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Efficient pseudo-five-component coupling-Fiesselmann synthesis of luminescent oligothiophenes and their modification

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1 GENERAL CONSIDERATIONS

All cross-coupling reactions were carried out in oven-dried *Schlenk* glassware using septa and syringes under nitrogen or argon atmosphere. THF was dried using MBraun system MB-SPS-800, and triethylamine was refluxed under argon over ketyl sodium, distilled and stored in a *Schlenk* flask over potassium hydroxide pellets under argon atmosphere.

Commercial grade reagents were used as supplied without further purification and were purchased from Sigma-Aldrich Chemie GmbH, Fluka AG, ABCR GmBH & Co. KG, Alfa Aesar GmbH & Co. KG and Riedel-de Haën. Some of the alkynes **4** and the catalysts were prepared according to literature.

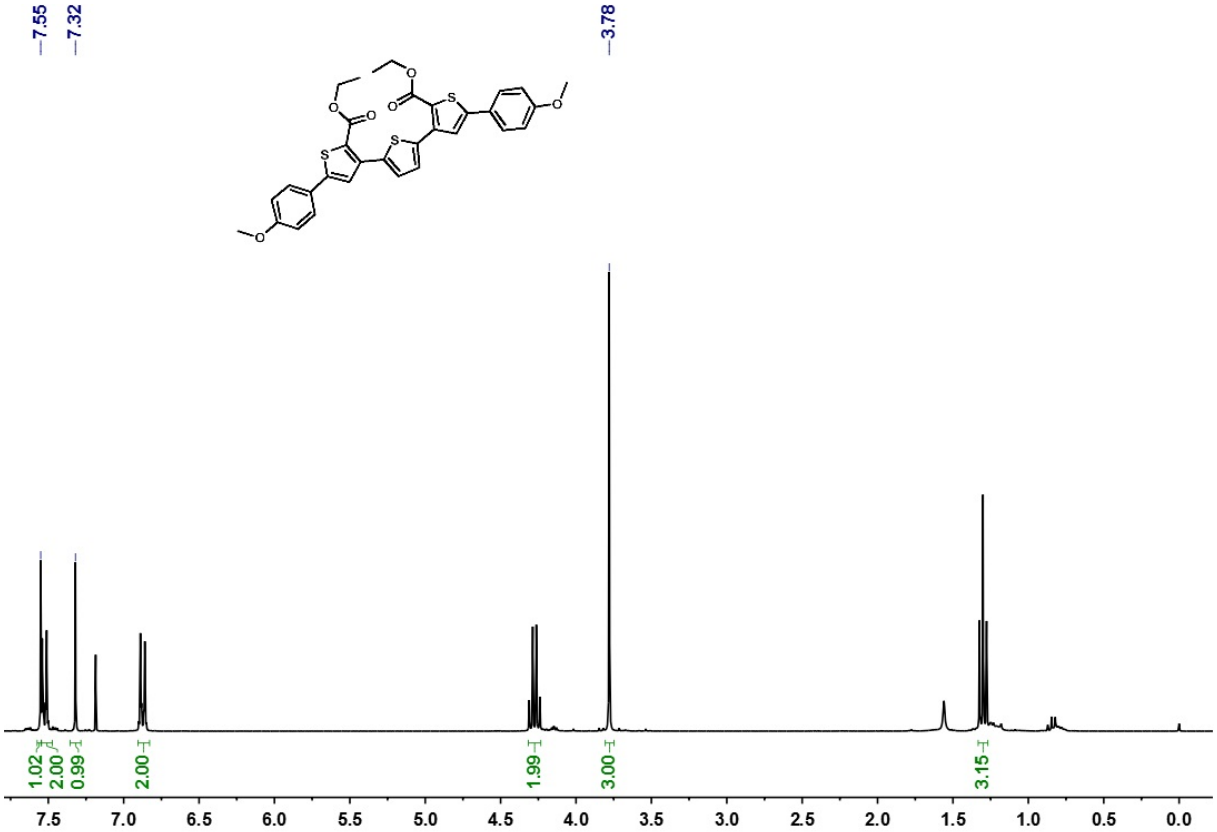
The purification of products was performed on silica gel 60 (0.04-0.063 mm) from Macherey-Nagel GmbH & Co. KG using flash technique and under pressure of 2 bar or using the automatic chromatography system Biotage SP4.

The crude mixtures were adsorbed on Celite® 545 (0.02-0.10 mm) from Merck KGaA Darmstadt or silica gel 60 (0.2-0.5 mm) from Merck Darmstadt prior to chromatographic purification.

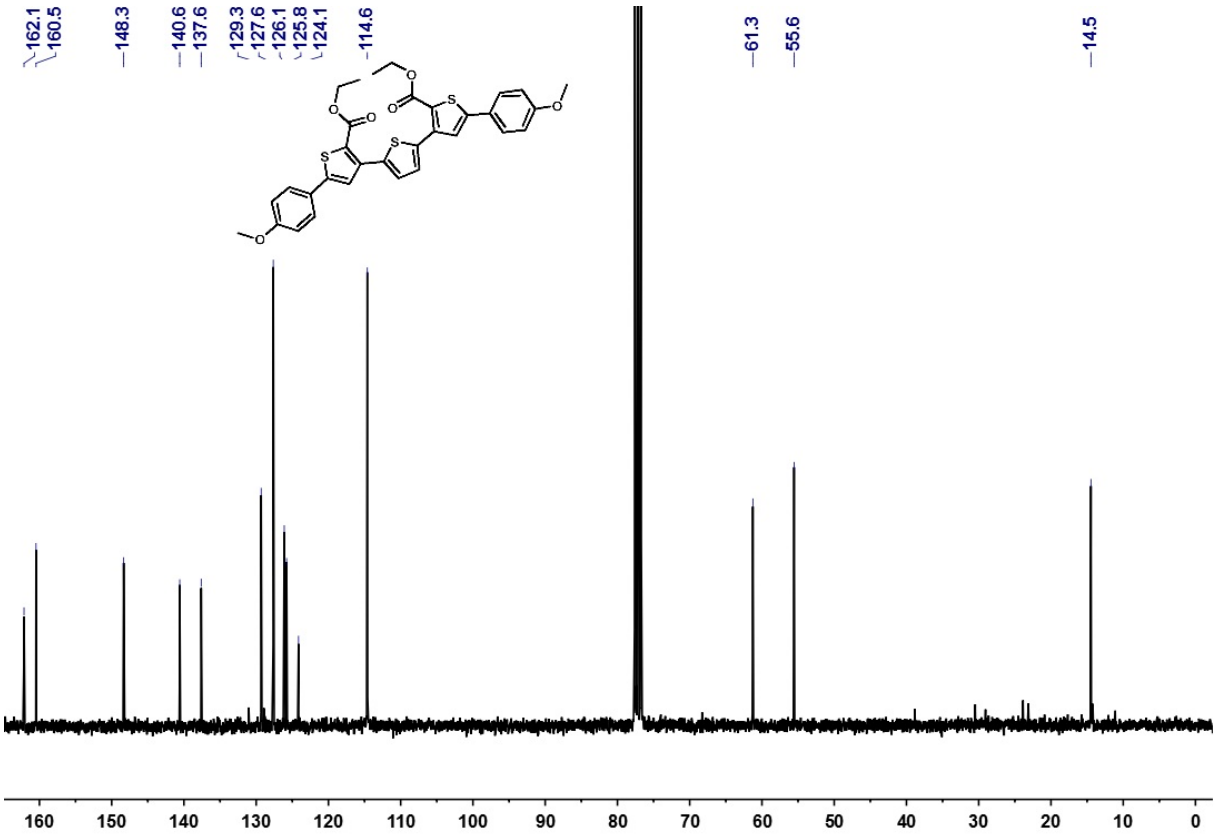
The reaction progress was monitored qualitatively using TLC Silica gel 60 F254 5 x 7.5 cm aluminium sheets obtained by Merck KGaA Darmstadt. The spots were detected with UV light at 254 nm and using aqueous potassium permanganate solution.

^{1}H, ^{13}C, and 135-DEPT NMR spectra were recorded on Bruker DRX 500 spectrometer. $CDCl_3$ and DMSO-d^6 were used as deuterated solvents. TMS was used as reference (δ = 0.0) or the resonances of the solvents were locked as internal standards ($CDCl_3$: ^{1}H δ 7.24, ^{13}C δ 77.2). The multiplicities of signals were abbreviated as follows: s: singlet; d: doublet; t: triplet; dd: doublet of doublets, m: multiplet and br: broad signal. The type of carbon nuclei was determined on the basis of 135-DEPT NMR spectra. EI mass spectra were measured on Finnigan MAT 8200 spectrometer. IR spectra were obtained on Bruker Vector 22 FT-IR. The solids were measured as KBr pellets and oils as films on KBr plates. The intensity of signals is abbreviated as follows: s (strong), m (medium) and w (weak). The melting points (uncorrected) were measured on Reichert-Jung Thermovar. Combustion analyses were carried out on Perkin Elmer Series II Analyser 2400 in the microanalytical laboratory of Institut für Pharmazeutische und Medizinische Chemie at the Heinrich-Heine-Universität Düsseldorf.

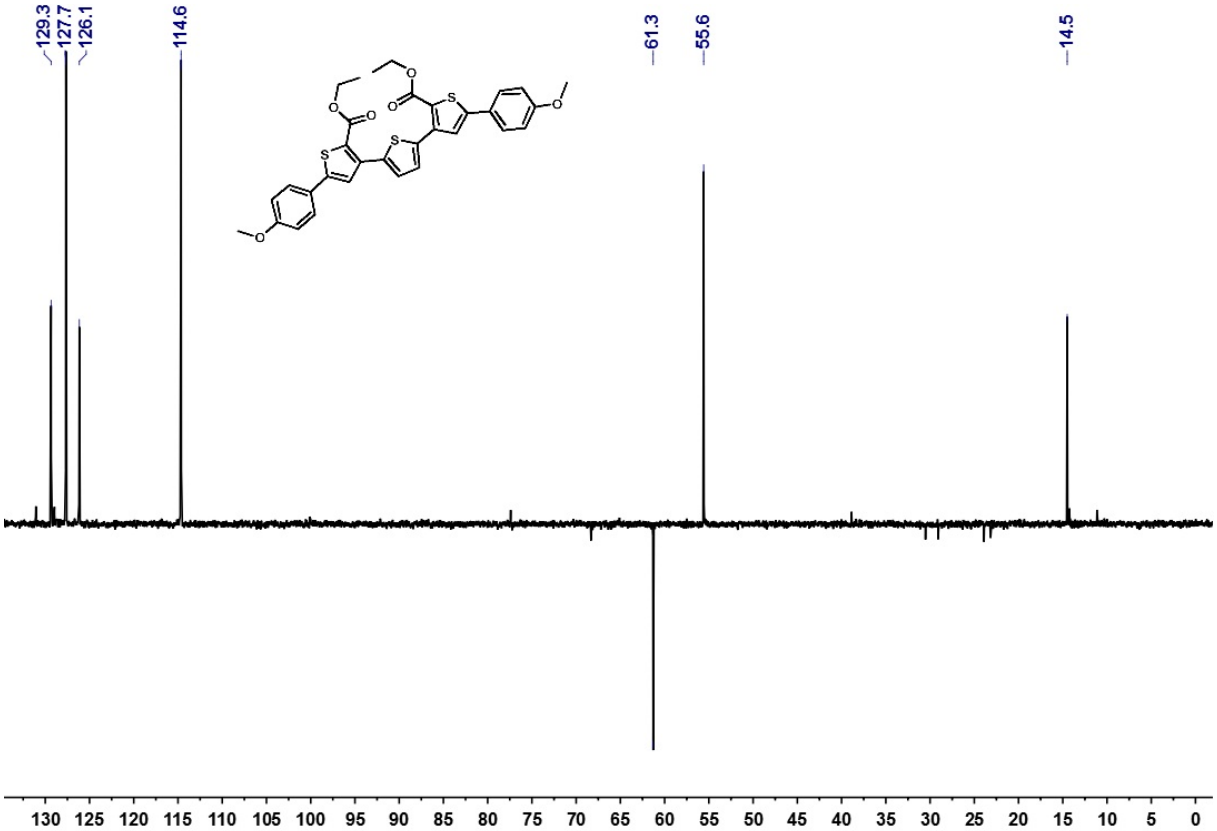
2 ^{1}H AND ^{13}C NMR SPECTRA



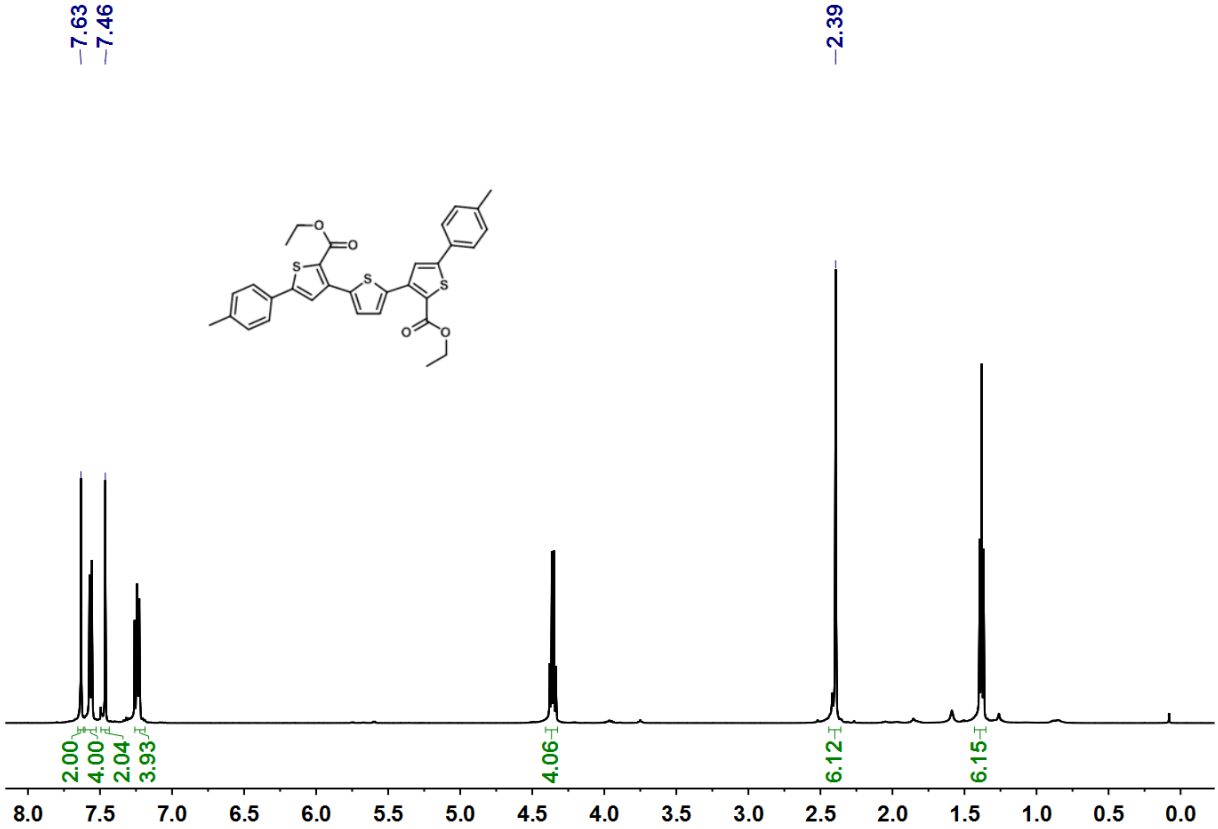
^{1}H-NMR of **6a** ($CDCl_3$, 500 MHz, 298 K)



^{13}C-NMR of **6a** ($CDCl_3$, 125 MHz, 298 K)



135-DEPT of **6a** ($CDCl_3$, 125 MHz, 298 K)



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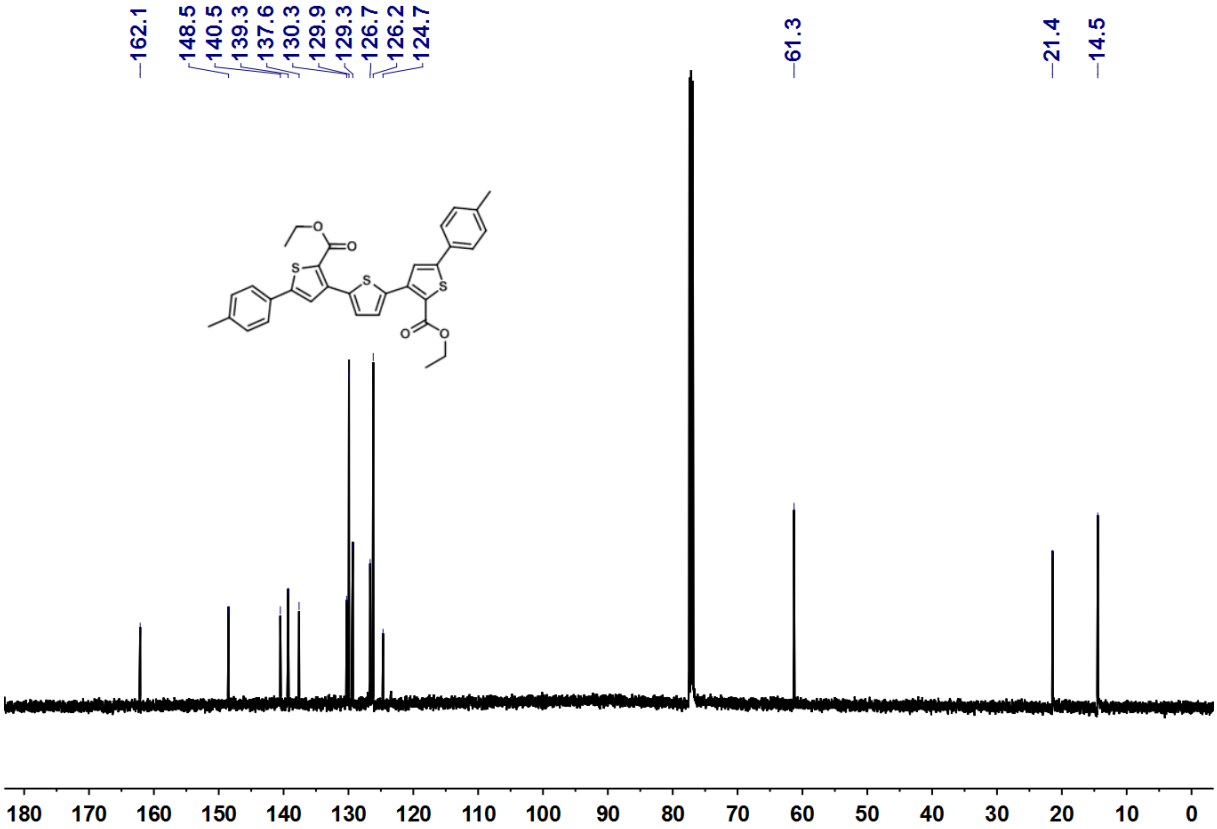

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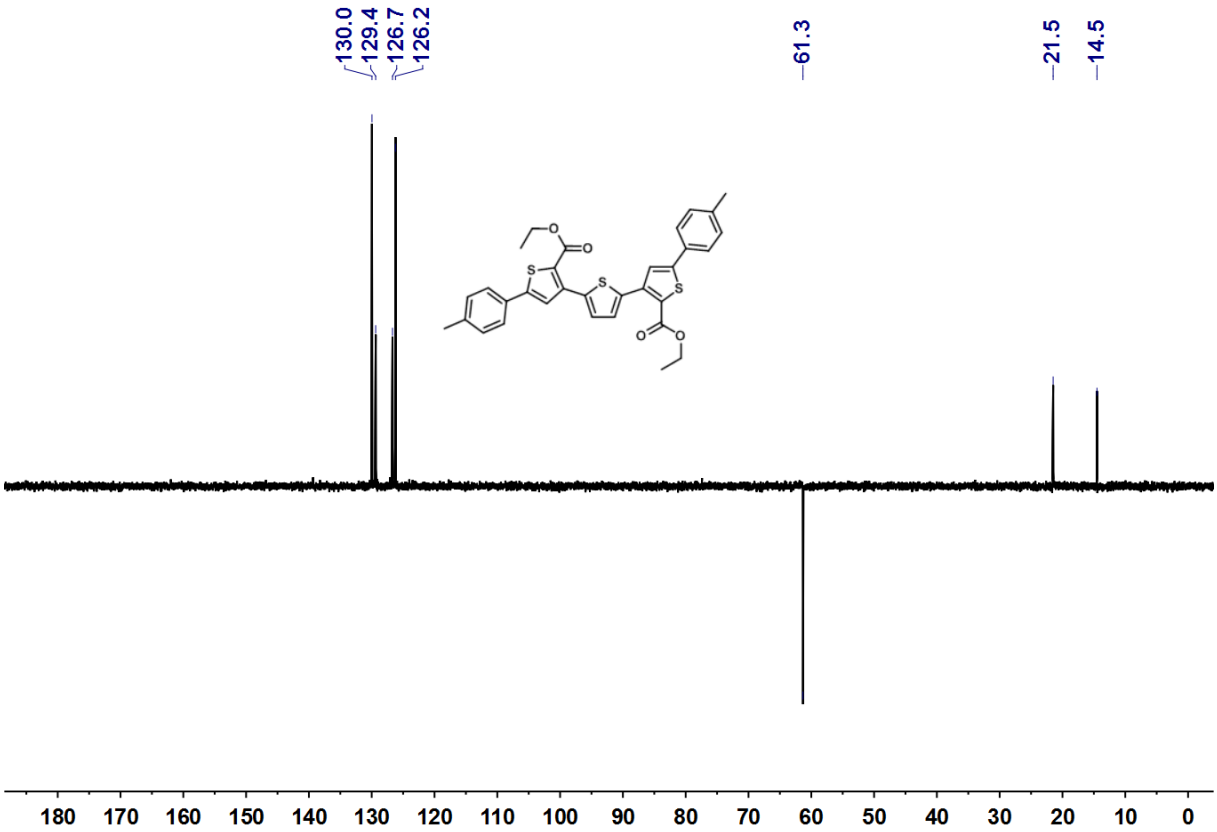
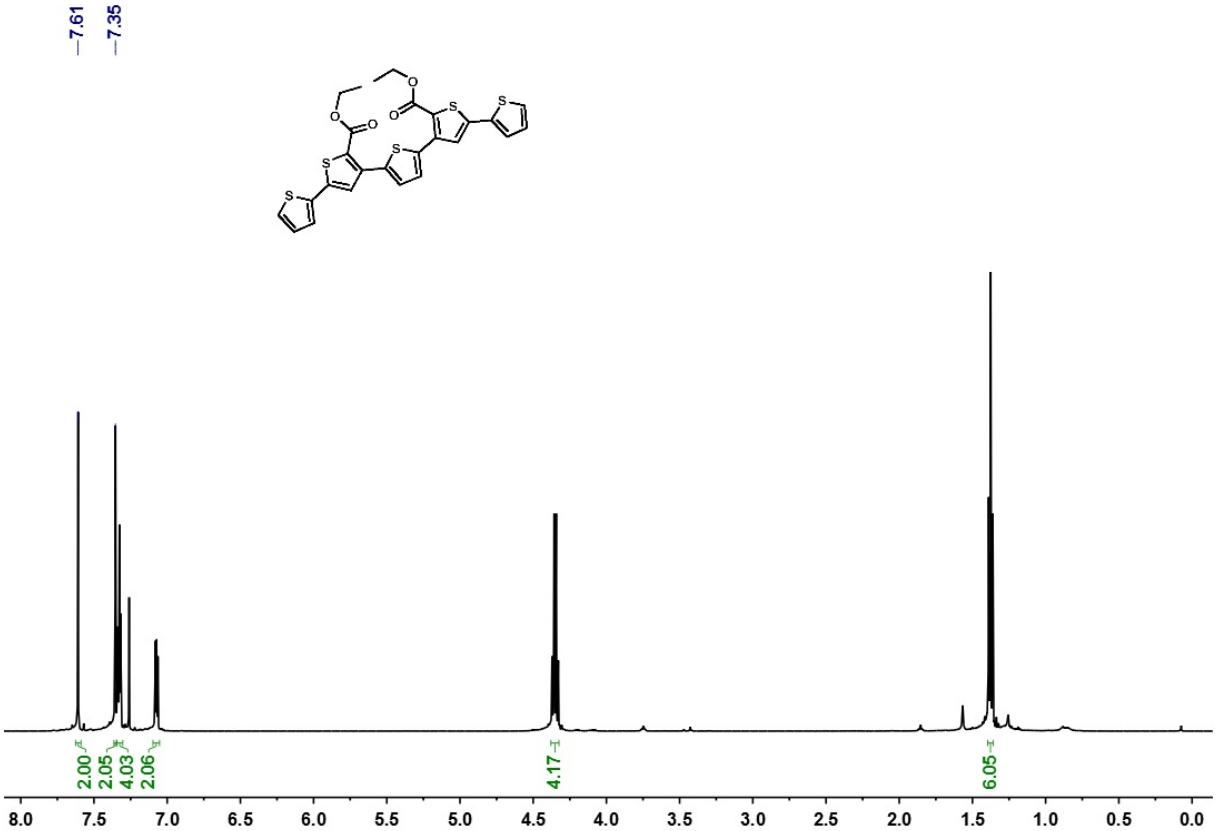


135-DEPT of **6b** ($CDCl_3$, 125 MHz, 298 K)



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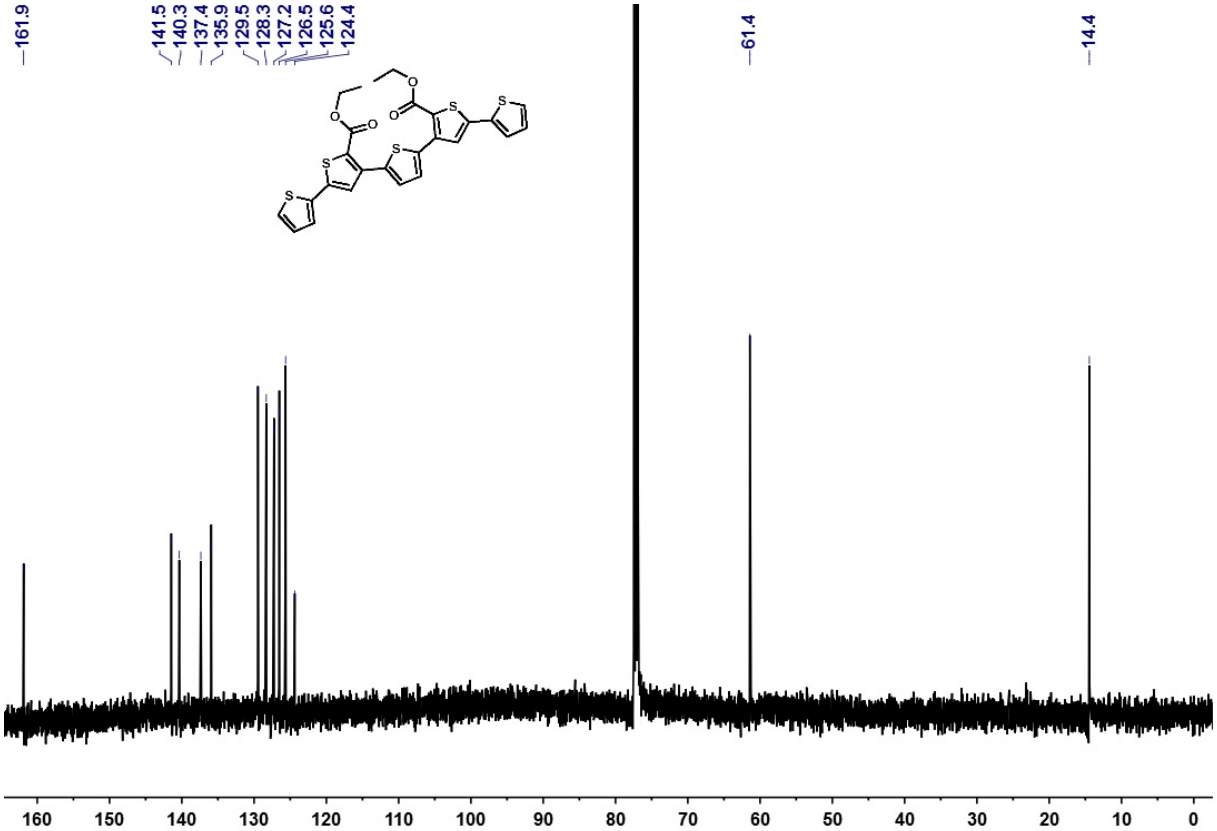
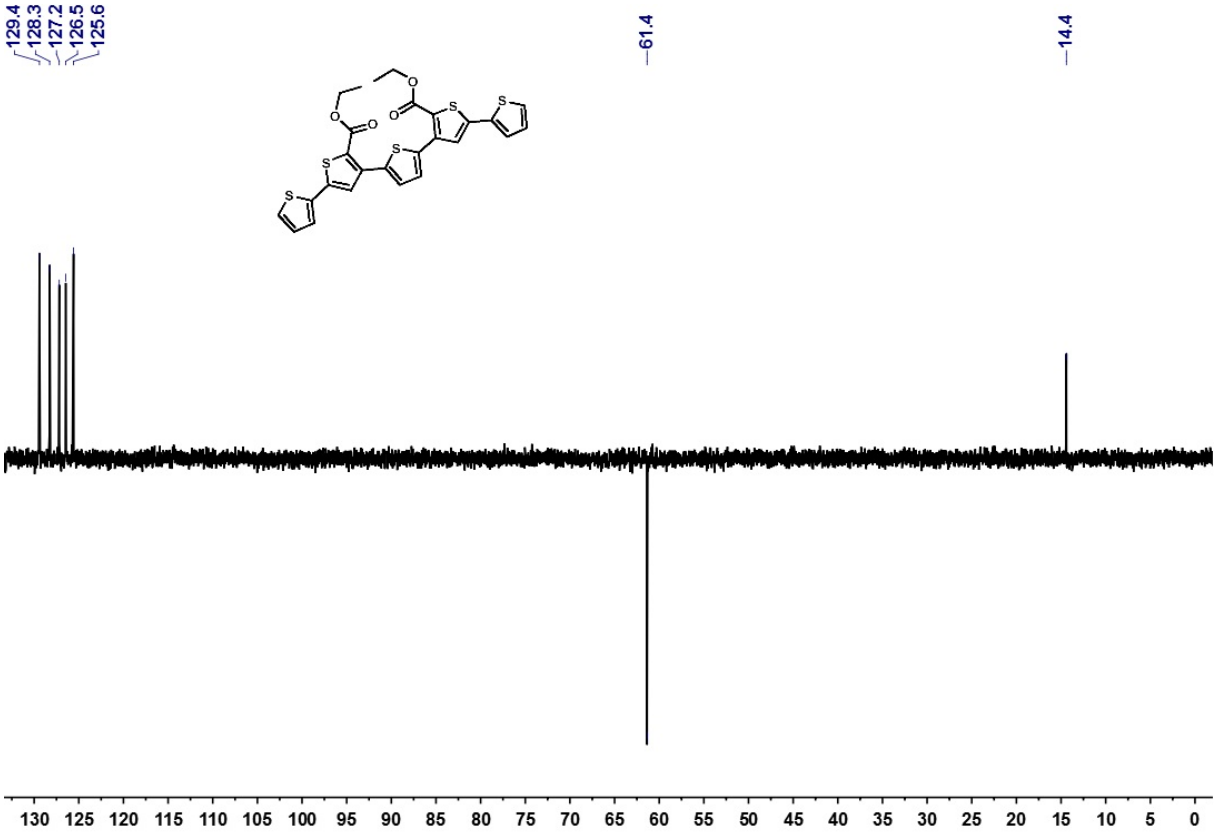
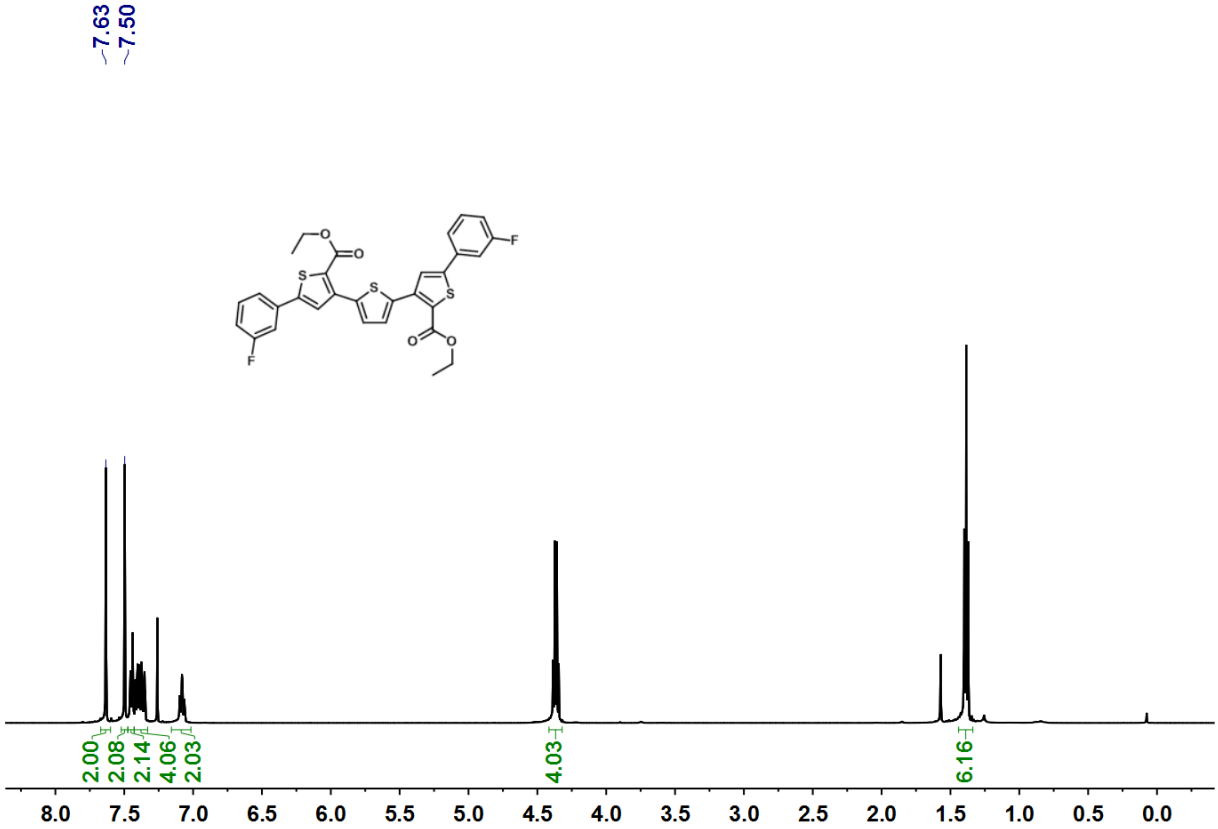


^{13}C-NMR of **6c** ($CDCl_3$, 125 MHz, 298 K)



135-DEPT of **6c** ($CDCl_3$, 125 MHz, 298 K)



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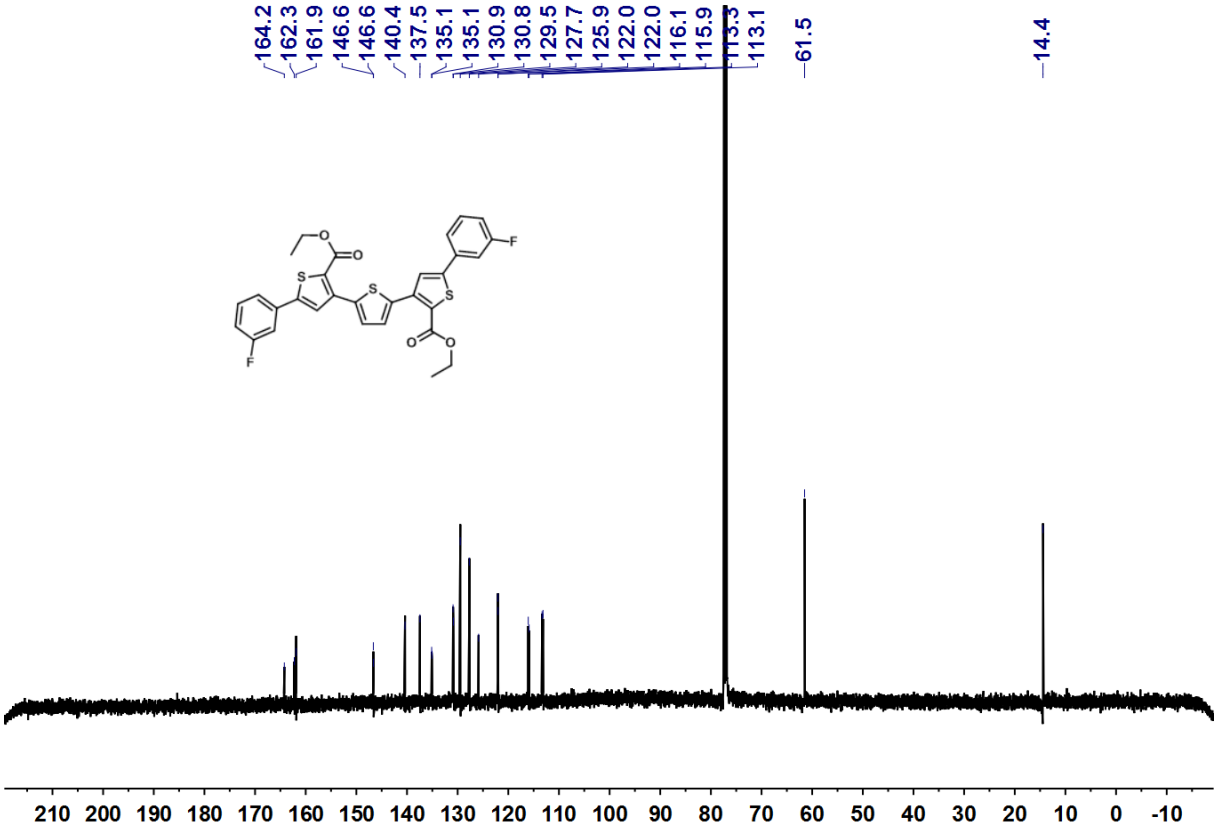

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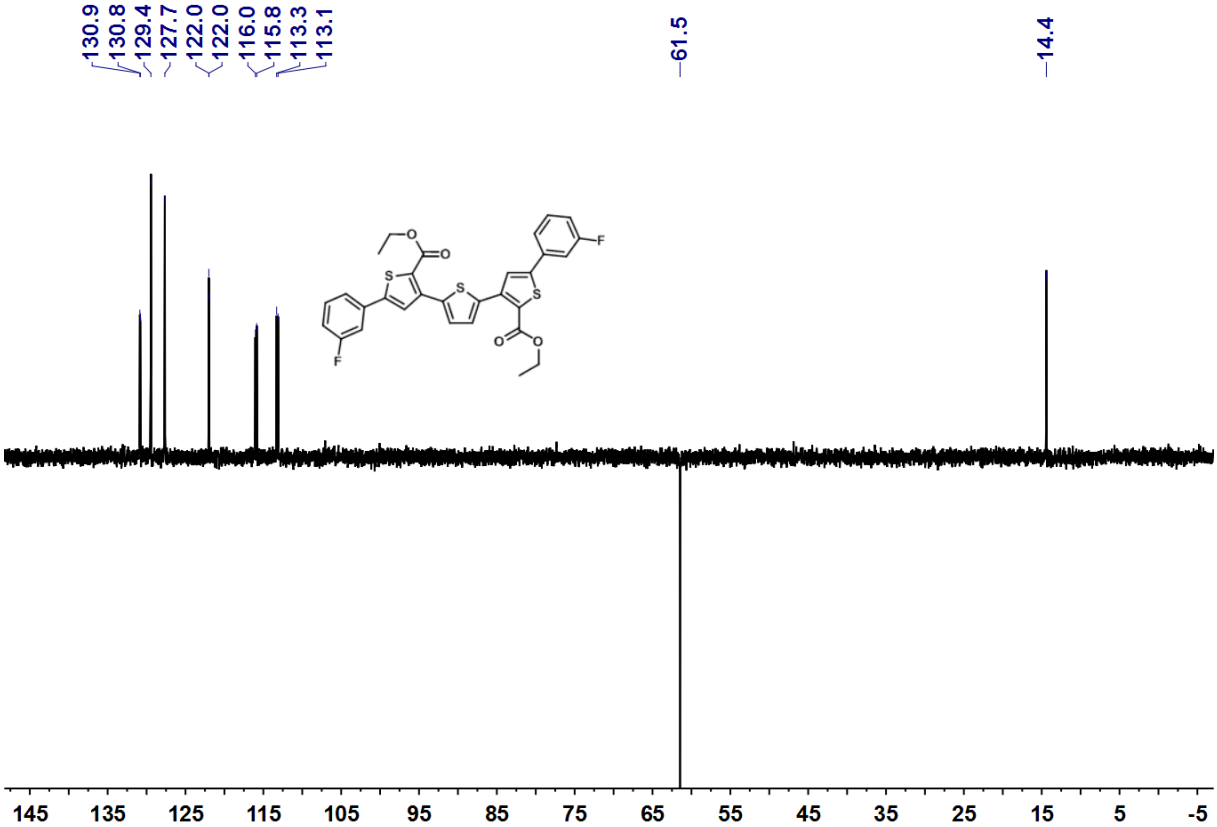
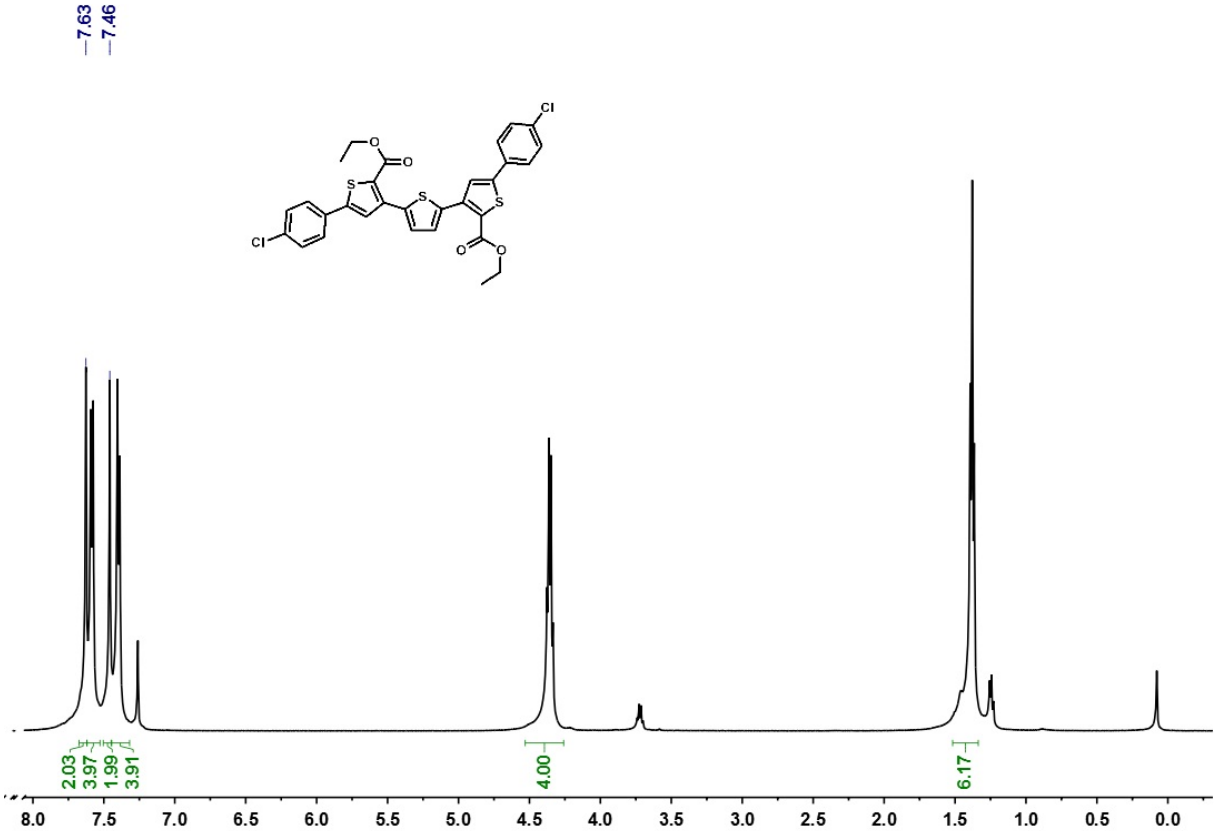


135-DEPT of **6d** ($CDCl_3$, 125 MHz, 298 K)



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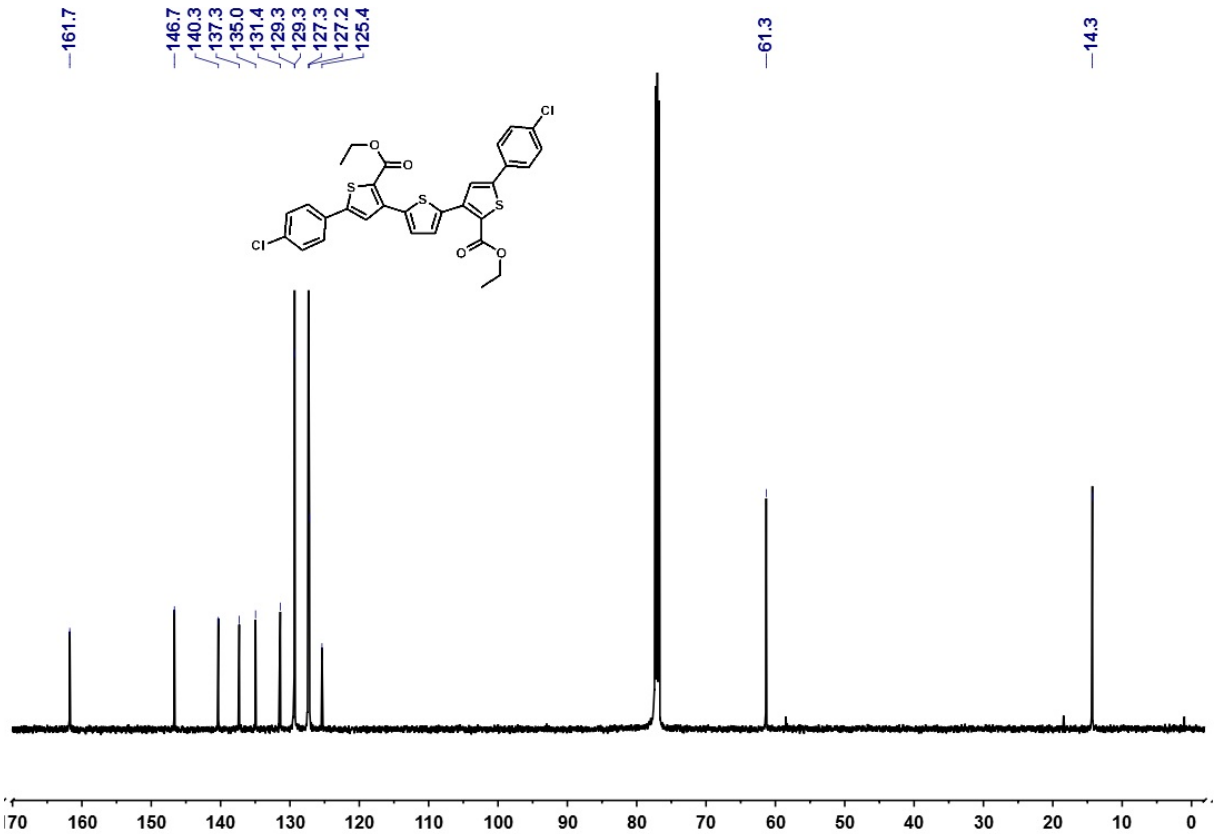
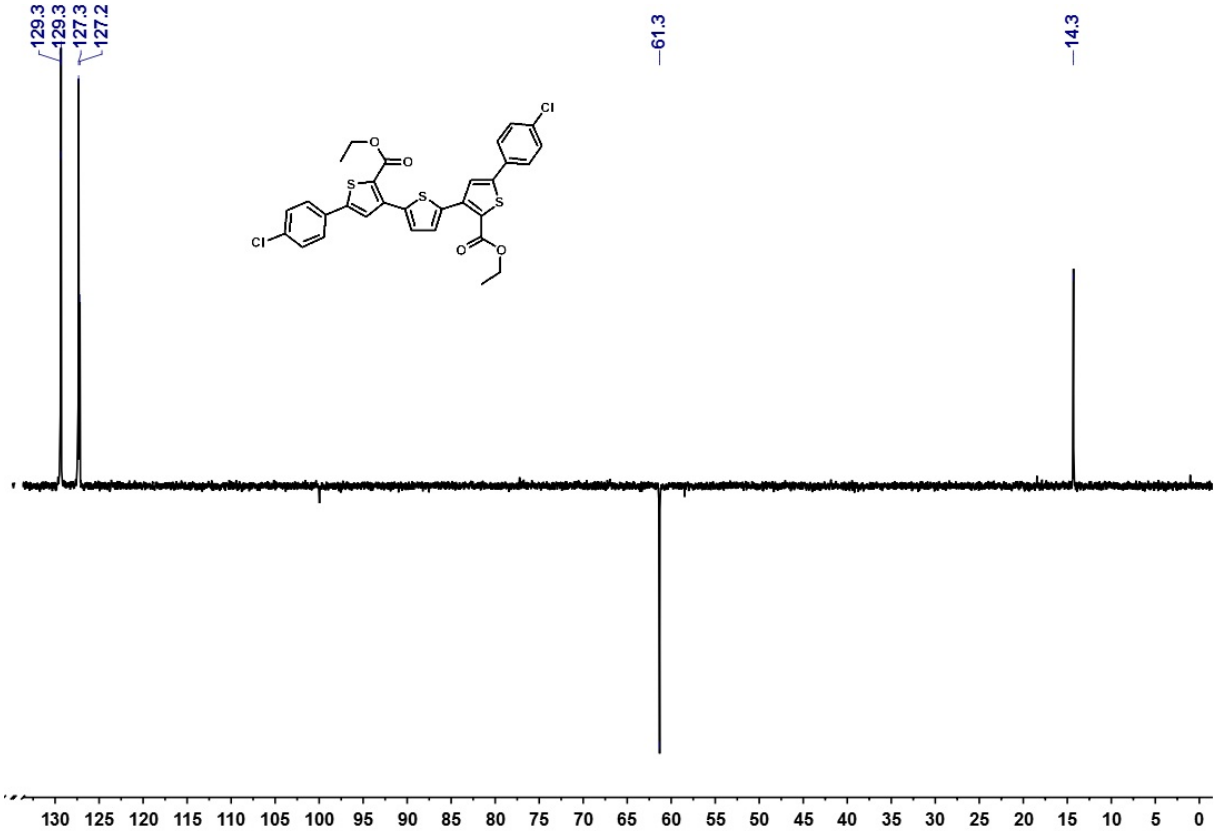
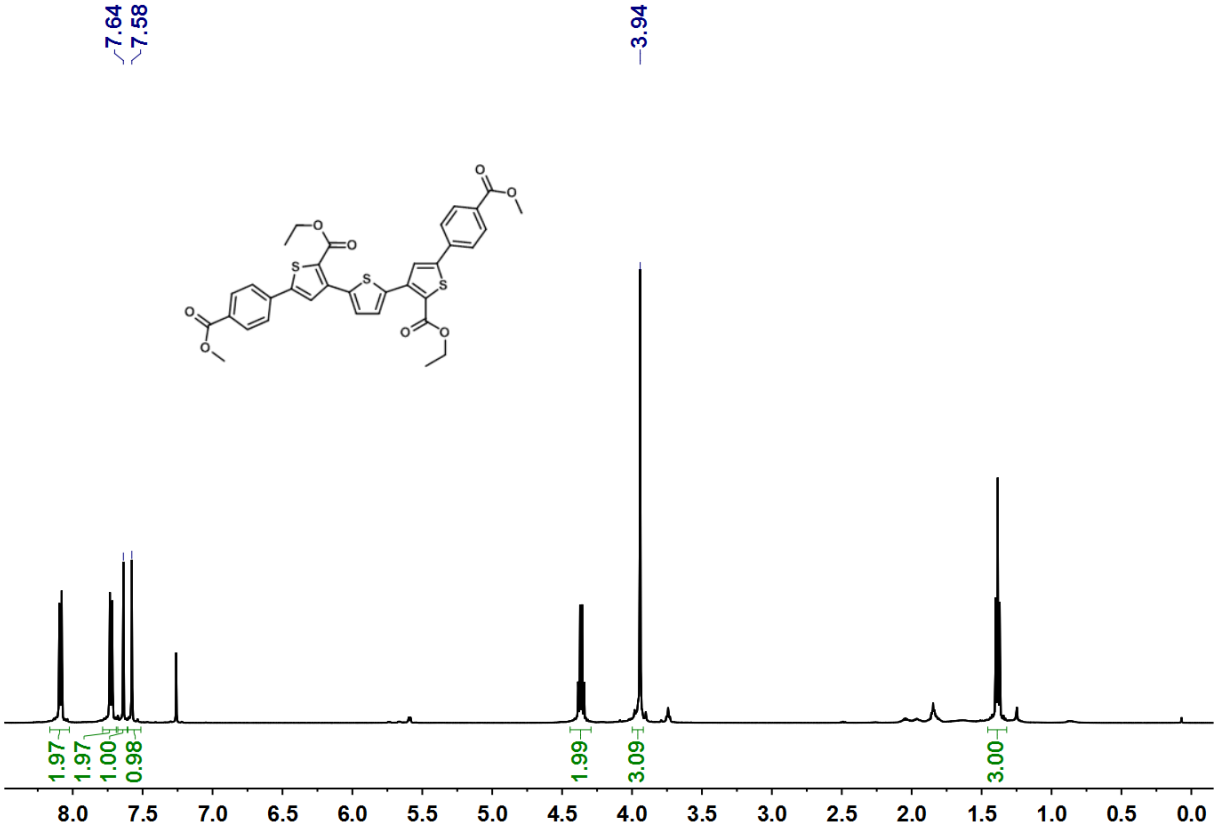


^{13}C-NMR of **6e** ($CDCl_3$, 125 MHz, 298 K)



135-DEPT of **6e** ($CDCl_3$, 125 MHz, 298 K)



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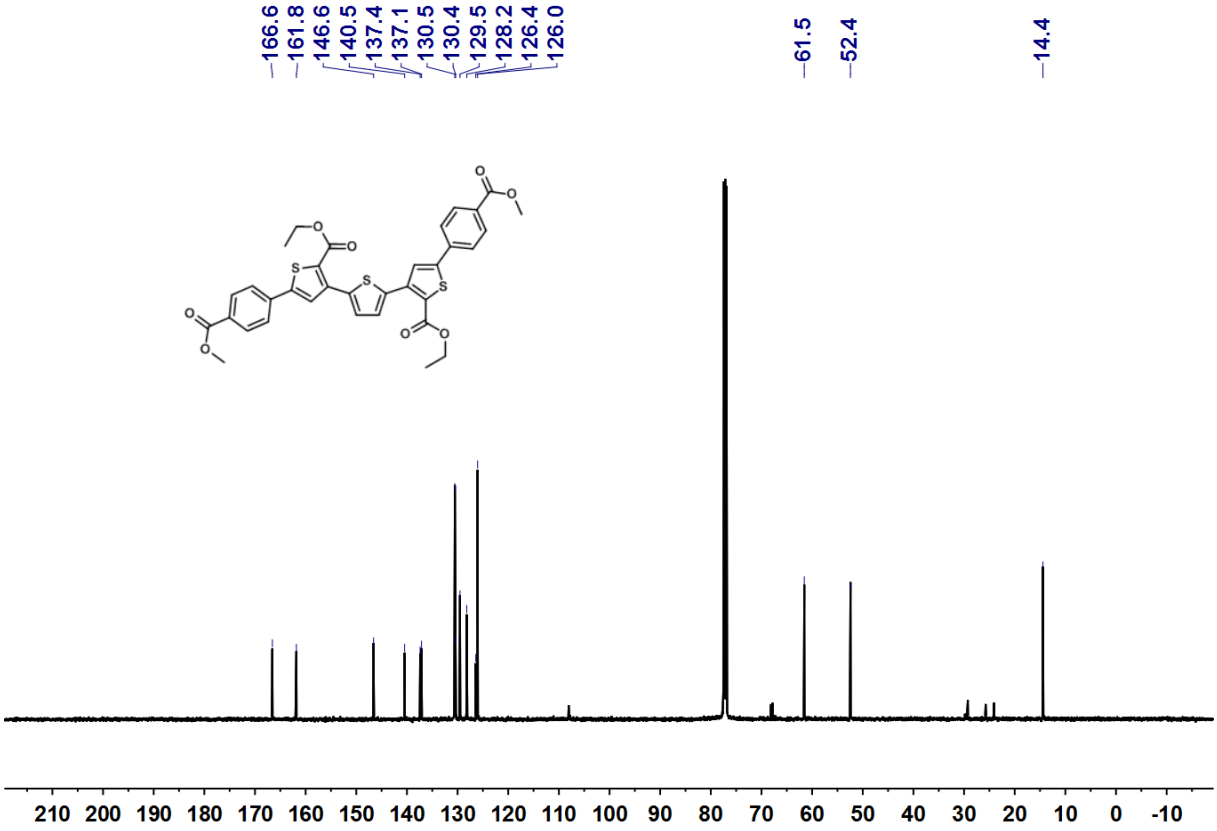
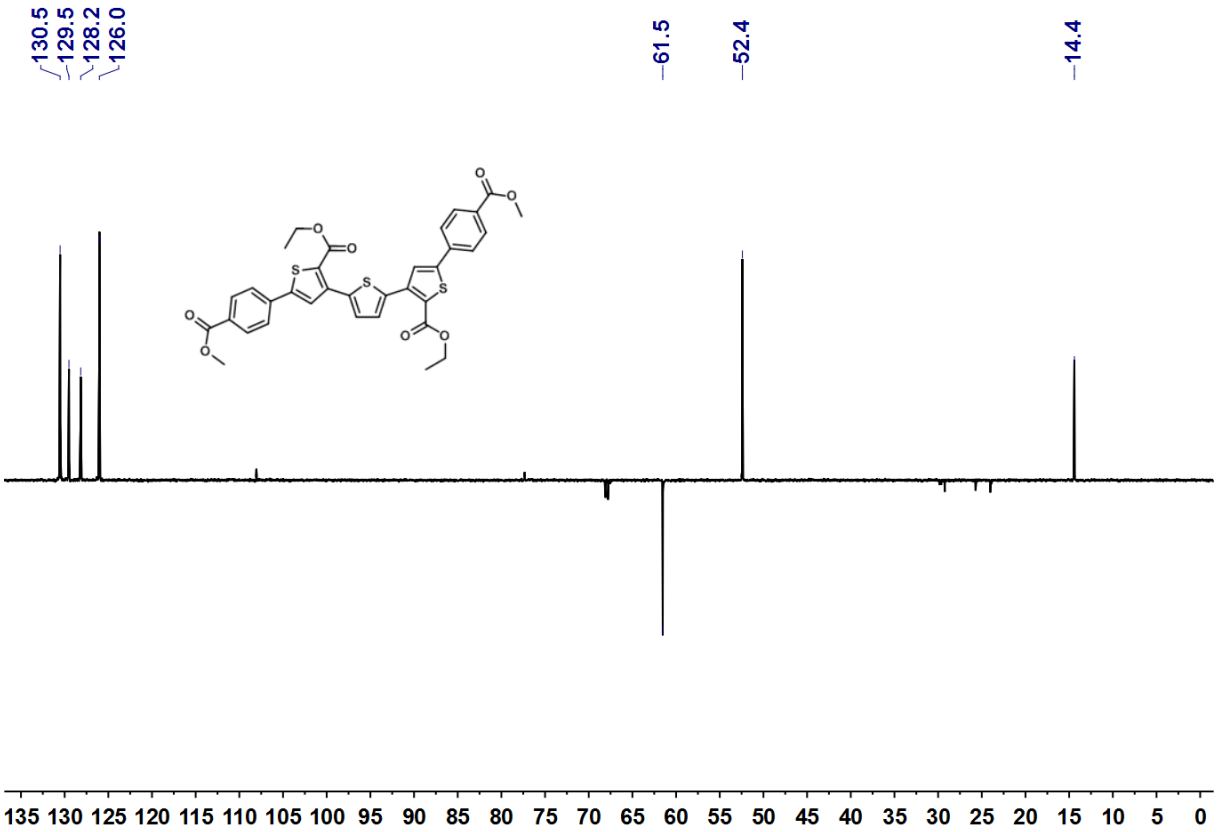
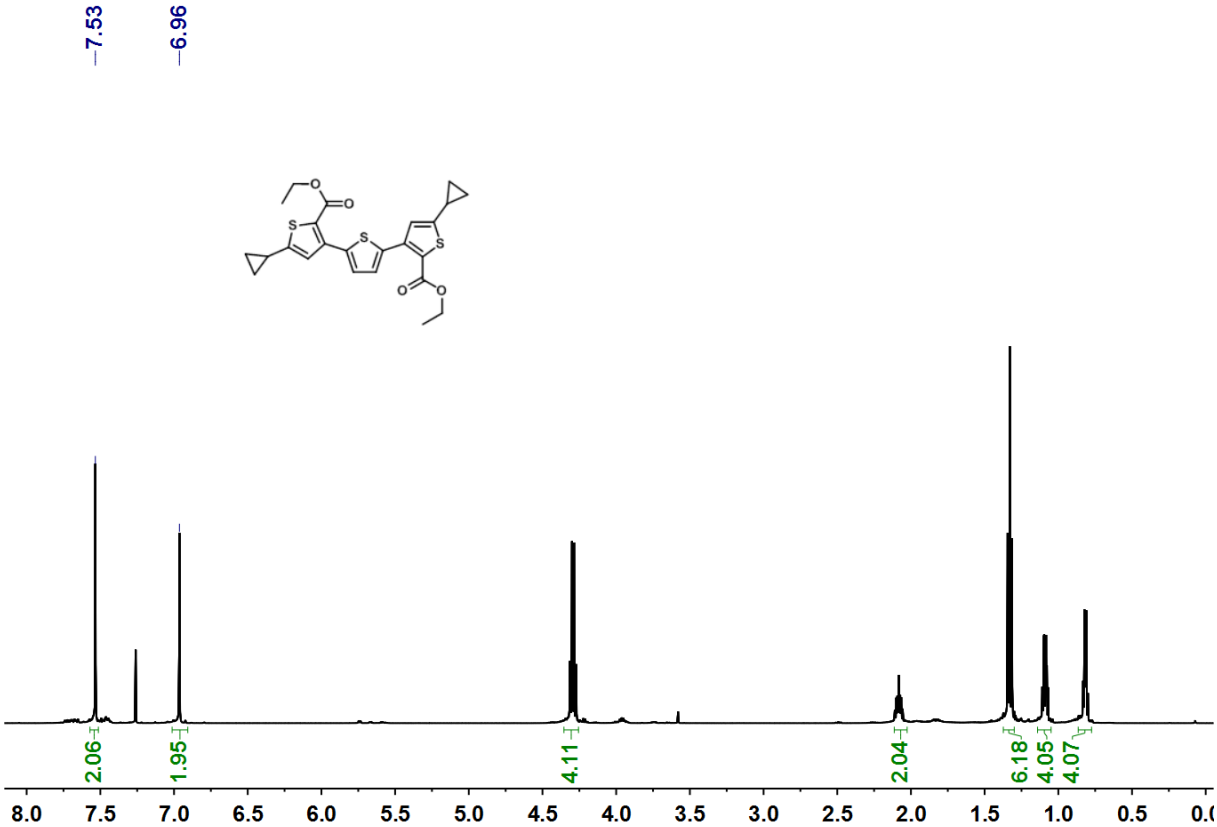


^{13}C-NMR of **6f** ($CDCl_3$, 125 MHz, 298 K)



135-DEPT of **6f** ($CDCl_3$, 125 MHz, 298 K)



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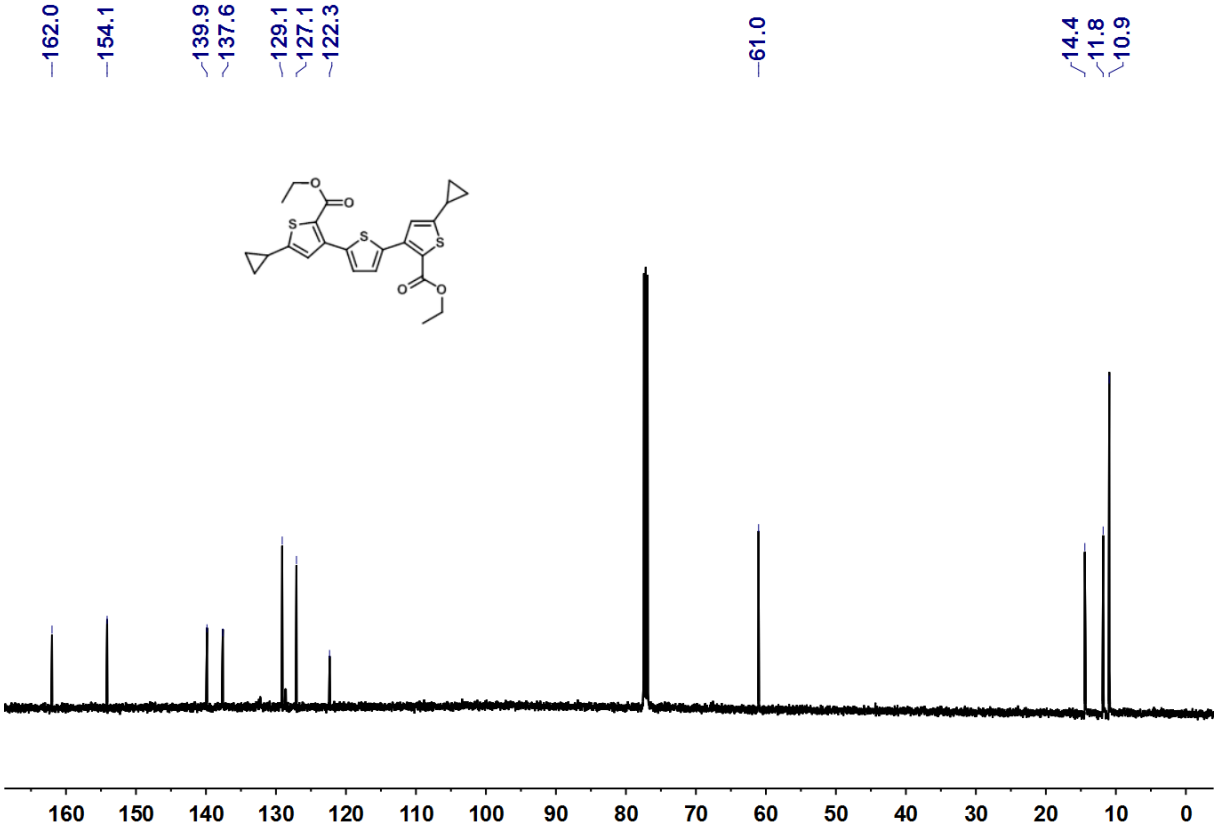
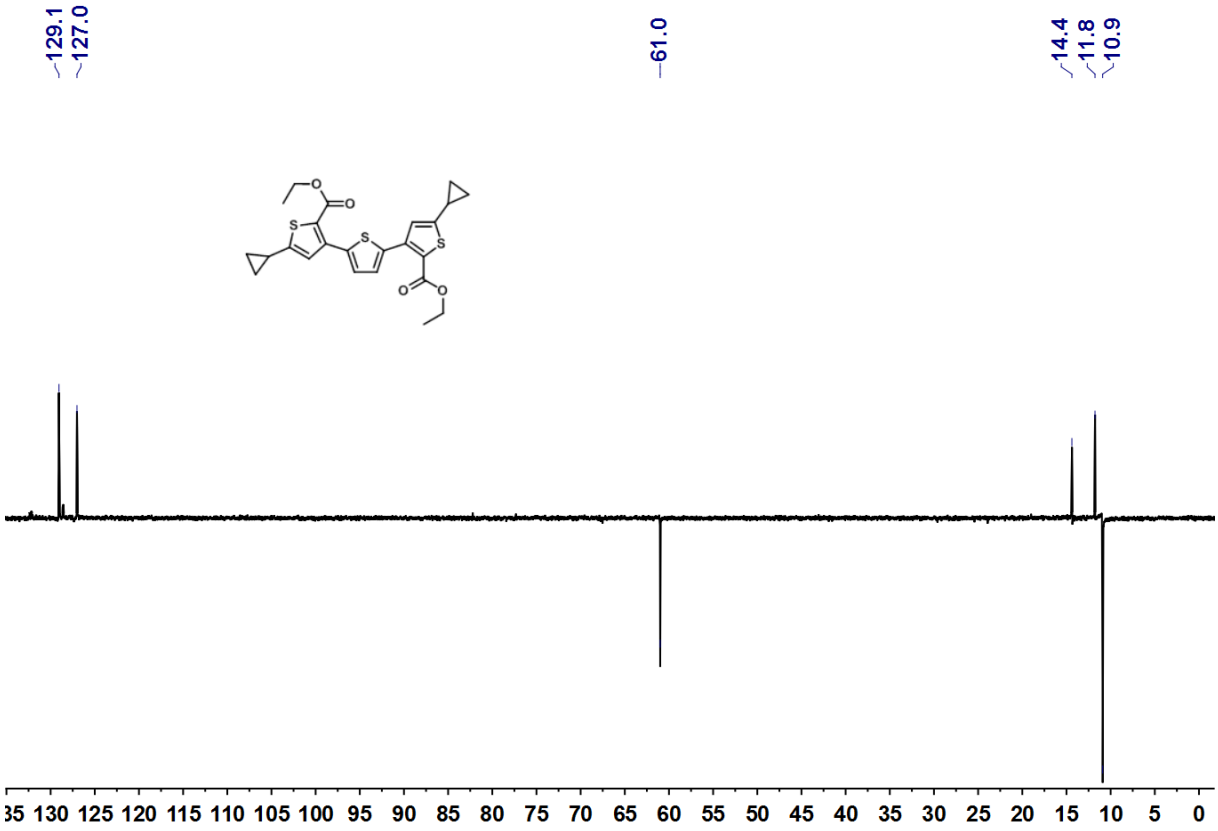
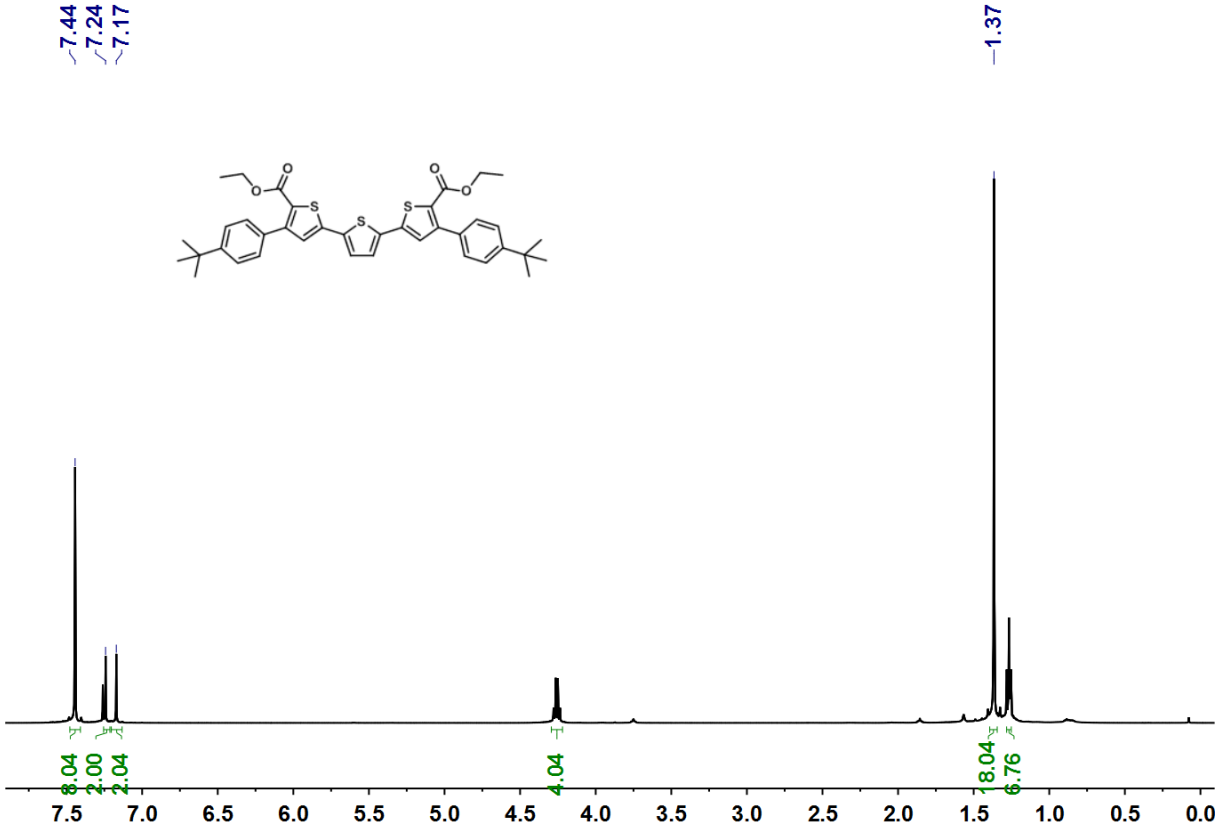


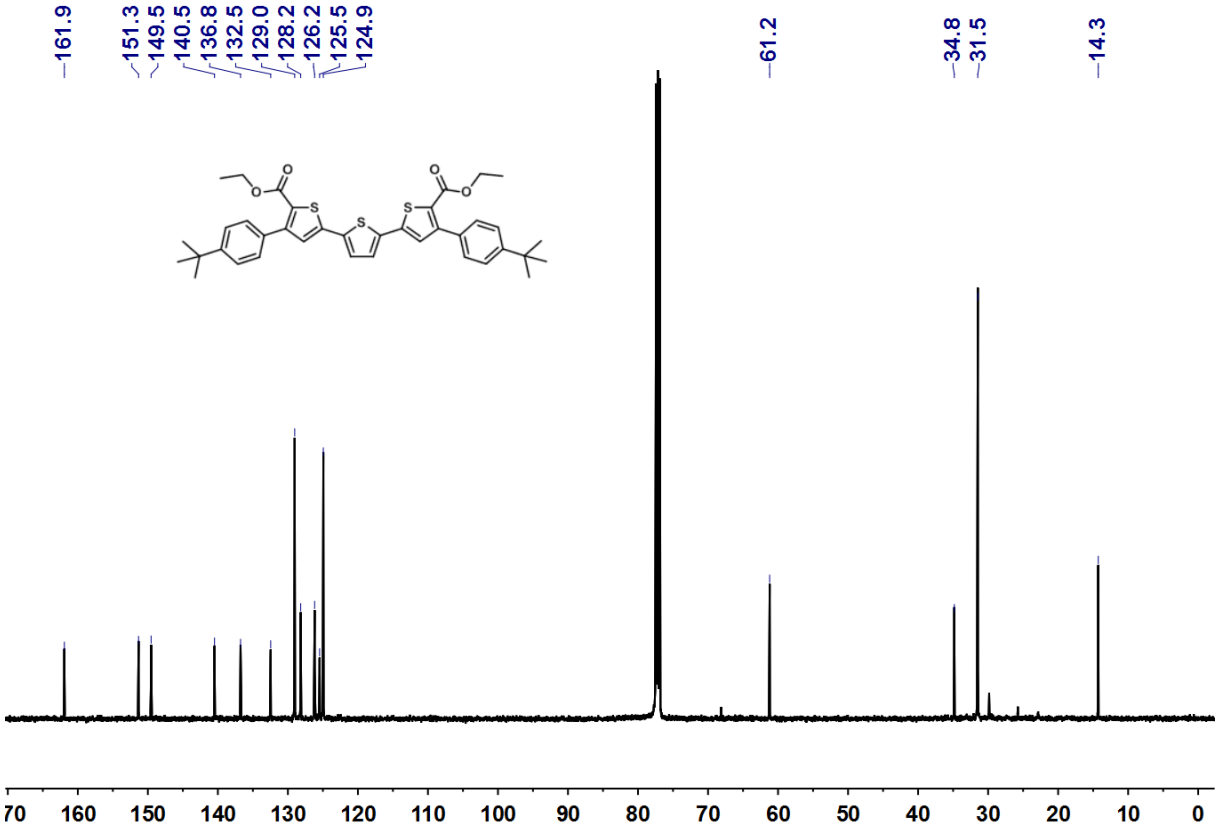
^{13}C-NMR of **6g** ($CDCl_3$, 125 MHz, 298 K)



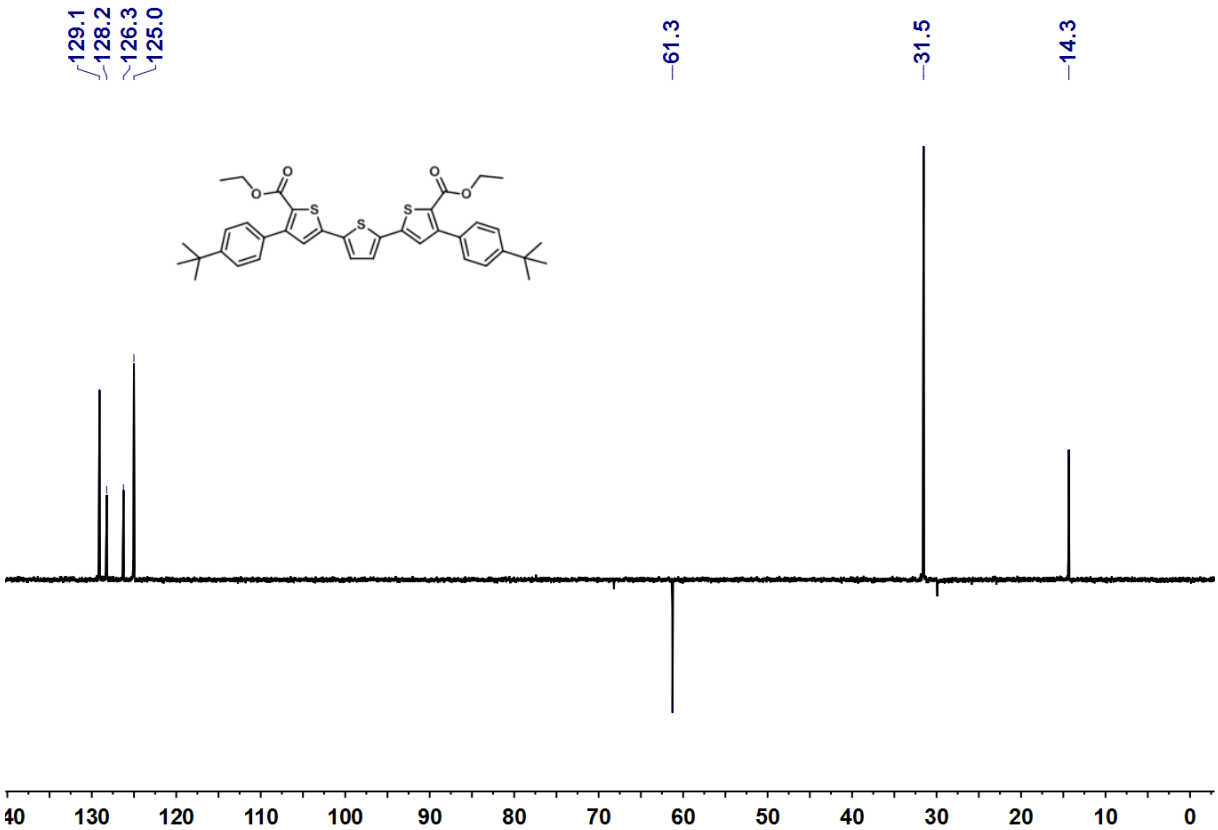
135-DEPT of **6g** ($CDCl_3$, 125 MHz, 298 K)



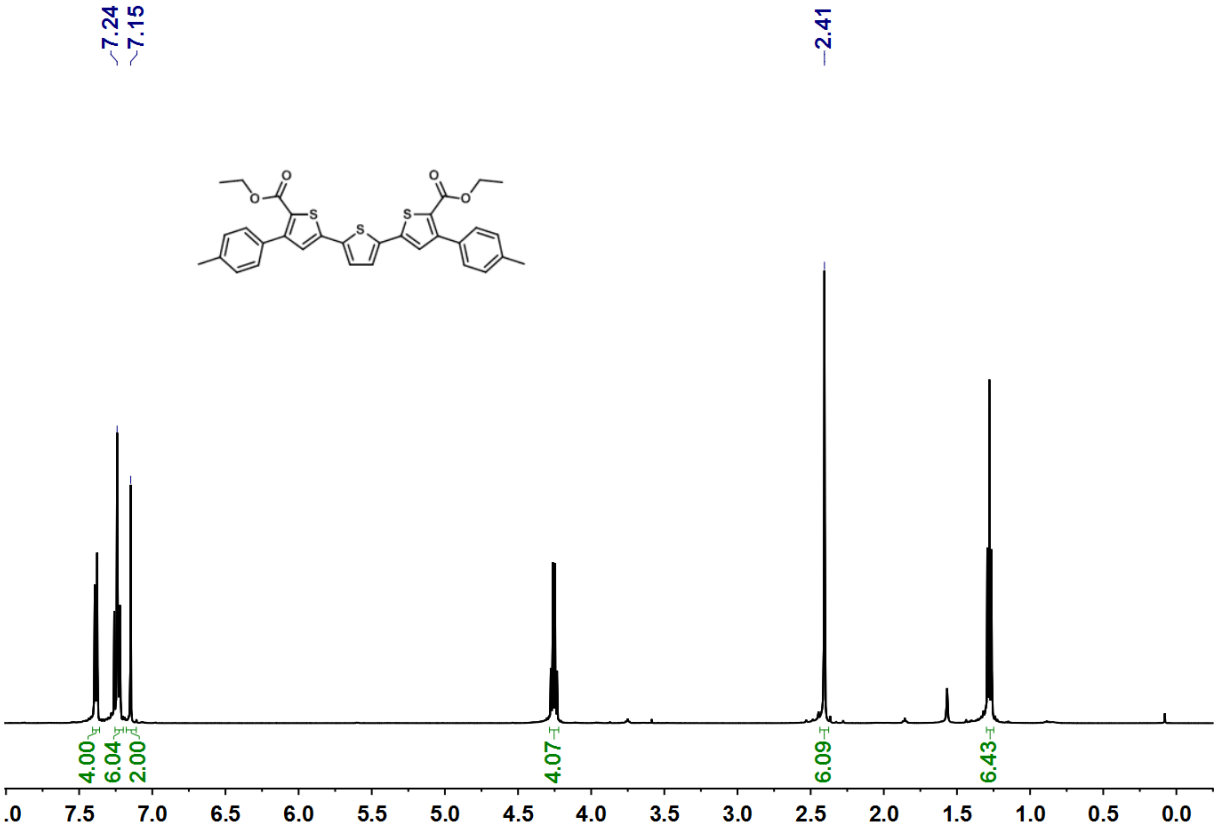
^{1}H-NMR of **8a** ($CDCl_3$, 500 MHz, 298 K)



^{13}C-NMR of **8a** ($CDCl_3$, 125 MHz, 298 K)



135-DEPT of **8a** ($CDCl_3$, 125 MHz, 298 K)



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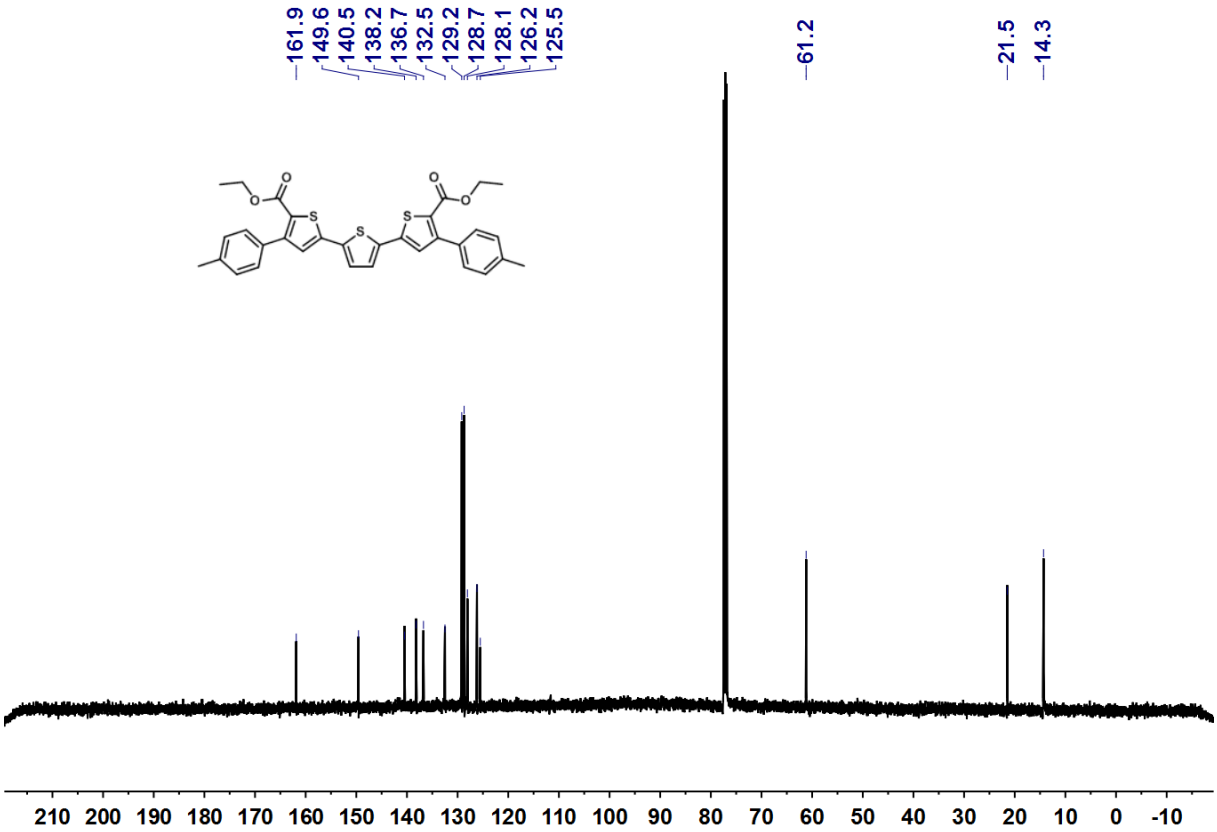
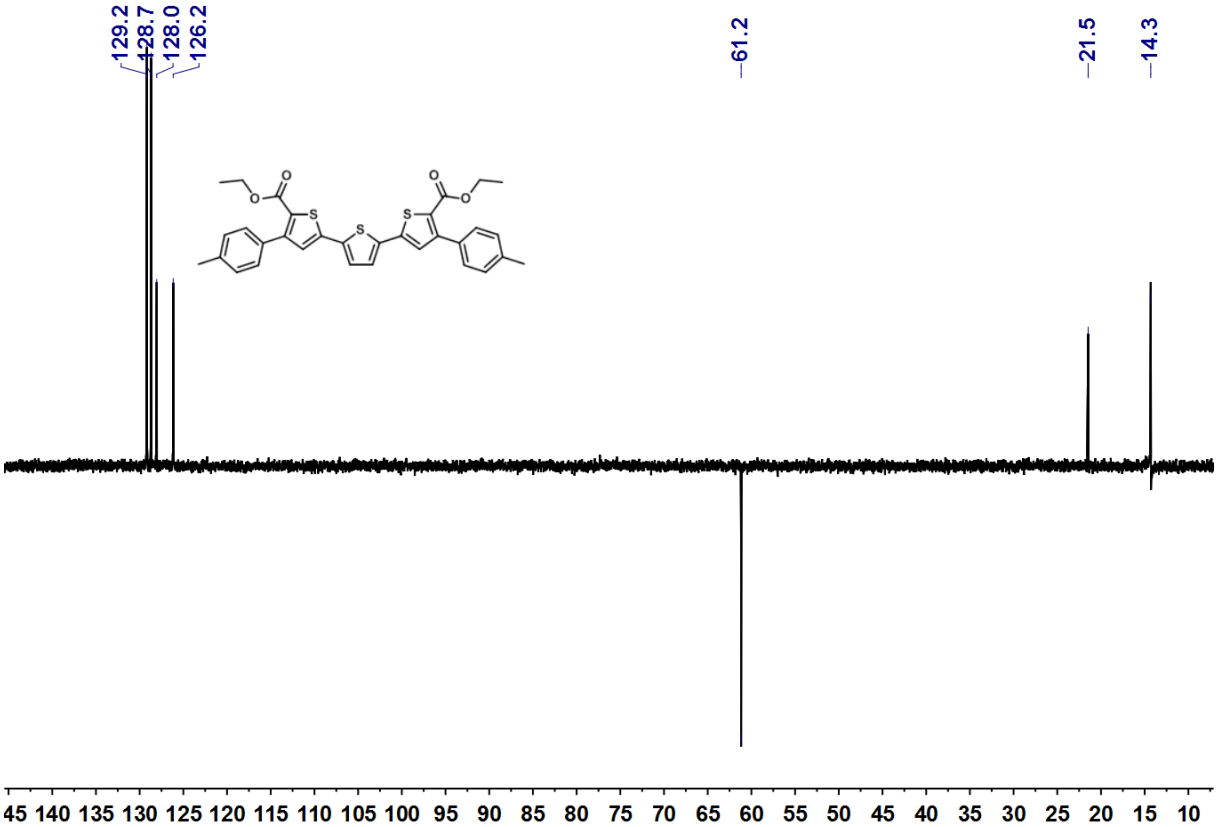
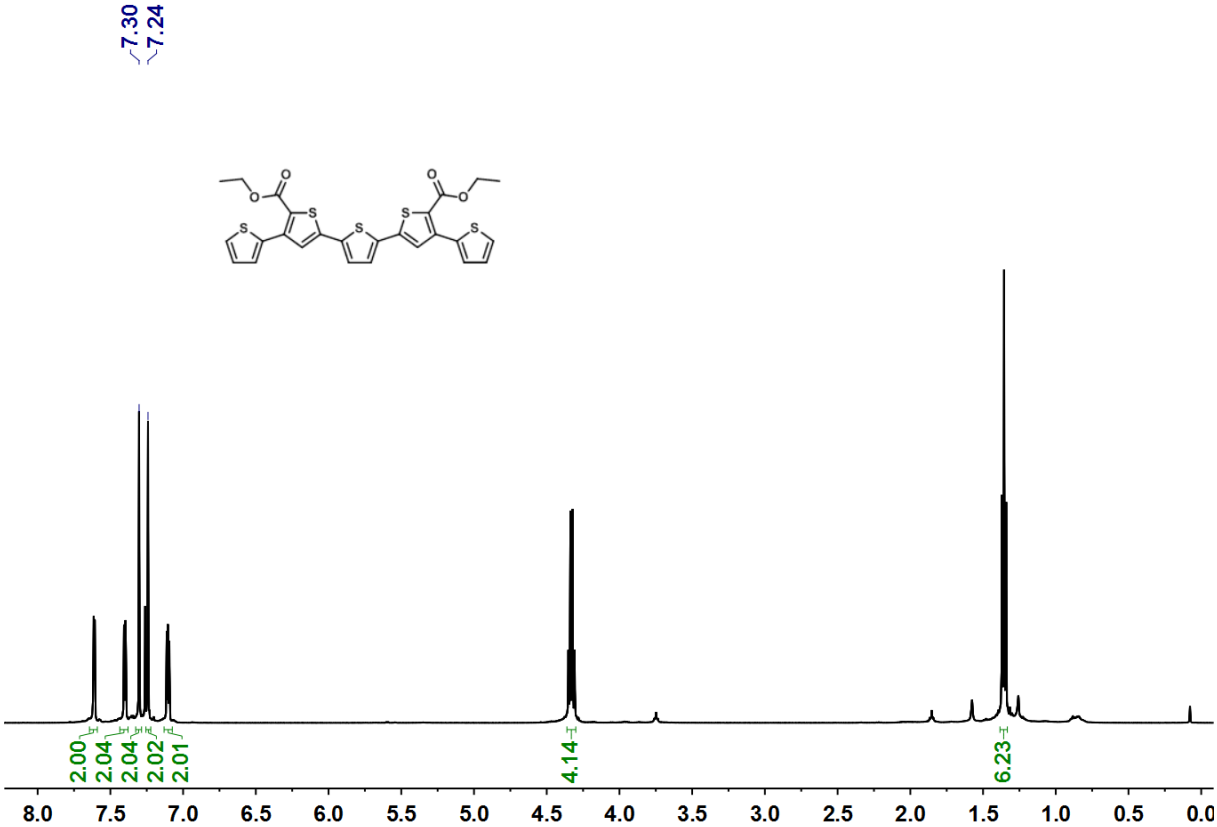


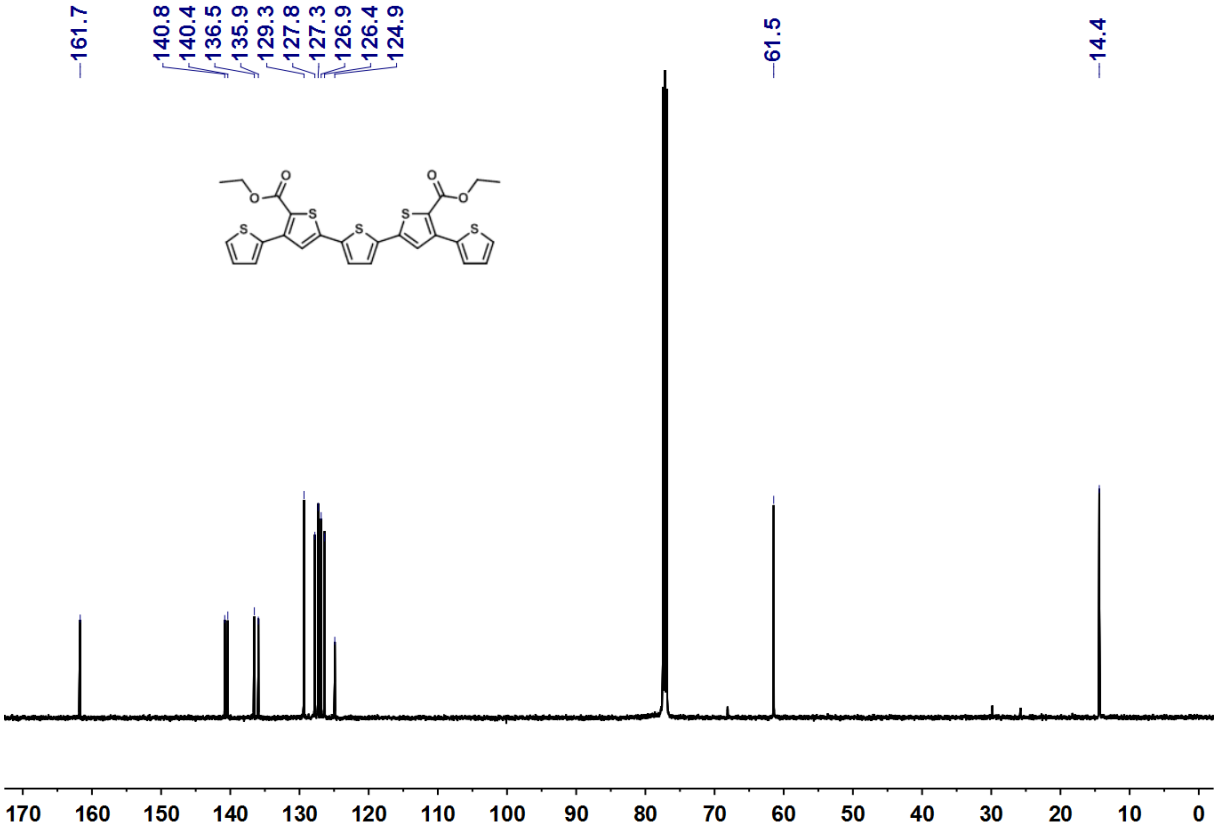
^{13}C-NMR of **8b** ($CDCl_3$, 125 MHz, 298 K)



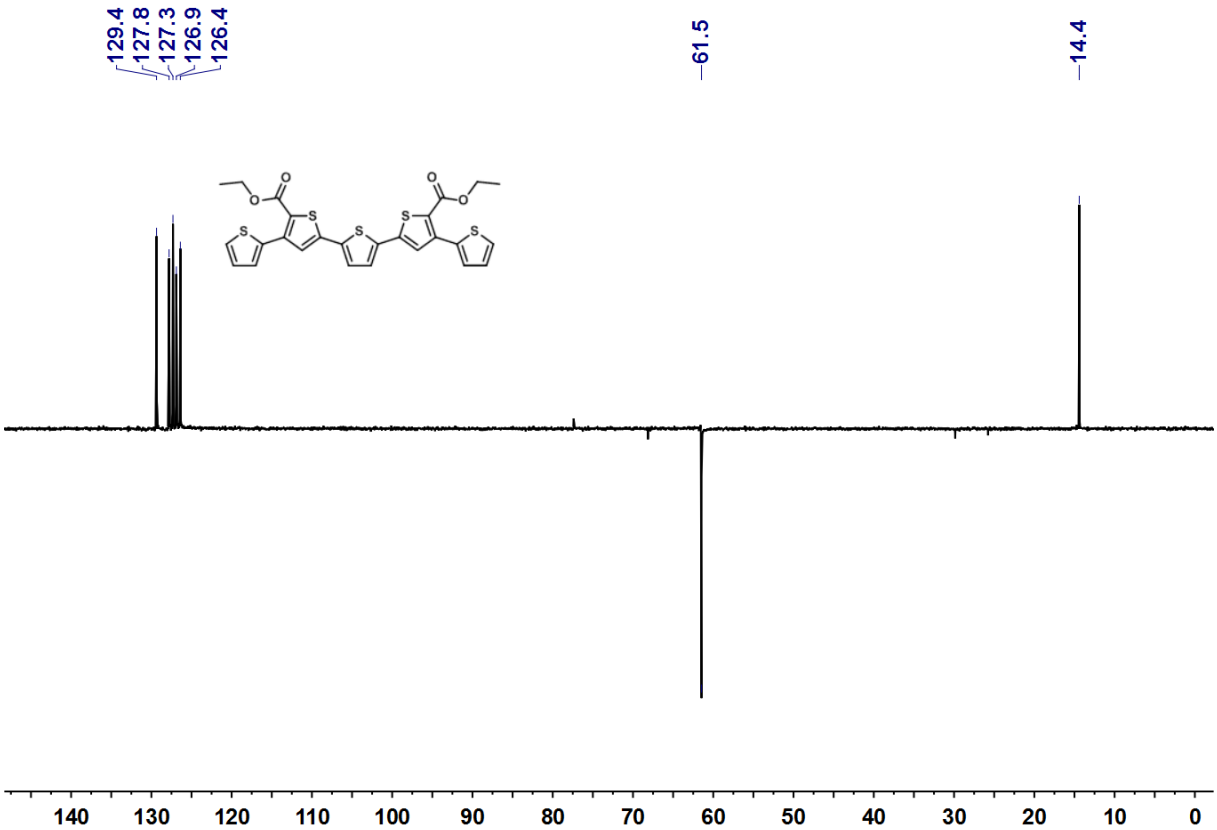
135-DEPT of **8b** ($CDCl_3$, 125 MHz, 298 K)



^{1}H-NMR of **8c** ($CDCl_3$, 500 MHz, 298 K)

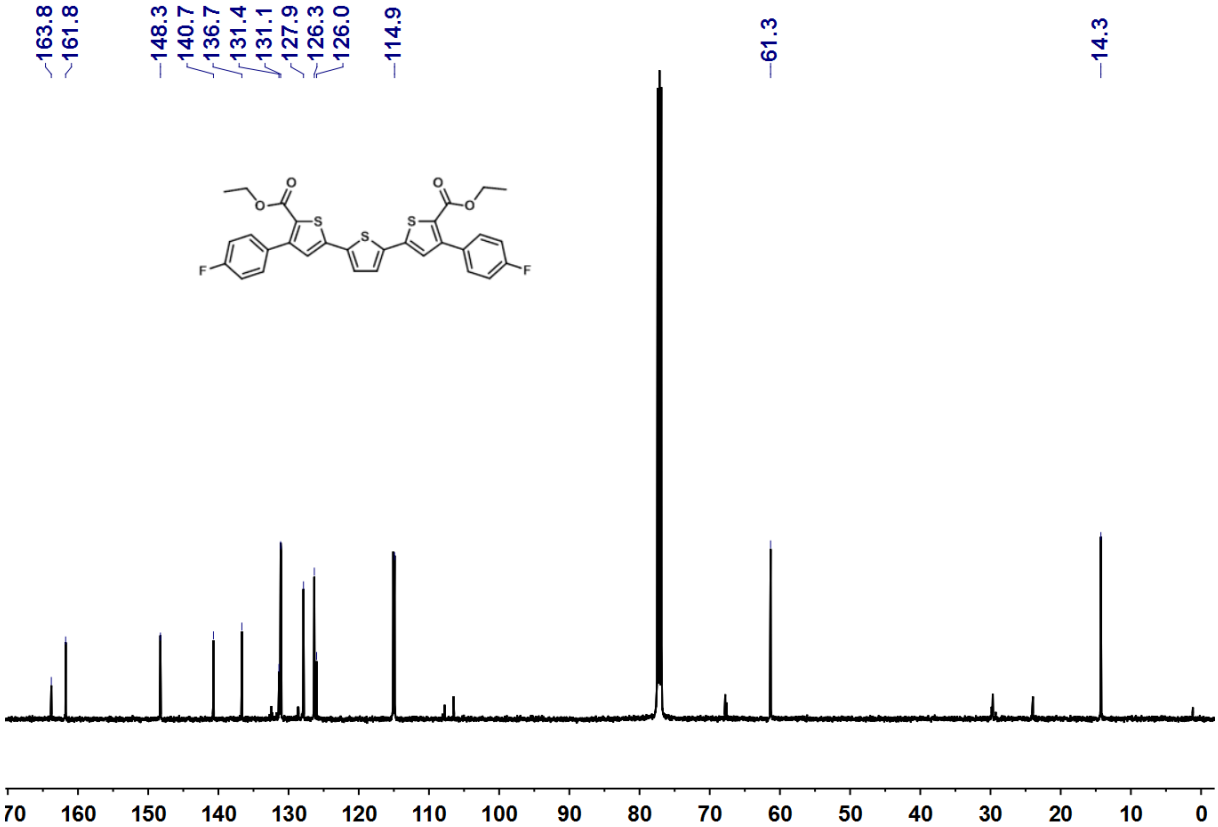


^{13}C-NMR of **8c** ($CDCl_3$, 125 MHz, 298 K)

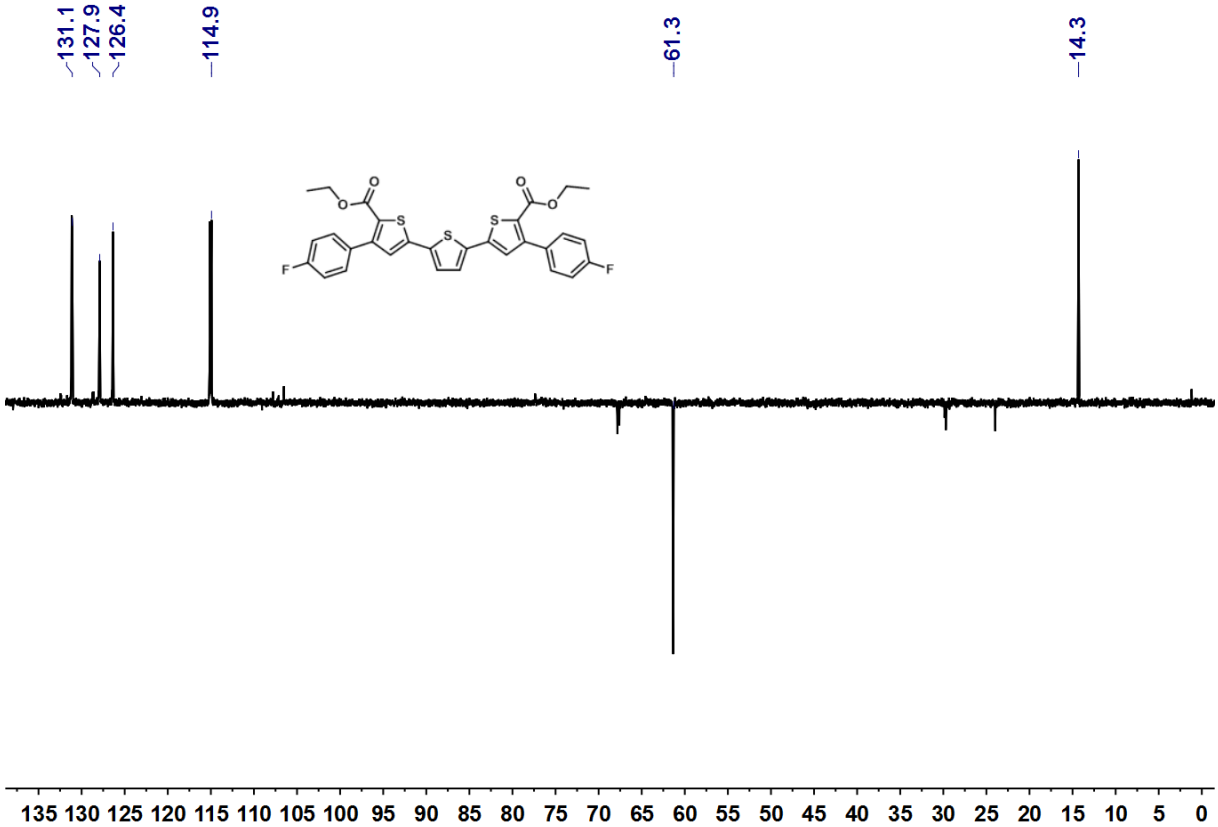


135-DEPT of **8c** ($CDCl_3$, 125 MHz, 298 K)

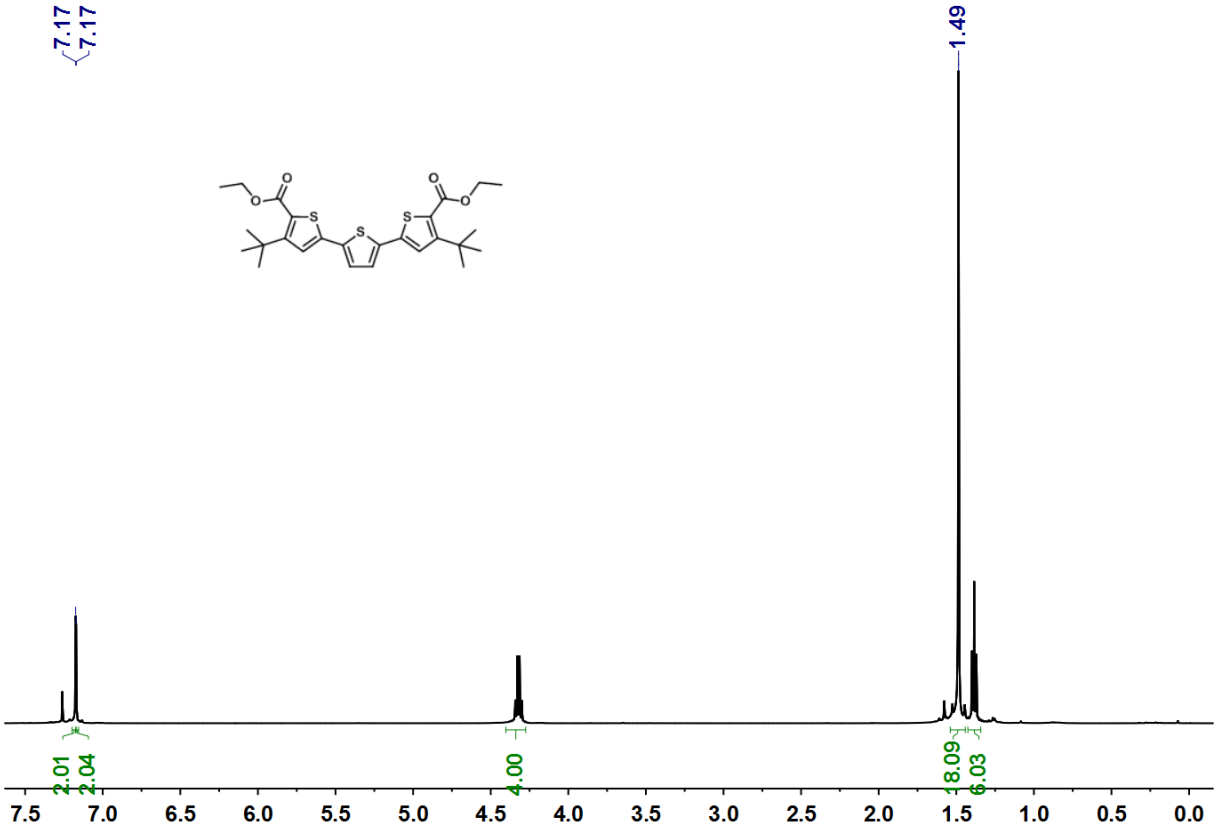
^{1}H-NMR of **8d** ($CDCl_3$, 500 MHz, 298 K)



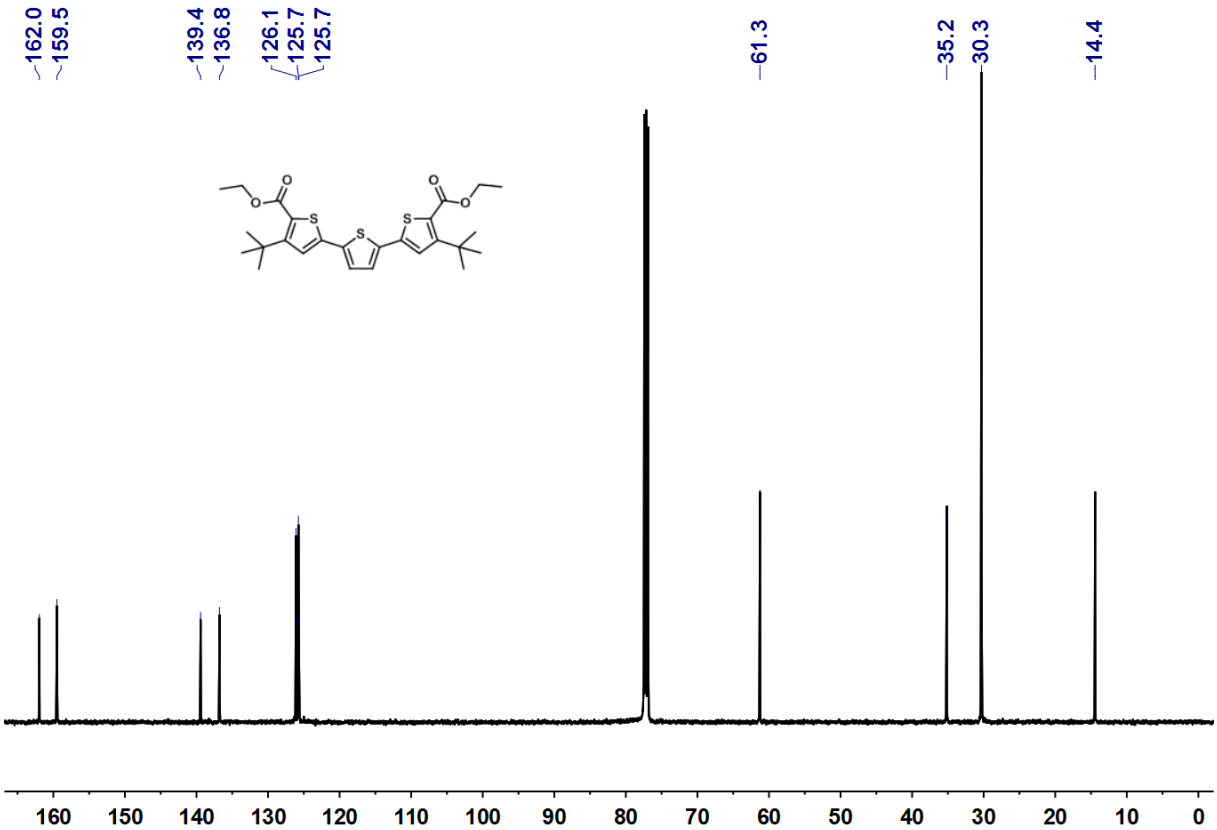
^{13}C-NMR of **8d** ($CDCl_3$, 125 MHz, 298 K)



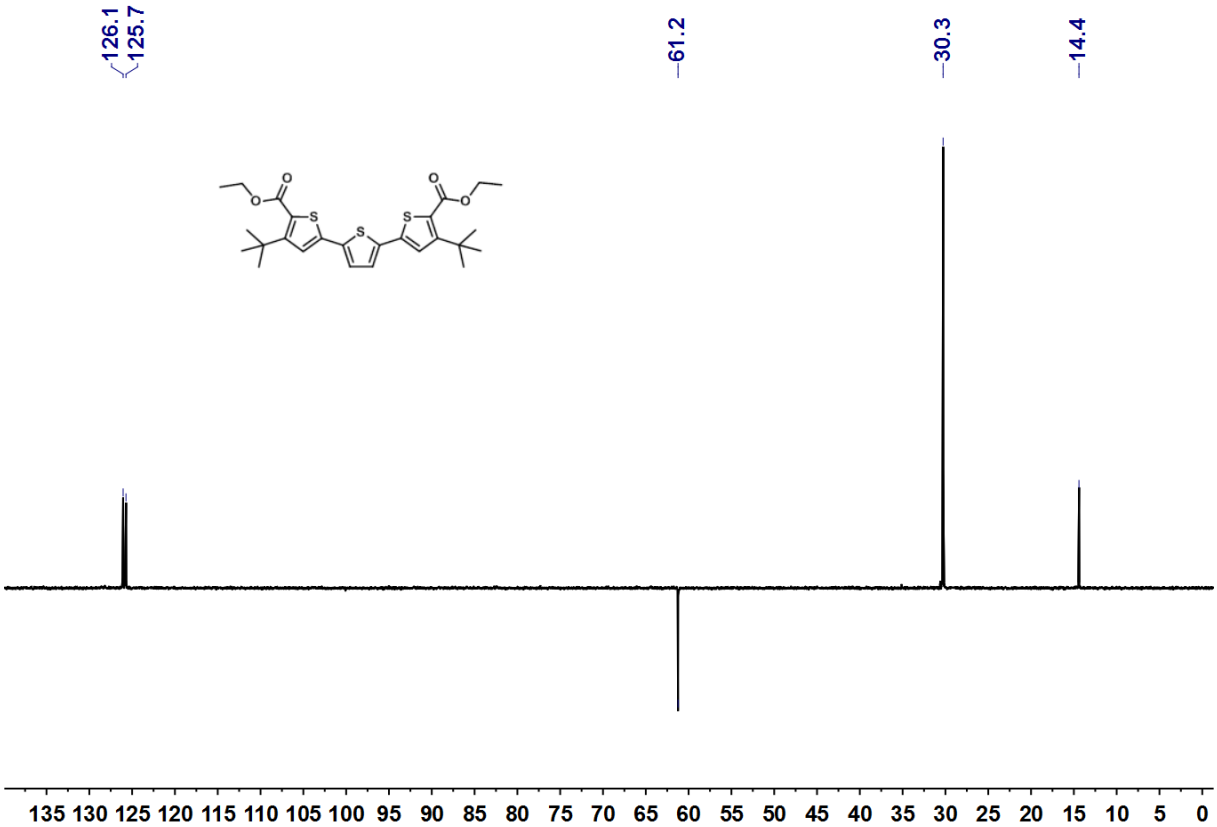
135-DEPT of **8d** ($CDCl_3$, 125 MHz, 298 K)



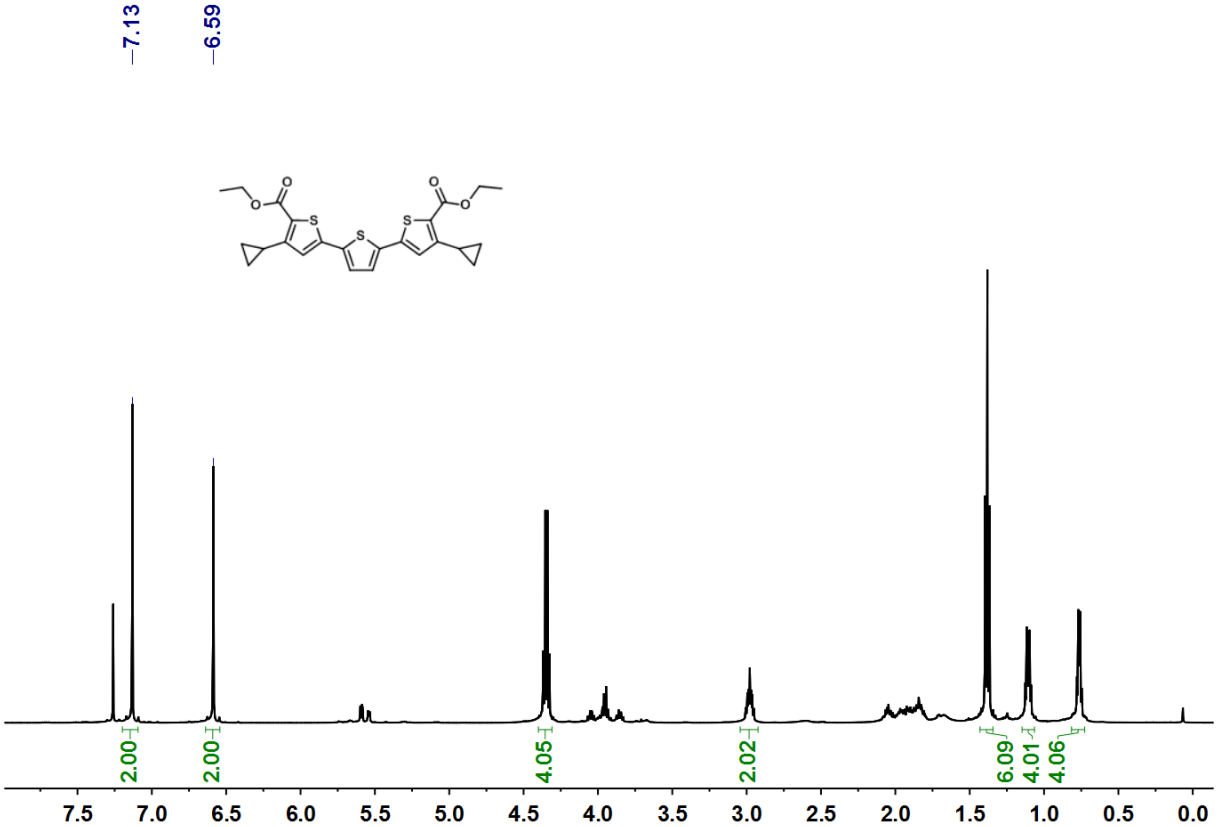
^{1}H-NMR of **8e** ($CDCl_3$, 500 MHz, 298 K)



^{13}C-NMR of **8e** ($CDCl_3$, 125 MHz, 298 K)



135-DEPT of **8e** ($CDCl_3$, 125 MHz, 298 K)



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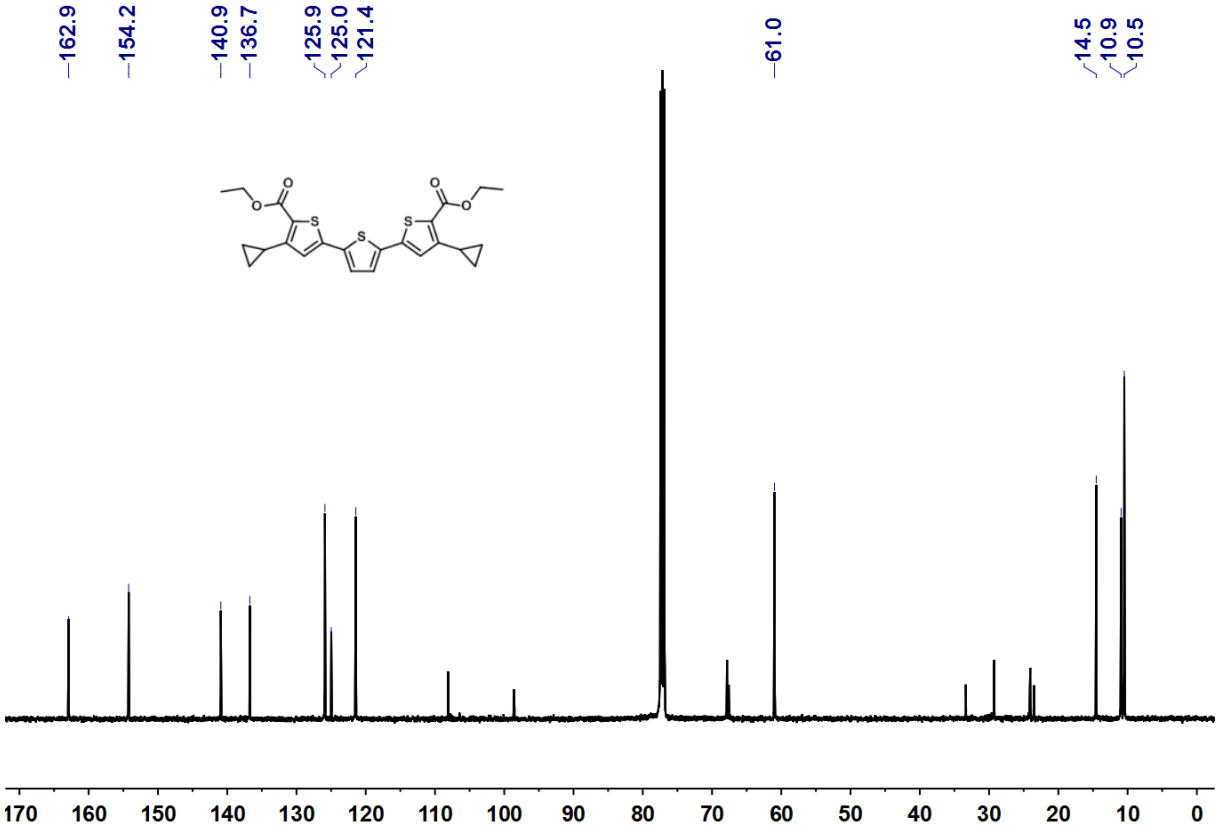
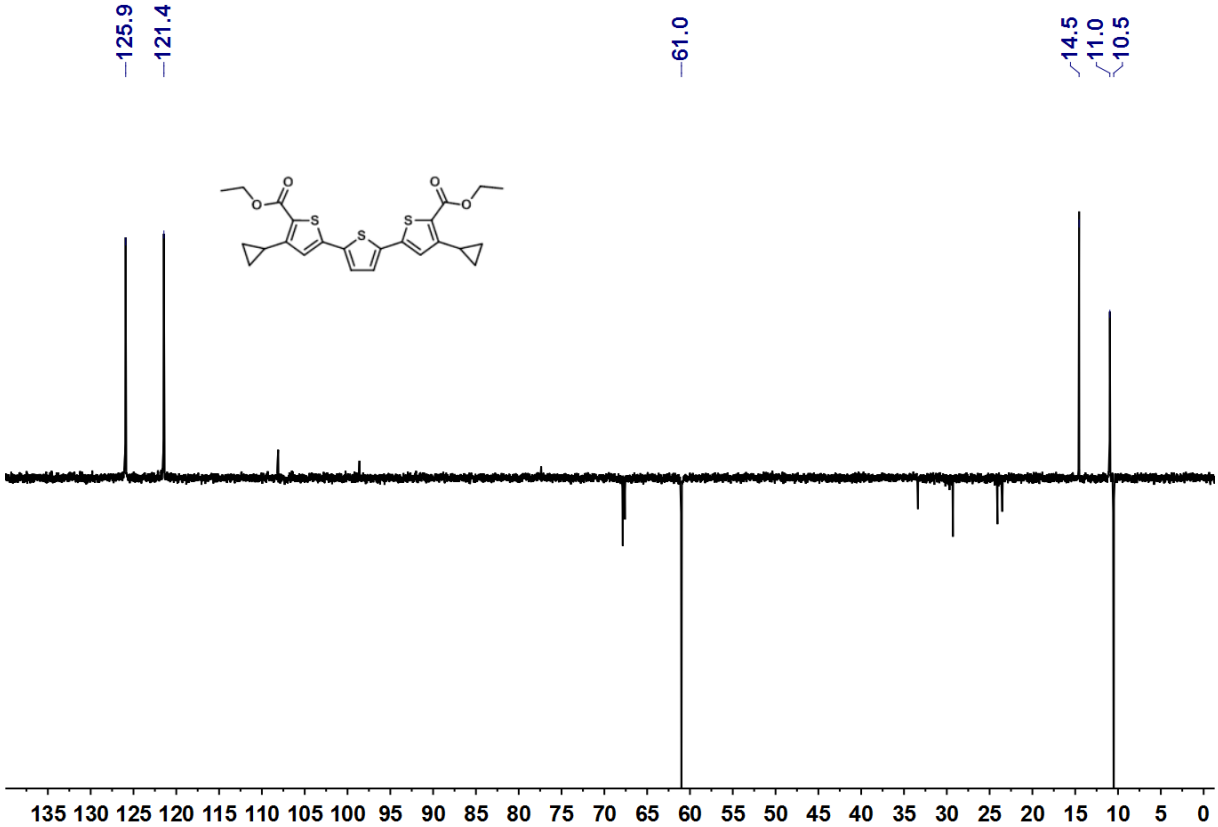
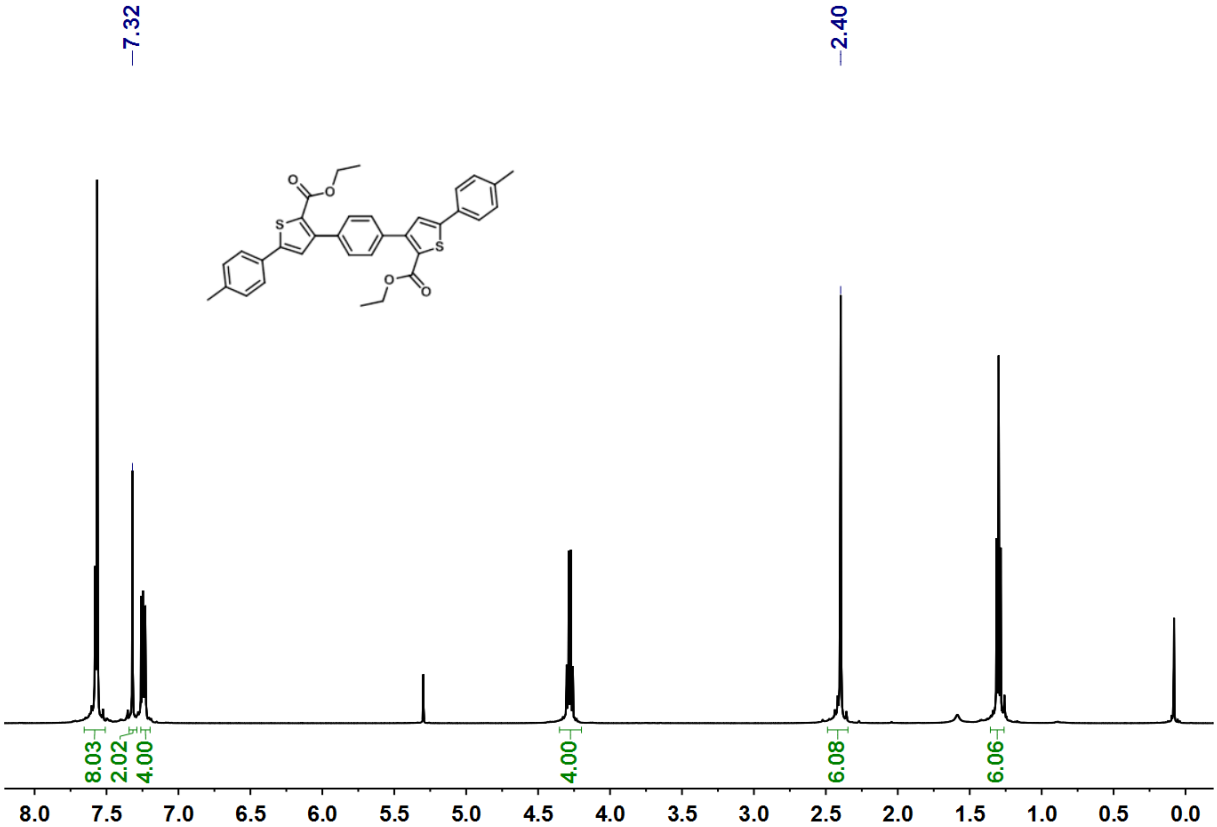


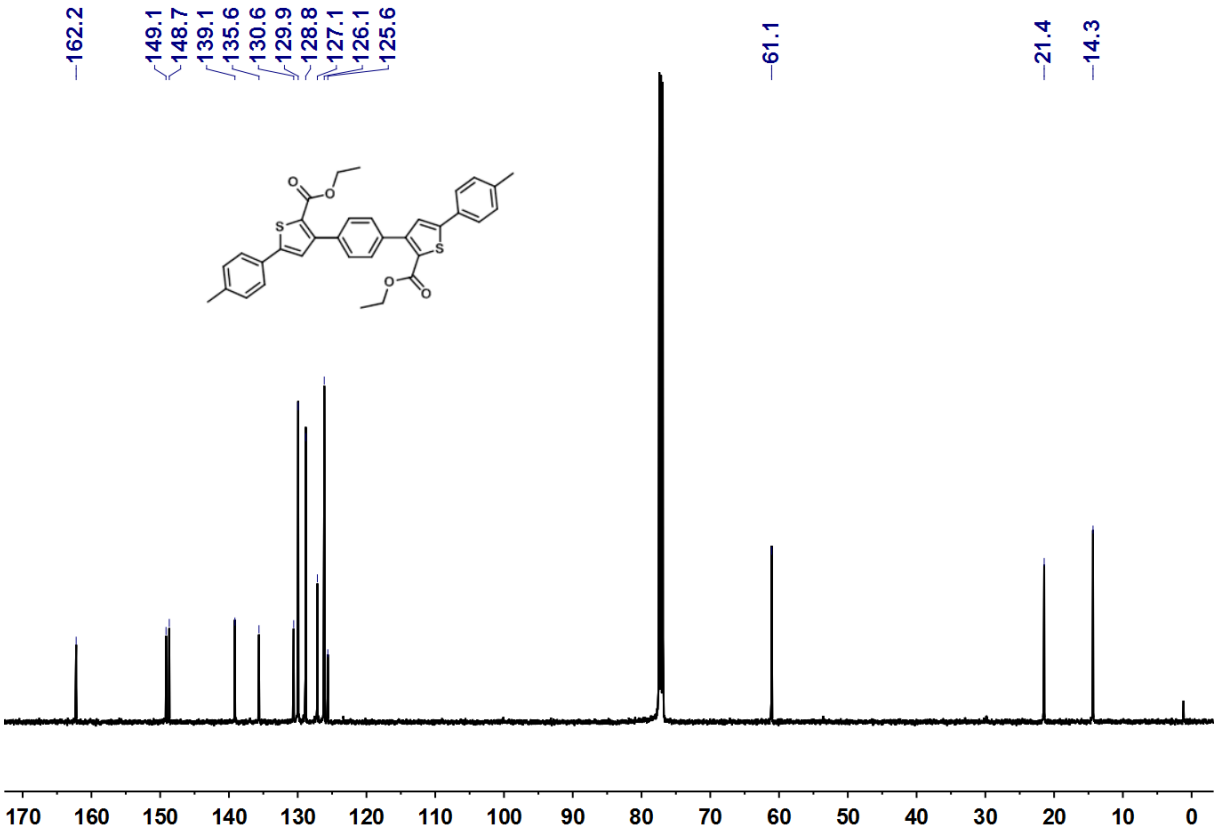
^{13}C-NMR of **8f** ($CDCl_3$, 125 MHz, 298 K)



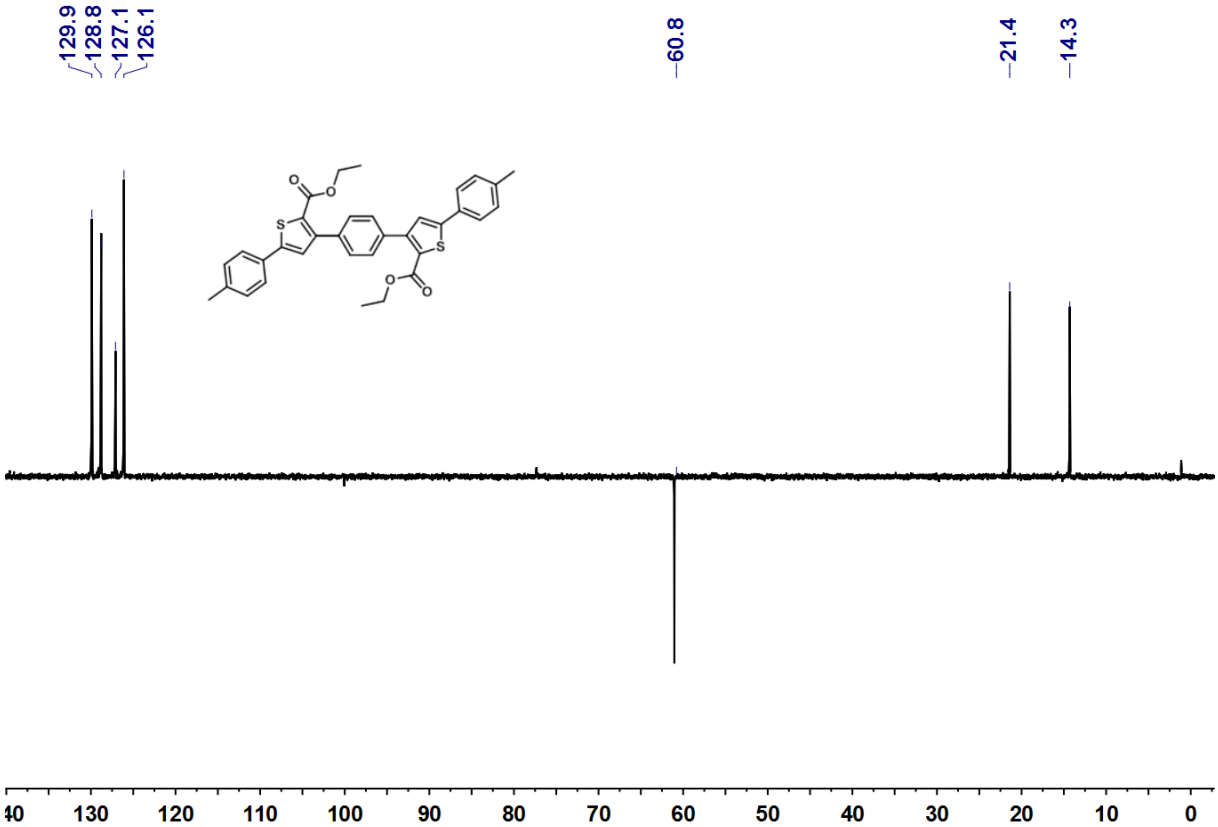
135-DEPT of **8f** ($CDCl_3$, 125 MHz, 298 K)



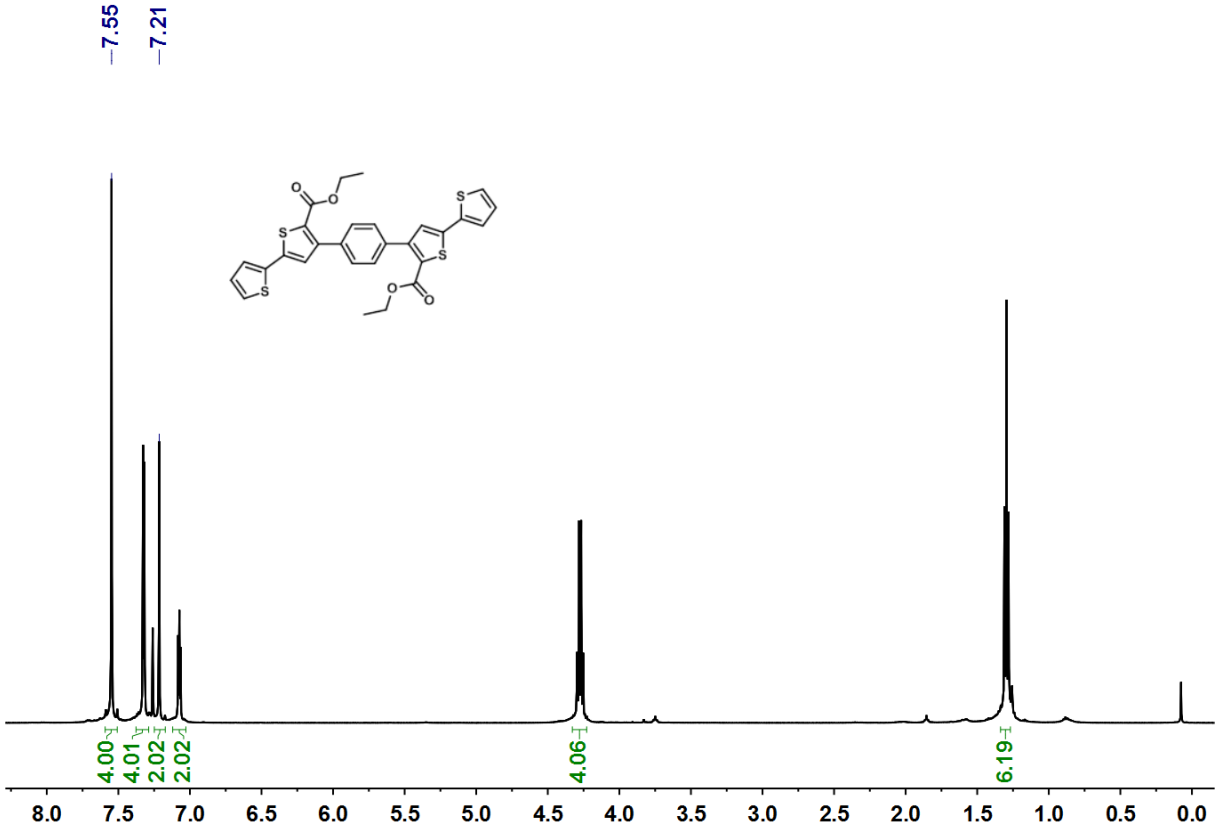
^{1}H-NMR of **10a** ($CDCl_3$, 500 MHz, 298 K)



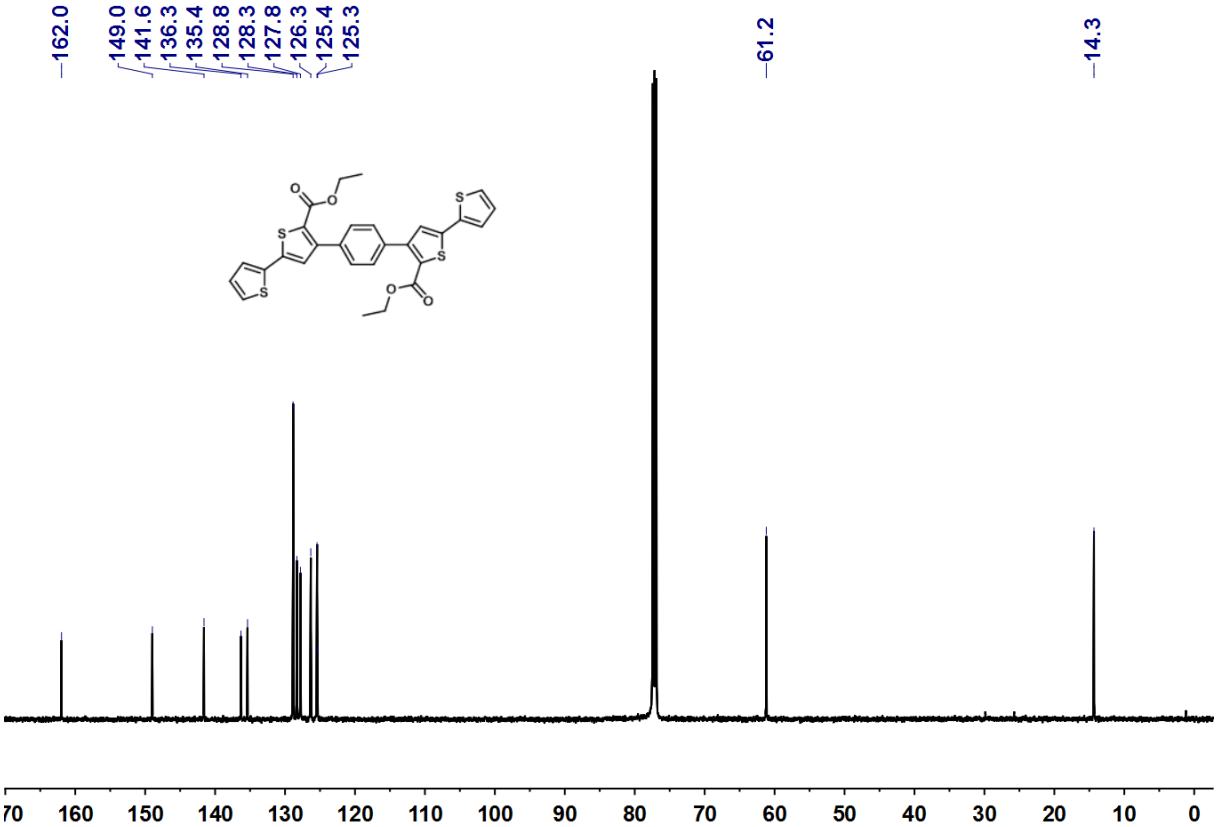
^{13}C-NMR of **10a** ($CDCl_3$, 125 MHz, 298 K)



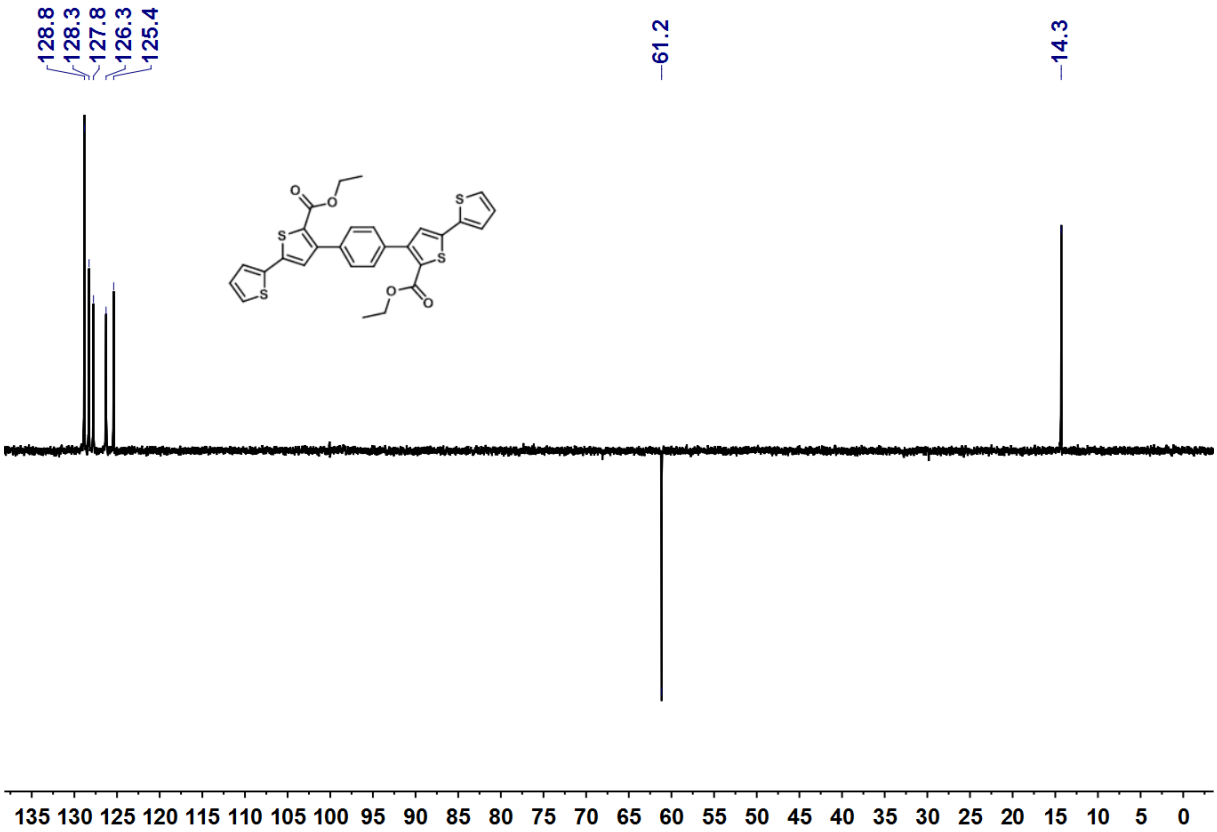
135-DEPT of **10a** ($CDCl_3$, 125 MHz, 298 K)



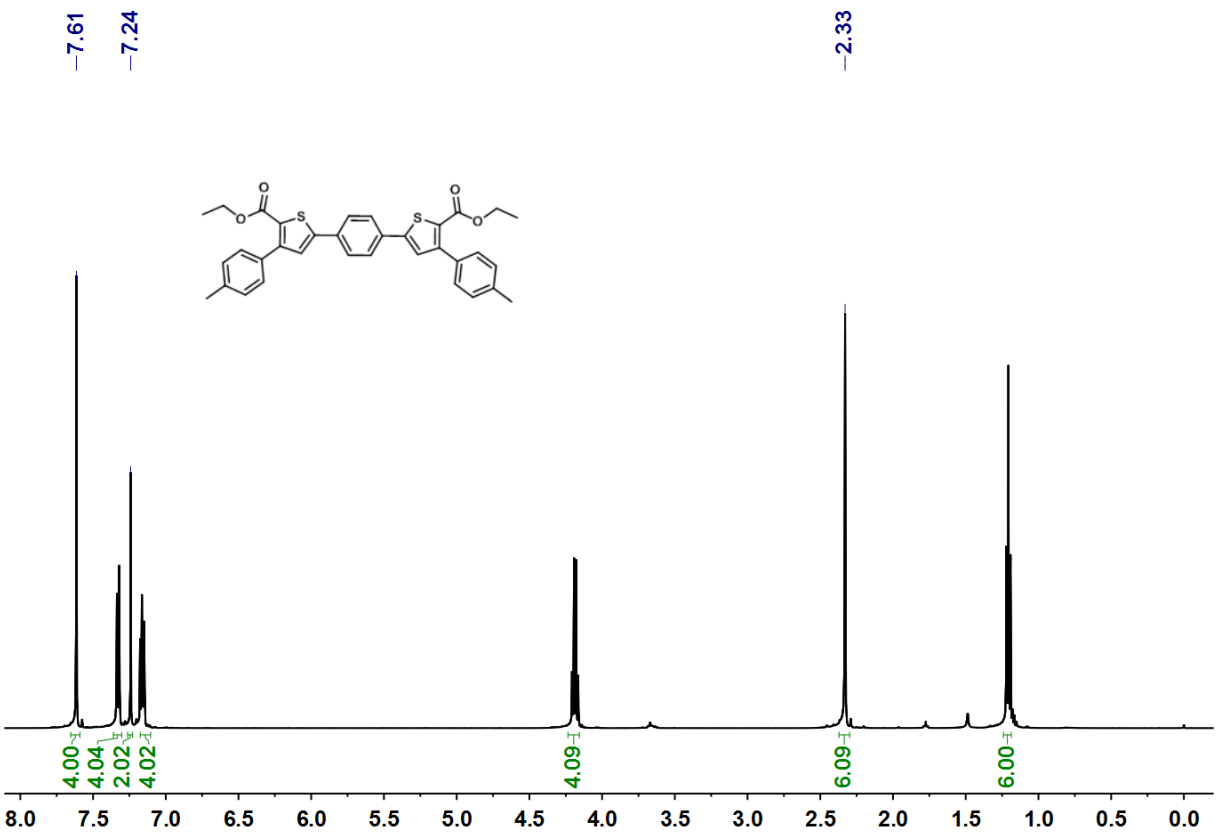
^{1}H-NMR of **10b** ($CDCl_3$, 500 MHz, 298 K)



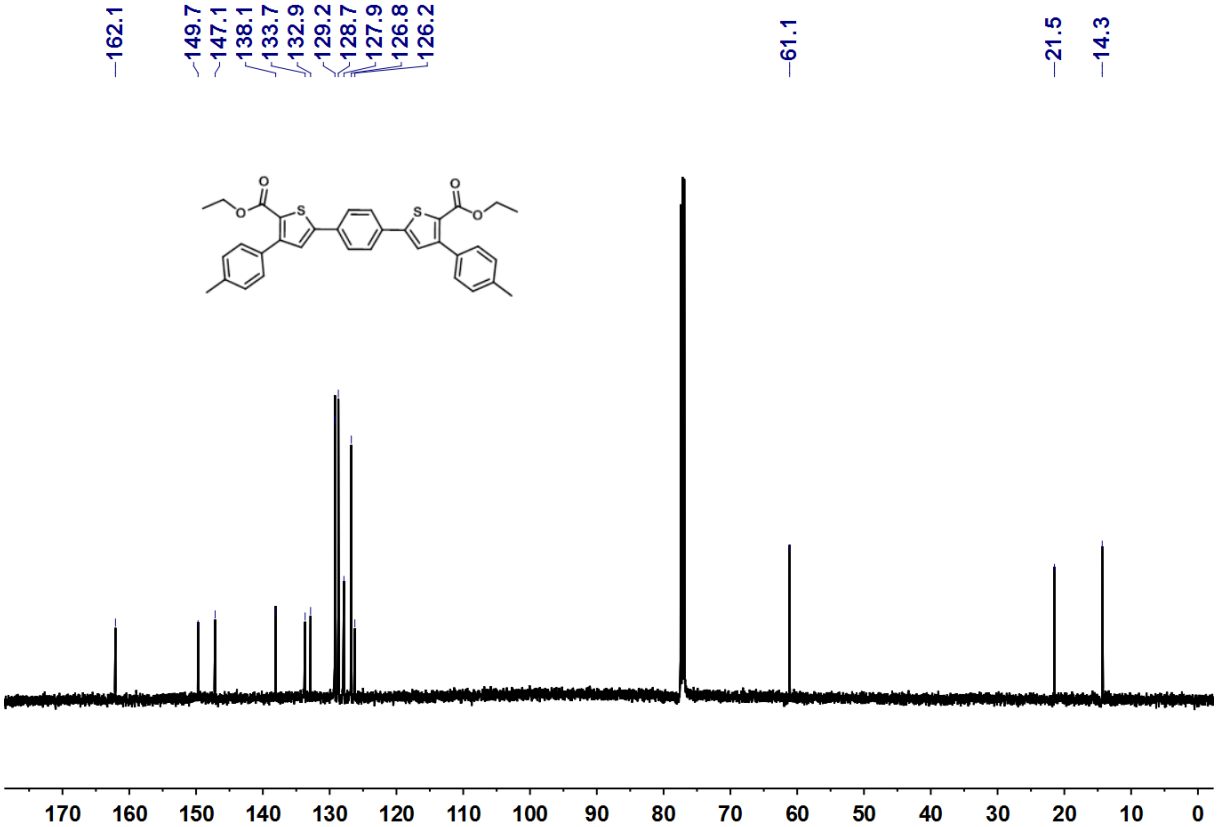
^{13}C-NMR of **10b** ($CDCl_3$, 125 MHz, 298 K)



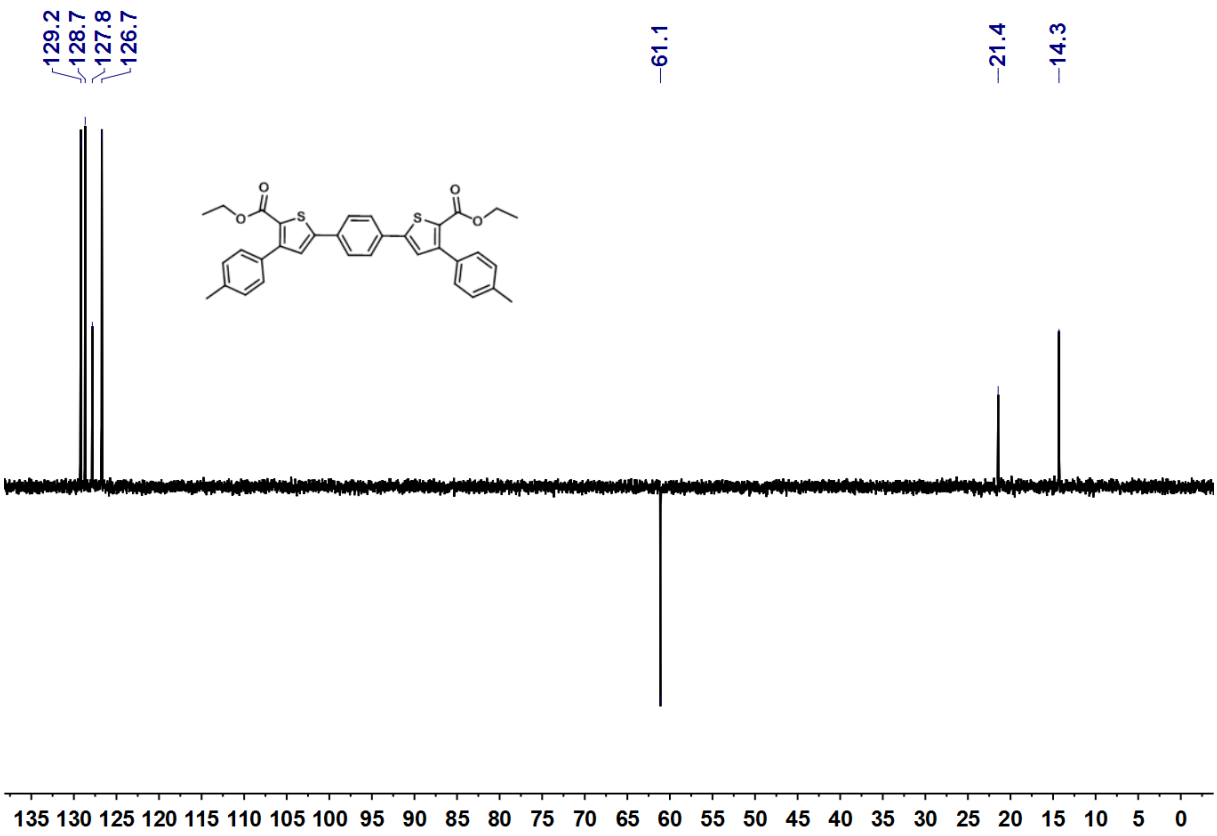
135-DEPT of **10b** ($CDCl_3$, 125 MHz, 298 K)



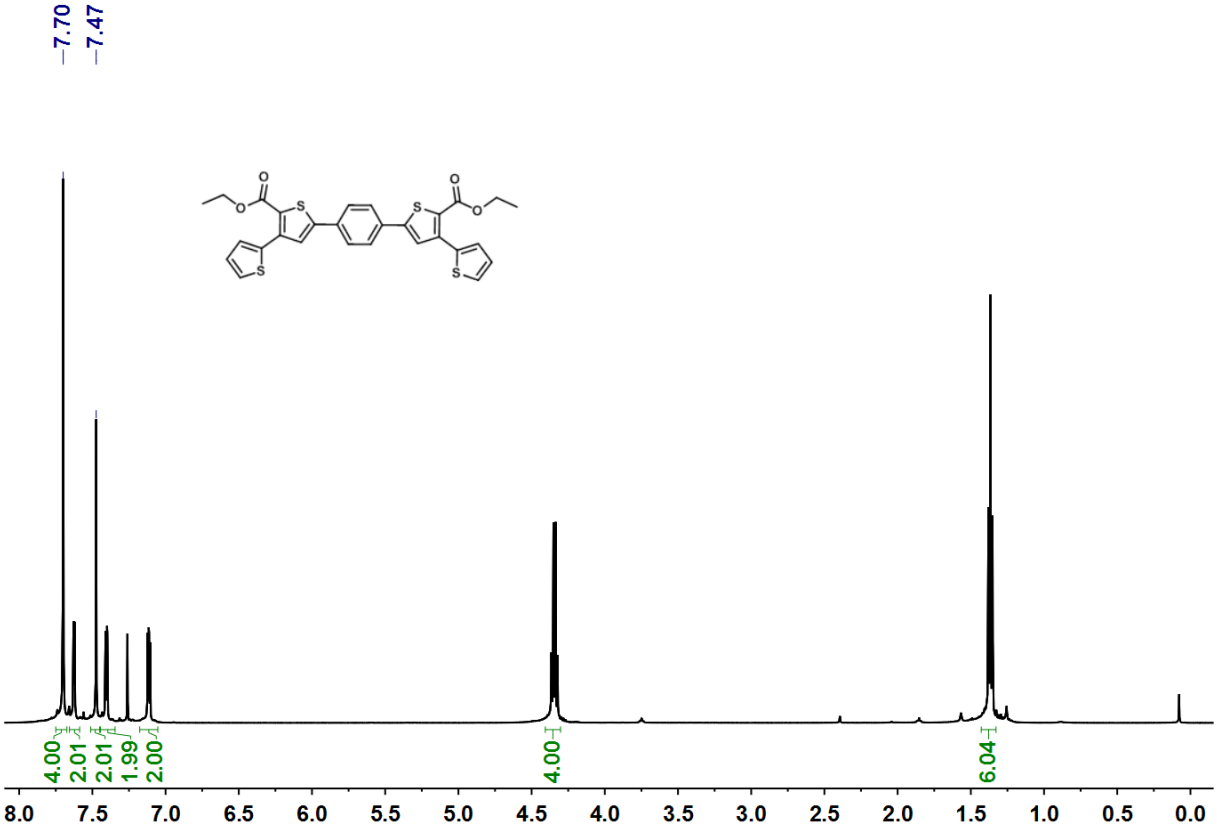
^{1}H-NMR of **12a** ($CDCl_3$, 500 MHz, 298 K)



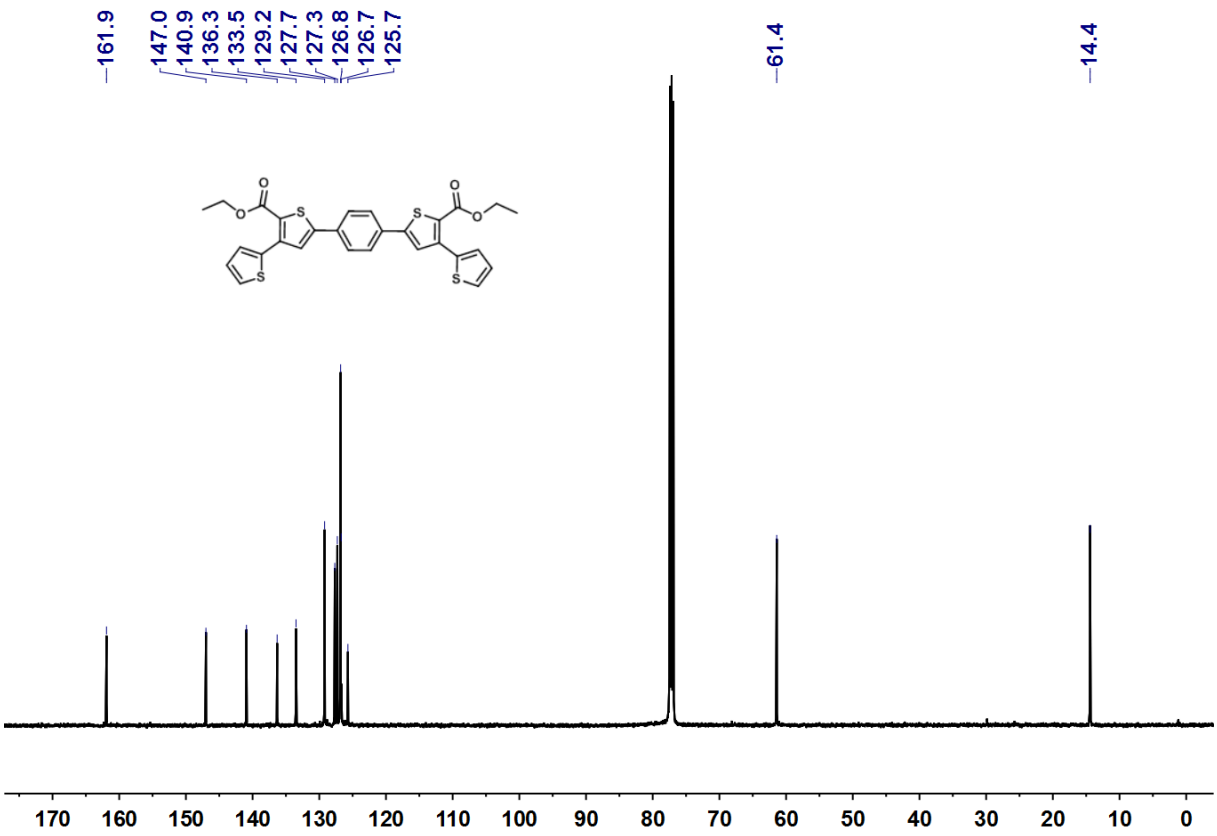
^{13}C-NMR of **12a** ($CDCl_3$, 125 MHz, 298 K)



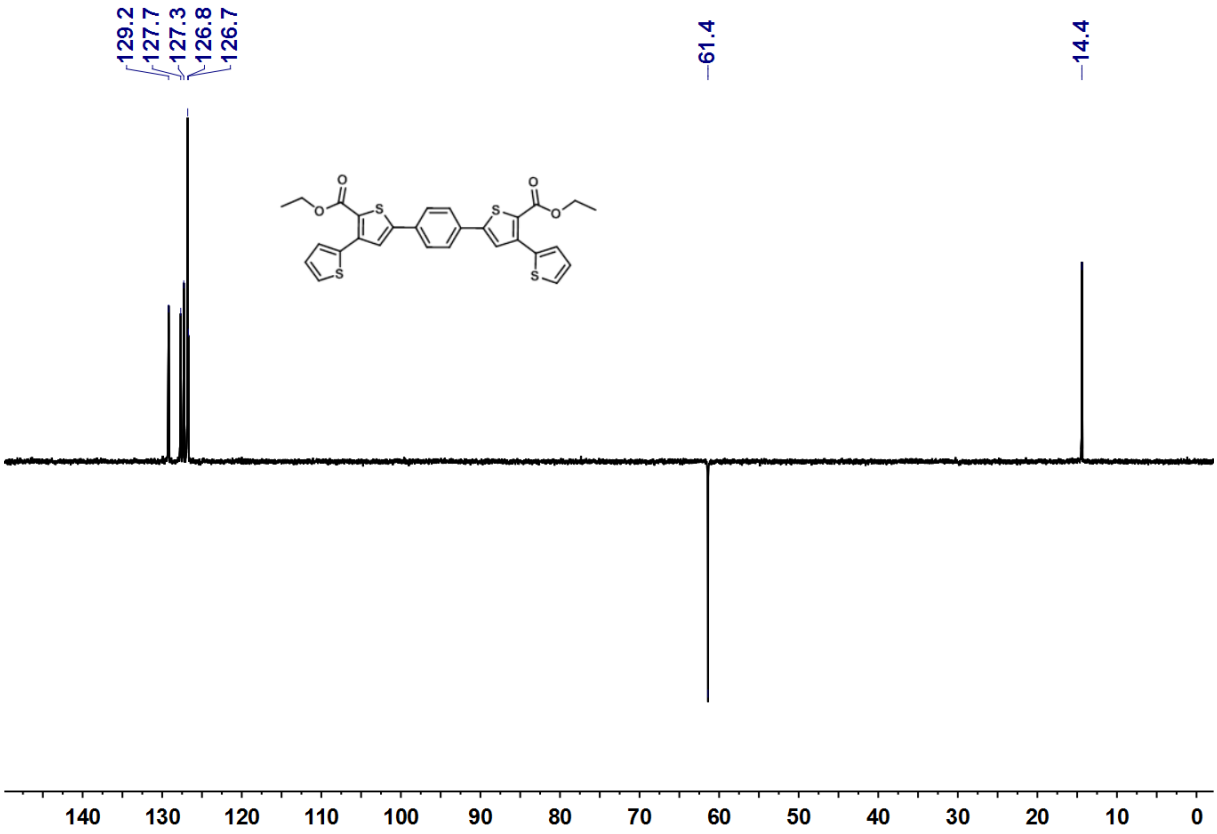
135-DEPT of **12a** ($CDCl_3$, 125 MHz, 298 K)



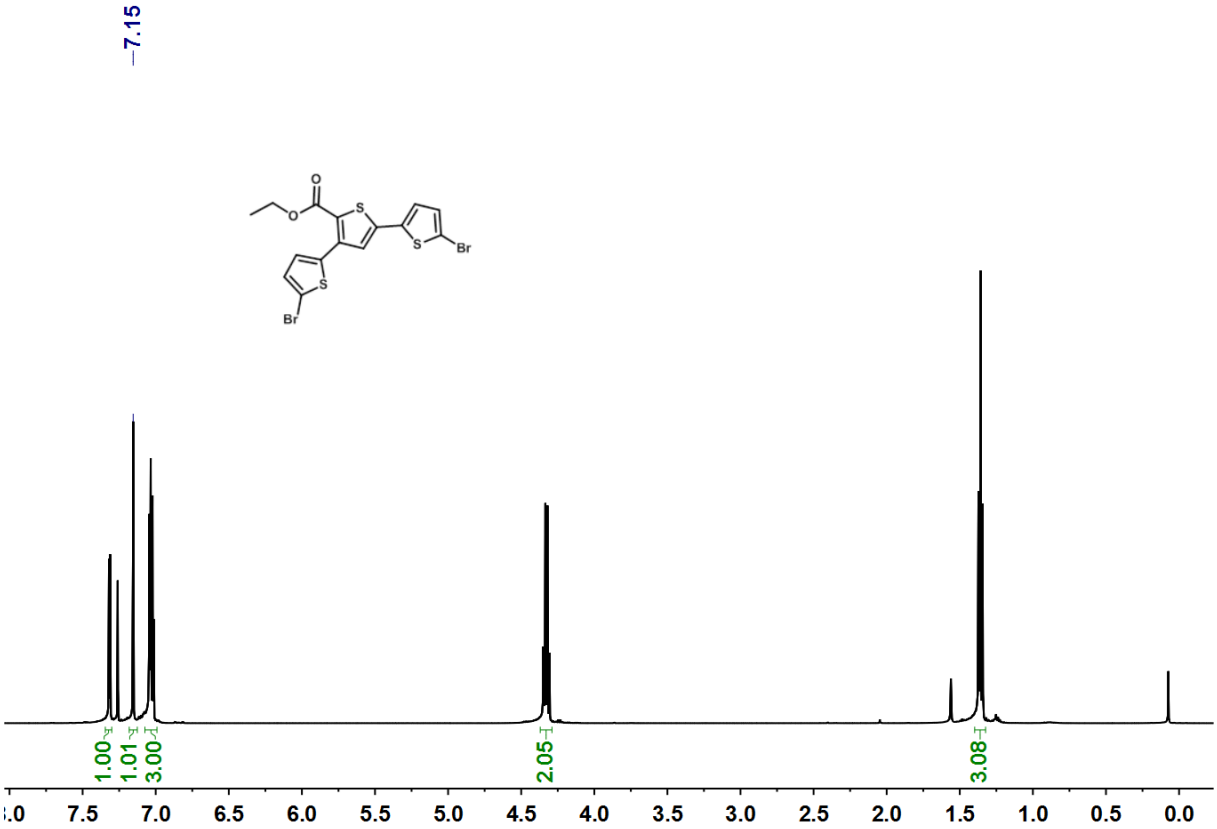
^{1}H-NMR of **12b** ($CDCl_3$, 500 MHz, 298 K)



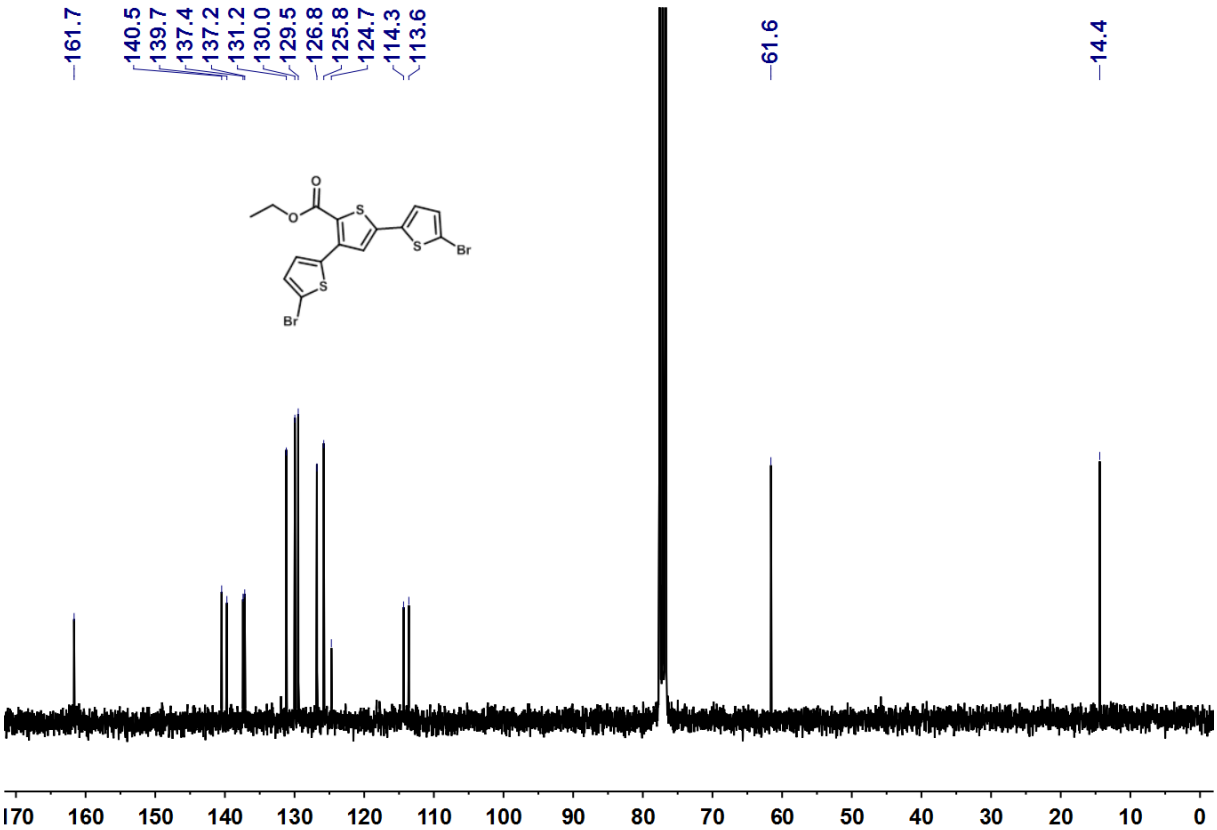
^{13}C-NMR of **12b** ($CDCl_3$, 125 MHz, 298 K)



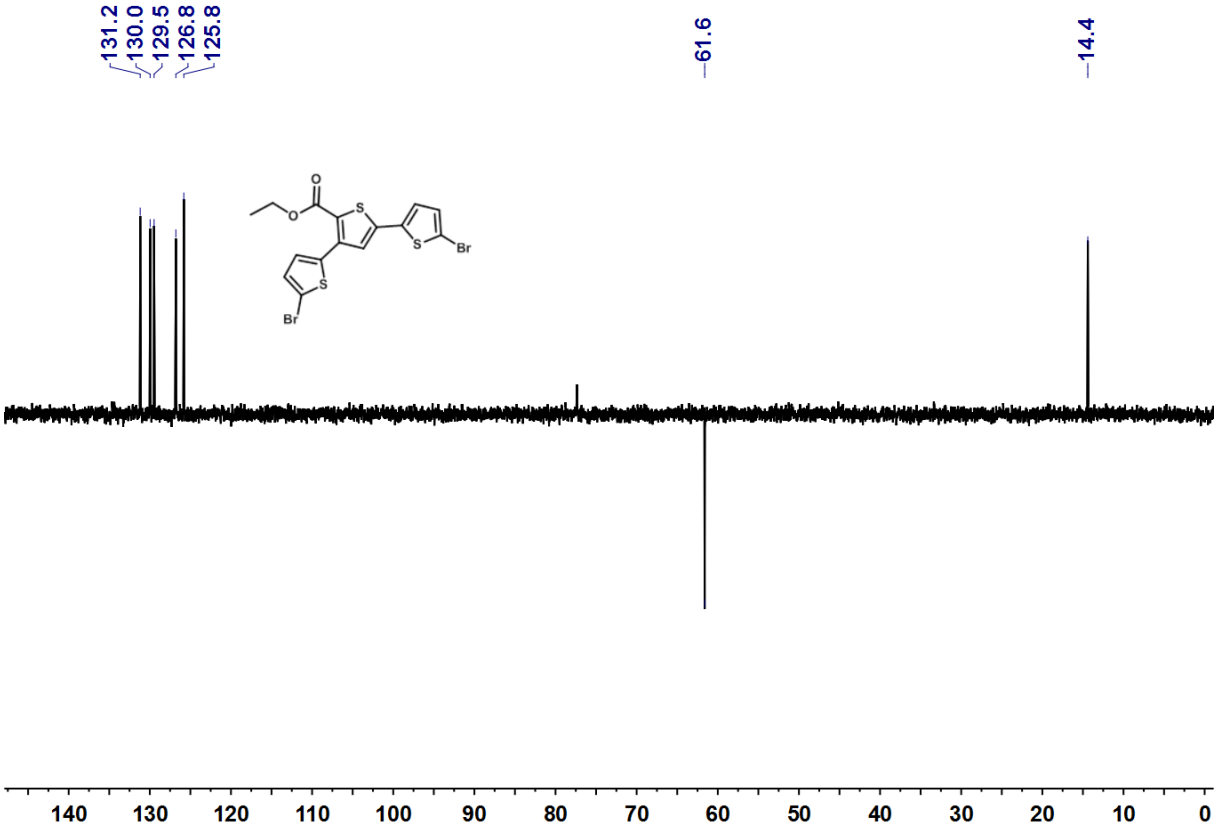
135-DEPT of **12b** ($CDCl_3$, 125 MHz, 298 K)



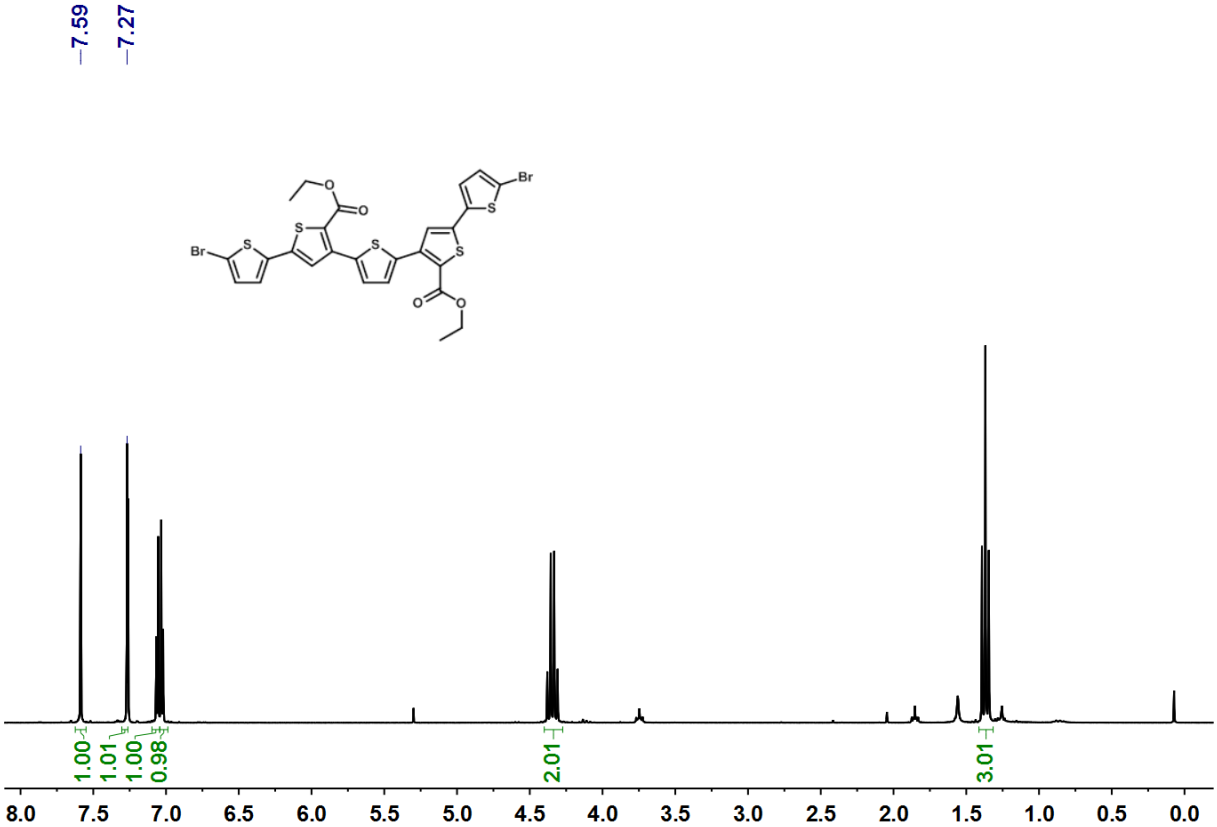
^{1}H-NMR of **13** ($CDCl_3$, 500 MHz, 298 K)



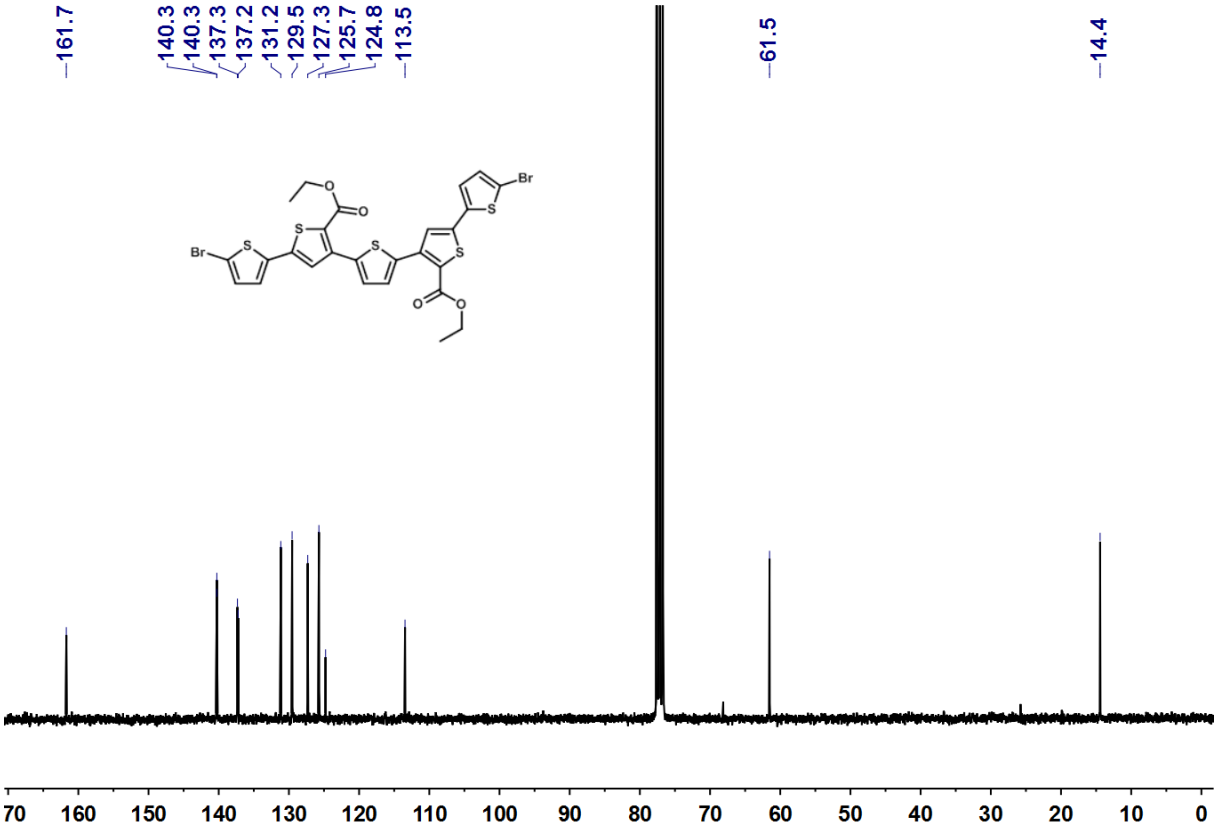
^{13}C-NMR of **13** ($CDCl_3$, 125 MHz, 298 K)



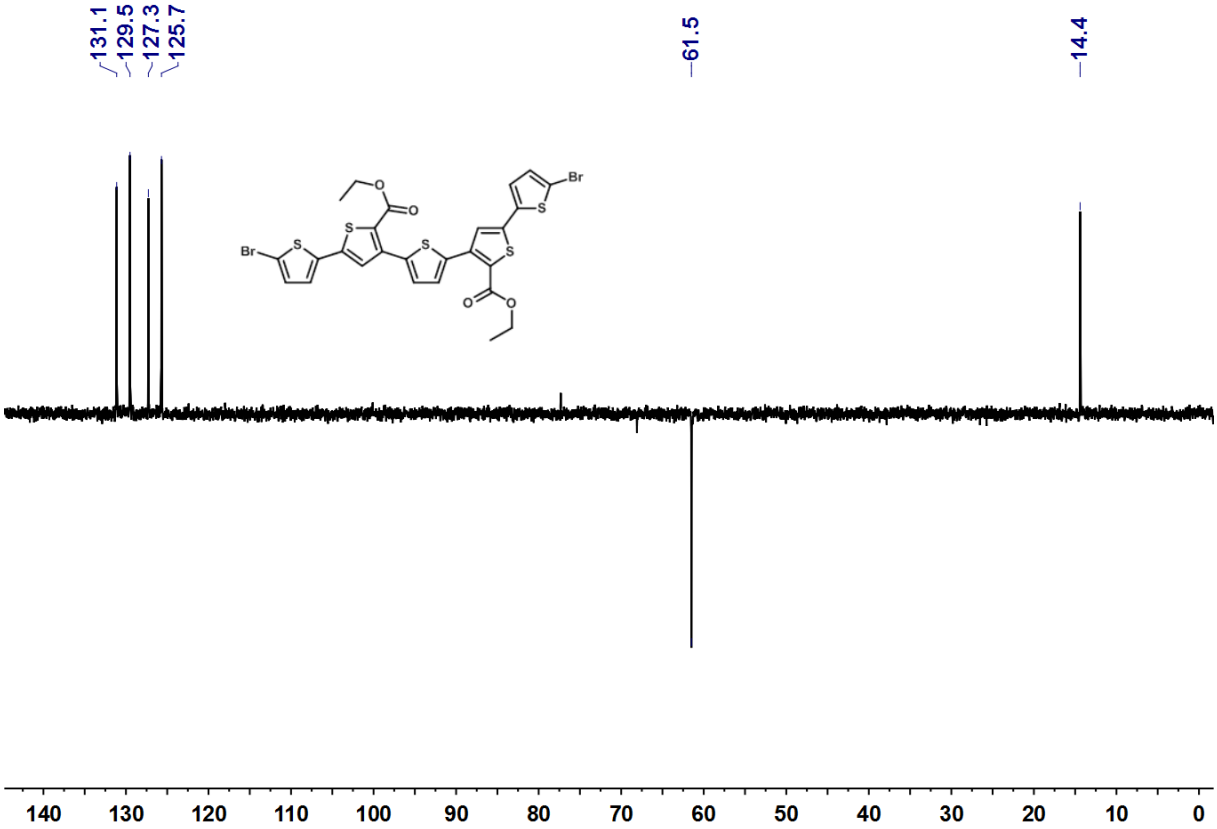
135-DEPT of **13** ($CDCl_3$, 125 MHz, 298 K)



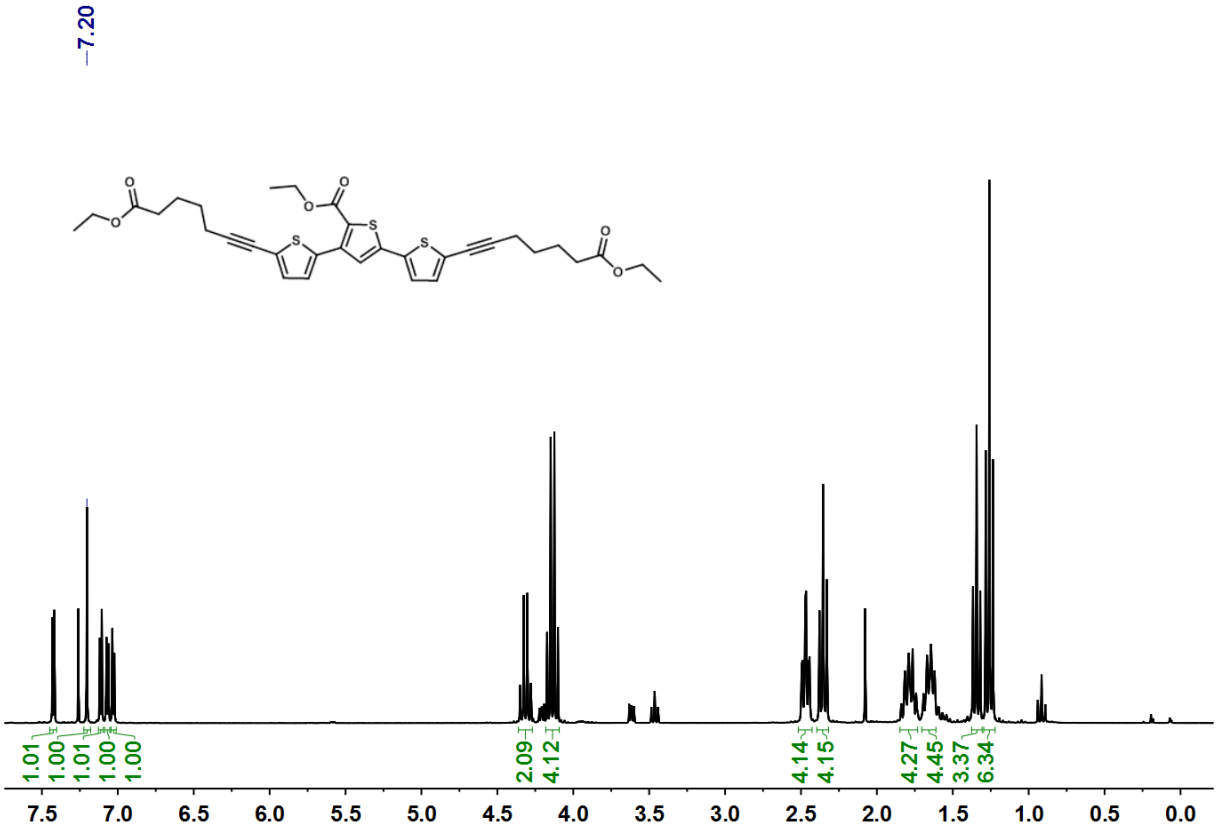
^{1}H-NMR of **14** ($CDCl_3$, 500 MHz, 298 K)



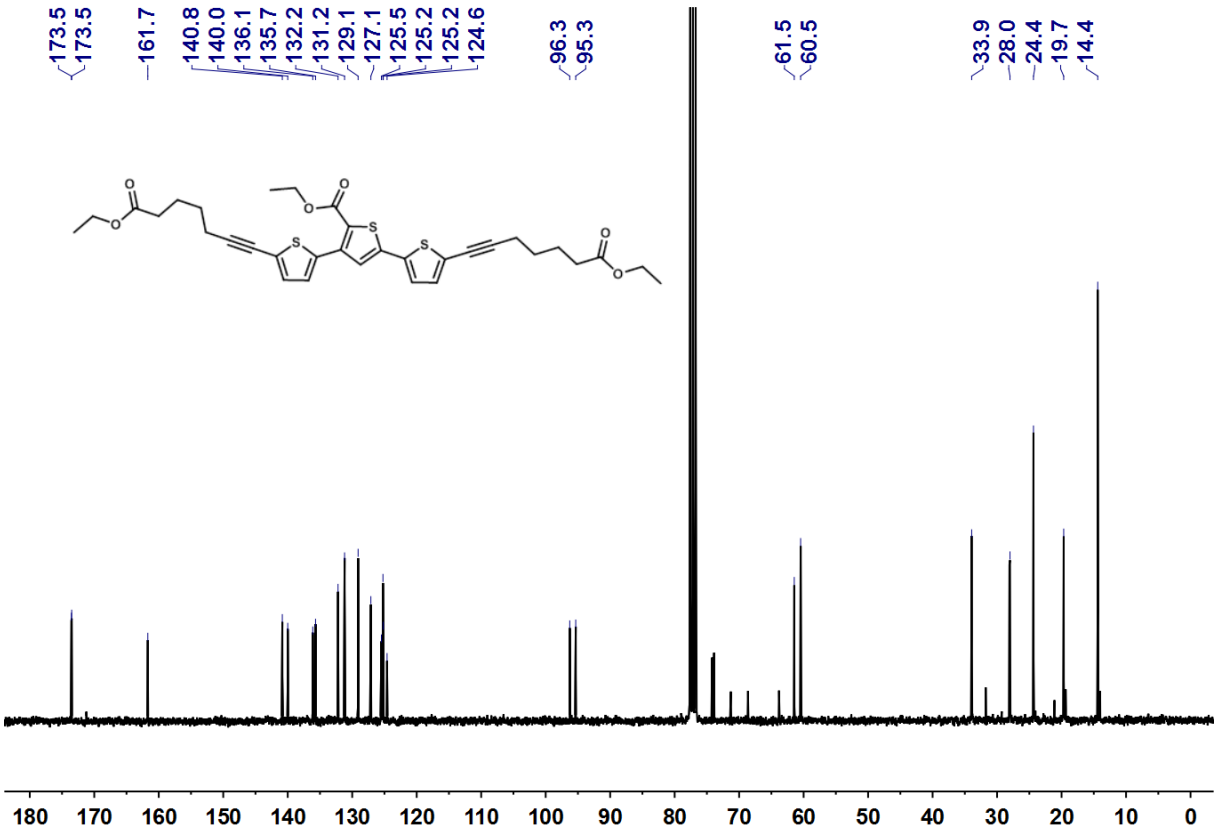
^{13}C-NMR of **14** ($CDCl_3$, 125 MHz, 298 K)



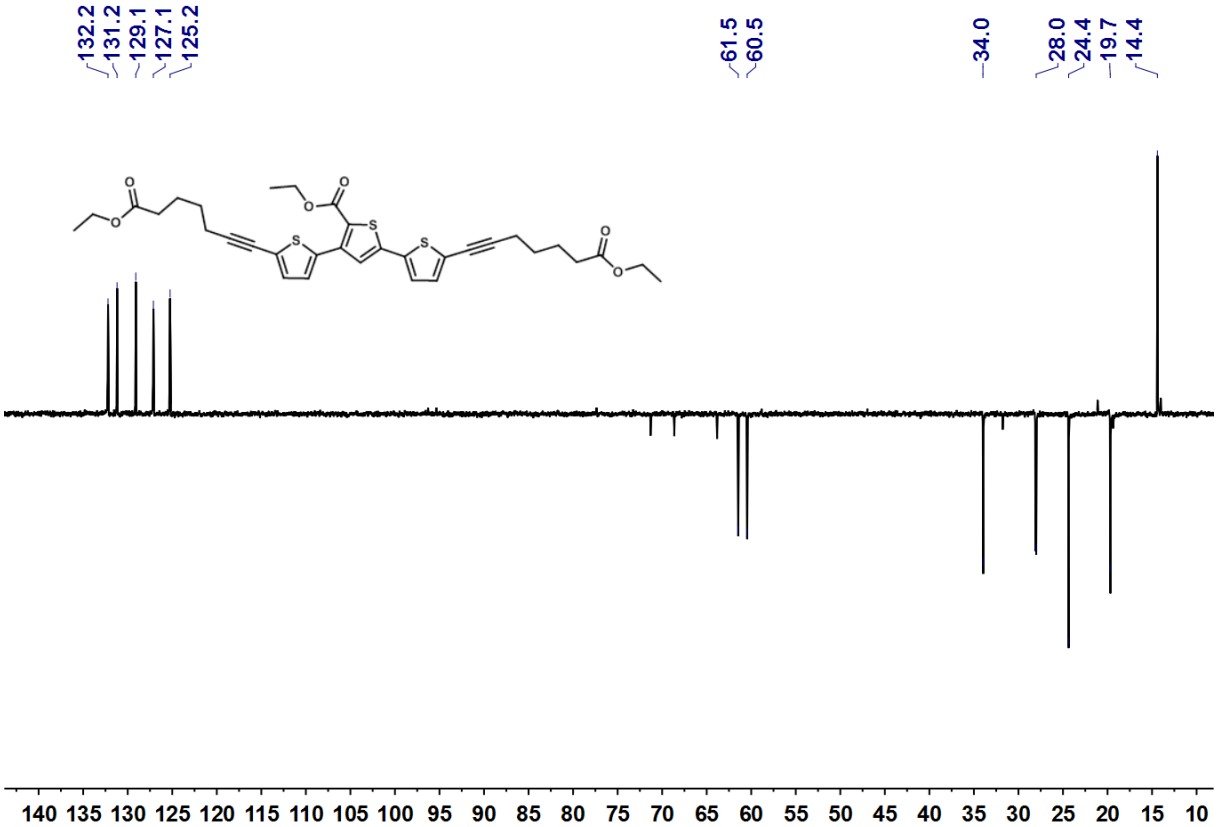
135-DEPT of **14** ($CDCl_3$, 125 MHz, 298 K)



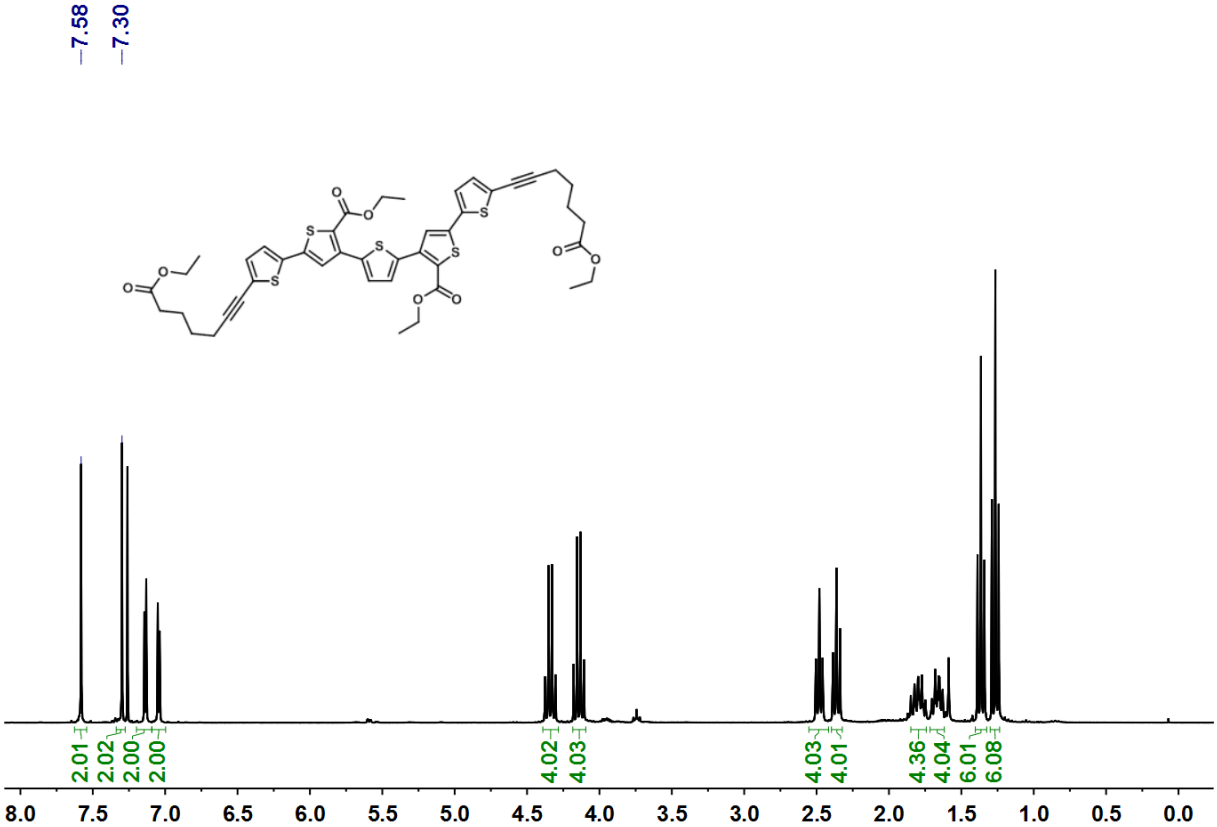
^{1}H-NMR of **15** ($CDCl_3$, 500 MHz, 298 K)



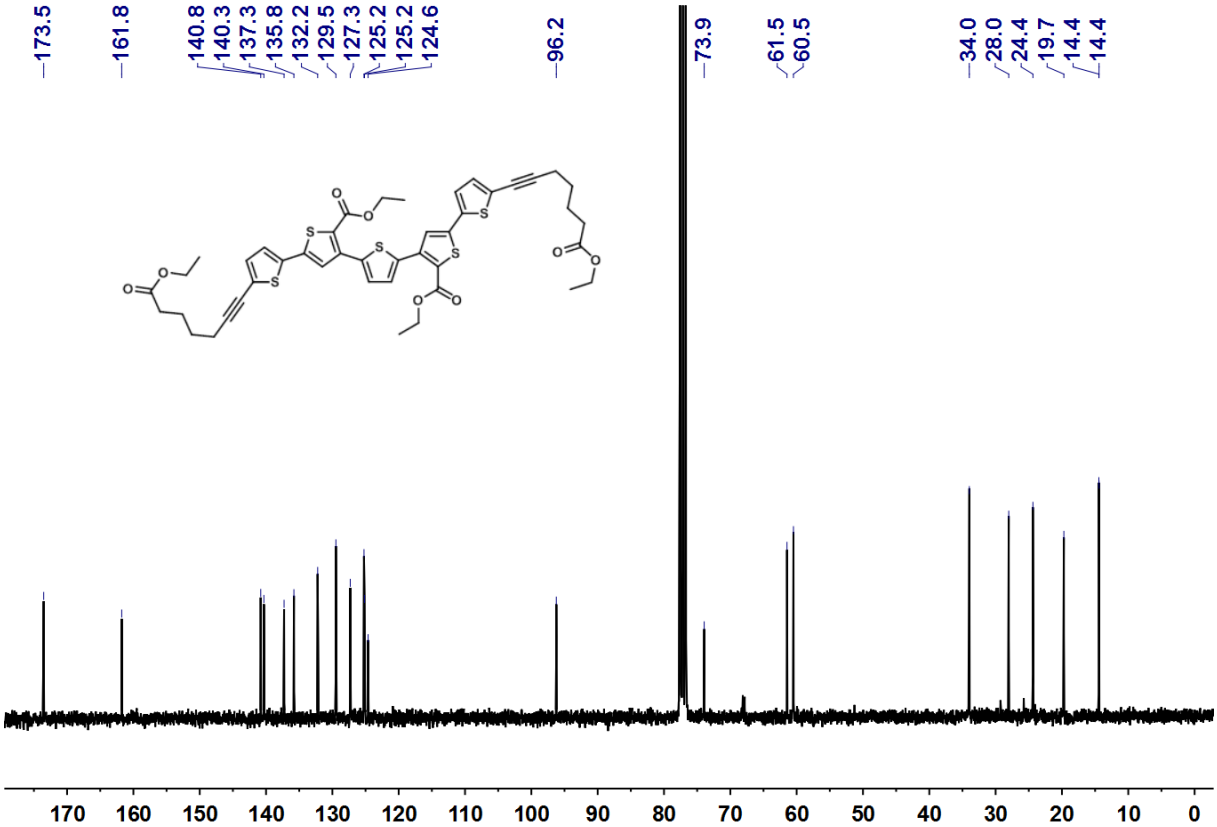
^{13}C-NMR of **15** ($CDCl_3$, 125 MHz, 298 K)



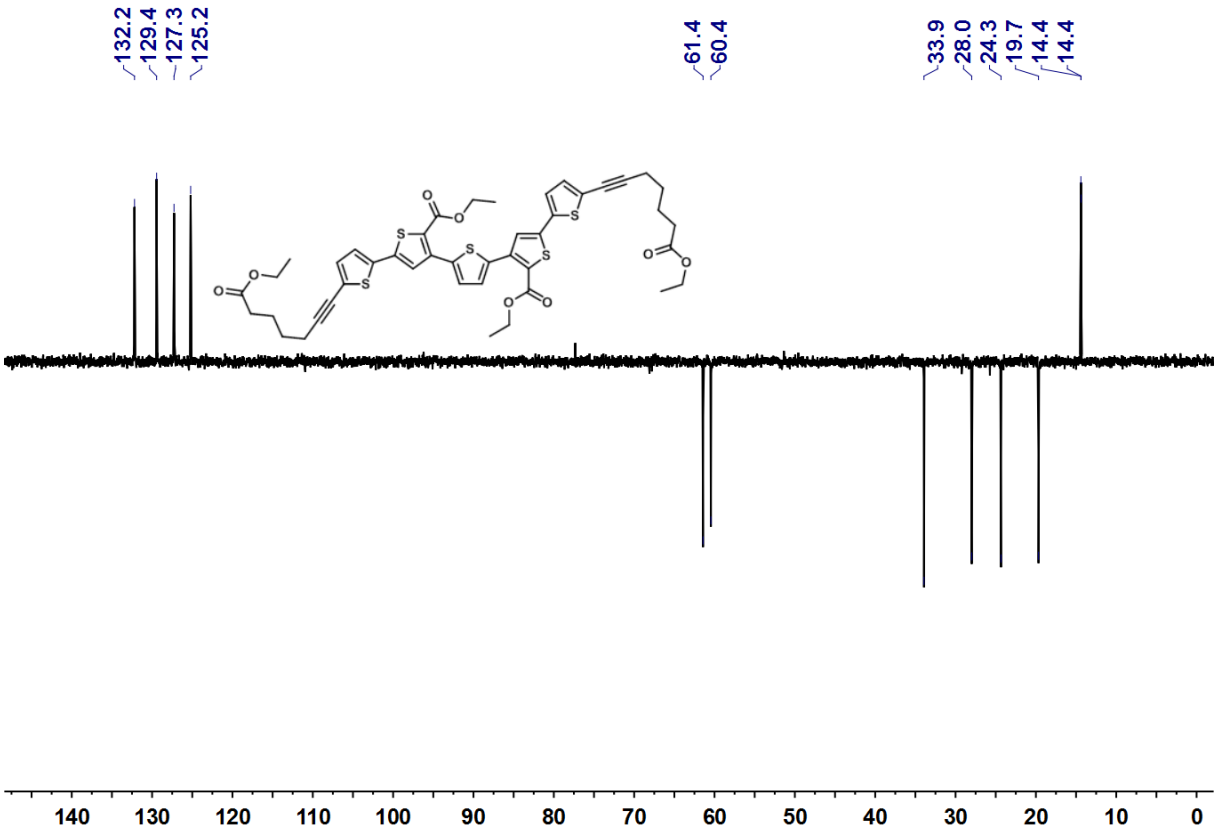
135-DEPT of **15** ($CDCl_3$, 125 MHz, 298 K)



^{1}H-NMR of **16** ($CDCl_3$, 500 MHz, 298 K)



^{13}C-NMR of **16** ($CDCl_3$, 125 MHz, 298 K)



135-DEPT of **16** ($CDCl_3$, 125 MHz, 298 K)

3 UV-VIS AND FLUORESCENCE SPECTRA

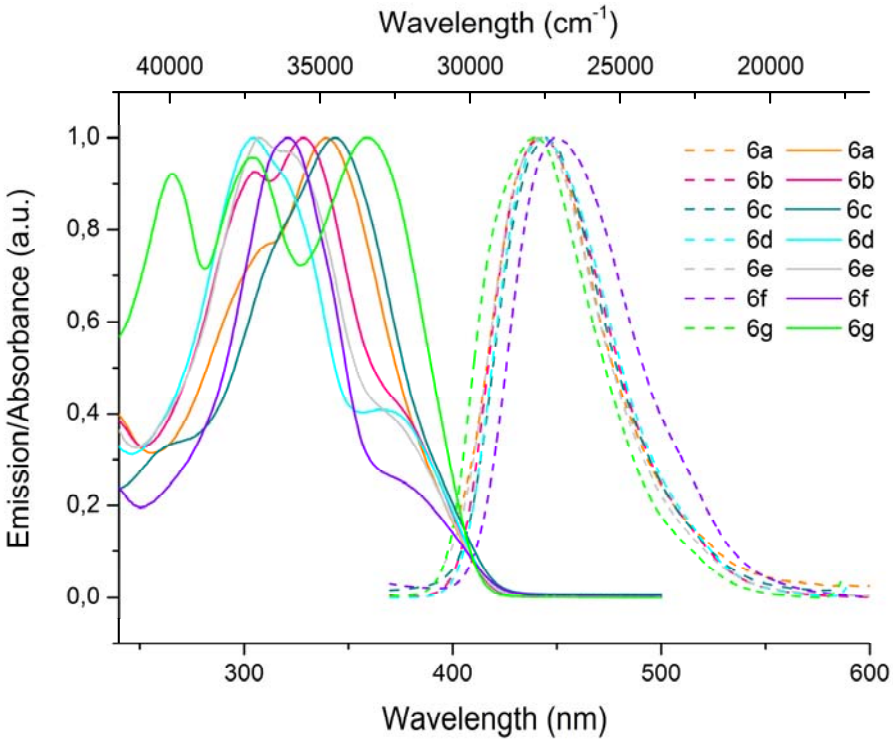


Figure 1: Absorption (solid line) and emission (dashed line) spectra of compounds **6** in CH_2Cl_2, T= 293 K.

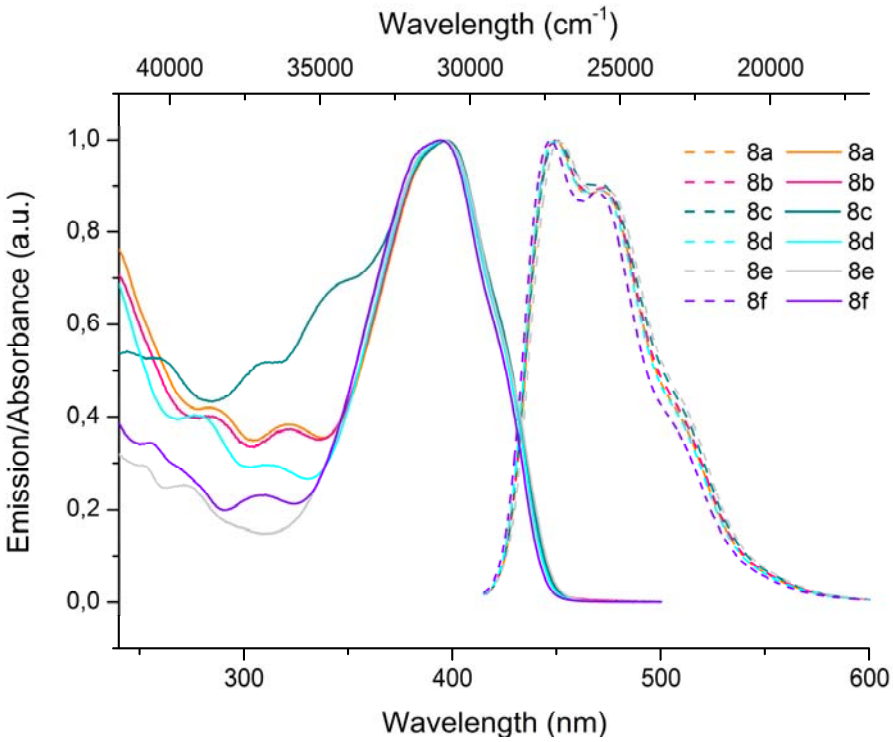


Figure 2: Absorption (solid line) and emission (dashed line) spectra of compounds **8** in CH_2Cl_2, T= 293 K.

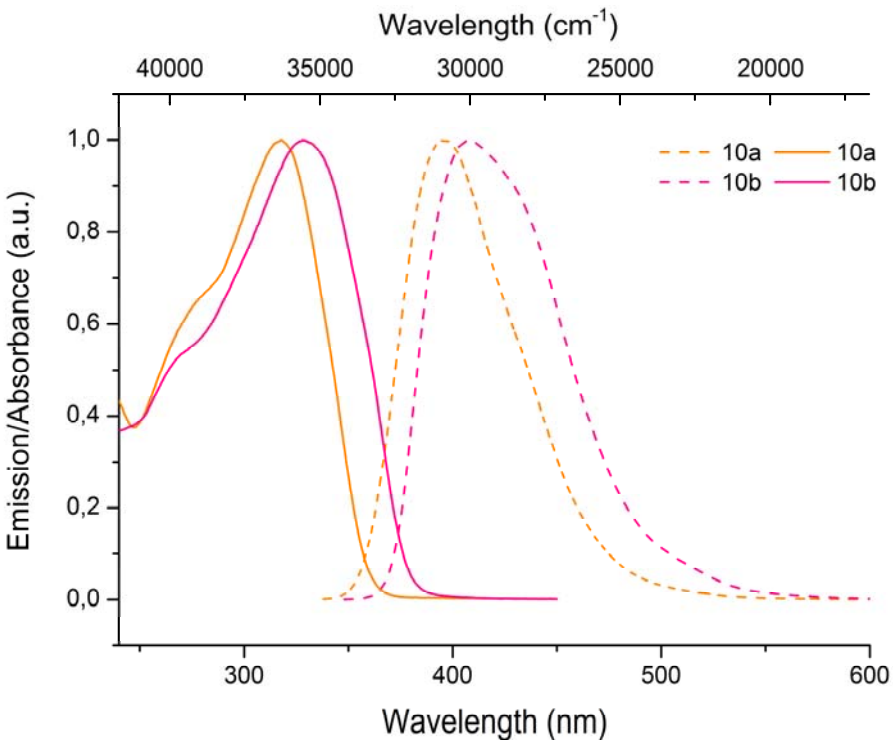


Figure 3: Absorption (solid line) and emission (dashed line) spectra of compounds **10** in CH_2Cl_2, T= 293 K.

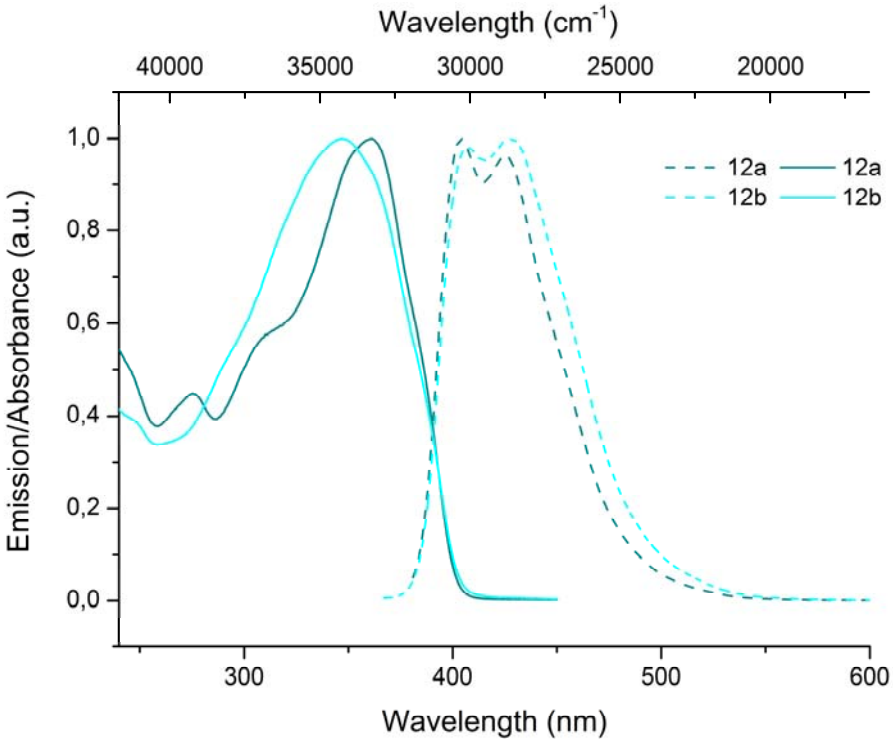


Figure 4: Absorption (solid line) and emission (dashed line) spectra of compounds **12** in CH_2Cl_2, T= 293 K.

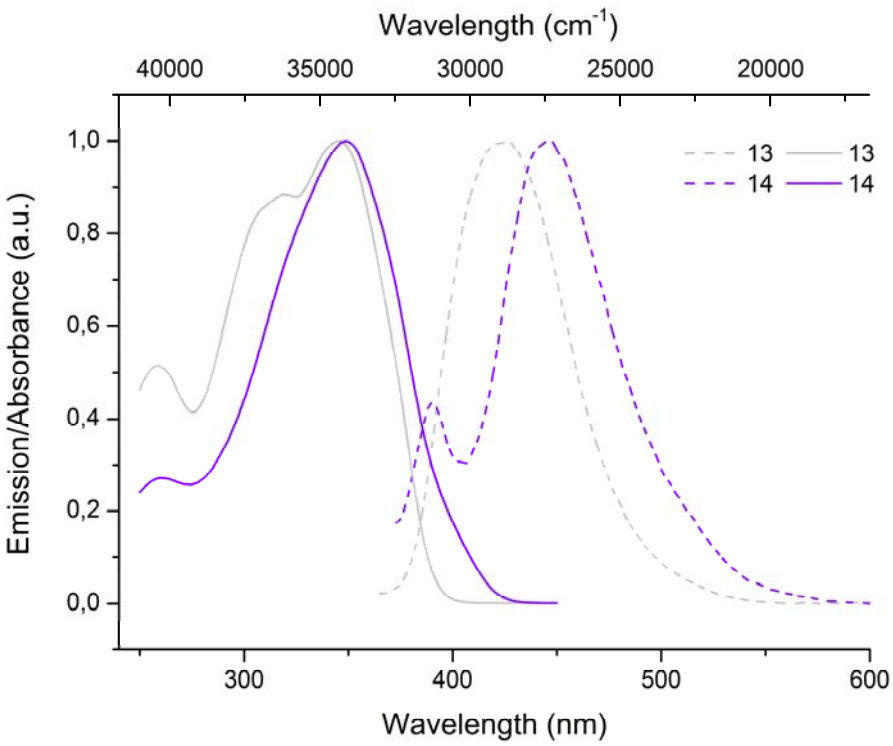


Figure 5: Absorption (solid line) and emission (dashed line) spectra of compounds **13** and **14** in CH_2Cl_2, T= 293 K.

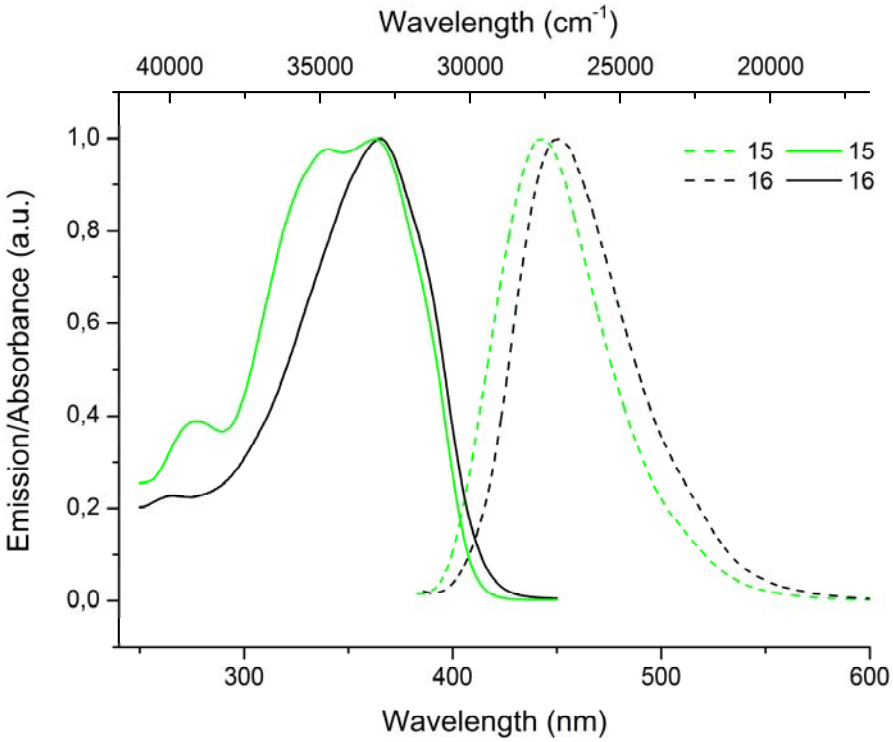


Figure 6: Absorption (solid line) and emission (dashed line) spectra of compounds **15** and **16** in CH_2Cl_2, T= 293 K.