**Supporting Information**

**Design, synthesis and anticancer activity of novel 3,6-diunsaturated 2,5-diketopiperazines**

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**Copies of NMR spectra for the derivatives**

Note: **3** and **7** are known compounds1-2

Figure **S1** 1H NMR of **1**

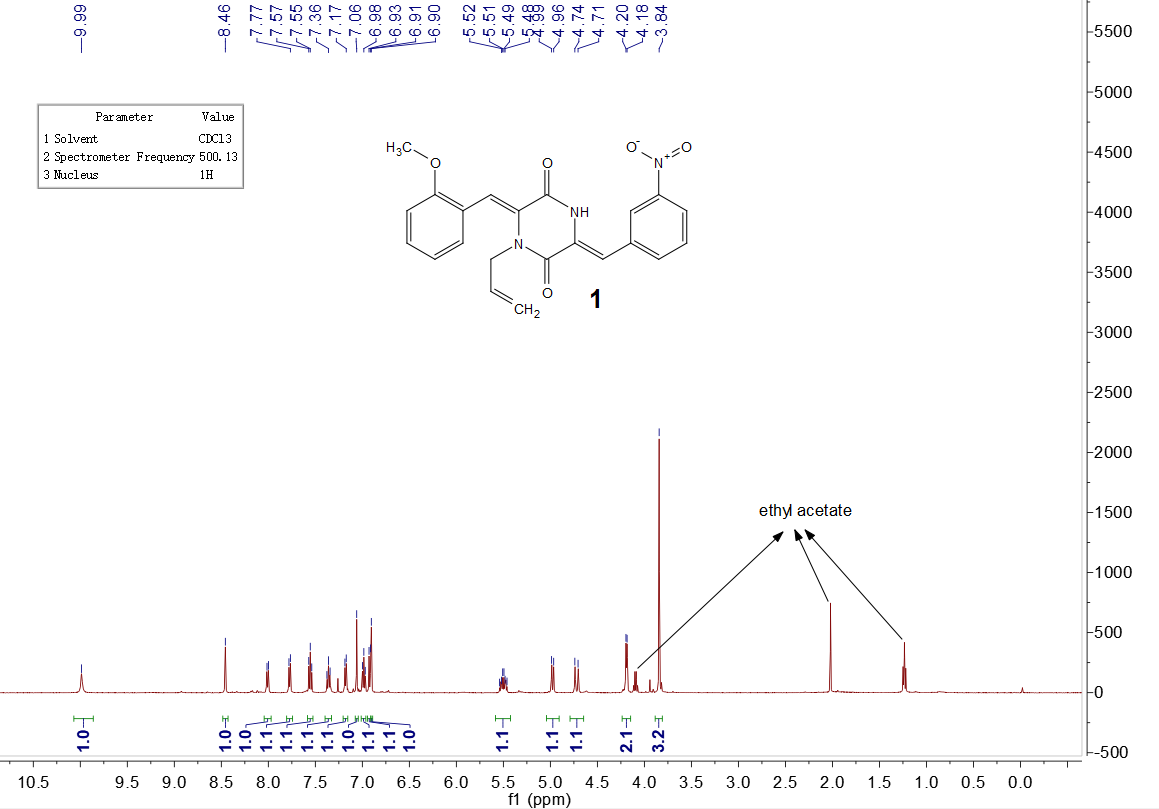


Figure **S2** 13C NMR of **1**

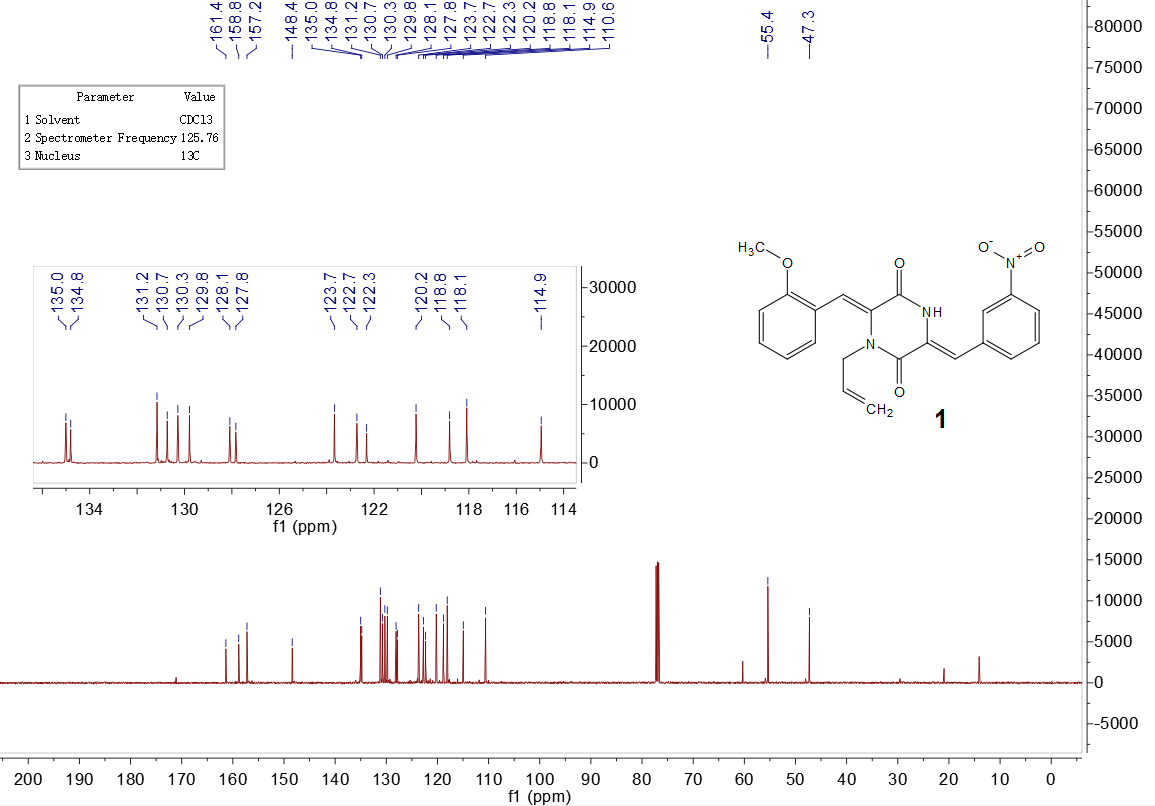


Figure **S3** 1H NMR of **2**

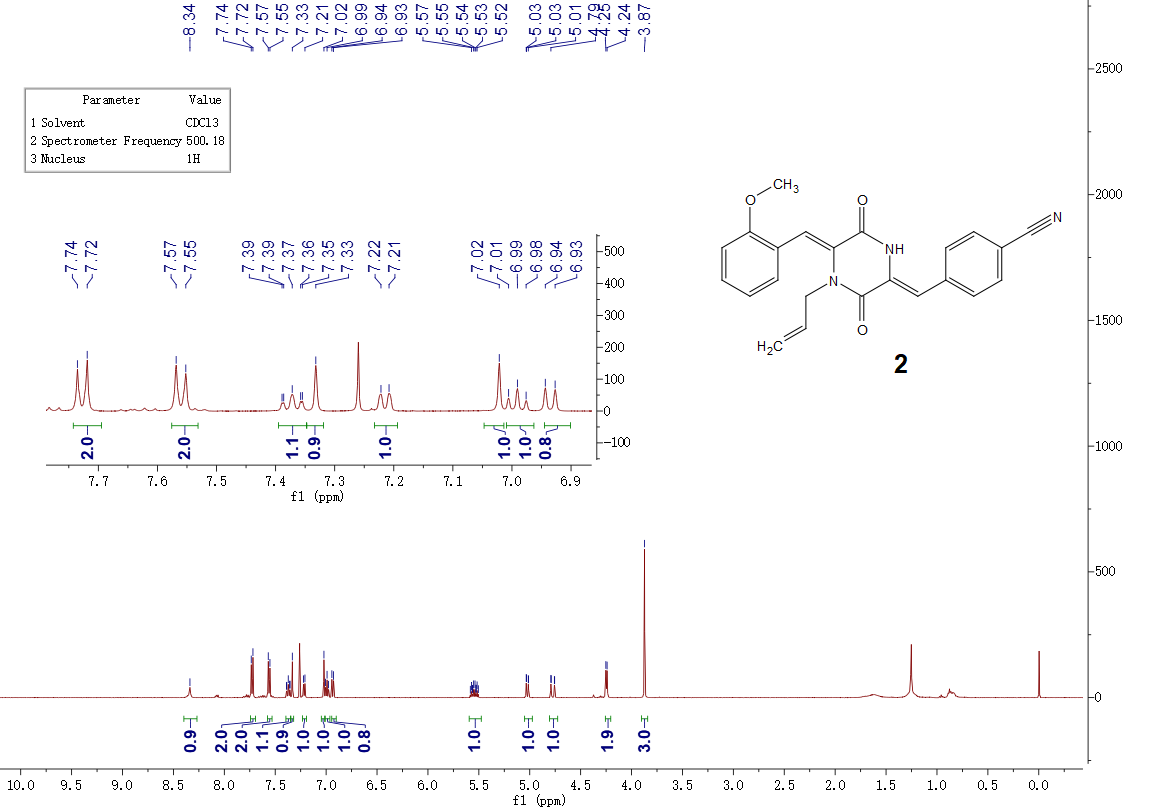


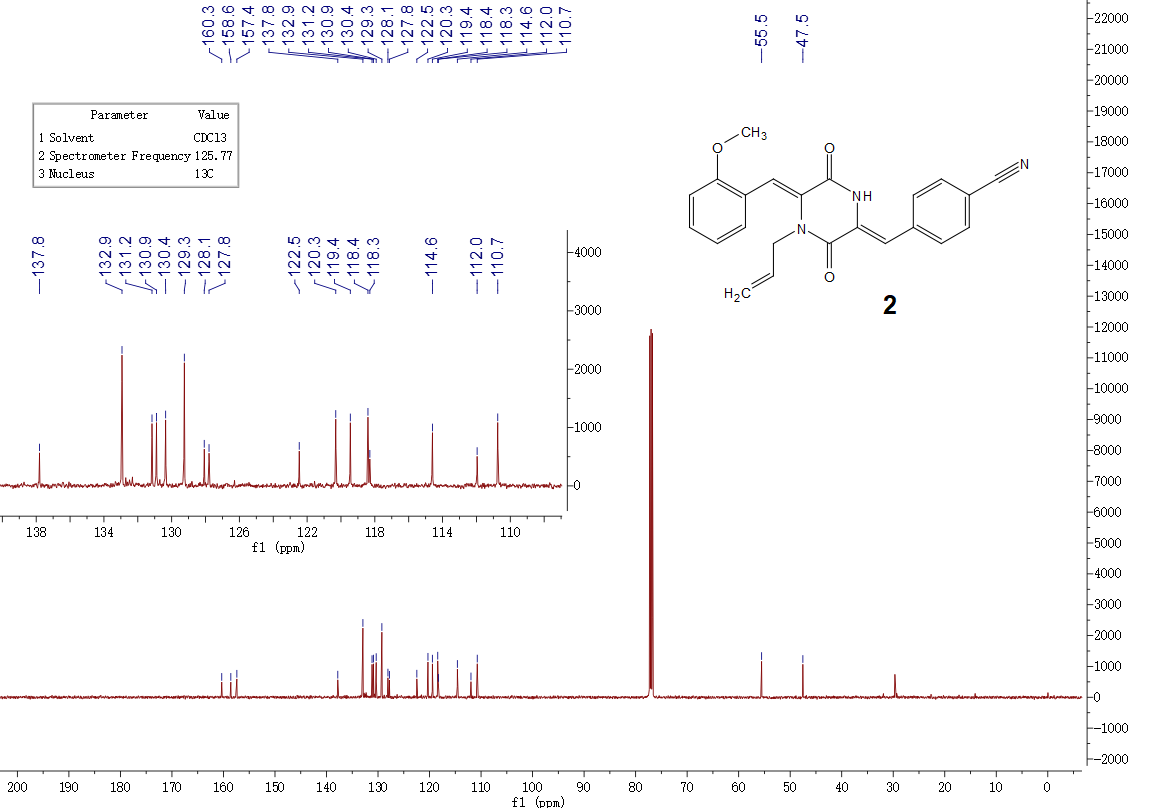
Figure **S4** 13C NMR of **2** 

Figure **S5** 1H NMR of **4**

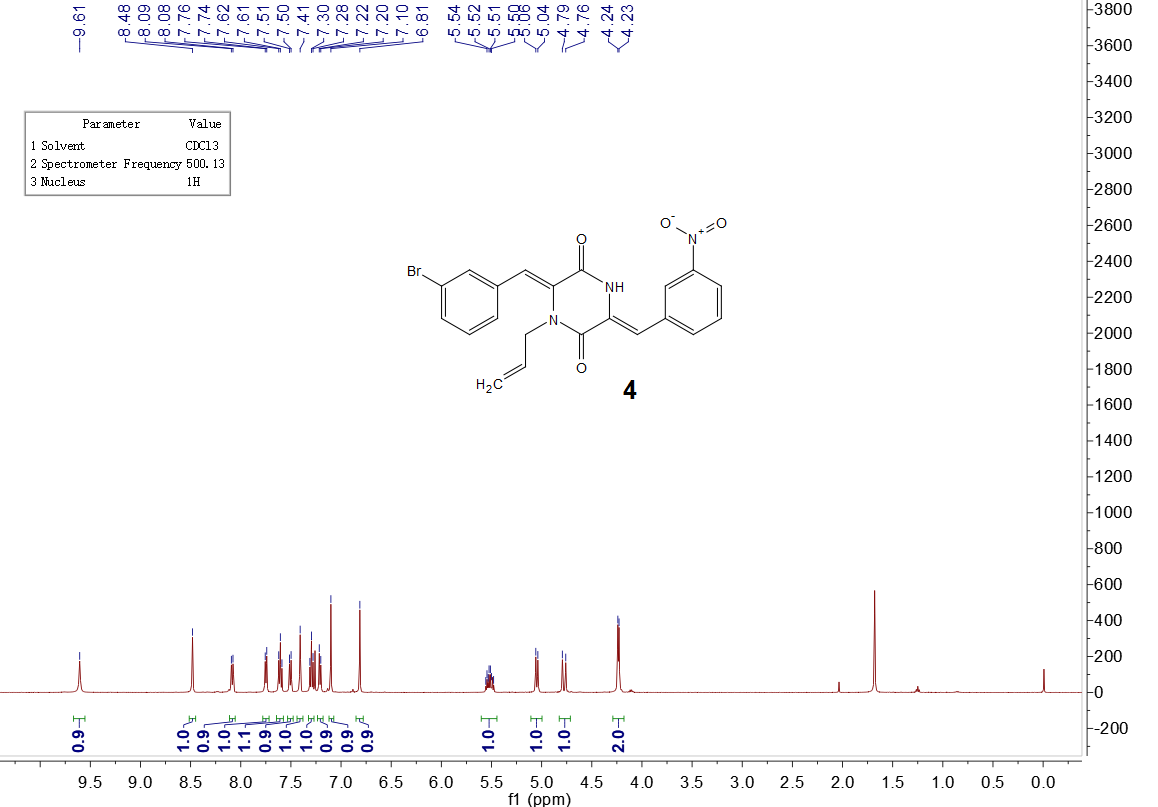


Figure **S6** 13C NMR of **4**

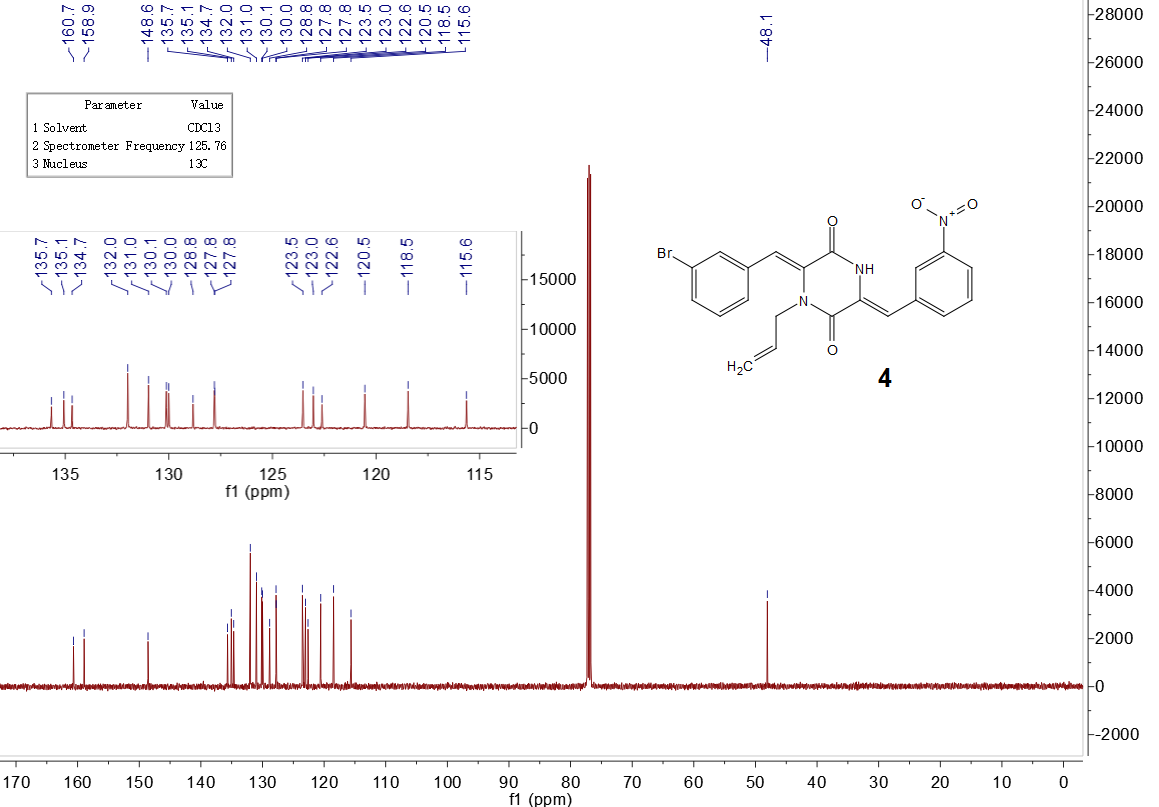


Figure **S7** 1H NMR of **5**

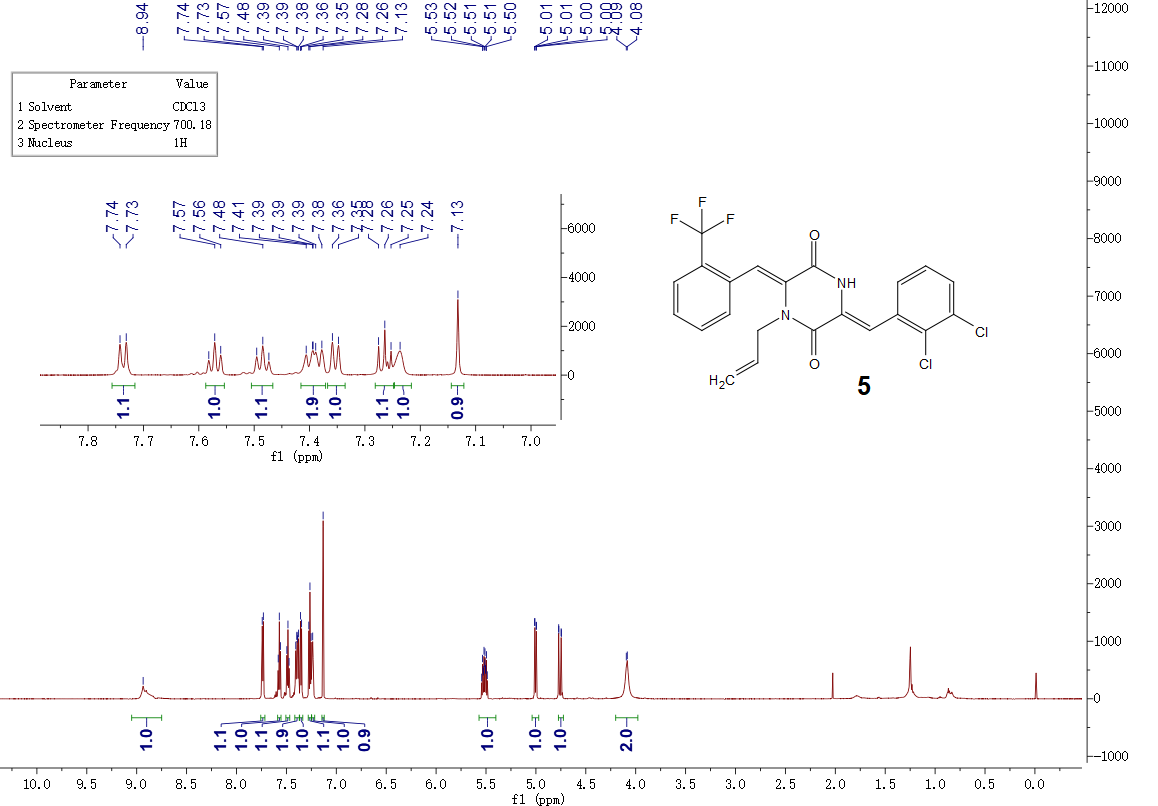


Figure **S8** 13C NMR of **5**

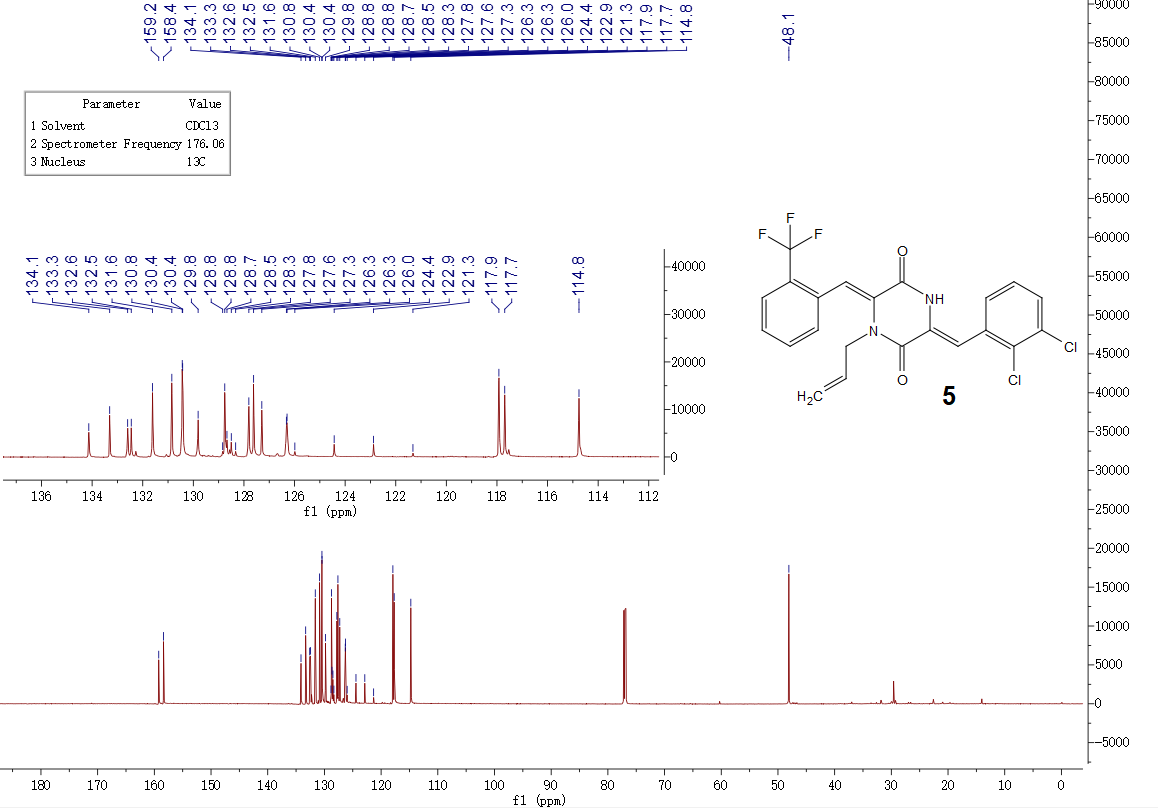


Figure **S9** 1H NMR of **6**

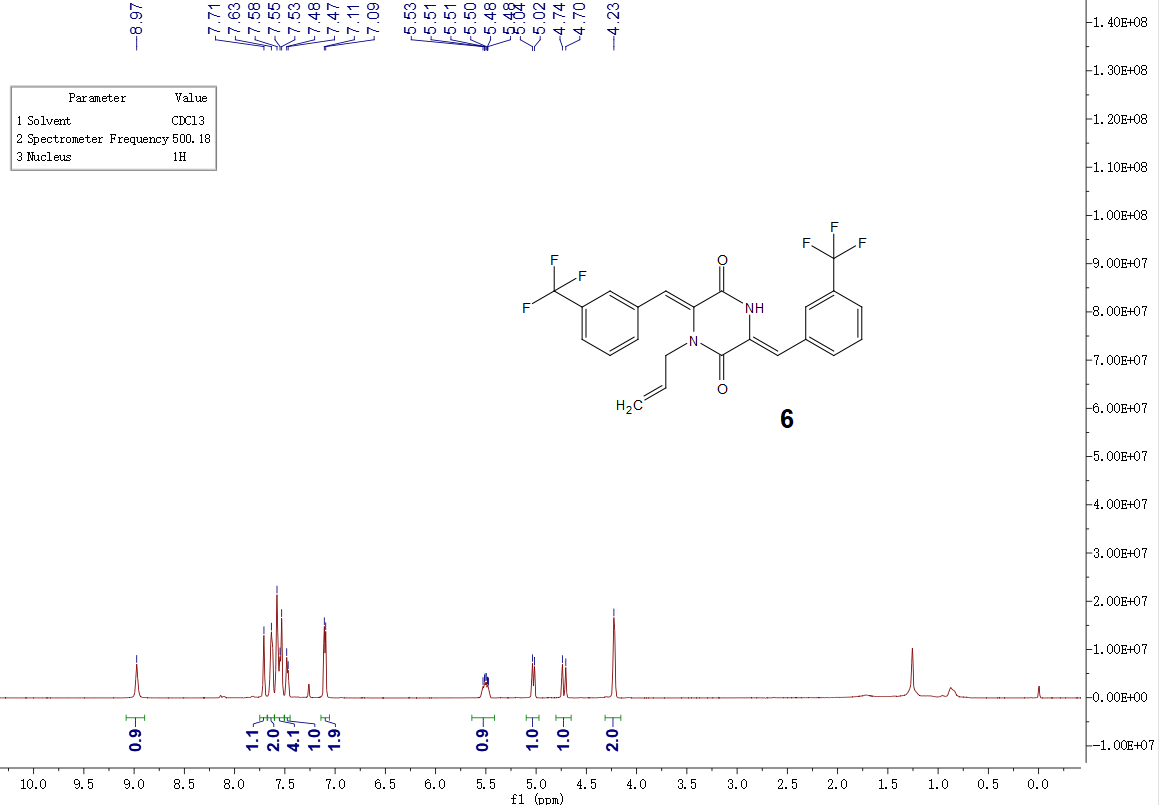


Figure **S10** 13C NMR of **6**

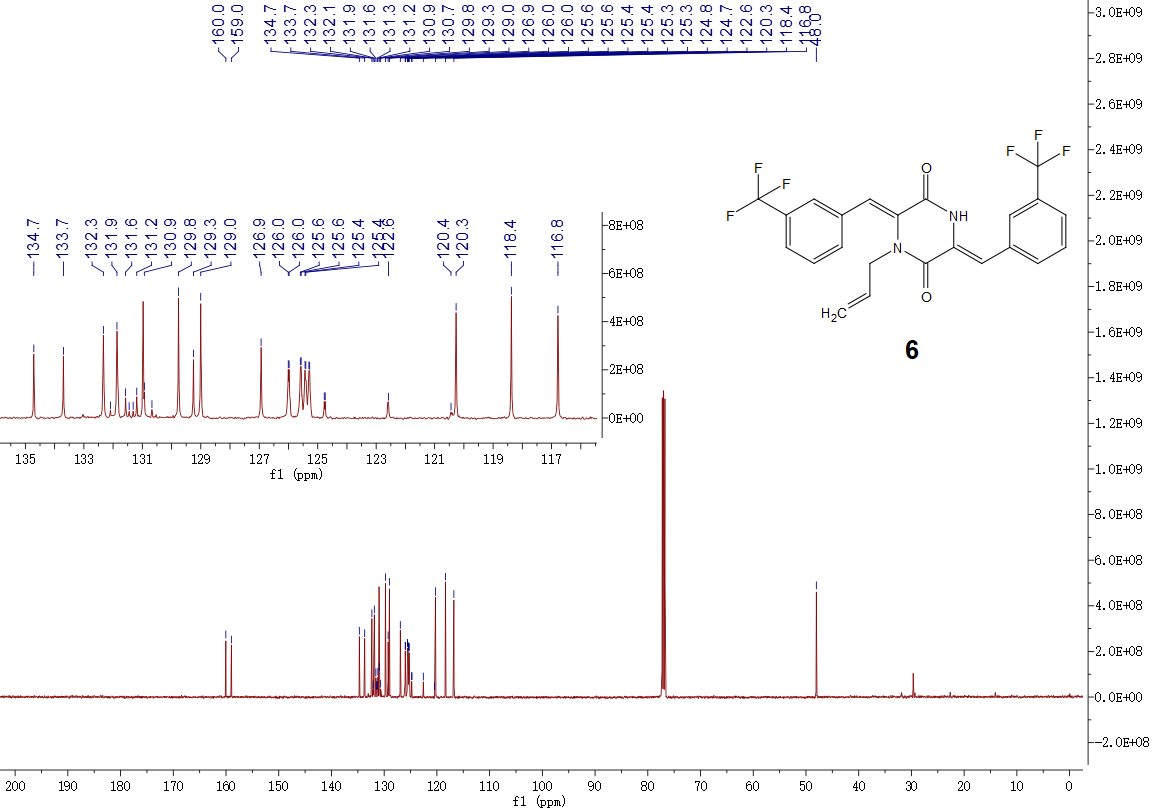


Figure **S11** 1H NMR of **8**

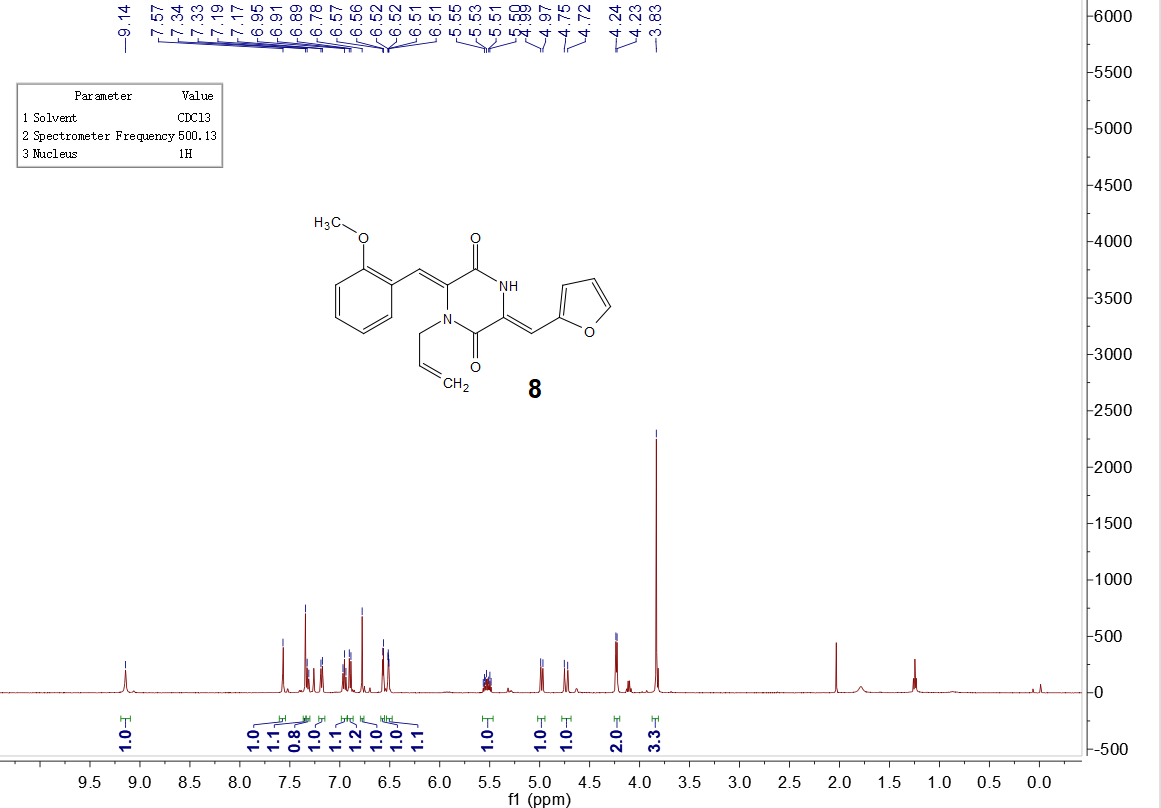


Figure **S12** 13C NMR of **8**

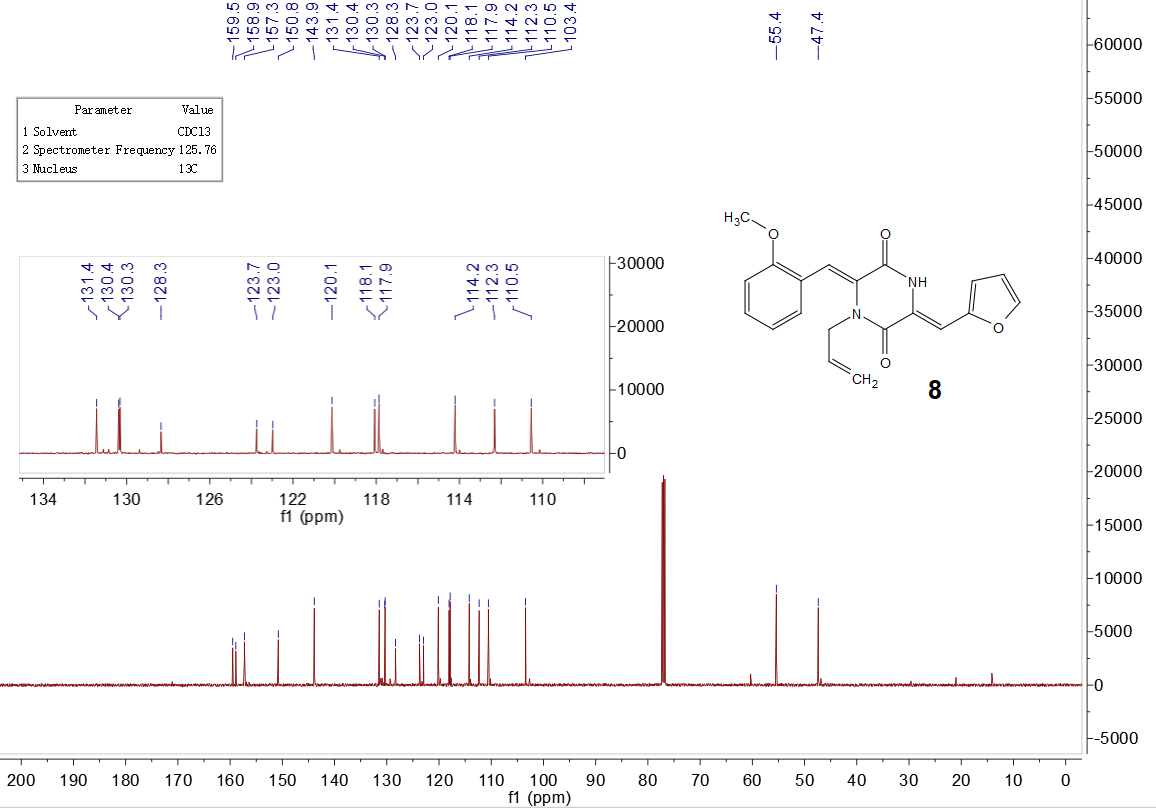


Figure **S13** 1H NMR of **9**

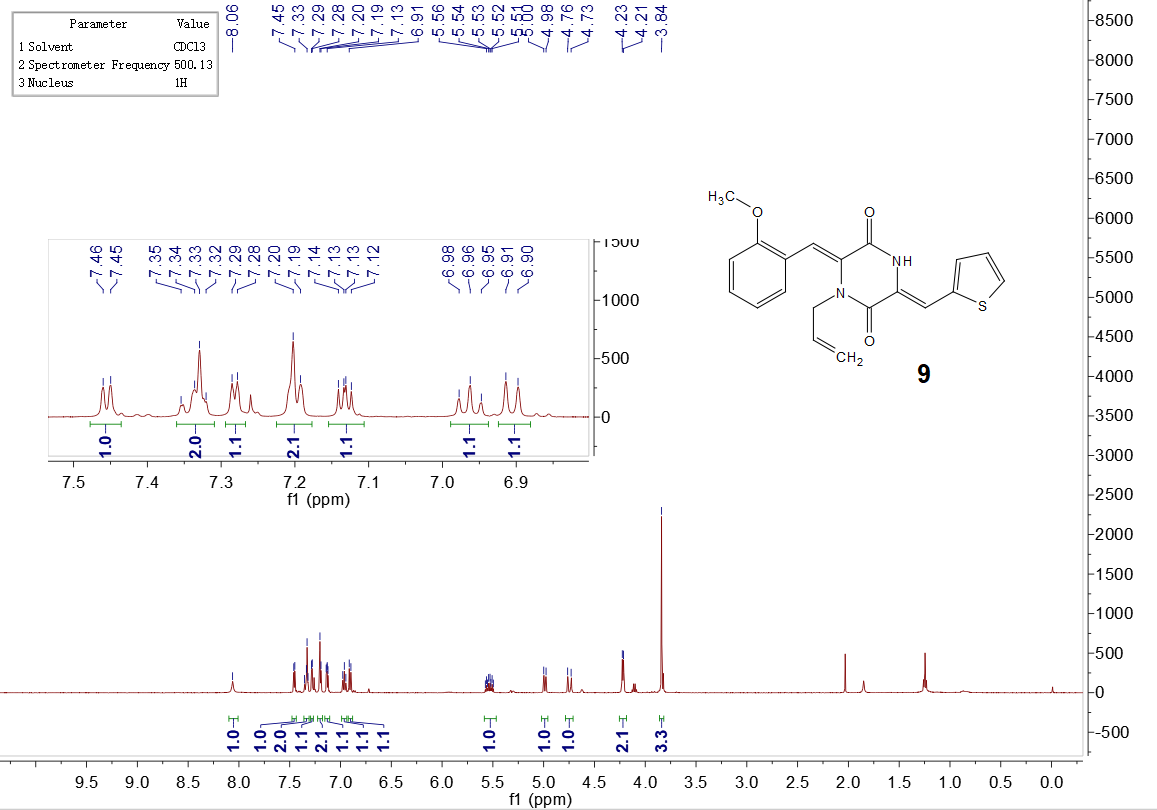


Figure **S14** 13C NMR of **9**

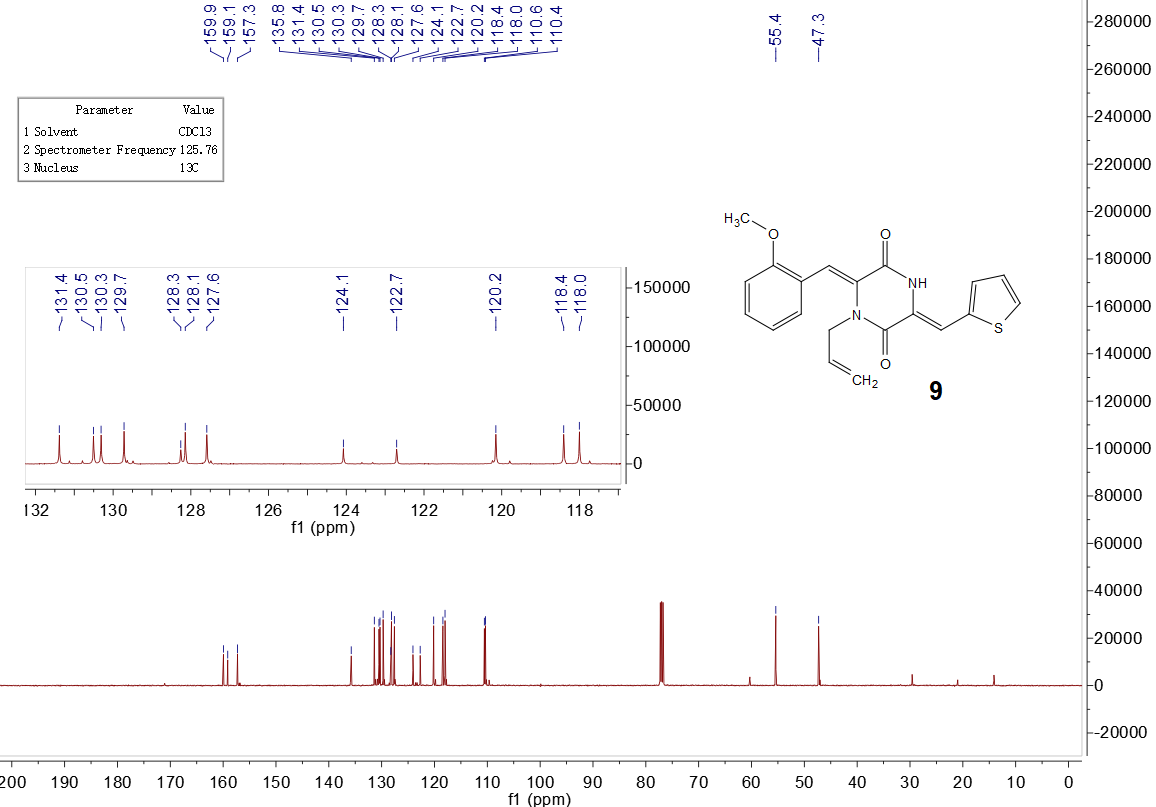


Figure **S15** 1H NMR of **10**

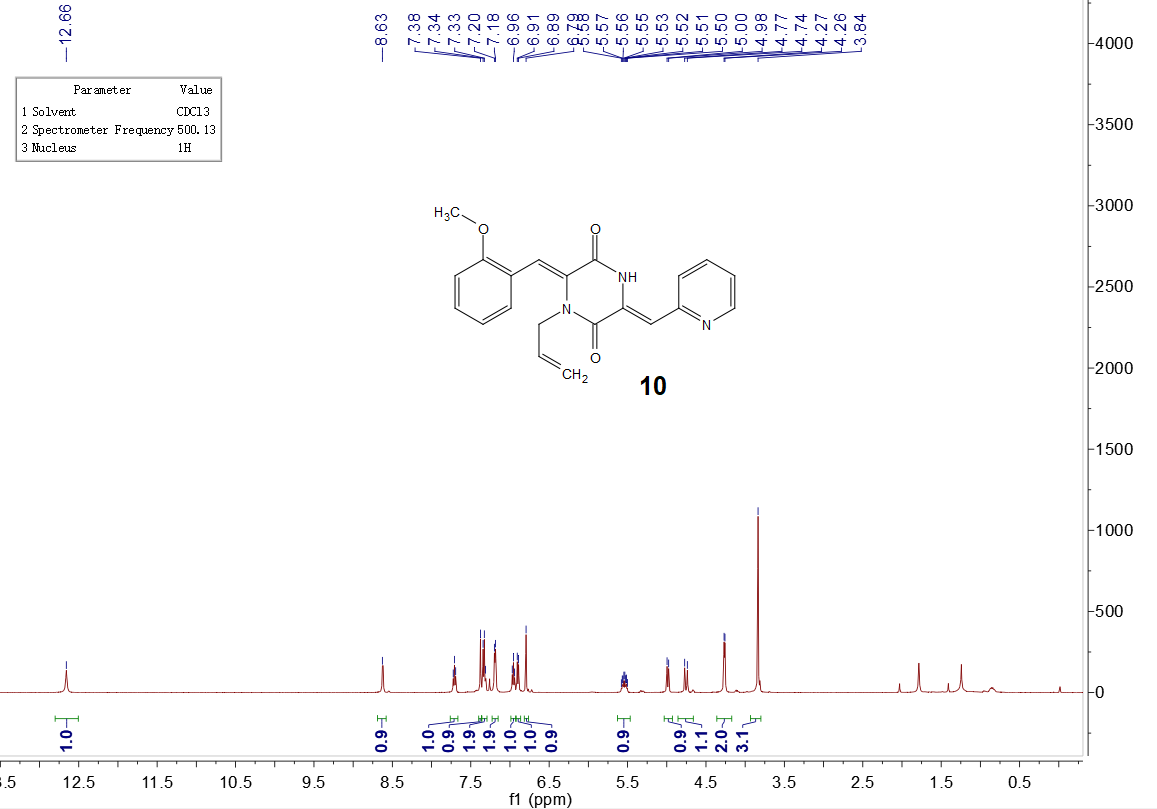


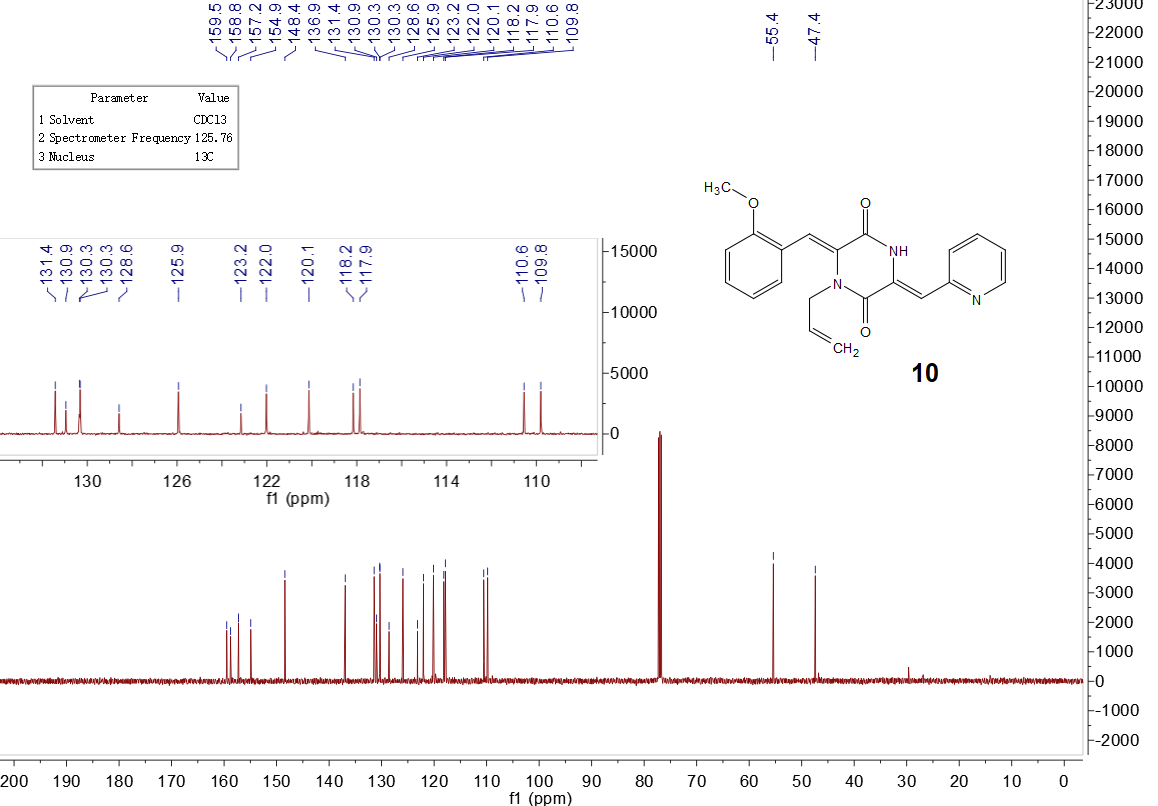
Figure **S16** 13C NMR of **10** 

Figure **S17** 1H NMR of **11**

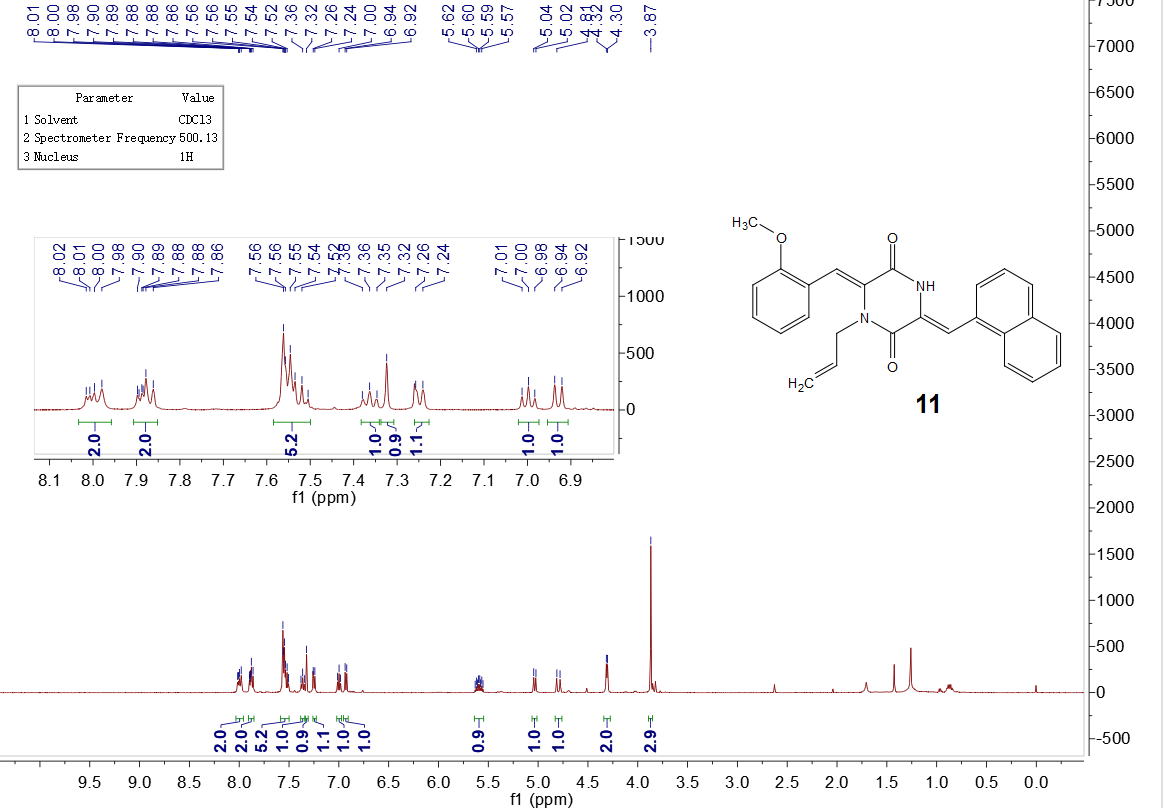


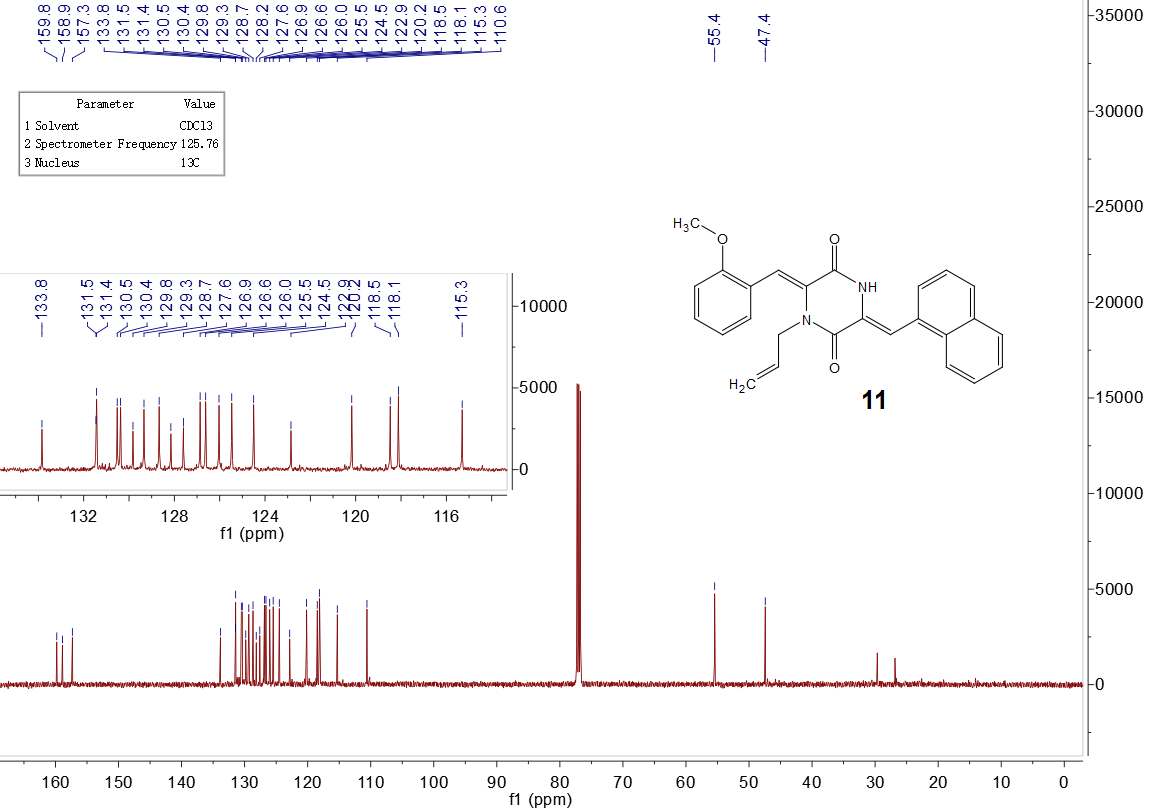
Figure **S18** 13C NMR of **11** 

Figure **S19** 1H NMR of **12**

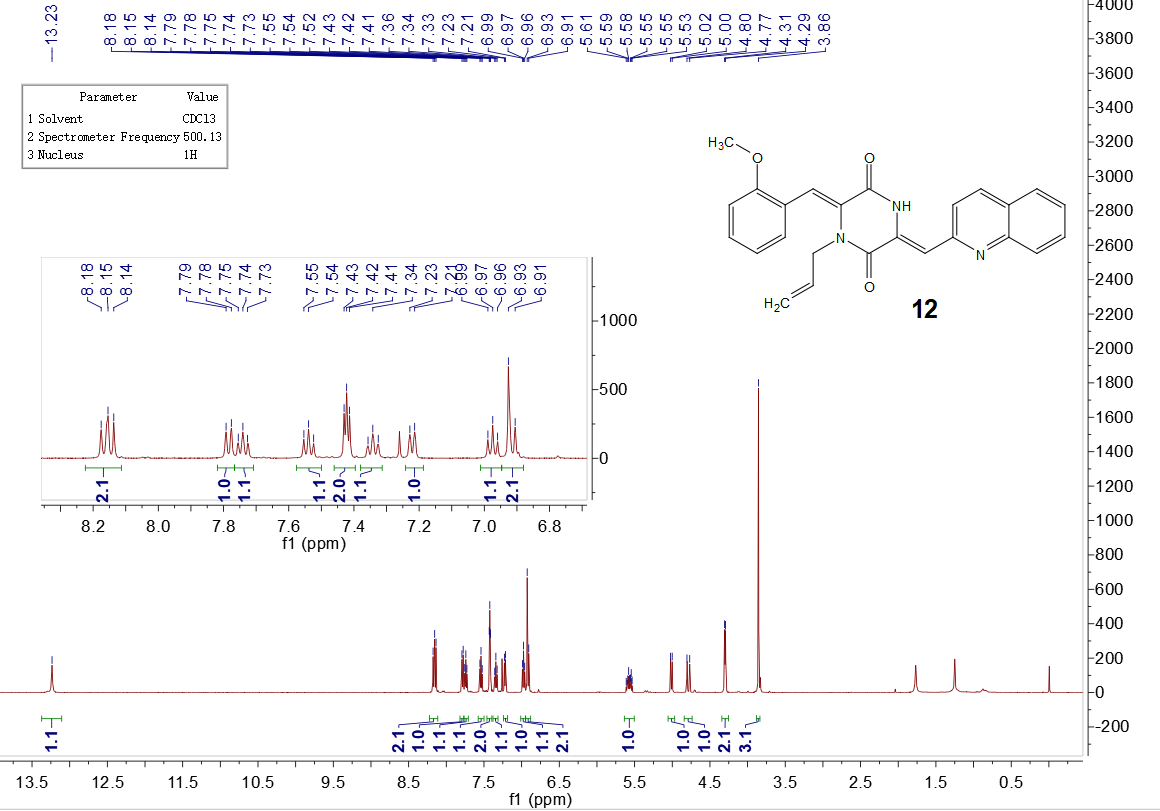


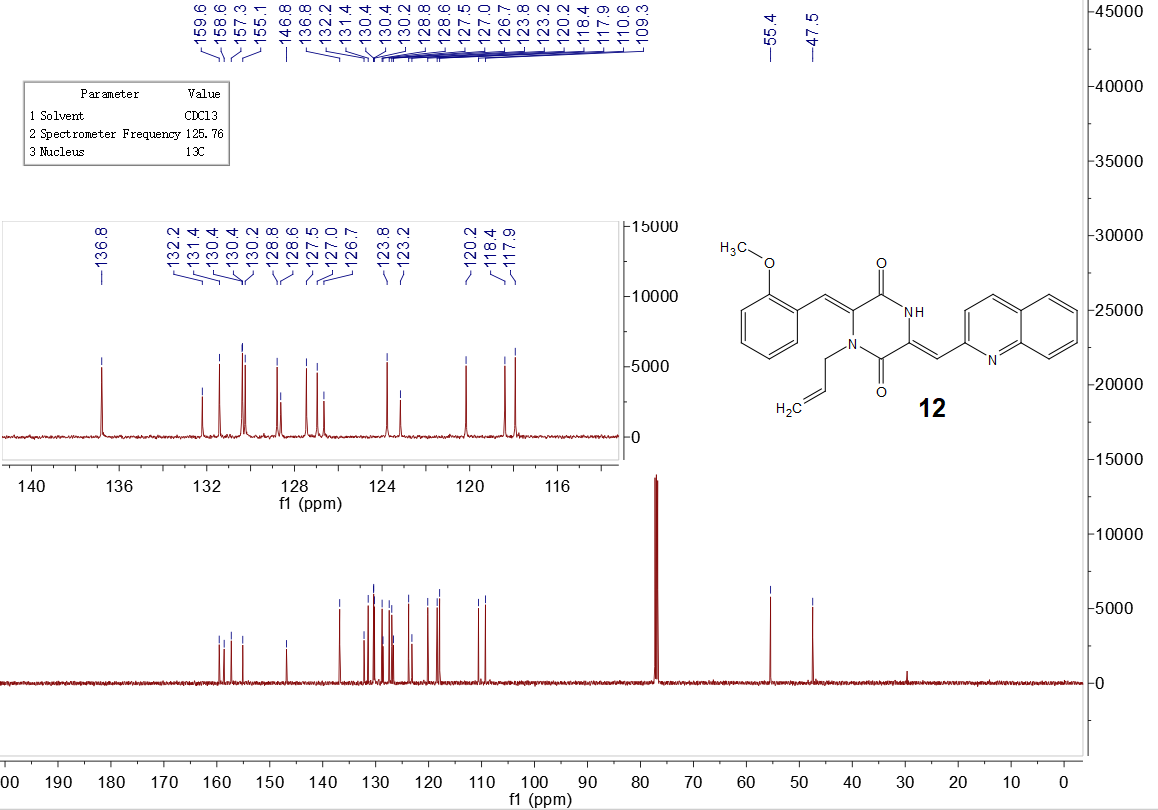
Figure **S20** 13C NMR of **12** 

Figure **S21** 1H NMR of **13**

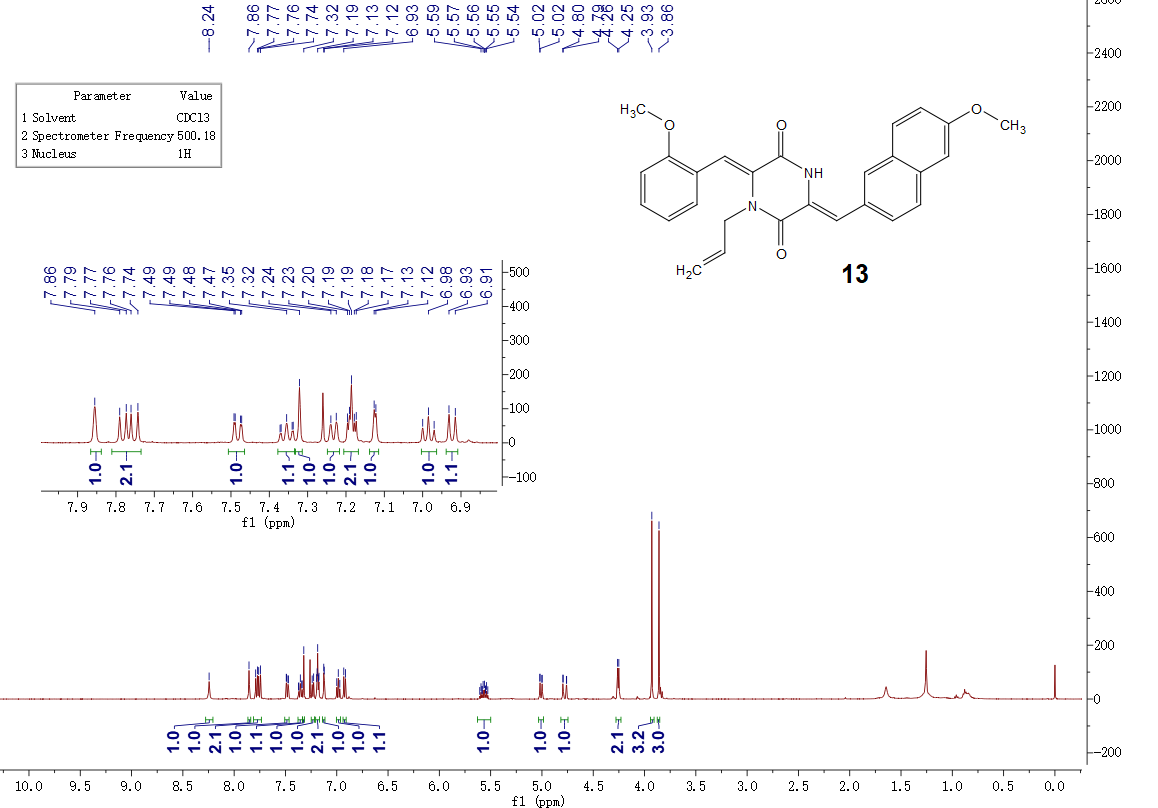


Figure **S22** 13C NMR of **13**

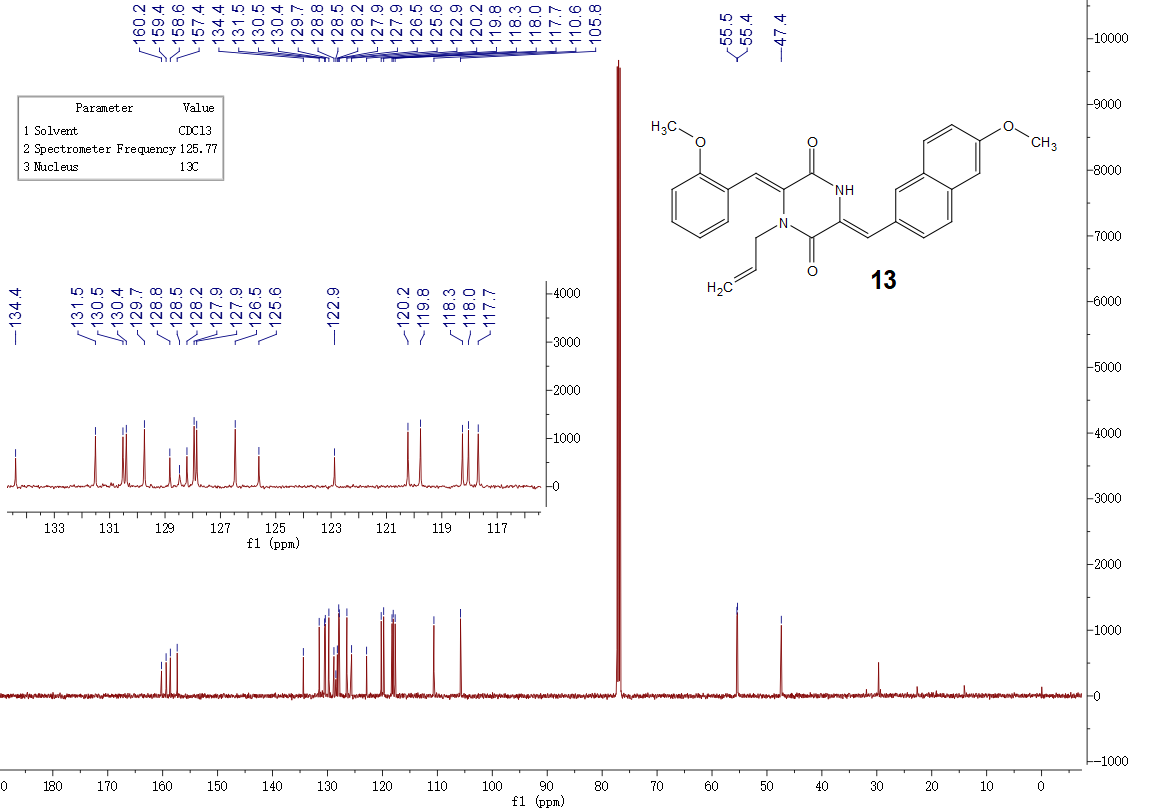


Figure **S23** 1H NMR of **14**

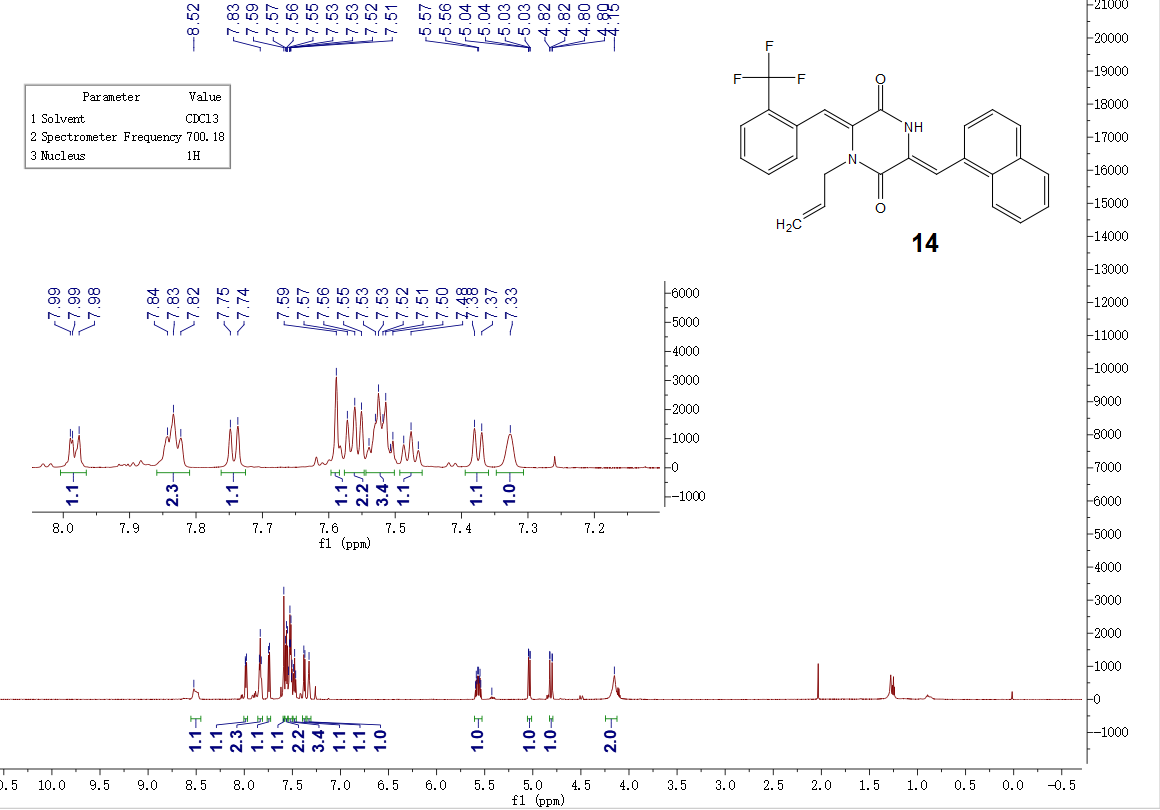


Figure **S24** 13C NMR of **14**



Figure **S25** 1H NMR of **15**

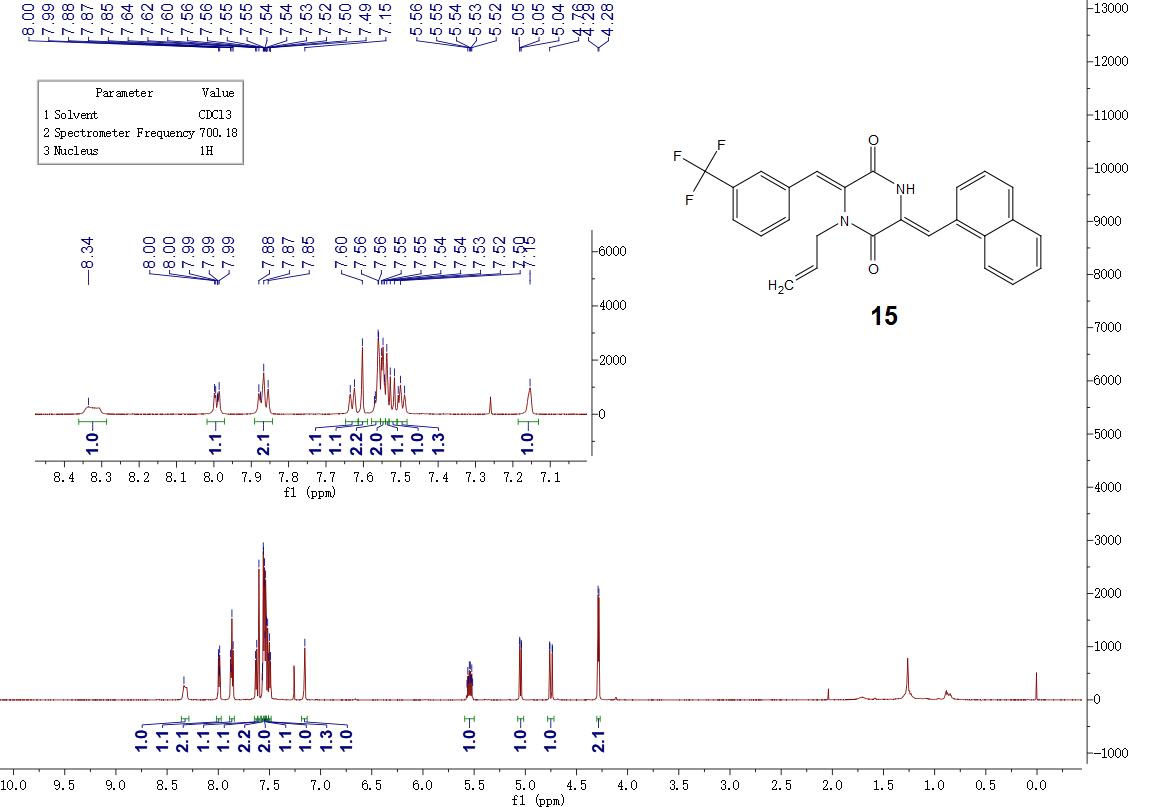


Figure **S26** 13C NMR of **15**

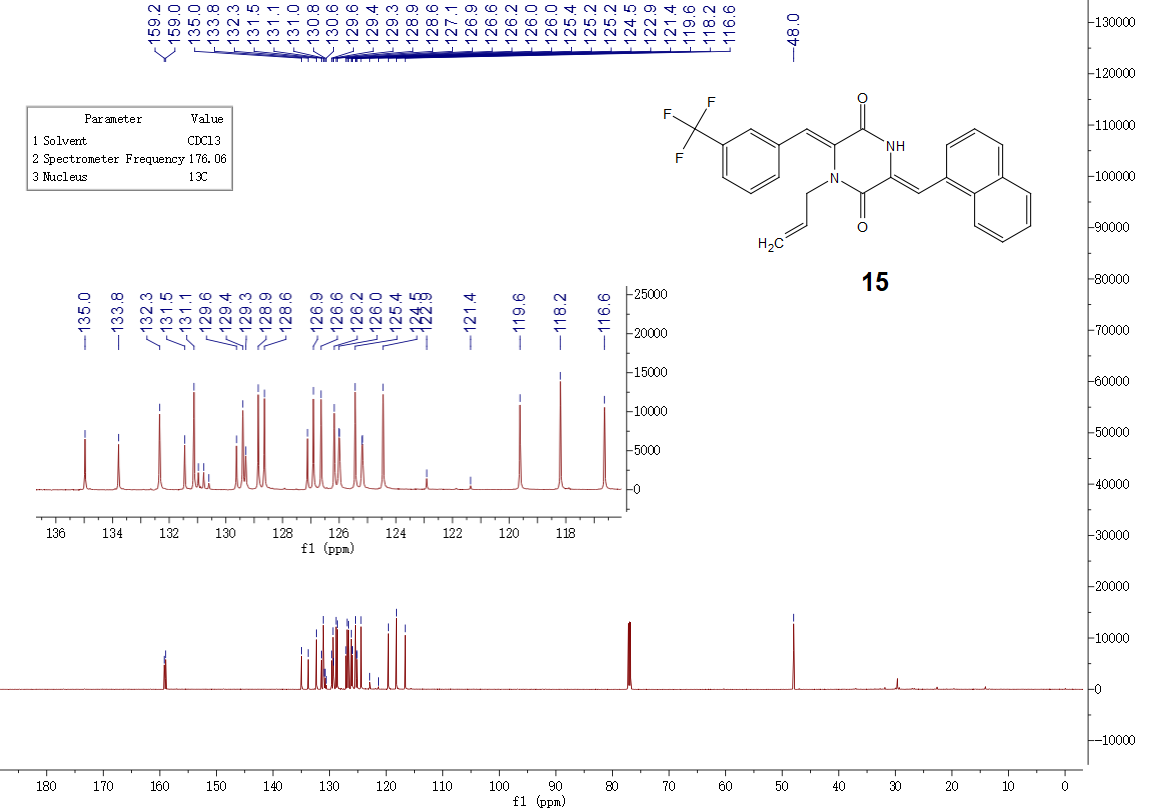


Figure **S27** 1H NMR of **16**

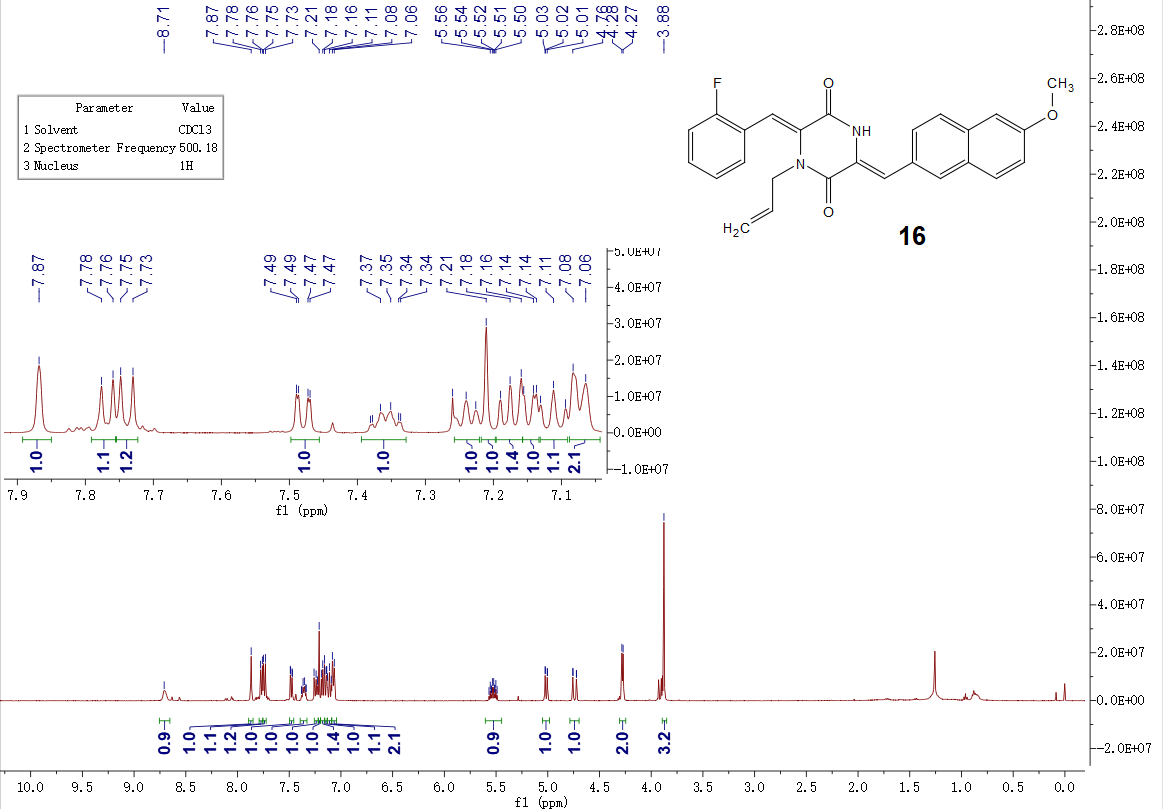
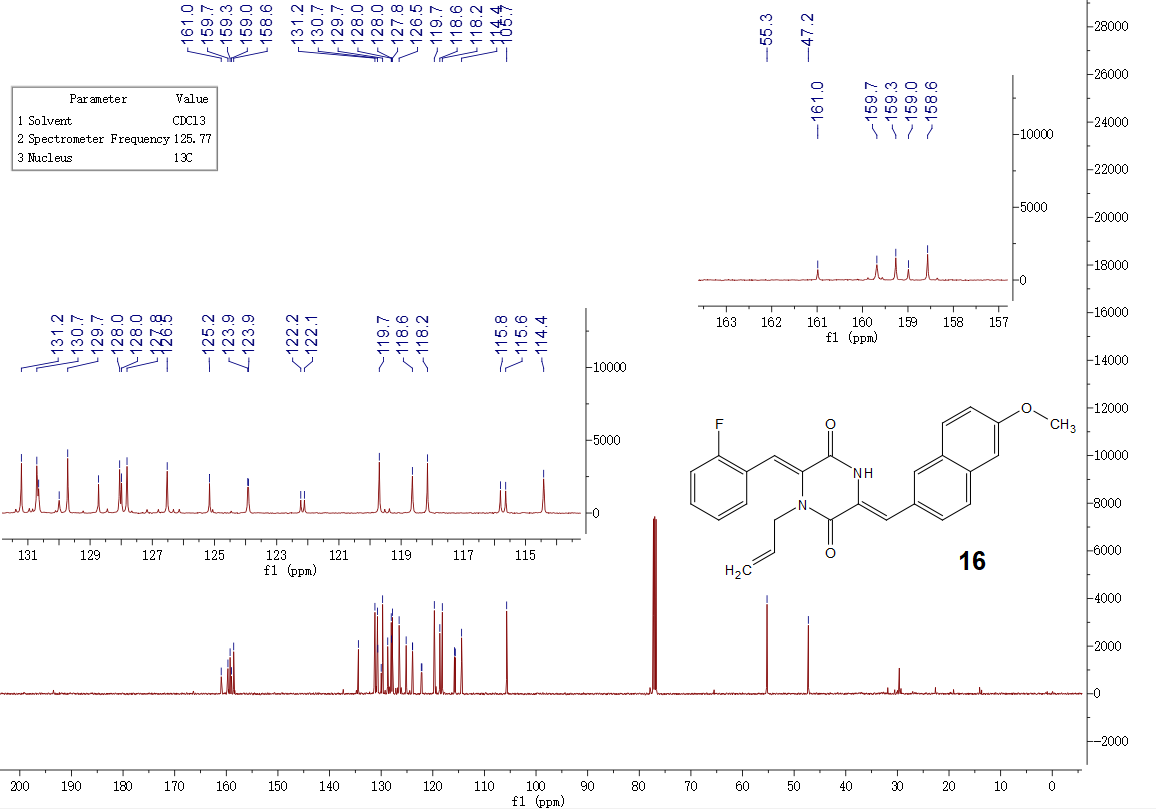


Figure **S28** 13C NMR of **16**



Ref:

1 Liao, S. R.; Du, L. J.; Qin, X. C.; Xu, L.; Wang, J. F.; Zhou, X. F.; Tu, Z. C.; Li, J.; Liu, Y. H., *Tetrahedron* **2016,** 72, (8), 1051-1057.

2 Liao, S. R.; Xu, Y.; Tang, Y.; Wang, J. F.; Zhou, X. F.; Xu, L.; Liu, Y. H., *Rsc Adv* **2015,** 5, (63), 51020-51026.