

## Supporting Information

# Sulfuration of C(sp<sup>2</sup>)-H Bond of Enaminones: A Protocol for The Synthesis of Thioether Using Elemental Sulfur as Sulfurating Reagent †

Li-Fang Yang, Cheng-Guo Liu, Xiao-Ping Xu\* and Shun-Jun Ji\*

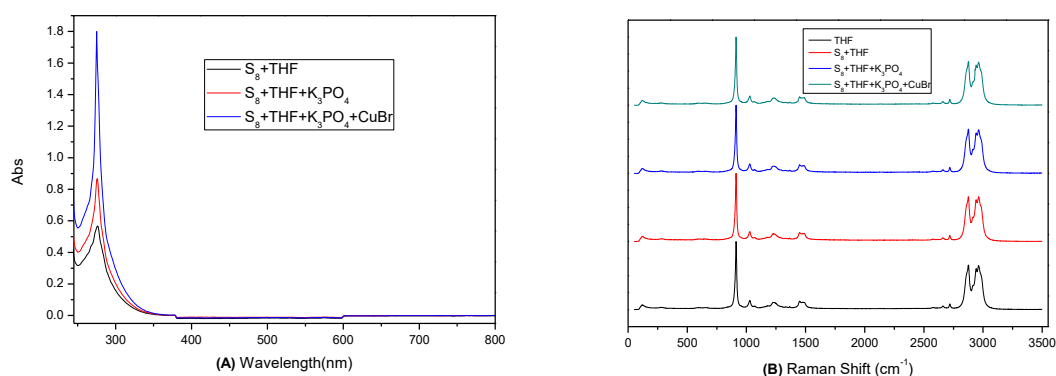
Key Laboratory of Organic Synthesis of Jiangsu Province, College of Chemistry, Chemical Engineering and Materials Science & Collaborative Innovation Center of Suzhou Nano Science and Technology, Soochow University, Suzhou, 215123, People's Republic of China.

Fax: (+)86 512-65880307; Tel: +86-512-65880307

[xuxp@suda.edu.cn](mailto:xuxp@suda.edu.cn); [shunjun@suda.edu.cn](mailto:shunjun@suda.edu.cn)

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## Electronic (UV-Visible) and vibrational (Raman) experiments



**Figure S1.** (A) UV-Visible absorption spectra and (B) Raman spectra experiments

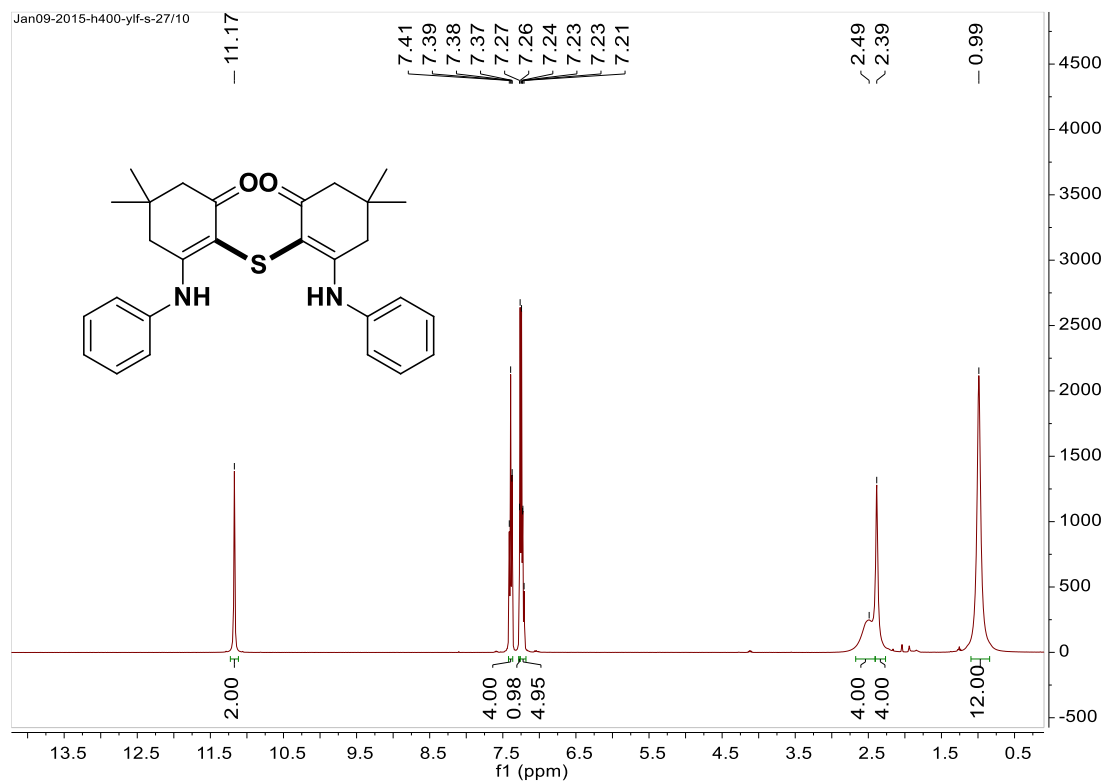
**Notes:** Beside electron spin resonance (ESR) spectroscopy, Chivers and co-workers reported that electronic (UV-Visible) and vibrational (IR and Raman) are also the useful spectroscopic techniques for the characterization of the trisulfur radical anion.<sup>1</sup> They observed that the bright blue  $S_3^-$  is associated with a visible absorption band in the range 595–620 nm in UV-visible absorption spectra<sup>2</sup>. We conducted the following experiments (Figure 1). The reaction mixture excluding enaminone was detected with the UV-vis. spectroscopy, but no absorption peak in the range of 595–620 nm was observed. Further more, in Raman spectroscopy, the characteristic peak at 535  $cm^{-1}$  associated with trisulfur radical anion<sup>1, 2b, 3</sup> was not observed, either. These results from UV-visible absorption spectra and Raman spectra suggest that the  $S_3^-$  did not exist in our reaction system.

1 T. Chivers and P. J. W. Elder, *Chem. Soc. Rev.* **2013**, *42*, 5996.

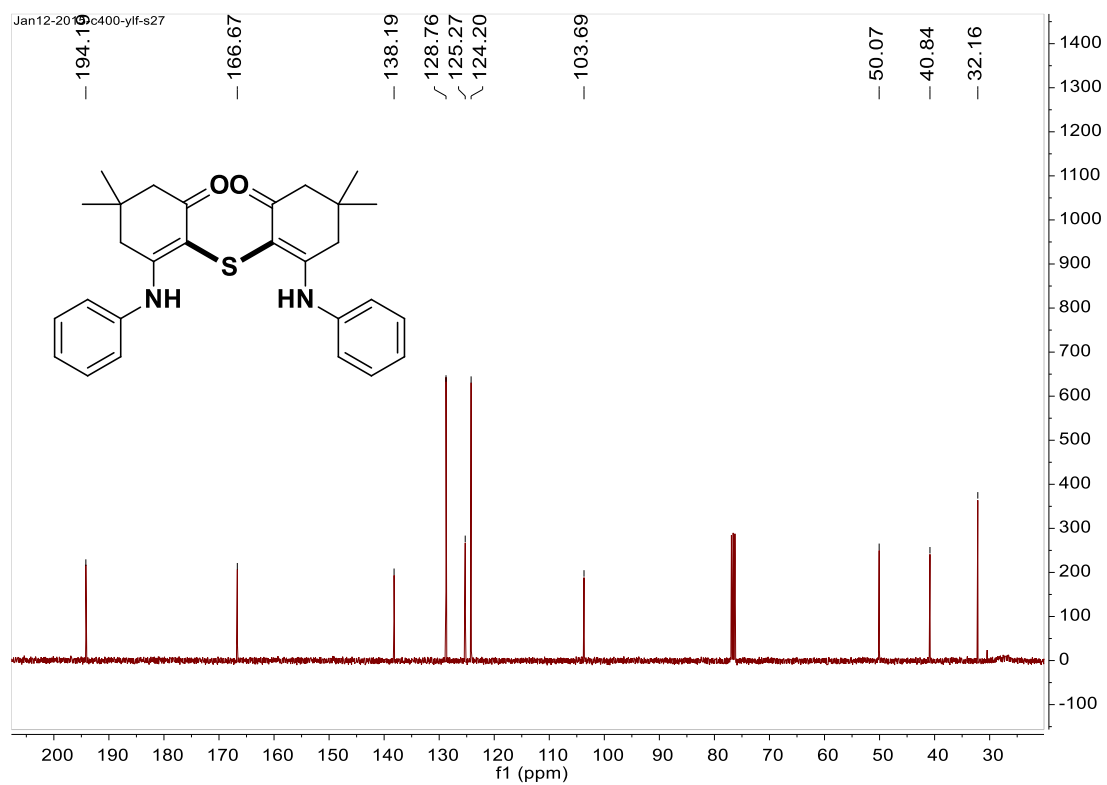
2 (a) T. Chivers, in *Homoatomic Rings, Chains and Macromolecules of Main-Group Elements*, ed. A. L. Rheingold, Elsevier, Amsterdam, 1977, ch. 22, pp. 499–537; (b) T. Chivers, *Nature* **1974**, *252*, 32.

3. (a) J.-T. Yeon, J.-Y. Jang, J.-G. Han, J. Cho, K. T. Lee, N.-S. Choi, *J. Electrochem. Soc.* **2012**, *159*, 1308; (b) R. J. H. Clark, D. G. Cobbold, *Inorg. Chem.* **1978**, *17*, 3169; (c) T. Chivers, C. Lau, *Inorg. Chem.* **1982**, *21*, 453;

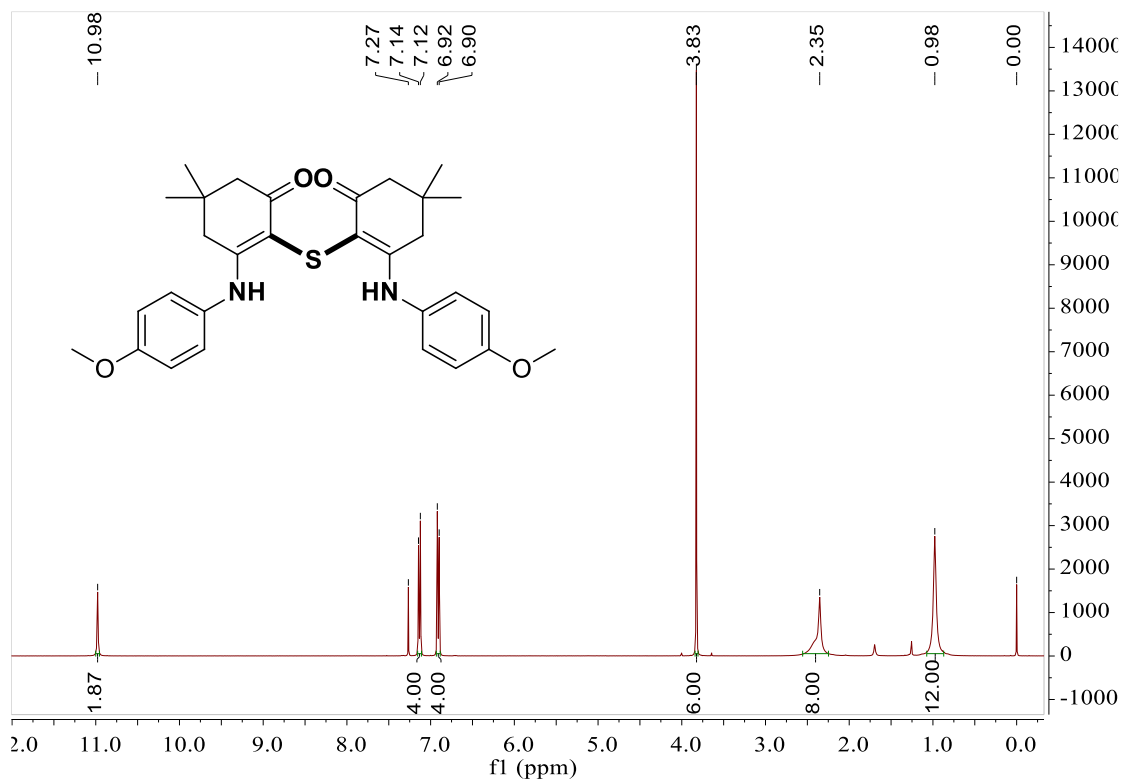
## NMR spectrum of compounds 3



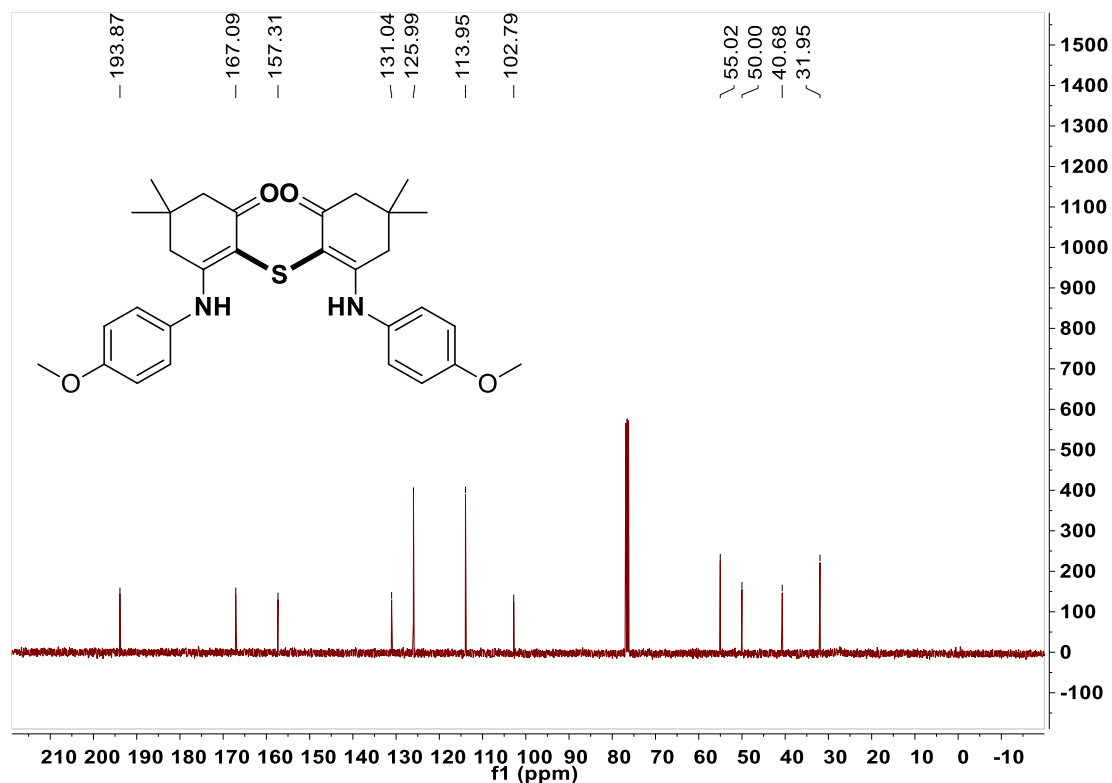
<sup>1</sup>H NMR spectrum of compound 3aa



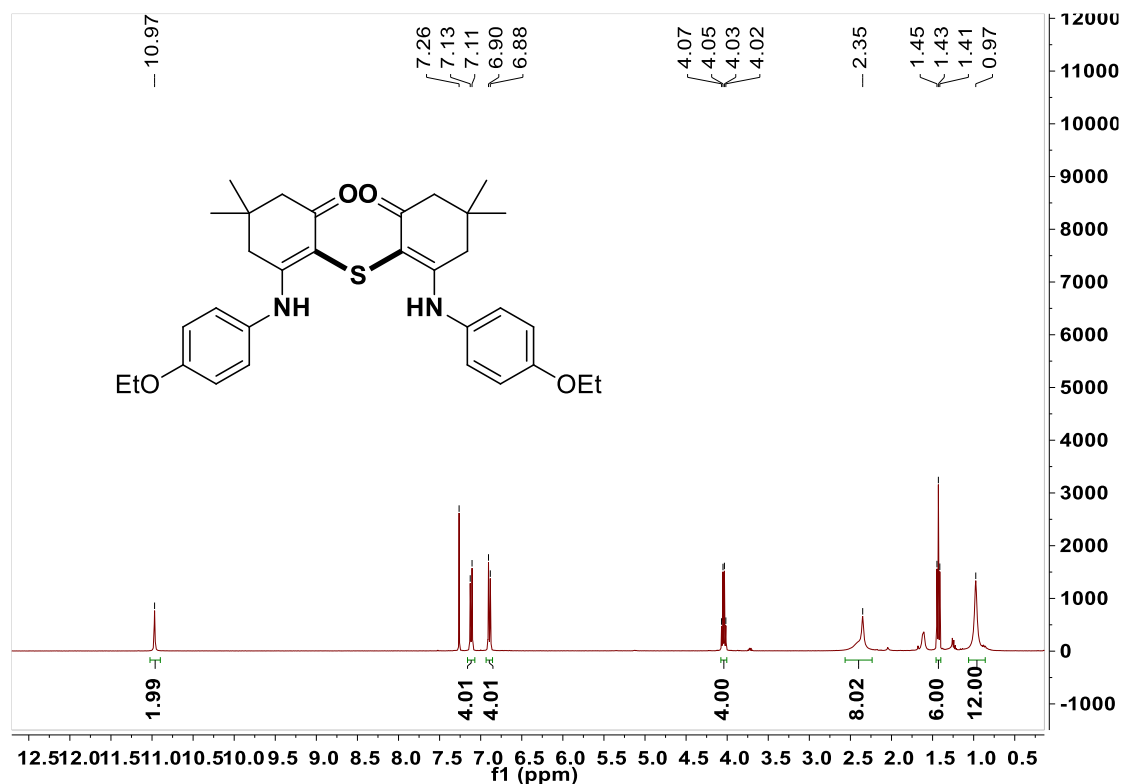
<sup>13</sup>C NMR spectrum of compound 3aa



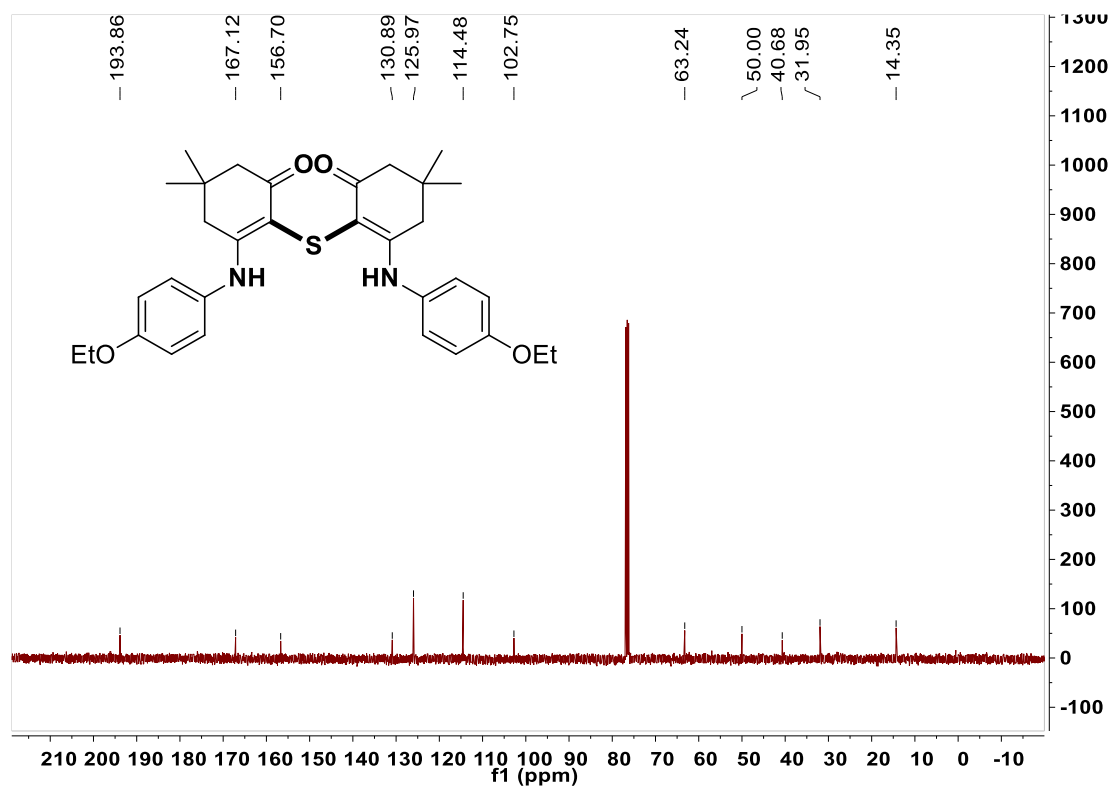
**<sup>1</sup>H NMR spectrum of compound 3ba**



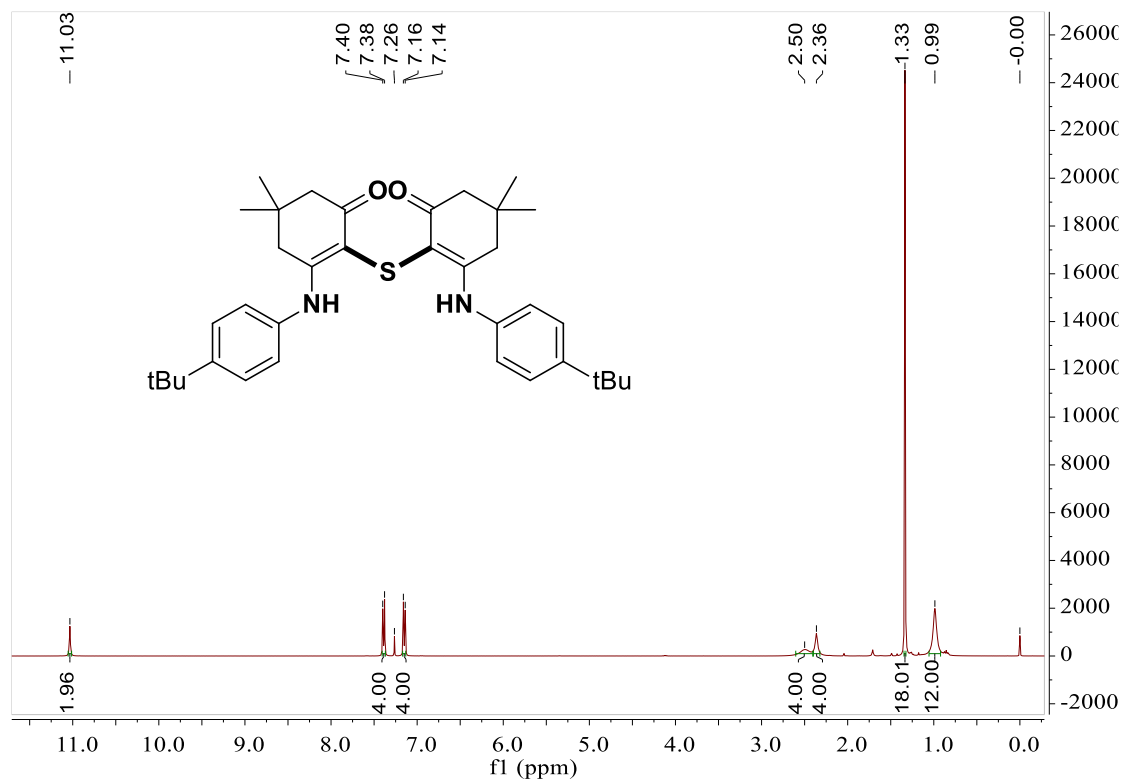
**<sup>13</sup>C NMR spectrum of compound 3ba**



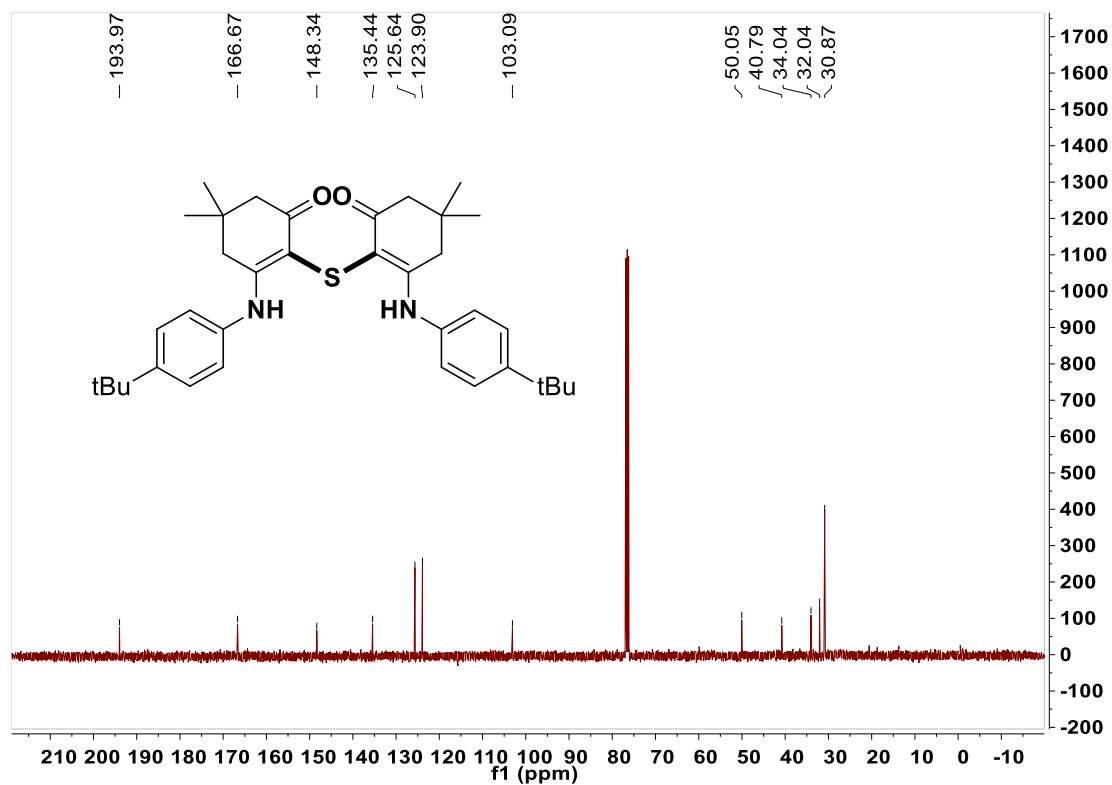
**<sup>1</sup>H NMR spectrum of compound 3ca**



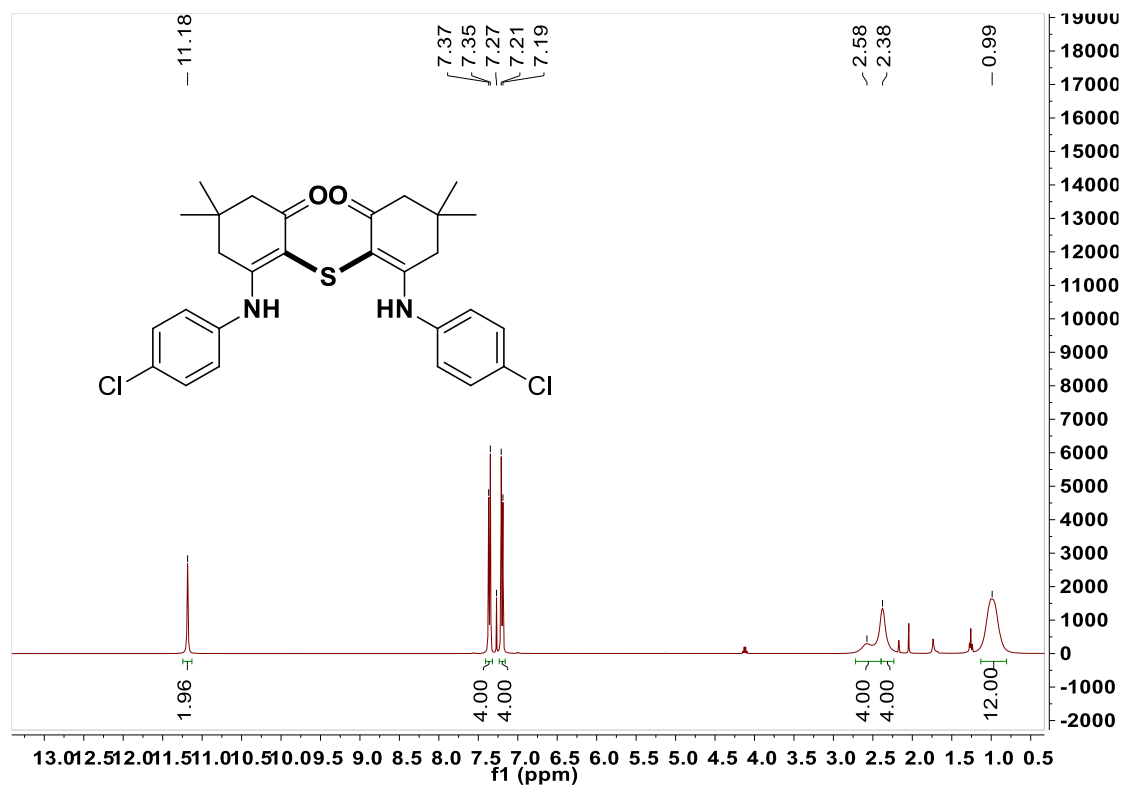
**<sup>13</sup>C NMR spectrum of compound 3ca**



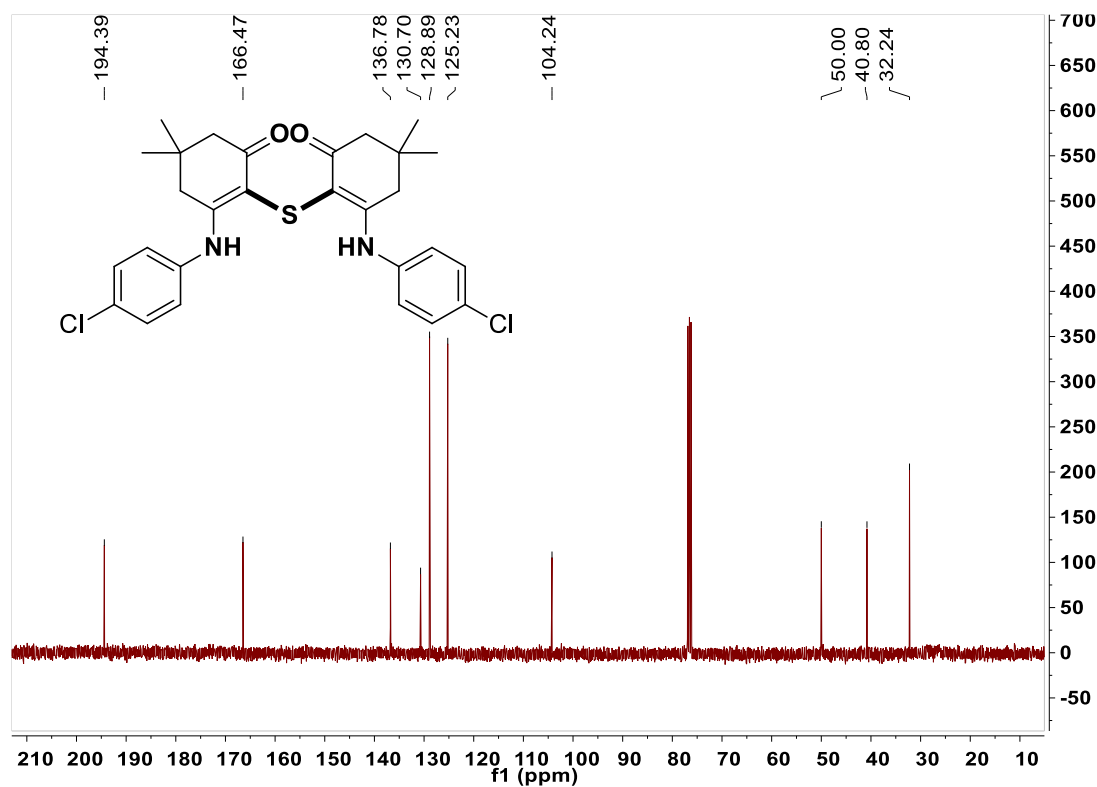
**<sup>1</sup>H NMR spectrum of compound 3da**



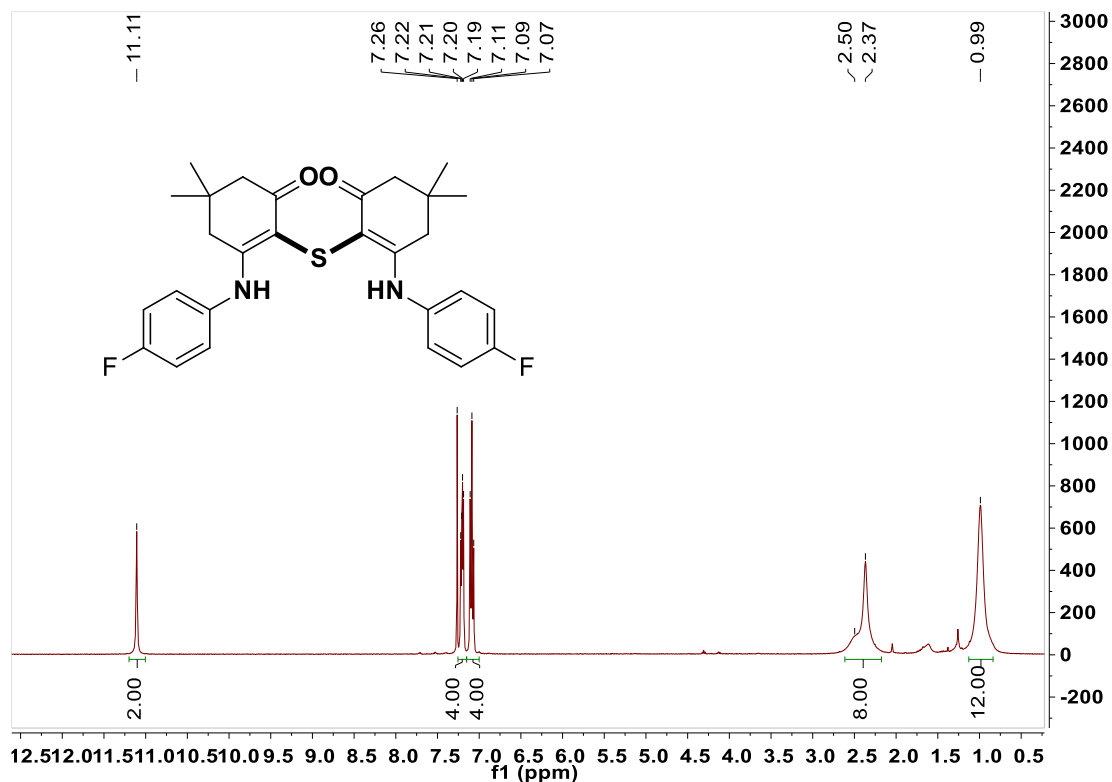
**<sup>13</sup>C NMR spectrum of compound 3da**



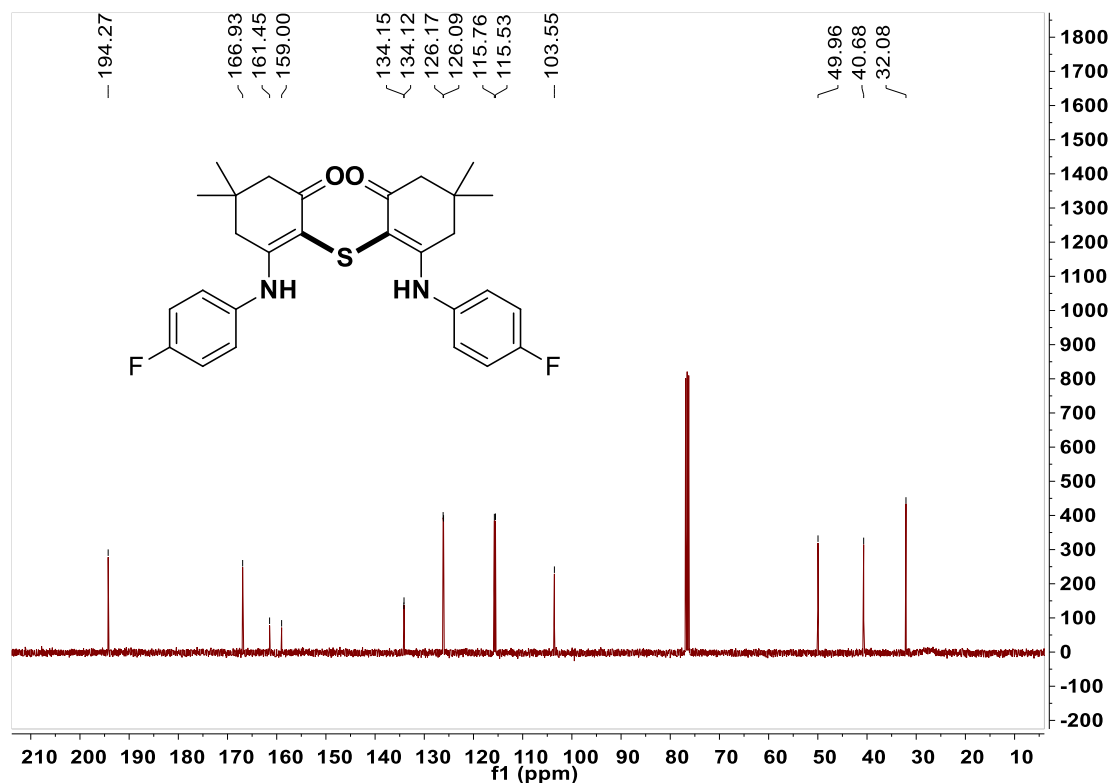
<sup>1</sup>H NMR spectrum of compound 3ea



<sup>13</sup>C NMR spectrum of compound 3ea

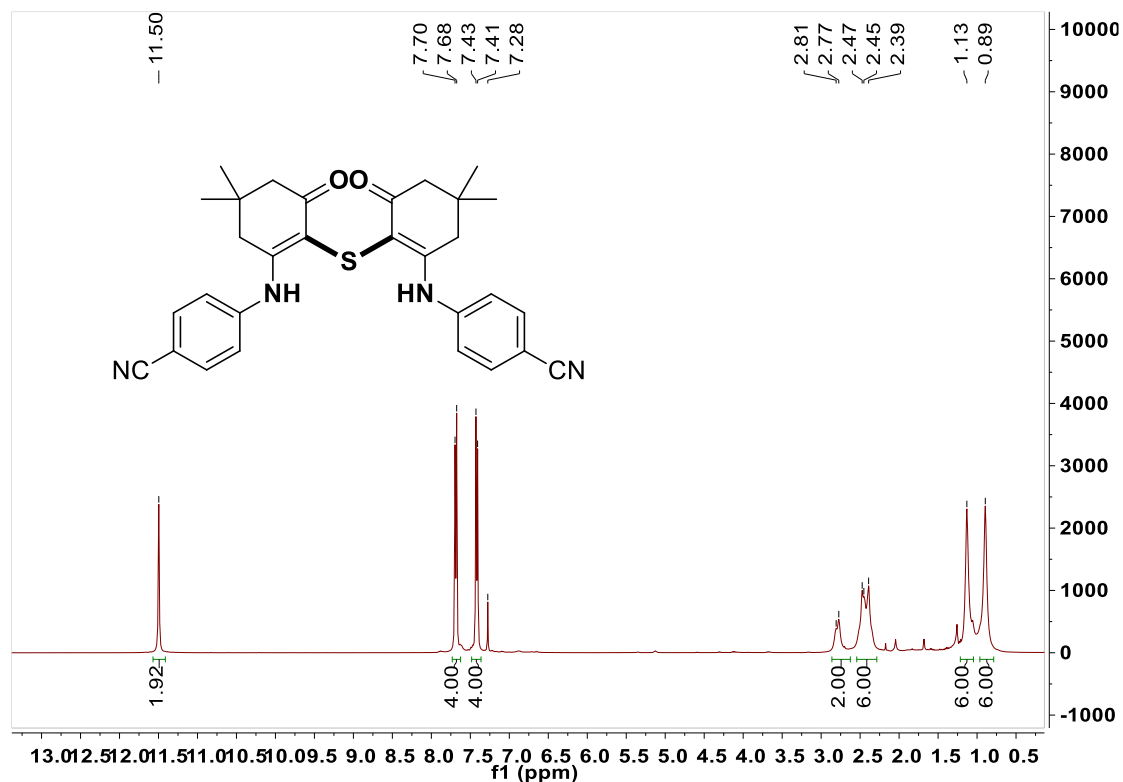


**<sup>1</sup>H NMR spectrum of compound 3fa**

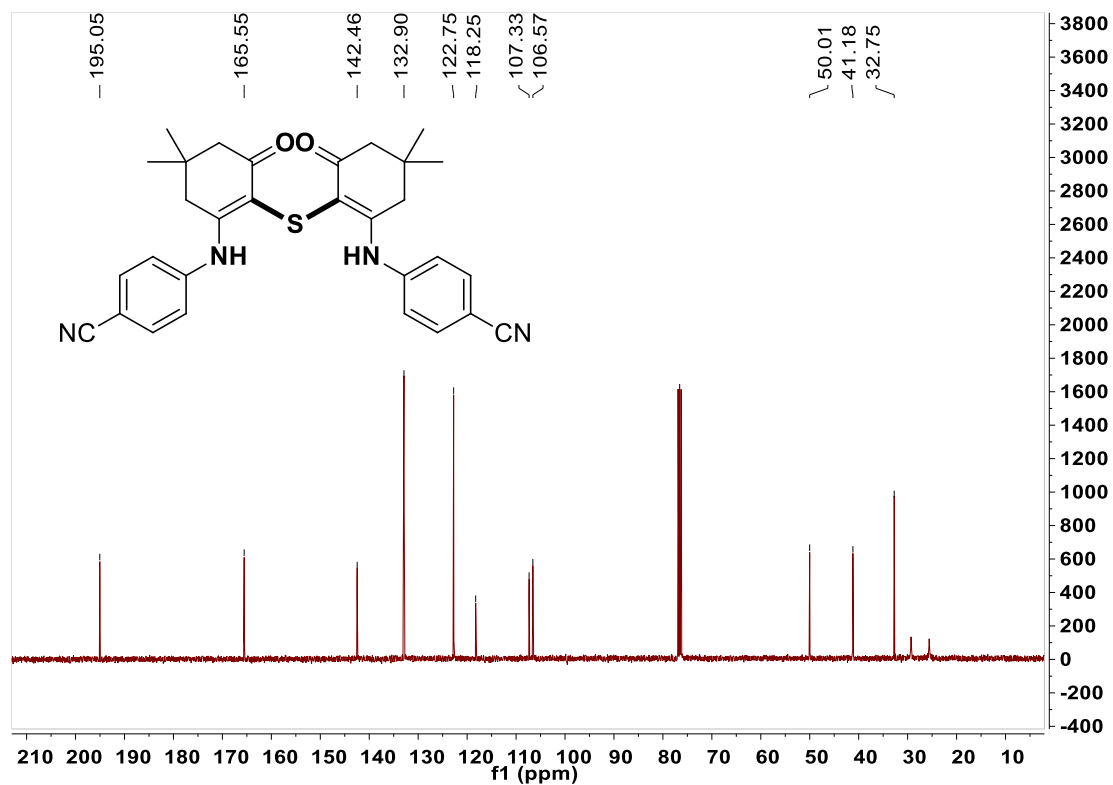


**<sup>13</sup>C NMR spectrum of compound 3fa**

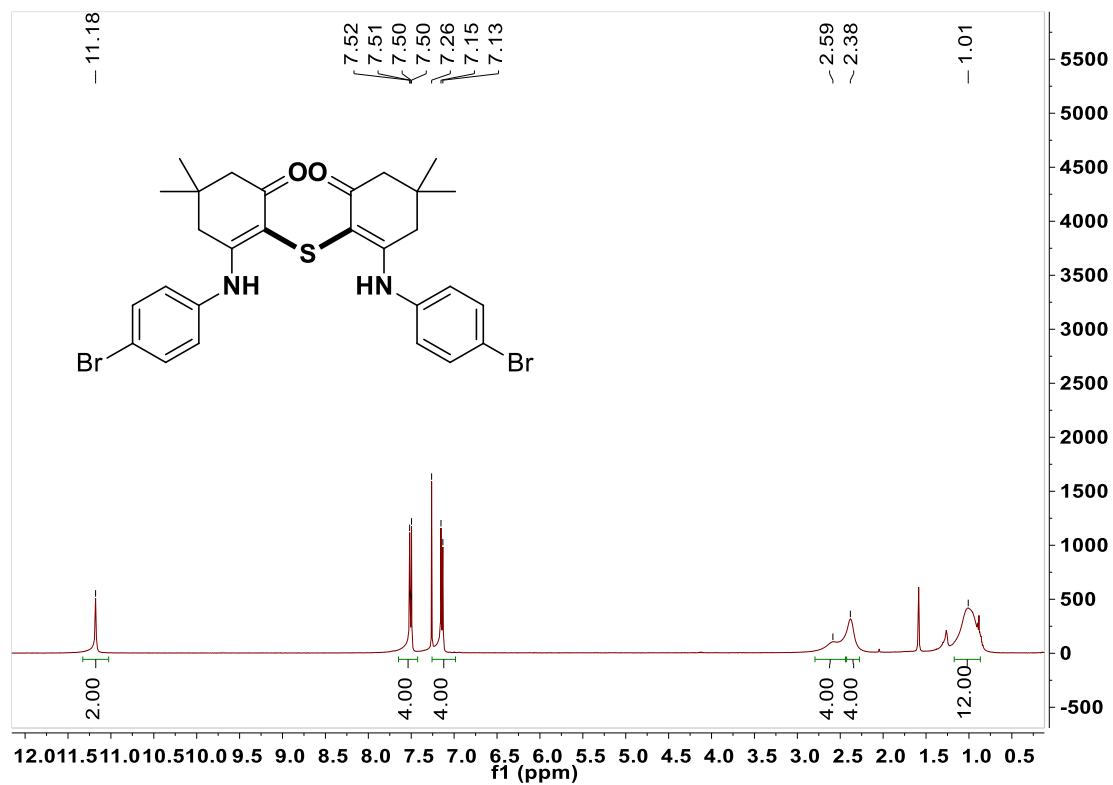




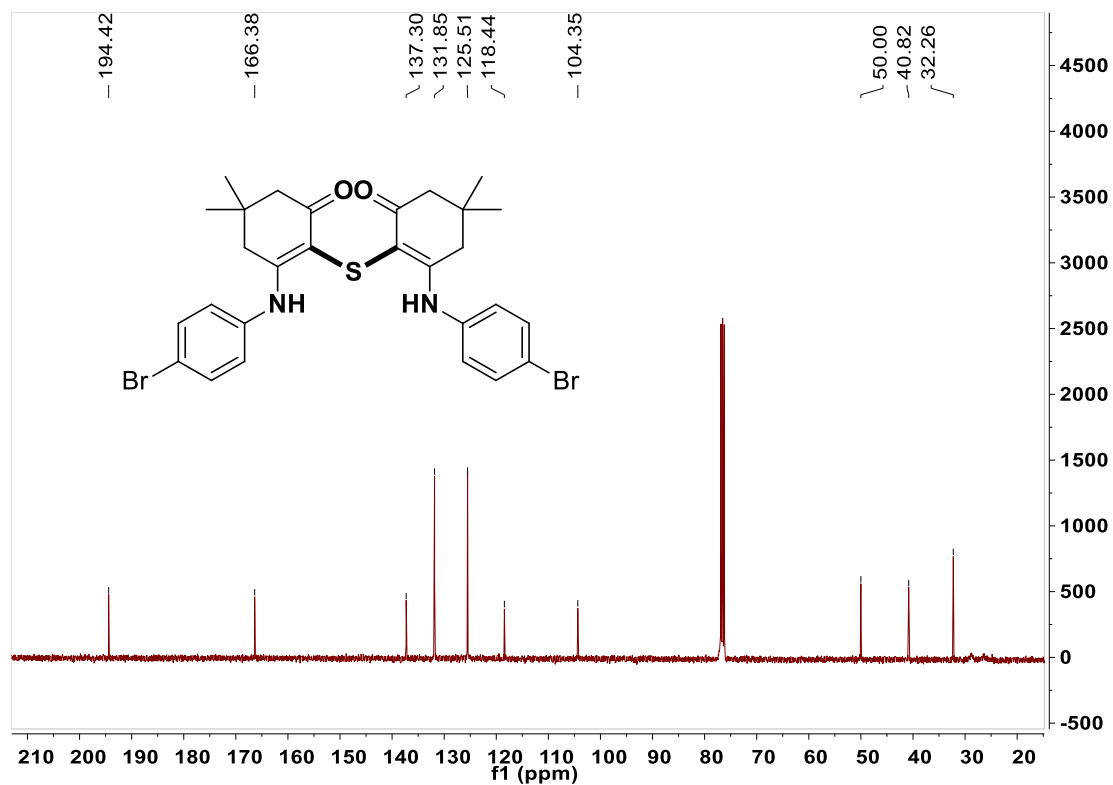
<sup>1</sup>H NMR spectrum of compound 3ga



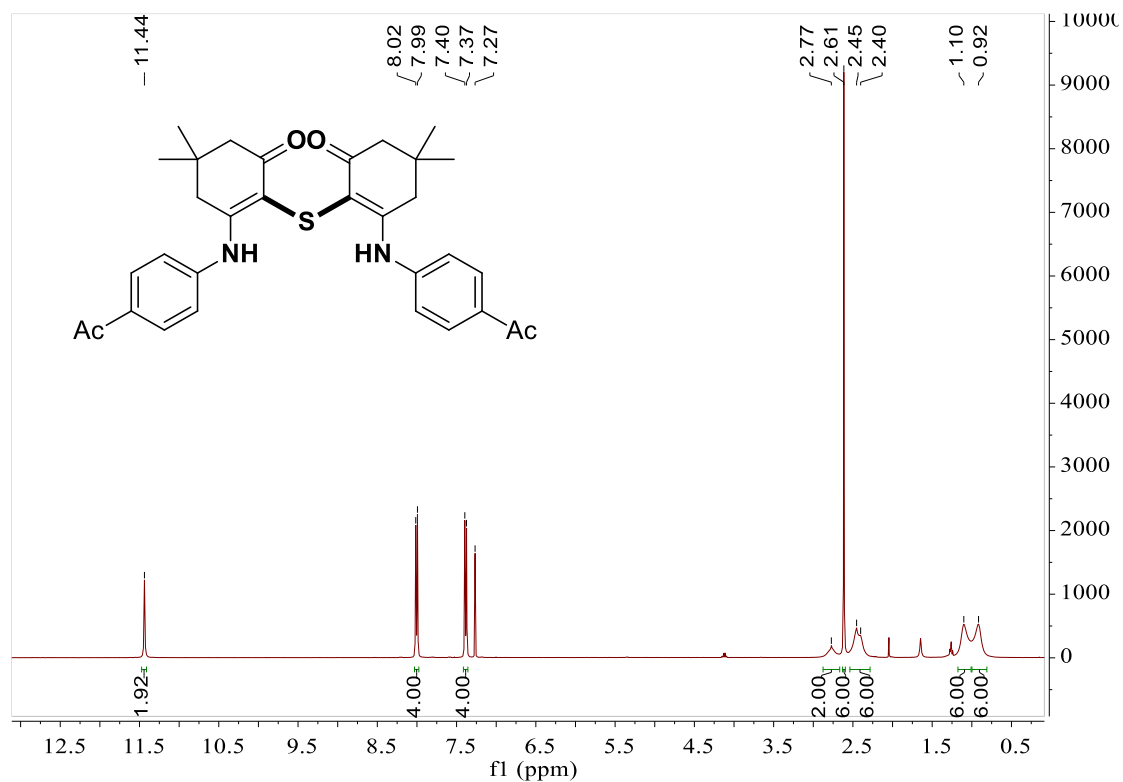
<sup>13</sup>C NMR spectrum of compound 3ga



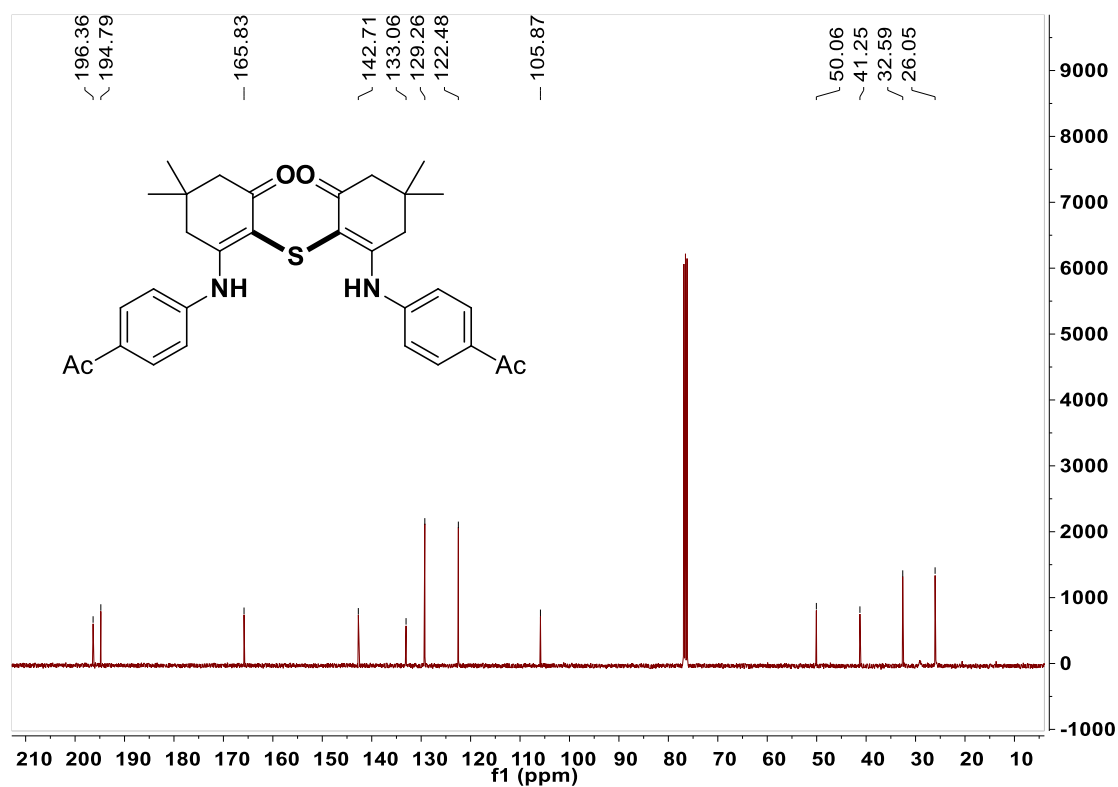
**<sup>1</sup>H NMR spectrum of compound 3ha**



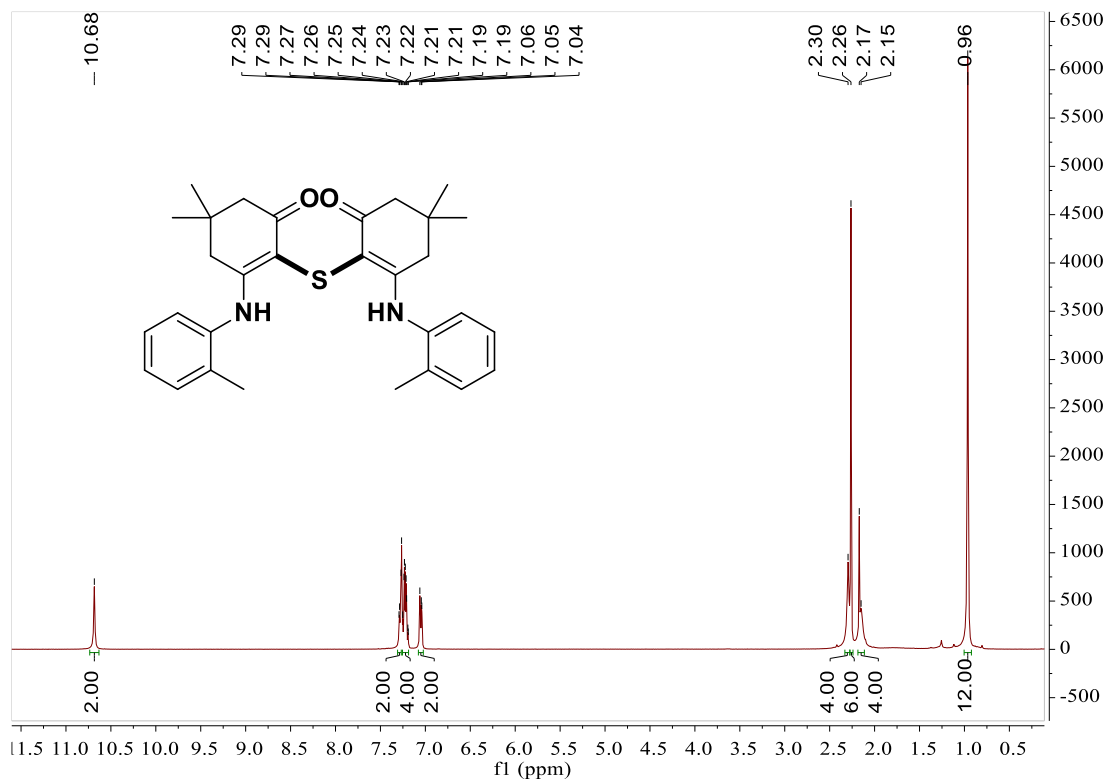
**<sup>13</sup>C NMR spectrum of compound 3ha**



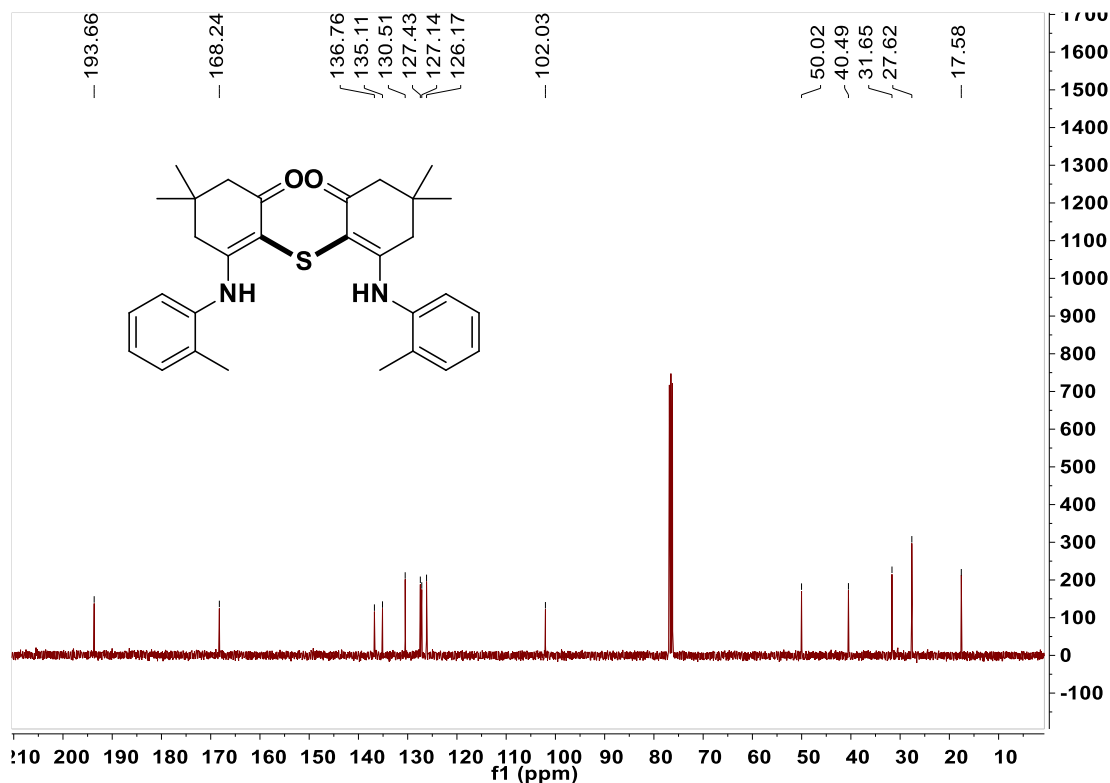
**<sup>1</sup>H NMR spectrum of compound 3ia**



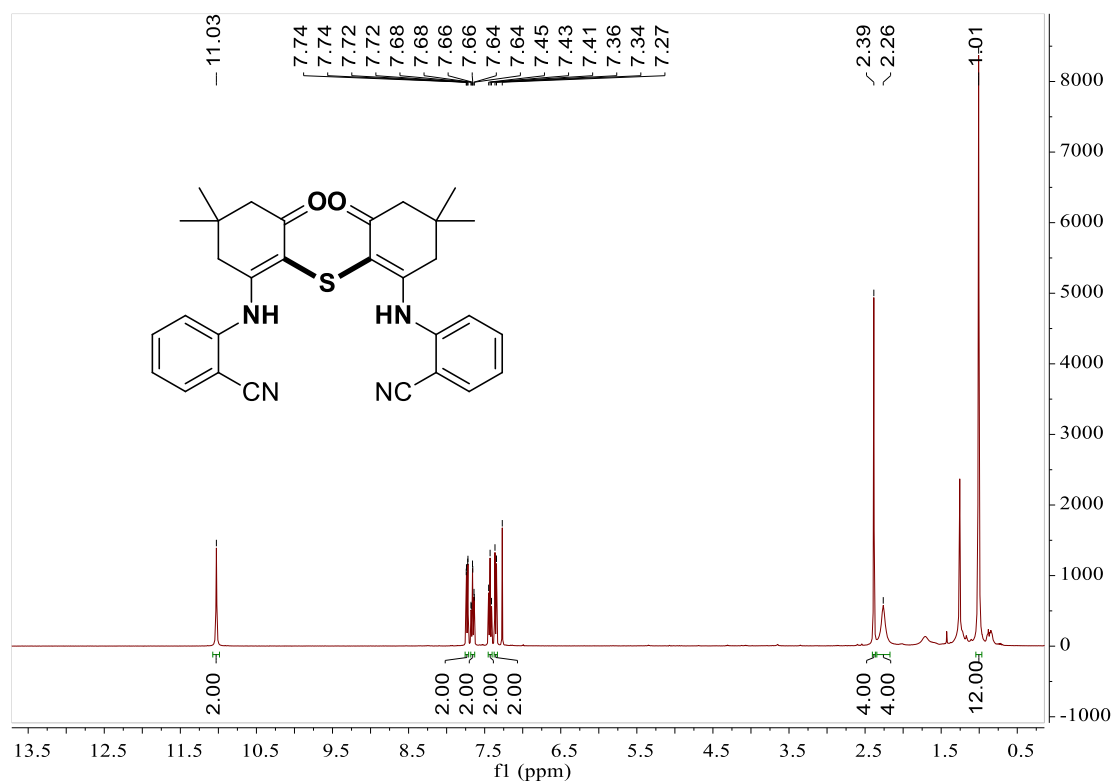
**<sup>13</sup>C NMR spectrum of compound 3ia**



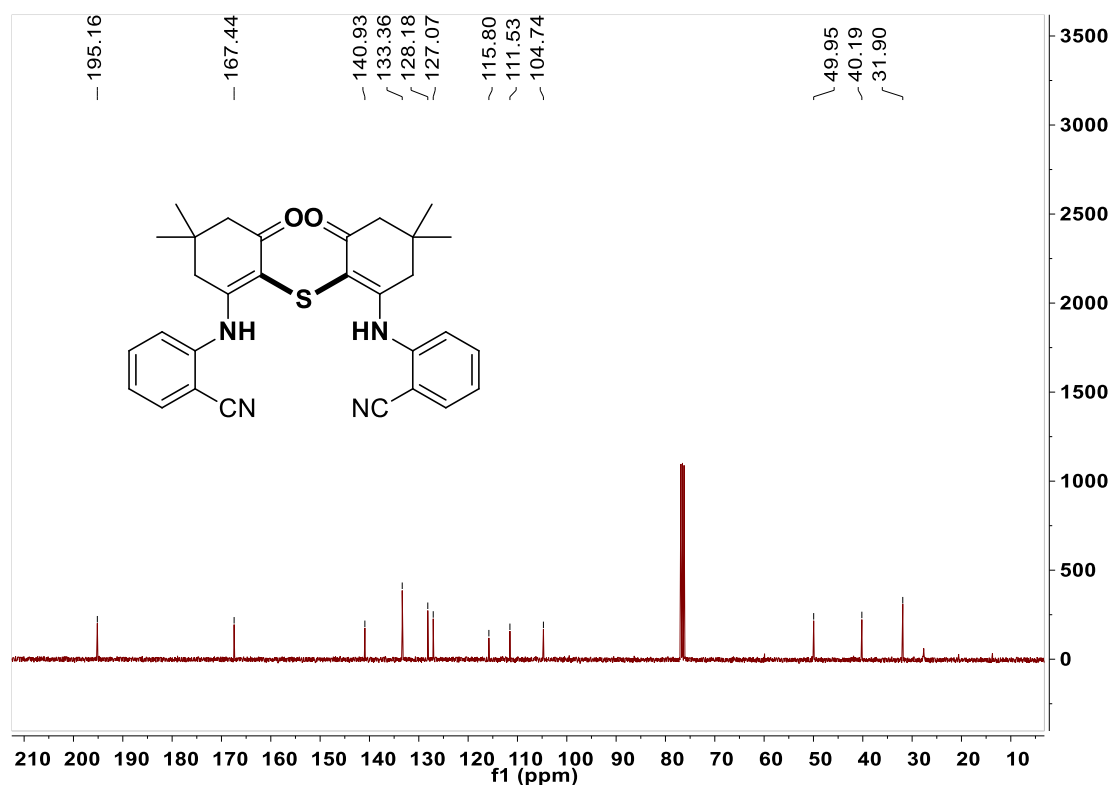
**<sup>1</sup>H NMR spectrum of compound 3ja**



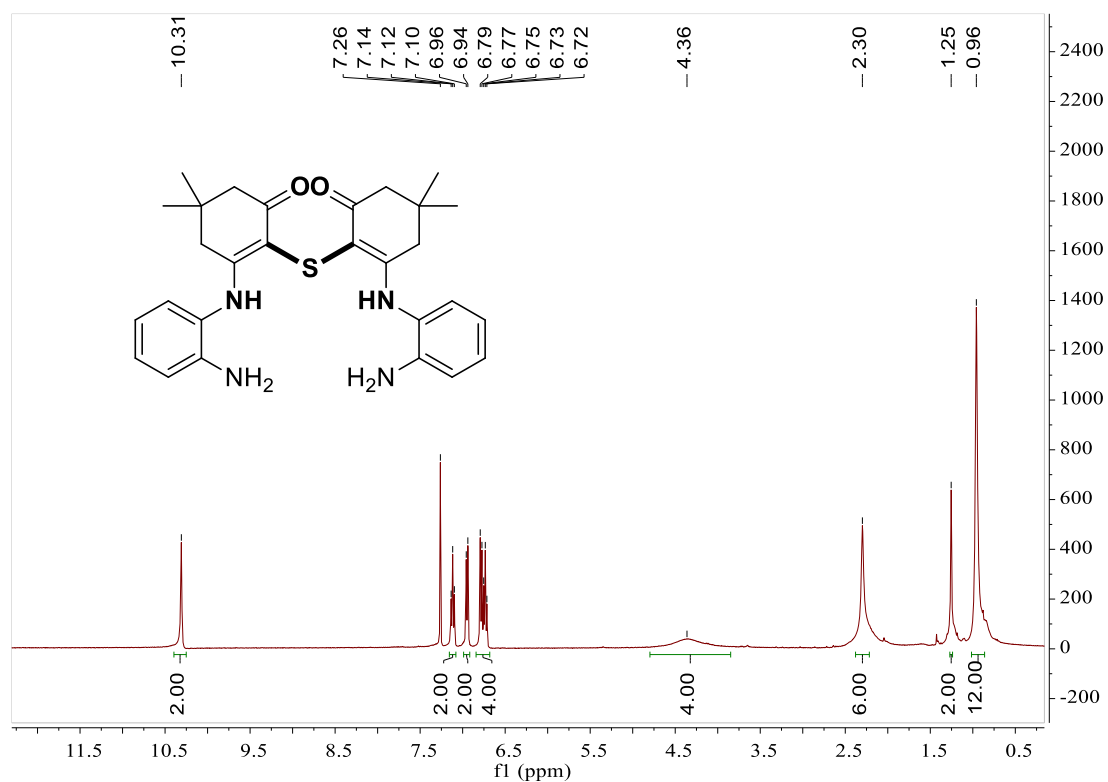
**<sup>13</sup>C NMR spectrum of compound 3ja**



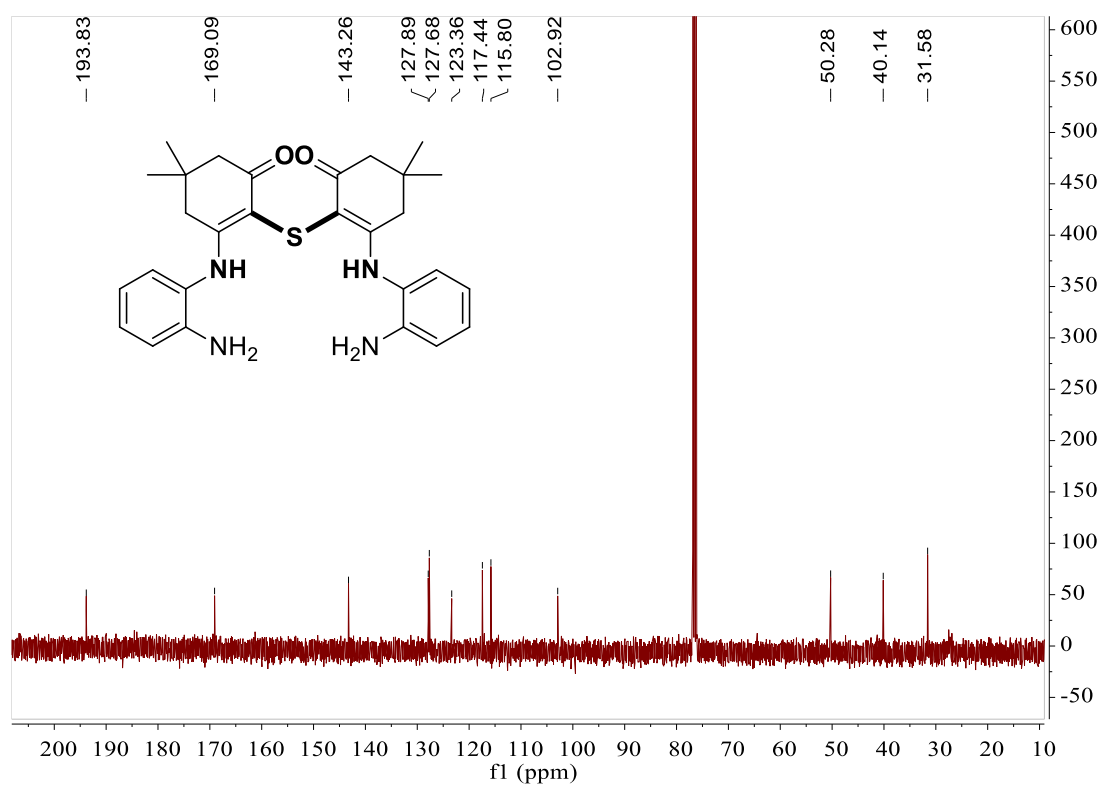
**<sup>1</sup>H NMR spectrum of compound 3ka**



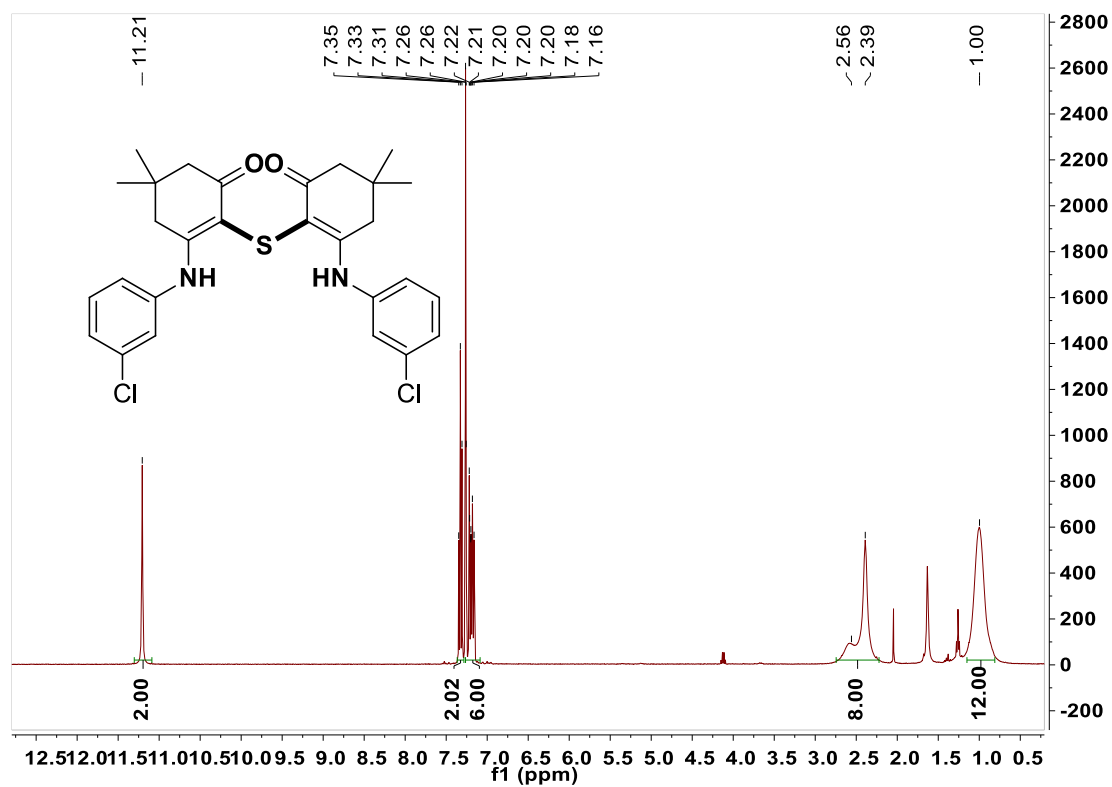
**<sup>13</sup>C NMR spectrum of compound 3ka**



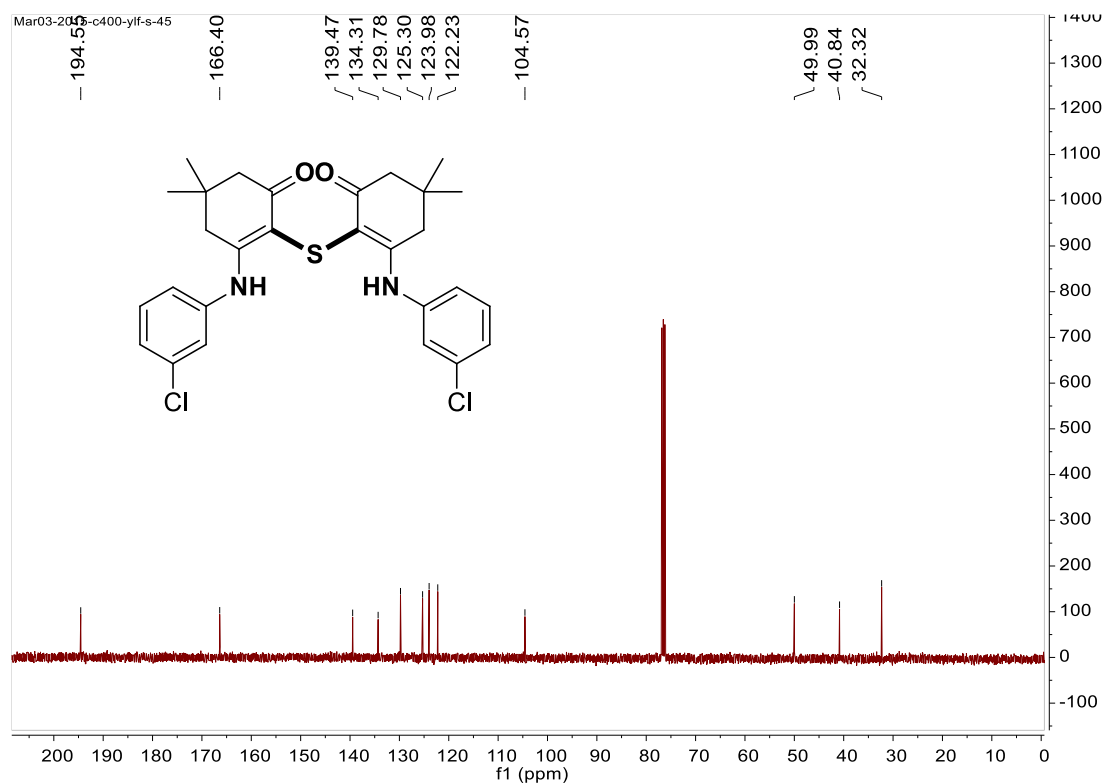
**<sup>1</sup>H NMR spectrum of compound 3la**



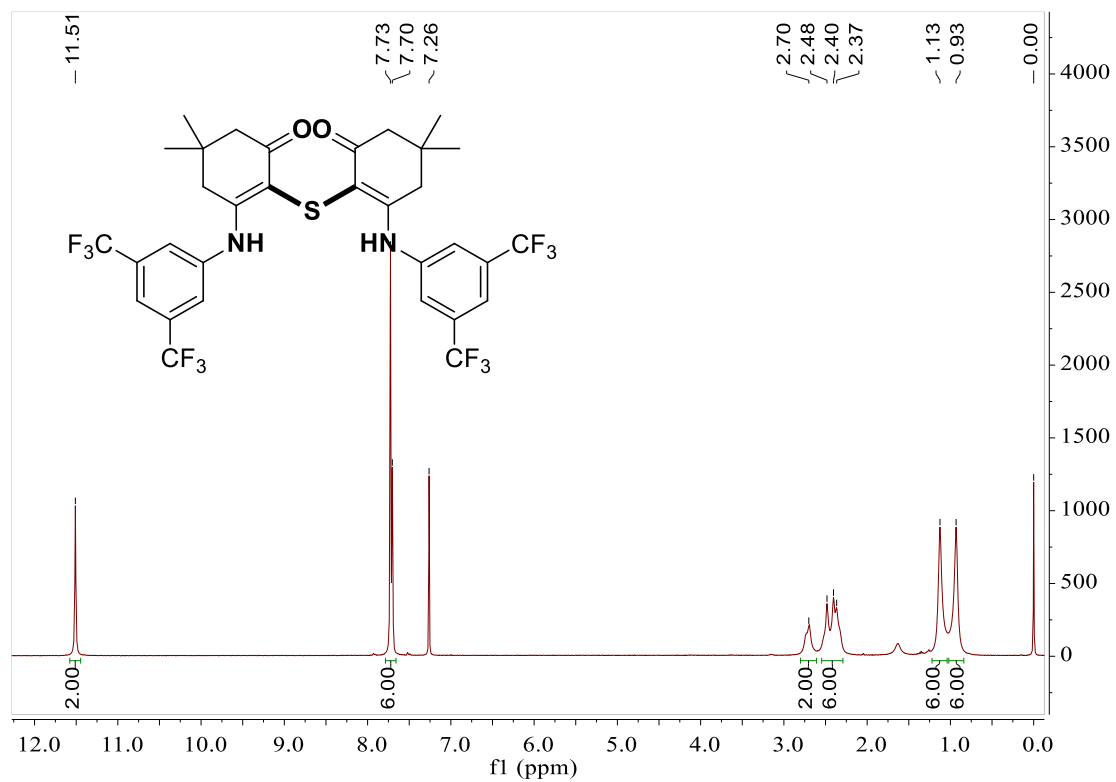
**<sup>13</sup>C NMR spectrum of compound 3la**



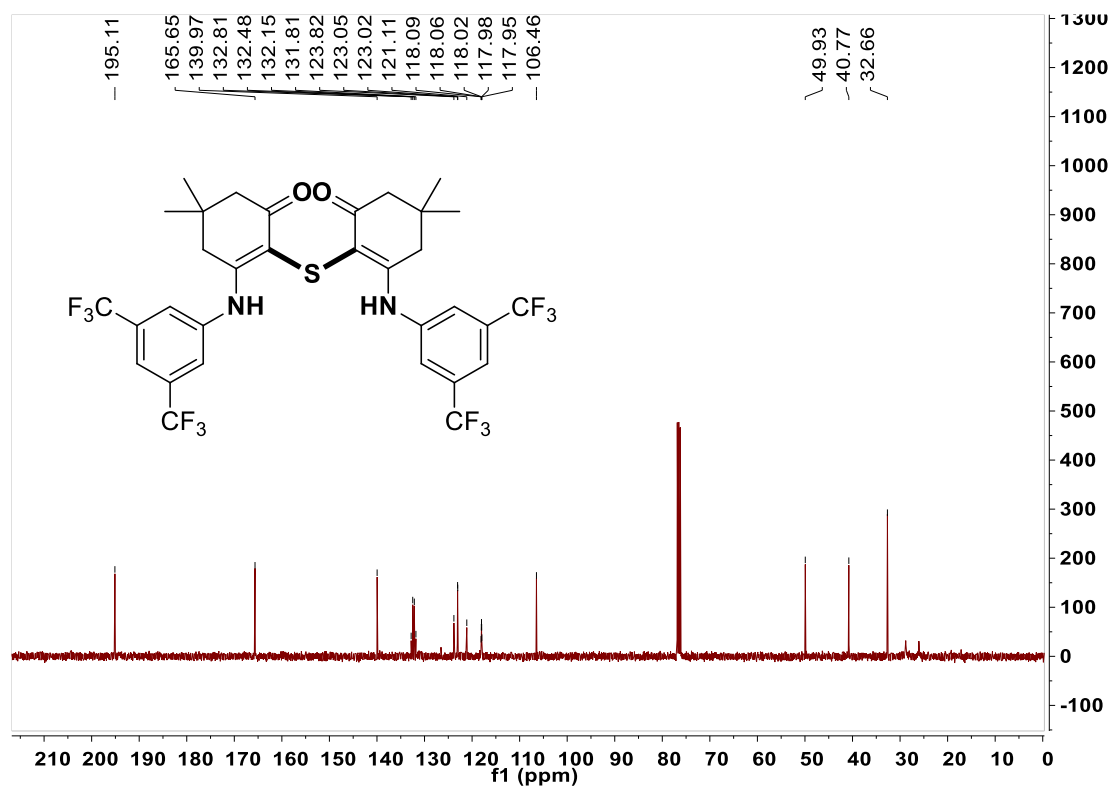
**<sup>1</sup>H NMR spectrum of compound 3ma**



**<sup>13</sup>C NMR spectrum of compound 3ma**

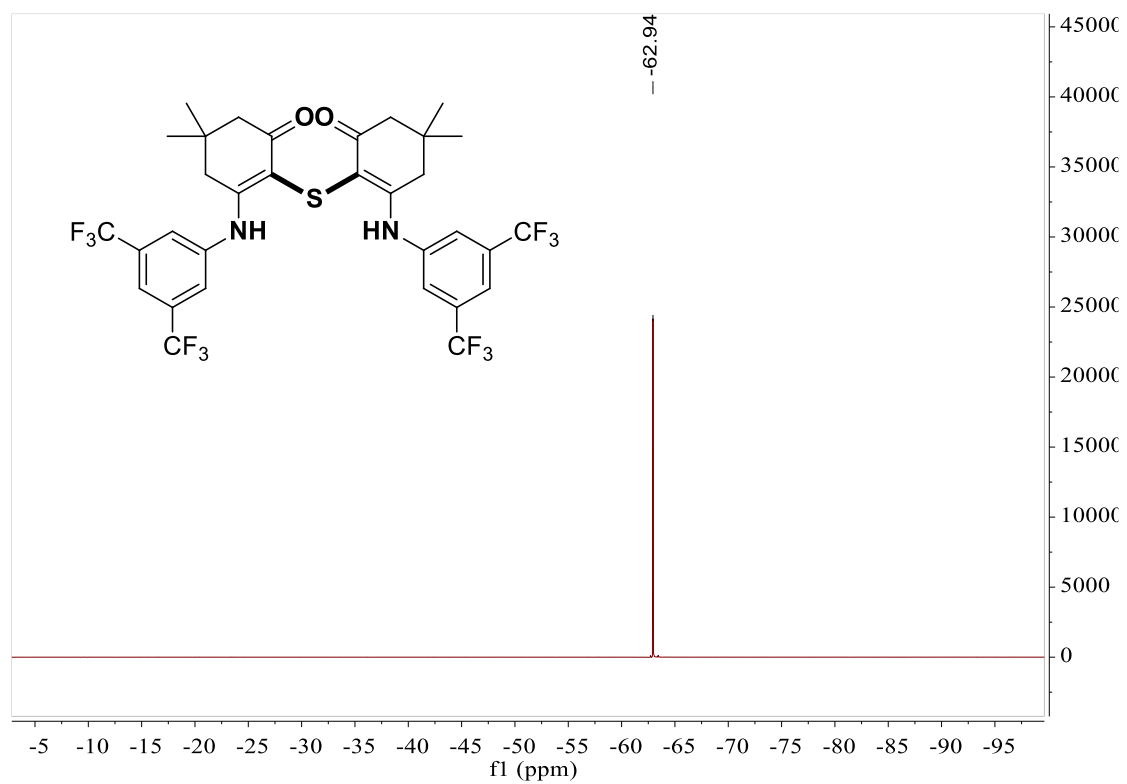


<sup>1</sup>H NMR spectrum of compound 3na

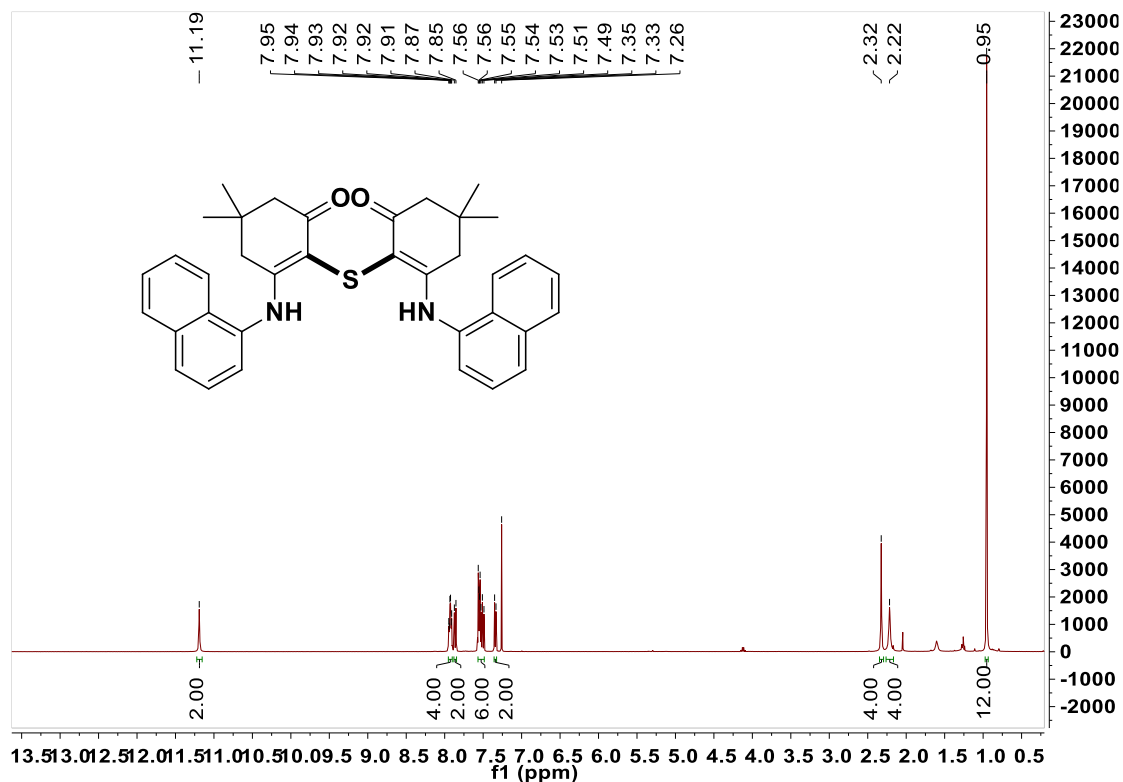


<sup>13</sup>C NMR spectrum of compound 3na

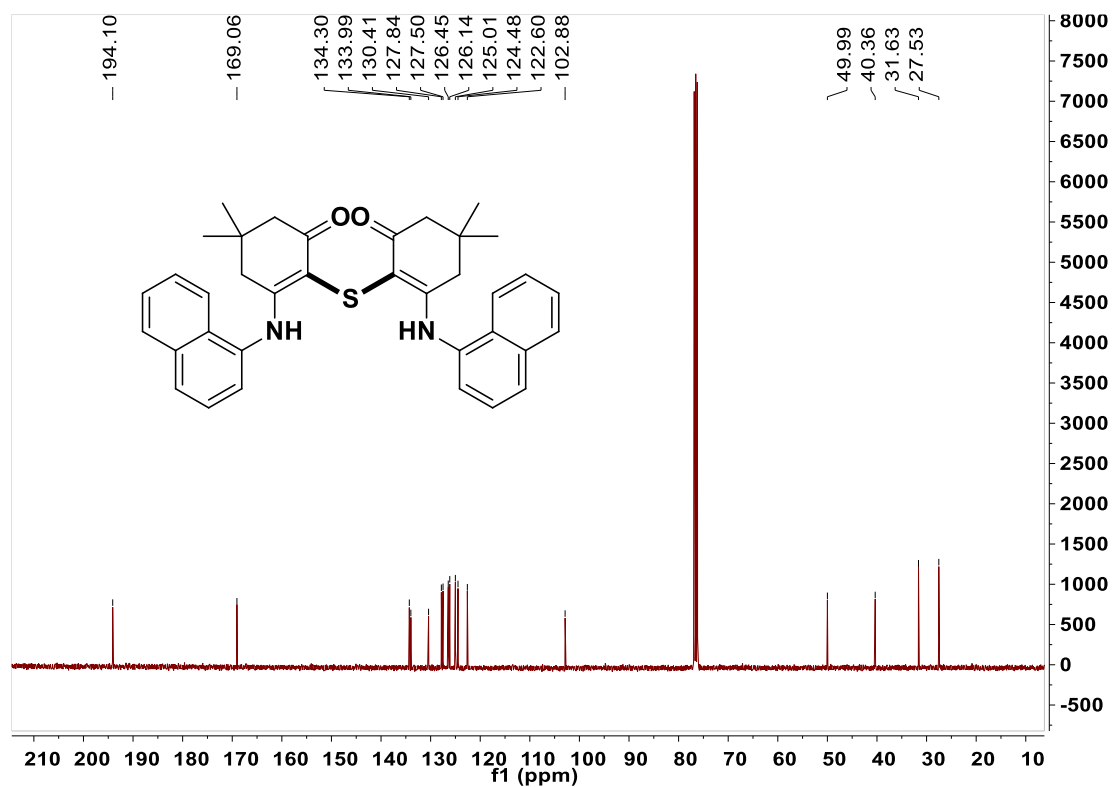




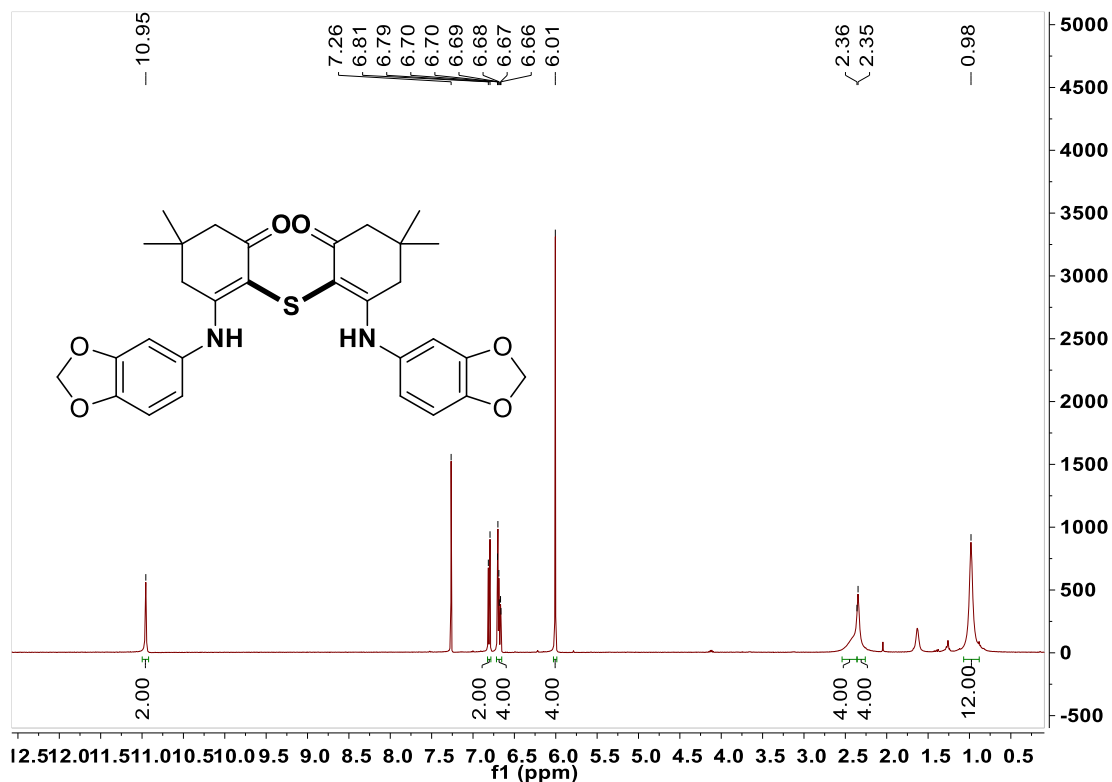
**$^{19}\text{F}$  NMR spectrum of compound 3na**



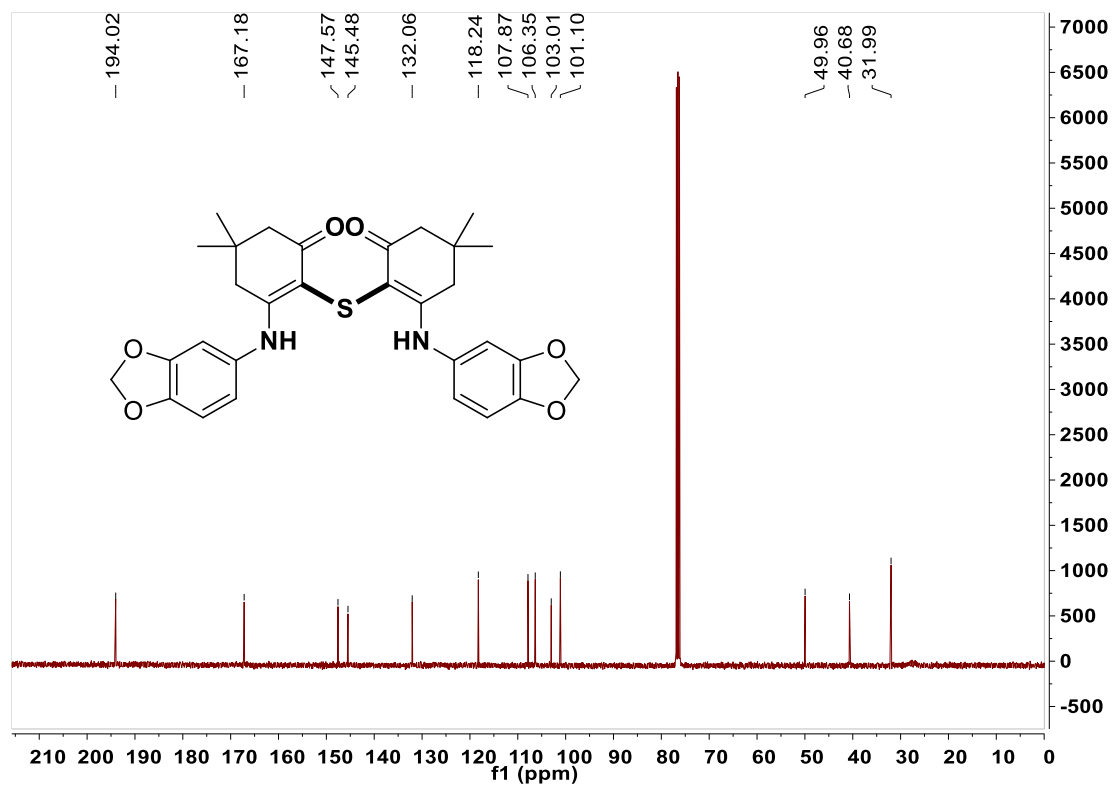
<sup>1</sup>H NMR spectrum of compound 30a



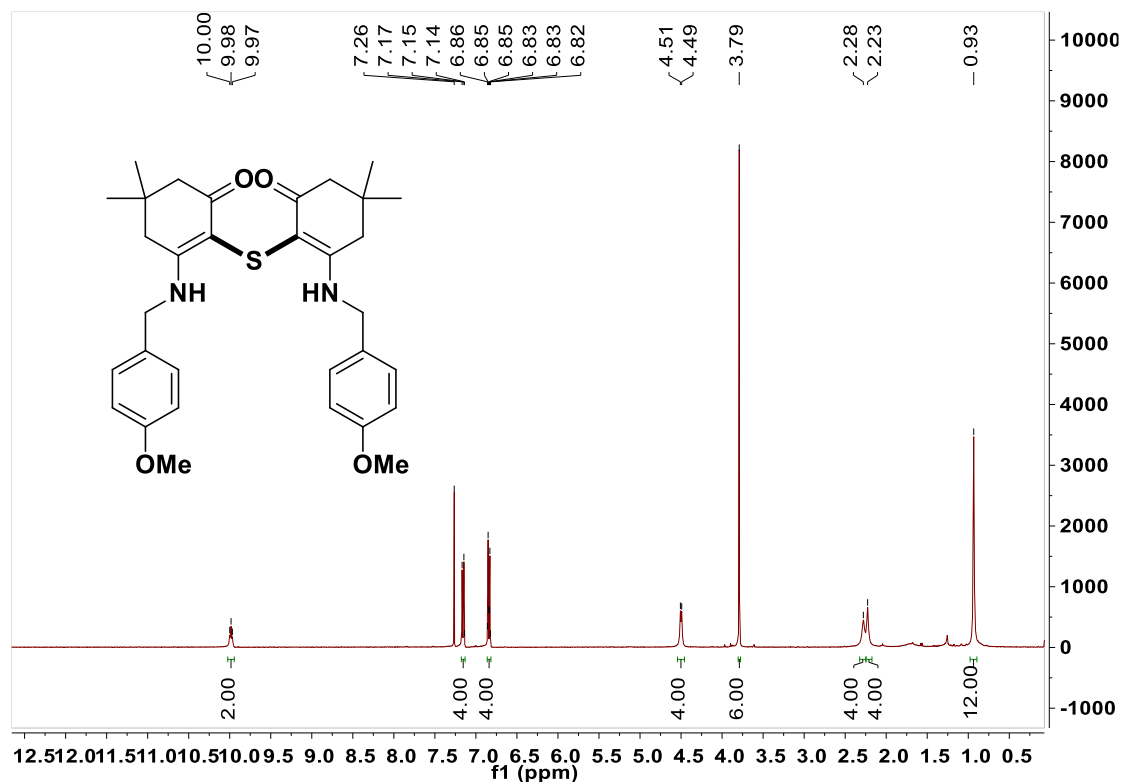
<sup>13</sup>C NMR spectrum of compound 30a



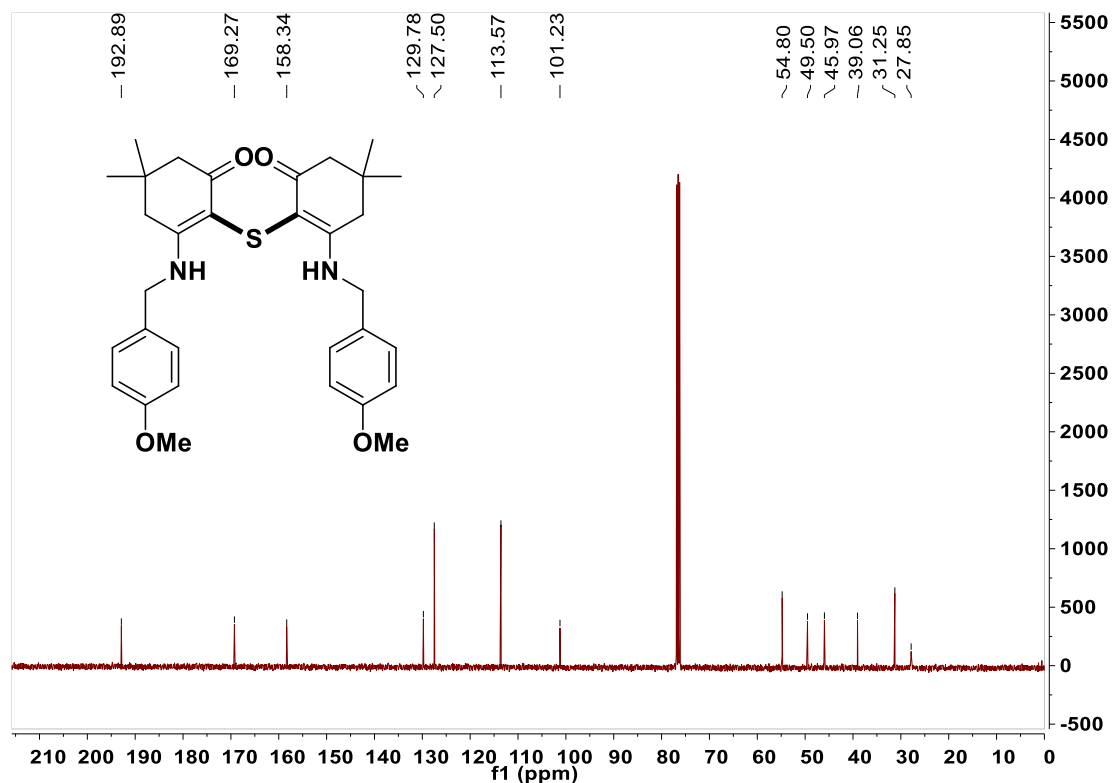
$^1\text{H}$  NMR spectrum of compound 3pa



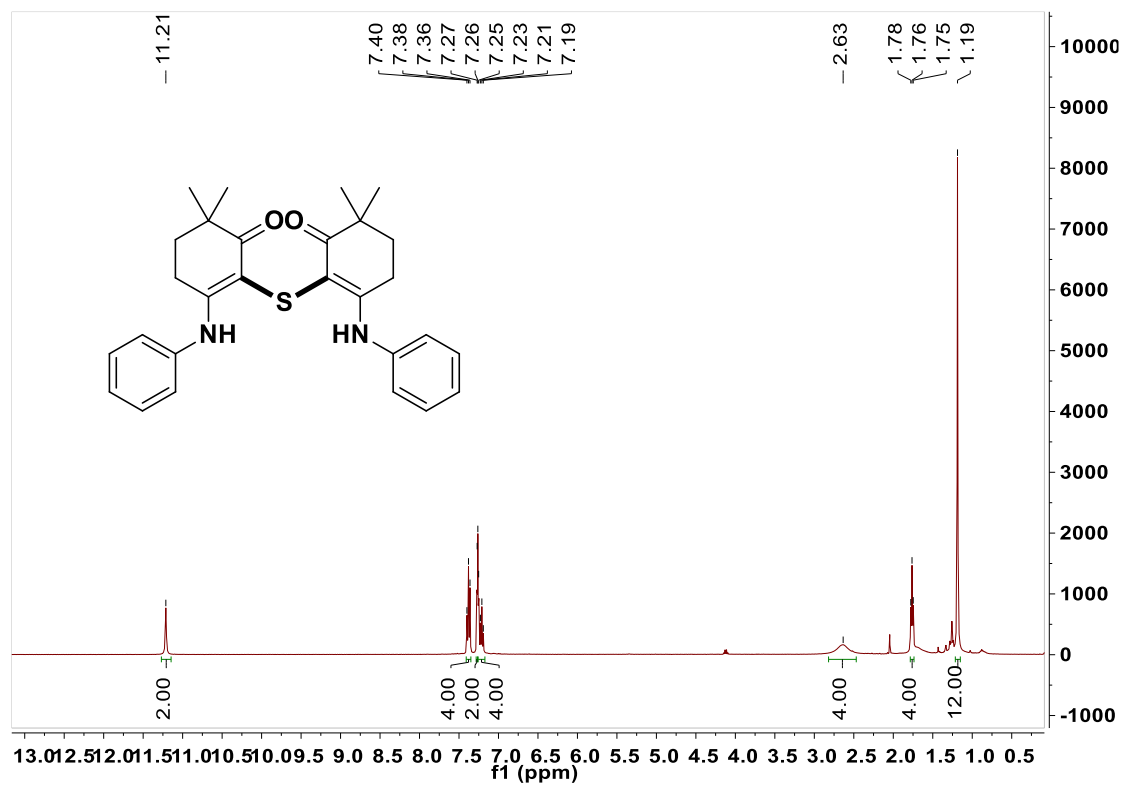
$^{13}\text{C}$  NMR spectrum of compound 3pa



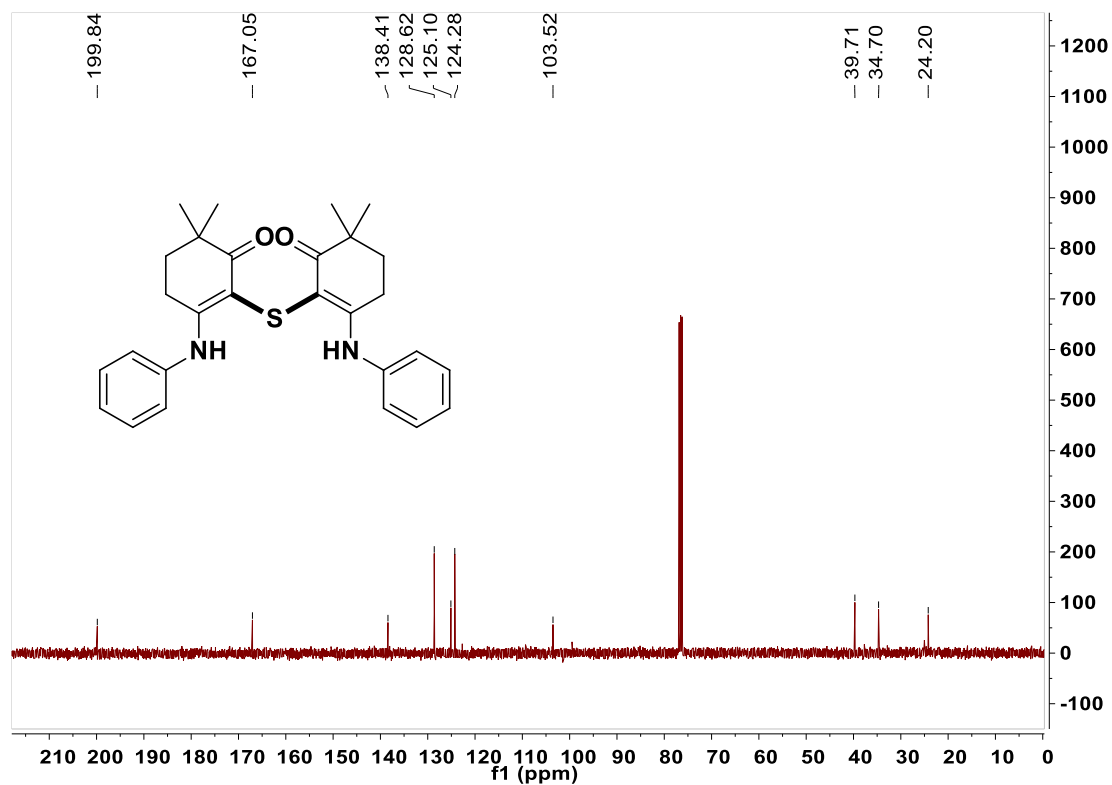
**<sup>1</sup>H NMR spectrum of compound 3qa**



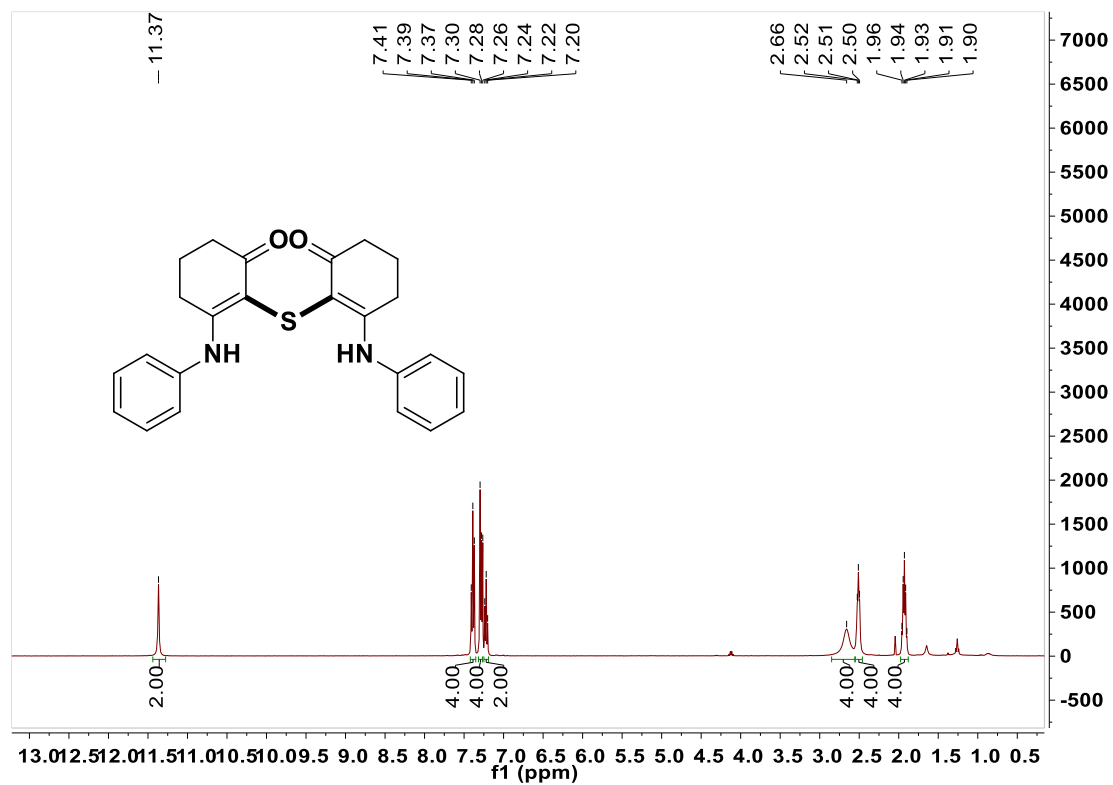
**<sup>13</sup>C NMR spectrum of compound 3qa**



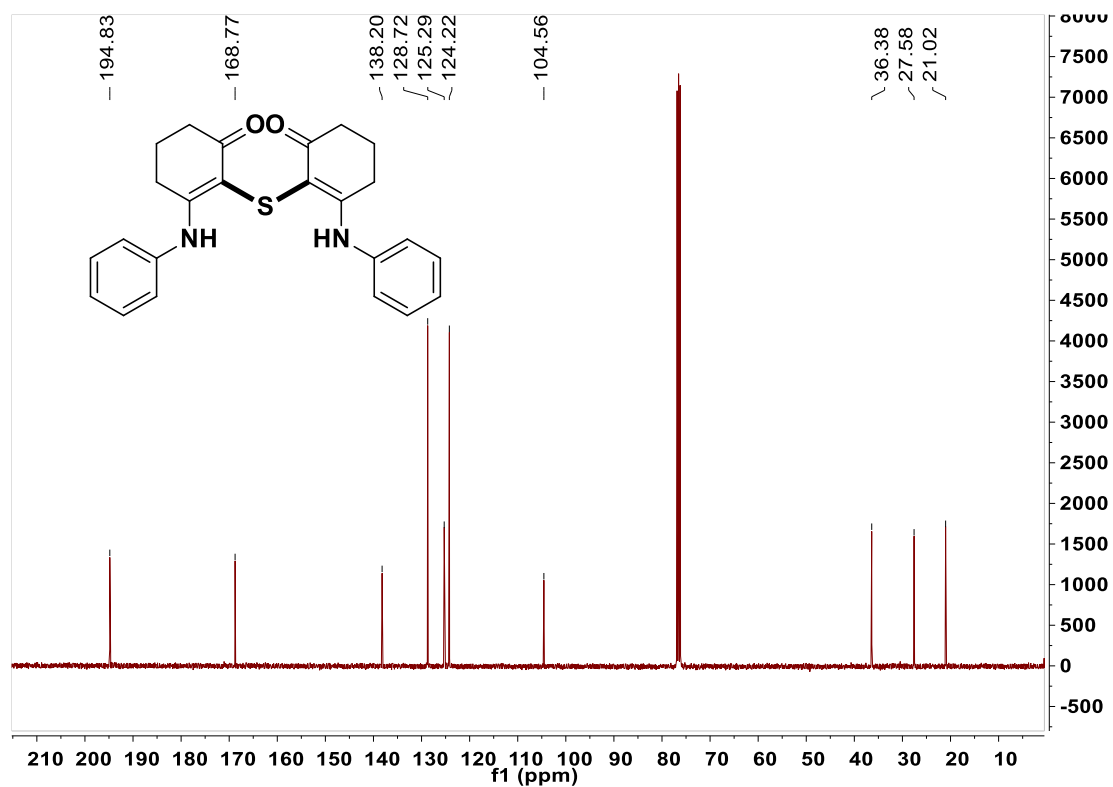
**<sup>1</sup>H NMR spectrum of compound 3ra**



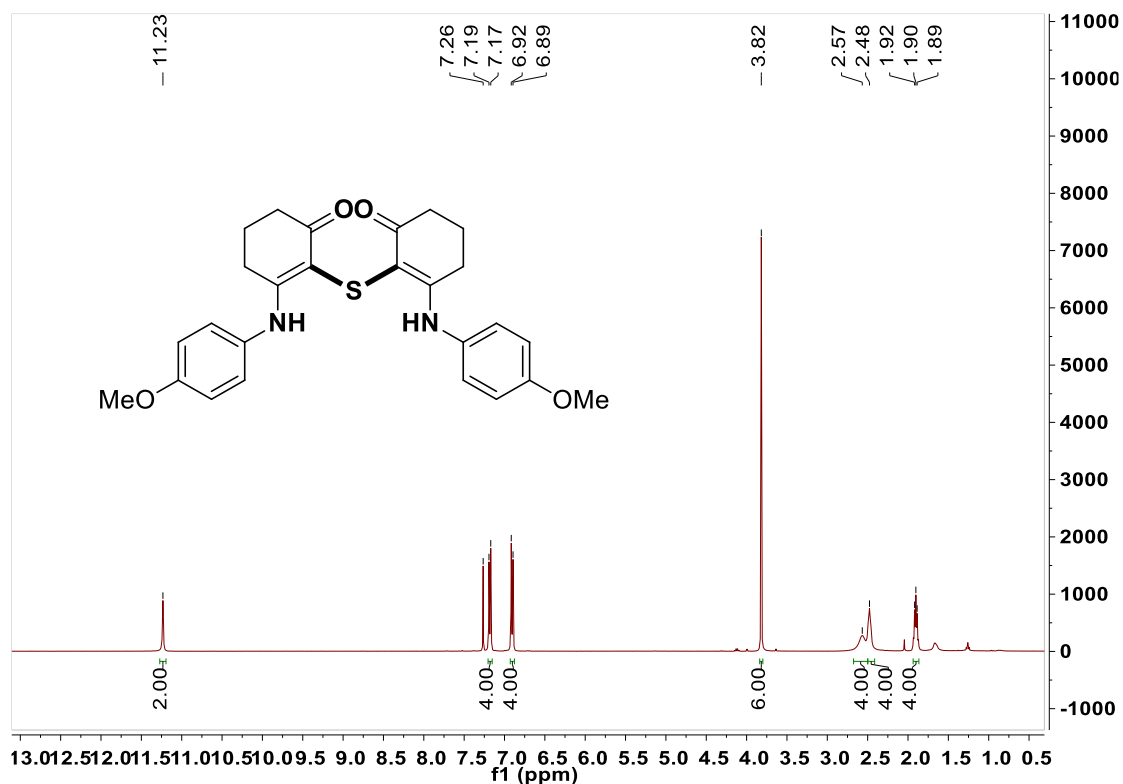
**<sup>13</sup>C NMR spectrum of compound 3ra**



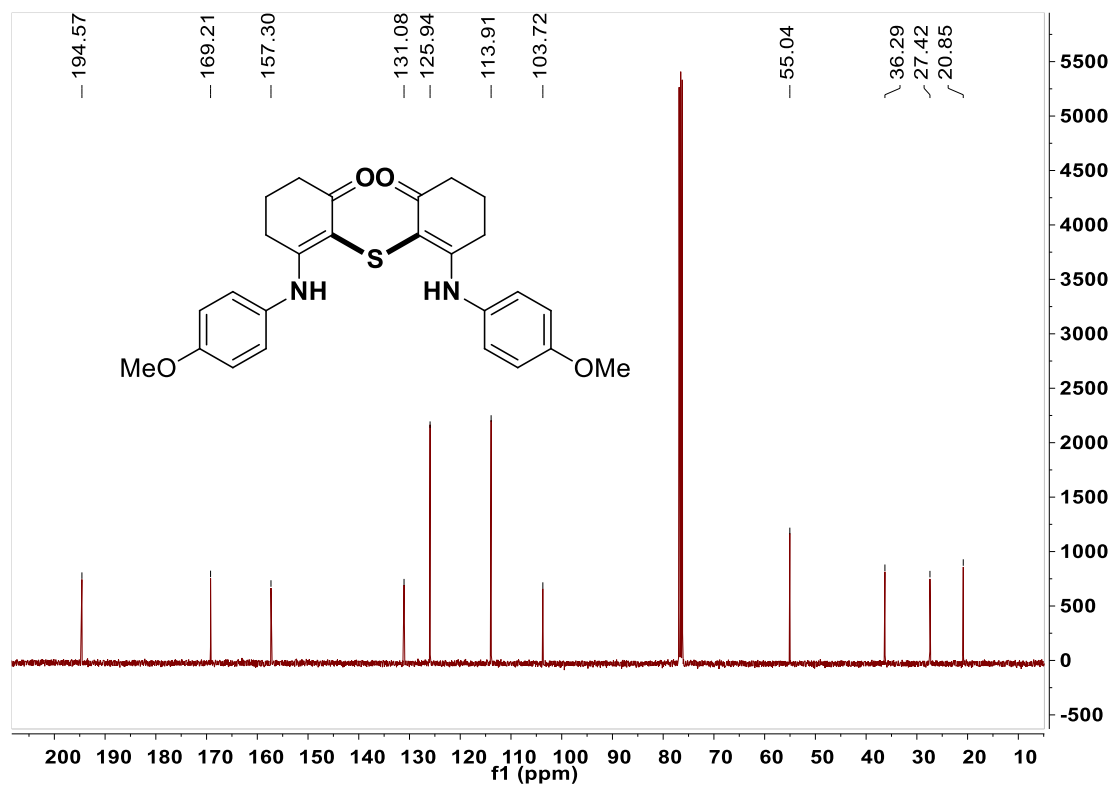
<sup>1</sup>H NMR spectrum of compound 3sa



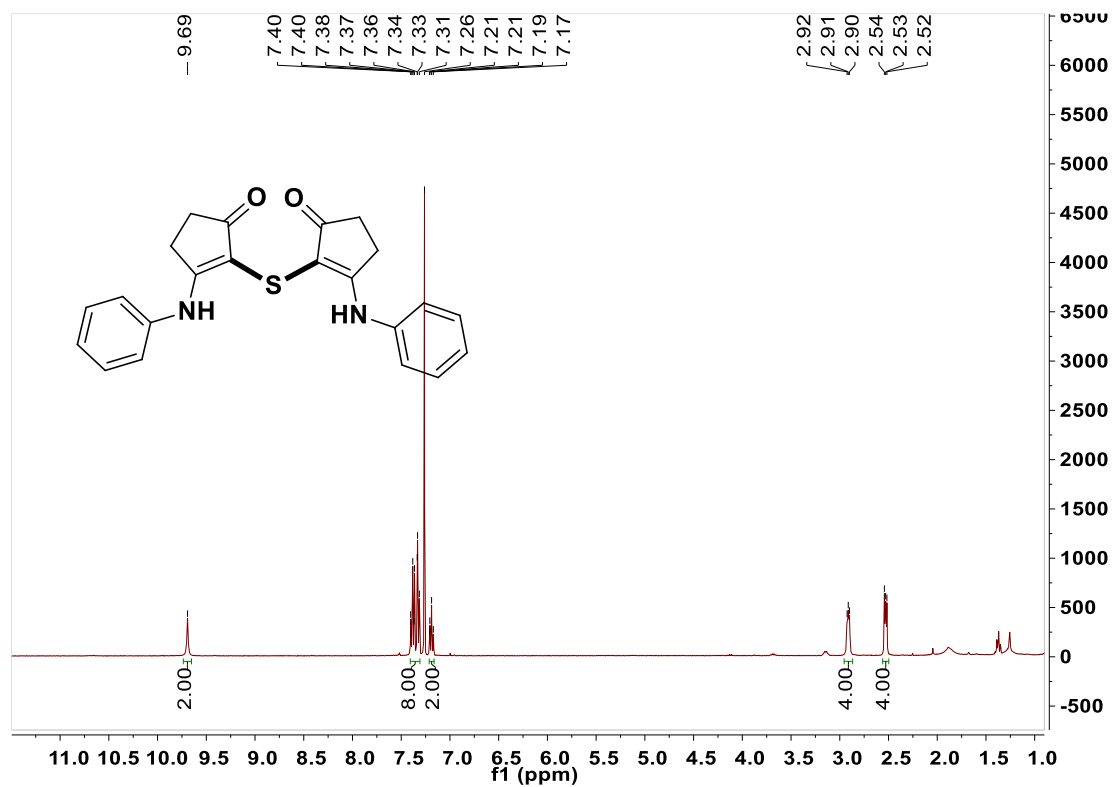
<sup>13</sup>C NMR spectrum of compound 3sa



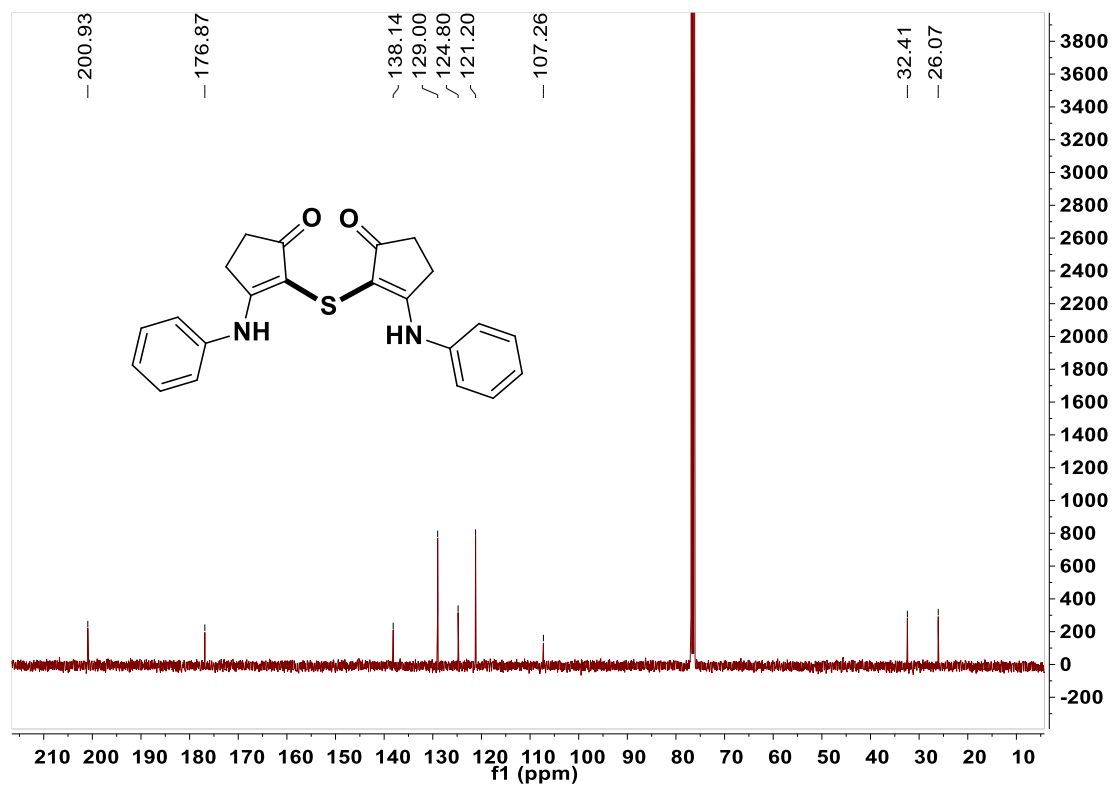
**<sup>1</sup>H NMR spectrum of compound 3ta**



**<sup>13</sup>C NMR spectrum of compound 3ta**

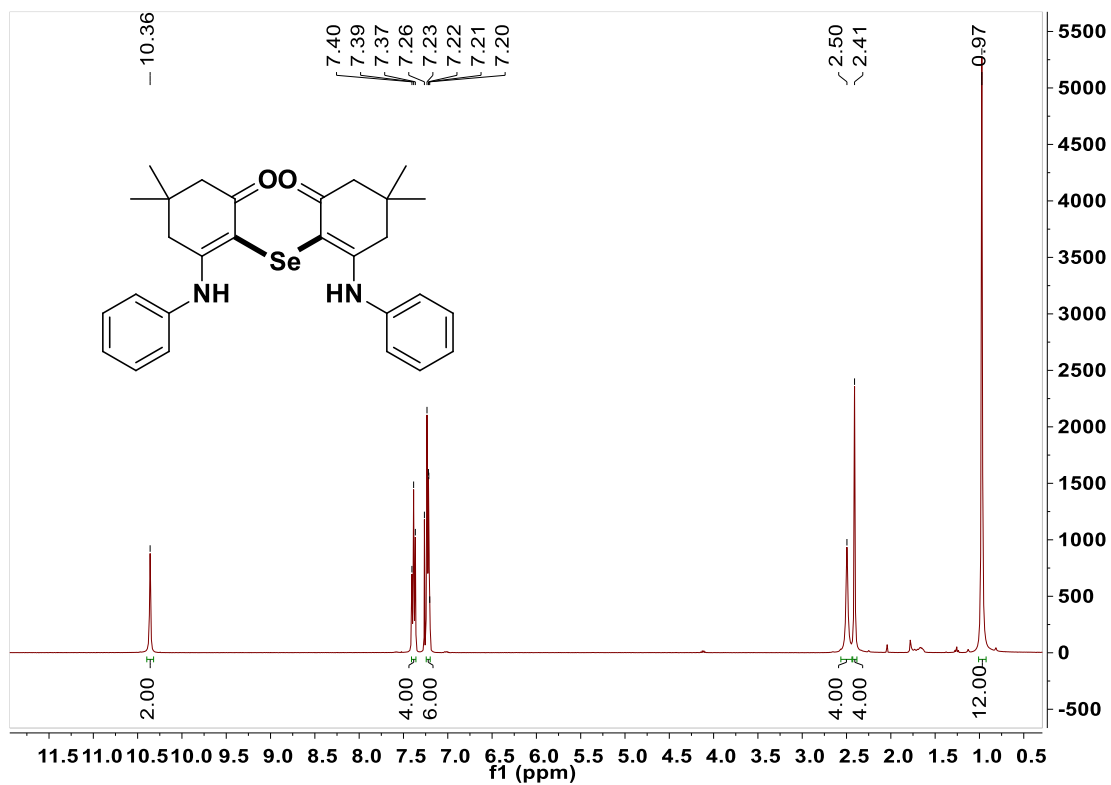


**<sup>1</sup>H NMR spectrum of compound 3ua**

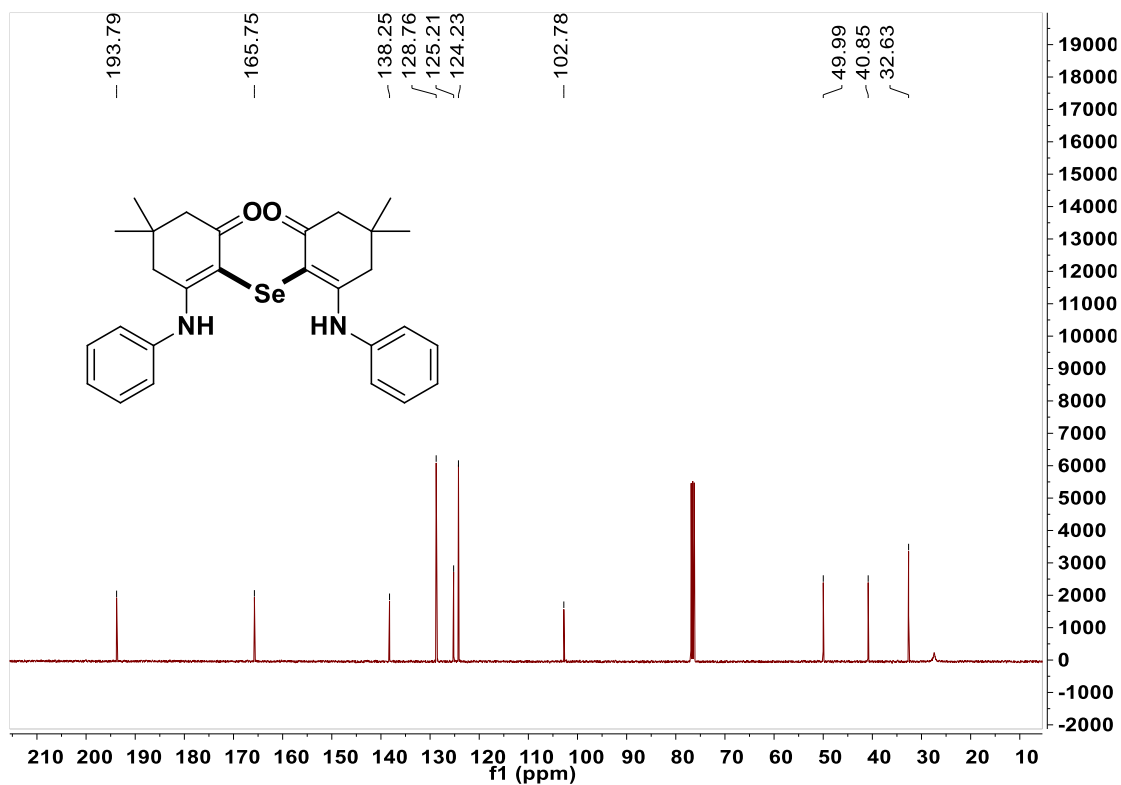


**<sup>13</sup>C NMR spectrum of compound 3ua**

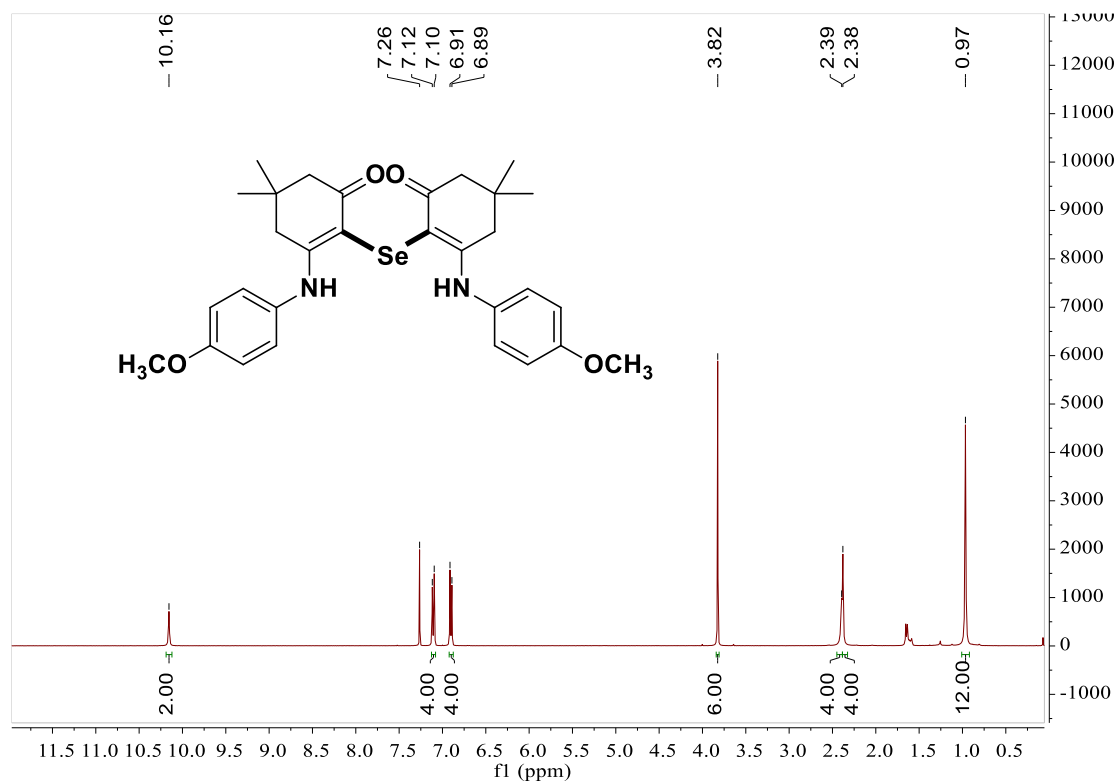




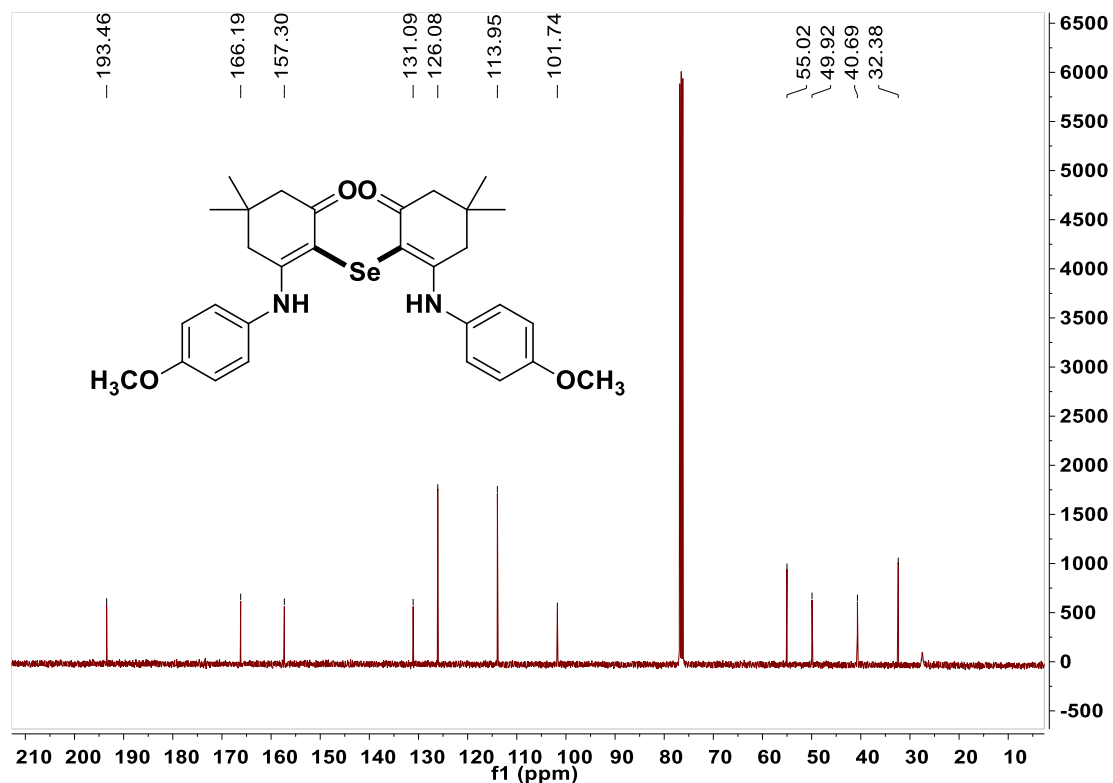
**<sup>1</sup>H NMR spectrum of compound 3ab**



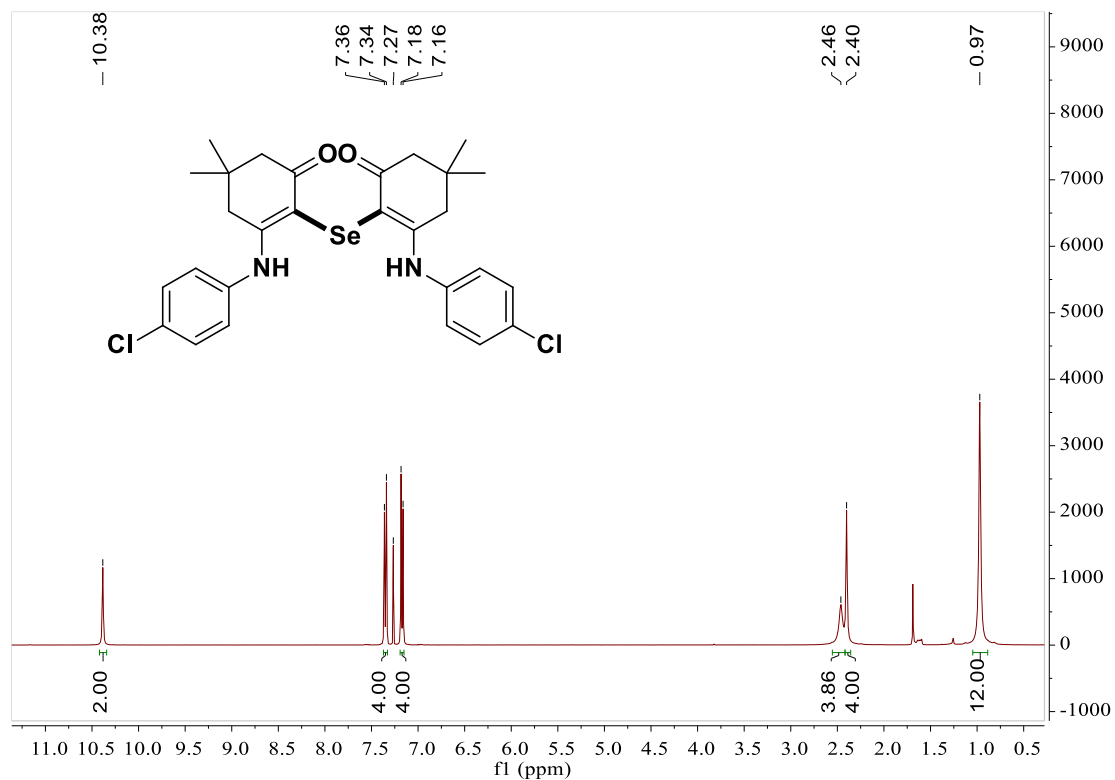
**<sup>13</sup>C NMR spectrum of compound 3ab**



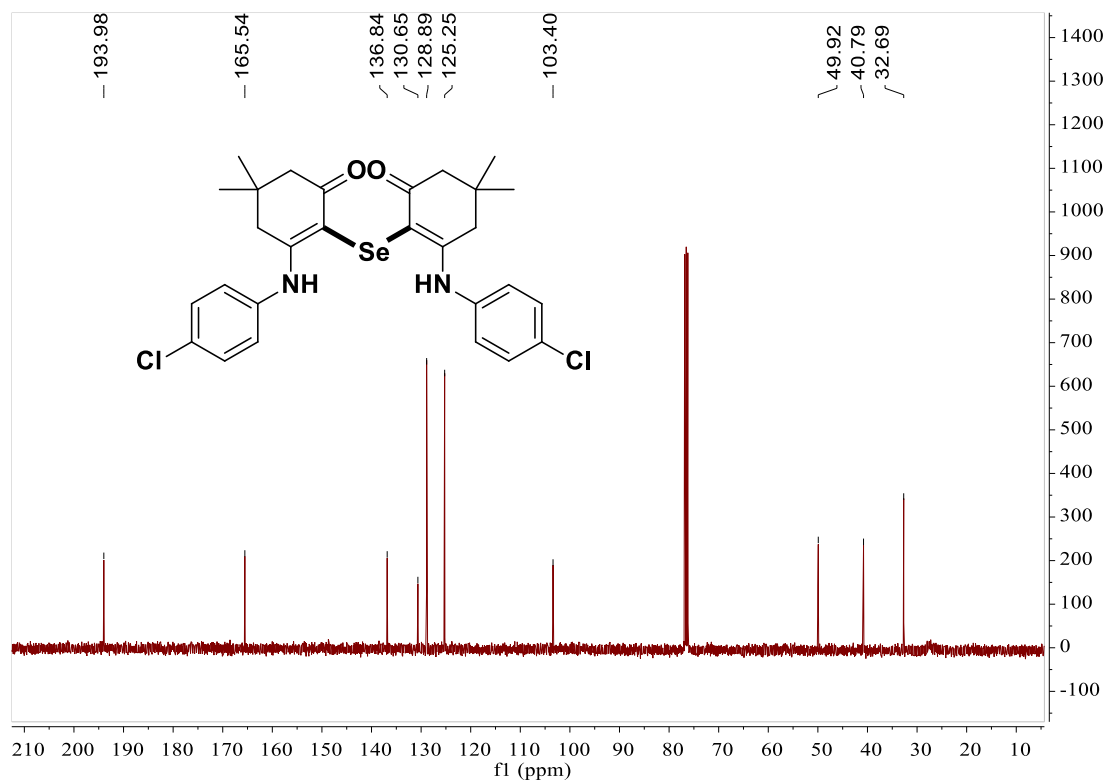
**<sup>1</sup>H NMR spectrum of compound 3bb**



**<sup>13</sup>C NMR spectrum of compound 3bb**



**<sup>1</sup>H NMR spectrum of compound 3eb**



**<sup>13</sup>C NMR spectrum of compound 3eb**