

Synthesis of Spiro-lactams and Polysubstituted Pyrroles via Ceric Ammonium Nitrate-Mediated Oxidative Cyclization of *N*-Furan-2-ylmethyl- β -Enaminones

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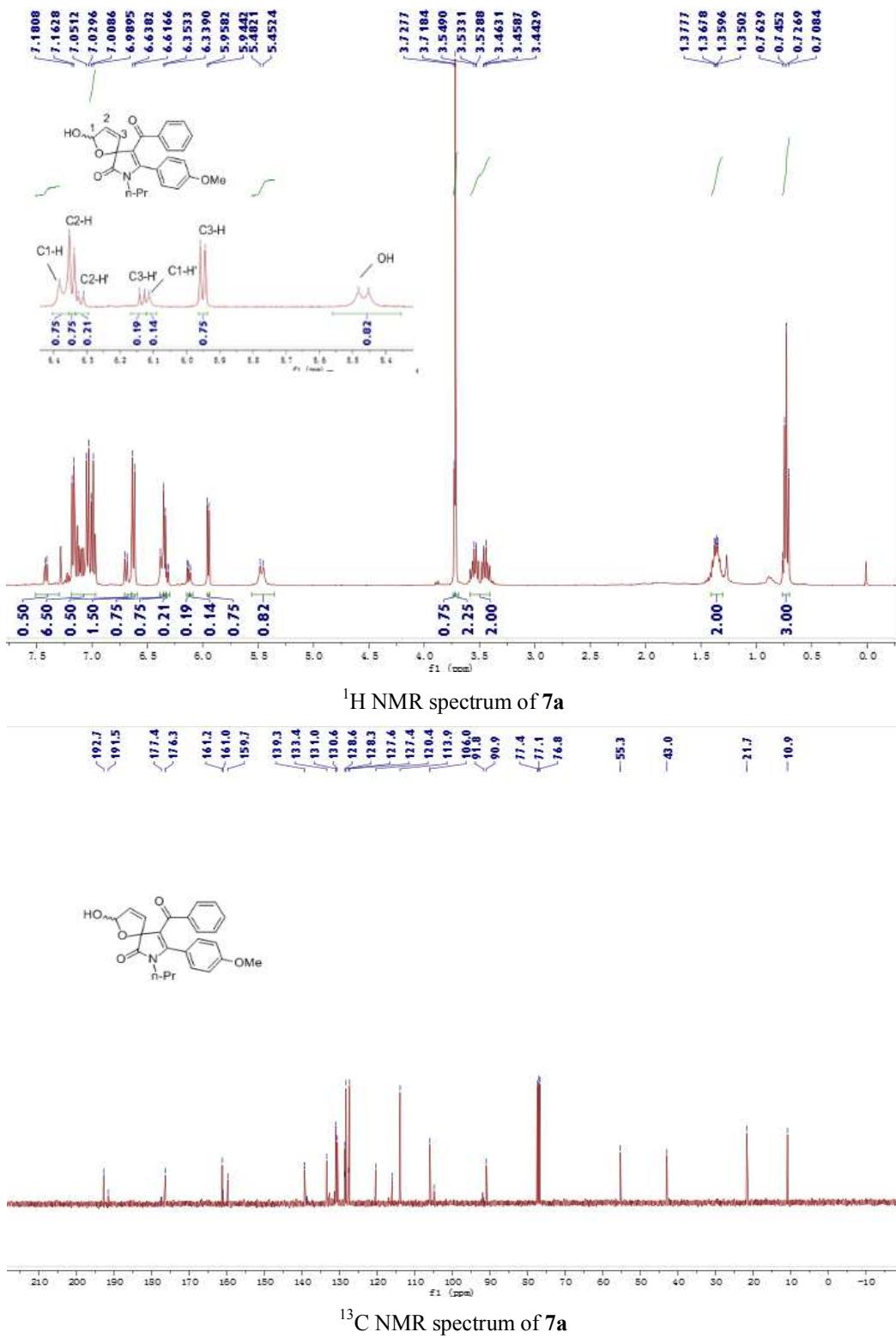
[†]School of Chemistry and Chemical Engineering, South China University of Technology, Guangzhou, P.R. China, 510640

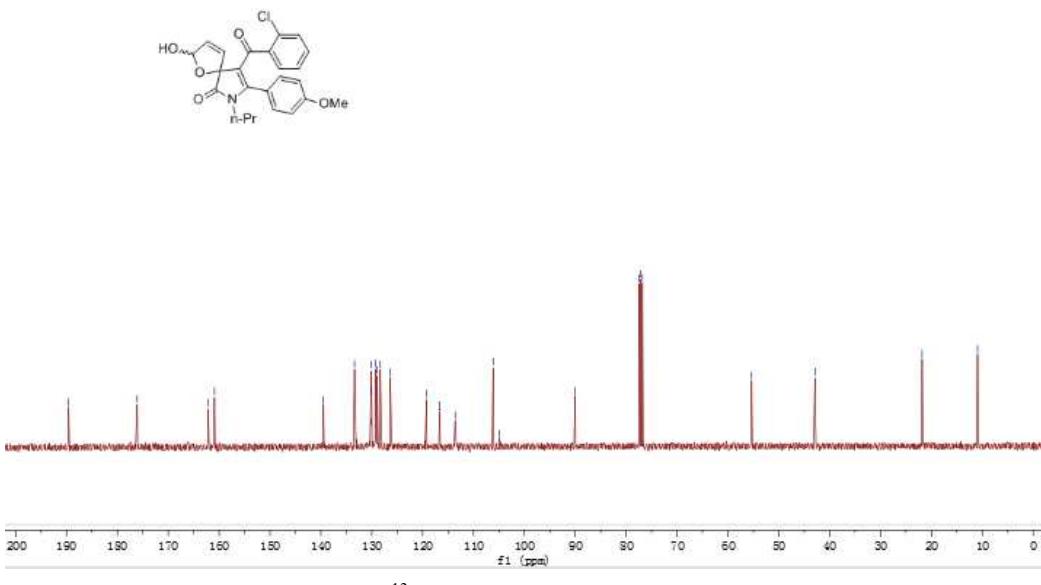
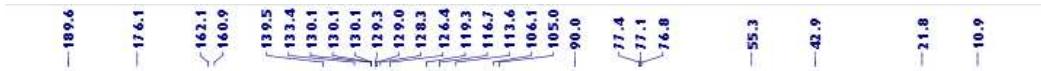
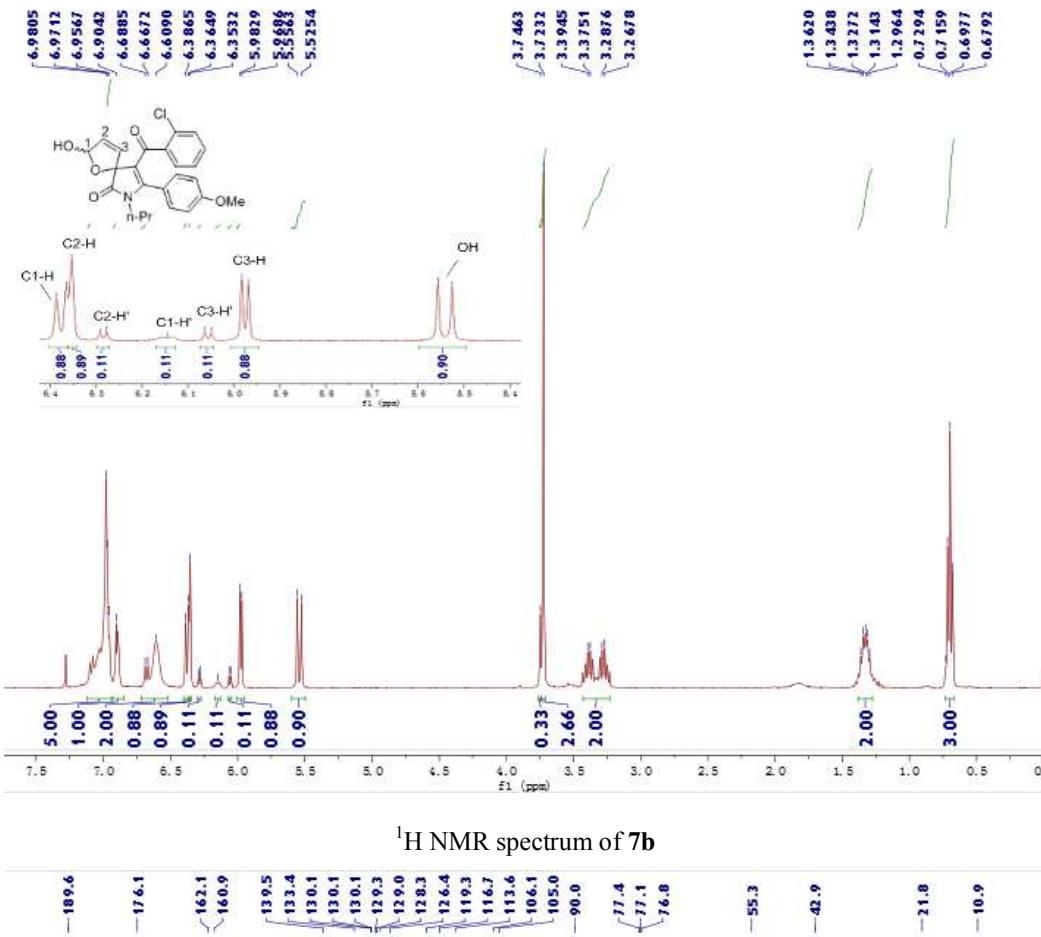
[‡]Guangdong Provincial Academy of Chinese Medical Sciences, Guangzhou, P.R. China, 510006

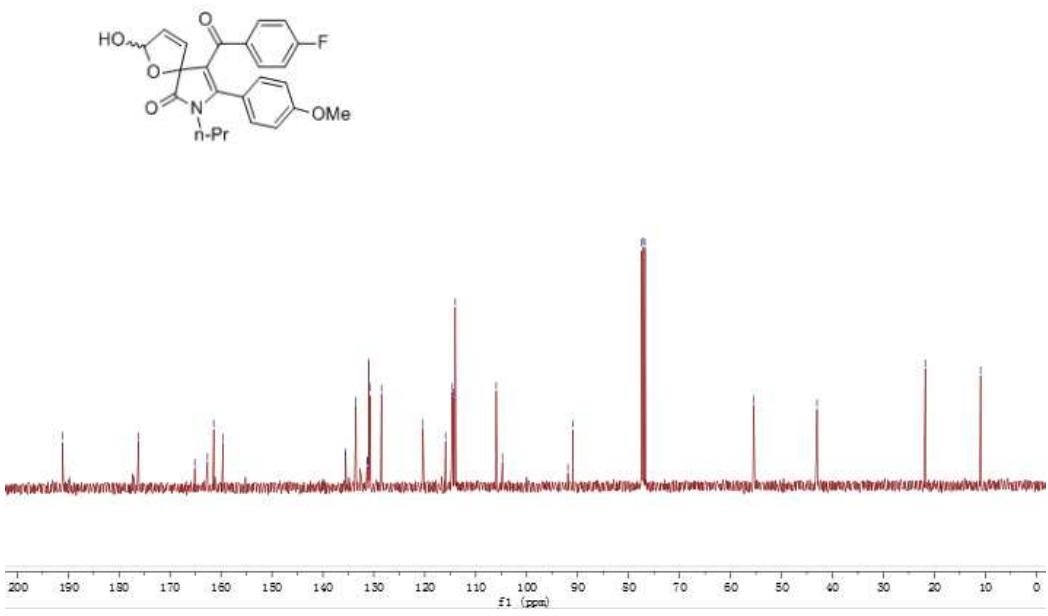
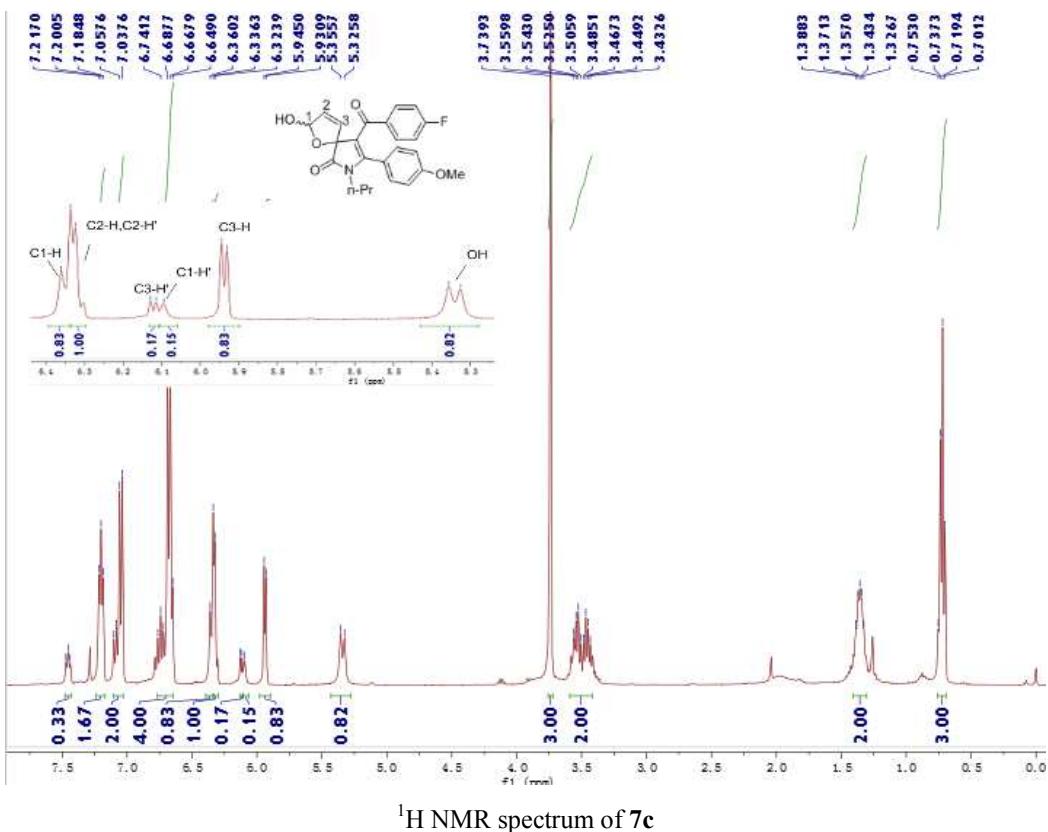
blyin@scut.edu.cn

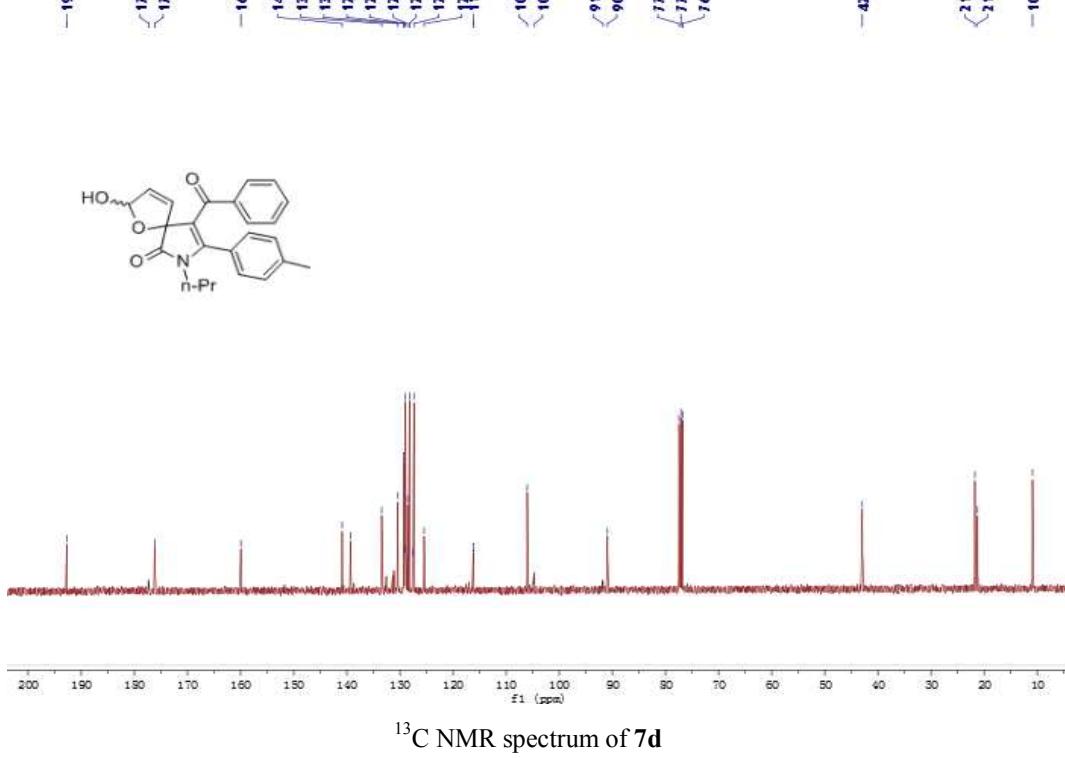
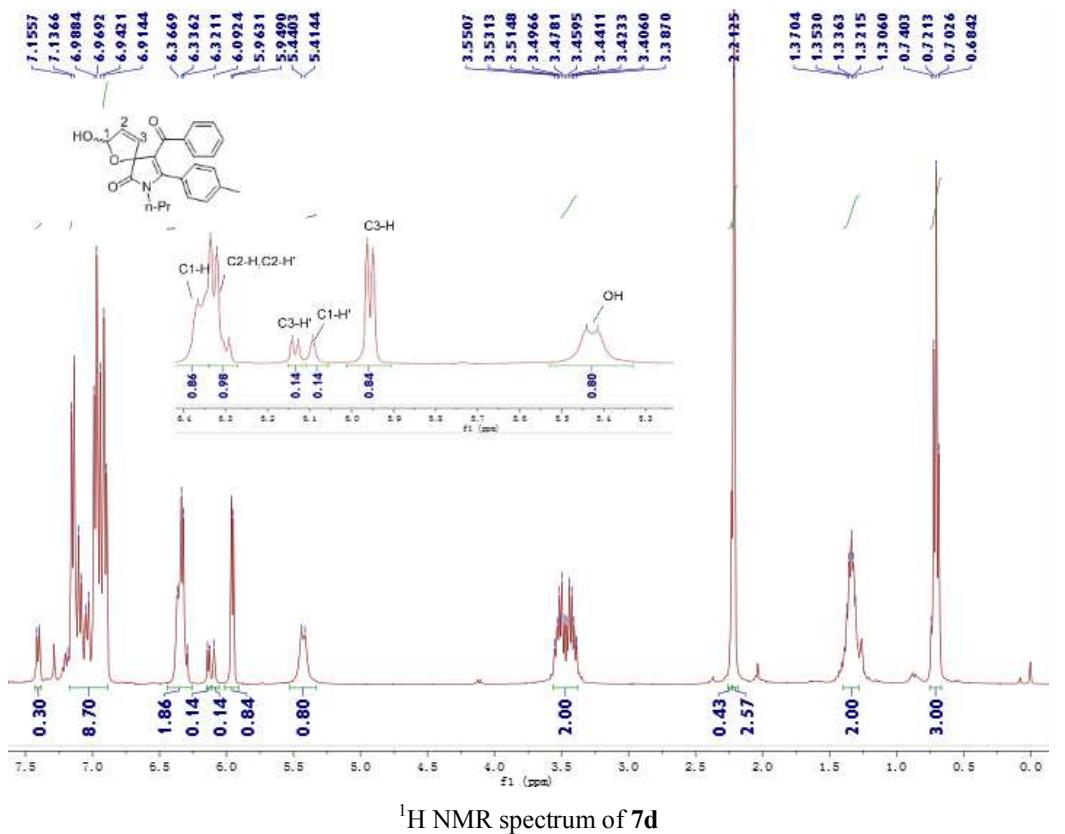
Content	Page
Spectra of 7	2-21
Spectra of 8	22-35
CIF information of 7c	36
HSQC of 7a , 7b , 7e , 7r	37-38

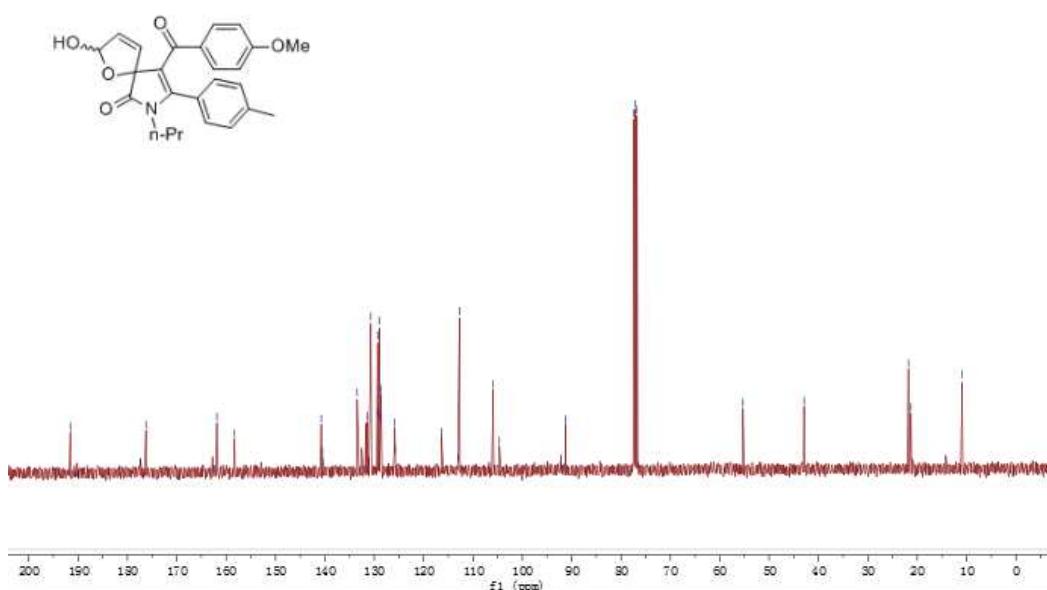
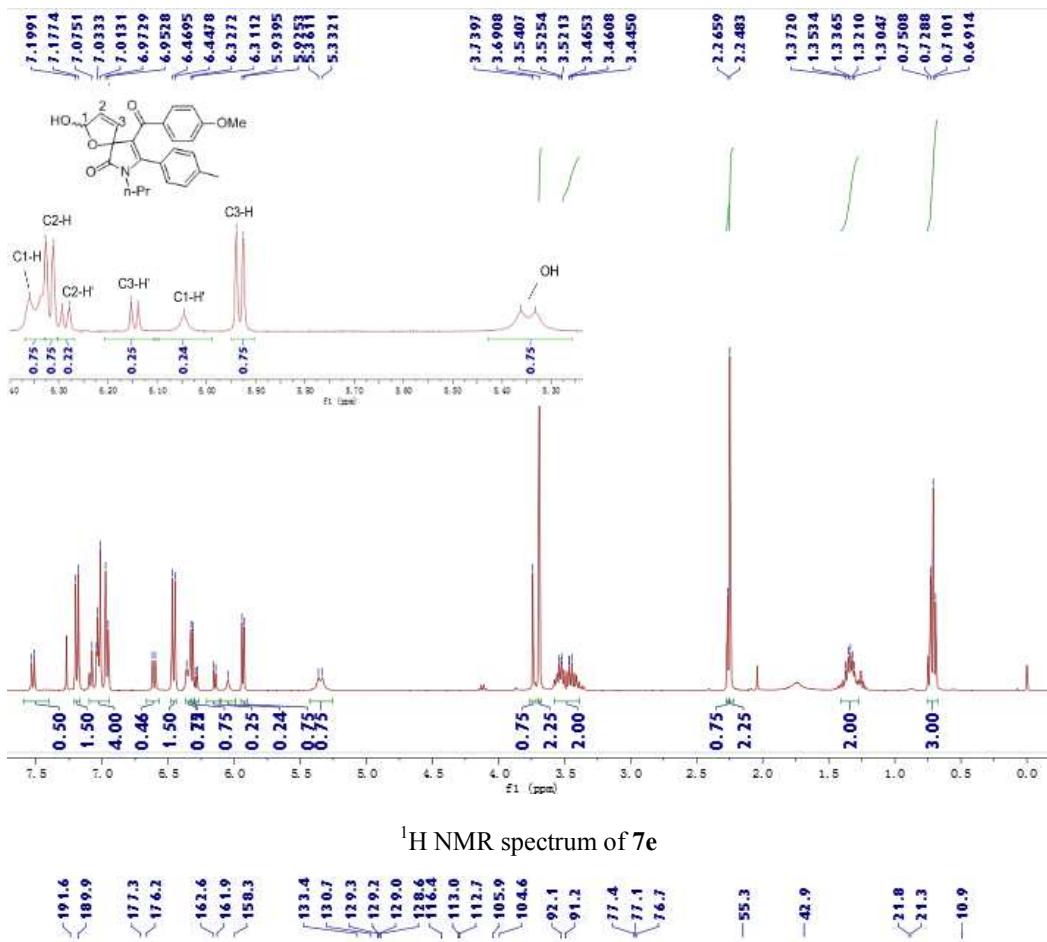
Spectra of 7

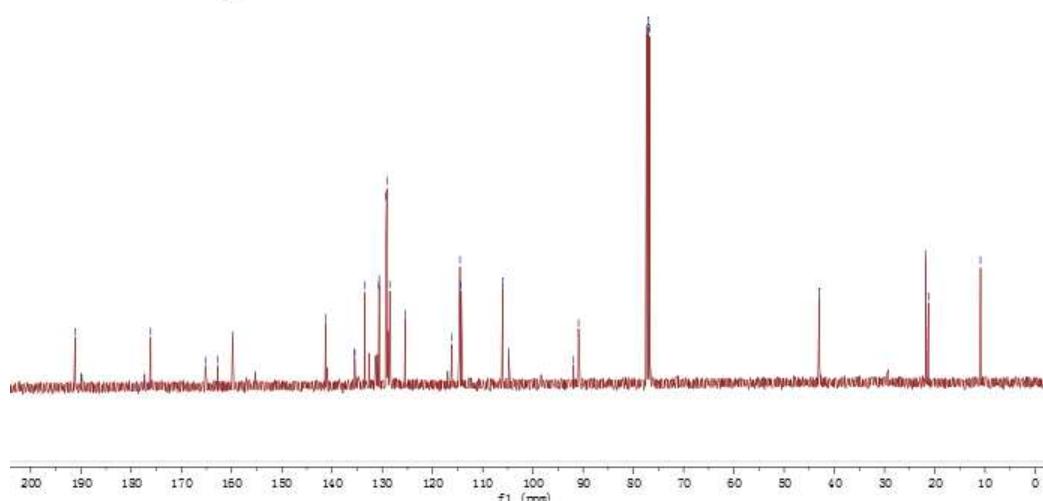
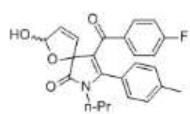
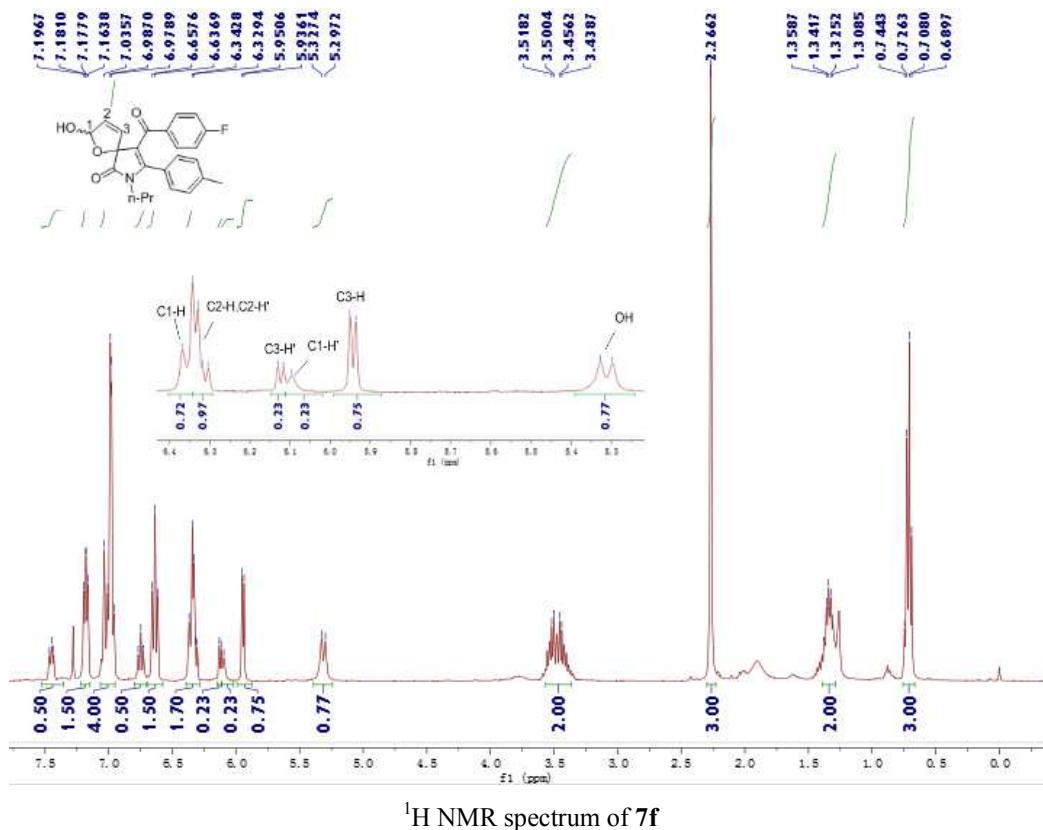




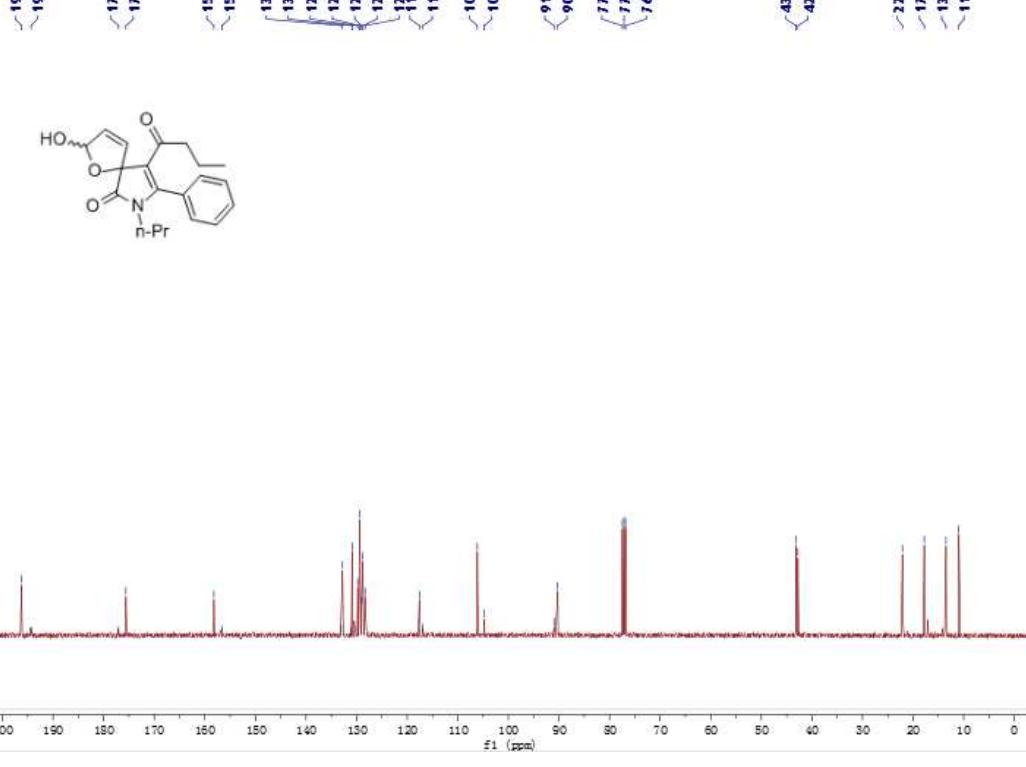
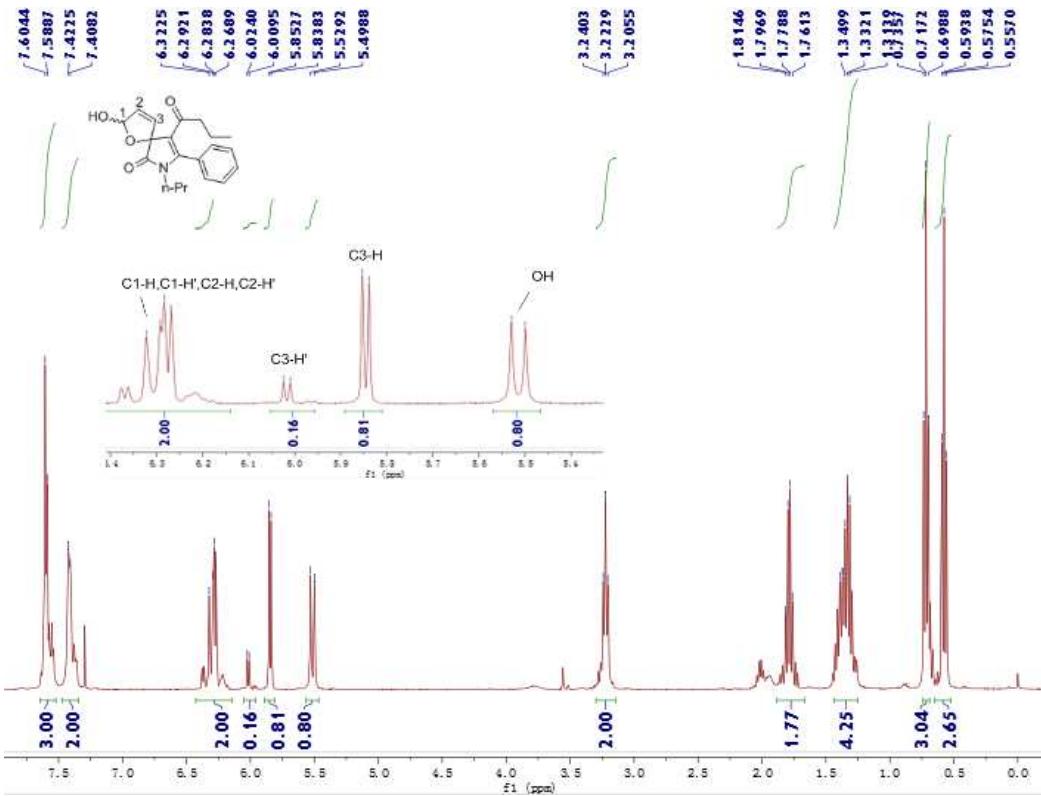


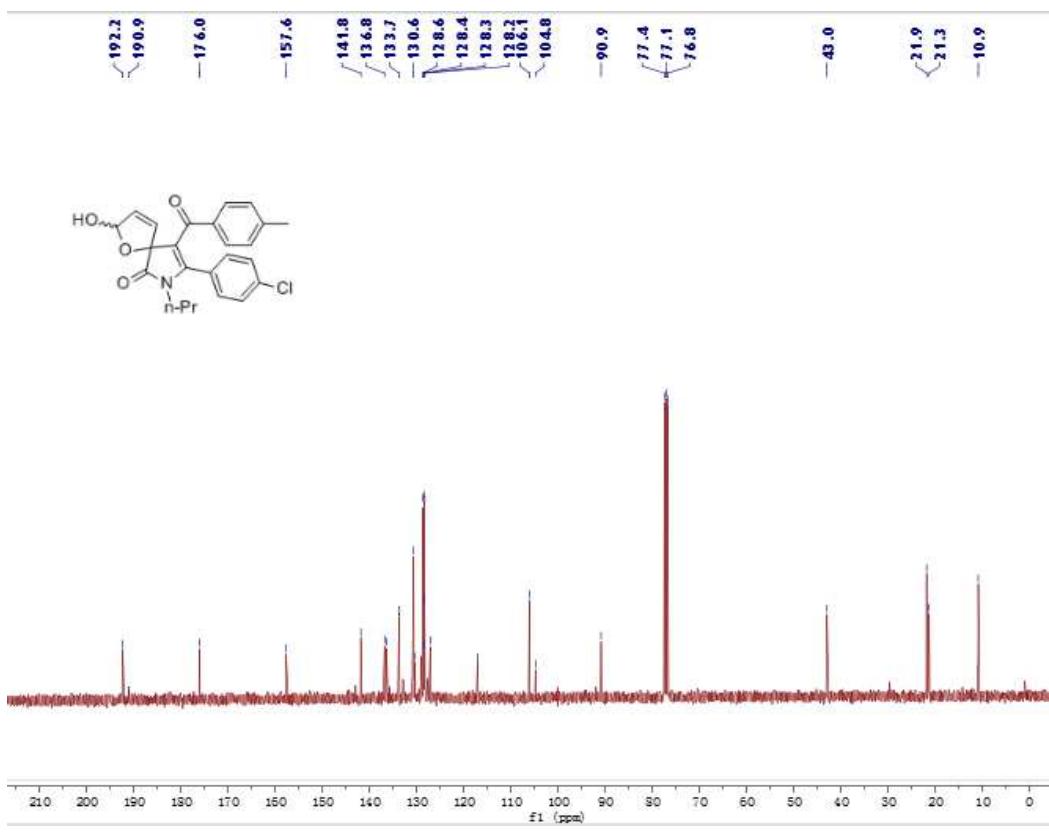
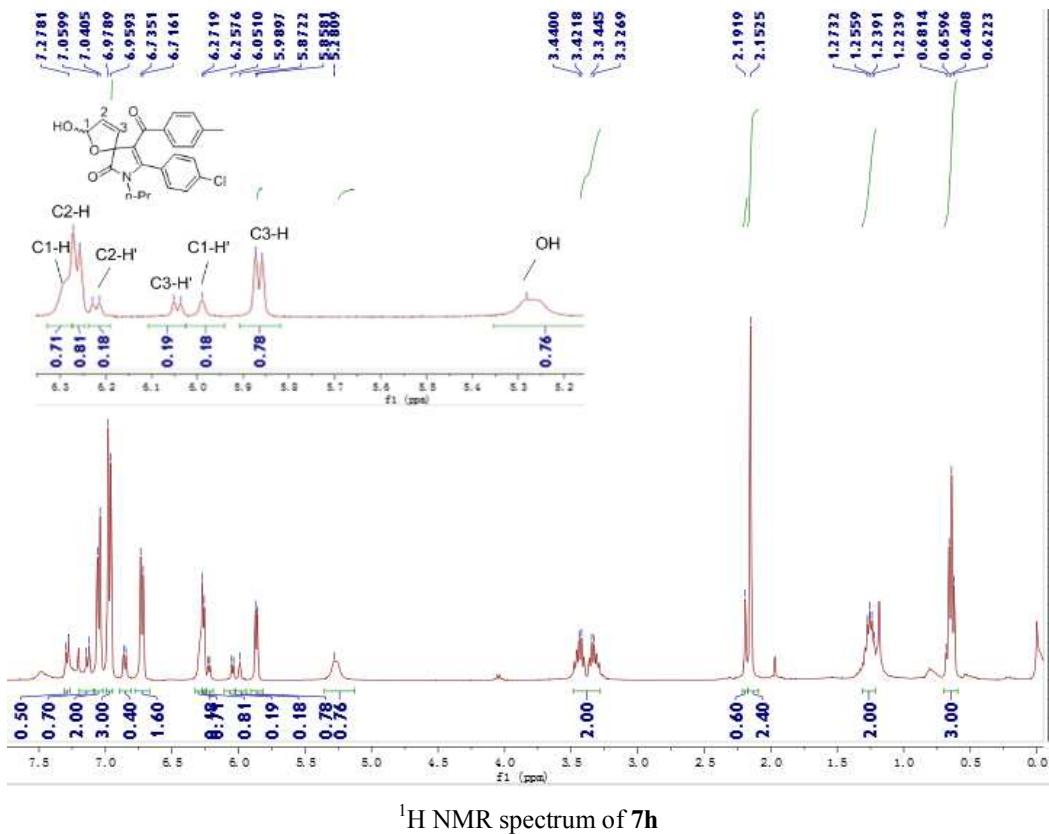


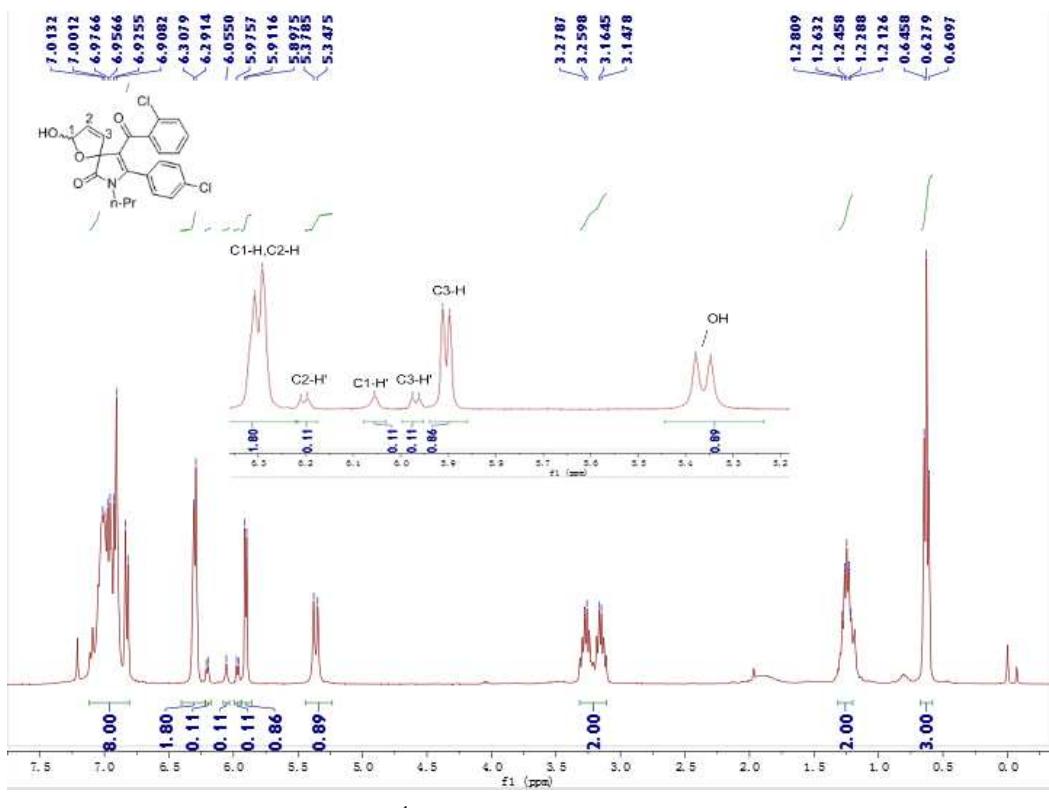




¹³C NMR spectrum of **7f**

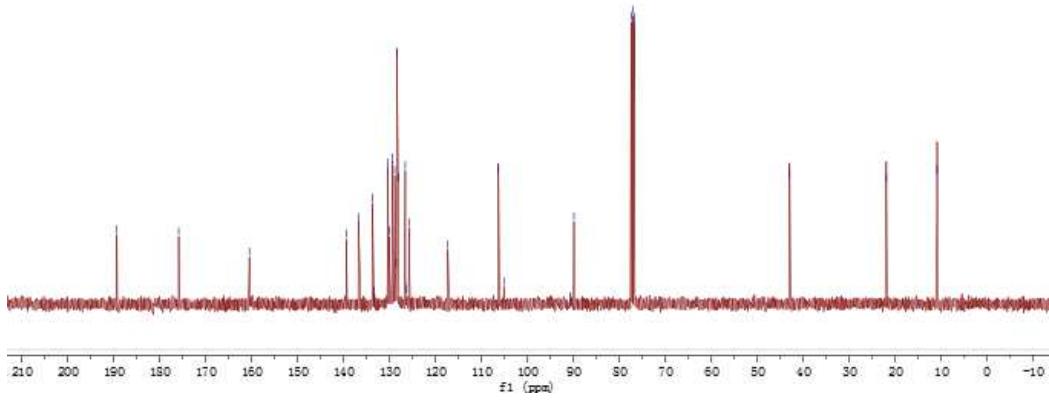
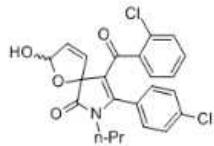




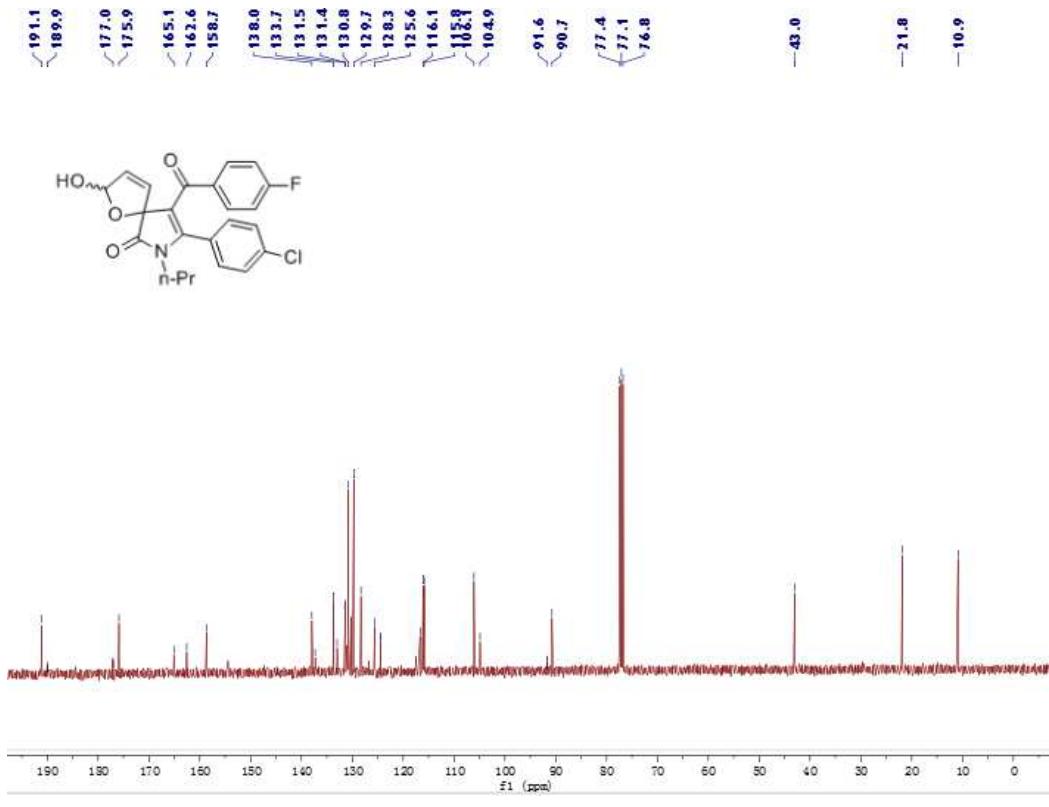
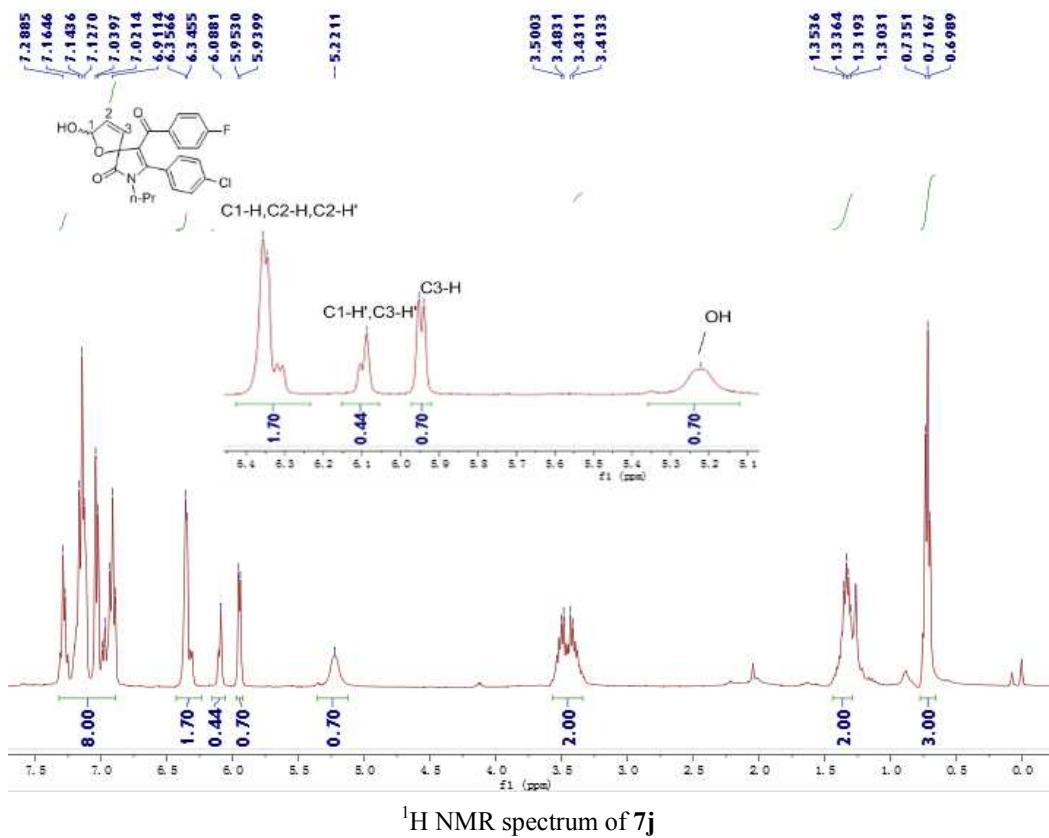


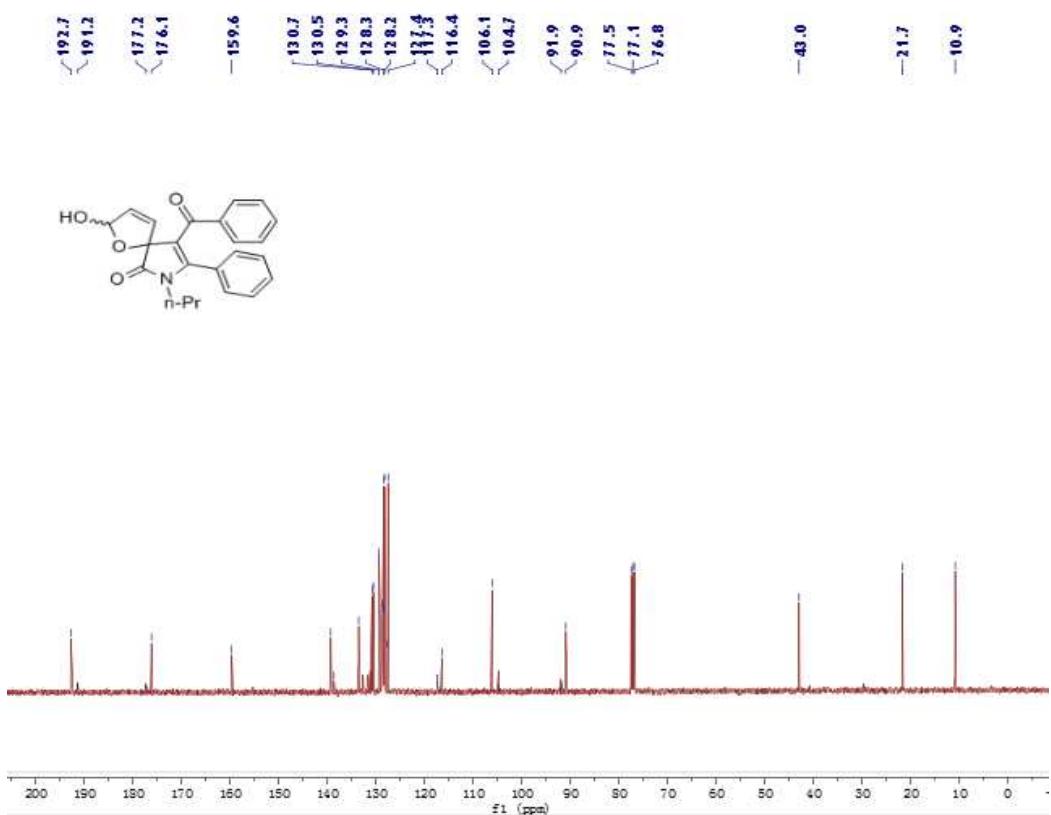
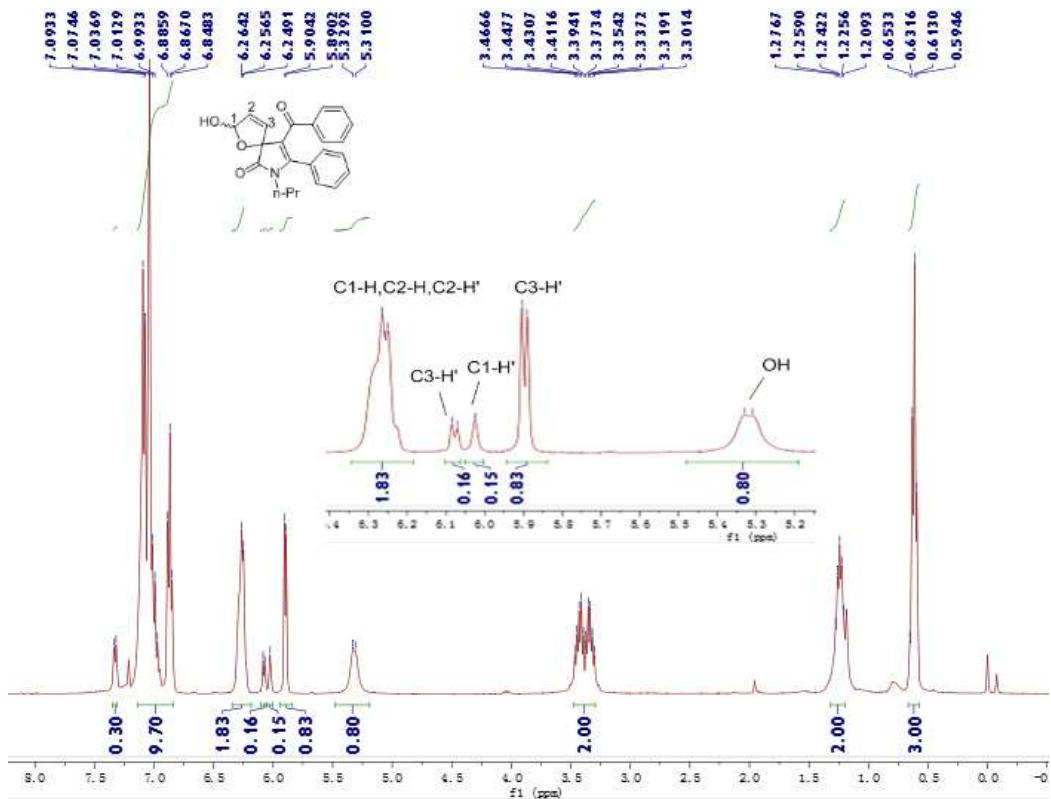
¹H NMR spectrum of 7i

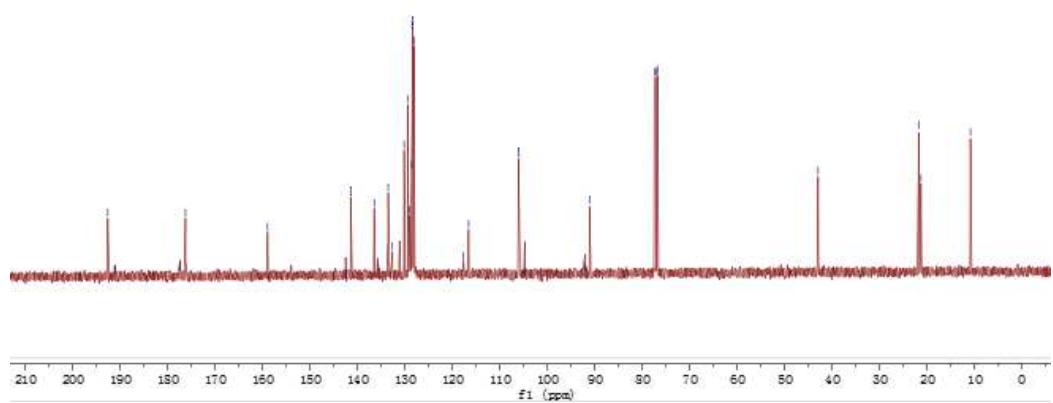
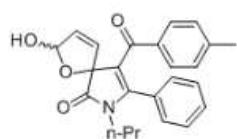
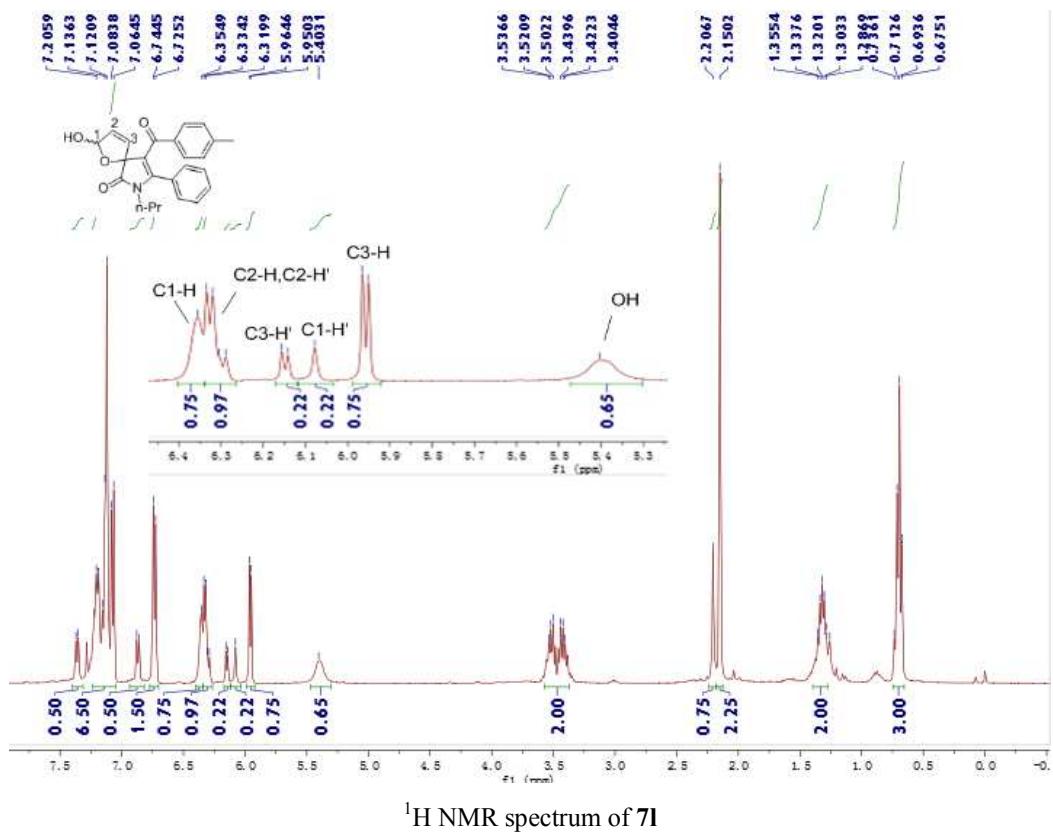
Peak labels (ppm): 8.00, 7.80, 7.58, 6.96, 6.89, 6.04, 5.96, 5.89, 5.67, 5.37, 5.33, 5.03, 4.93, 4.88, 4.84, 4.81, 4.65, 4.57, 4.53, 4.49, 4.45, 4.41, 4.37, 4.33, 4.29, 4.25, 4.21, 4.17, 4.13, 4.09, 4.05, 4.01, 3.97, 3.93, 3.89, 3.85, 3.81, 3.77, 3.73, 3.69, 3.65, 3.61, 3.57, 3.53, 3.49, 3.45, 3.41, 3.37, 3.33, 3.29, 3.25, 3.21, 3.17, 3.13, 3.09, 3.05, 3.01, 2.97, 2.93, 2.89, 2.85, 2.81, 2.77, 2.73, 2.69, 2.65, 2.61, 2.57, 2.53, 2.49, 2.45, 2.41, 2.37, 2.33, 2.29, 2.25, 2.21, 2.17, 2.13, 2.09, 2.05, 2.01, 1.97, 1.93, 1.89, 1.85, 1.81, 1.77, 1.73, 1.69, 1.65, 1.61, 1.57, 1.53, 1.49, 1.45, 1.41, 1.37, 1.33, 1.29, 1.25, 1.21, 1.17, 1.13, 1.09, 1.05, 1.01, 0.97, 0.93, 0.89, 0.85, 0.81, 0.77, 0.73, 0.69, 0.65, 0.61, 0.57, 0.53, 0.49, 0.45, 0.41, 0.37, 0.33, 0.29, 0.25, 0.21, 0.17, 0.13, 0.09, 0.05, 0.01, -0.05.



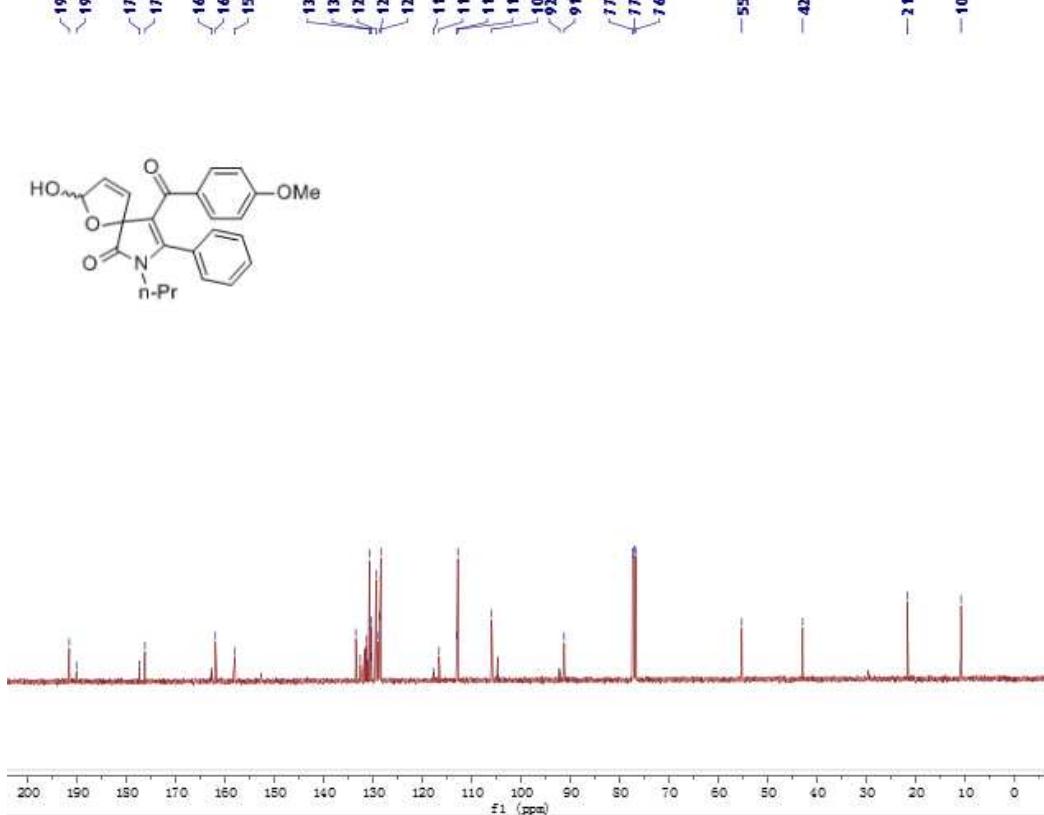
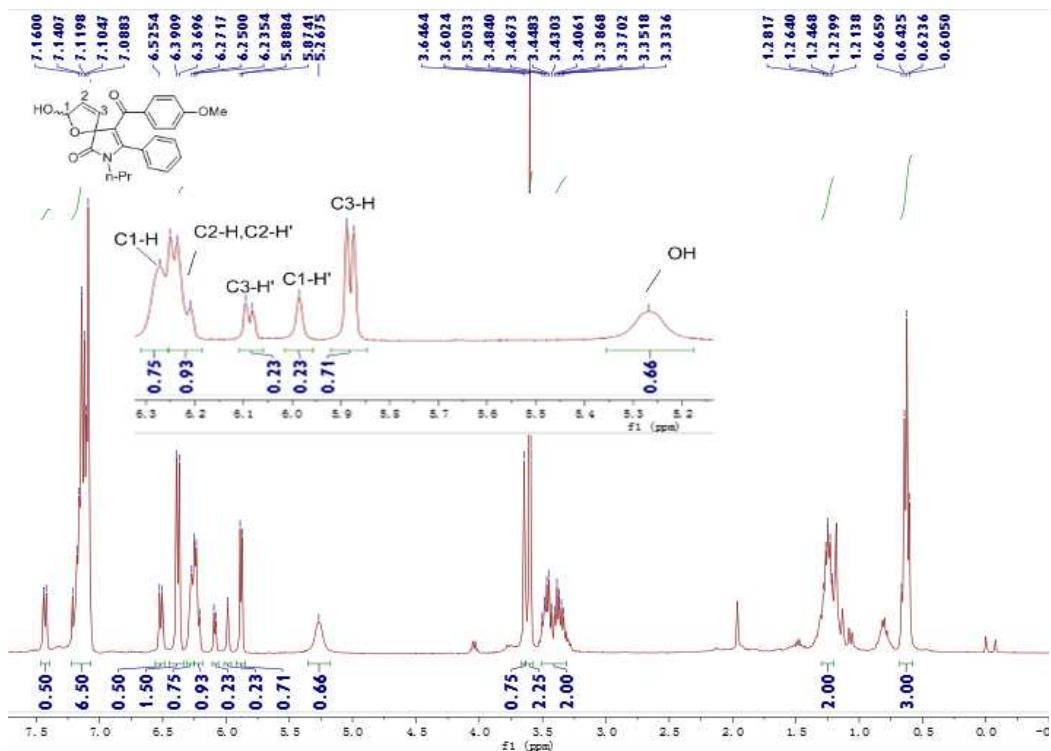
¹³C NMR spectrum of 7i

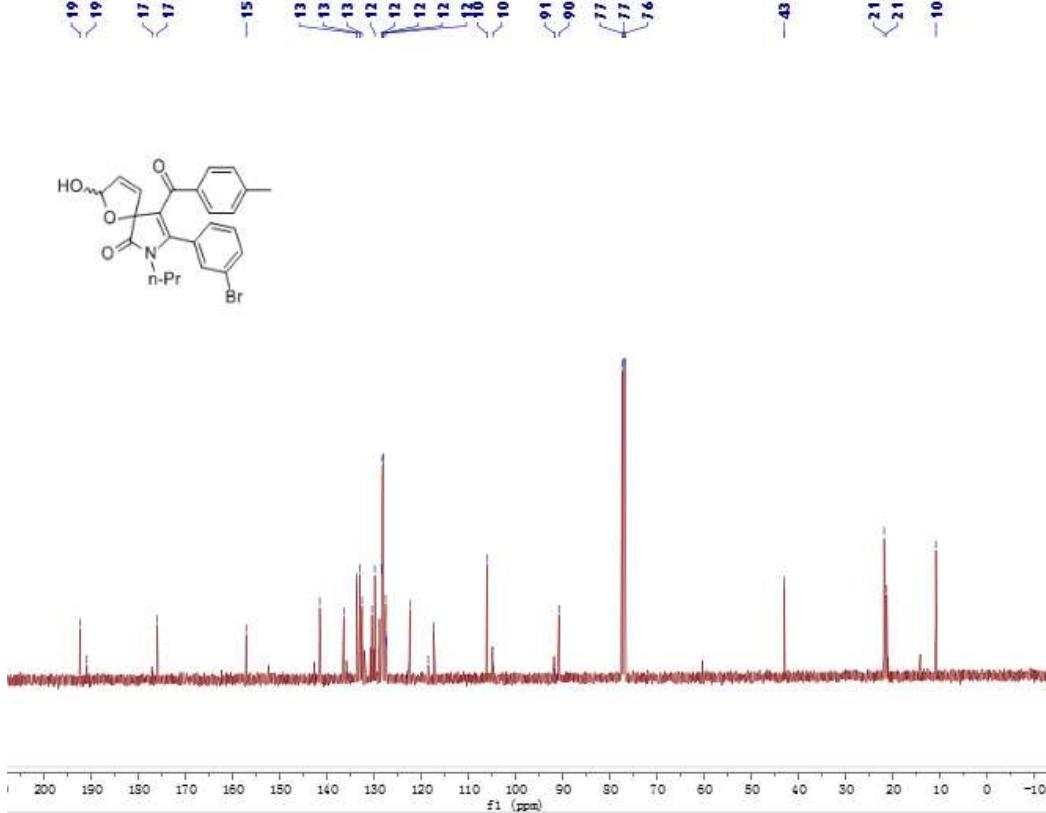
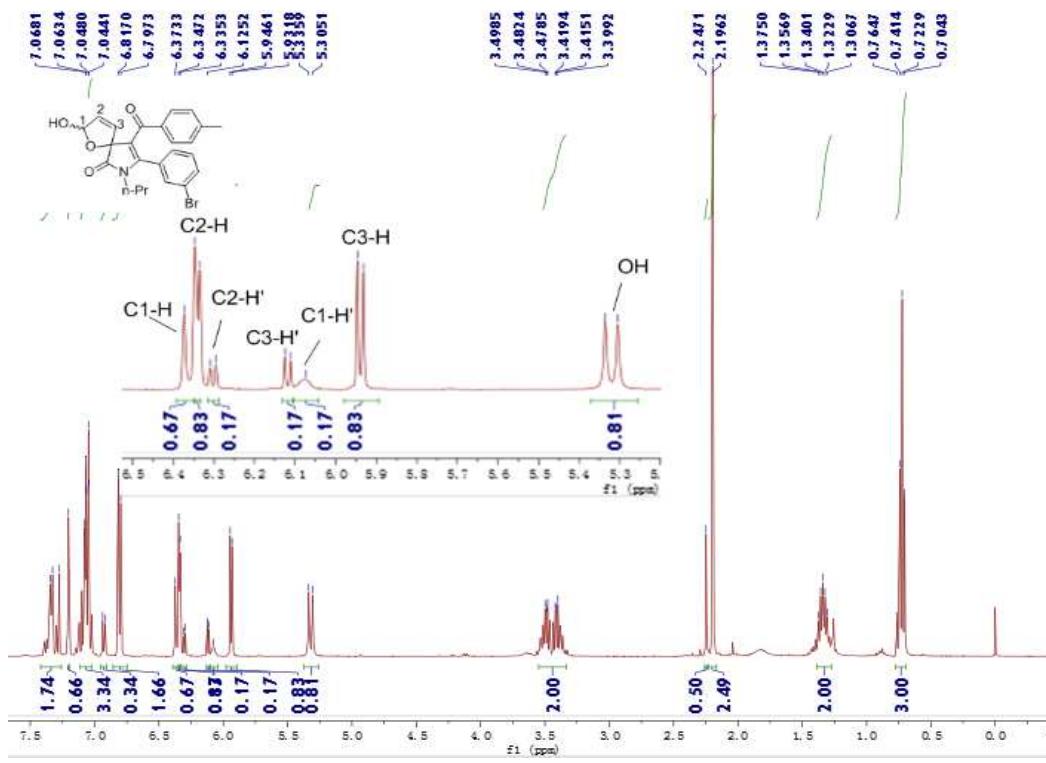


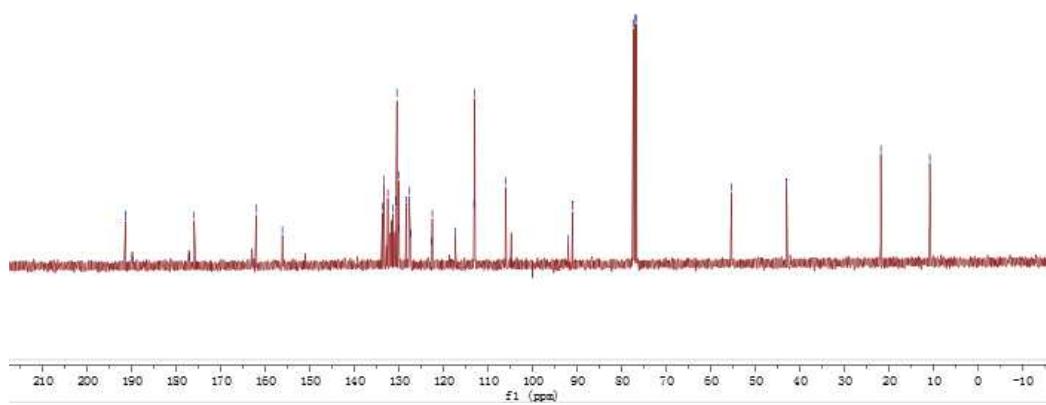
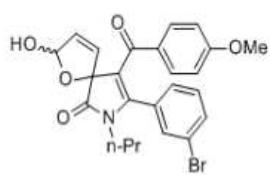
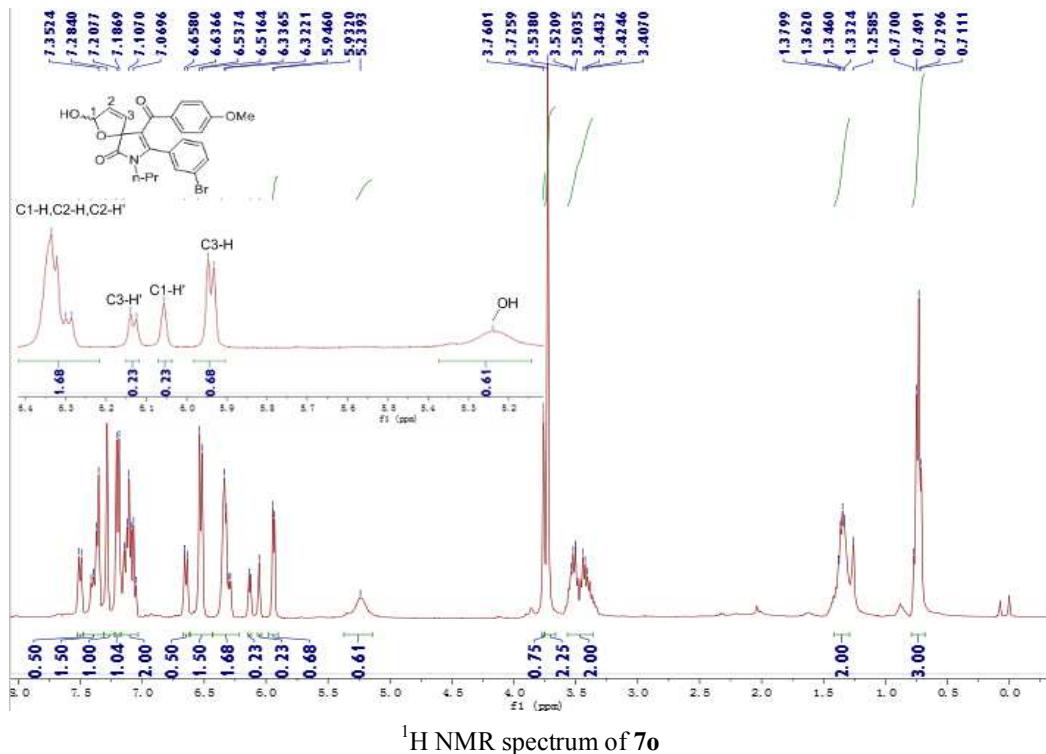




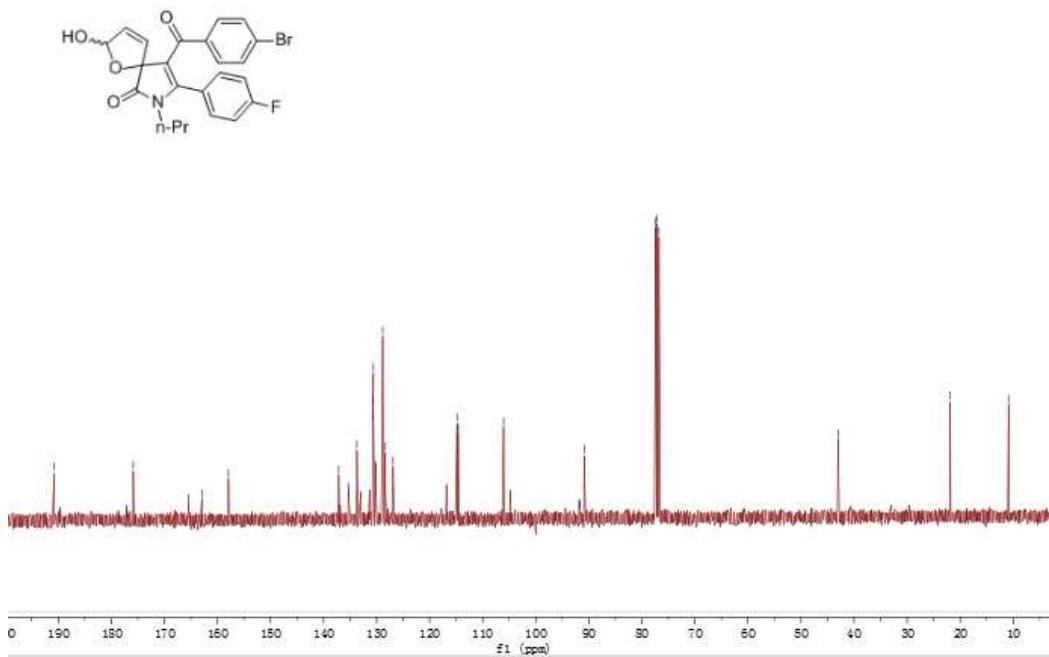
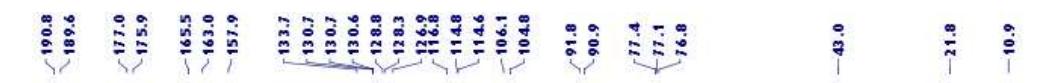
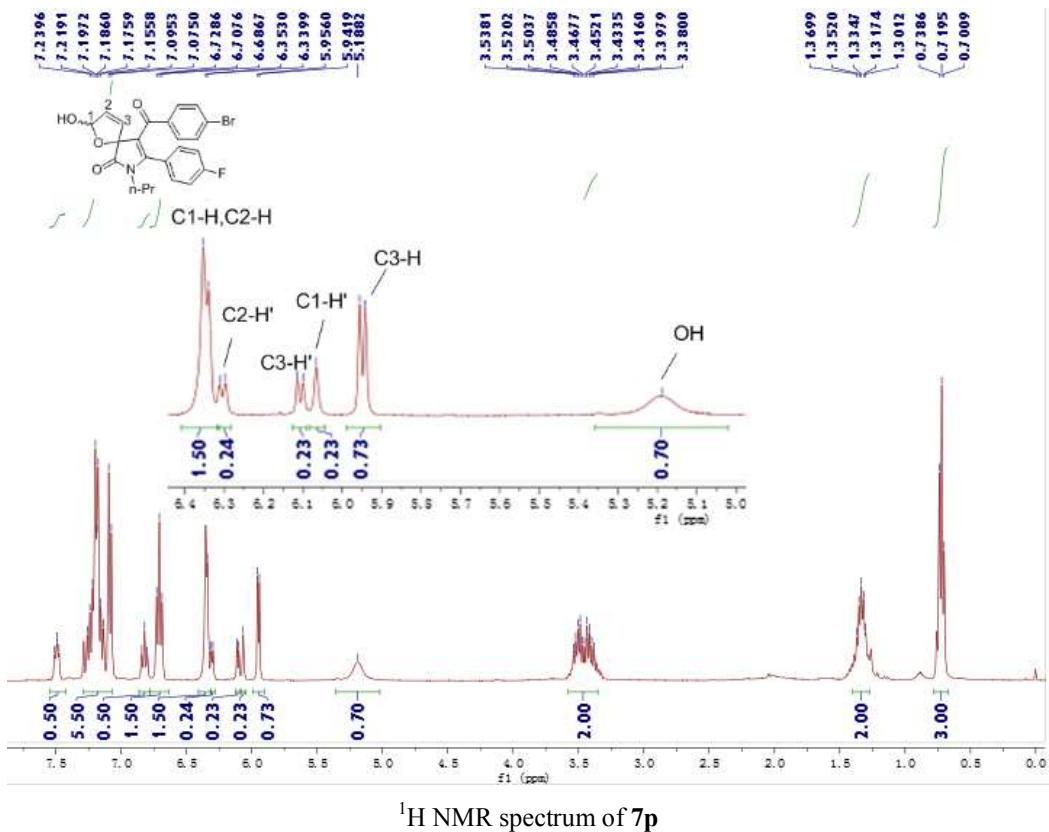
¹³C NMR spectrum of **7I**

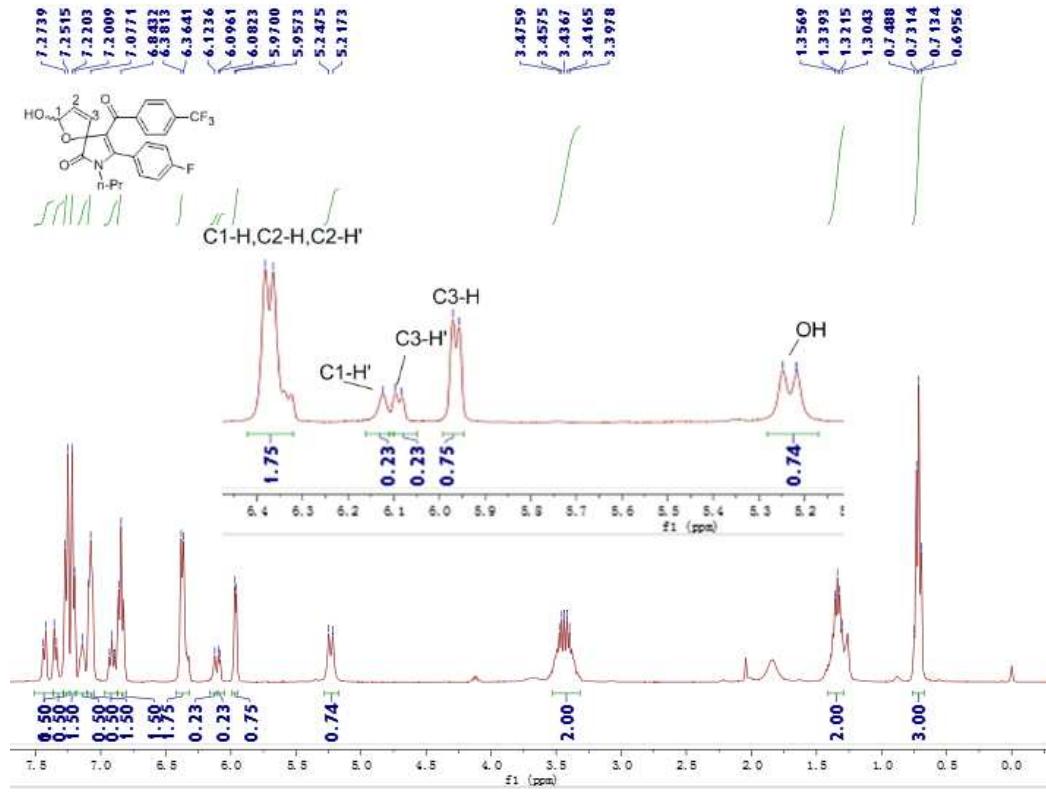




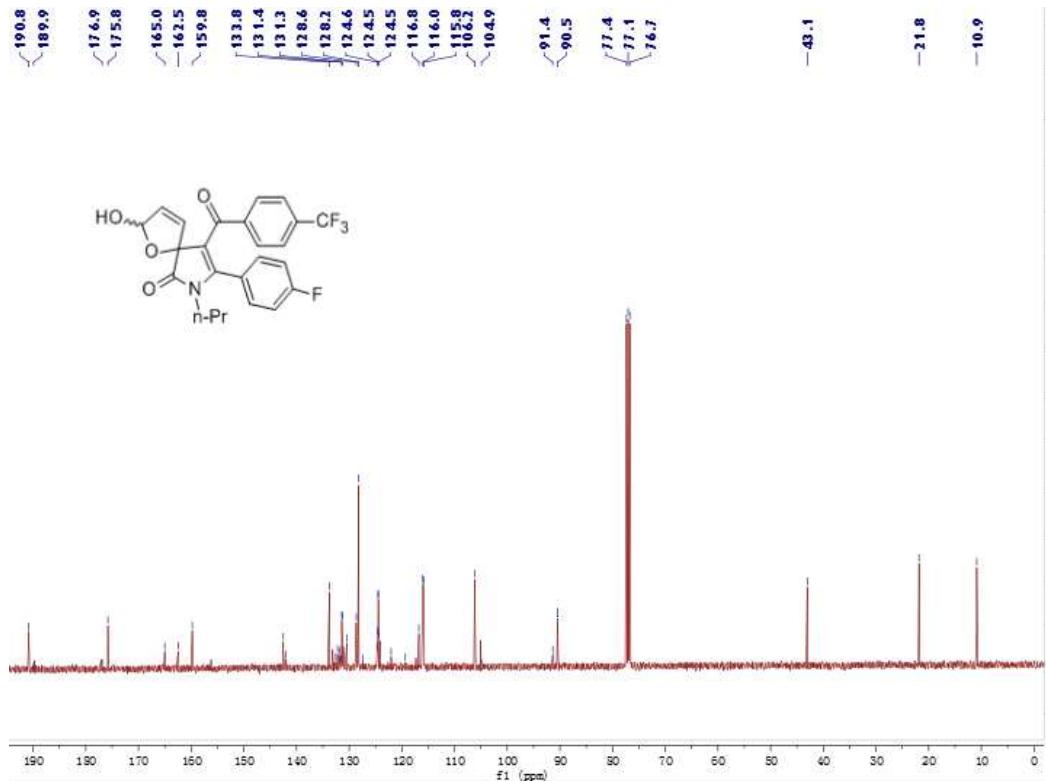


¹³C NMR spectrum of **7o**

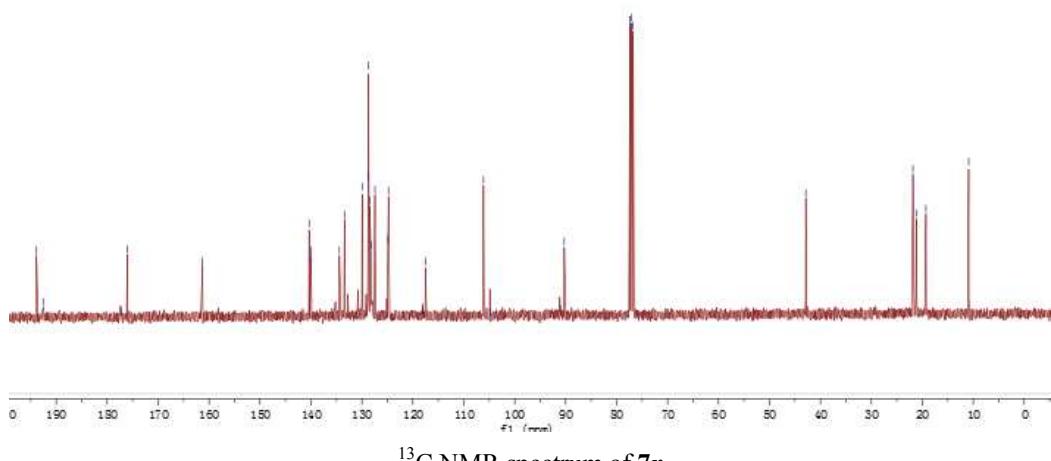
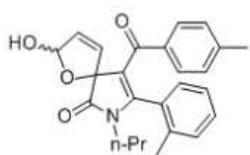
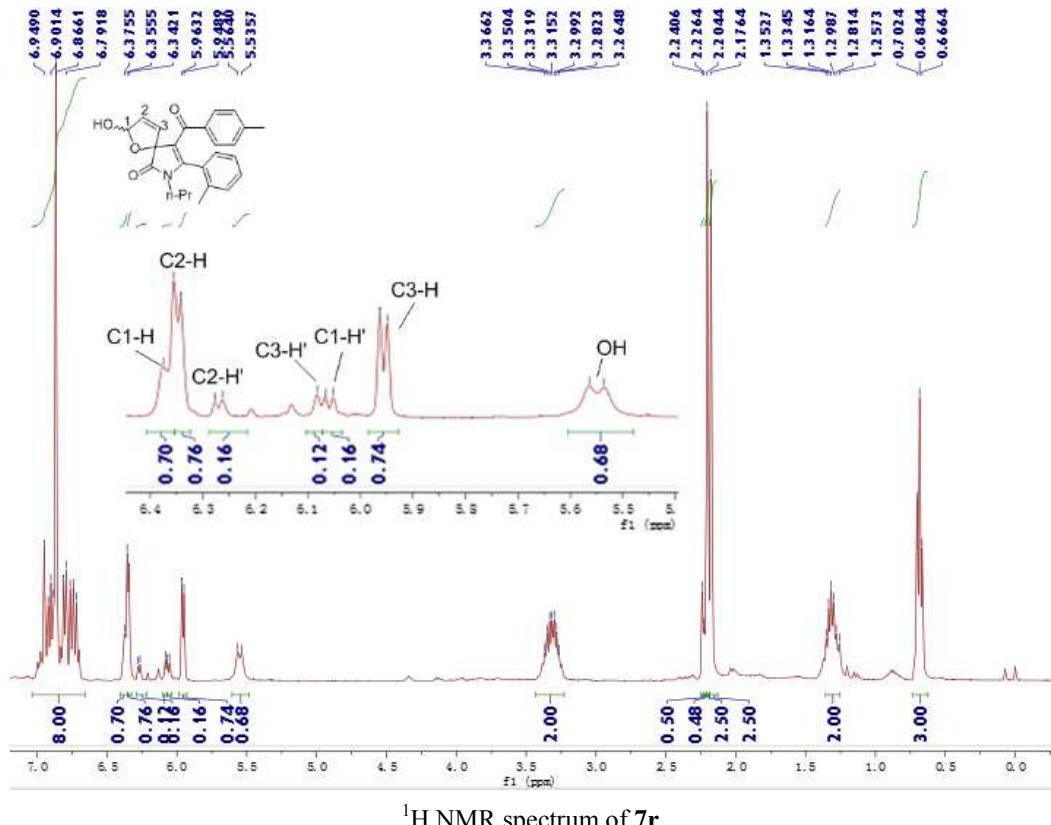


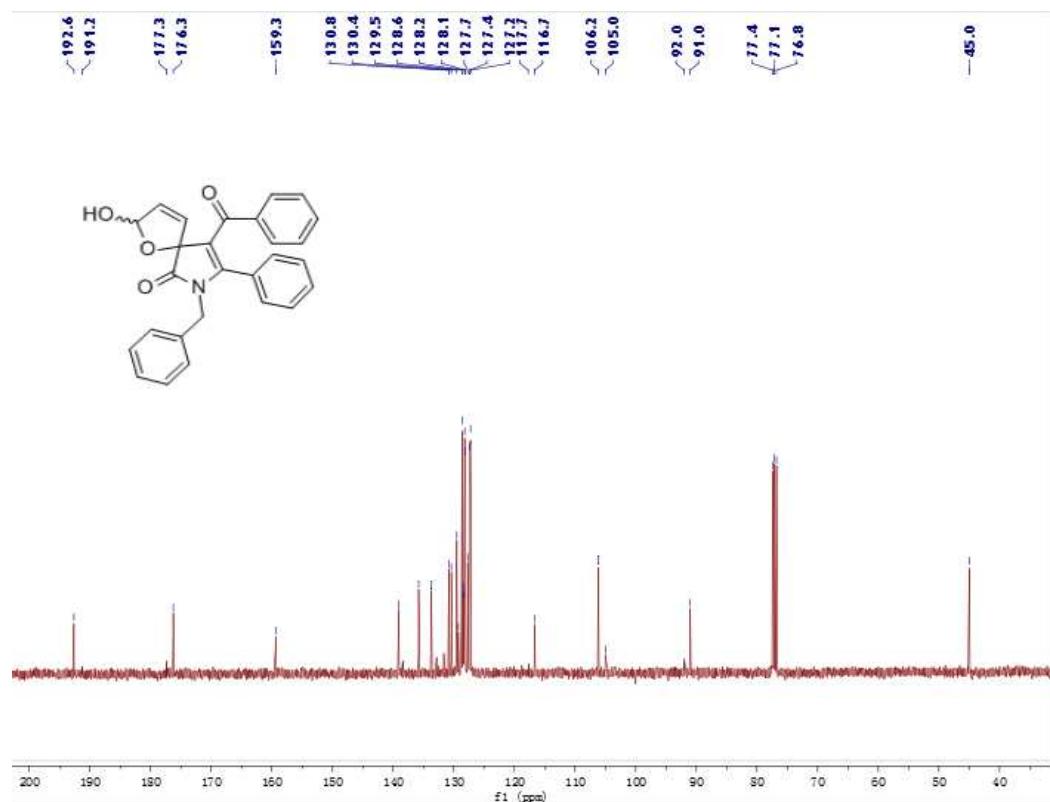
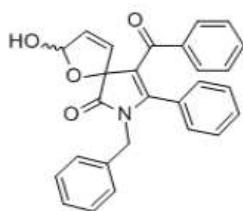
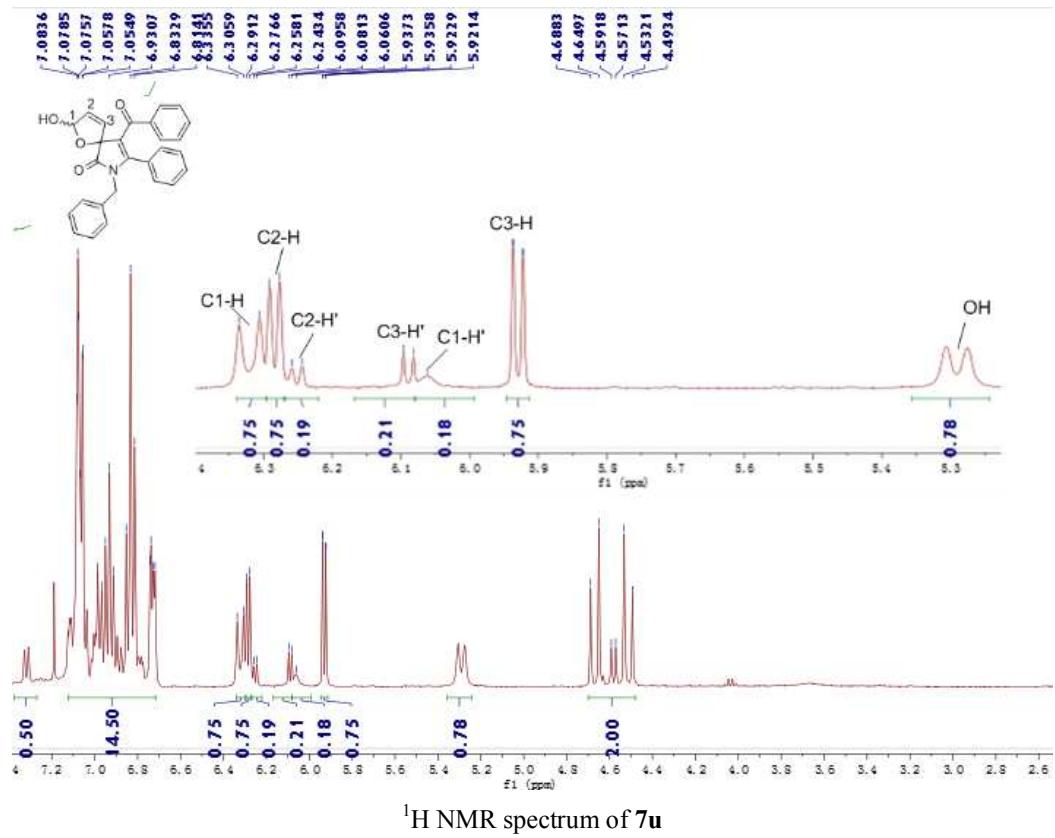


¹H NMR spectrum of 7q



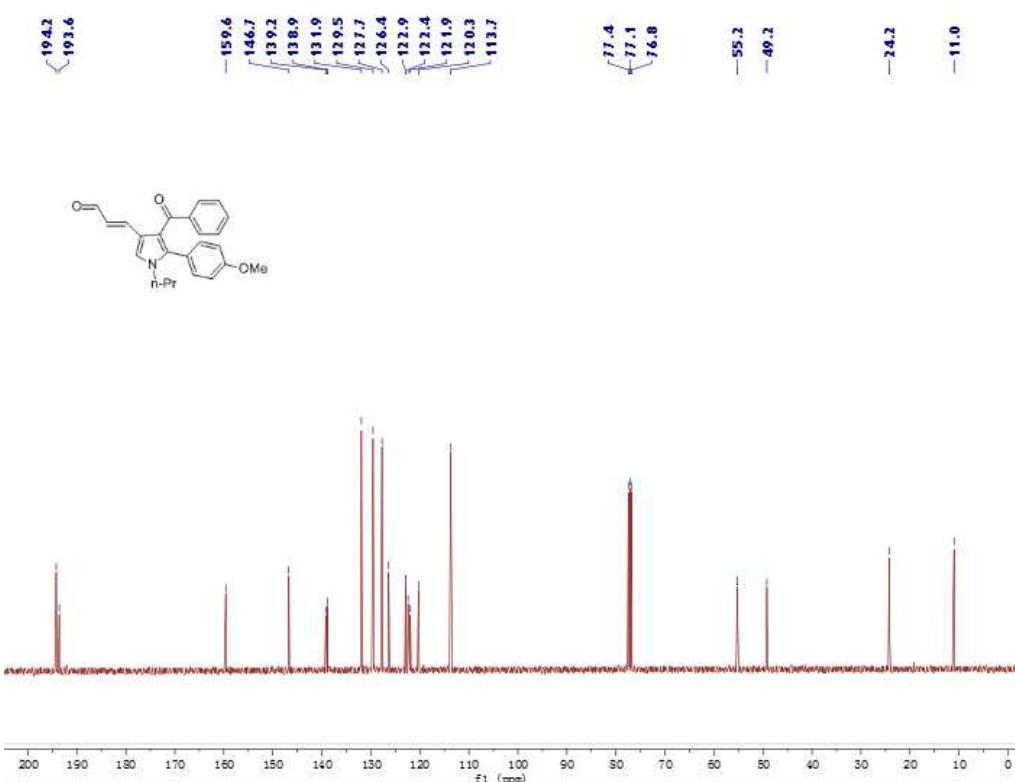
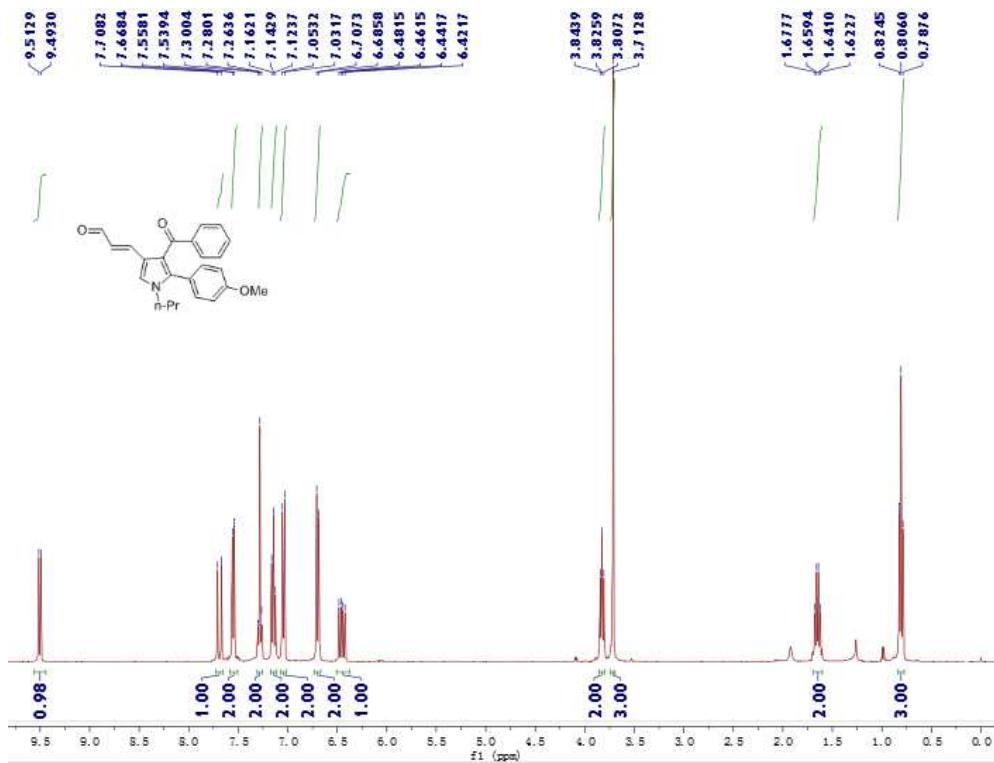
¹³C NMR spectrum of 7q

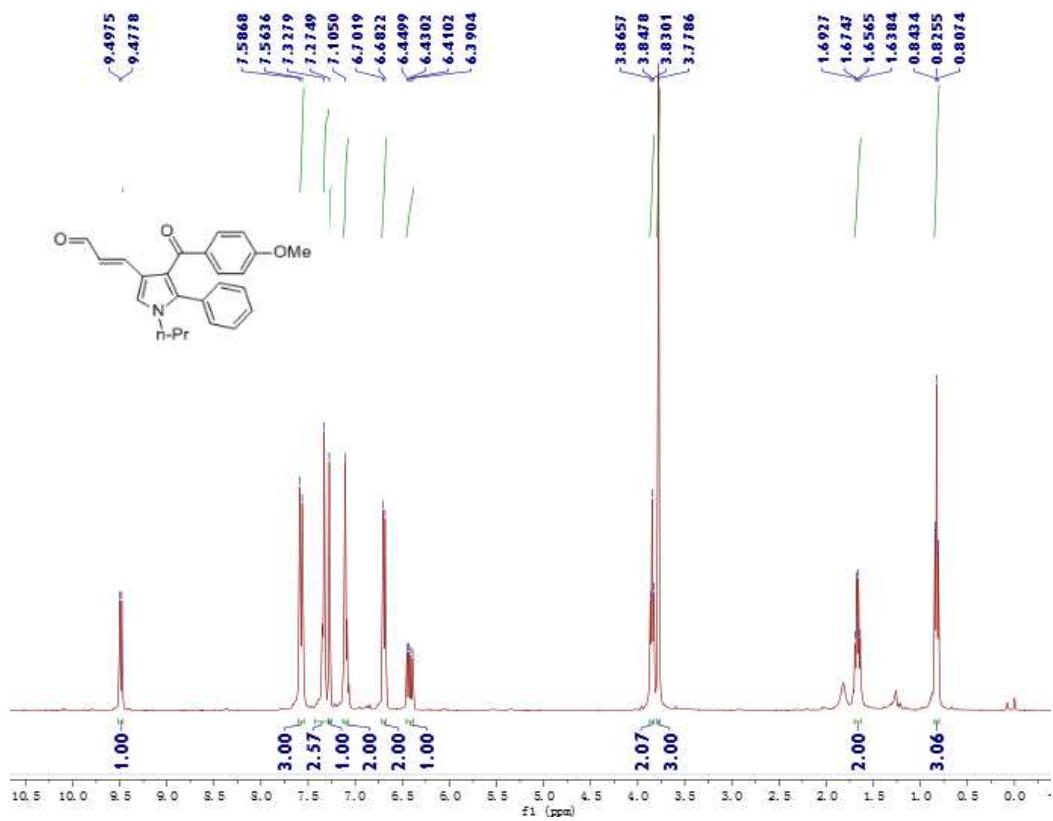




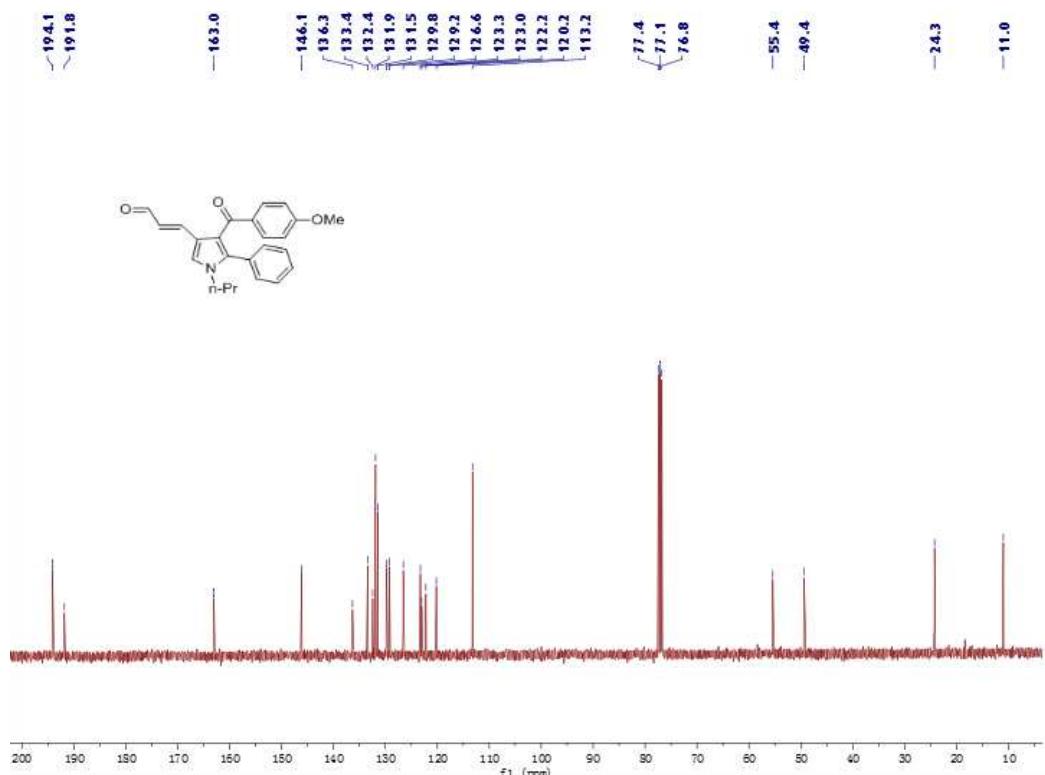
¹³C NMR spectrum of **7u**

Spectra of **8**

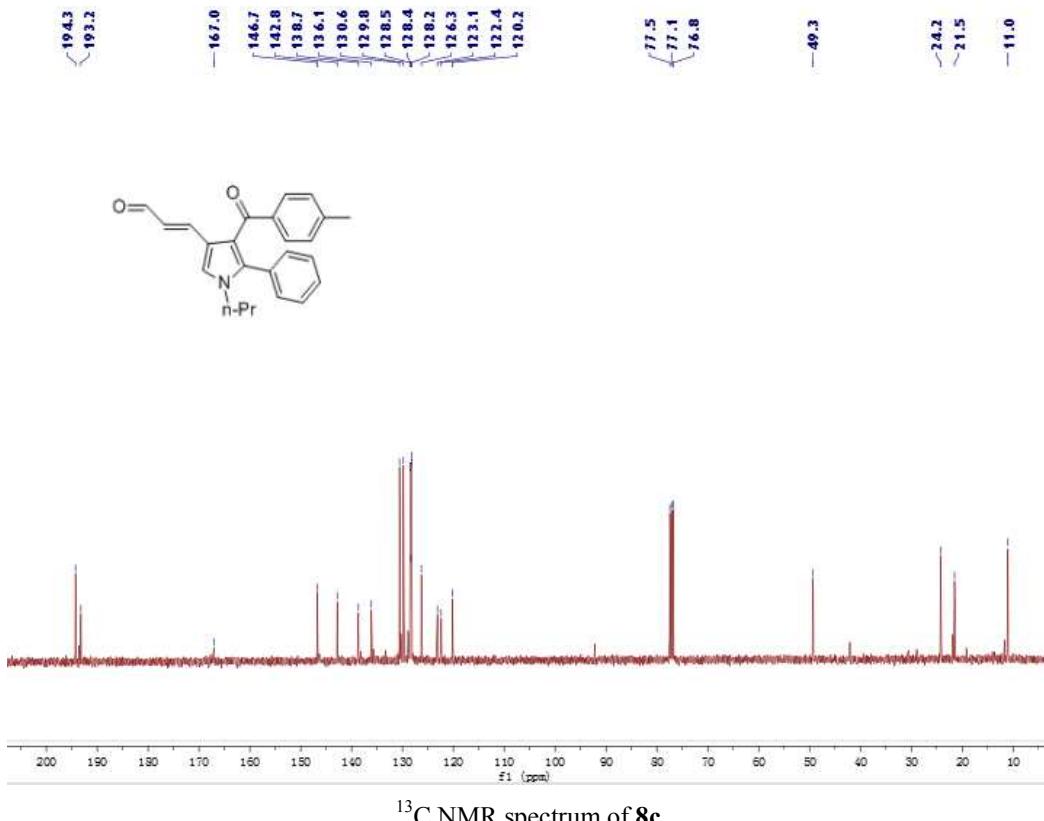
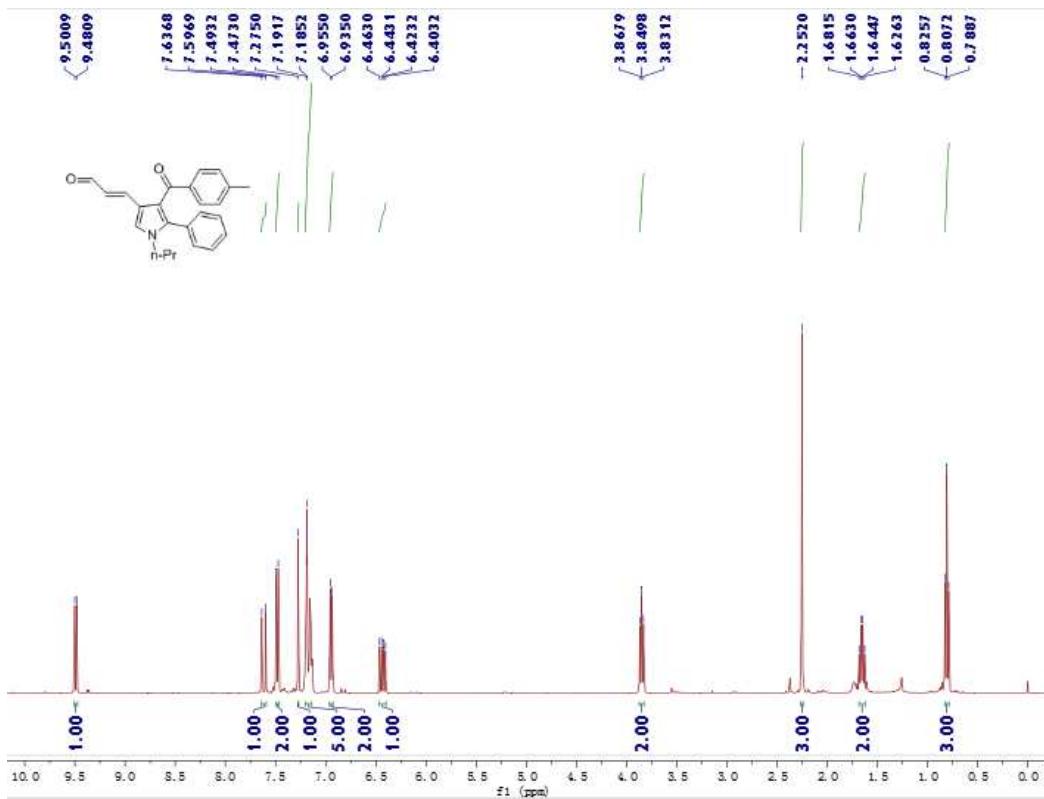


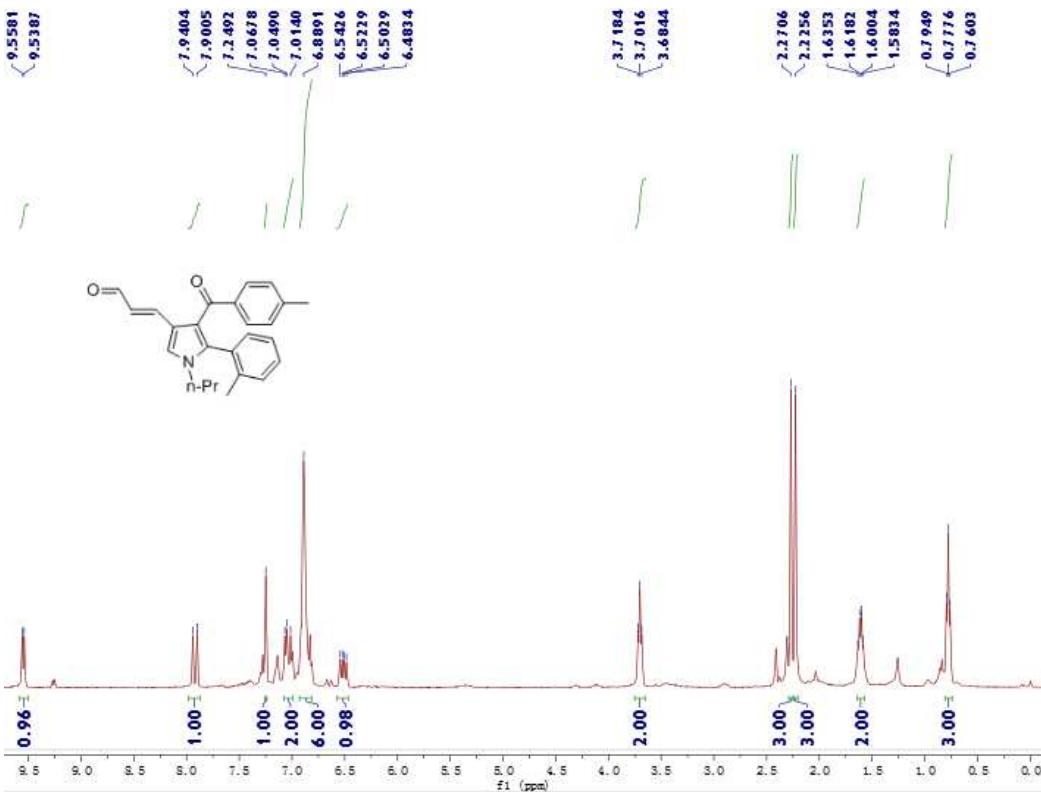


¹H NMR spectrum of **8b**

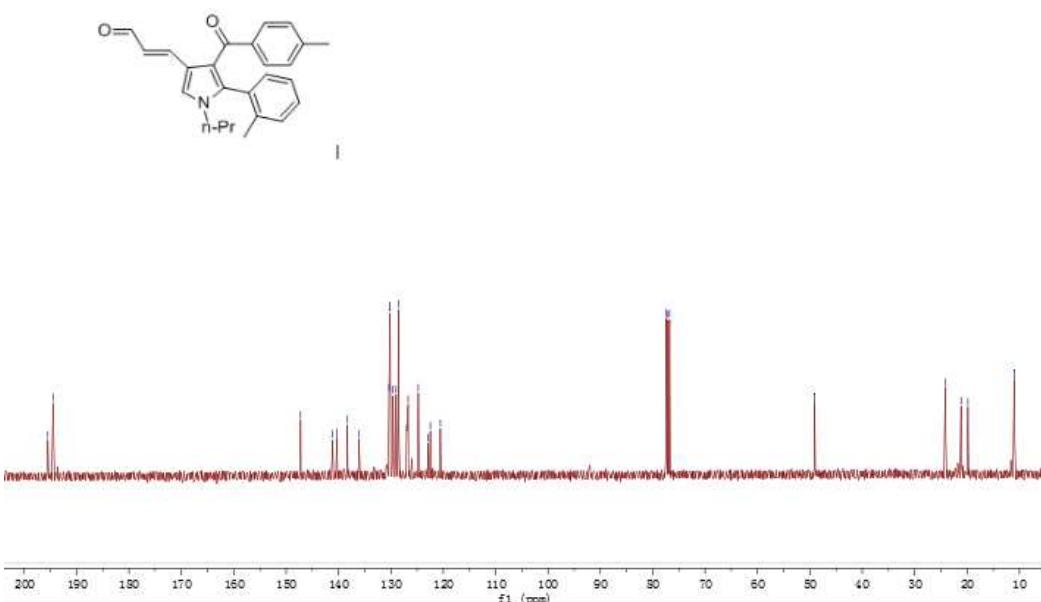


¹³C NMR spectrum of **8b**

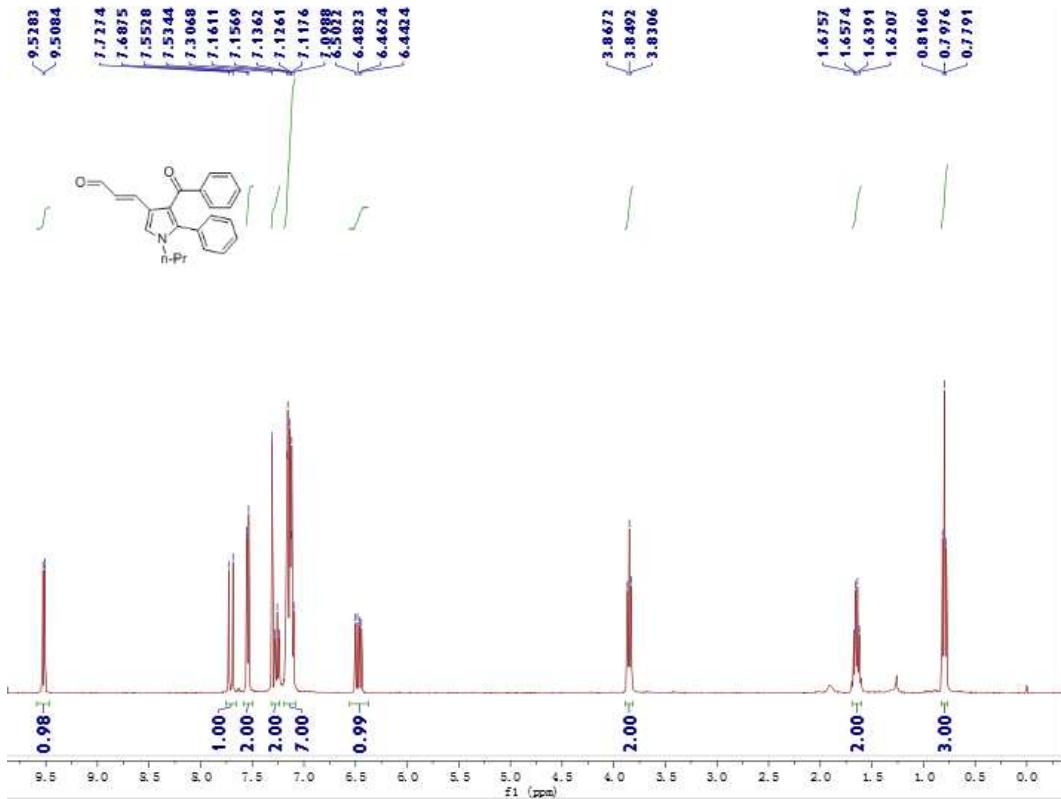




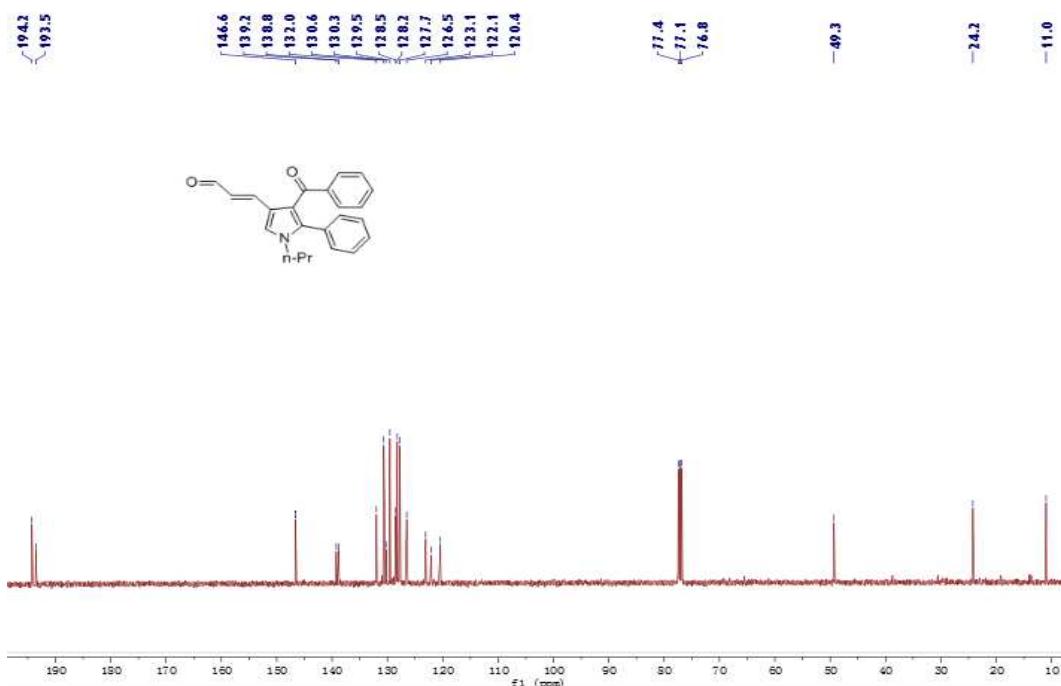
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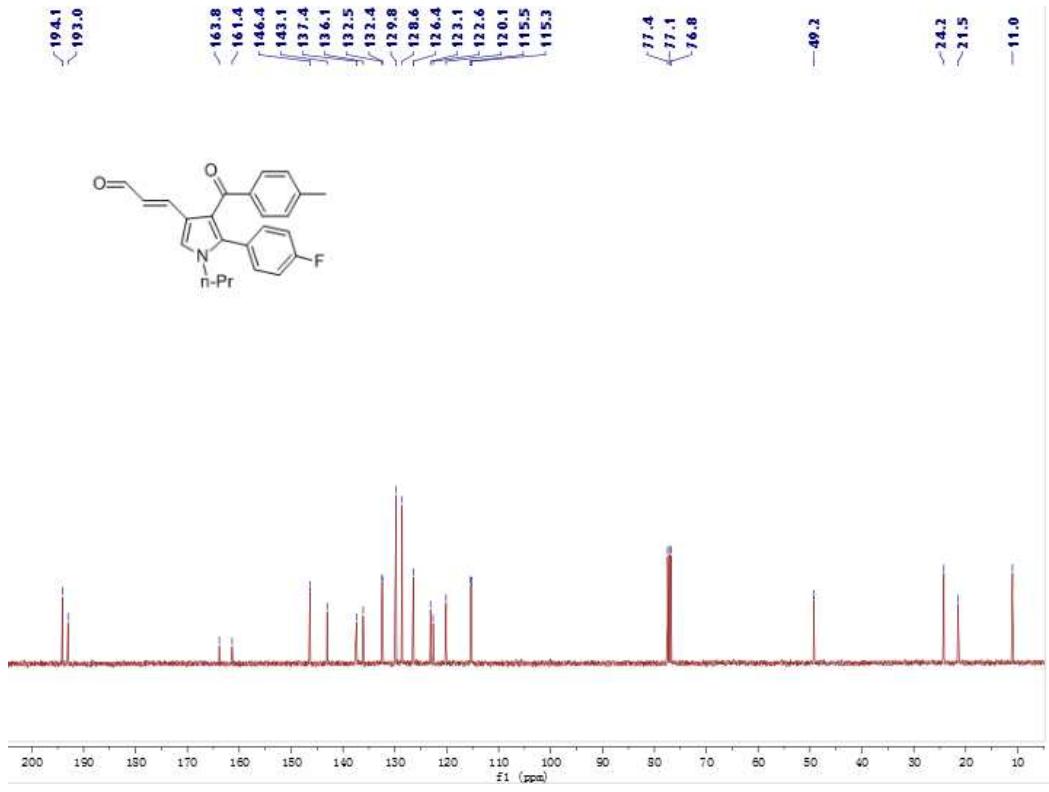
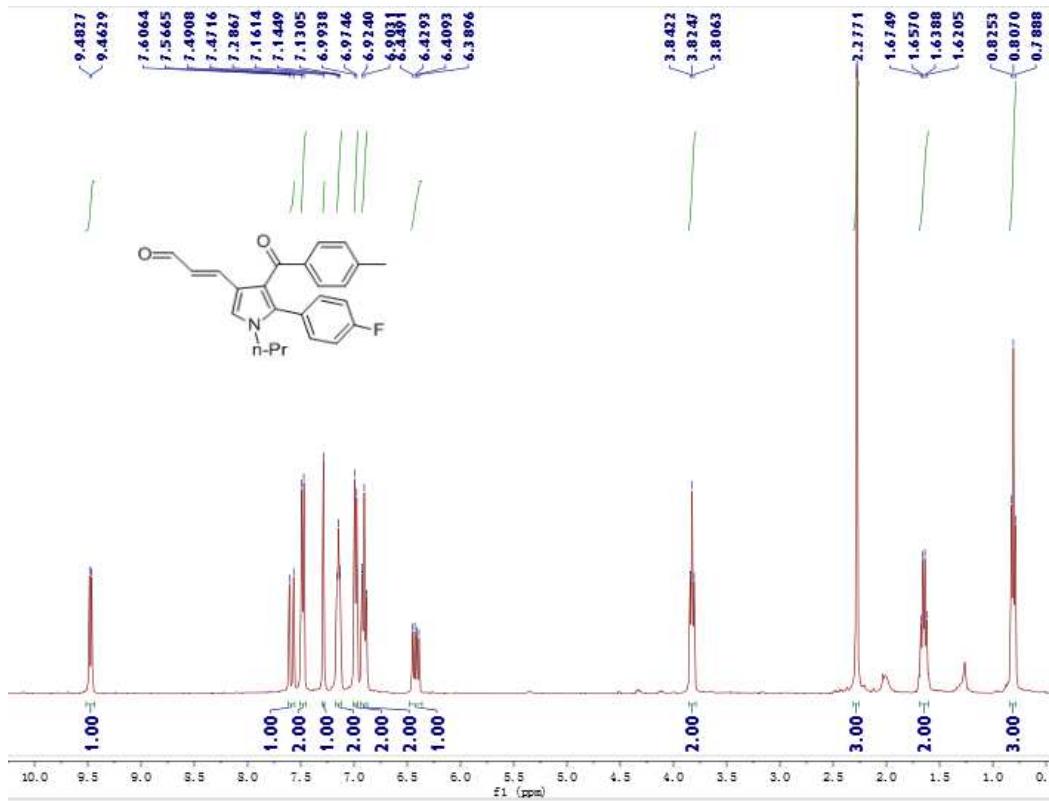
¹³C NMR spectrum of **8d**

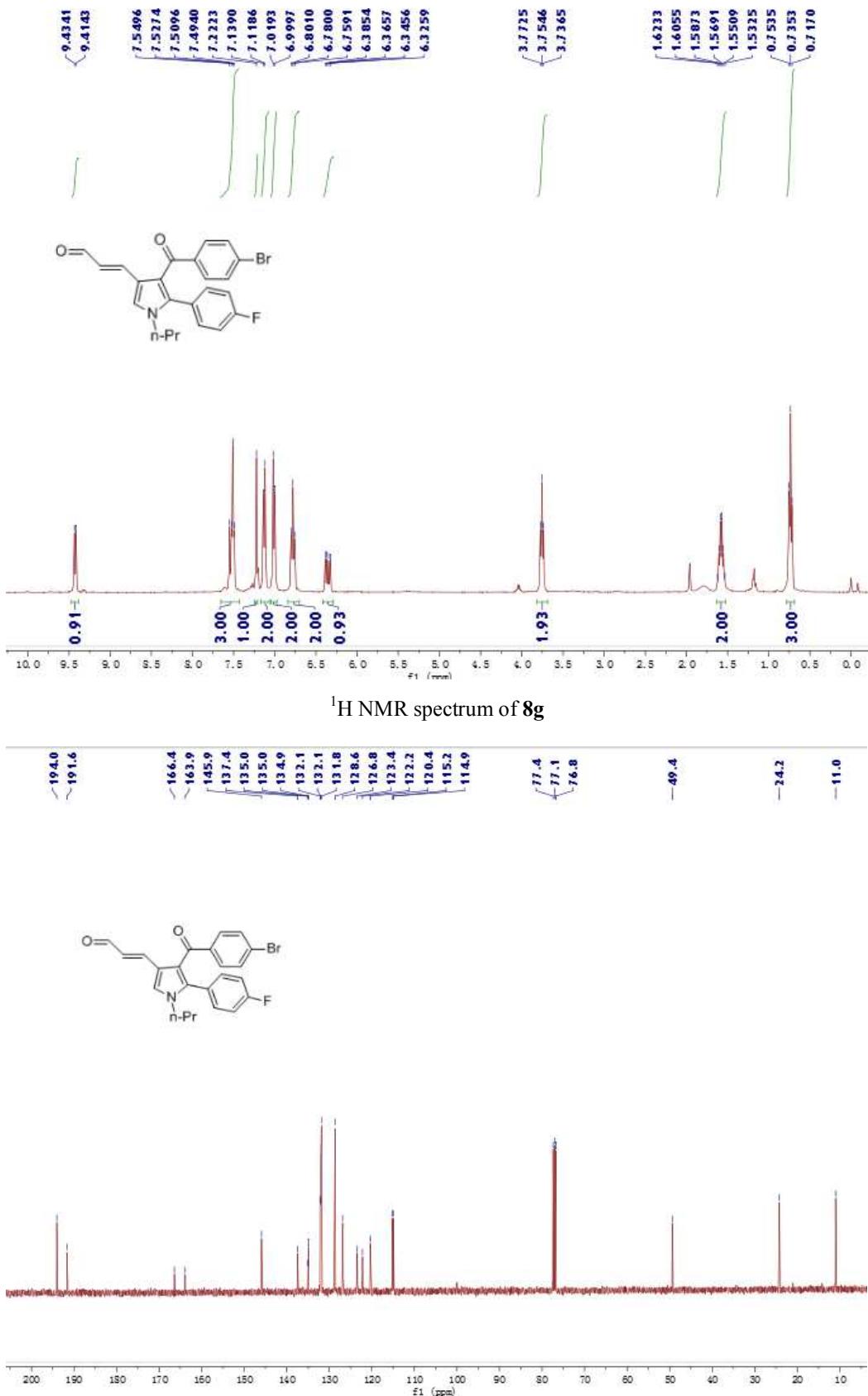


^1H NMR spectrum of **8e**

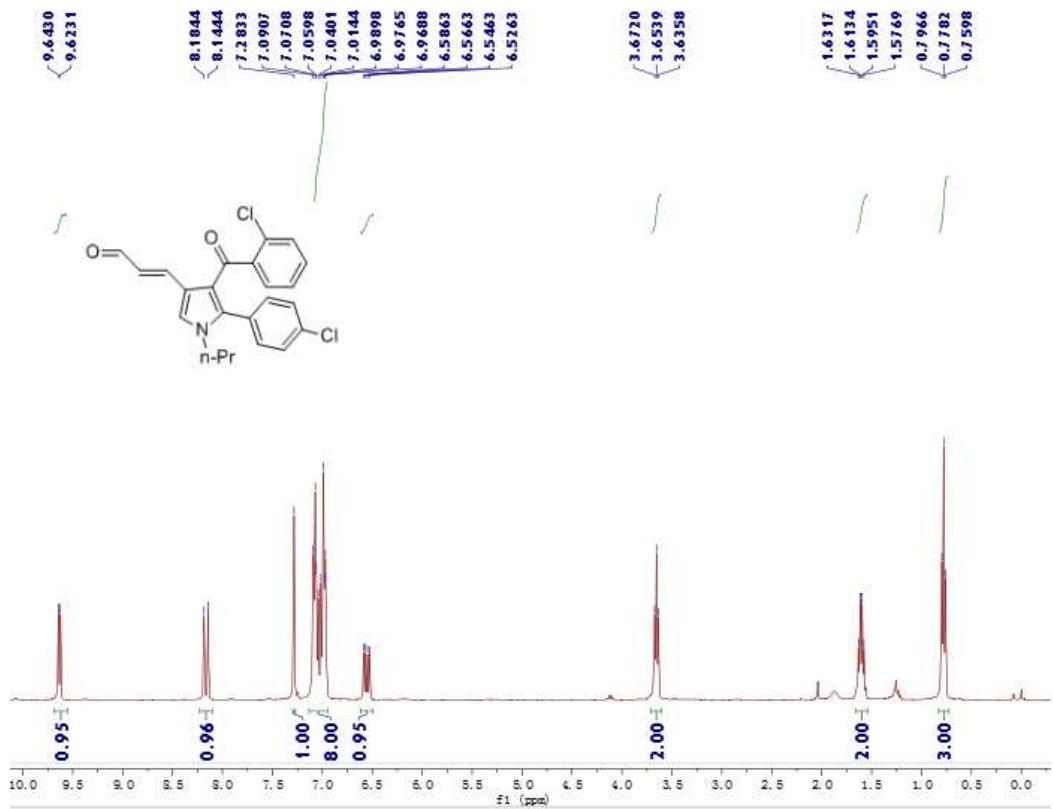


^{13}C NMR spectrum of **8e**

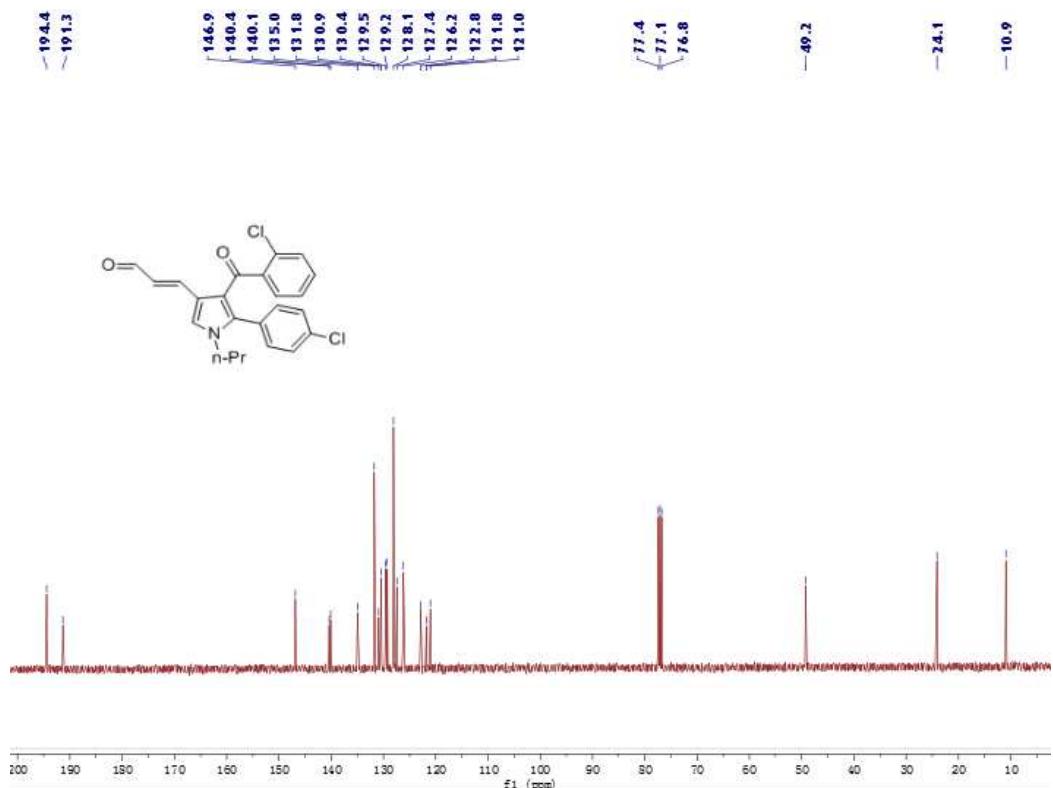




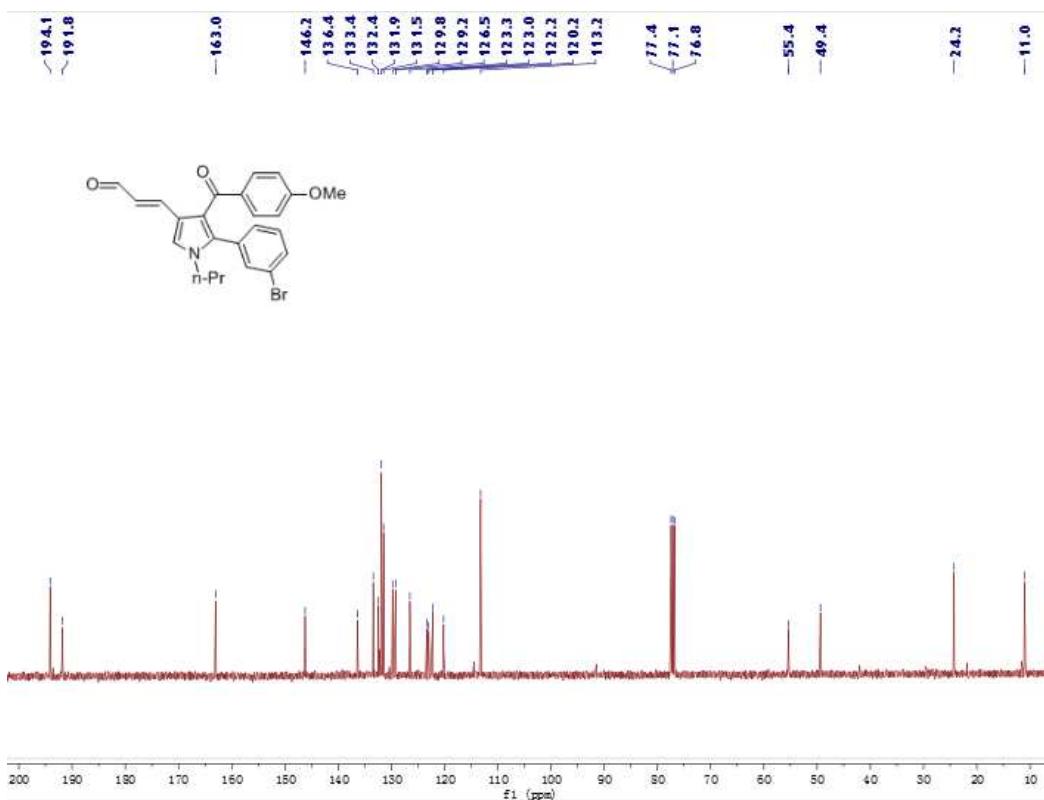
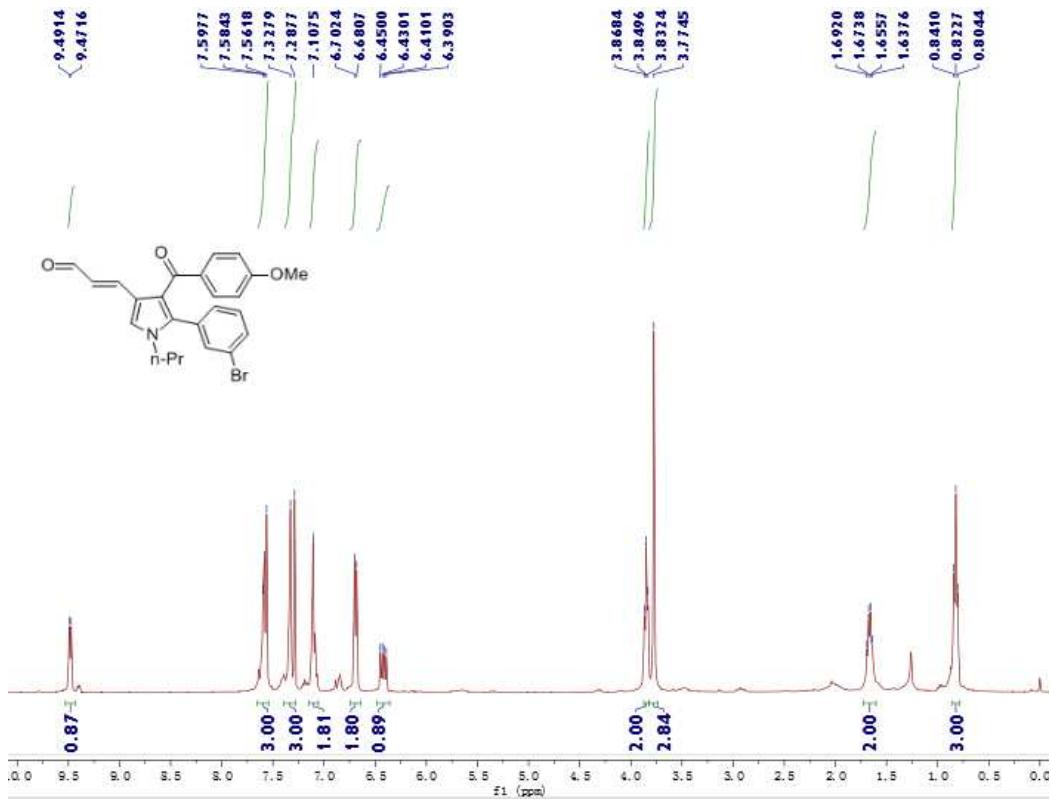
¹³C NMR spectrum of **8g**

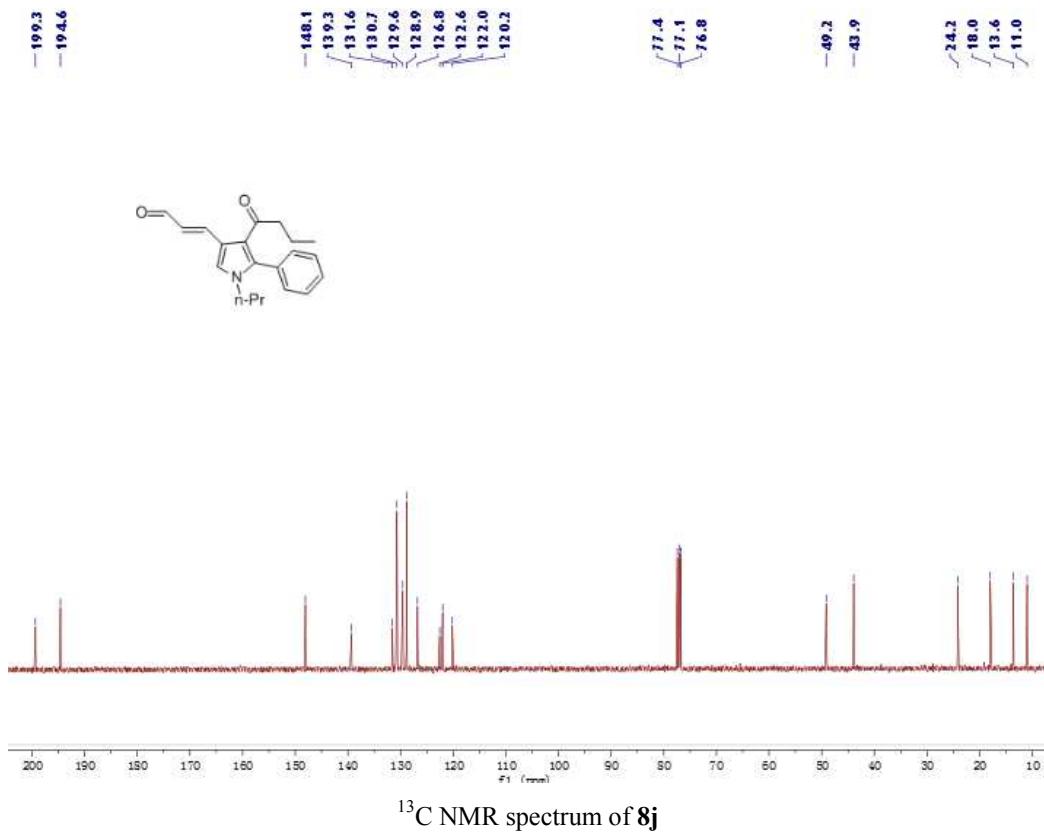
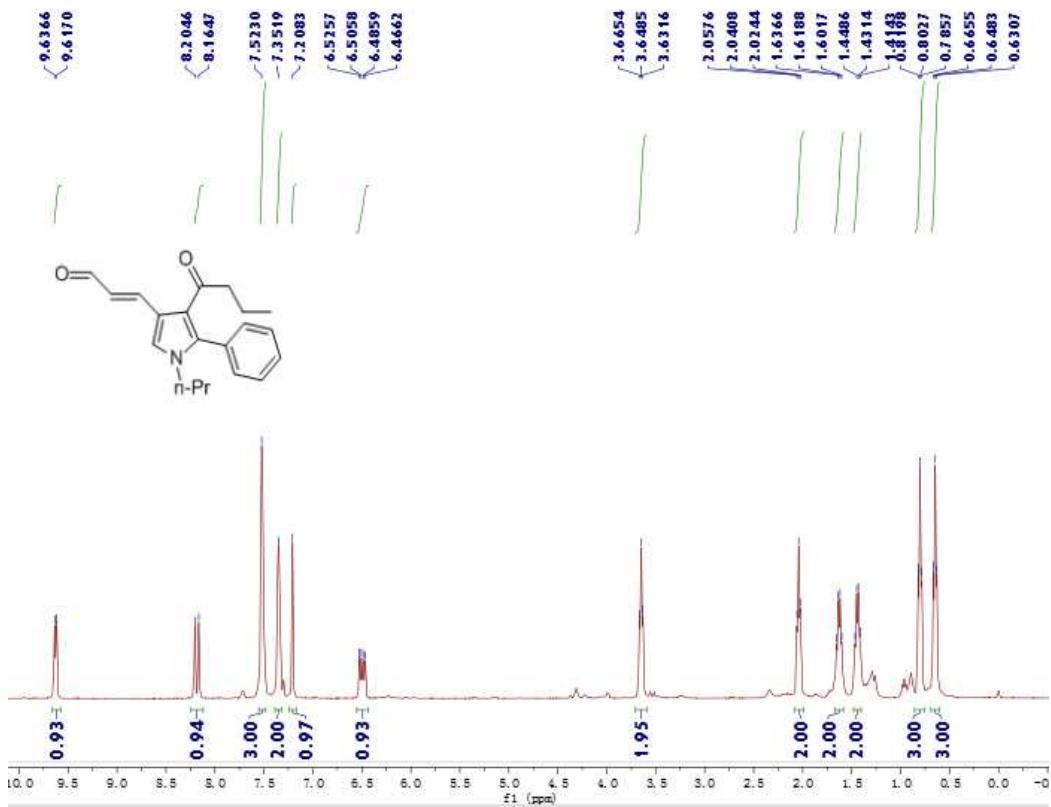


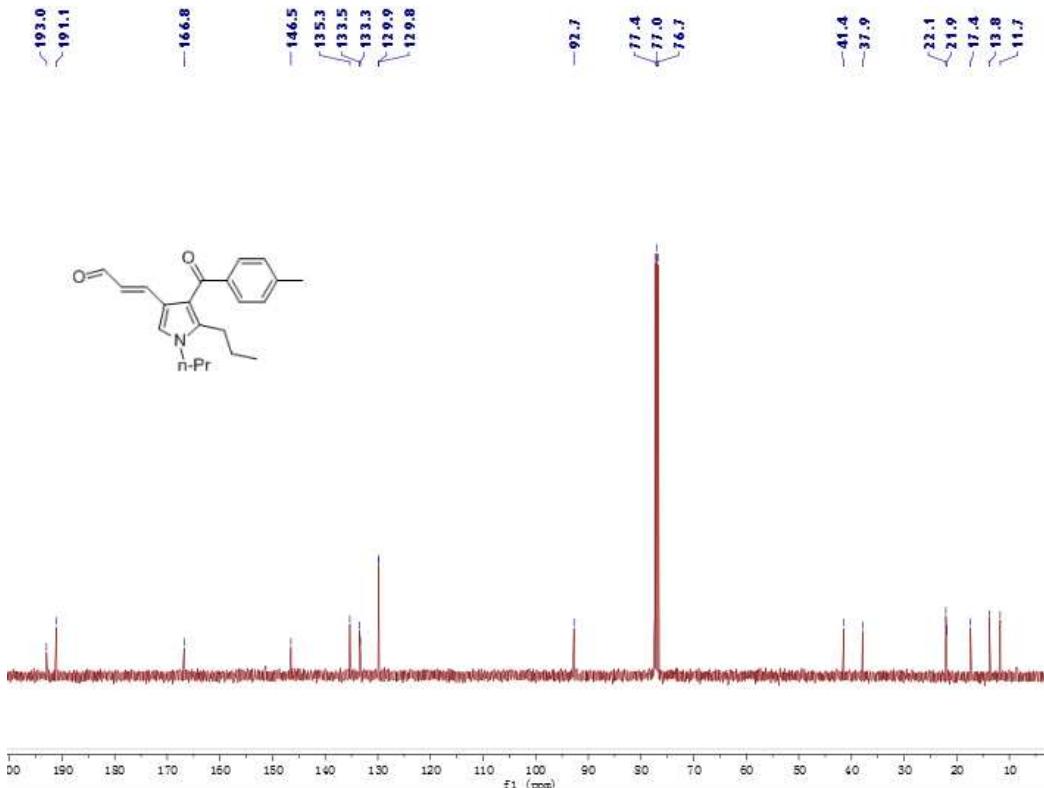
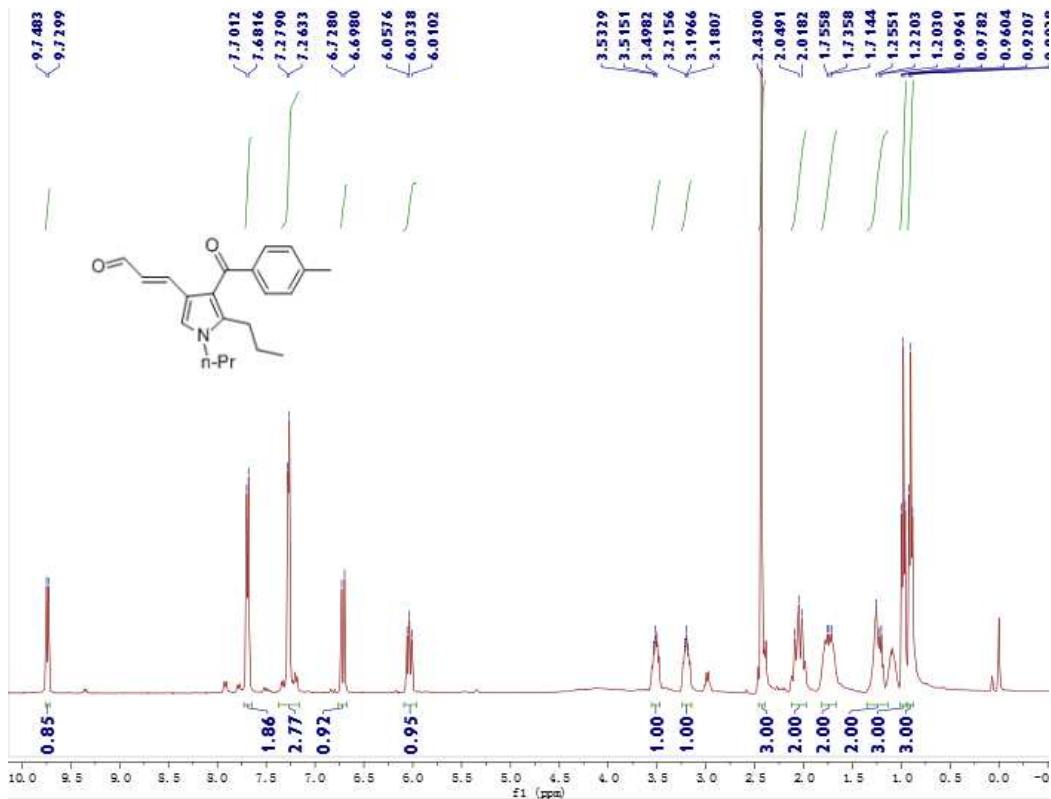
¹H NMR spectrum of **8h**



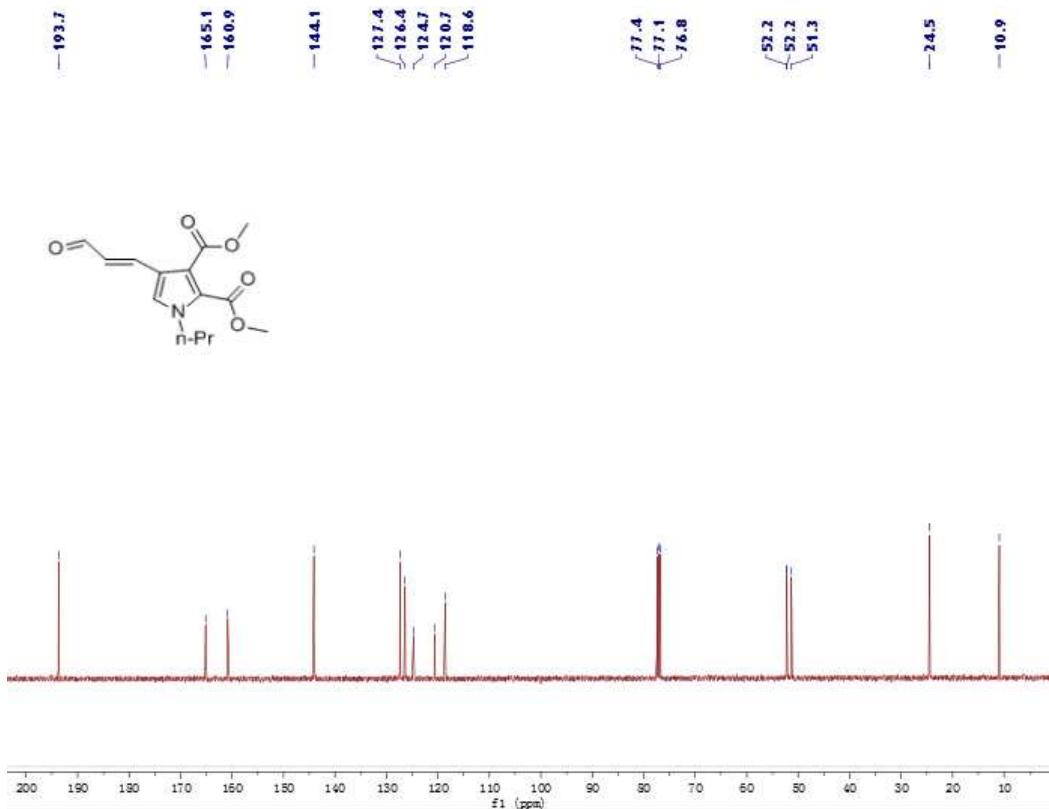
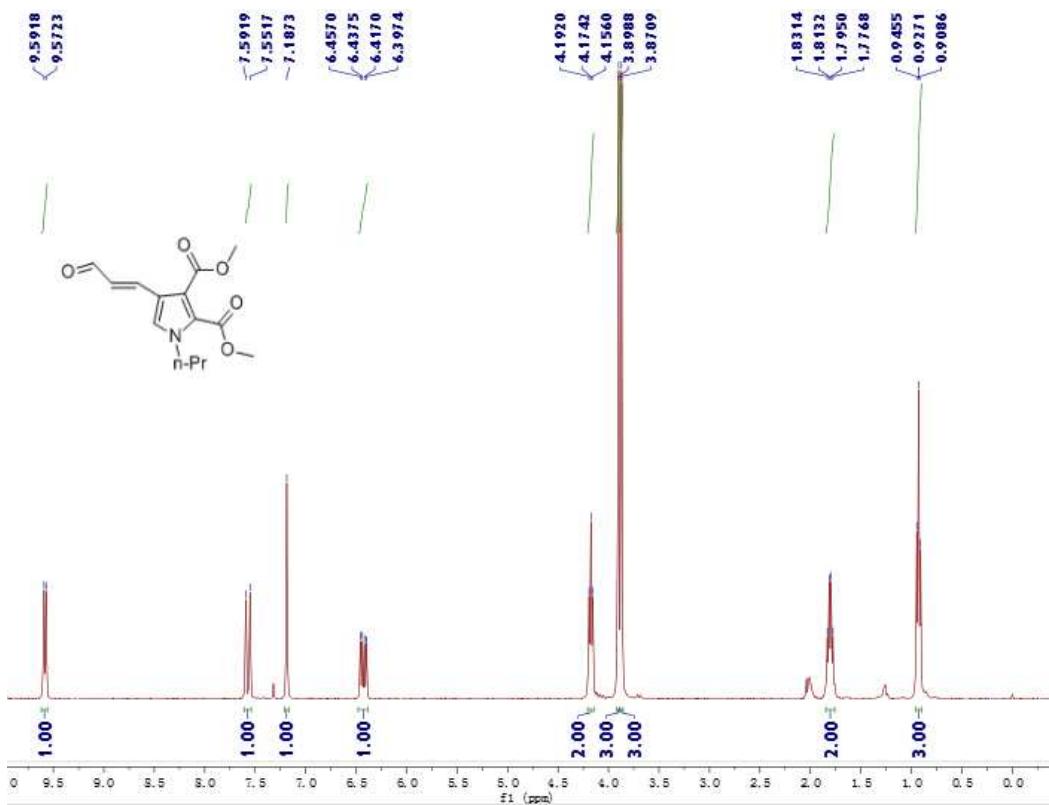
¹³C NMR spectrum of **8h**



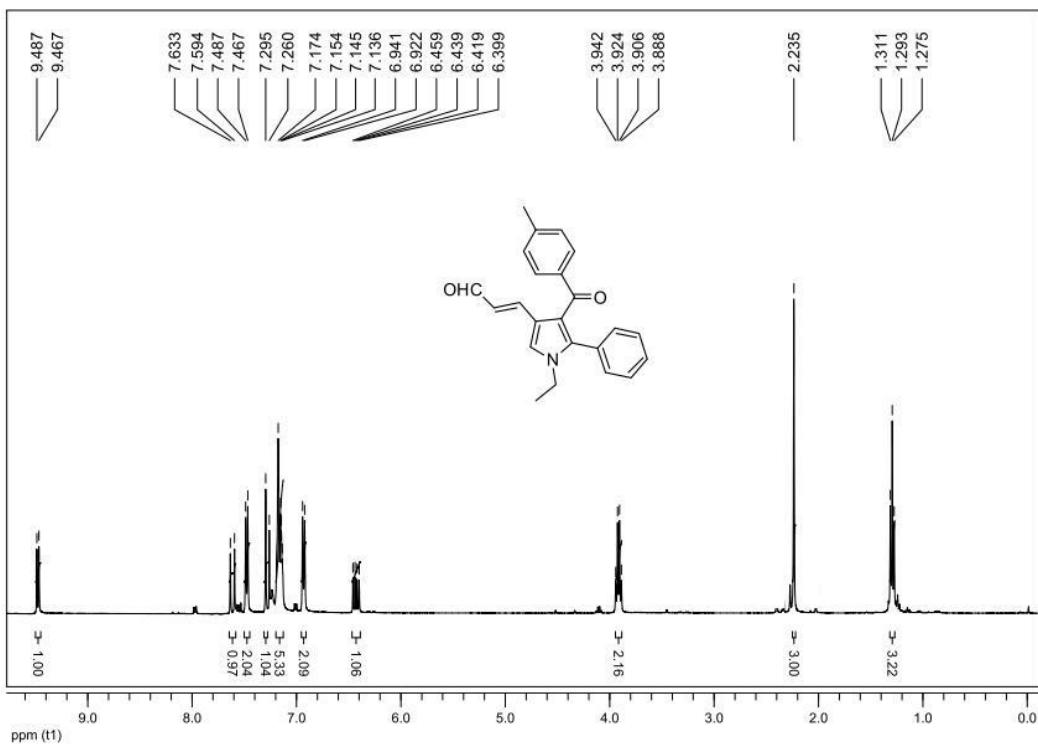




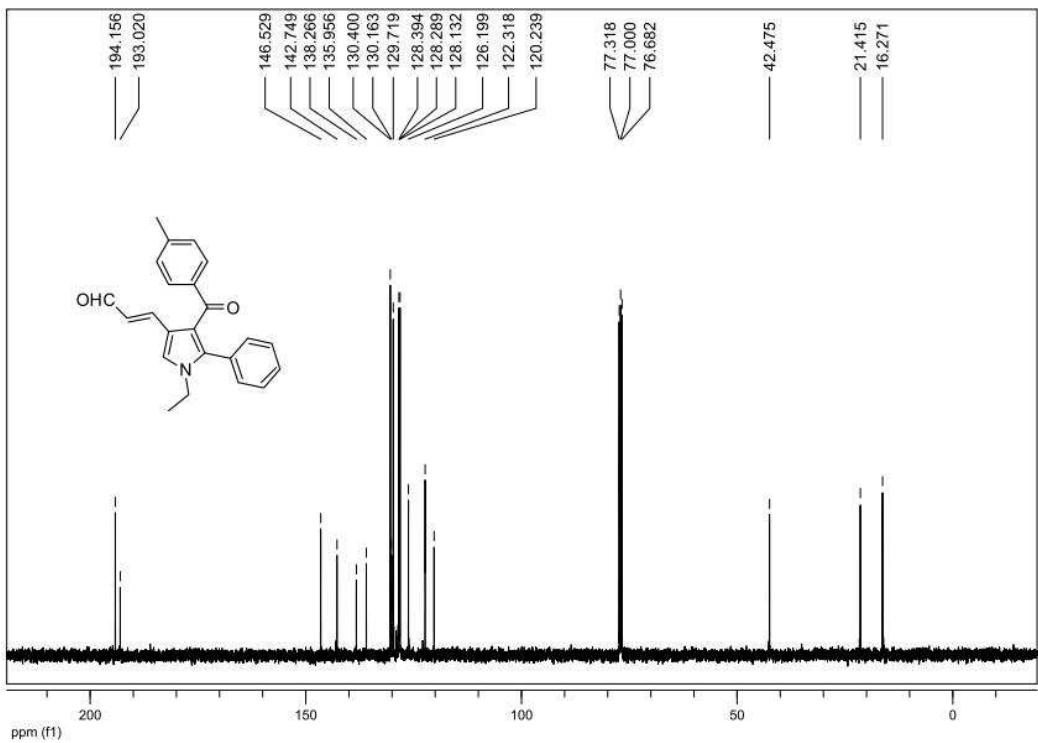
¹³C NMR spectrum of **8k**



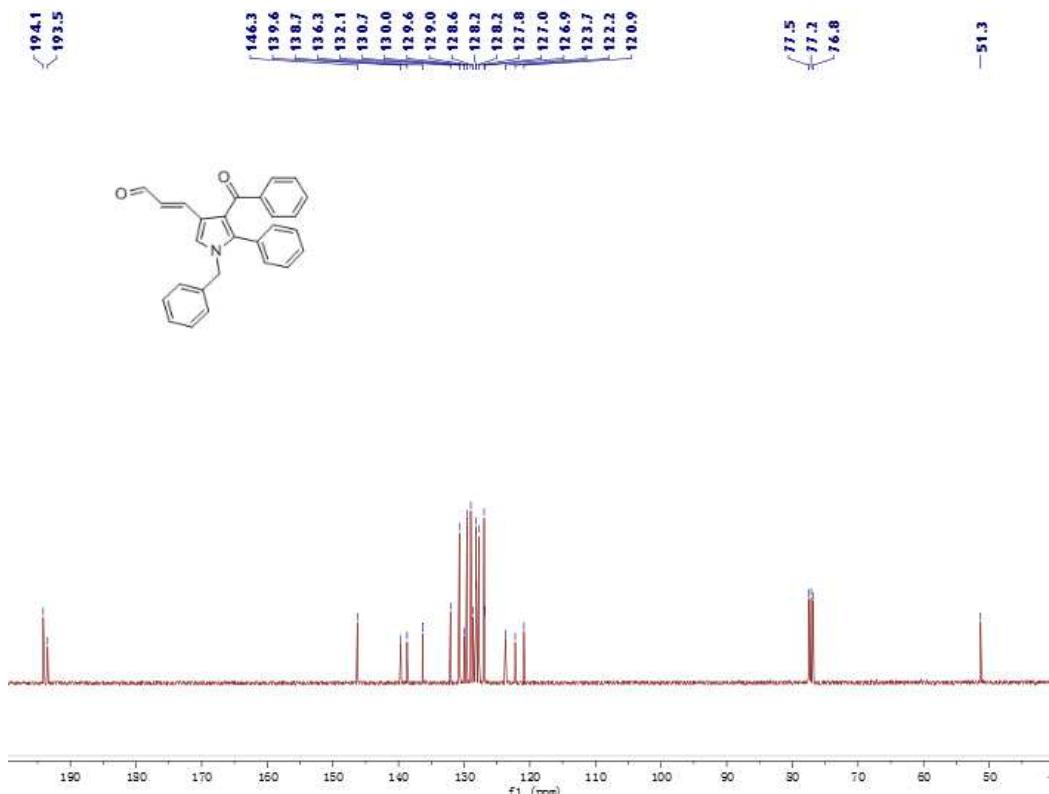
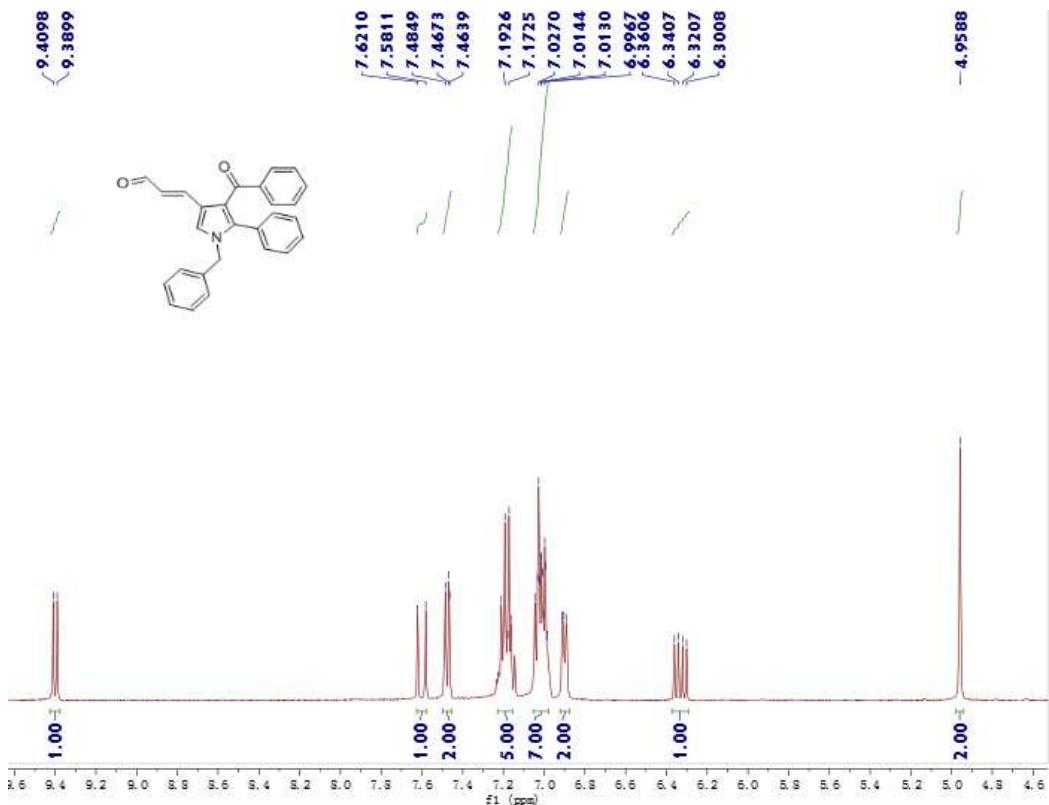
¹³C NMR spectrum of **8m**



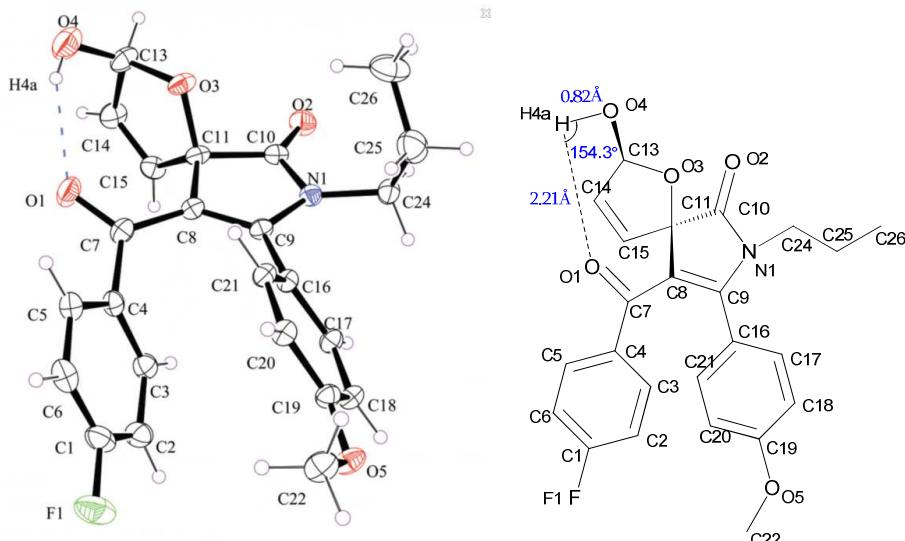
¹H NMR spectrum of **8o**



¹³C NMR spectrum of **8o**

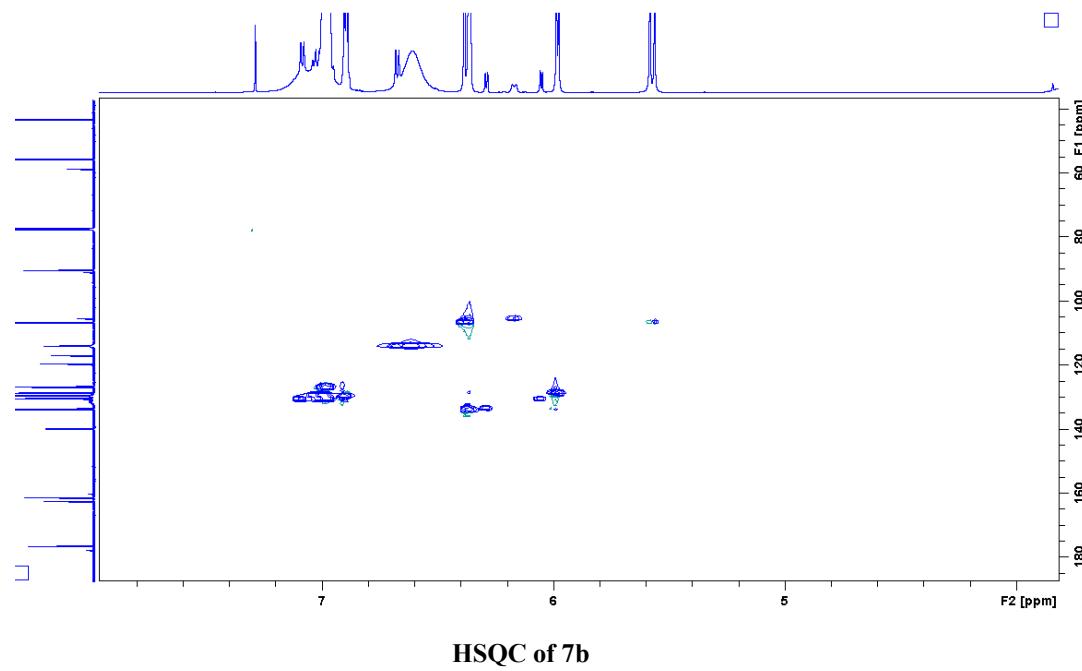
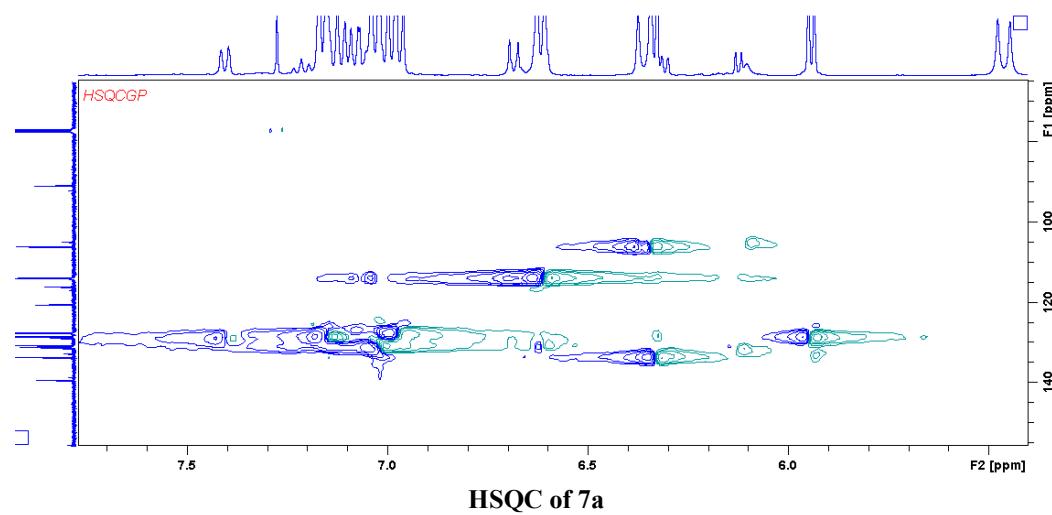


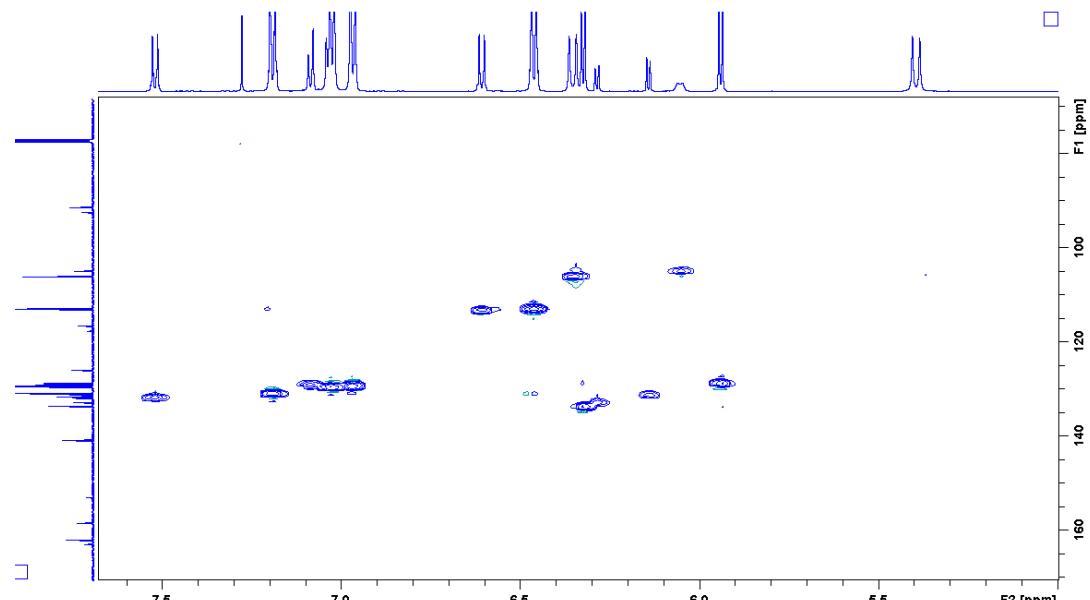
CIF information of 7c



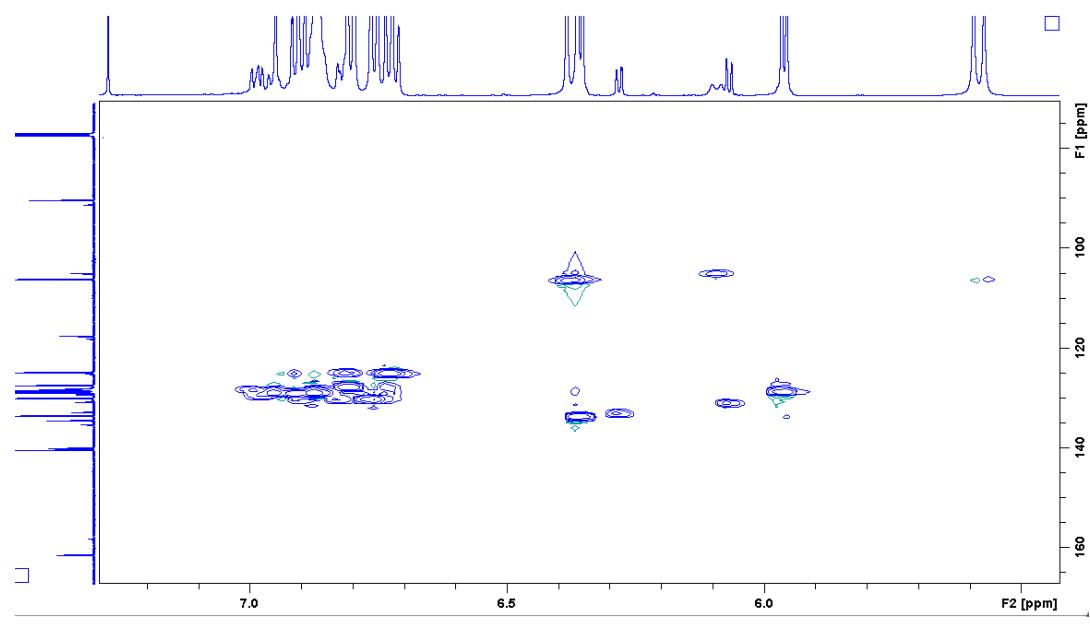
X-ray structure of 7c with 30% ellipsoid probability.

Identification code	Sad
Empirical formula	C24 H21 F N O5
Formula weight	422.42
Temperature	571(2) K
Wavelength	0.71073 Å
Crystal system, space group	Triclinic, P21/n
Unit cell dimensions	a = 10.5930(3) Å alpha = 90 deg. b = 11.1116(3) Å beta = 95.862(3) deg. c = 17.5737(5) Å gamma = 90 deg.
Volume	2057.70(10) Å ³
Z, Calculated density	4, 1.364 Mg/m ³
Absorption coefficient	0.102 mm ⁻¹
F(000)	884
Crystal size	0.25 x 0.20 x 0.18 mm
Theta range for data collection	2.15 to 24.25 deg.
Limiting indices	-12<=h<=12, -12<=k<=12, -20<=l<=20
Reflections collected / unique	32467 / 3287 [R(int) = 0.0545]
Completeness to theta = 24.25	98.8 %
Max. and min. transmission	0.9819 and 0.9750
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	3287 / 0 / 281
Goodness-of-fit on F ²	1.085
Final R indices [I>2sigma(I)]	R1 = 0.0693, wR2 = 0.1822
R indices (all data)	R1 = 0.0728, wR2 = 0.1849
Extinction coefficient	0.021(4)
Largest diff. peak and hole	1.429 and -0.353 e.Å ⁻³





HSQC of 7e



HSQC of 7r