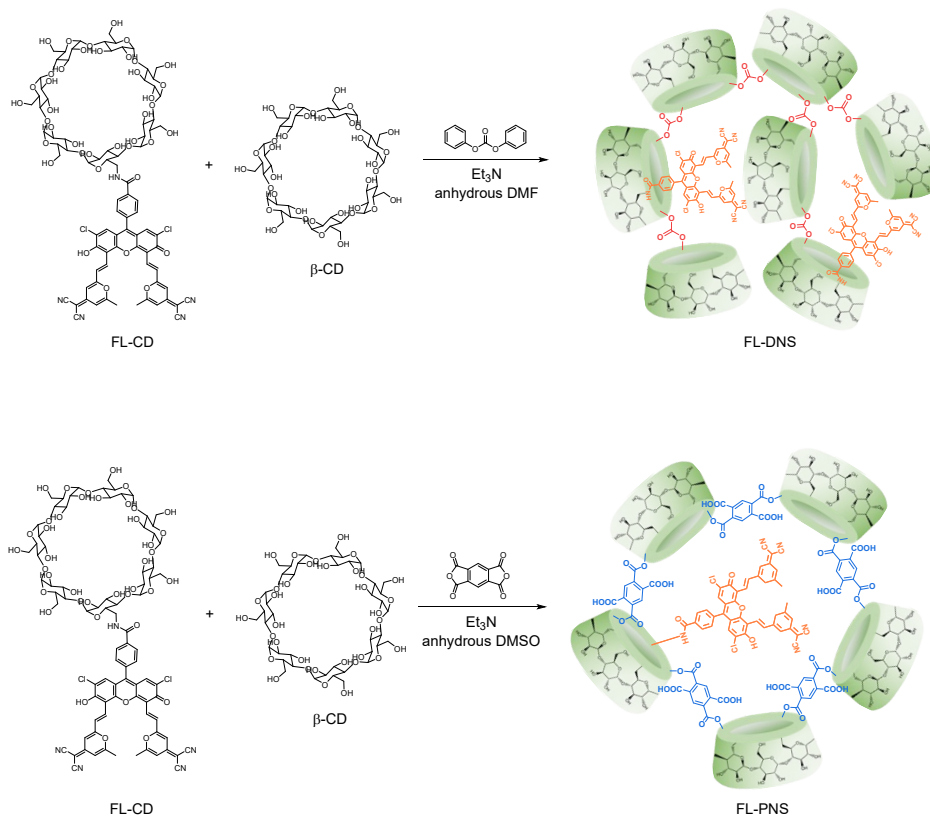
FL was synthesized according to the previously reported method<sup>1</sup>.

FL (76 mg, 0.1 mmol) and DIPEA (33  $\mu$ L, 0.2 mmol) were dissolved in anhydrous DMF (5.0 mL). HATU (57 mg, 0.15 mmol) in anhydrous DMF (1.0 mL) was added dropwise to the solution at 0 °C. After stirring for 30 min, mono-(6-amino-6-deoxy)-beta-cyclodextrin (136 mg, 0.12 mmol) in anhydrous DMF (2.0 mL) was added dropwise and the reaction mixture was gradually raised to room temperature and continued stirring for 24 hours. Once the reaction was complete, the reaction mixture was poured into acetone and filtrated under reduced pressure to obtain the crude product. The crude product was further purified by a medium-pressure liquid chromatography system using a gradient elution of deionized water and ethanol at a flow rate of 10 mL/min, with ethanol from 10% to 50% in 30 min. After removal of the solvent under reduced pressure, the target product was obtained as a black solid (65 mg, 34%).

<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  8.41 (s, 1H), 8.12 – 7.92 (m, 4H), 7.77 – 7.51 (m, 4H), 6.97 (d, *J* = 2.6 Hz, 2H), 6.61 (s, 2H), 6.45 (s, 2H), 5.82 – 5.65 (m, 14H), 5.00 – 4.81 (m, 7H), 4.49 – 4.38 (m, 6H), 3.75 – 3.54 (m, 28H), 3.46 – 3.35 (m, overlaps with HOD), 2.44 – 2.22 (m, 6H). <sup>13</sup>C NMR (101 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  171.9, 166.6, 163.5, 162.3, 161.4, 156.0, 154.4, 150.6, 135.8, 135.2, 129.8, 129.5, 128.8, 127.8, 126.9, 118.2, 115.4, 115.2, 110.4, 109.1, 105.6, 104.9, 102.0, 83.9, 81.7, 81.5, 73.1, 72.5, 72.2, 59.9, 54.9, 35.8, 30.8, 19.4. HRMS (ESI/TOF) *m/z* calcd for C<sub>84</sub>H<sub>90</sub>Cl<sub>2</sub>N<sub>5</sub>O<sub>40</sub><sup>-</sup> ([M-H]<sup>-</sup>)

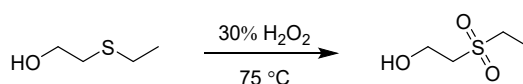
): 1880.4510, found: 1880.4507.

## 2.2 Synthesis of FL-DNS and FL-PNS



**Scheme S1** Synthesis of FL-DNS and FL-PNS

## 2.3 Synthesis of 2-ethylsulfonylethanol<sup>2</sup>

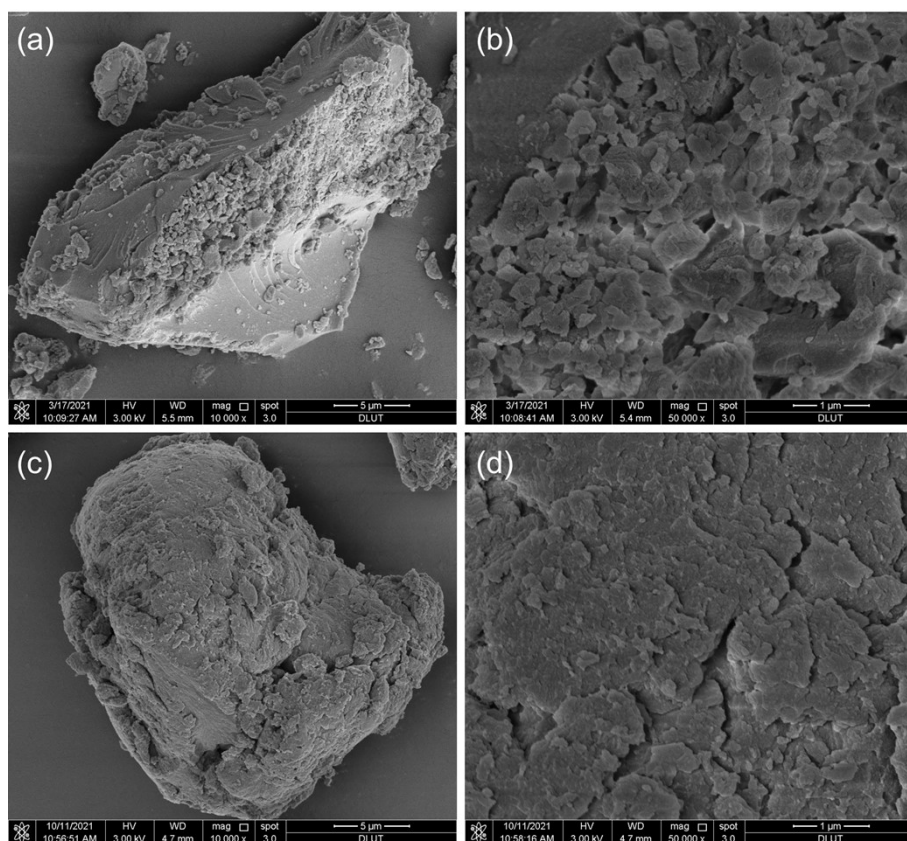


In order to better identify the overoxidation product from the reaction solution, 2-ethylsulfonylethanol was synthesized according to the previously reported method. Specifically, to ethyl 2-hydroxyethyl sulfide (20 mmol, 2.1 mL), a 30% aqueous solution of  $\text{H}_2\text{O}_2$  (5 mmol, 0.5 mL) was added dropwise, and the reaction mixture was heated at  $75\text{ }^\circ\text{C}$  for 3 h in a round-bottom flask equipped with a reflux condenser. After cooling to room temperature, a 30% aqueous solution of  $\text{H}_2\text{O}_2$  (20 mmol, 2 mL) was added and the reaction mixture was heated at  $75\text{ }^\circ\text{C}$  for further 12 h. Additional  $\text{H}_2\text{O}_2$  (20 mmol, 2 mL) was added when the total reaction time reached 15 h and 39 h, respectively. And the full conversion was reached in 63 h. After cooling to room

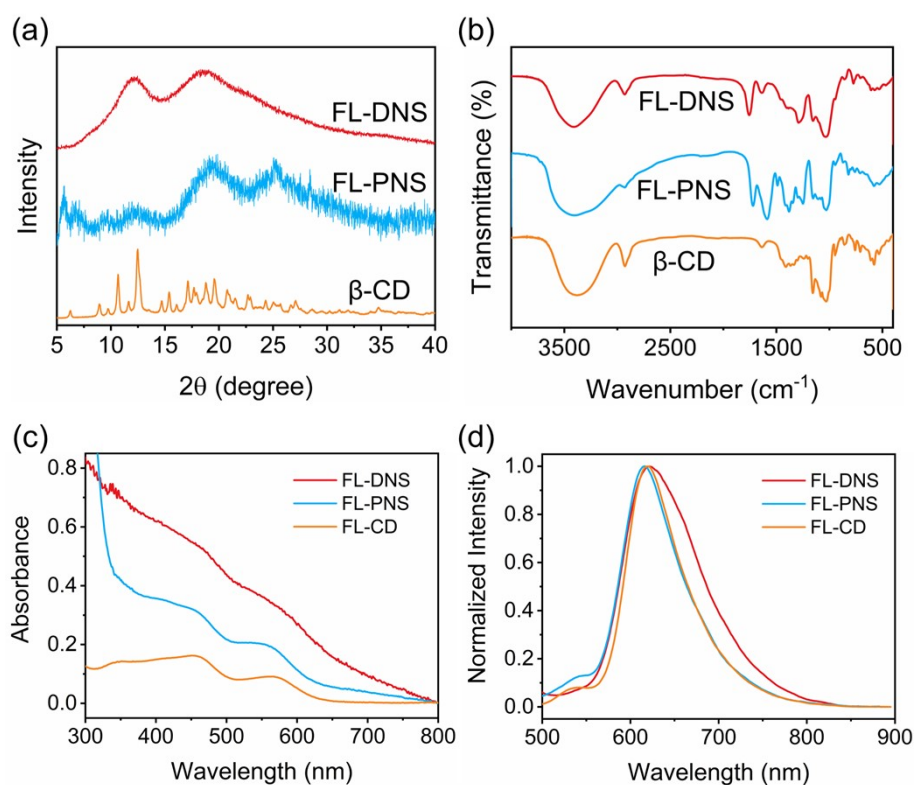
temperature,  $\text{Na}_2\text{SO}_3$  was added partially to neutralize excess  $\text{H}_2\text{O}_2$ . The mixture was extracted with  $\text{CH}_2\text{Cl}_2$ . The combined organic layers were washed with water and brine, and then dried over anhydrous  $\text{Na}_2\text{SO}_4$ . After removal of solvent under reduced pressure, the target product was obtained as a colorless liquid (2.30 g, 83%).

$^1\text{H}$  NMR (400 MHz, Methanol- $d_4$ )  $\delta$  3.99 – 3.94 (m, 2H), 3.22 (t,  $J = 5.6$  Hz, 2H), 3.16 (q,  $J = 7.5$  Hz, 2H), 1.34 (t,  $J = 7.5$  Hz, 3H).  $^{13}\text{C}$  NMR (101 MHz, Methanol- $d_4$ )  $\delta$  56.8, 55.5, 49.9, 6.5. HRMS (ESI/TOF)  $m/z$  calcd for  $\text{C}_4\text{H}_{10}\text{NaO}_3\text{S}^+$  ( $[\text{M}+\text{Na}]^+$ ): 161.0248, found: 161.0238.

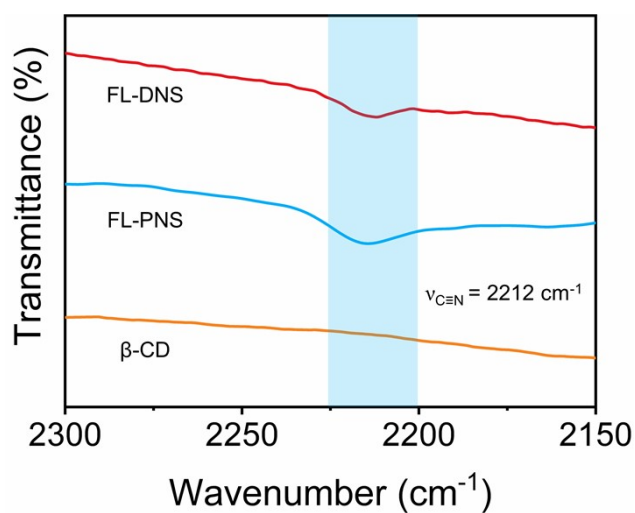
### 3 Characterization



**Fig. S1** SEM images of (a) FL-DNS and (c) FL-PNS. Scale bar = 5  $\mu\text{m}$ . (b) and (d) are the details with enlarged scale of (a) and (c), respectively. Scale bar = 1  $\mu\text{m}$ .



**Fig. S2** (a) PXRD profiles and (b) FT-IR spectra of FL-DNS, FL-PNS, and  $\beta$ -CD. (c) Absorption and (d) fluorescence spectra of FL-DNS, FL-PNS, and FL-CD.



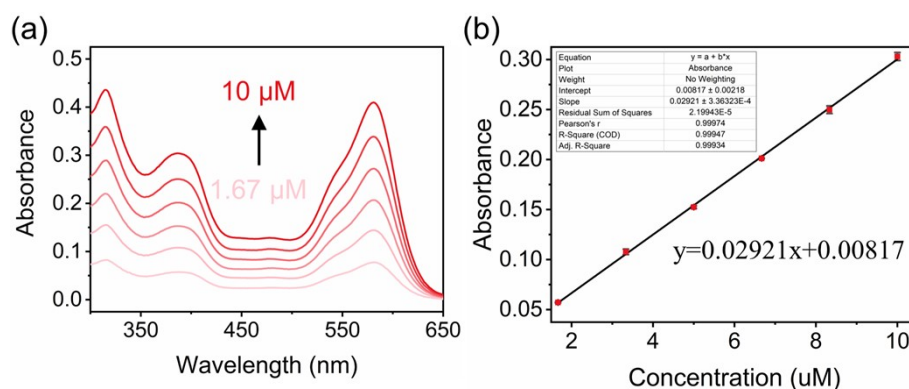
**Fig. S3** FT-IR spectra of FL-DNS, FL-PNS, and  $\beta$ -CD.

### 3.1 Determination of FL-CD content

The contents of FL-CD in FL-DNS and FL-PNS were determined by standard curve. FL-CD was dissolved in a mixed solvent of DMF,  $\text{H}_2\text{O}$ , and  $\text{NH}_3 \cdot \text{H}_2\text{O}$  (2.5 : 0.4 : 0.1,

V/V) to prepare a stock solution with a concentration of 1 mmol/L (1 mM). Taking 5, 10, 15, 20, 25, 30  $\mu\text{L}$  of the stock solution, respectively, and diluting to 3 mL with the above mentioned mixed solution to test the absorption spectra. Plotting the absorbance at 390 nm versus corresponding concentration to obtain the standard curve at the concentration range of 0 - 10  $\mu\text{M}$ .

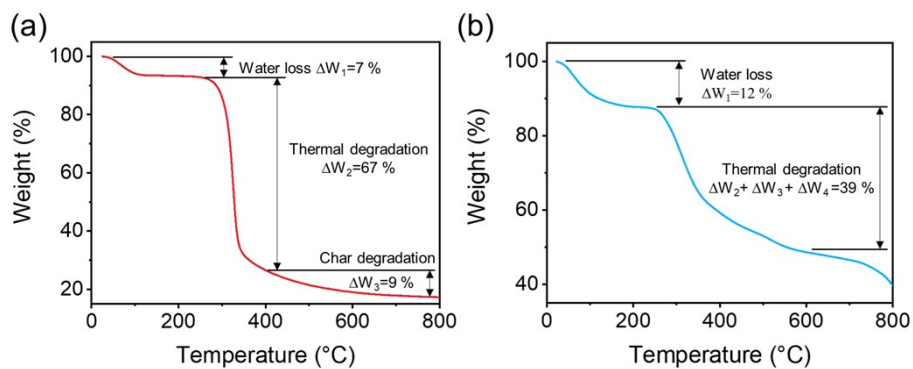
$\text{H}_2\text{O}$  (0.8 mL) and  $\text{NH}_3\cdot\text{H}_2\text{O}$  (0.2 mL) were added to the appropriate amount of FL-DNS or FL-PNS and the resulting suspension was sonicated for 1 h to get a clear solution. Taking 0.5 mL of the solution and diluting to 3 mL with DMF to test the absorption spectra. And the content of FL-CD in FL-DNS or FL-PNS was calculated by the absorbance at 390 nm according to the standard curve.



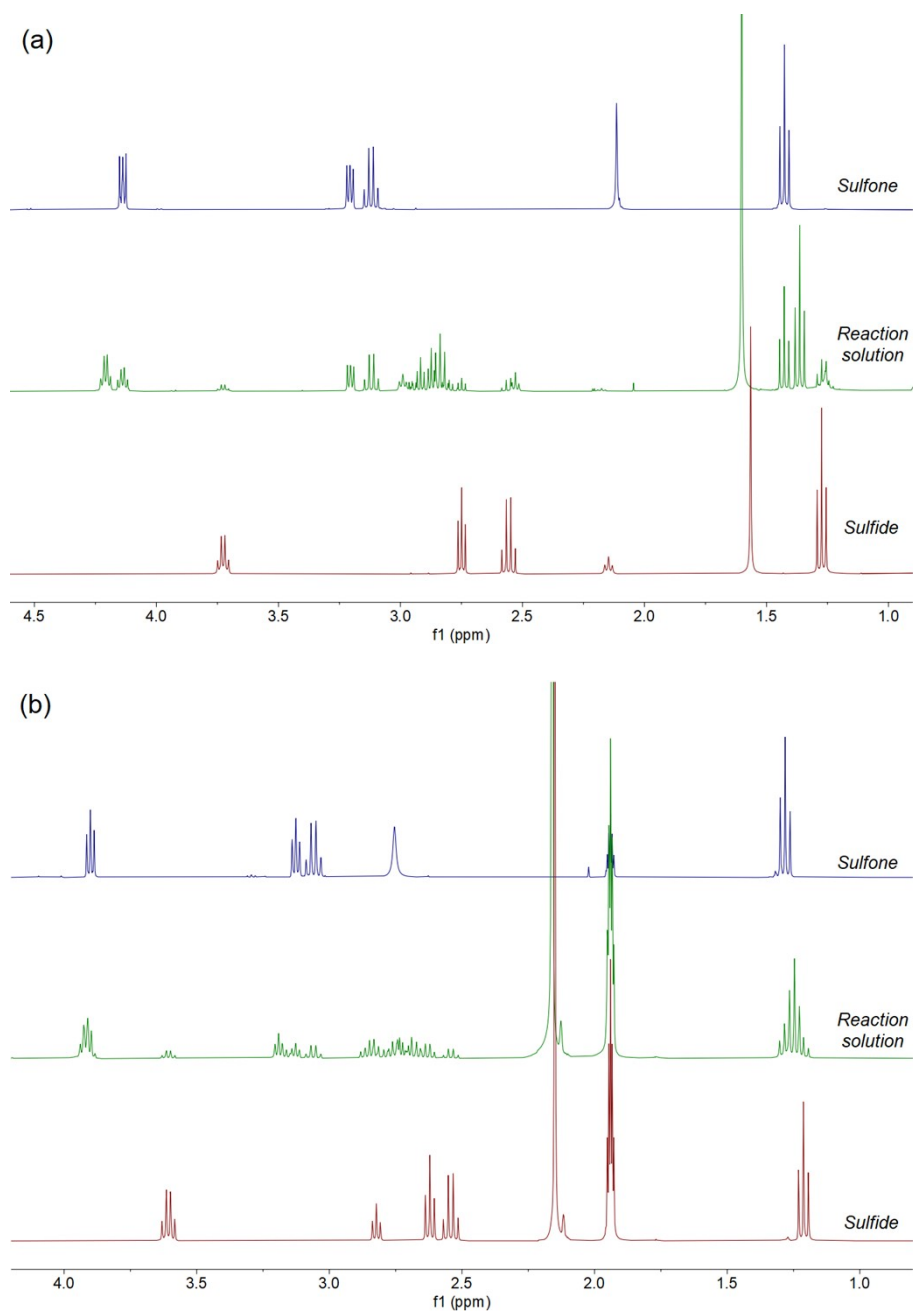
**Fig. S4** (a) Absorption spectra of FL-CD in a mixed solvent of DMF,  $\text{H}_2\text{O}$  and  $\text{NH}_3\cdot\text{H}_2\text{O}$  (2.5 : 0.4 : 0.1, V/V) at different concentrations. (b) The standard curve obtained by plotting the absorbance at 390 nm versus corresponding concentration.

**Table S1** The FL-CD contents in FL-DNS and FL-PNS.

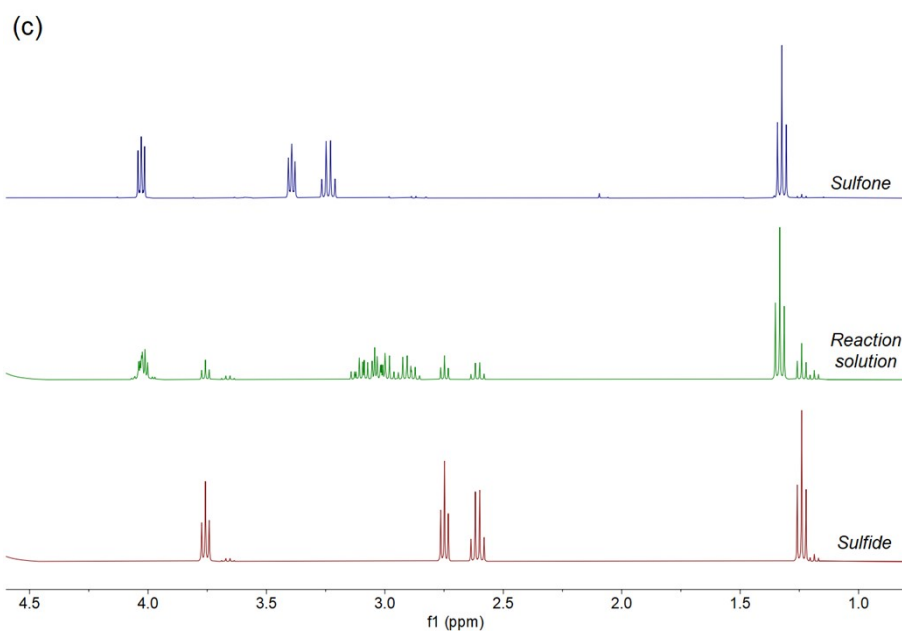
Photocatalyst	Weight /mg	$A_{390\text{nm}}$	Content / (nmol/mg)	Mean / (nmol/mg)
FL-DNS	4.00	0.146	7.08	7.20
	4.17	0.153	7.13	
	5.69	0.213	7.39	
FL-PNS	2.94	0.064	3.90	4.12
	3.34	0.078	4.29	
	3.60	0.081	4.16	



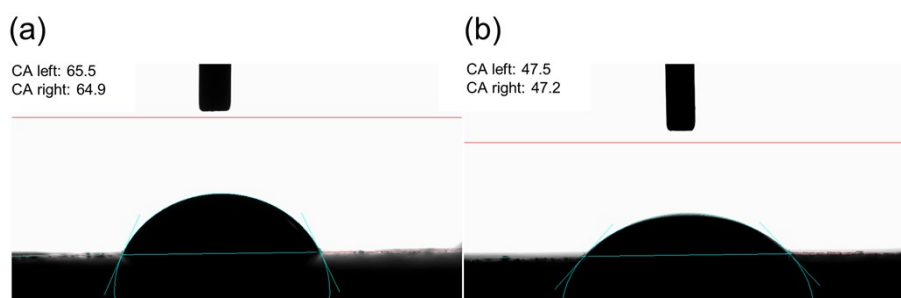
**Fig. S5** TG curves of (a) FL-DNS and (b) FL-PNS.







**Fig. S6**  $^1\text{H}$  NMR spectra of the reaction solutions when using (a)  $\text{CDCl}_3$  (b)  $\text{CD}_3\text{CN}$  and (c)  $\text{H}_2\text{O}$ , respectively, as solvents for photocatalytic aerobic oxidation of ethyl 2-hydroxyethyl sulfide to corresponding sulfoxide. Obviously, the catalytic reaction carried out in water was highly selective and no overoxidation product sulfone was detected.



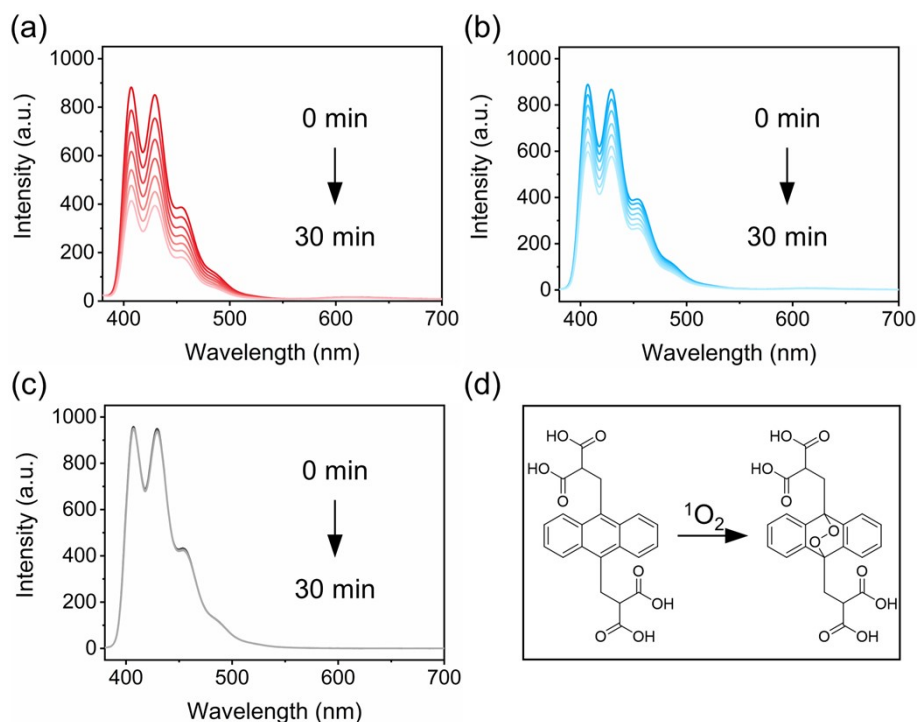
**Fig. S7** WCA measurements of (a) FL-DNS and (b) FL-PNS.

### 3.2 Verification of $^1\text{O}_2$ and $\text{O}_2^{\cdot-}$ generation.

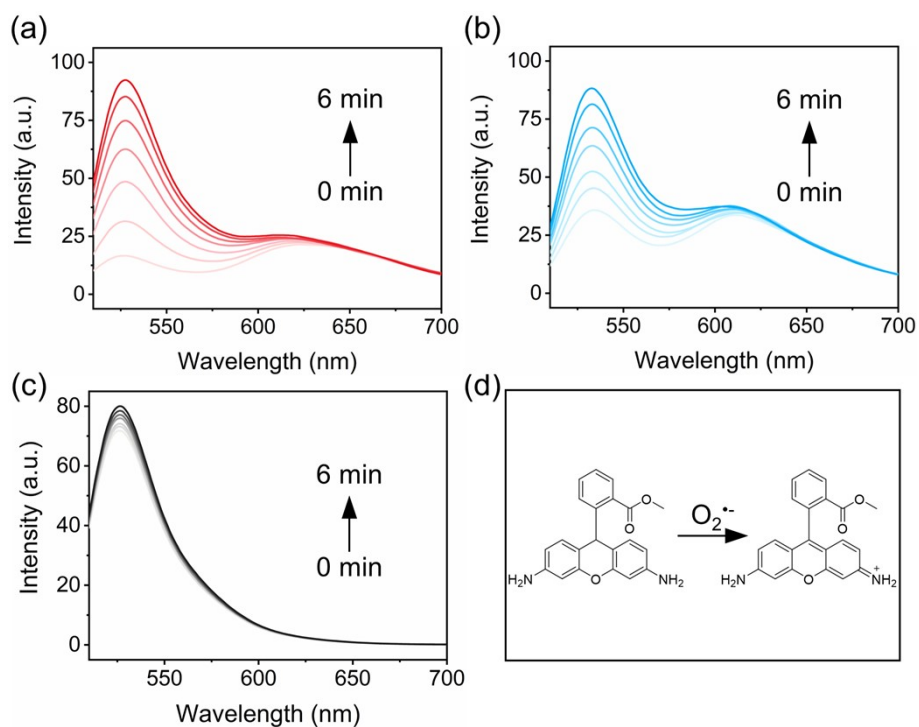
FL-DNS or FL-PNS was dispersed in  $\text{H}_2\text{O}$  (2 mL) at a concentration of  $5\ \mu\text{M}$  to test the generation of  $^1\text{O}_2$ .  $1\ \mu\text{L}$  ABDA (10 mM in DMSO) was added and the mixture was irradiated with a white-light LED ( $20\ \text{mW}\cdot\text{cm}^{-2}$ ) under stirring. The emission spectrum

of the mixture was recorded every 5 min with an excitation wavelength of 360 nm. The fluorescence intensity change of ABDA was defined as  $(I_t - I_0)/I_0$ , where  $I_t$  and  $I_0$  referred to the fluorescence intensity at 407 nm after irradiation for  $t$  min and before irradiation, respectively. Plotting the fluorescence intensity change of ABDA versus the irradiation time to reveal the ability of FL-DNS or FL-PNS to generate  $^1O_2$ .

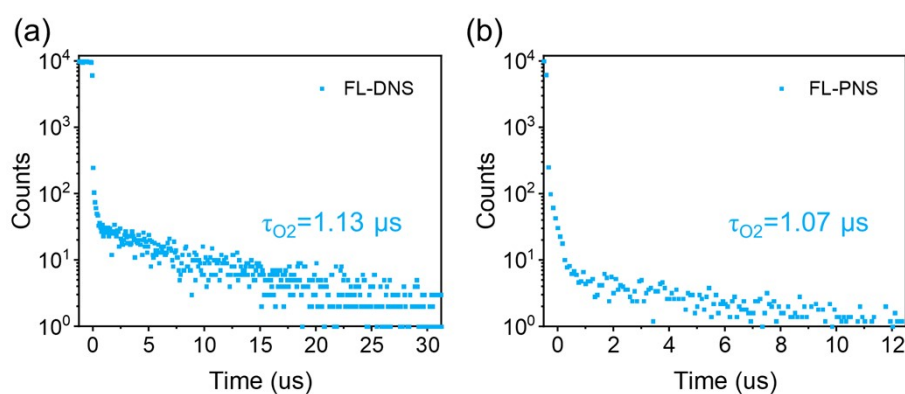
FL-DNS or FL-PNS was dispersed in  $H_2O$  (2 mL) at a concentration of  $5 \mu M$  to test the generation of  $O_2^{\cdot-}$ .  $8 \mu L$  of DHR 123 (2.5 mM in DMSO) was added and the mixture was irradiated with a white-light LED ( $20 mW \cdot cm^{-2}$ ) under stirring. The emission spectrum of the mixture was recorded every 1 min with an excitation wavelength of 488 nm. The fluorescence intensity change of DHR 123 was defined as  $(I_t - I_0)/I_0$ , where  $I_t$  and  $I_0$  referred to the fluorescence intensity at 528 nm after irradiation for  $t$  min and before irradiation, respectively. Plotting the fluorescence intensity change of DHR 123 versus the irradiation time to reveal the ability of FL-DNS or FL-PNS to generate  $O_2^{\cdot-}$ .



**Fig. S8** Time-course fluorescence intensity of (a) FL-DNS and ABDA, (b) FL-PNS and ABDA, and (c) ABDA alone after photoirradiation. (d) Chemical reactions of ABDA with  $^1O_2$ .



**Fig. S9** Time-course fluorescence intensity of (a) FL-DNS and DHR 123, (b) FL-PNS and DHR 123, and (c) DHR 123 alone after photoirradiation. (d) Chemical reactions of DHR123 with  $O_2^{\cdot-}$ .



**Fig. S10** Time-resolved decay spectra of (a) FL-DNS and (b) FL-PNS in  $O_2$ -saturated  $H_2O$ .

**Table S2** The luminescence lifetimes of FL-DNS and FL-PNS.

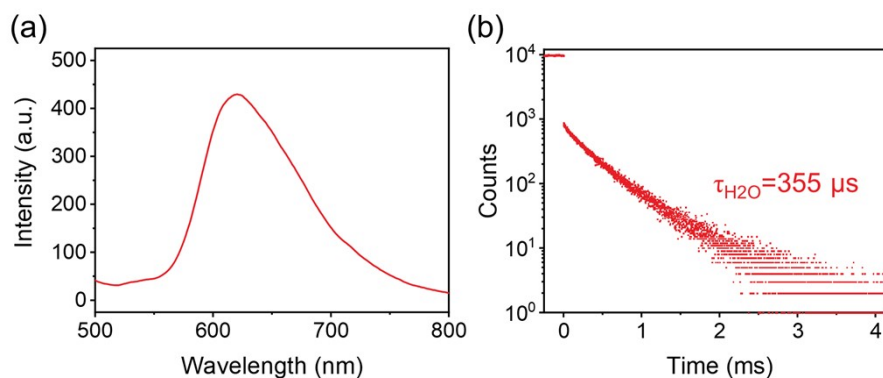
Catalysts	Solvents	$\tau_1/\mu\text{s}$	$B_1$	$\tau_2/\mu\text{s}$	$B_2$	$\tau_3/\mu\text{s}$	$B_3$	$\tau/\mu\text{s}$	$\chi^2$
FL-DNS	CH <sub>3</sub> CN <sup>a</sup>	74.41	0.71	246.05	0.29	/	/	124.18	1.02
	H <sub>2</sub> O <sup>a</sup>	207.51	0.49	546.72	0.51	/	/	380.51	1.02
	H <sub>2</sub> O <sup>b</sup>	0.13	0.87	7.82	0.13	/	/	1.13	0.96
FL-PNS	CH <sub>3</sub> CN <sup>a</sup>	139.07	0.60	675.58	0.34	2732.17	0.06	477.07	1.00
	H <sub>2</sub> O <sup>a</sup>	88.06	1	/	/	/	/	88.06	0.98
	H <sub>2</sub> O <sup>b</sup>	0.19	0.85	6.08	0.15	/	/	1.07	1.12

<sup>a</sup>In argon ambience.<sup>b</sup>In oxygen ambience.

## 4 Photocatalytic aerobic oxidation of sulfides into sulfoxides

### 4.1 Recycling experiments

For the recovery and reuse of FL-DNS, ethyl 2-hydroxyethyl sulfide (0.1 mmol, 10  $\mu\text{L}$ ) and FL-DNS (0.2 mol%, based on FLCDC contained in the photocatalyst) were suspended in O<sub>2</sub>-saturated H<sub>2</sub>O (5 mL) in a 10 mL glass vessel equipped with an O<sub>2</sub> balloon. Then the reaction vessel was irradiated with a white-light LED (20 mW $\cdot\text{cm}^{-2}$ ) for 80 min under stirring. After recovering by centrifugation and washing three times with H<sub>2</sub>O, FL-DNS was reused in the next cycle. And the yield of 2-hydroxyethyl ethyl sulfoxide for each cycle was determined by <sup>1</sup>H NMR.

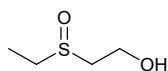
**Fig. S11** (a) Fluorescence spectrum and (b) time-resolved decay spectrum of FL-DNS in deaerated H<sub>2</sub>O after stirring in the dark for ten hours.

**Table S3** The luminescence lifetime of FL-DNS in deaerated H<sub>2</sub>O.

Conditions	$\tau_1/\mu\text{s}$	$B_1$	$\tau_2/\mu\text{s}$	$B_2$	$\tau/\mu\text{s}$	$\chi^2$
stirring in the dark	189.34	0.50	520.33	0.50	354.84	1.01
after five cycles	91.59	0.37	301.43	0.63	223.79	0.99

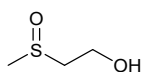
## 4.2 NMR data of sulfoxides

2-(ethylsulfinyl)ethan-1-ol (1b)<sup>3</sup>



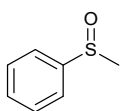
<sup>1</sup>H NMR (400 MHz, H<sub>2</sub>O+D<sub>2</sub>O)  $\delta$  4.06 – 3.95 (m, 2H), 3.13 – 2.83 (m, 4H), 1.31 (t, J = 7.5 Hz, 3H). <sup>13</sup>C NMR (101 MHz, H<sub>2</sub>O+D<sub>2</sub>O)  $\delta$  54.8, 52.9, 44.7, 6.0.

2-(methylsulfinyl)ethan-1-ol (2b)<sup>4</sup>



<sup>1</sup>H NMR (400 MHz, H<sub>2</sub>O+D<sub>2</sub>O)  $\delta$  3.83 – 3.77 (m, 2H), 2.97 – 2.81 (m, 2H), 2.55 (s, 3H). <sup>13</sup>C NMR (101 MHz, H<sub>2</sub>O+D<sub>2</sub>O)  $\delta$  55.7, 54.8, 37.1.

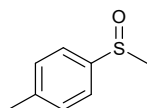
(methylsulfinyl)benzene (3b)<sup>4</sup>



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.68 – 7.63 (m, 2H), 7.57 – 7.48 (m, 3H), 2.73 (s, 3H).

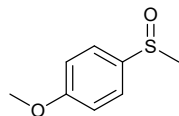
<sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  145.8, 131.1, 129.4, 123.6, 44.0.

1-methyl-4-(methylsulfinyl)benzene (4b)<sup>4</sup>



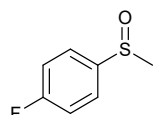
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.56 – 7.52 (m, 2H), 7.33 (d, J = 8.0 Hz, 2H), 2.71 (s, 3H), 2.42 (s, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>)  $\delta$  142.6, 141.5, 130.1, 123.6, 44.0, 21.4.

1-methoxy-4-(methylsulfinyl)benzene (5b)<sup>4</sup>



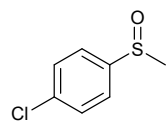
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.63 – 7.57 (m, 2H), 7.07 – 7.00 (m, 2H), 3.86 (s, 3H), 2.70 (s, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 162.0, 136.6, 125.5, 114.9, 55.5, 44.0.

1-fluoro-4-(methylsulfinyl)benzene (6b)<sup>5</sup>



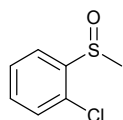
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.70 – 7.63 (m, 2H), 7.27 – 7.20 (m, 2H), 2.72 (s, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 164.3 (d, J = 251.3 Hz), 141.1, 125.9, 125.8, 116.8, 116.6, 44.2 (d, J = 1.4 Hz).

1-chloro-4-(methylsulfinyl)benzene (7b)<sup>4</sup>



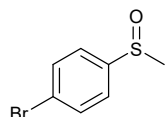
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.62 – 7.57 (m, 2H), 7.54 – 7.49 (m, 2H), 2.72 (s, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 144.3, 137.3, 129.7, 125.0, 44.1

1-chloro-2-(methylsulfinyl)benzene (8b)<sup>4</sup>



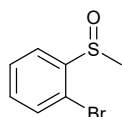
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.96 (dd, J = 7.8, 1.6 Hz, 1H), 7.54 (td, J = 7.5, 1.3 Hz, 1H), 7.48 – 7.38 (m, 2H), 2.83 (s, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 143.7, 132.0, 129.8, 129.8, 128.2, 125.4, 41.7.

1-bromo-4-(methylsulfinyl)benzene (9b)<sup>5</sup>



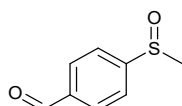
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.70 – 7.65 (m, 2H), 7.56 – 7.50 (m, 2H), 2.72 (s, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 144.9, 132.6, 125.5, 125.2, 44.0.

1-bromo-2-(methylsulfinyl)benzene (10b)<sup>5</sup>



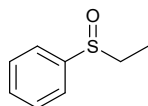
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.95 (dd, J = 7.8, 1.7 Hz, 1H), 7.63 – 7.54 (m, 2H), 7.38 (td, J = 7.8, 1.7 Hz, 1H), 2.83 (s, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 145.4, 133.0, 132.3, 128.8, 125.7, 118.5, 41.9.

4-(methylsulfinyl)benzaldehyde (11b)<sup>5</sup>



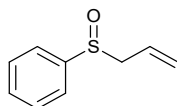
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 10.10 (s, 1H), 8.09 – 8.03 (m, 2H), 7.87 – 7.81 (m, 2H), 2.79 (s, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 191.1, 152.5, 138.2, 130.4, 124.2, 43.8.

(ethylsulfinyl)benzene (12b)<sup>4</sup>



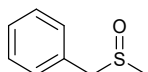
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.64 – 7.59 (m, 2H), 7.51 (qd, J = 8.3, 4.8 Hz, 3H), 2.96 – 2.72 (m, 2H), 1.20 (t, J = 7.4 Hz, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 143.4, 131.0, 129.2, 124.2, 50.4, 6.0.

(allylsulfinyl)benzene (13b)<sup>5</sup>



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.60 (dt, J = 6.7, 2.6 Hz, 2H), 7.56 – 7.47 (m, 3H), 5.65 (ddt, J = 17.5, 10.2, 7.5 Hz, 1H), 5.34 (dd, J = 10.2, 1.4 Hz, 1H), 5.20 (dt, J = 17.0, 1.3 Hz, 1H), 3.61 – 3.47 (m, 2H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 143.0, 131.1, 129.1, 125.3, 124.4, 123.9, 60.9.

((methylsulfinyl)methyl)benzene (14b)<sup>4</sup>



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.35 – 7.18 (m, 5H), 3.99 (d, J = 12.8 Hz, 1H), 3.85 (d, J = 12.8 Hz, 1H), 2.38 (s, 3H). <sup>13</sup>C NMR (101 MHz, CDCl<sub>3</sub>) δ 130.0, 129.7, 129.0, 128.5, 60.4, 37.3.

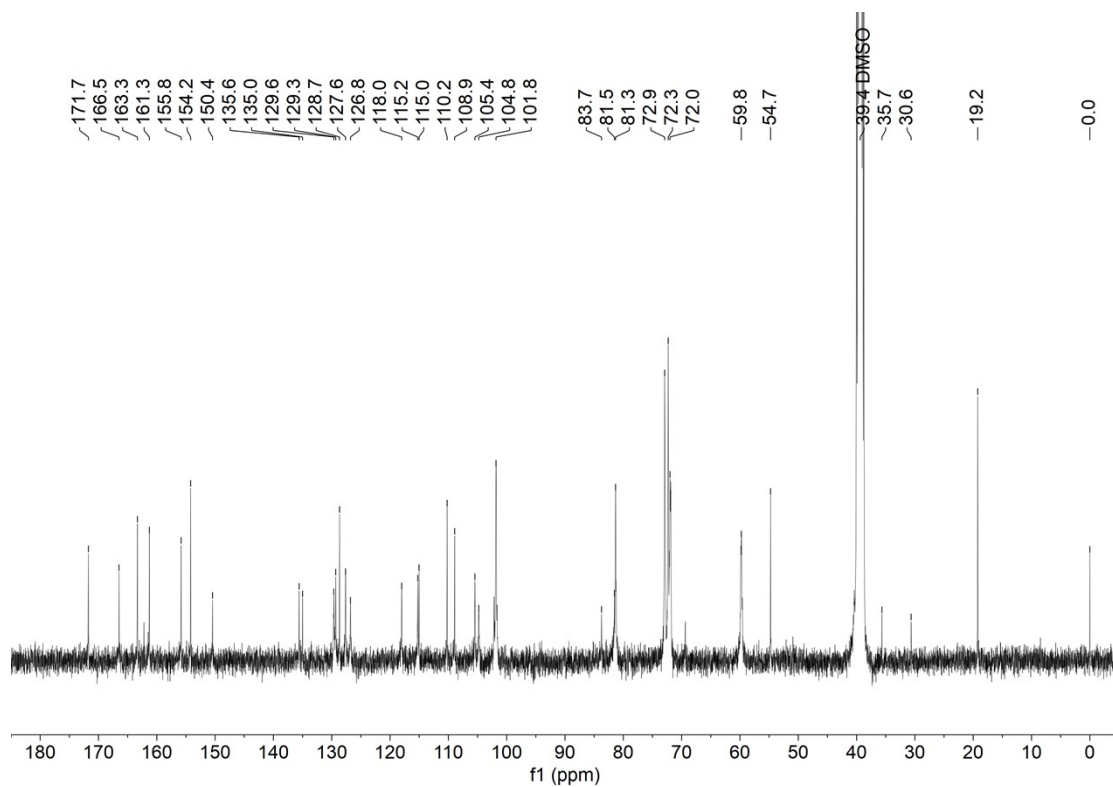
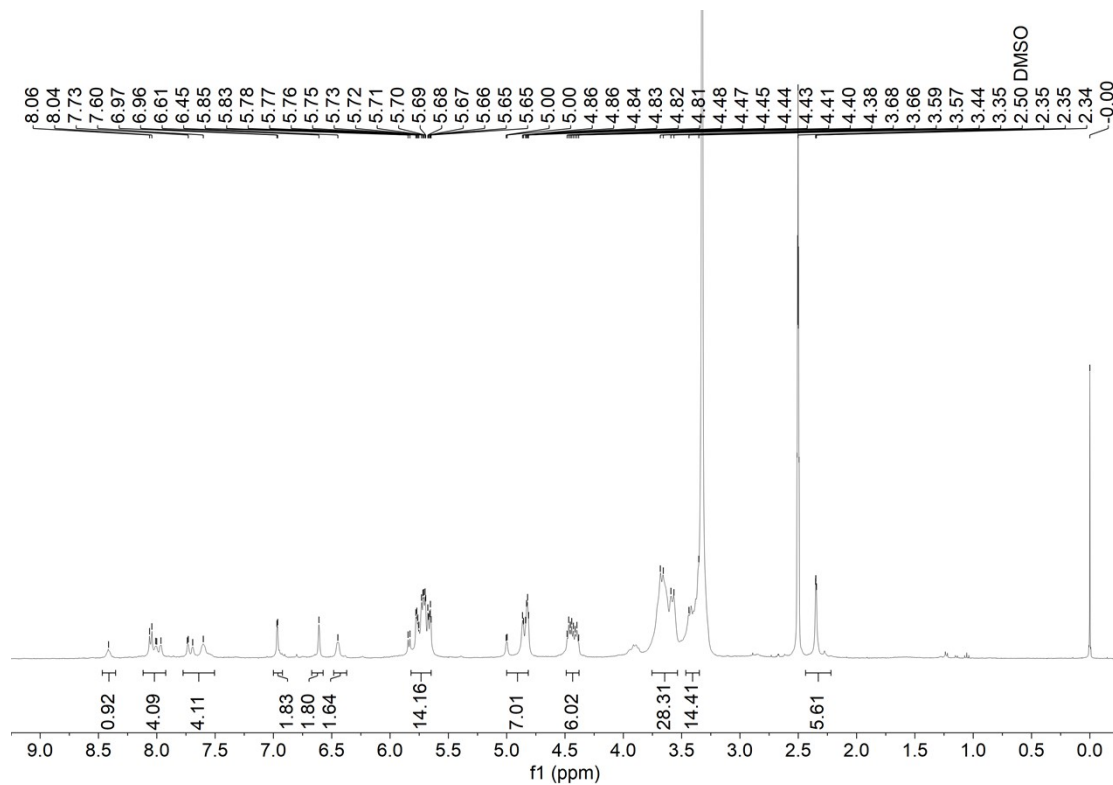
## 5. References

1. Z. Liu, F. Song, B. Song, L. Jiao, J. An, J. Yuan and X. Peng, *Sens. Actuator B-Chem.*, 2018, **262**, 958-965.
2. M. Jereb, *Green Chem.*, 2012, **14**, 3047-3052.
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4. C. Yang, Q. Jin, H. Zhang, J. Liao, J. Zhu, B. Yu and J. Deng, *Green Chem.*, 2009, **11**, 1401-1405.
5. N. Amri and T. Wirth, *J Org. Chem.*, 2021, **86**, 15961-15972.

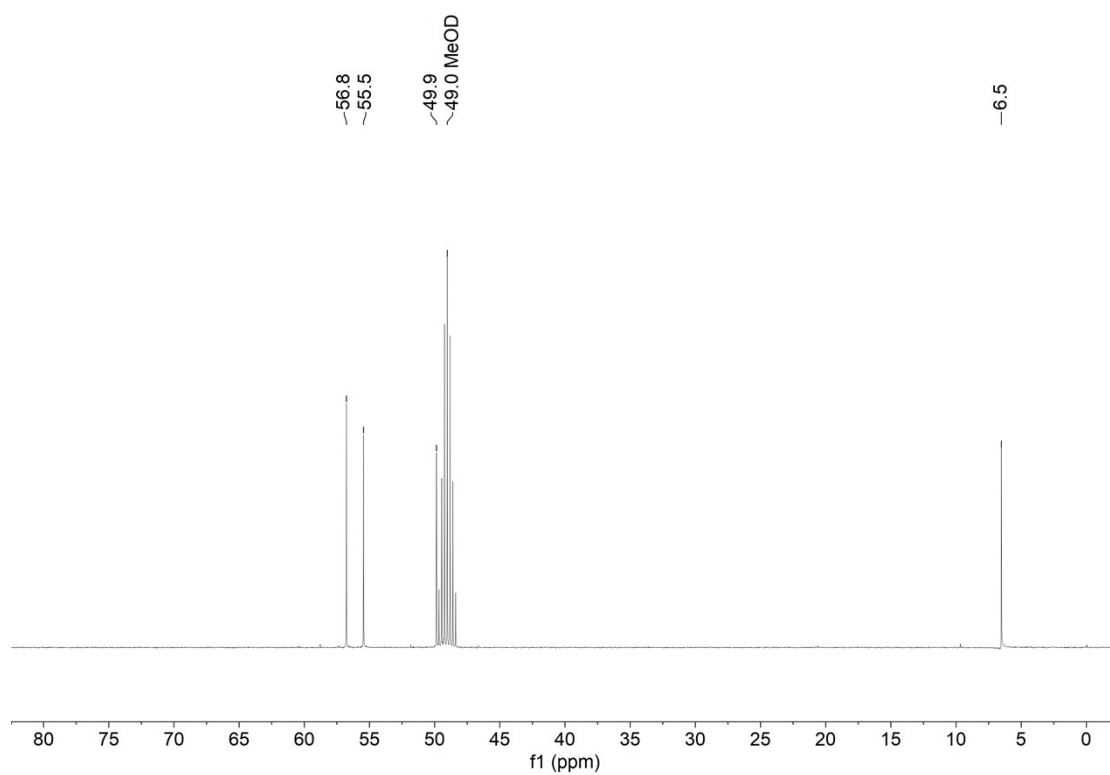
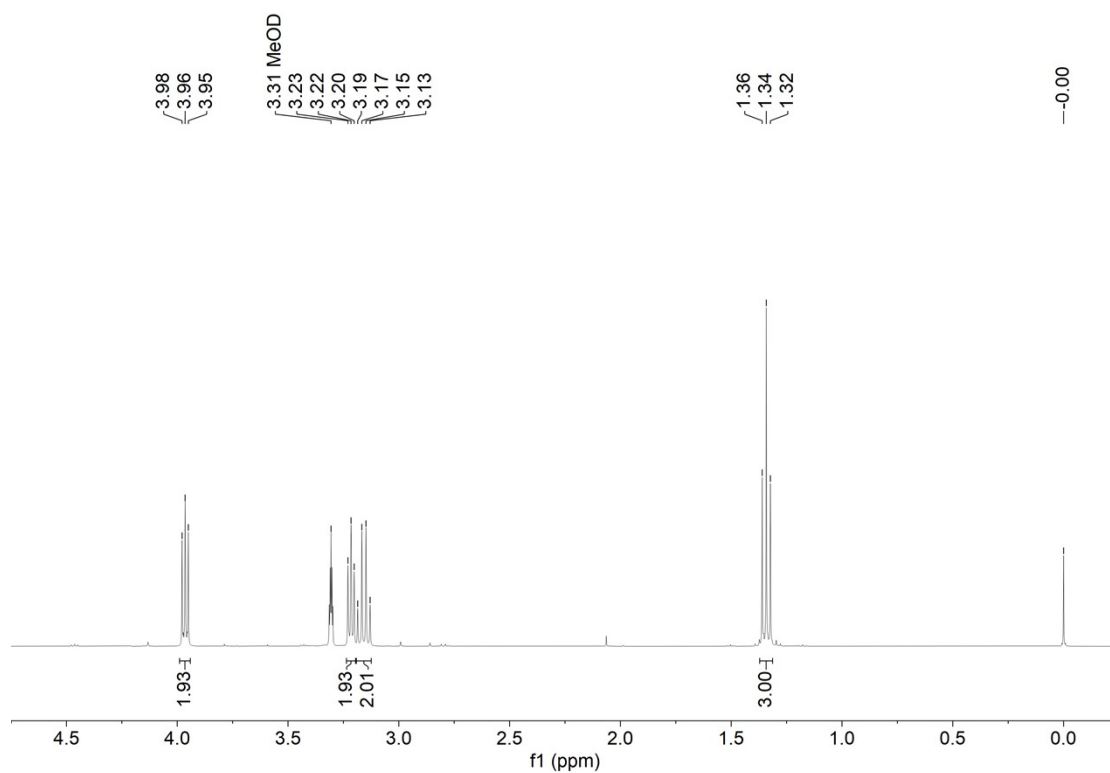


## Appendix

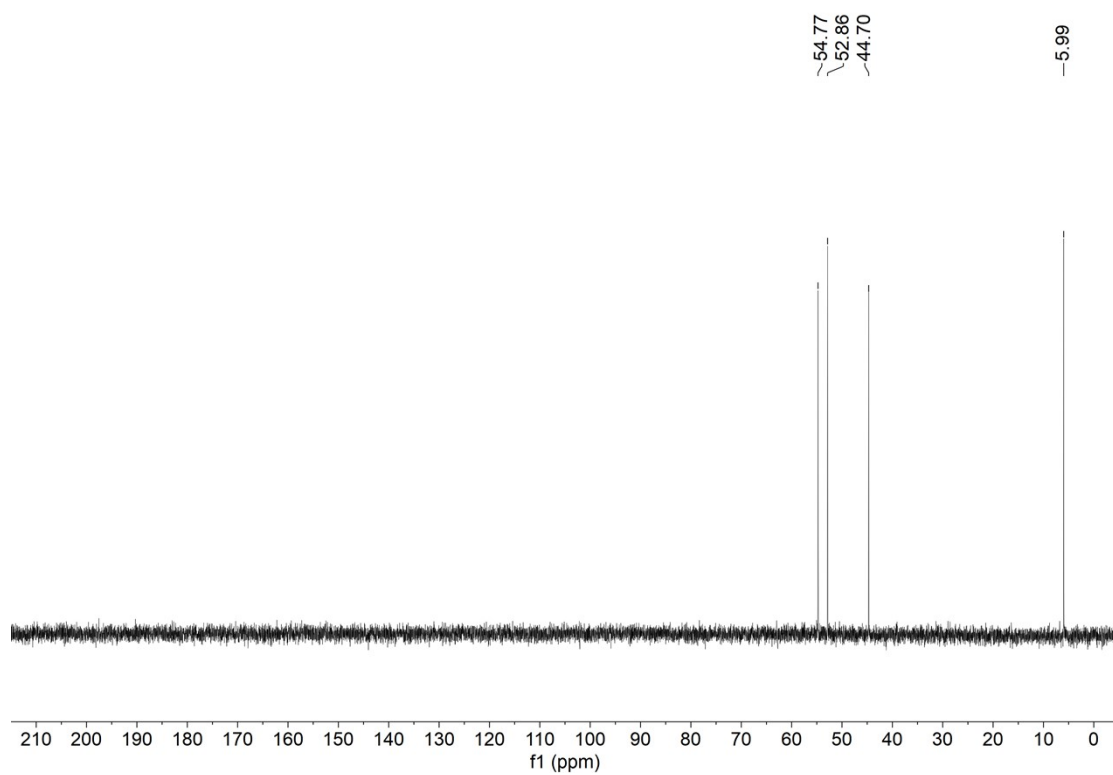
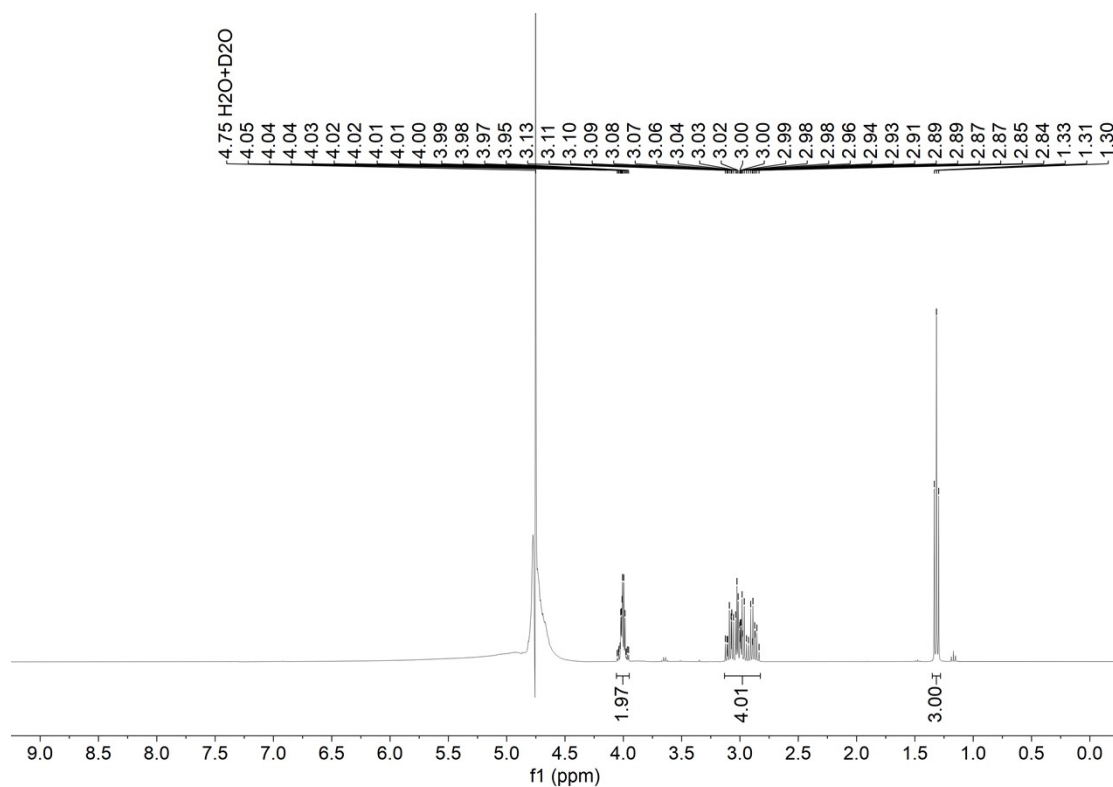
$^1\text{H}$  NMR (400 MHz,  $\text{DMSO-}d_6$ ) and  $^{13}\text{C}$  NMR (101 MHz,  $\text{DMSO-}d_6$ ) spectra of FL-CD.



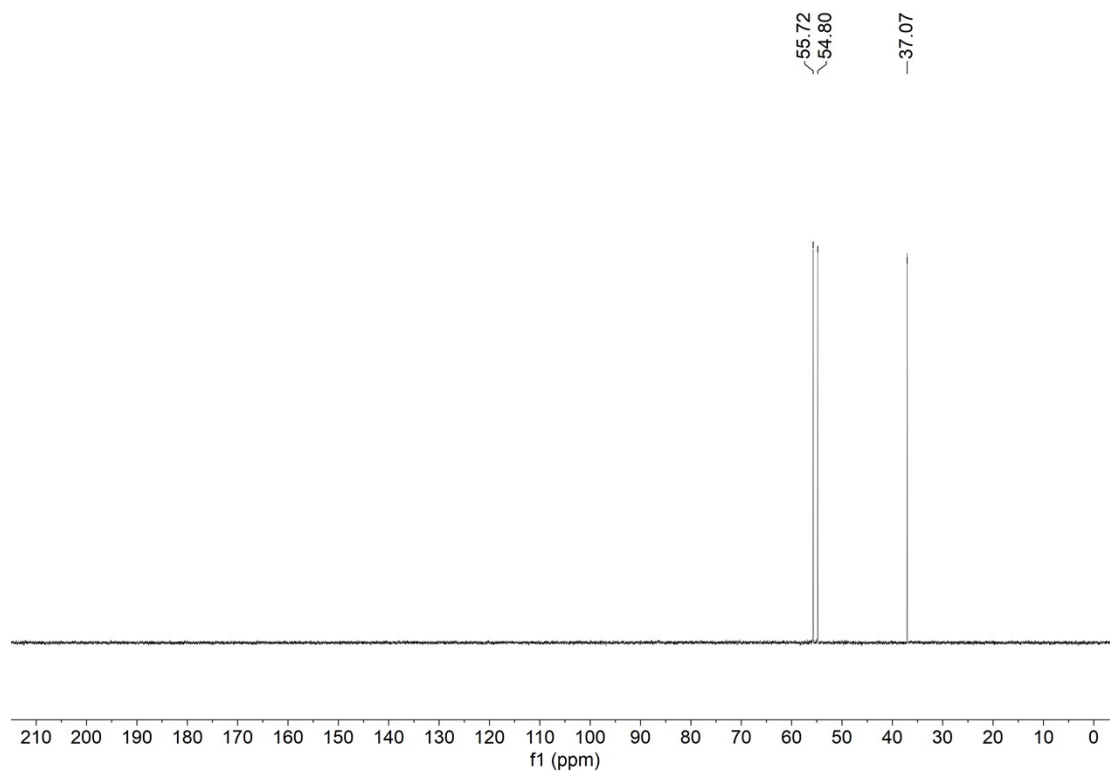
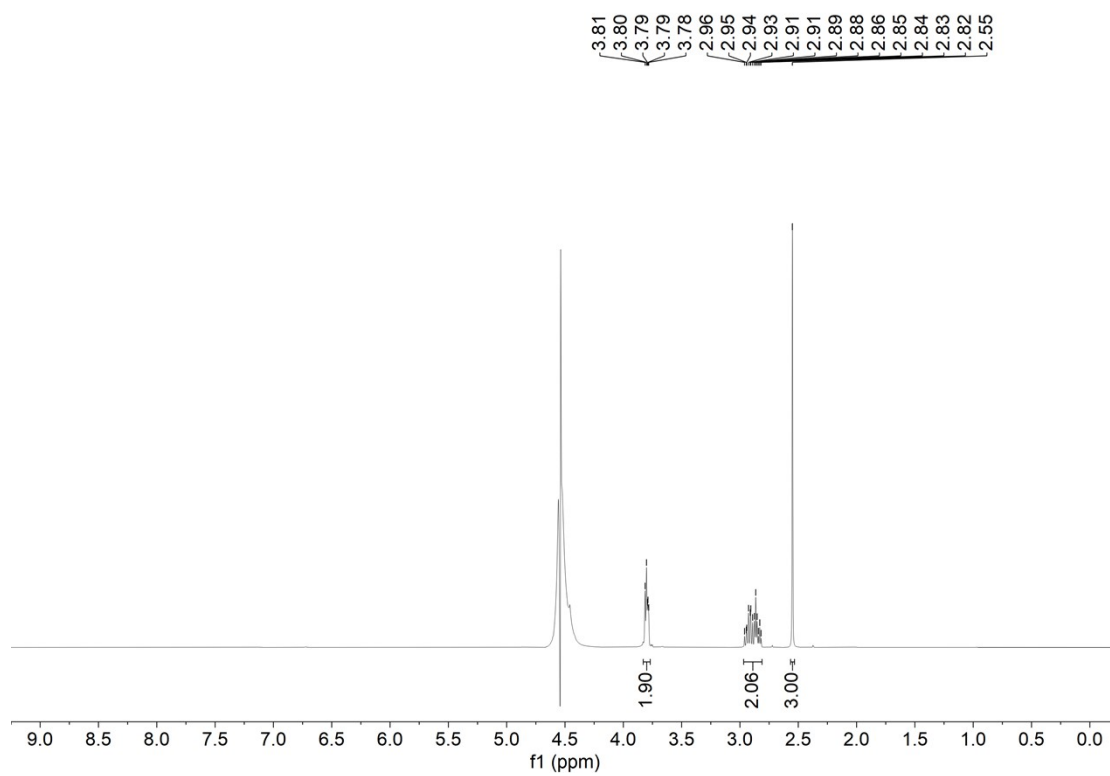
$^1\text{H}$  NMR (400 MHz, MeOD) and  $^{13}\text{C}$  NMR (101 MHz, MeOD) spectra of 2-ethylsulfonylethanol.



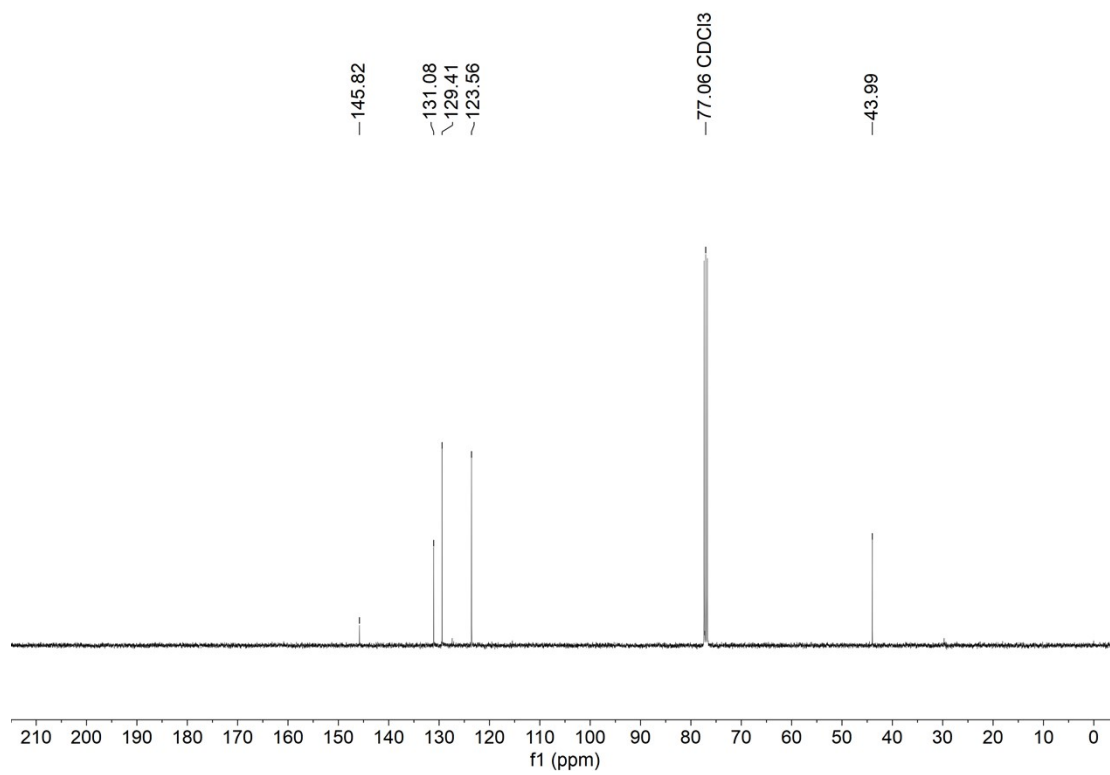
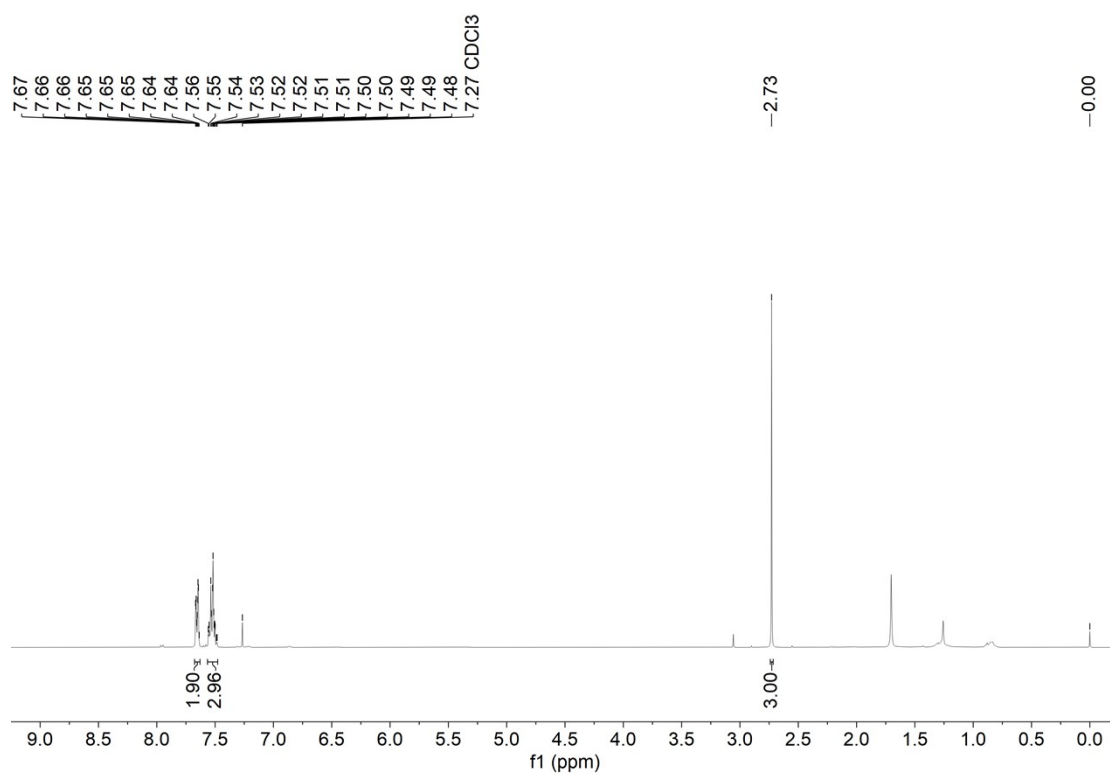
$^1\text{H}$  NMR (400 MHz,  $\text{H}_2\text{O}+\text{D}_2\text{O}$ ) and  $^{13}\text{C}$  NMR (101 MHz,  $\text{H}_2\text{O}+\text{D}_2\text{O}$ ) spectra of 1b.



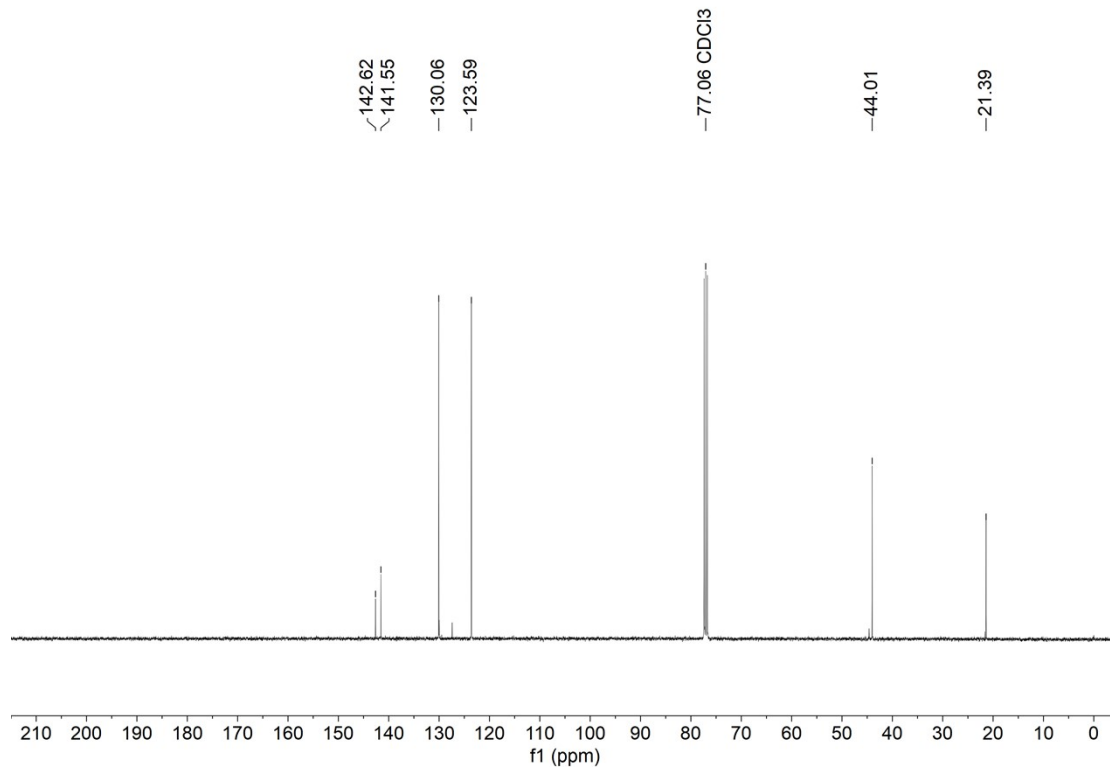
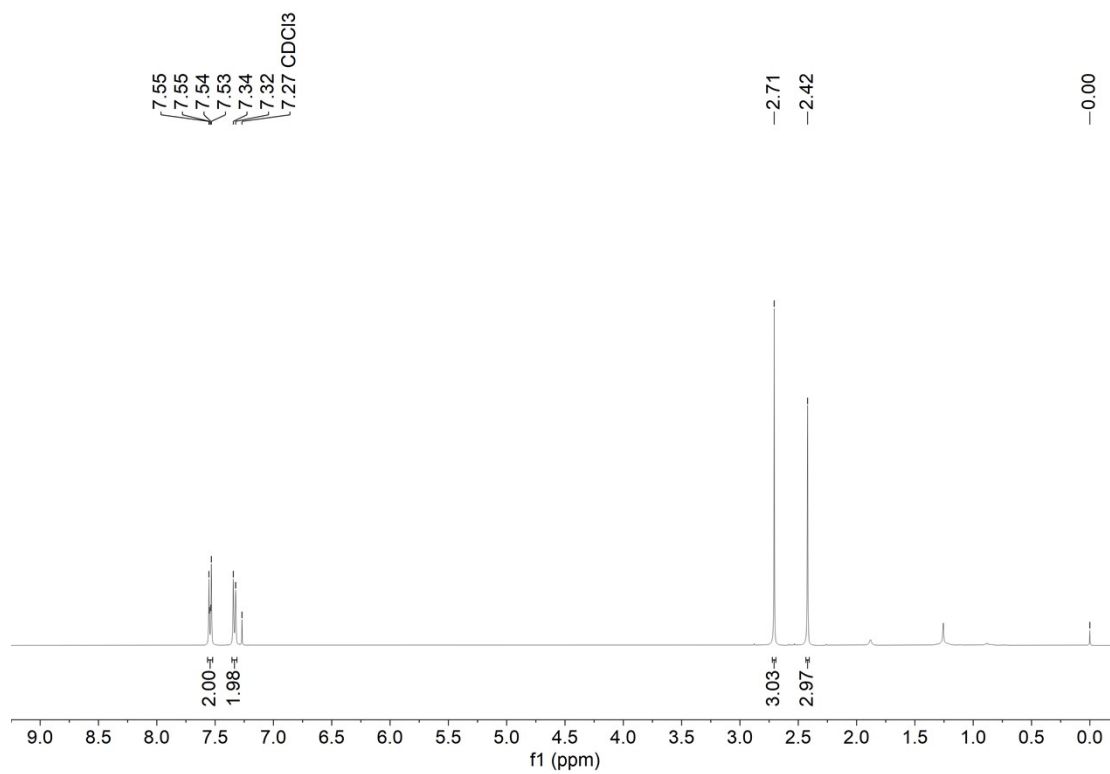
$^1\text{H}$  NMR (400 MHz,  $\text{H}_2\text{O}+\text{D}_2\text{O}$ ) and  $^{13}\text{C}$  NMR (101 MHz,  $\text{H}_2\text{O}+\text{D}_2\text{O}$ ) spectra of 2b.



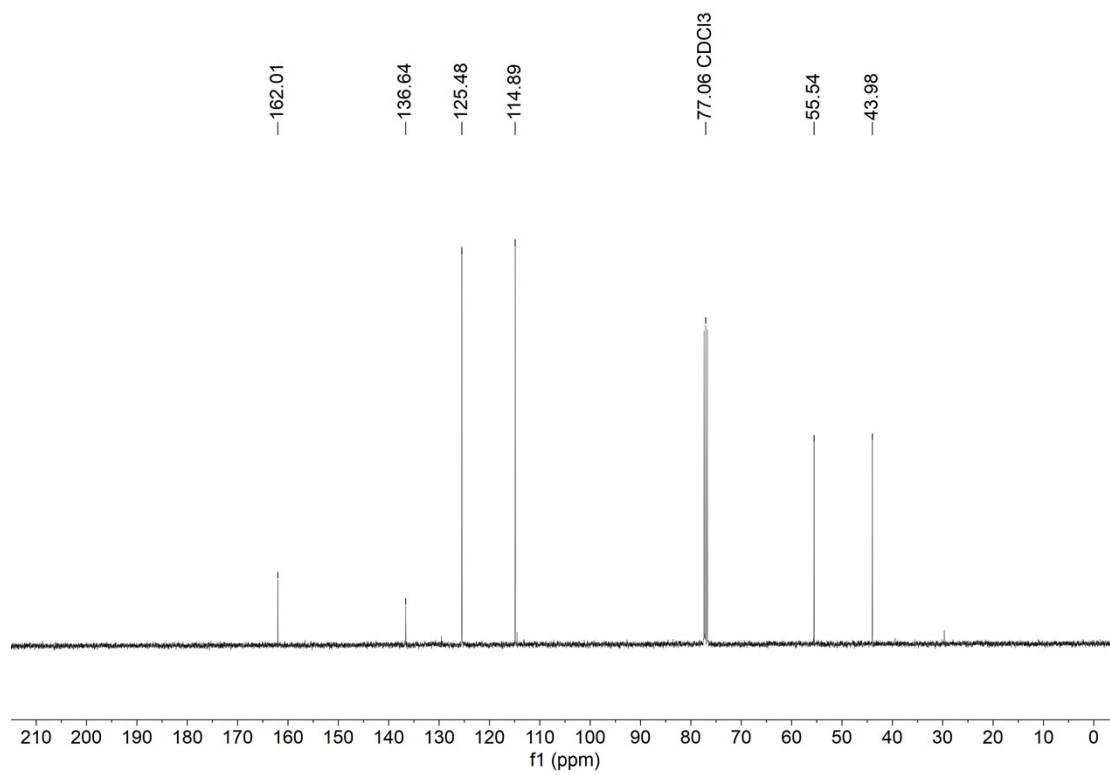
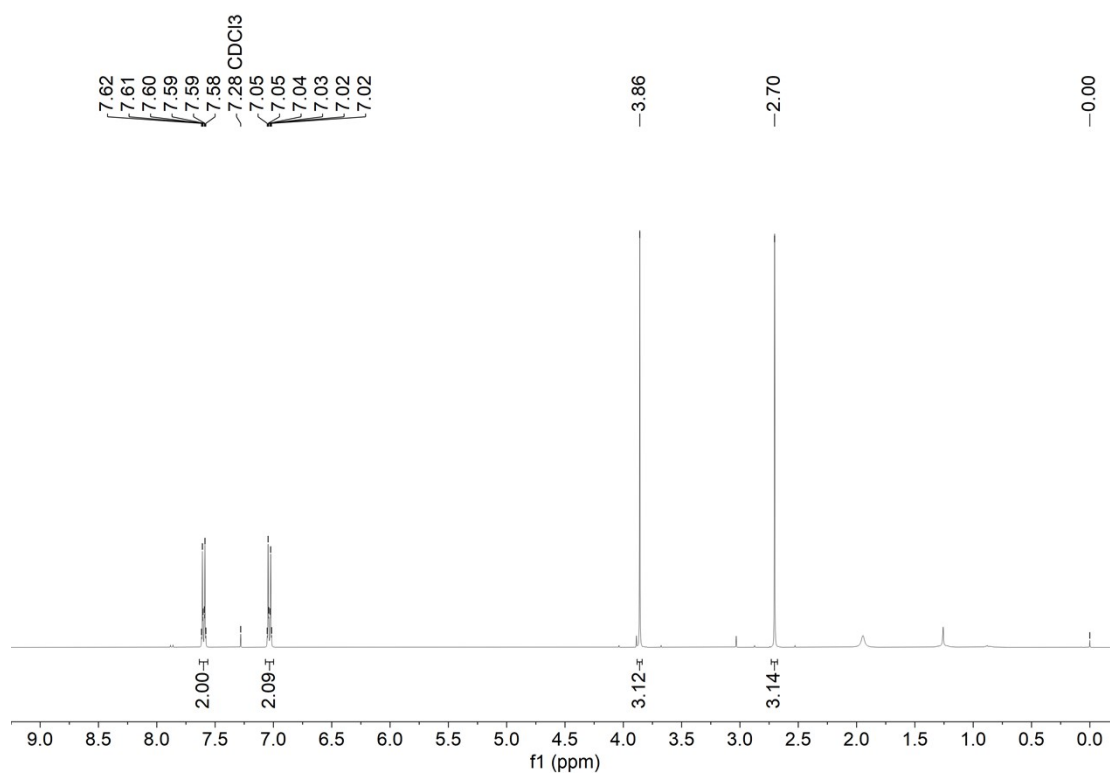
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectra of 3b.



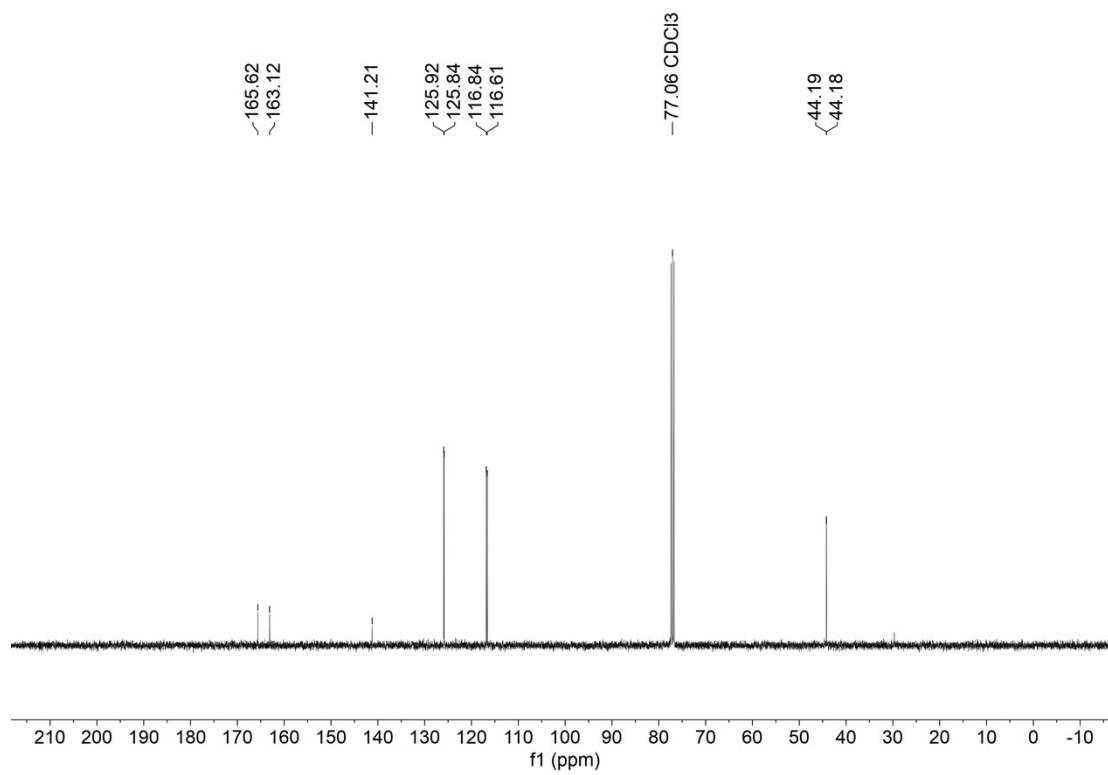
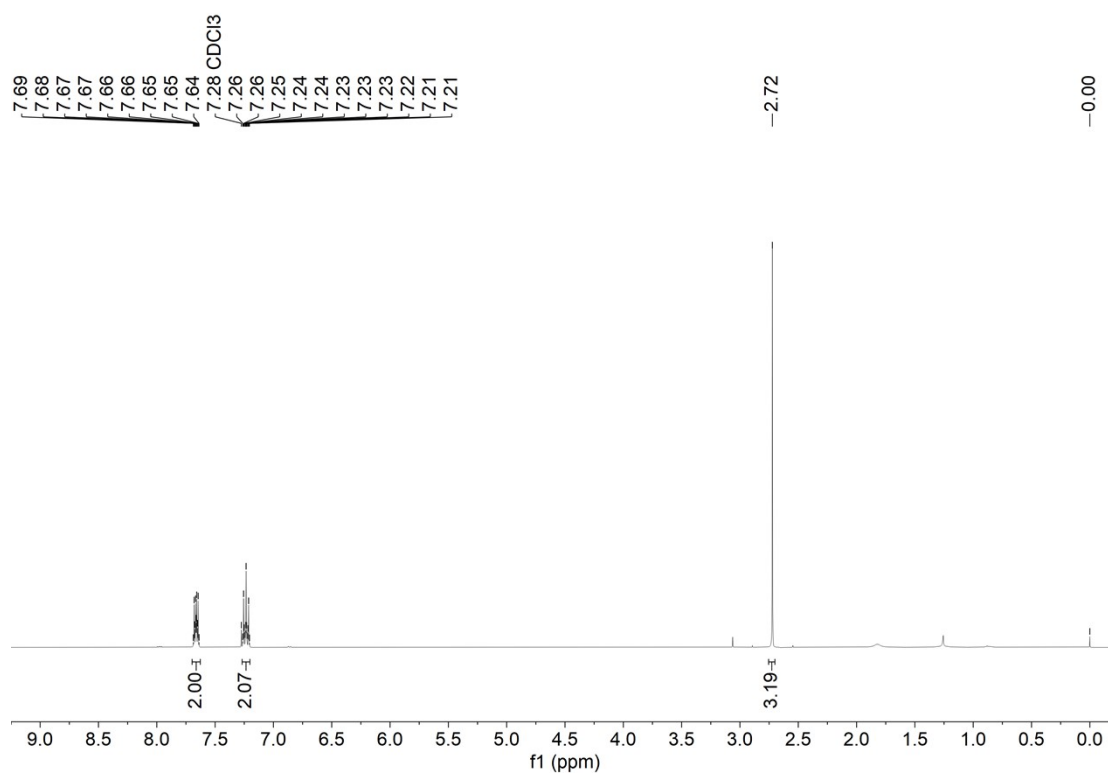
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectra of 4b.



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectra of 5b.

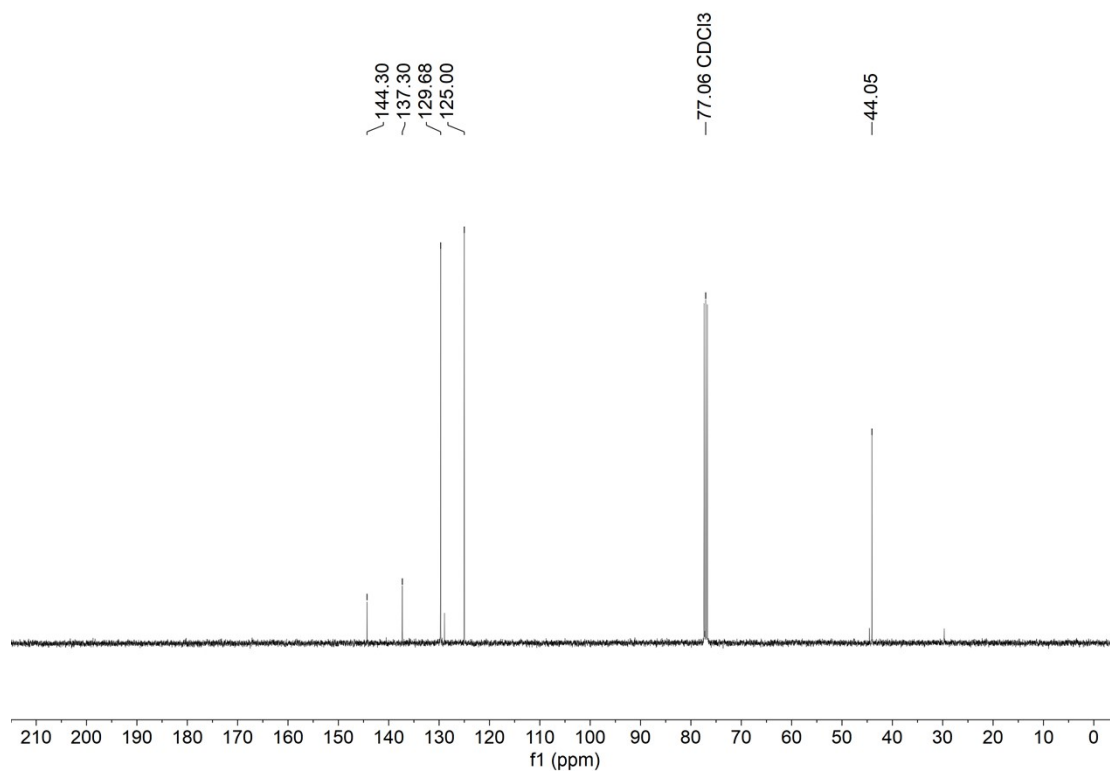
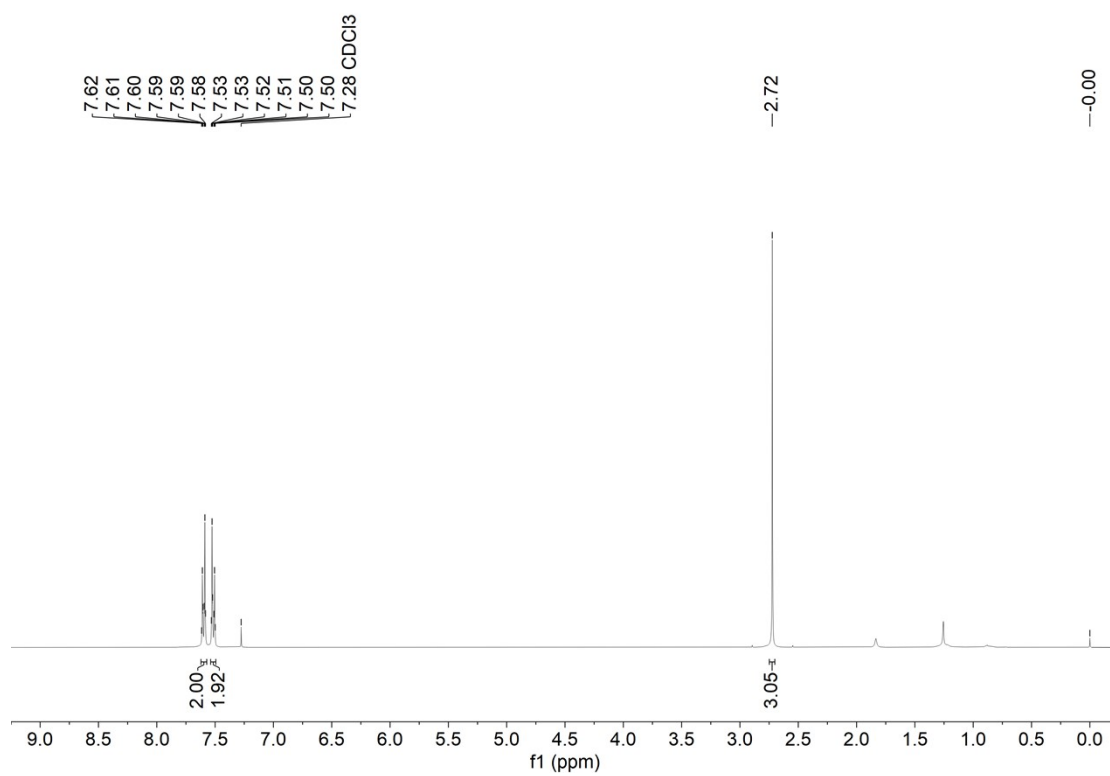


$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectra of 6b.

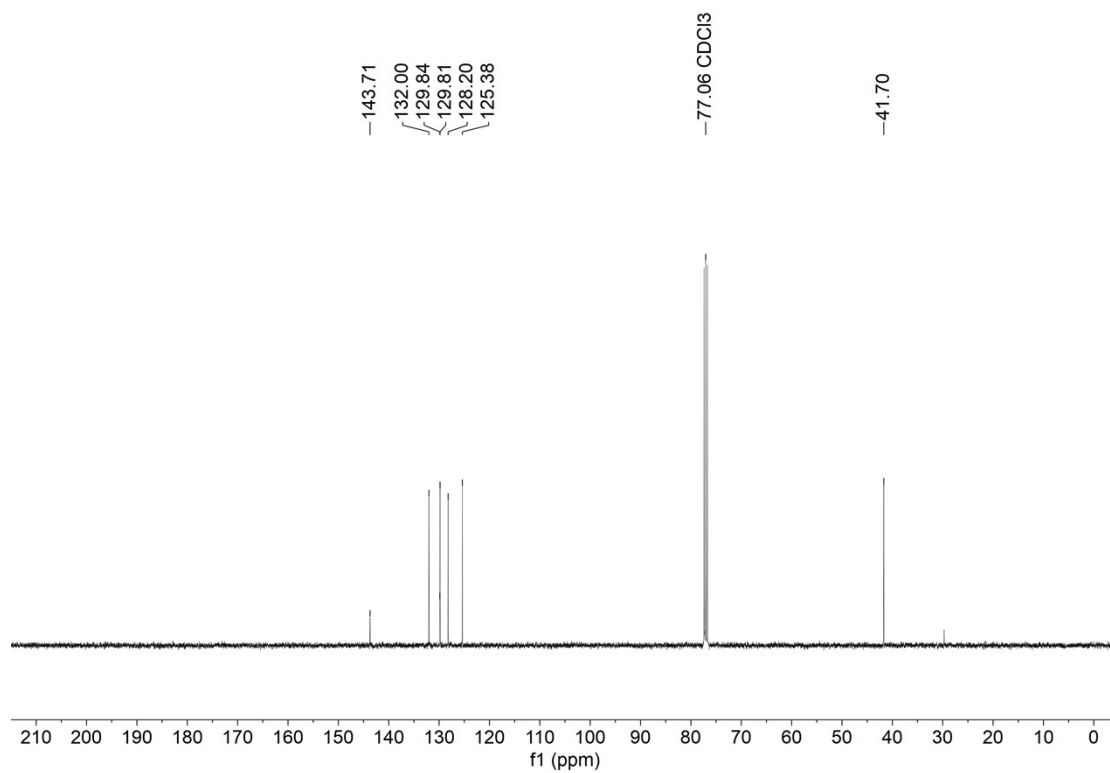
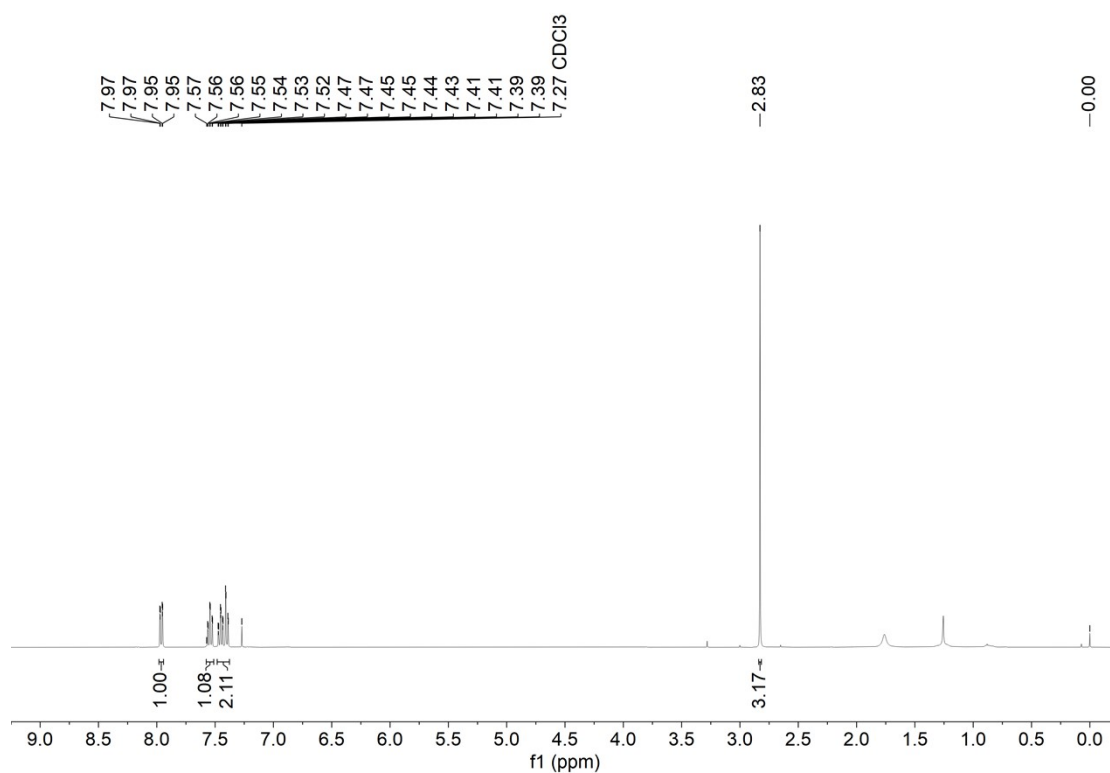




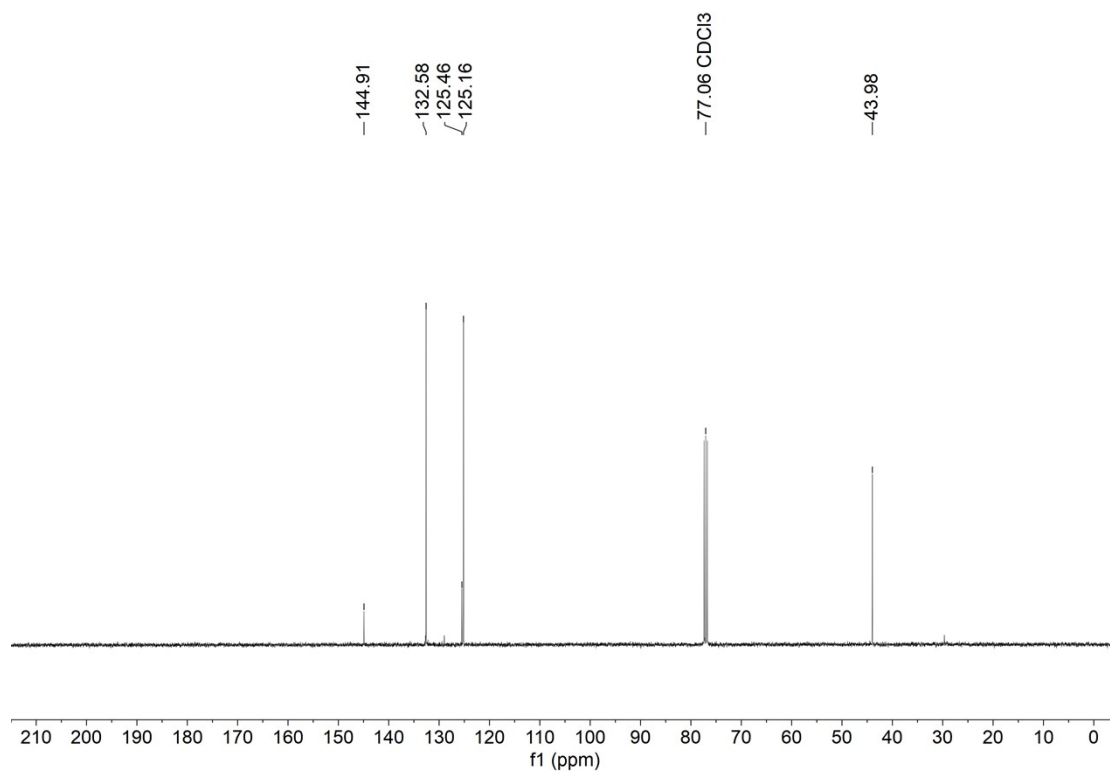
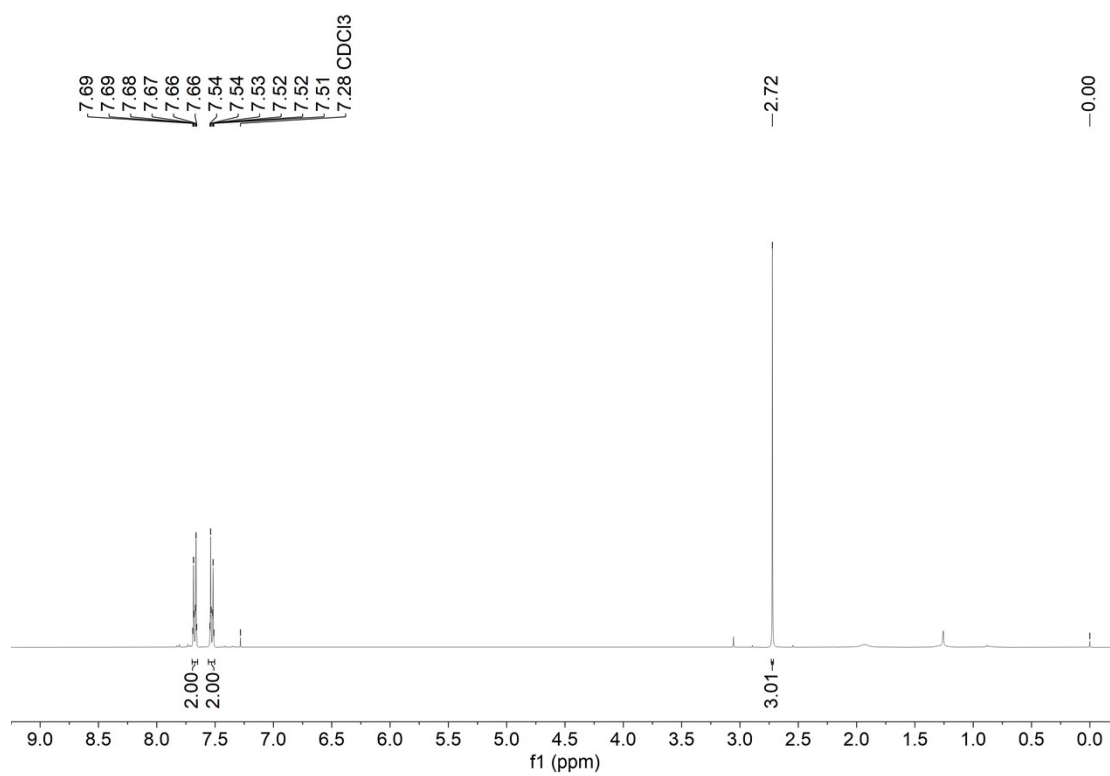
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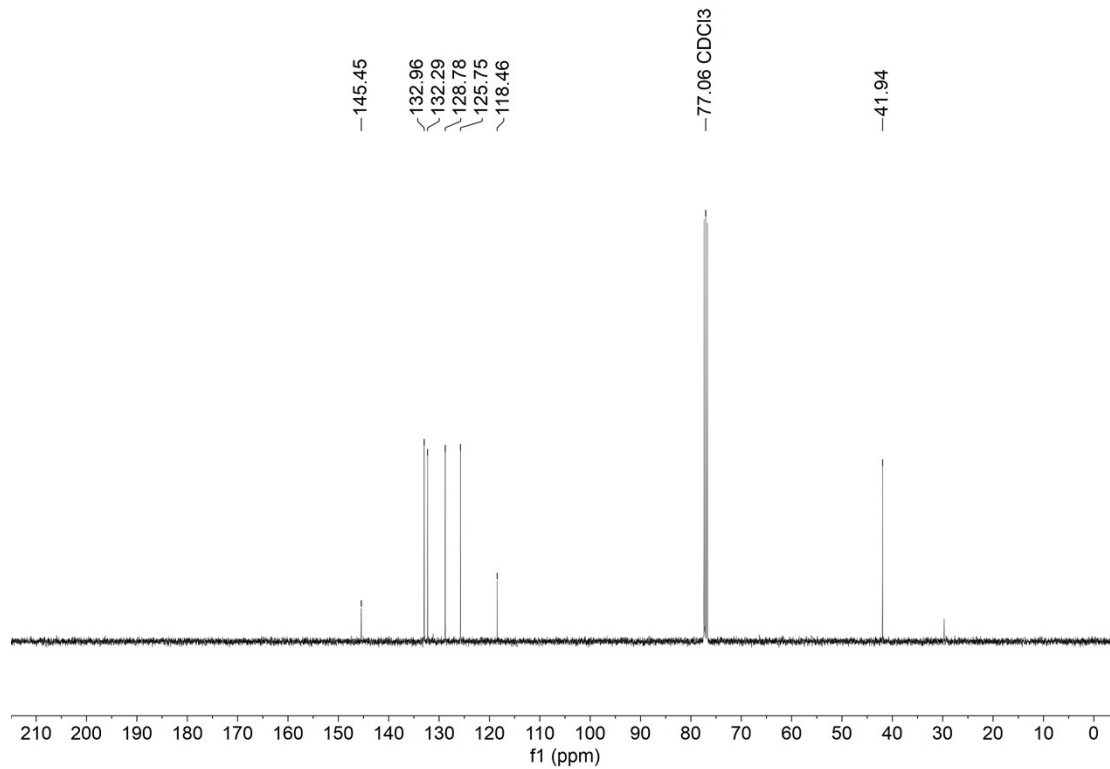
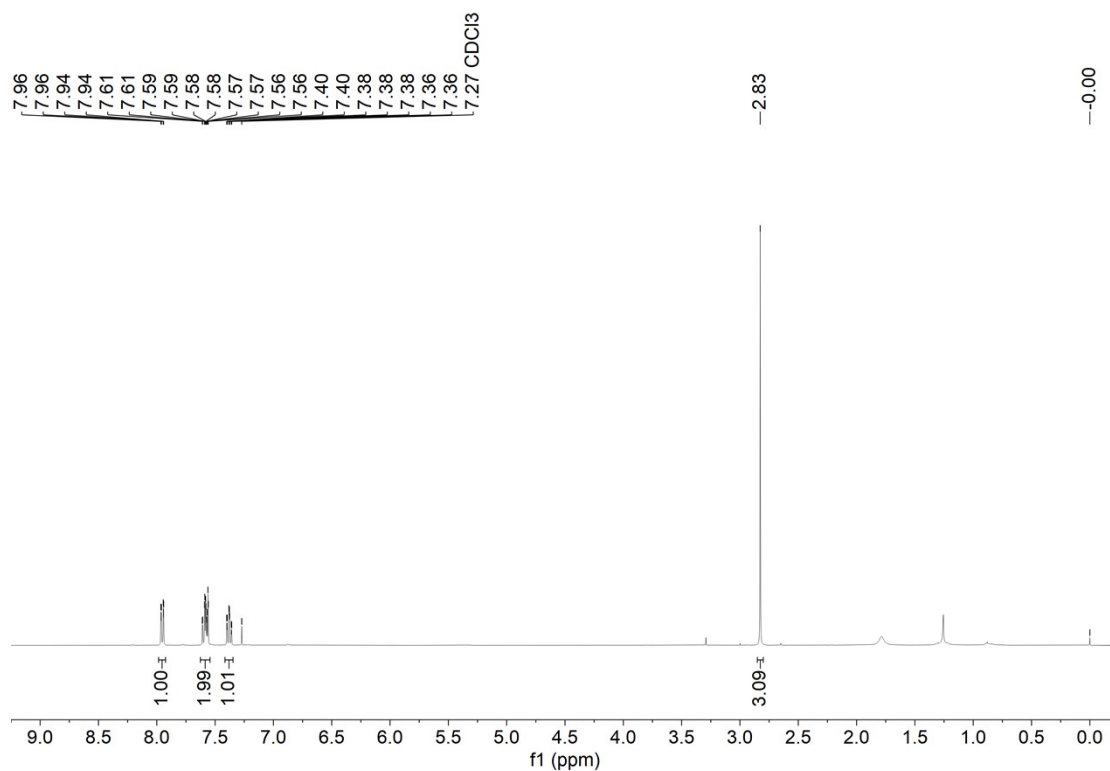
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectra of 8b.



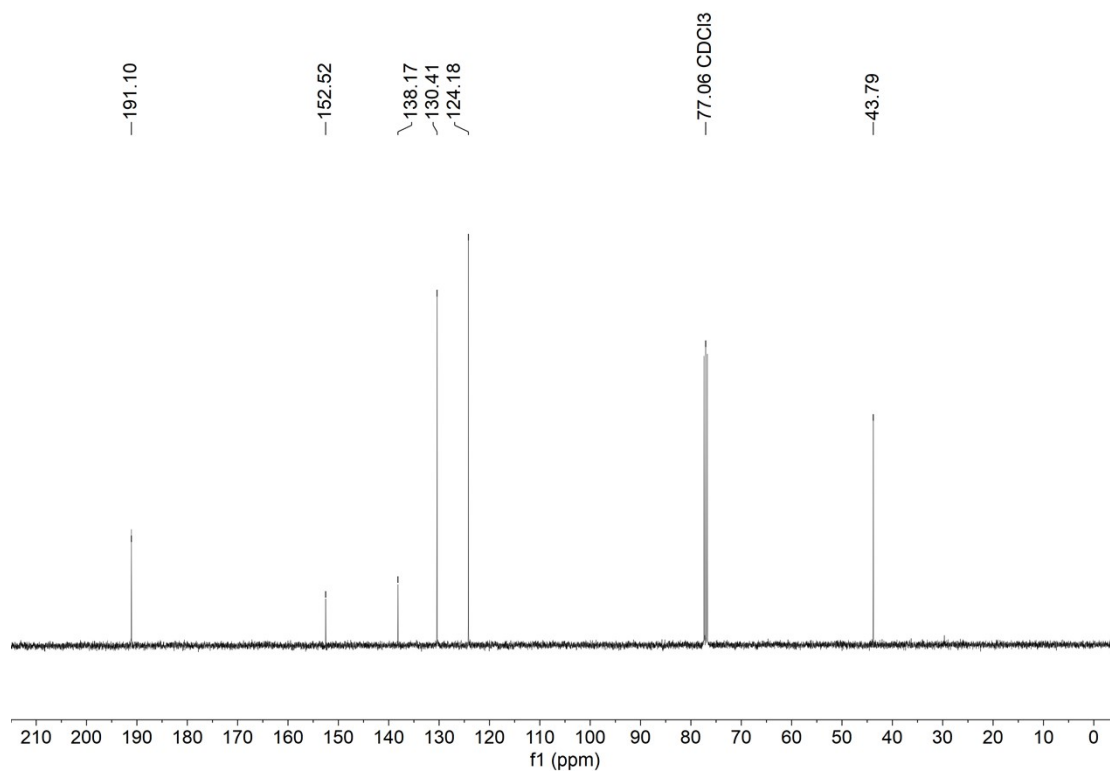
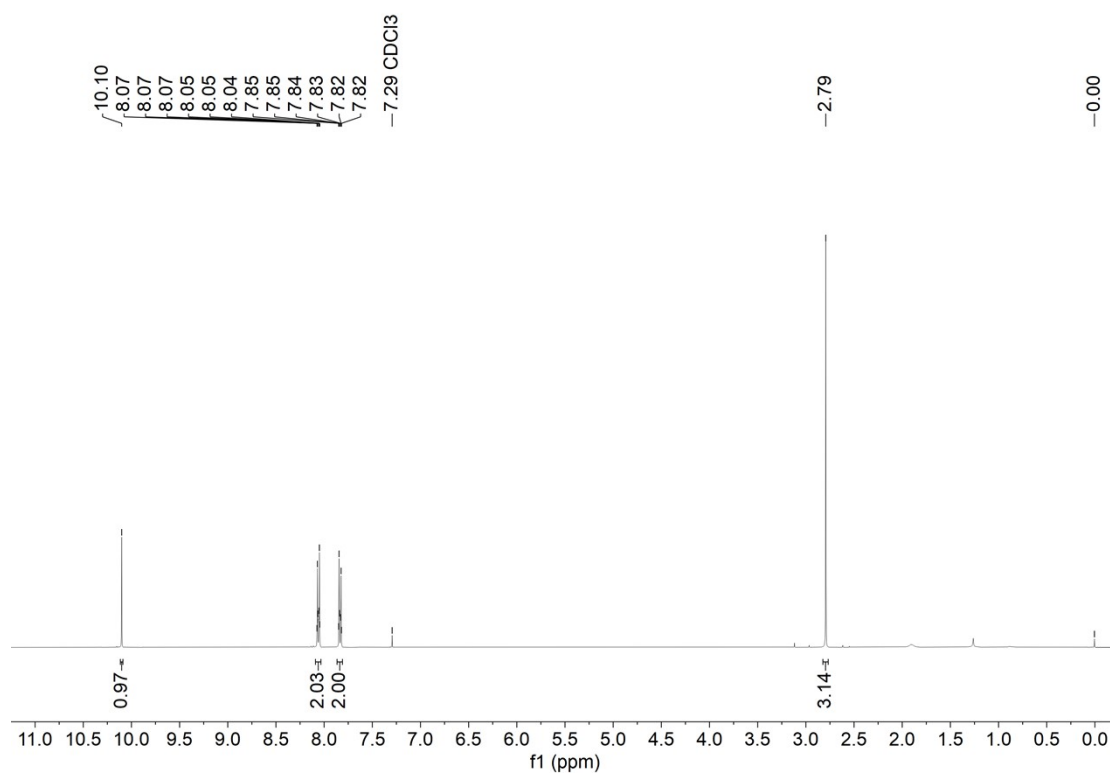
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectra of 9b.



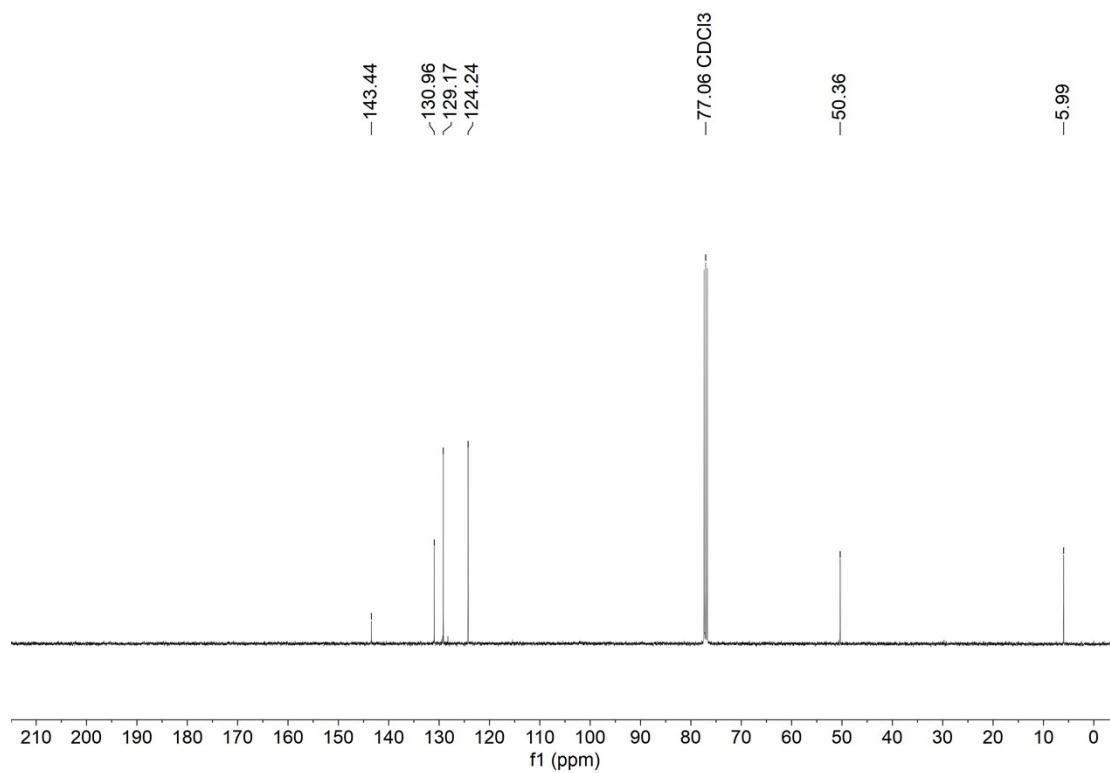
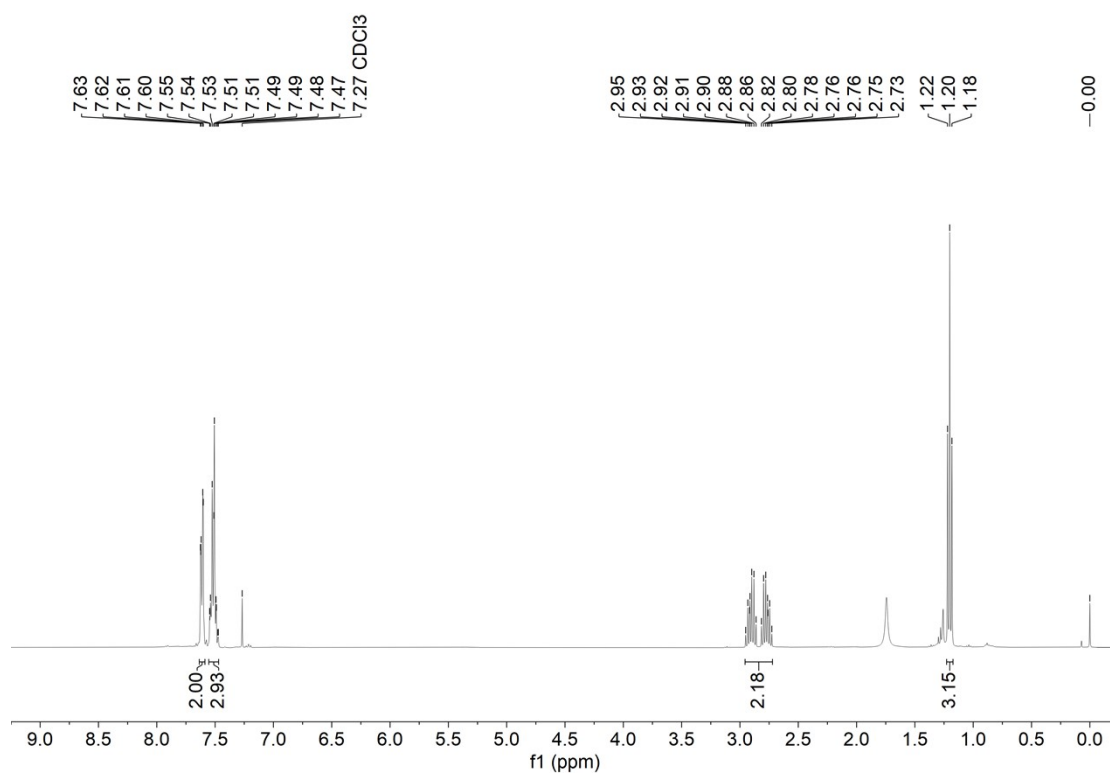
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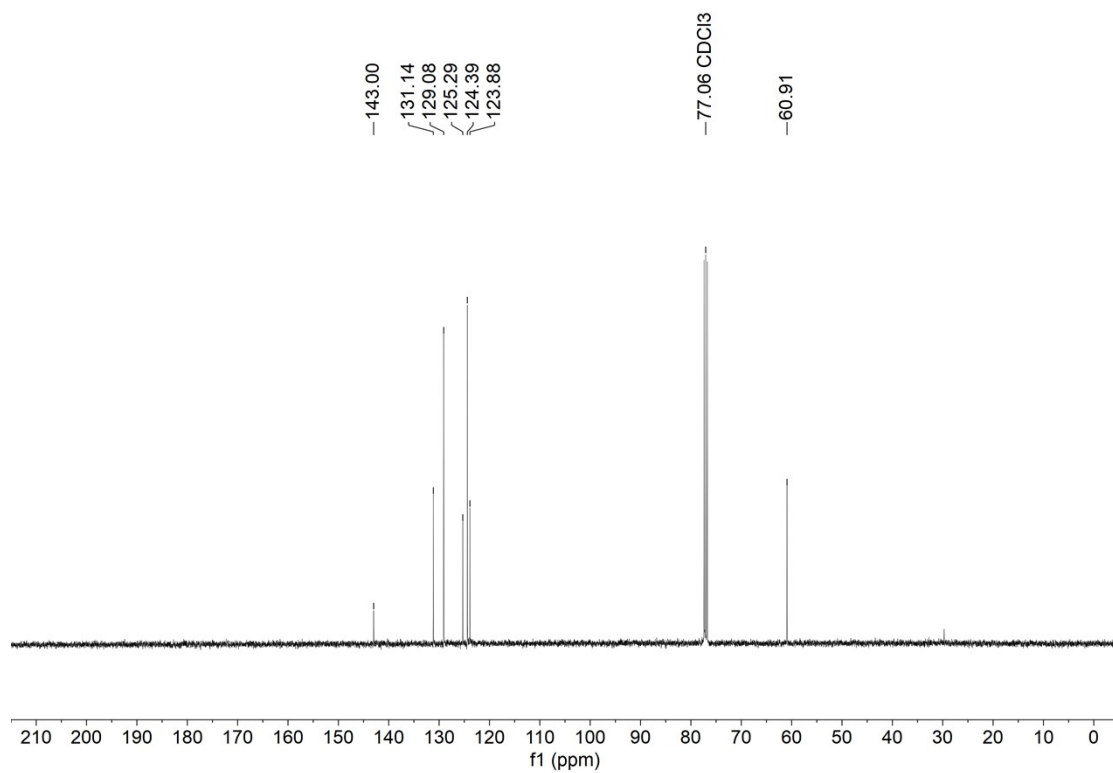
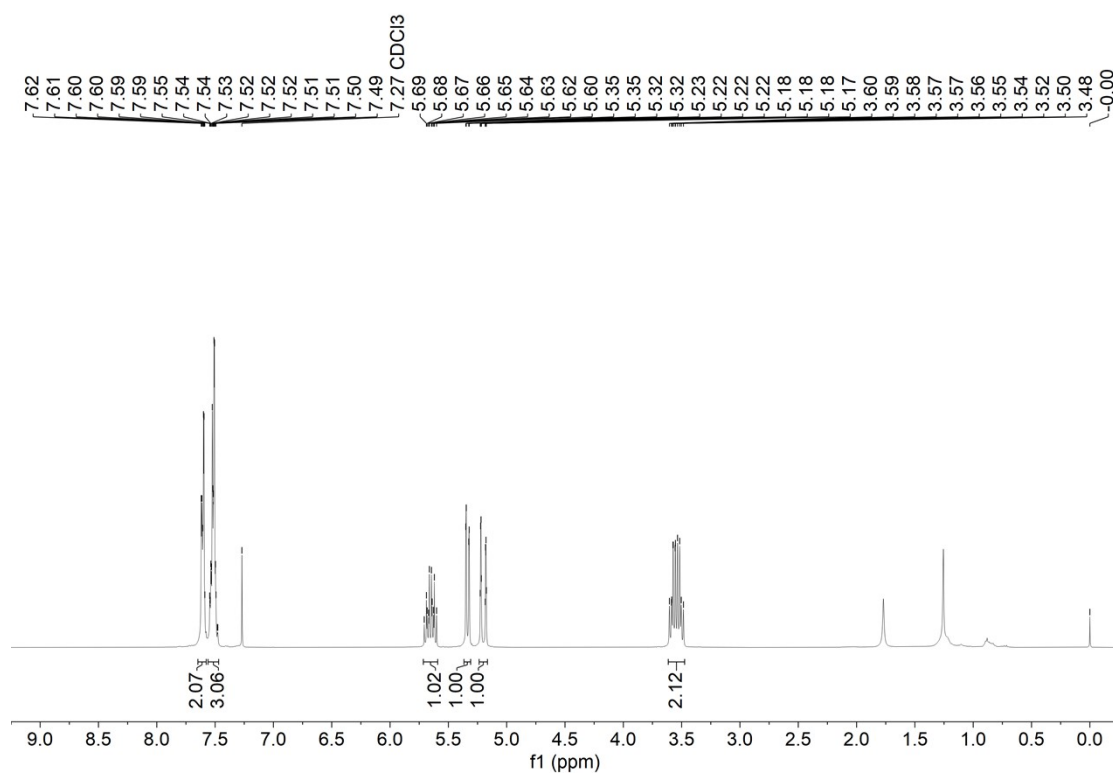
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectra of 11b.



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectra of 12b.



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectra of 13b.



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) and  $^{13}\text{C}$  NMR (101 MHz,  $\text{CDCl}_3$ ) spectra of 14b.

