Supporting Information for

General Approach to Construct Azepino[2,3-b:4,5-*b'*]diindoles, Azocino[2,3-b:4,5-*b'*]diindoles and Azonino[2,3-b:4,5-*b'*]diindolesvia Rh(II)-Catalyzed Reactions of 3-Diazoindolin-2-imines with 3-(Bromoalkyl)indoles

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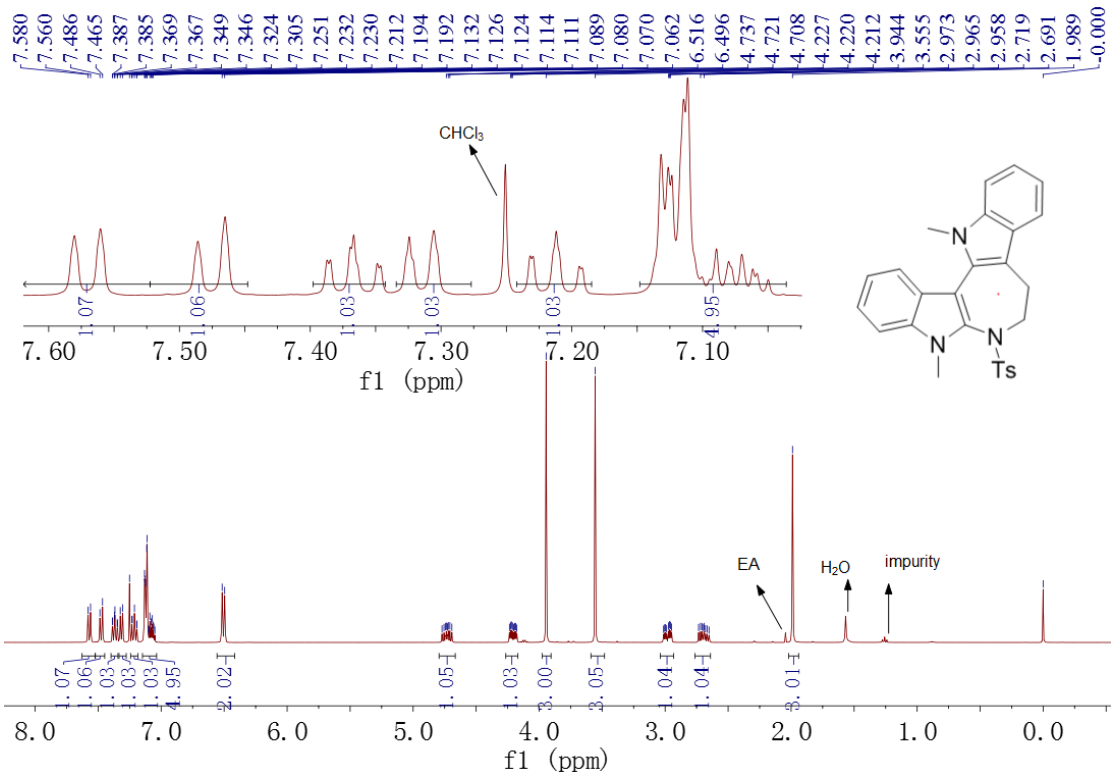
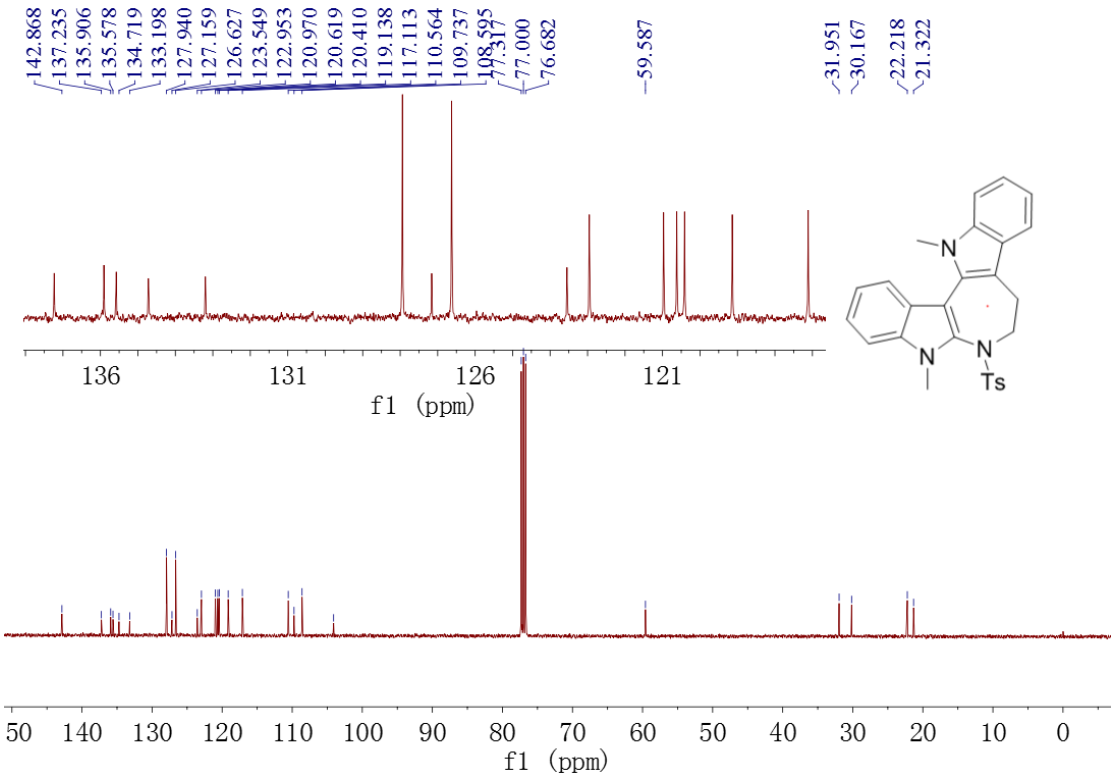
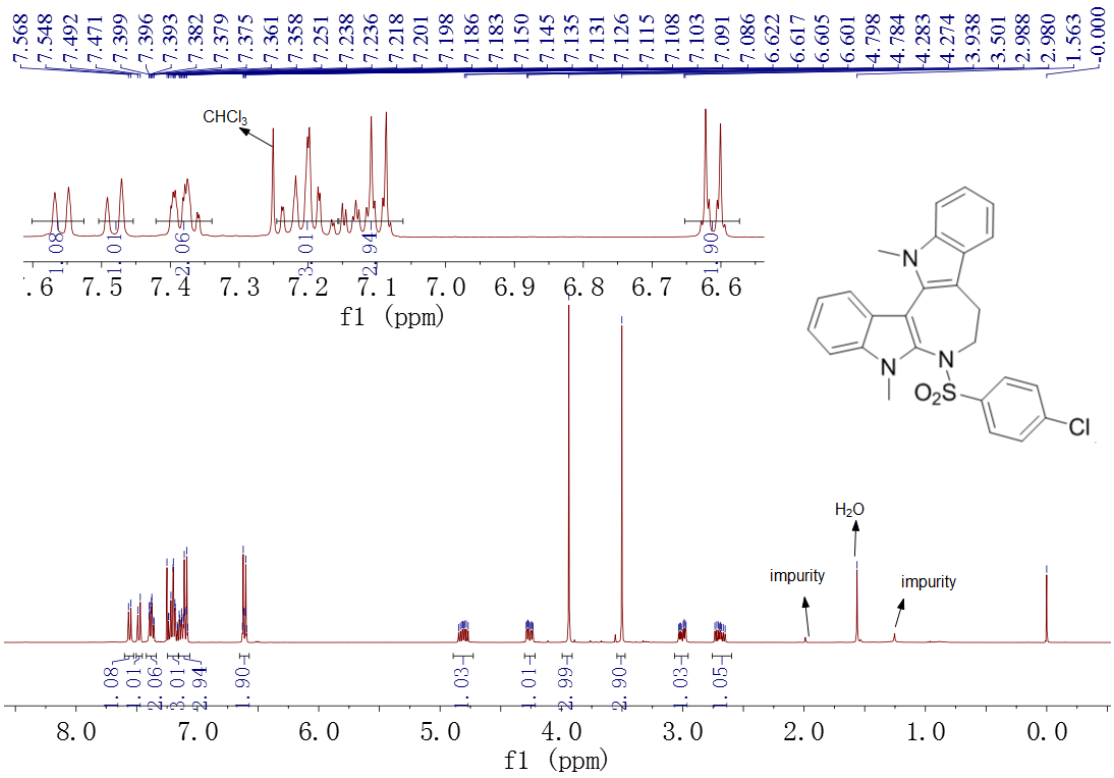
Copies of ^{1}H NMR & ^{13}C NMR Spectra



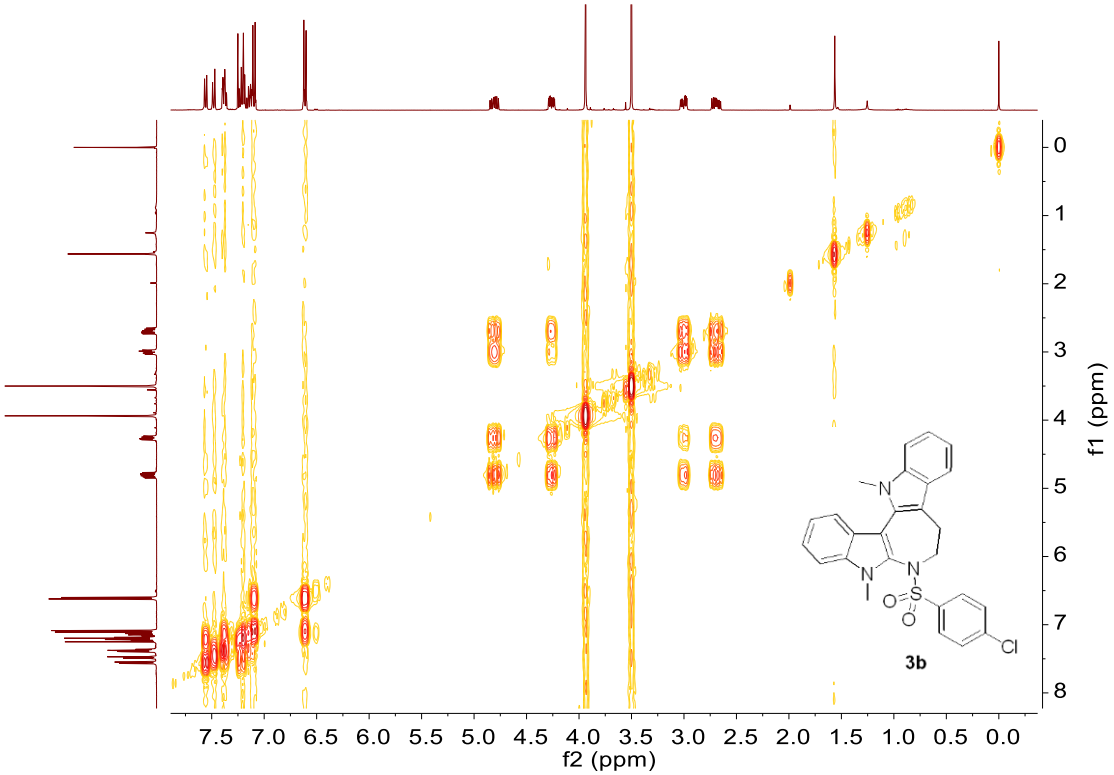
^{1}H NMR Spectrum for 3a ($CDCl_3$, 400 MHz)



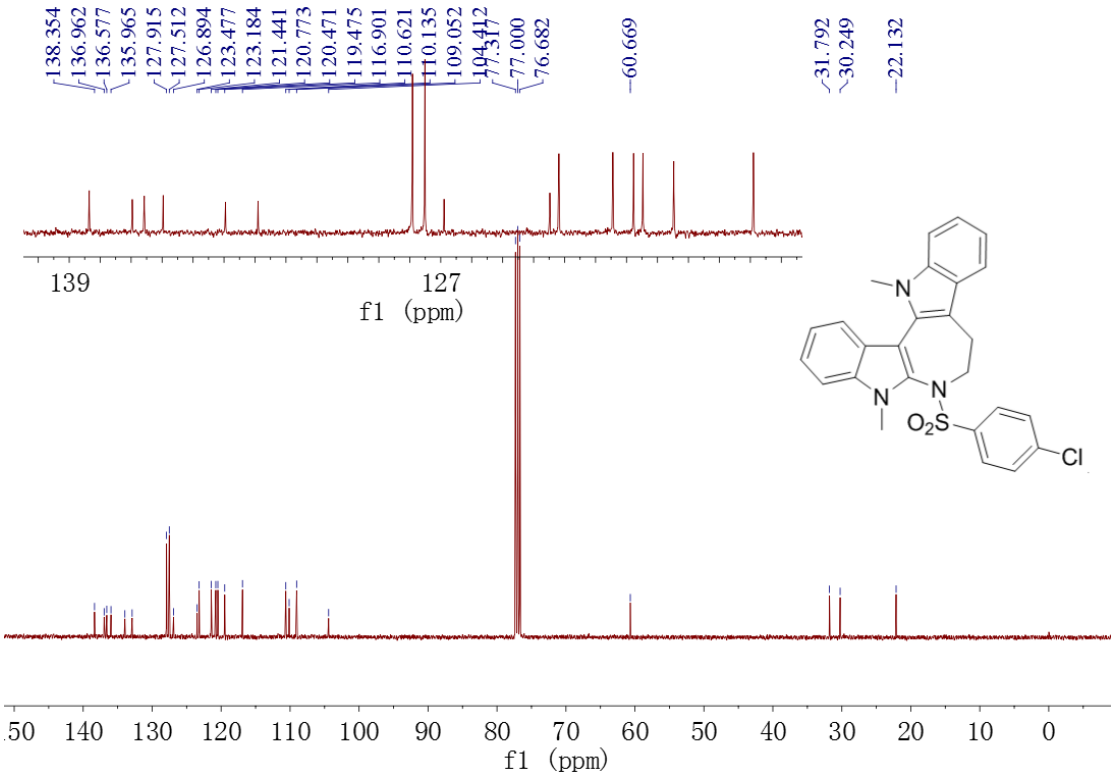
^{13}C NMR Spectrum for 3a ($CDCl_3$, 100 MHz)



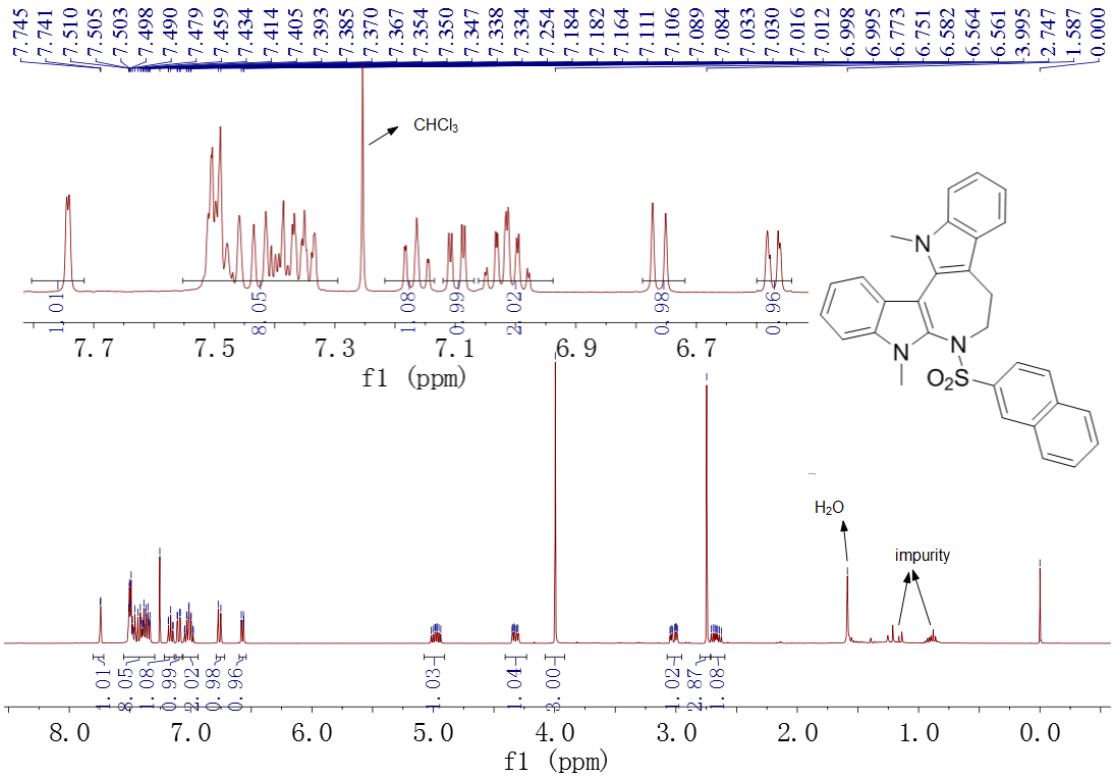
^{1}H NMR Spectrum for 3b ($CDCl_3$, 400 MHz)



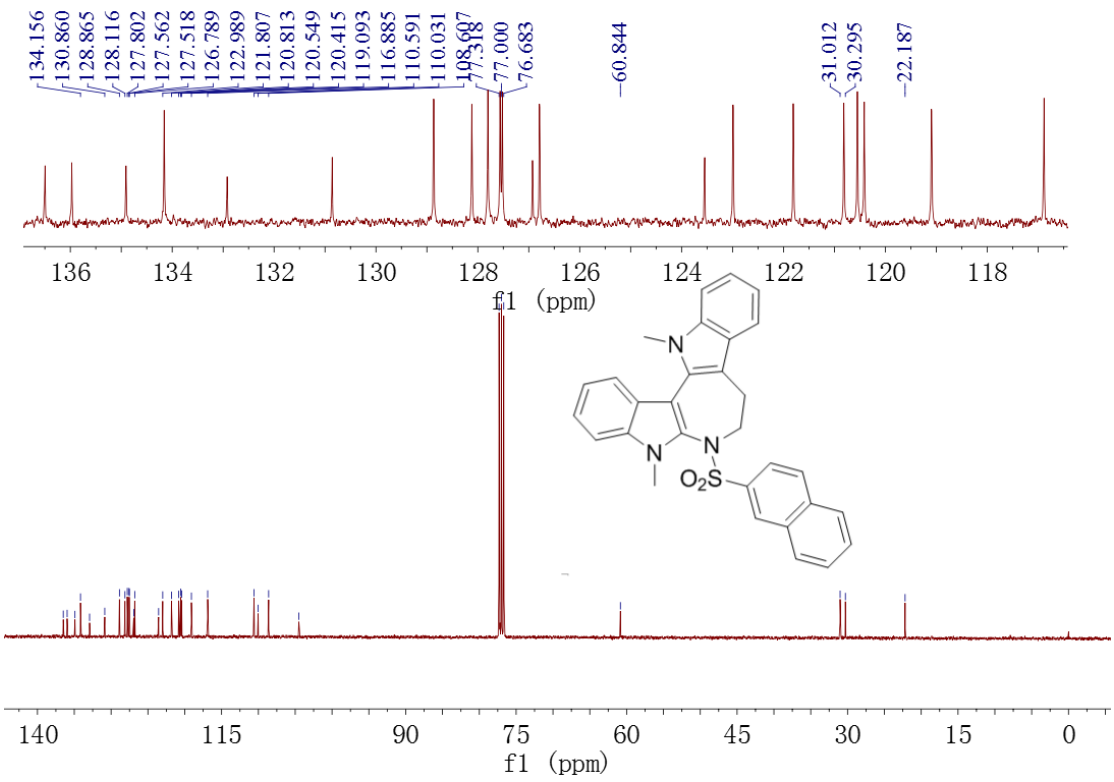
COSY Spectrum for 3b ($CDCl_3$, 400 MHz)



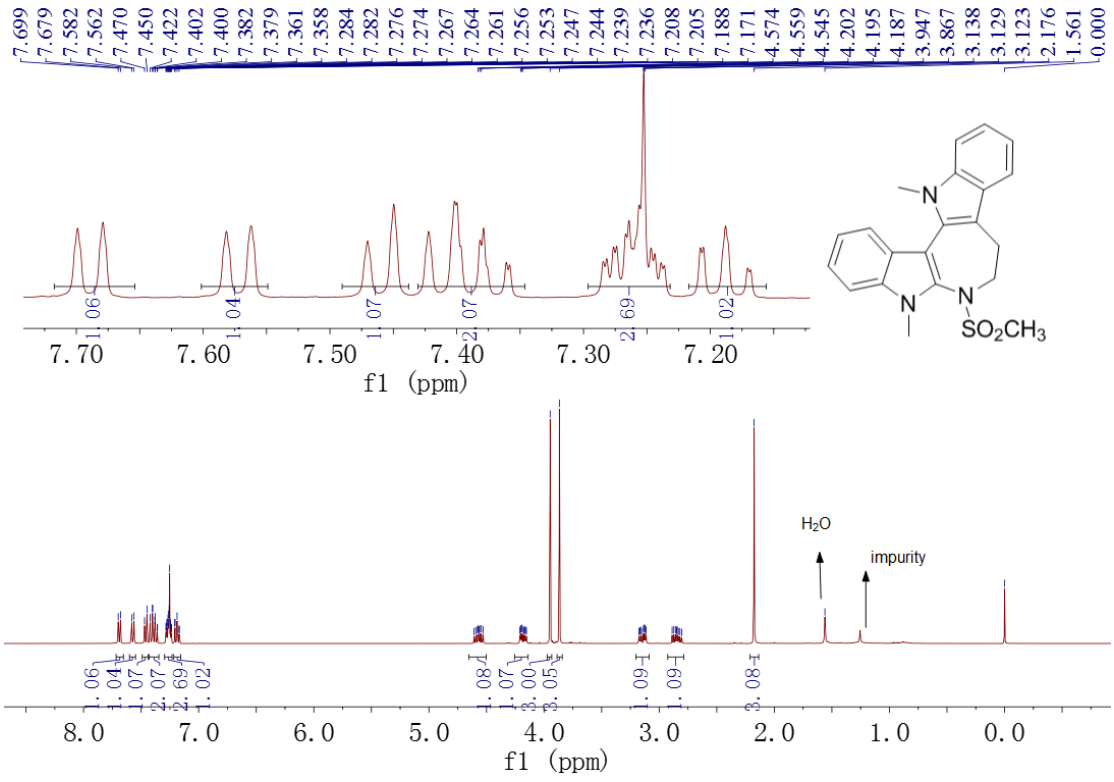
^{13}C NMR Spectrum for 3b ($CDCl_3$, 100 MHz)



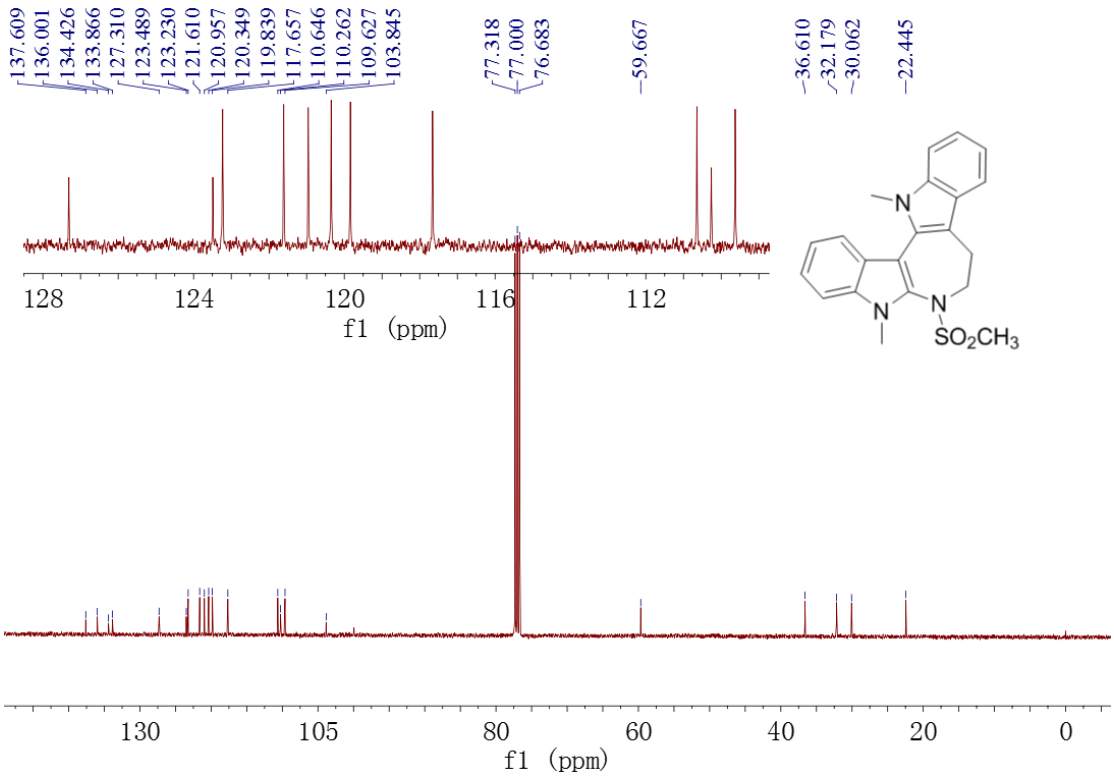
^{1}H NMR Spectrum for 3c ($CDCl_3$, 400 MHz)



^{13}C NMR Spectrum for 3c ($CDCl_3$, 100 MHz)



^{1}H NMR Spectrum for 3d ($CDCl_3$, 400 MHz)



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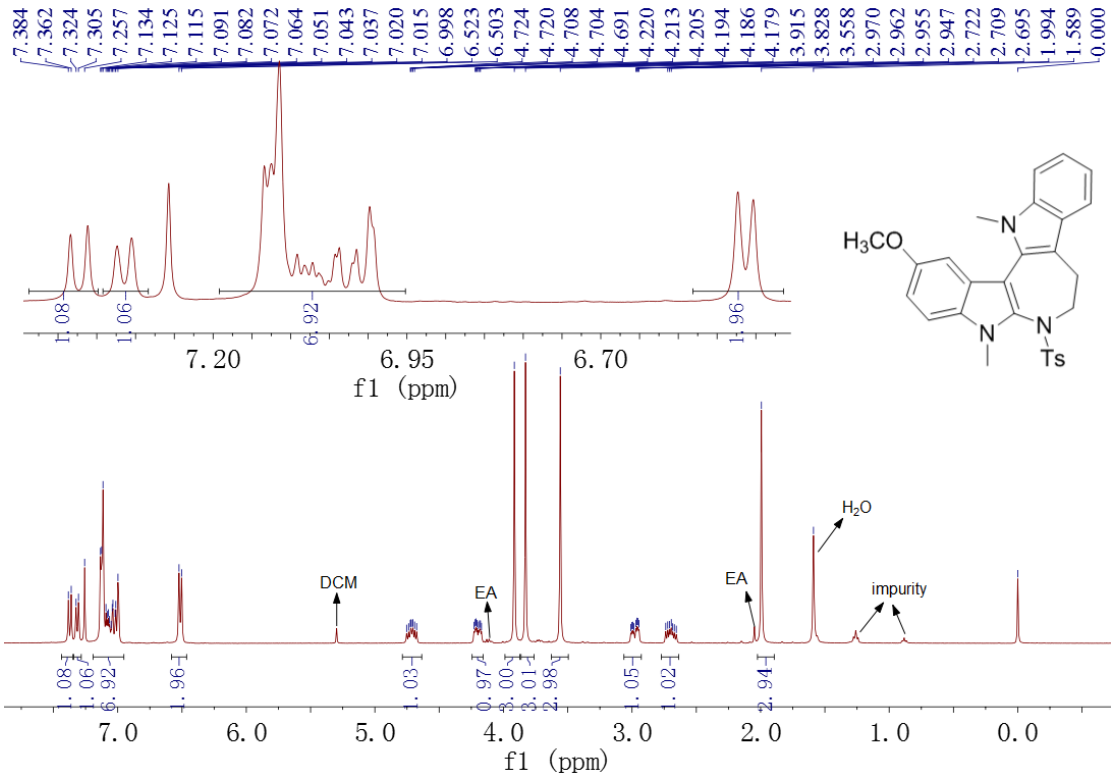
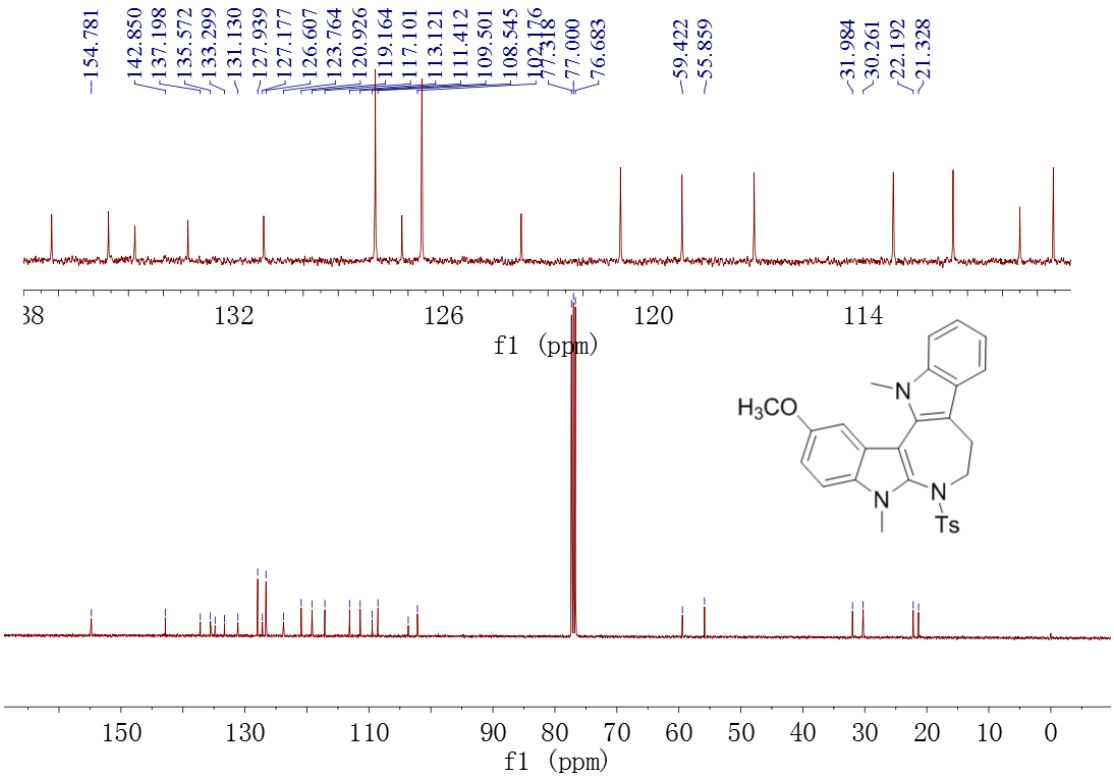


^{1}H NMR Spectrum for 3e ($CDCl_3$, 400 MHz)



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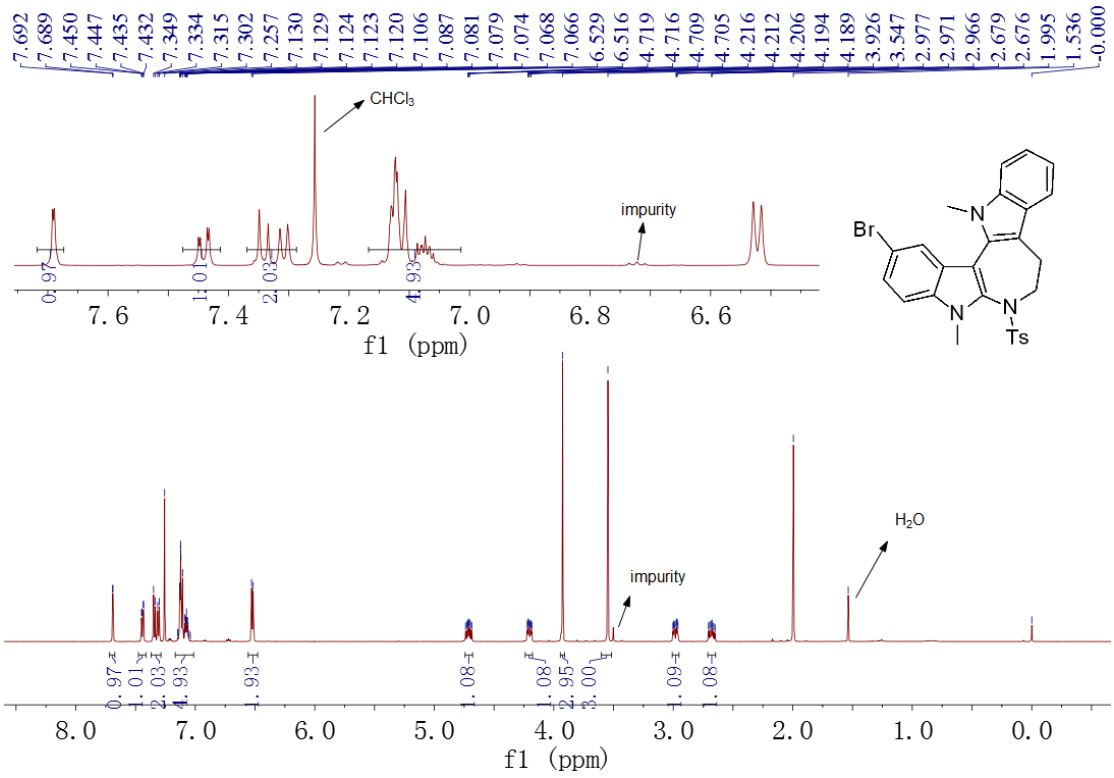
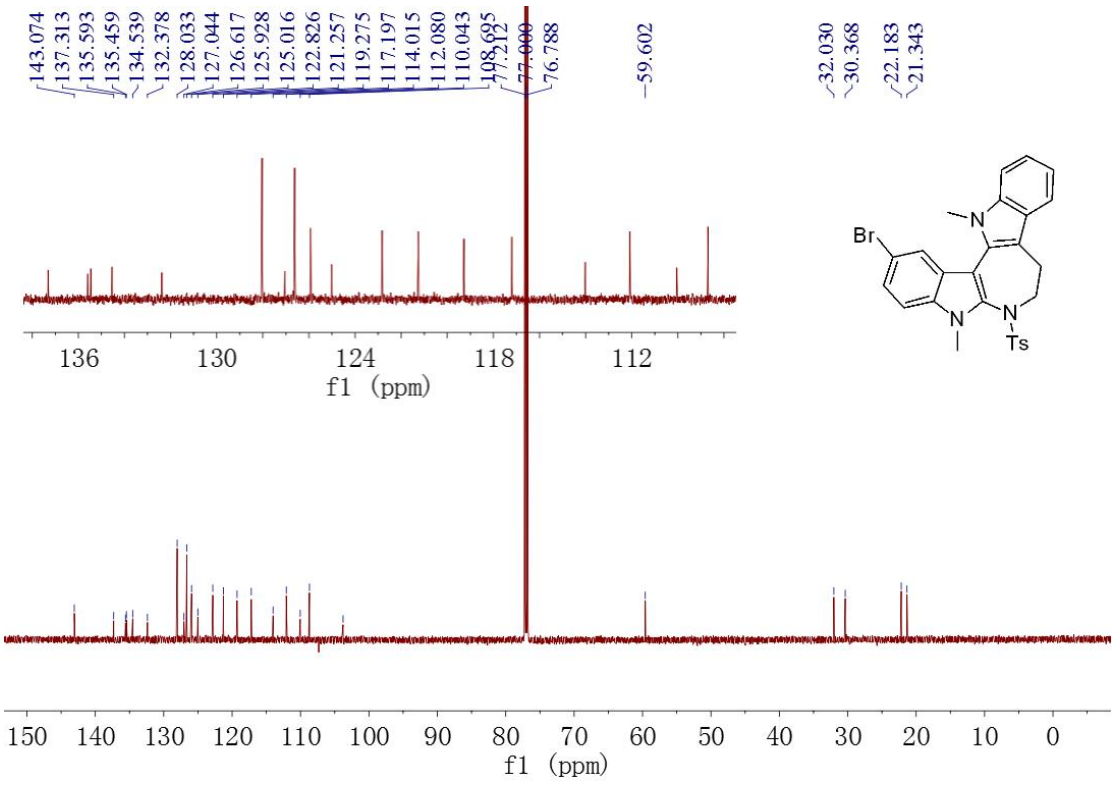
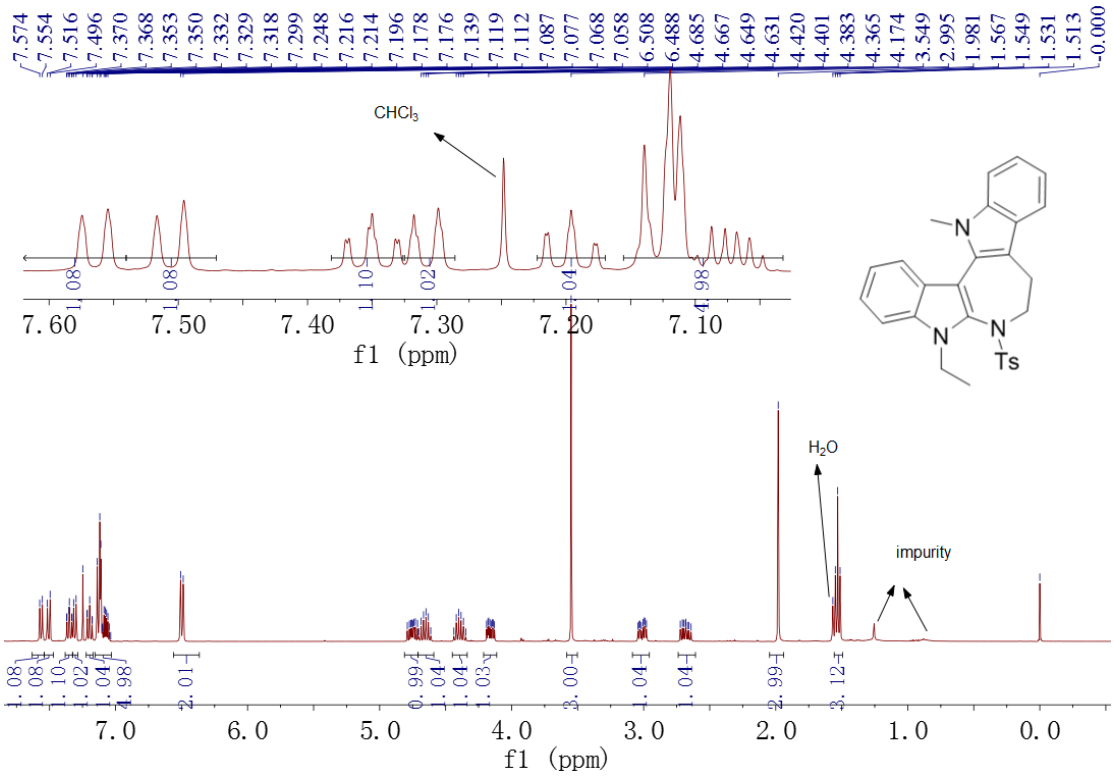


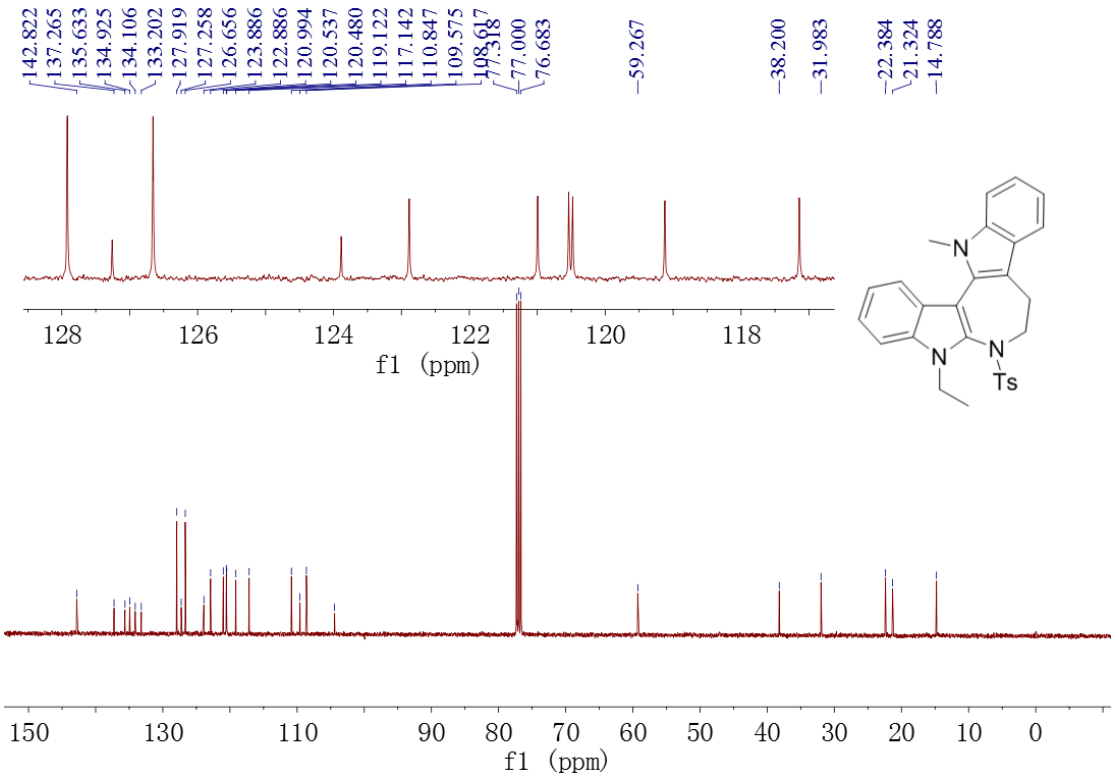
^{1}H NMR Spectrum for 3f ($CDCl_3$, 600 MHz)



^{13}C NMR Spectrum for 3f ($CDCl_3$, 150 MHz)



^{1}H NMR Spectrum for 3g ($CDCl_3$, 400 MHz)



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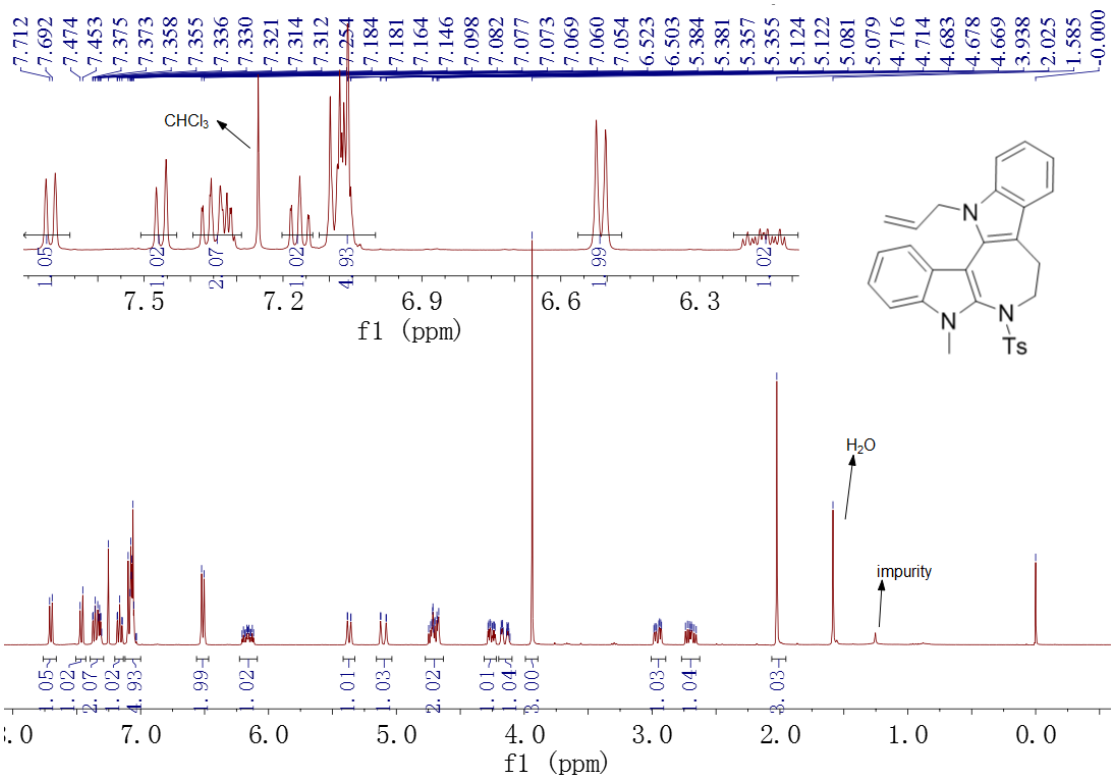
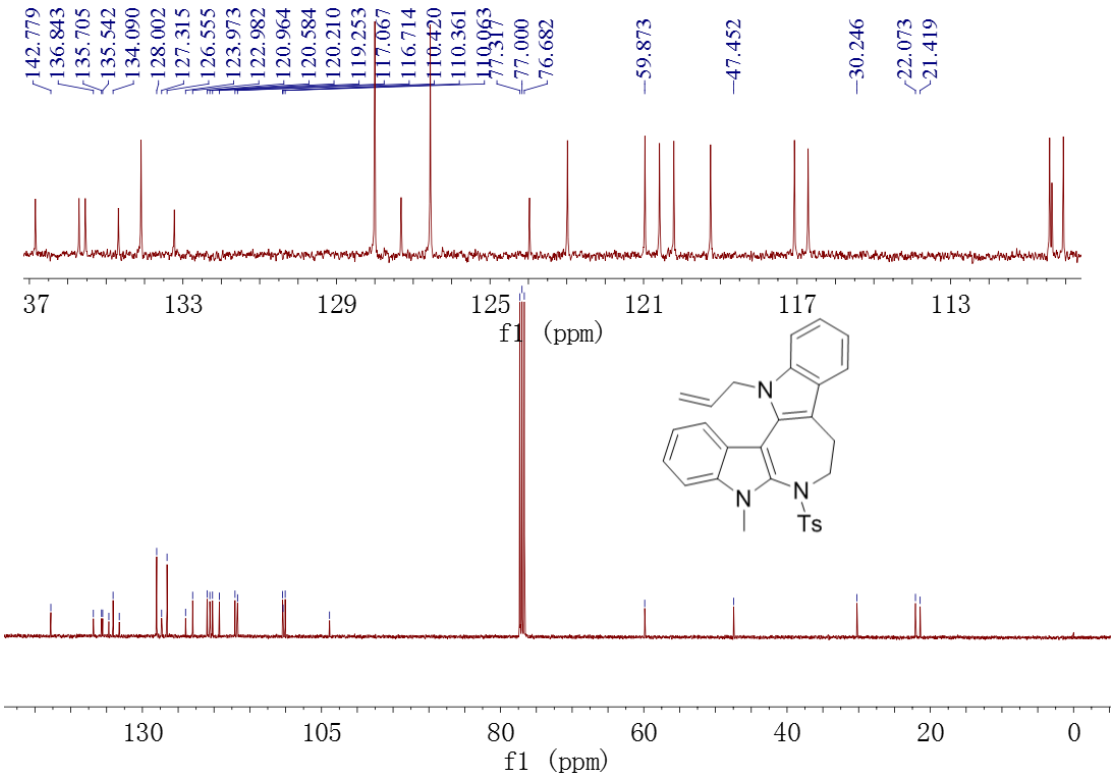
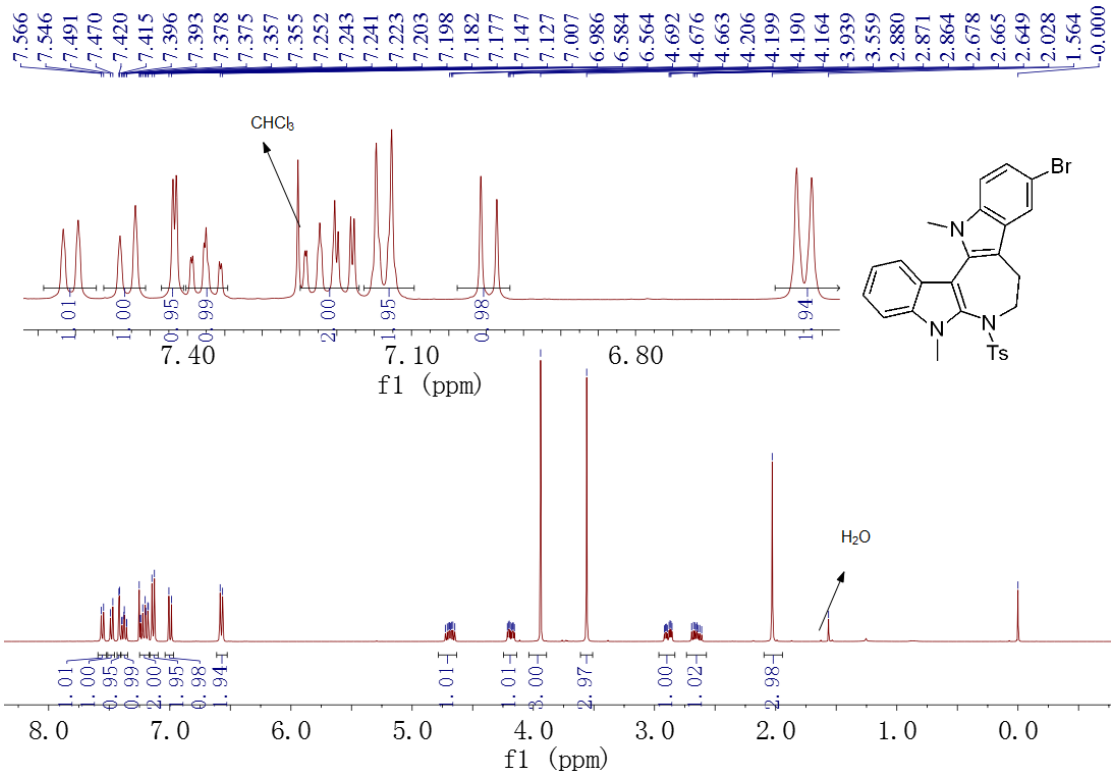


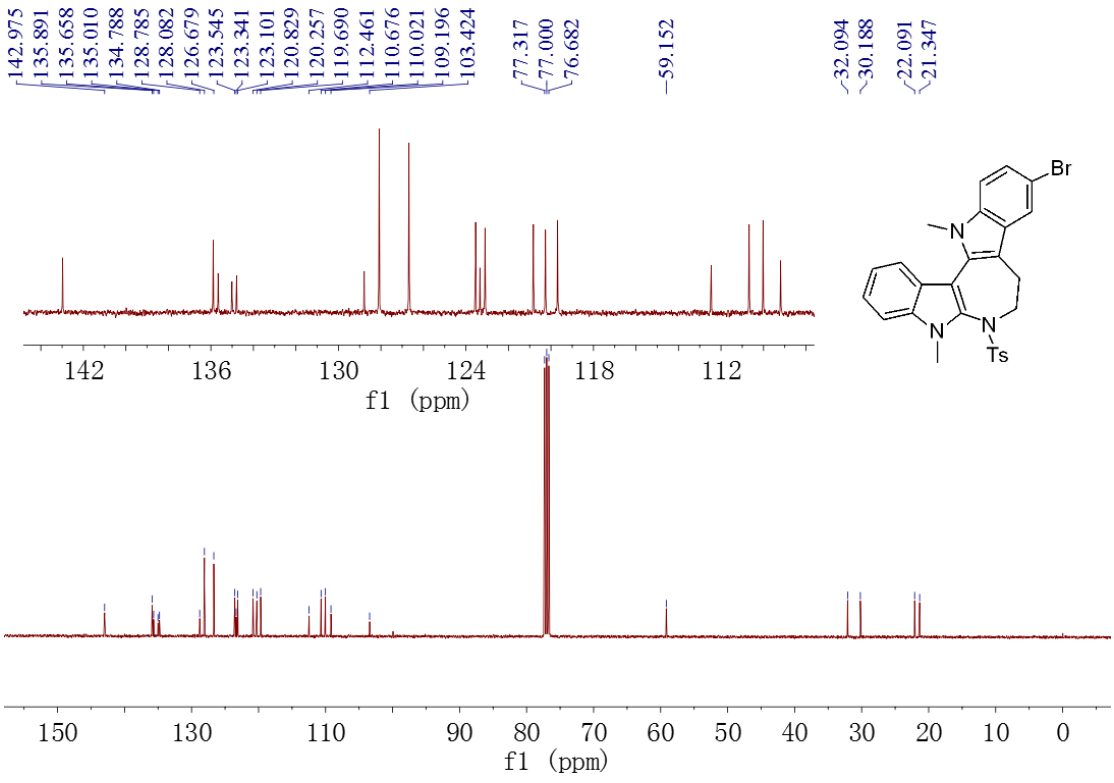
^{1}H NMR Spectrum for 3h ($CDCl_3$, 400 MHz)



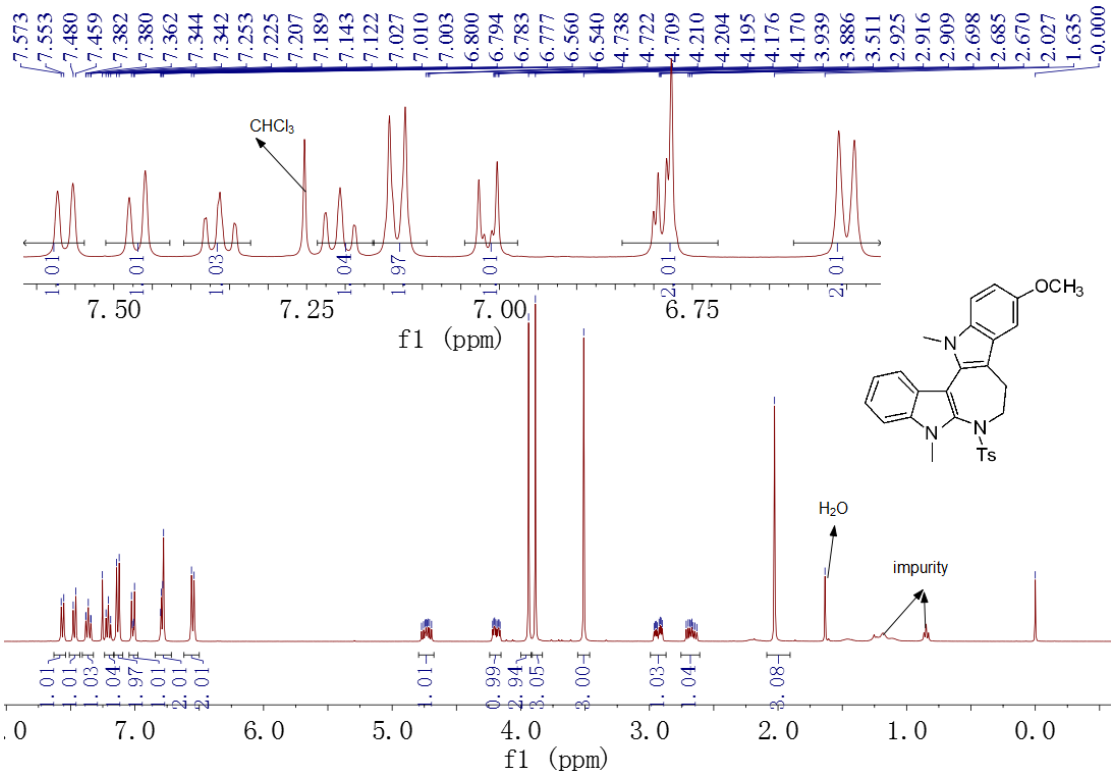
^{13}C NMR Spectrum for 3h ($CDCl_3$, 100 MHz)



^{1}H NMR Spectrum for 3i ($CDCl_3$, 400 MHz)



^{13}C NMR Spectrum for 3i ($CDCl_3$, 100 MHz)



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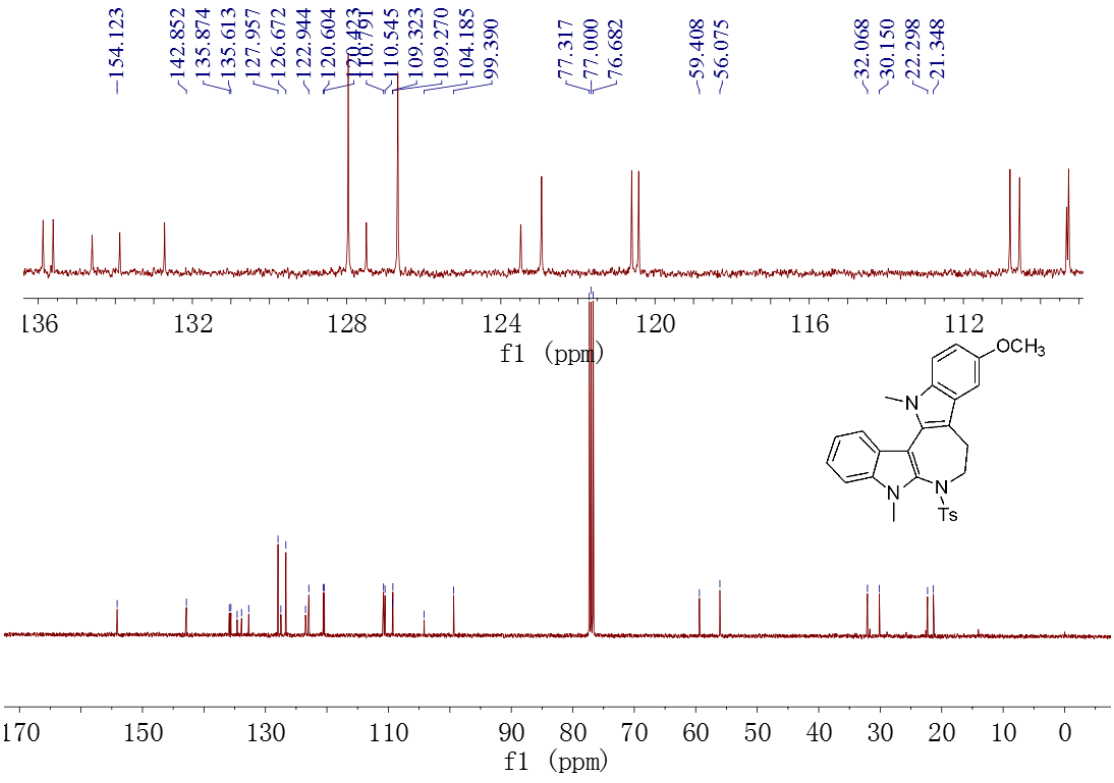
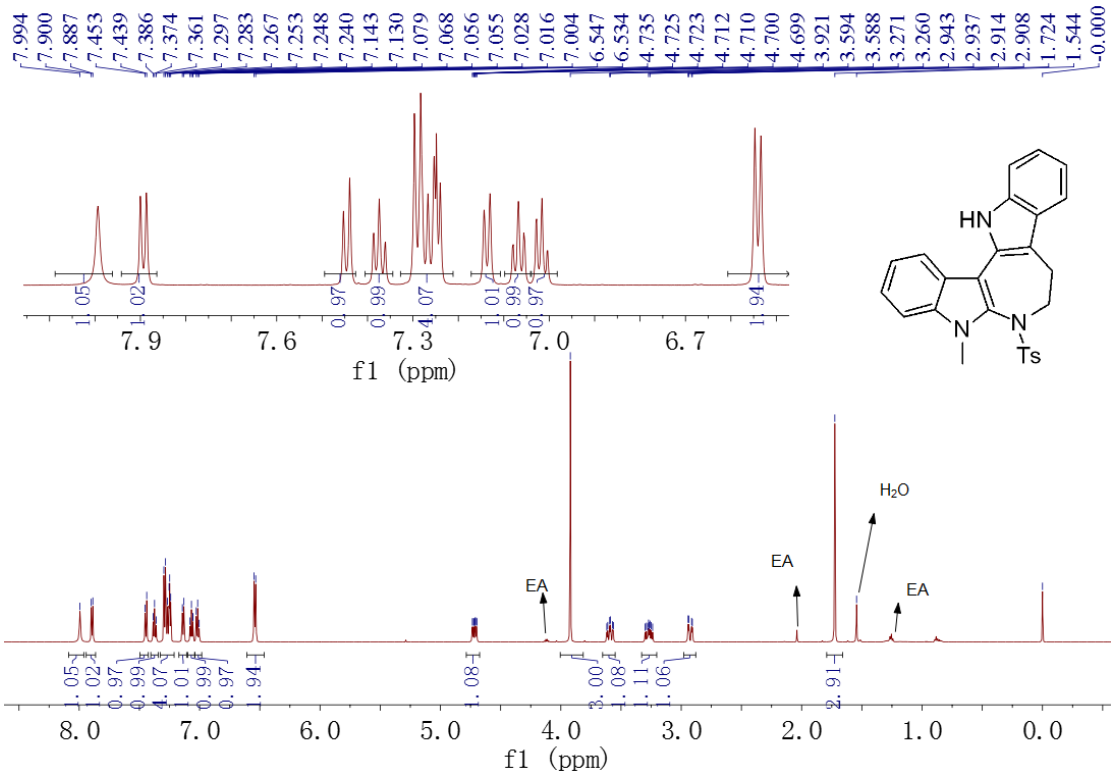
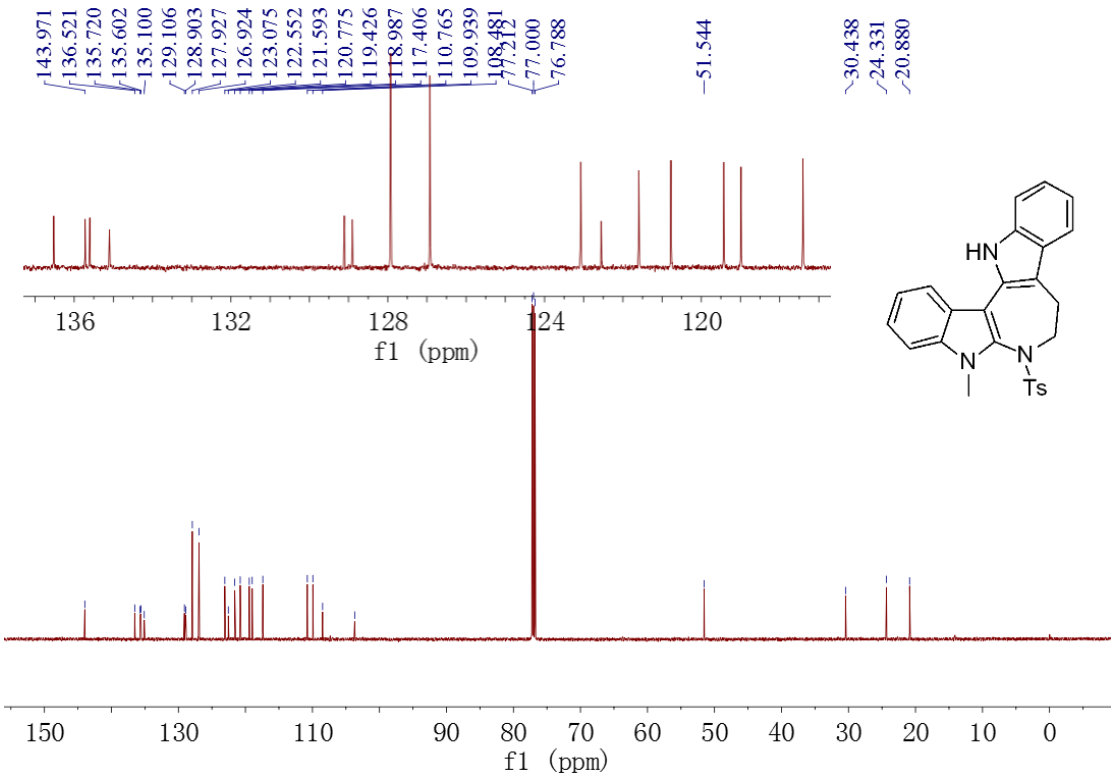


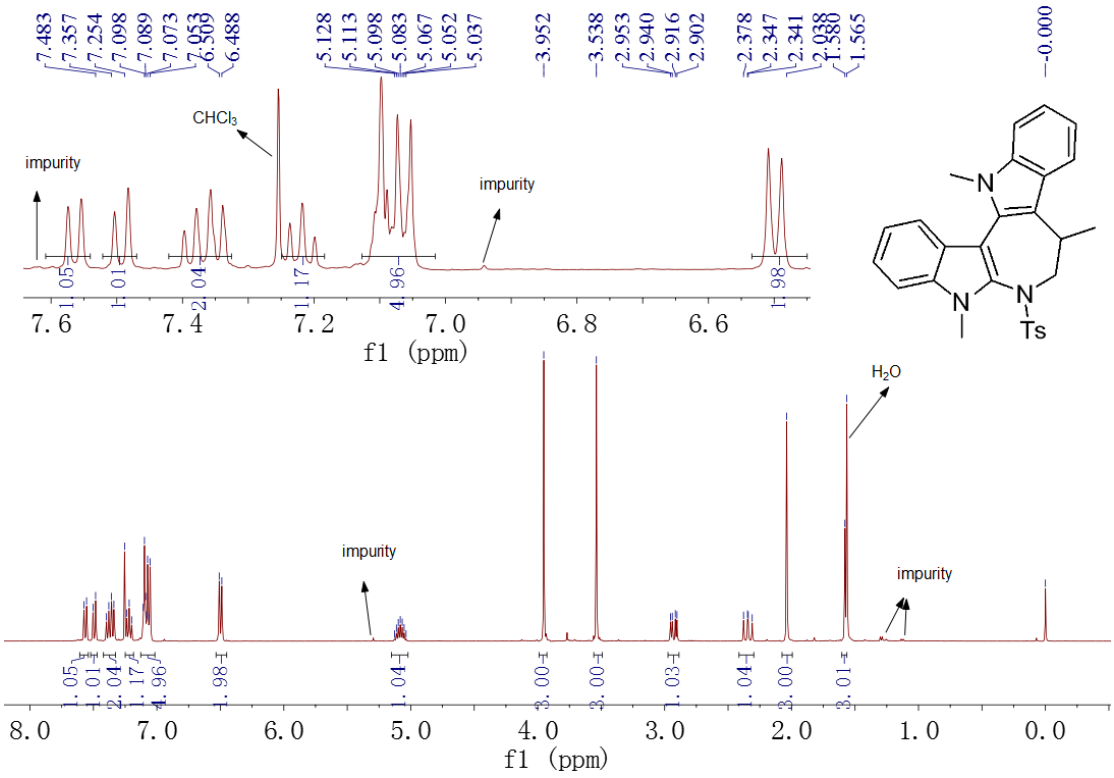
^{13}C NMR Spectrum for 3j ($CDCl_3$, 100 MHz)



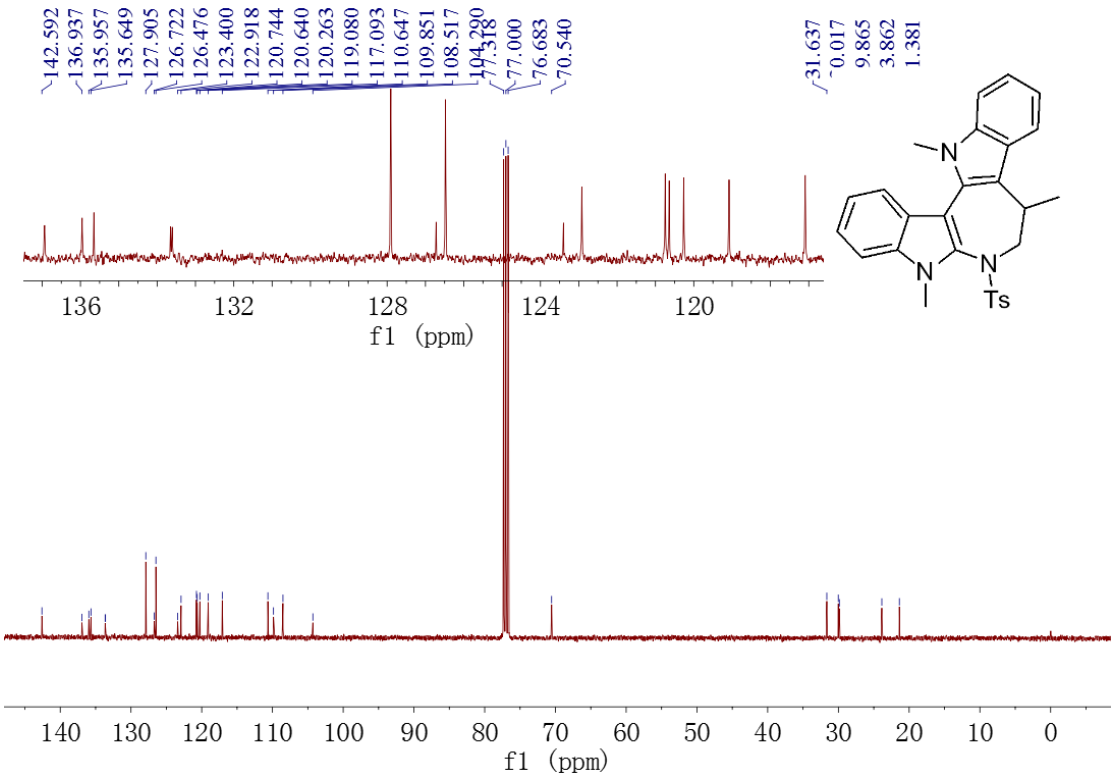
^{1}H NMR Spectrum for 3k ($CDCl_3$, 600 MHz)



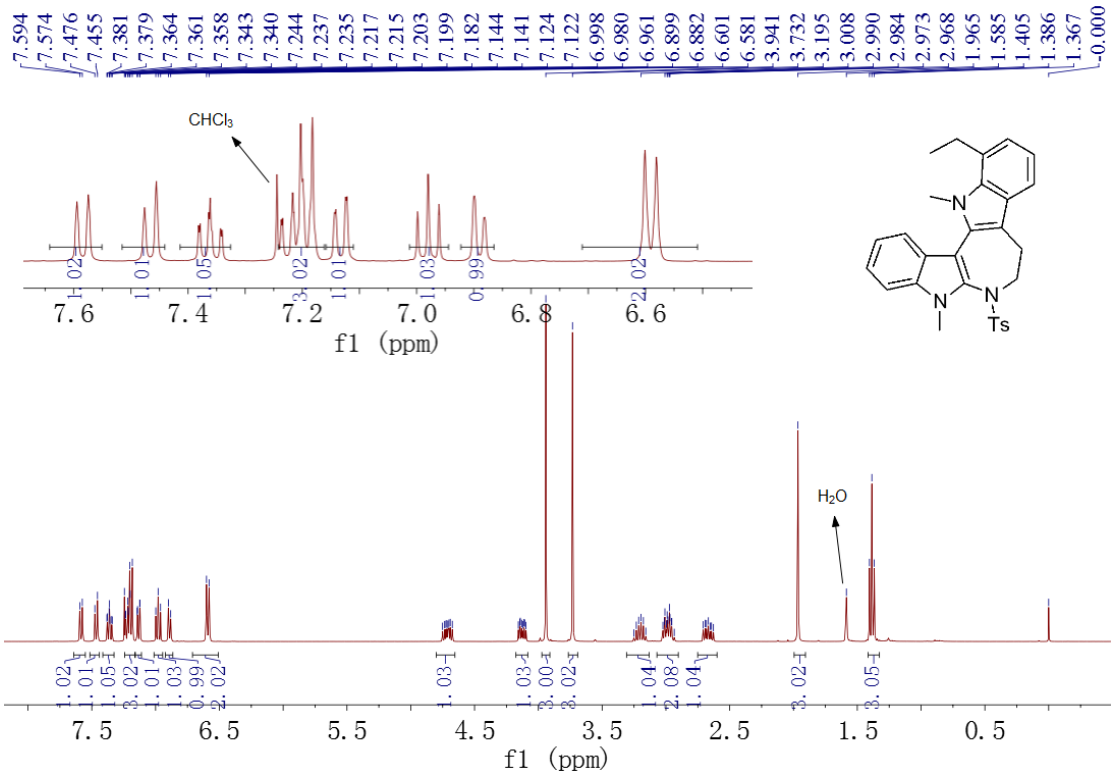
^{13}C NMR Spectrum for 3k ($CDCl_3$, 150 MHz)



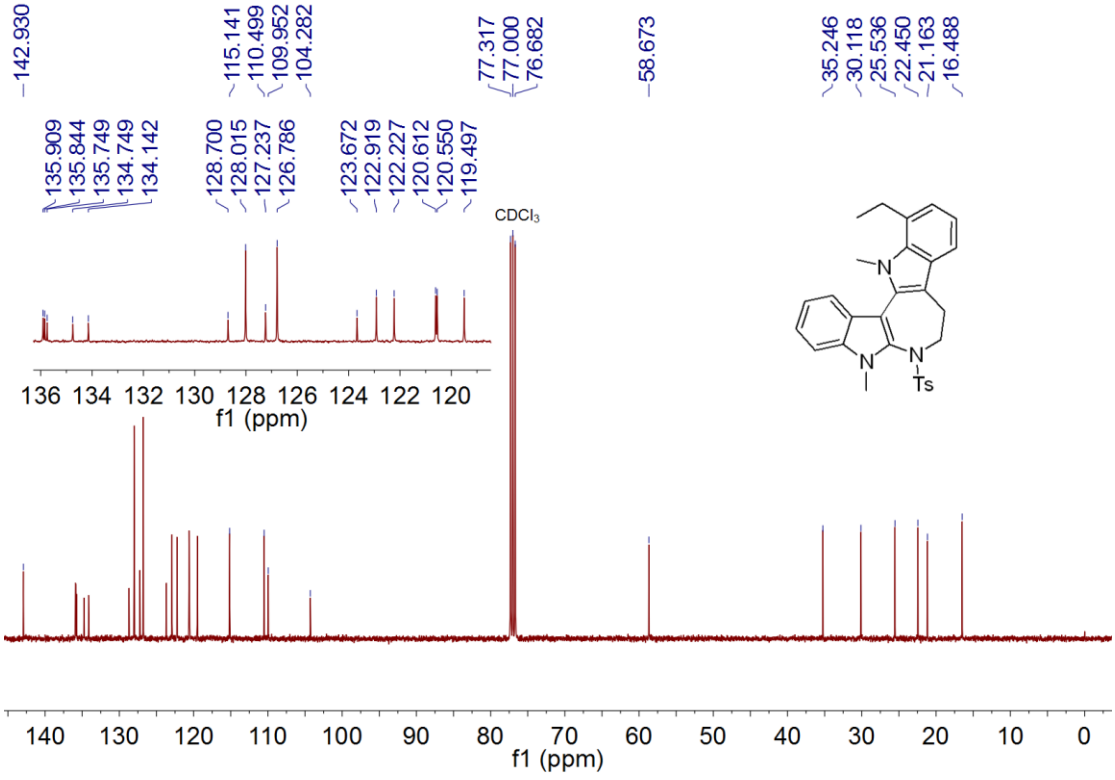
^{1}H NMR Spectrum for 3l ($CDCl_3$, 400 MHz)



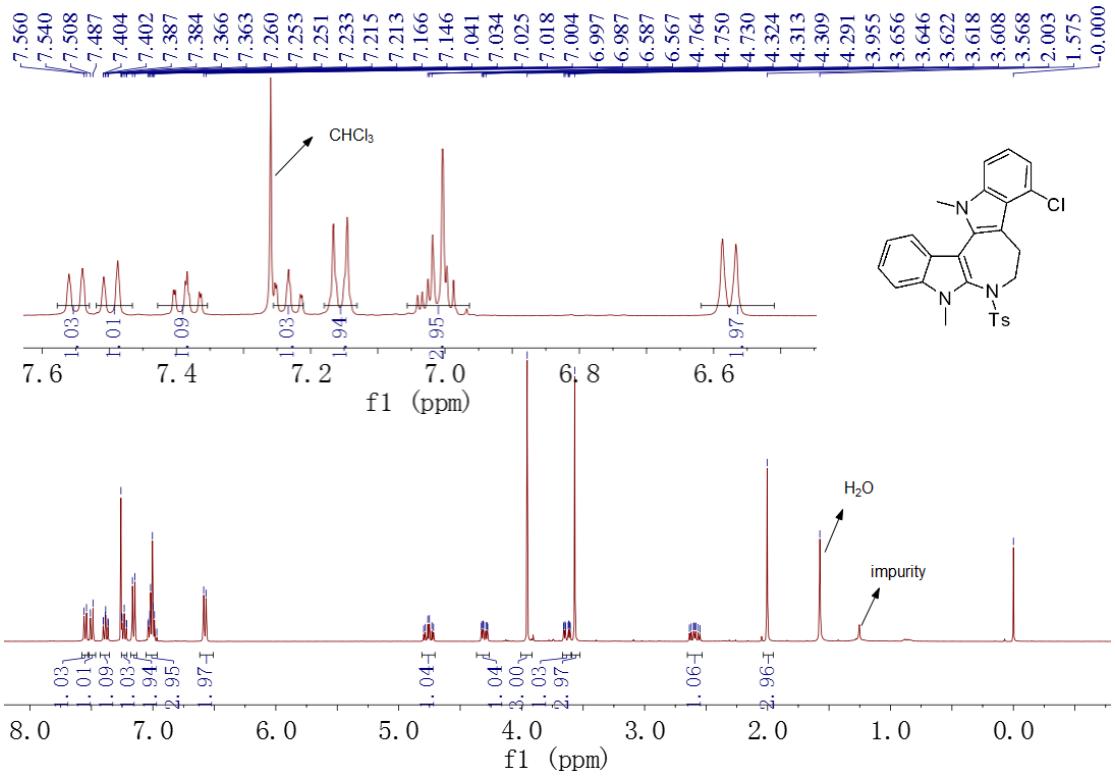
^{13}C NMR Spectrum for 3l ($CDCl_3$, 100 MHz)



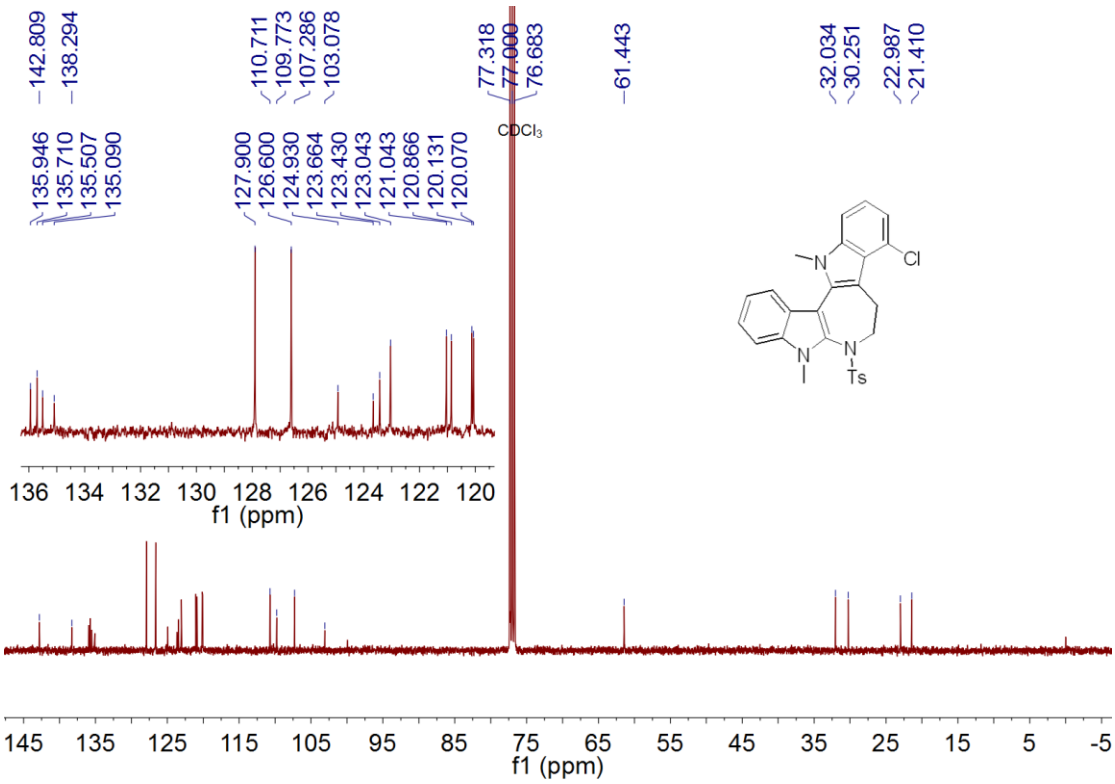
^{1}H NMR Spectrum for 3m ($CDCl_3$, 400 MHz)



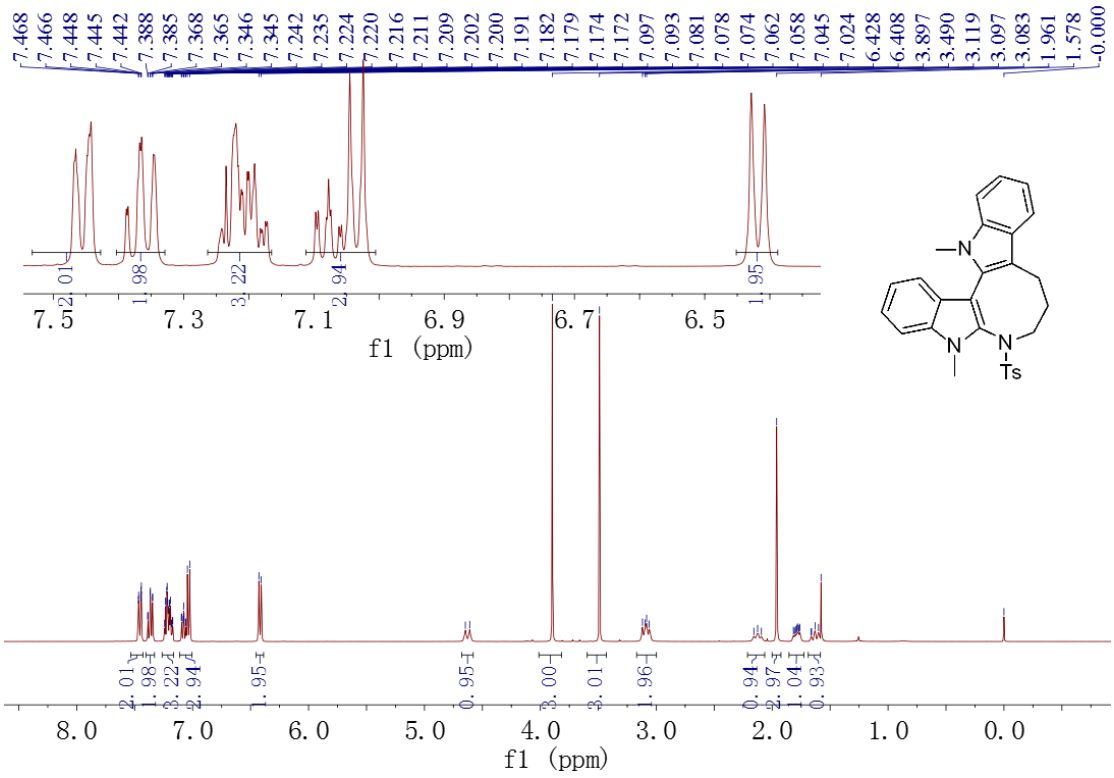
^{13}C NMR Spectrum for 3m ($CDCl_3$, 100 MHz)



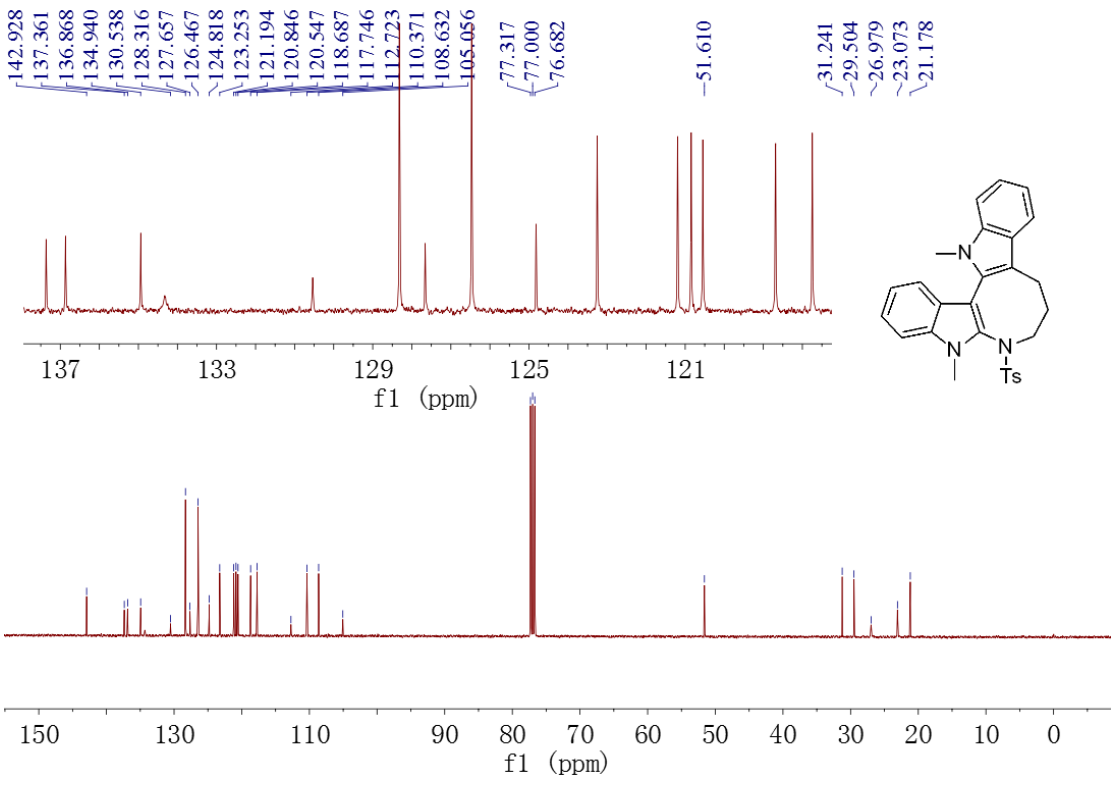
^{1}H NMR Spectrum for 3n ($CDCl_3$, 400 MHz)



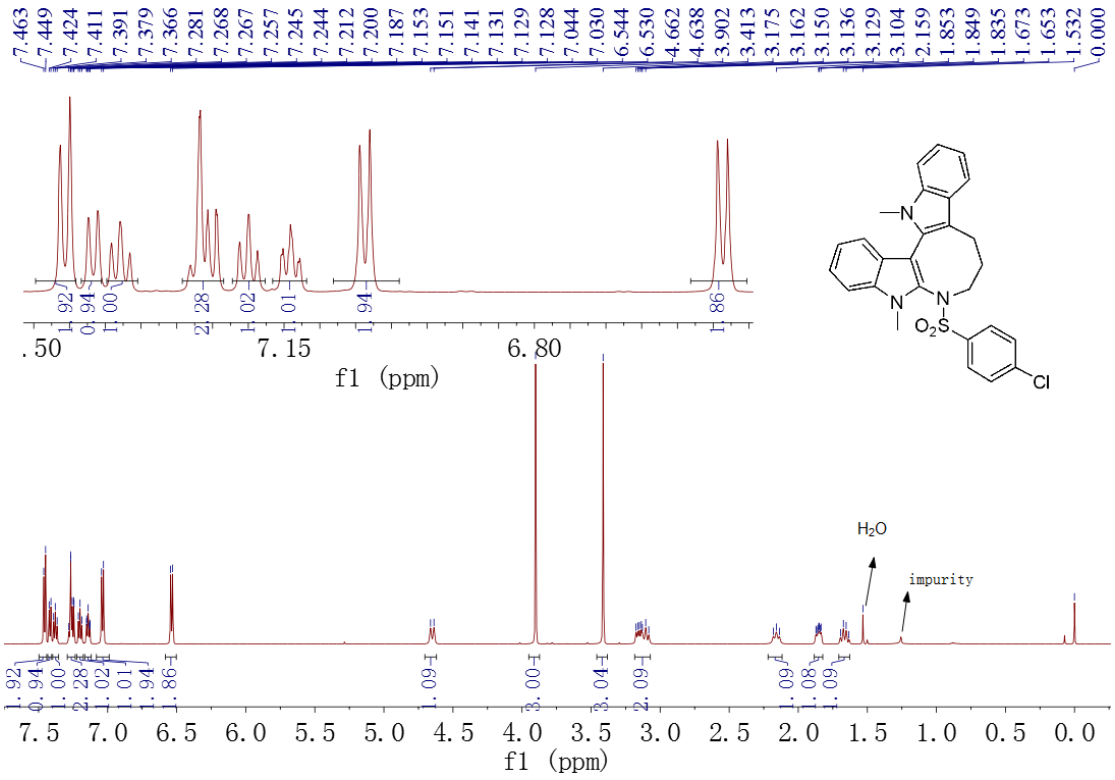
^{13}C NMR Spectrum for 3n ($CDCl_3$, 100 MHz)



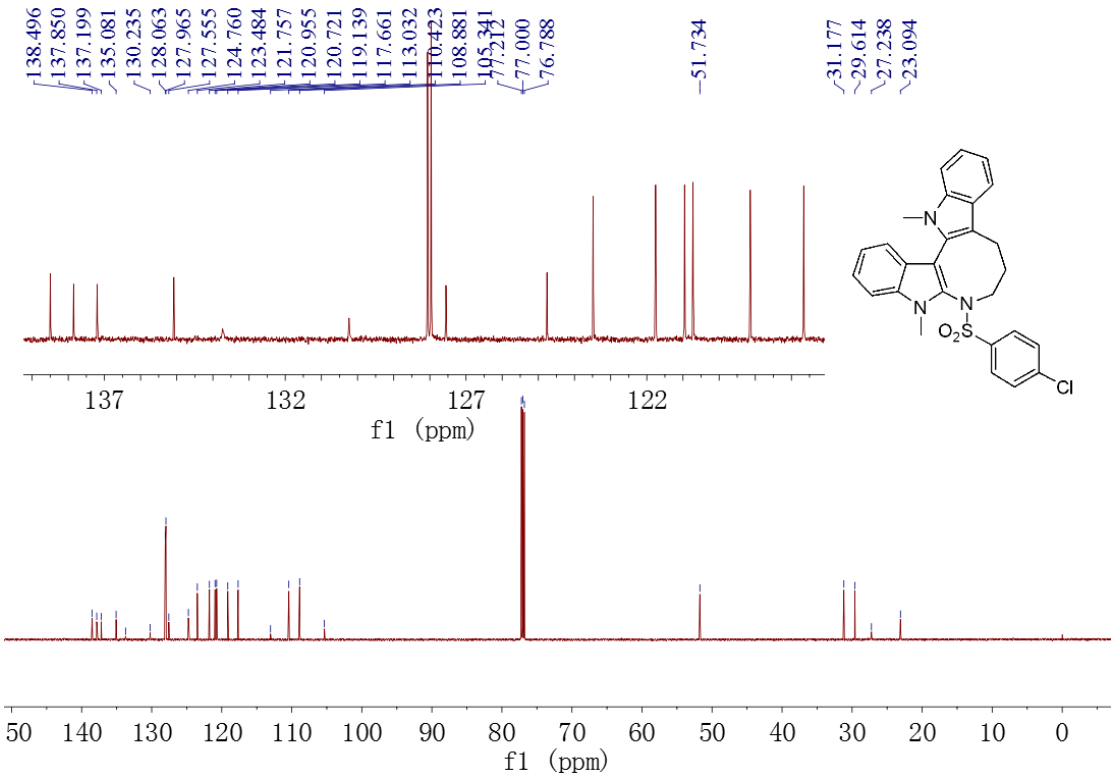
^{1}H NMR Spectrum for 5a ($CDCl_3$, 400 MHz)



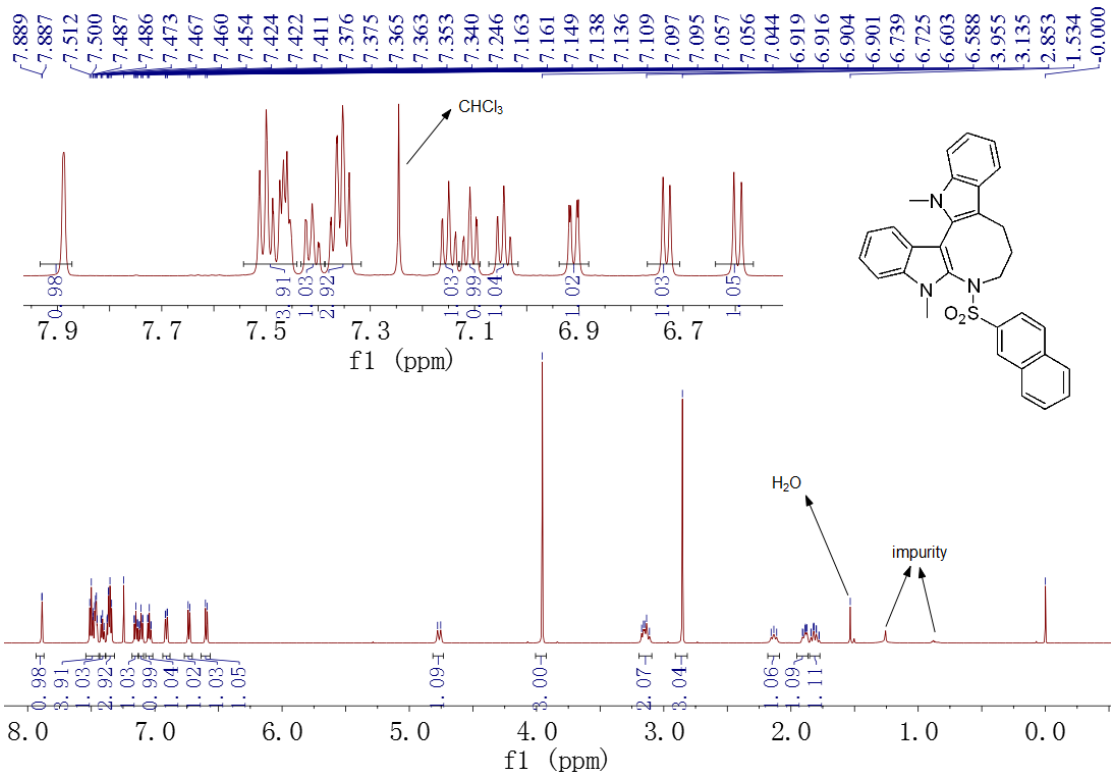
^{13}C NMR Spectrum for 5a ($CDCl_3$, 100 MHz)



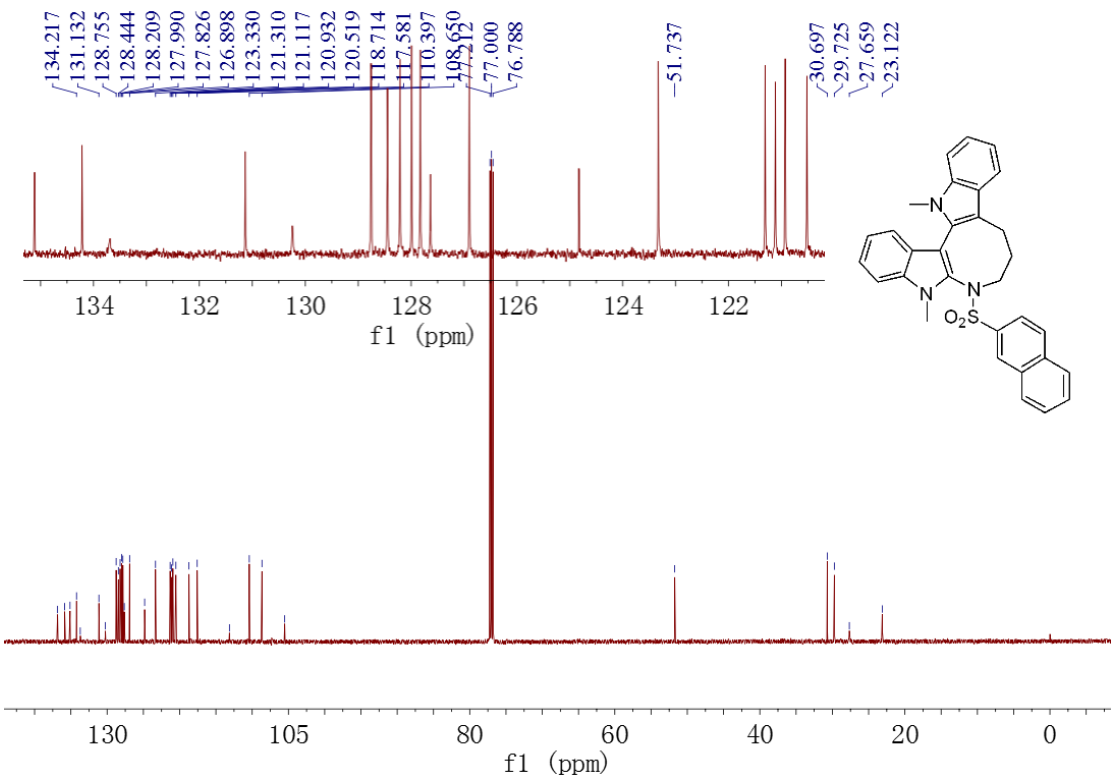
^{1}H NMR Spectrum for 5b ($CDCl_3$, 600 MHz)



^{13}C NMR Spectrum for 5b ($CDCl_3$, 150 MHz)



^{1}H NMR Spectrum for 5c ($CDCl_3$, 600 MHz)



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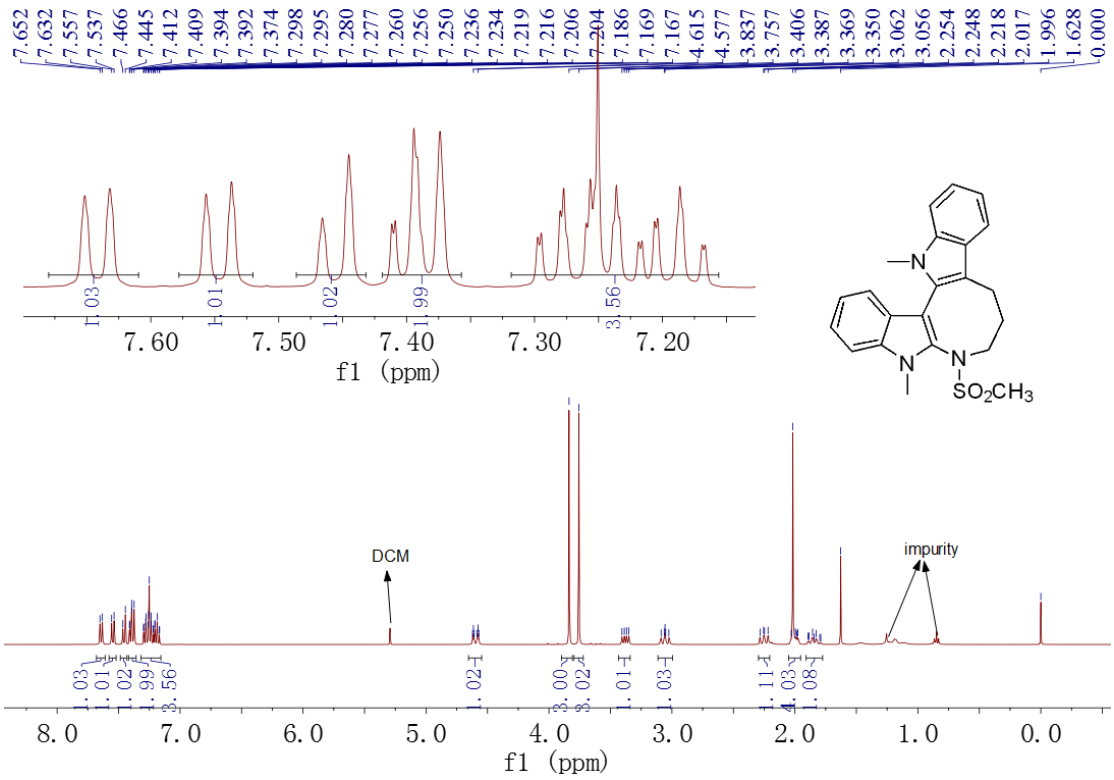

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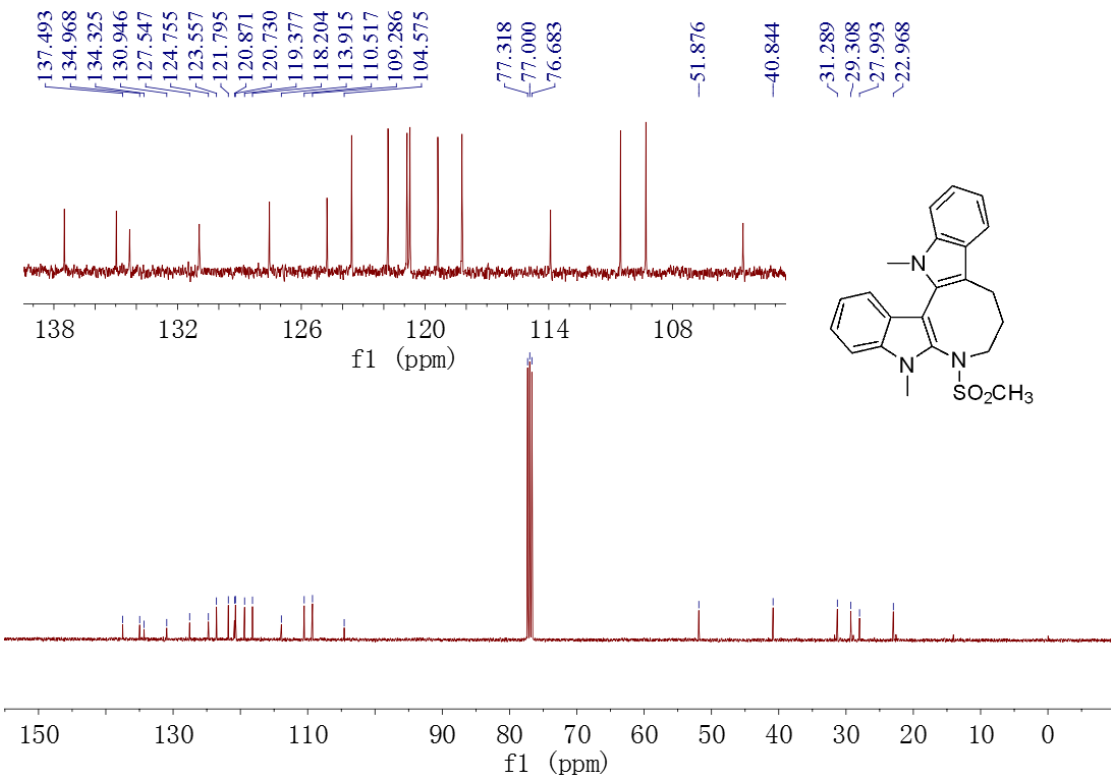
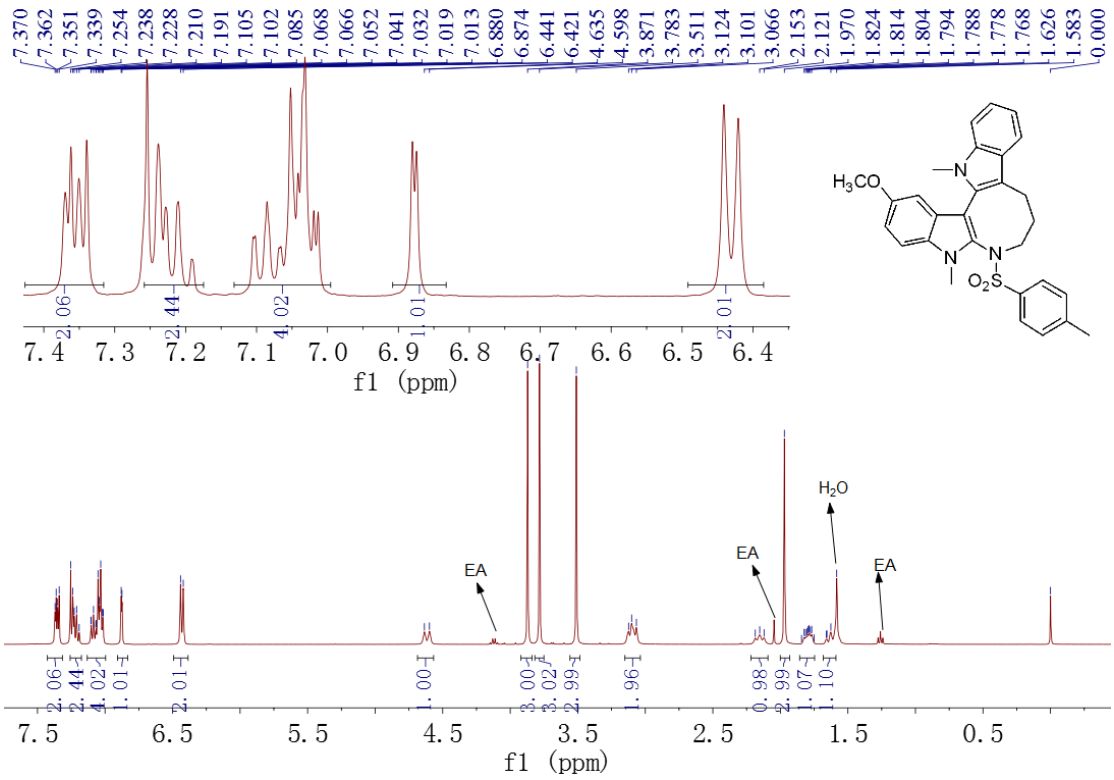
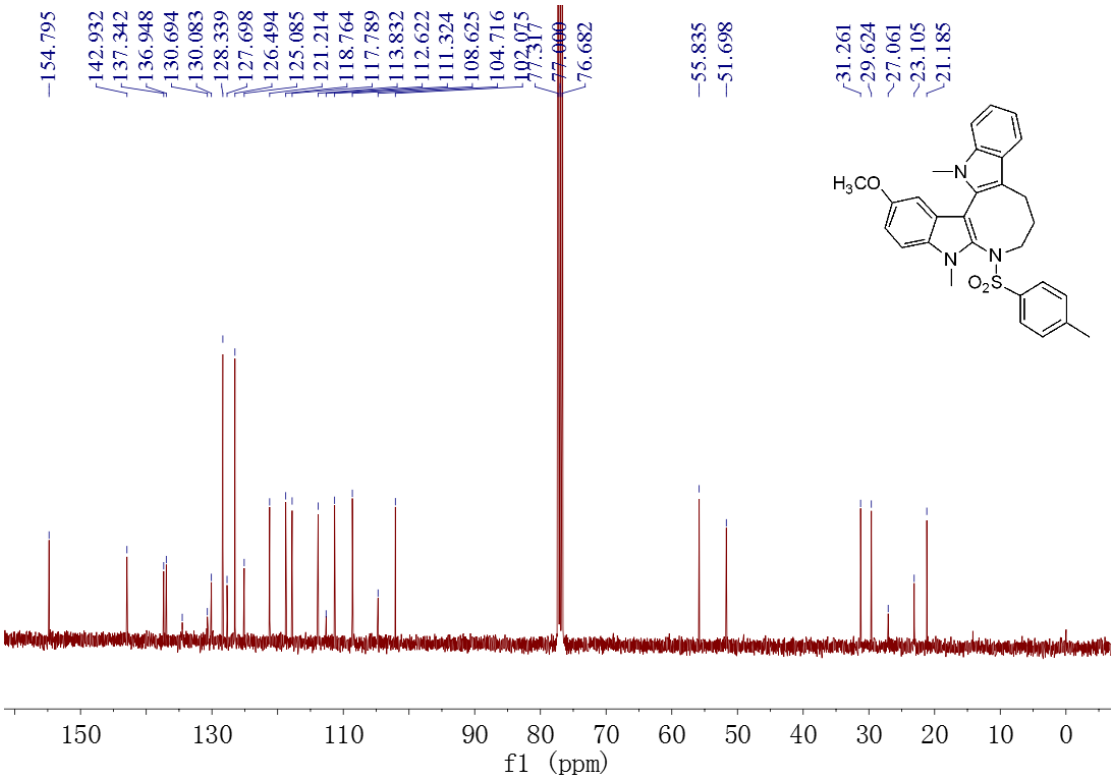


^{13}C NMR Spectrum for 5d ($CDCl_3$, 100 MHz)



^{1}H NMR Spectrum for 5e ($CDCl_3$, 400 MHz)



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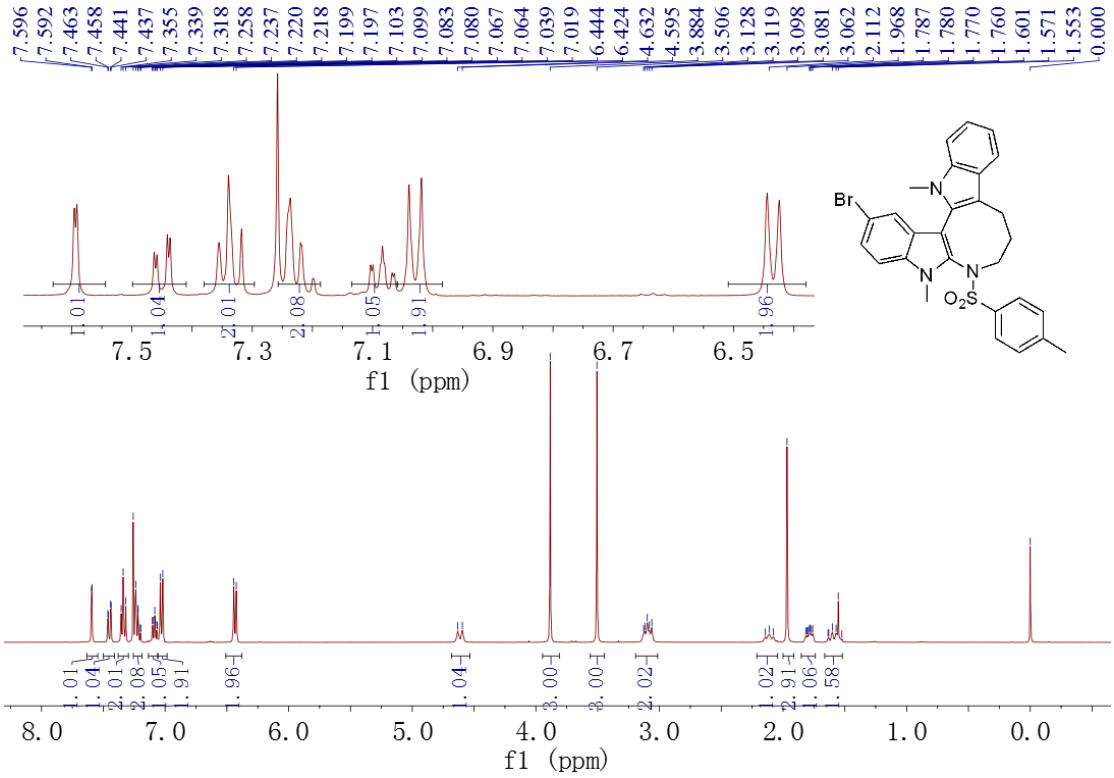
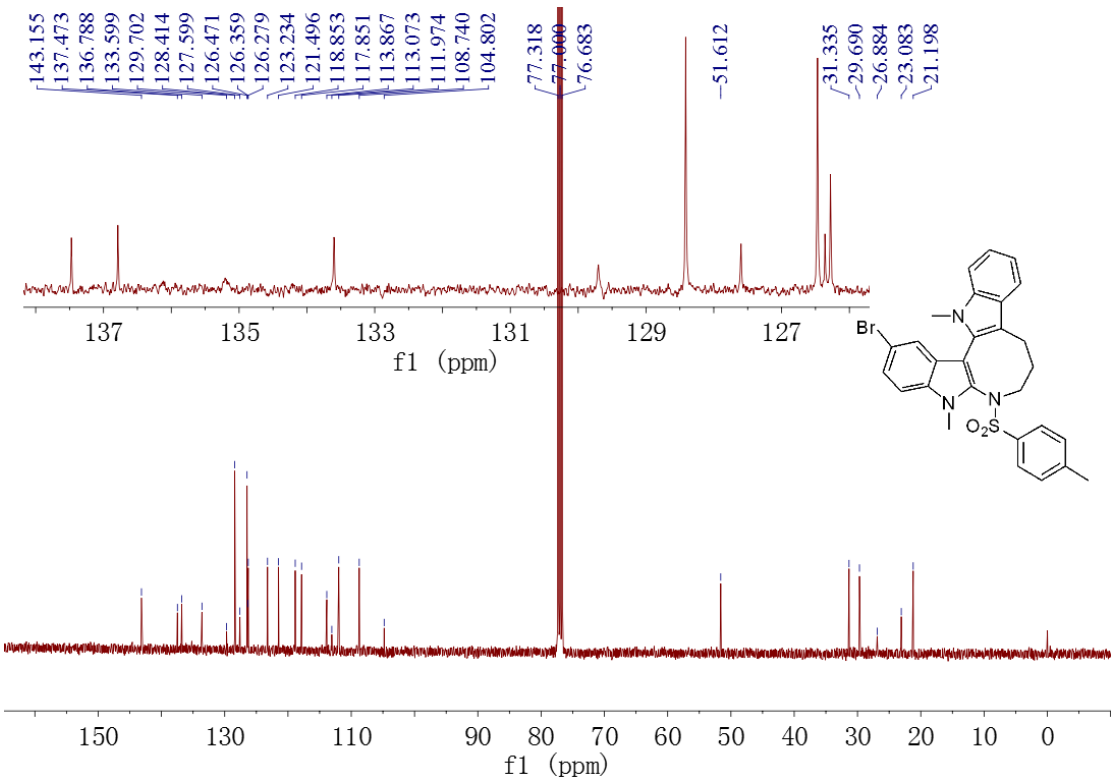


^{1}H NMR Spectrum for 5f ($CDCl_3$, 400 MHz)



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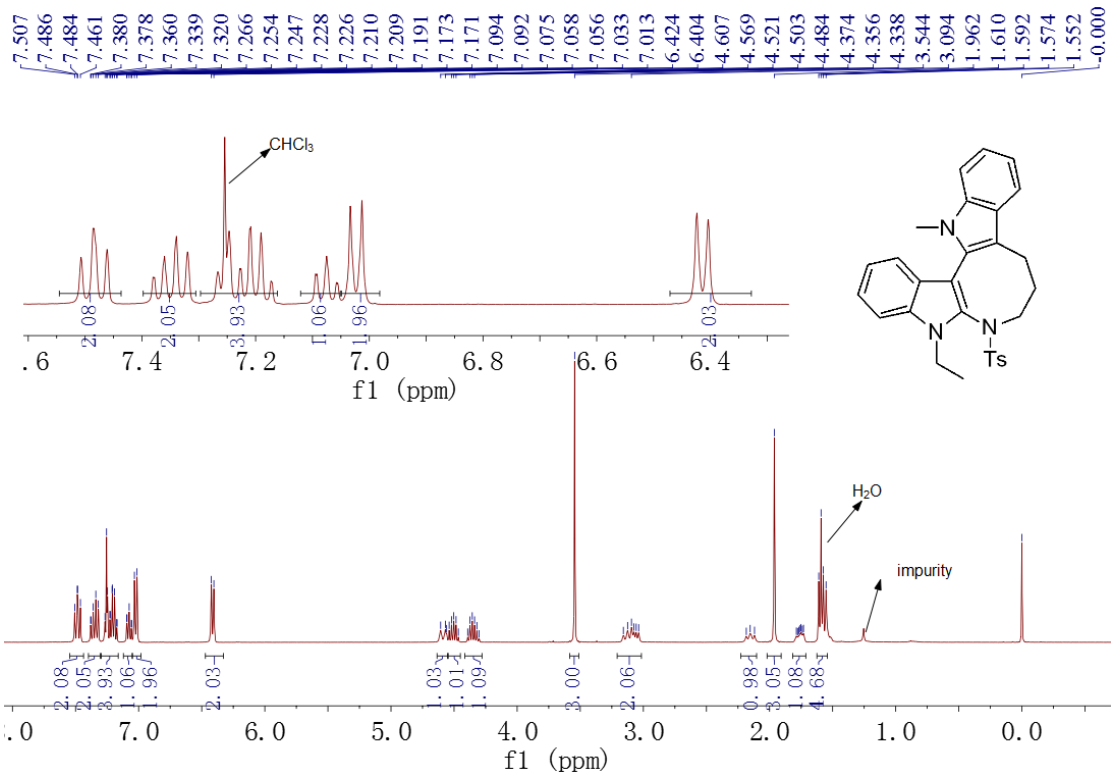
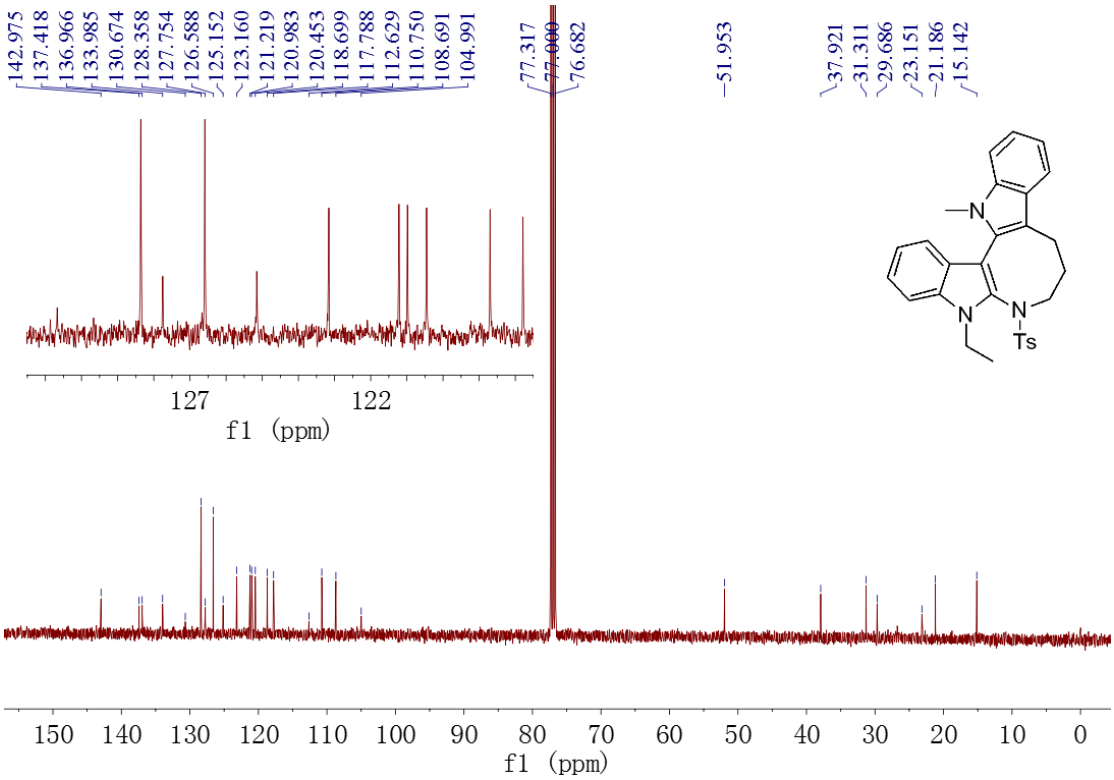
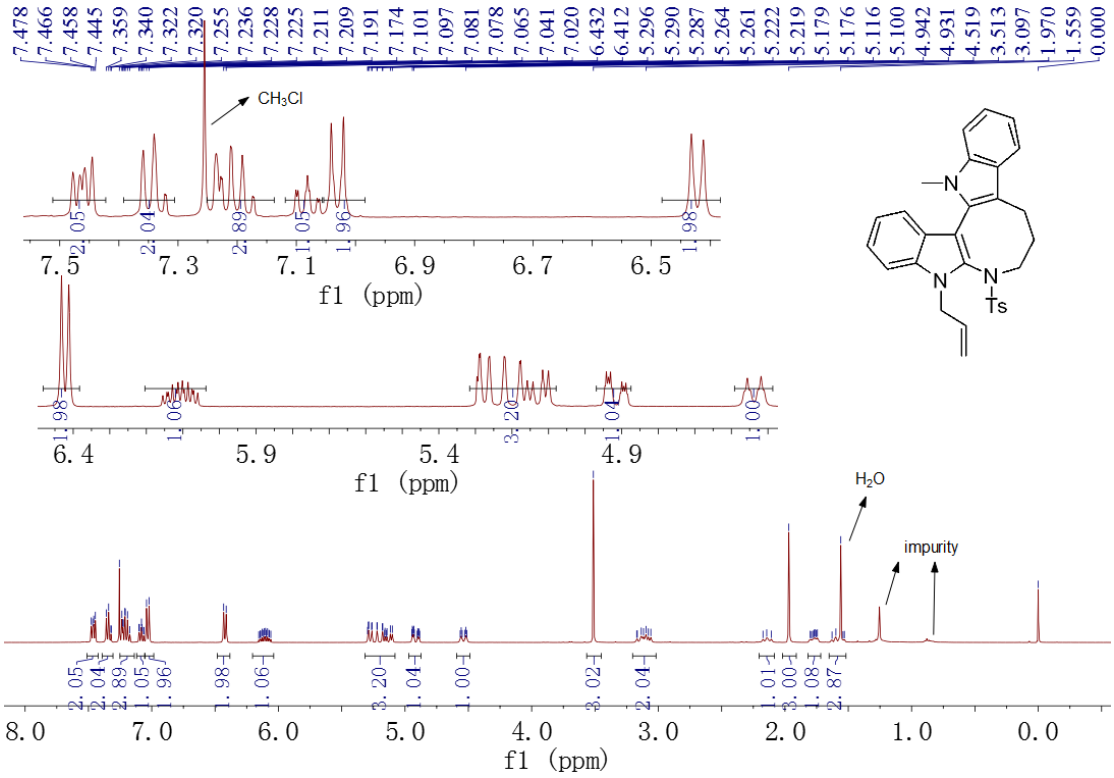


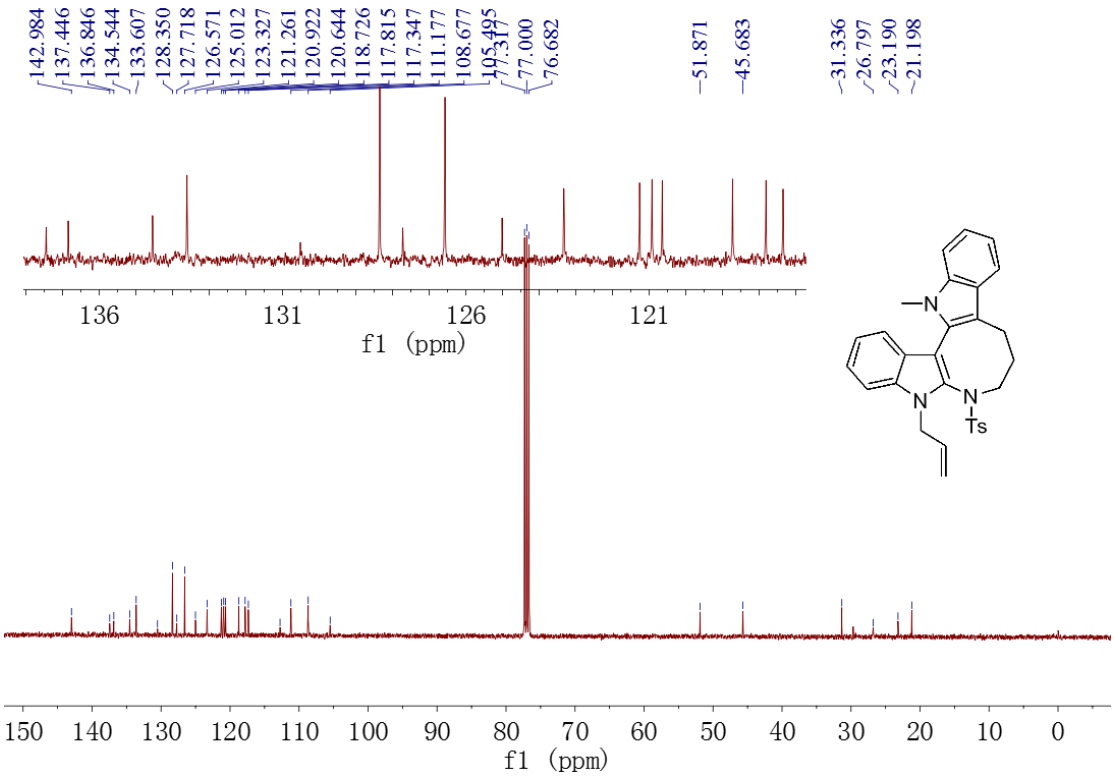
^{1}H NMR Spectrum for 5g ($CDCl_3$, 400 MHz)



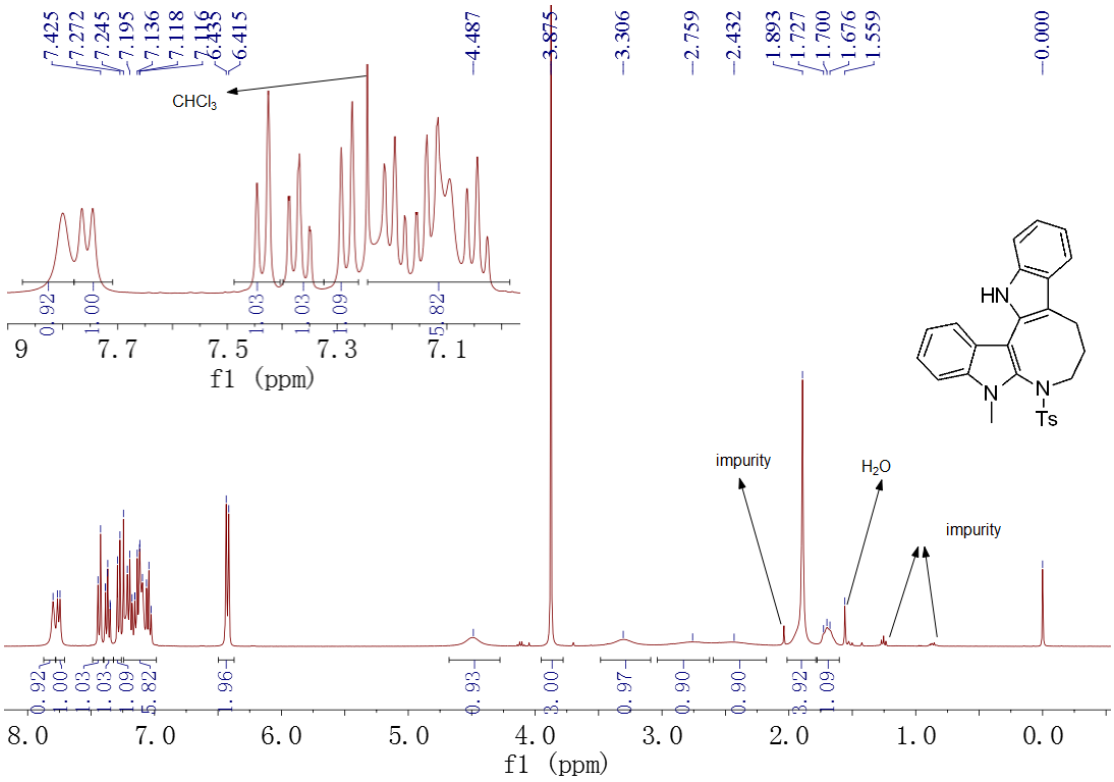
^{13}C NMR Spectrum for 5g ($CDCl_3$, 100 MHz)



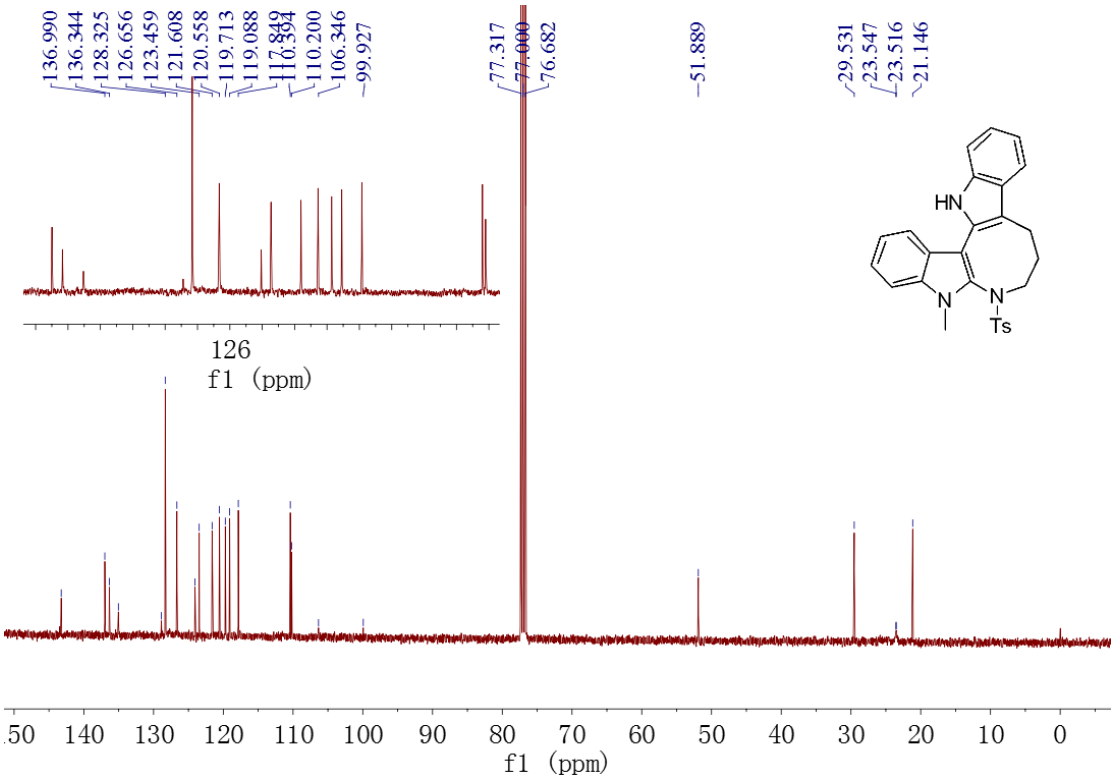
^{1}H NMR Spectrum for 5h ($CDCl_3$, 400 MHz)



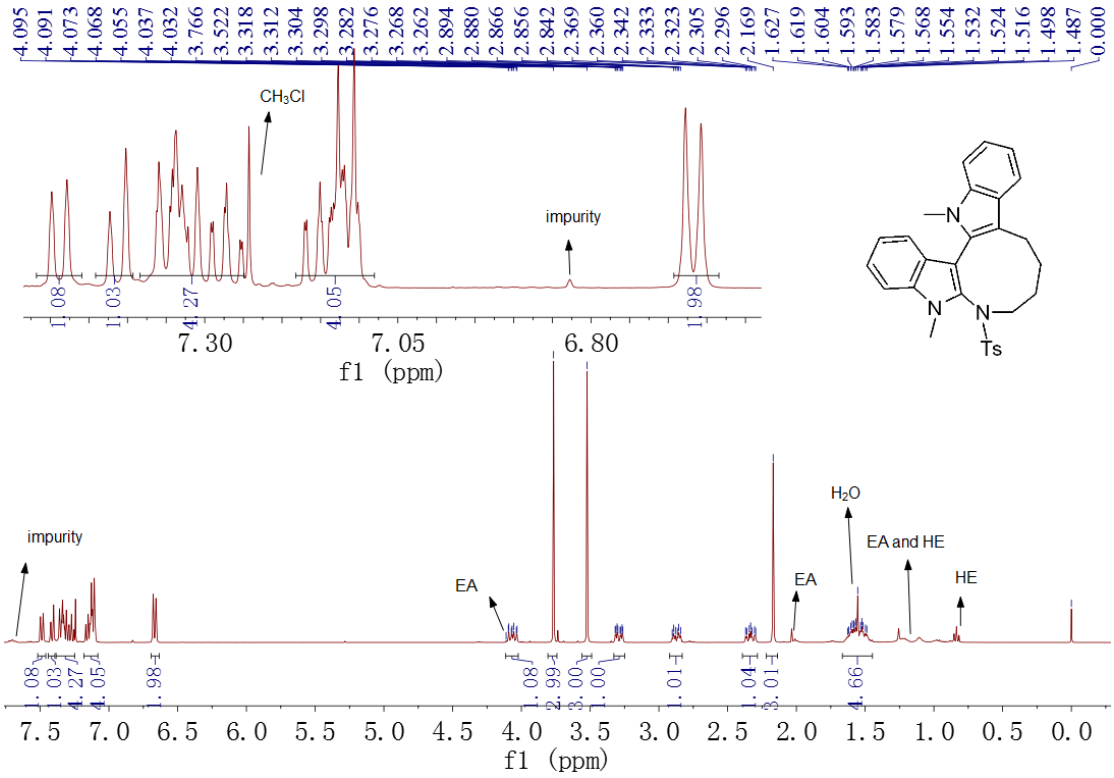
^{13}C NMR Spectrum for 5h ($CDCl_3$, 100 MHz)



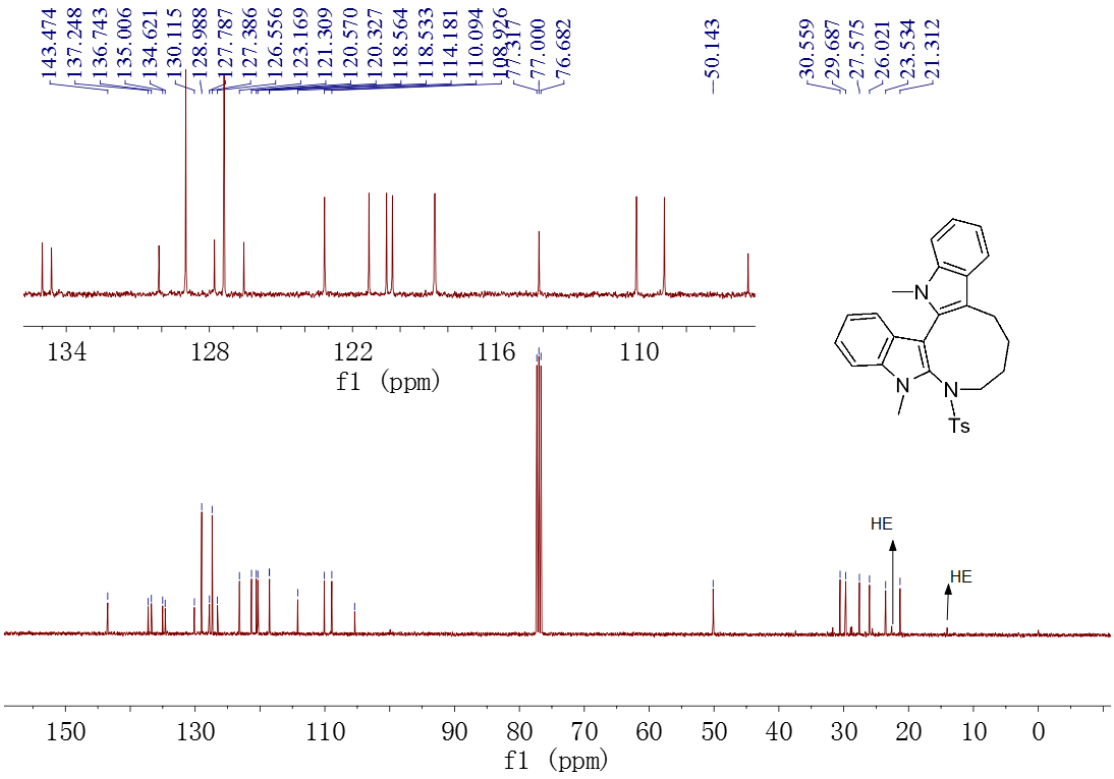
^{1}H NMR Spectrum for 5i ($CDCl_3$, 400 MHz)



^{13}C NMR Spectrum for 5i ($CDCl_3$, 100 MHz)



^{1}H NMR Spectrum for 7a ($CDCl_3$, 400 MHz)



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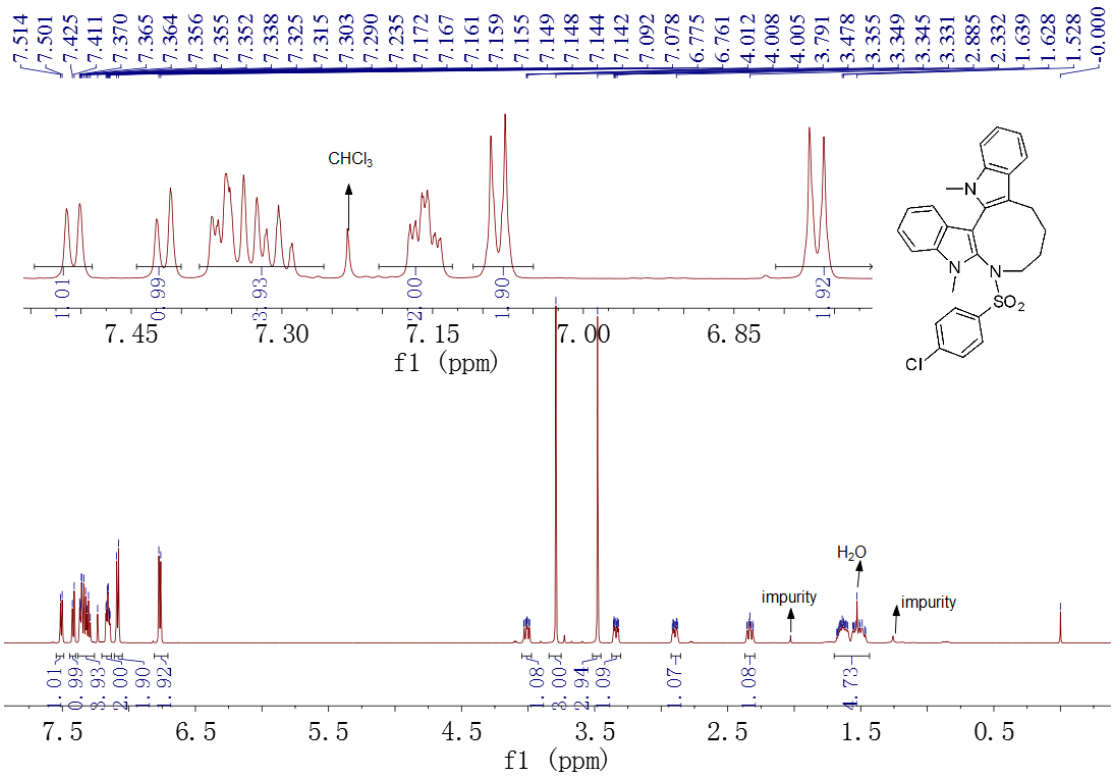
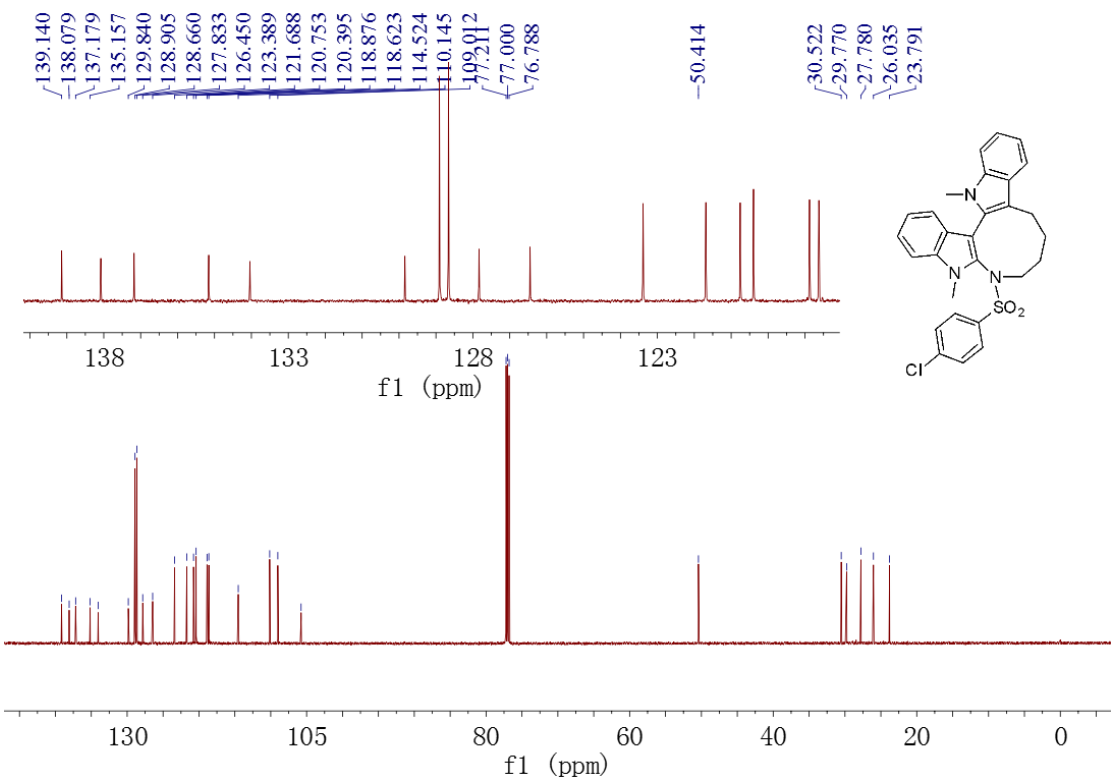
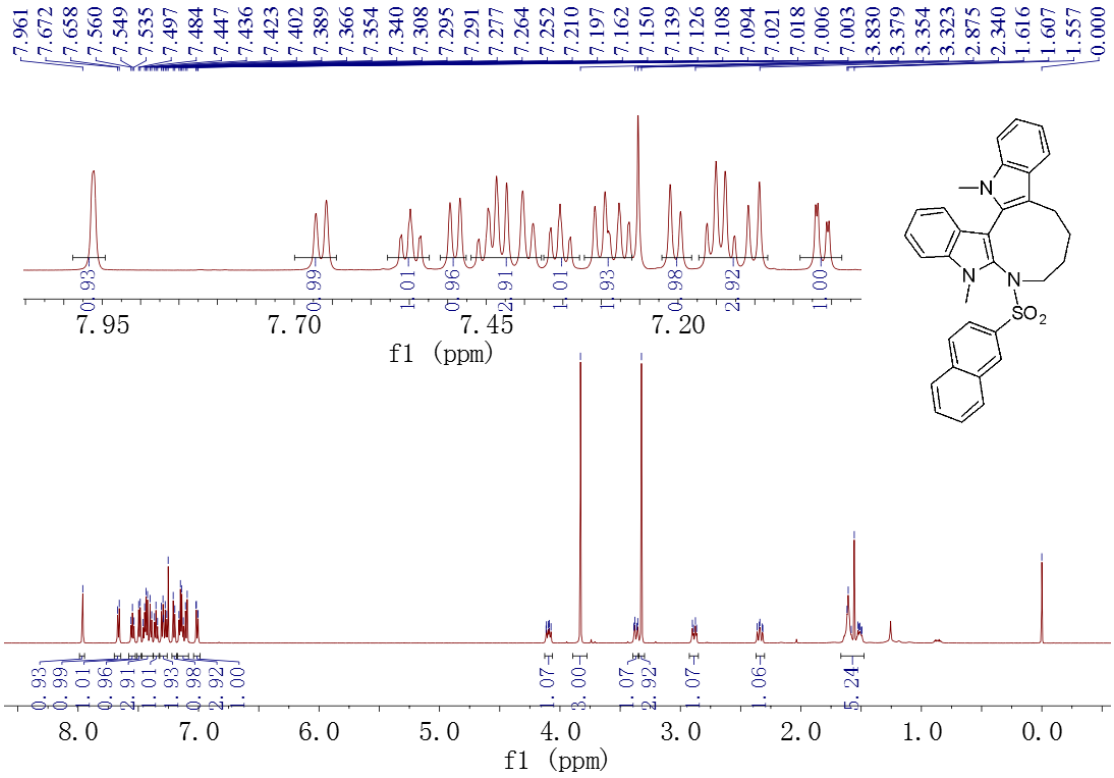


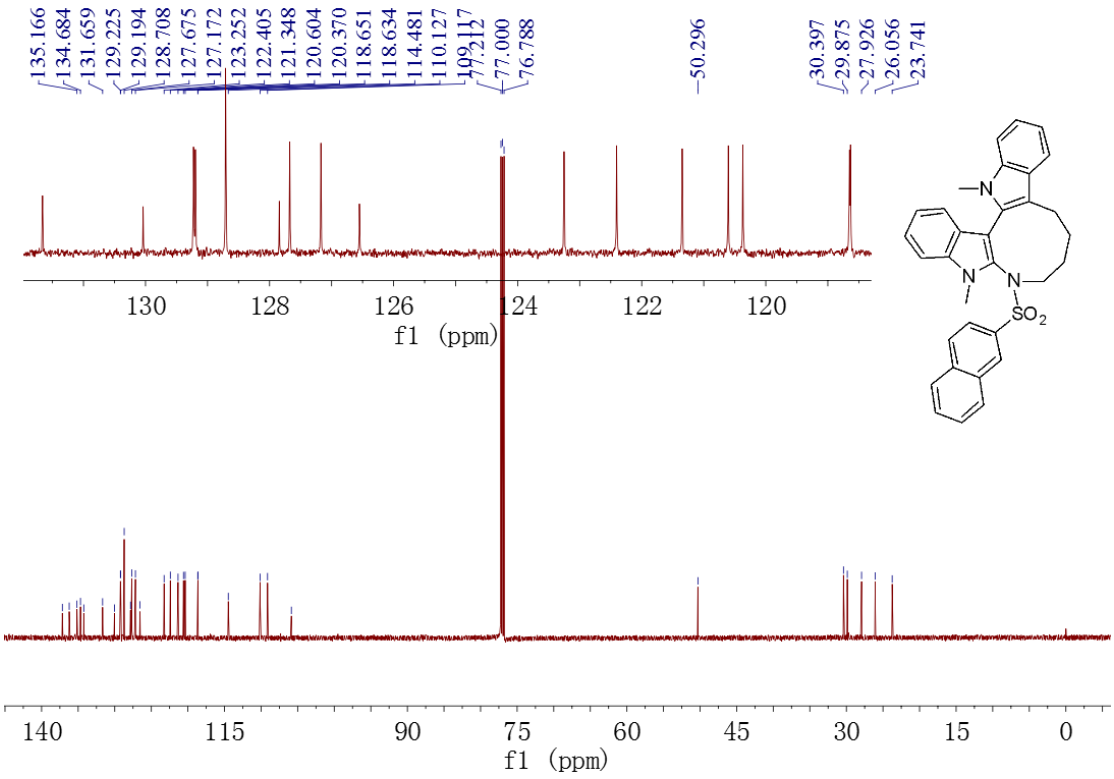
^{1}H NMR Spectrum for 7b ($CDCl_3$, 600 MHz)



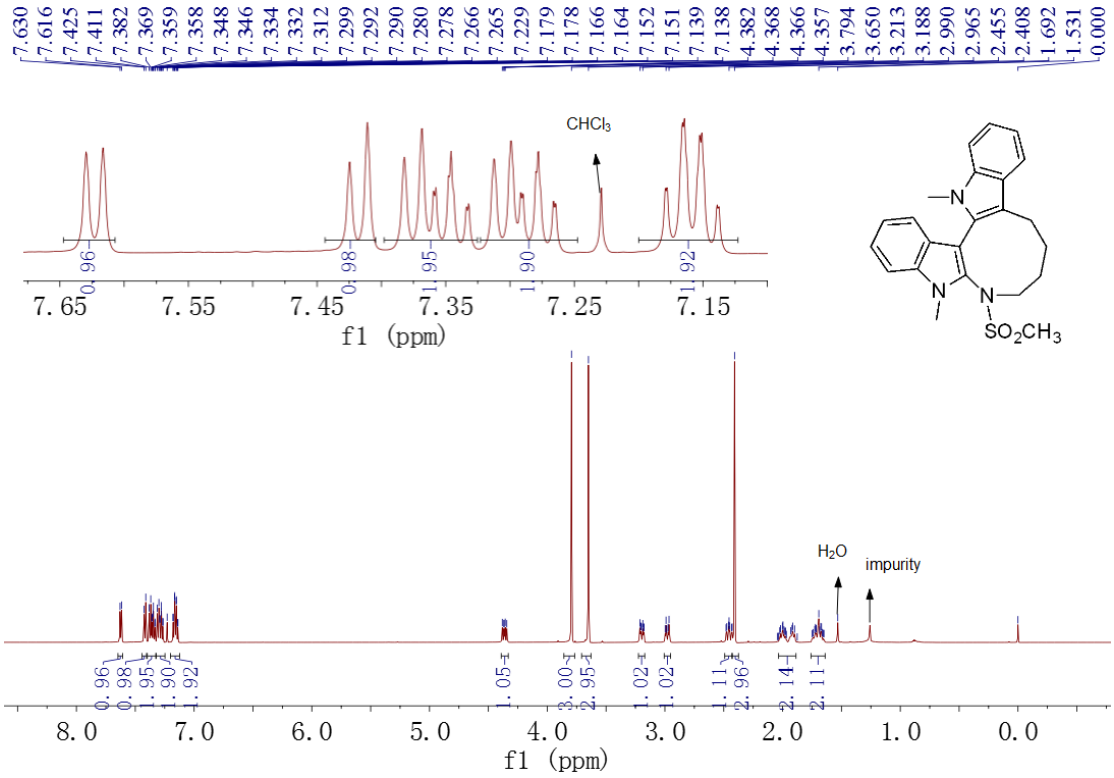
^{13}C NMR Spectrum for 7b ($CDCl_3$, 150 MHz)



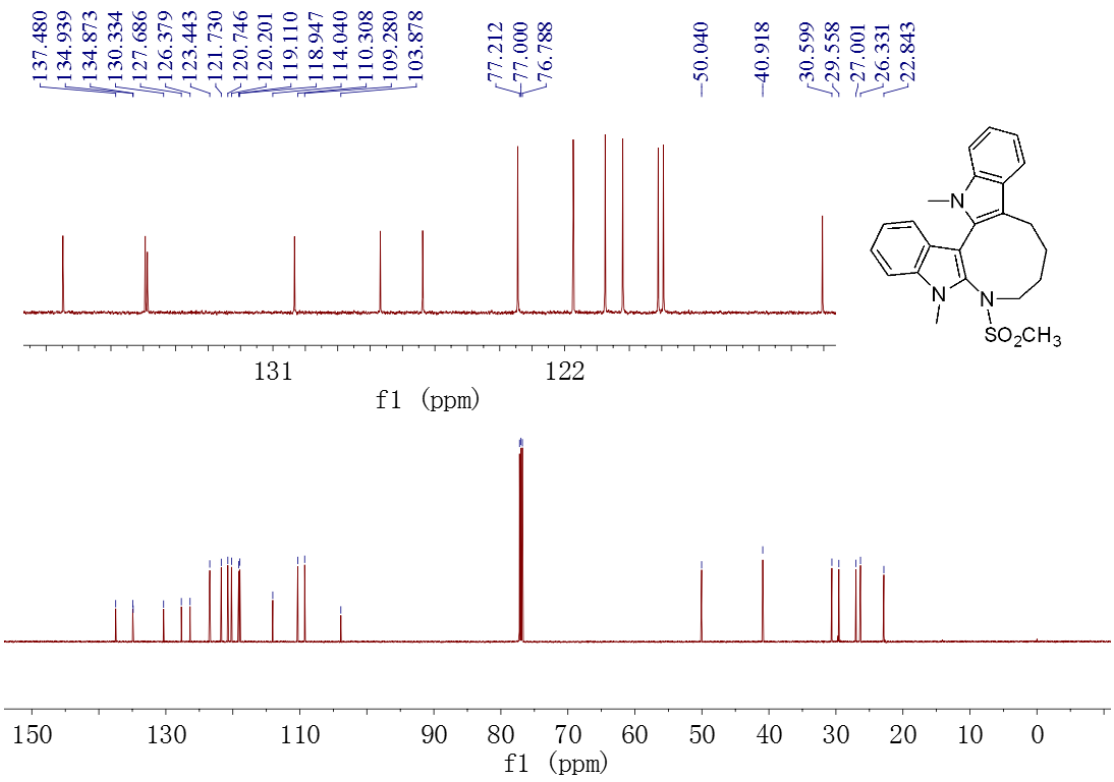
^{1}H NMR Spectrum for 7c ($CDCl_3$, 600 MHz)



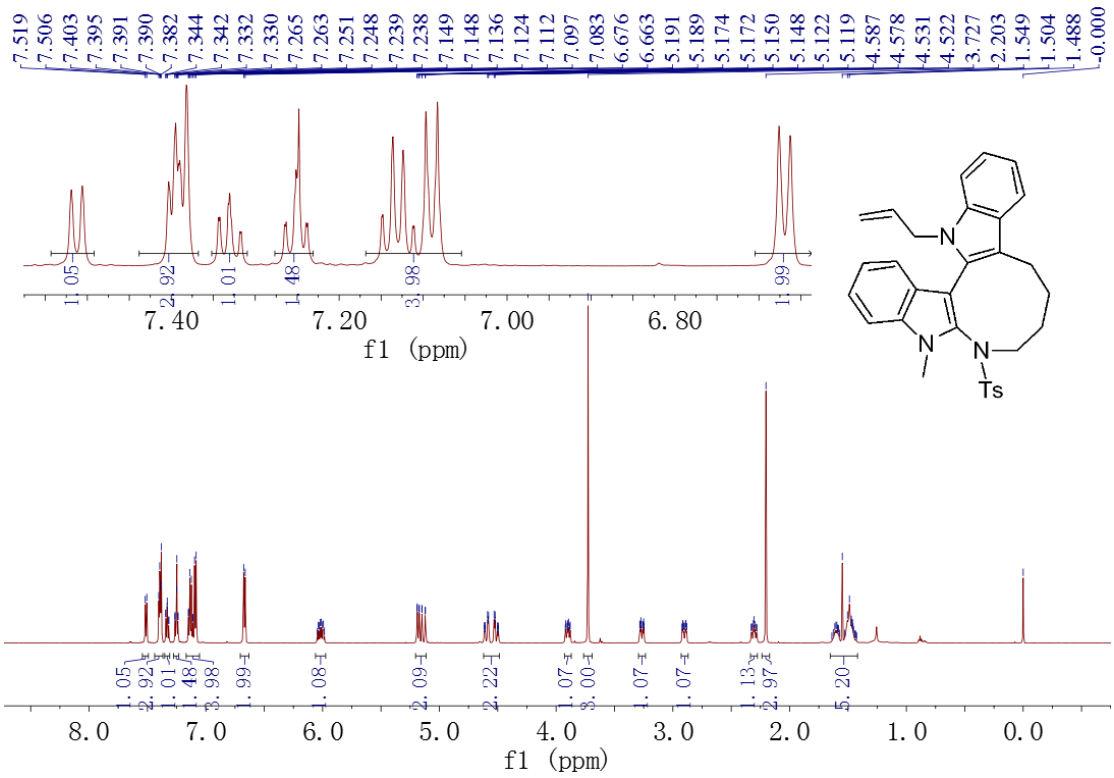
^{13}C NMR Spectrum for 7c ($CDCl_3$, 150 MHz)



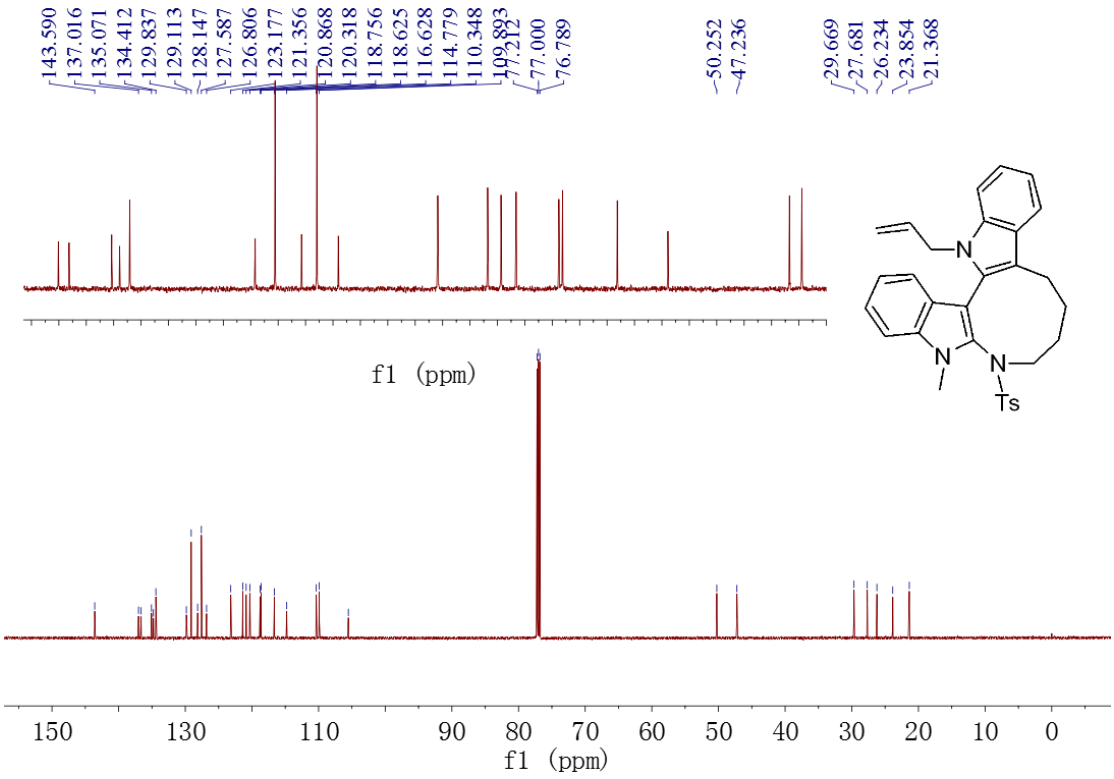
^{1}H NMR Spectrum for 7d ($CDCl_3$, 600 MHz)



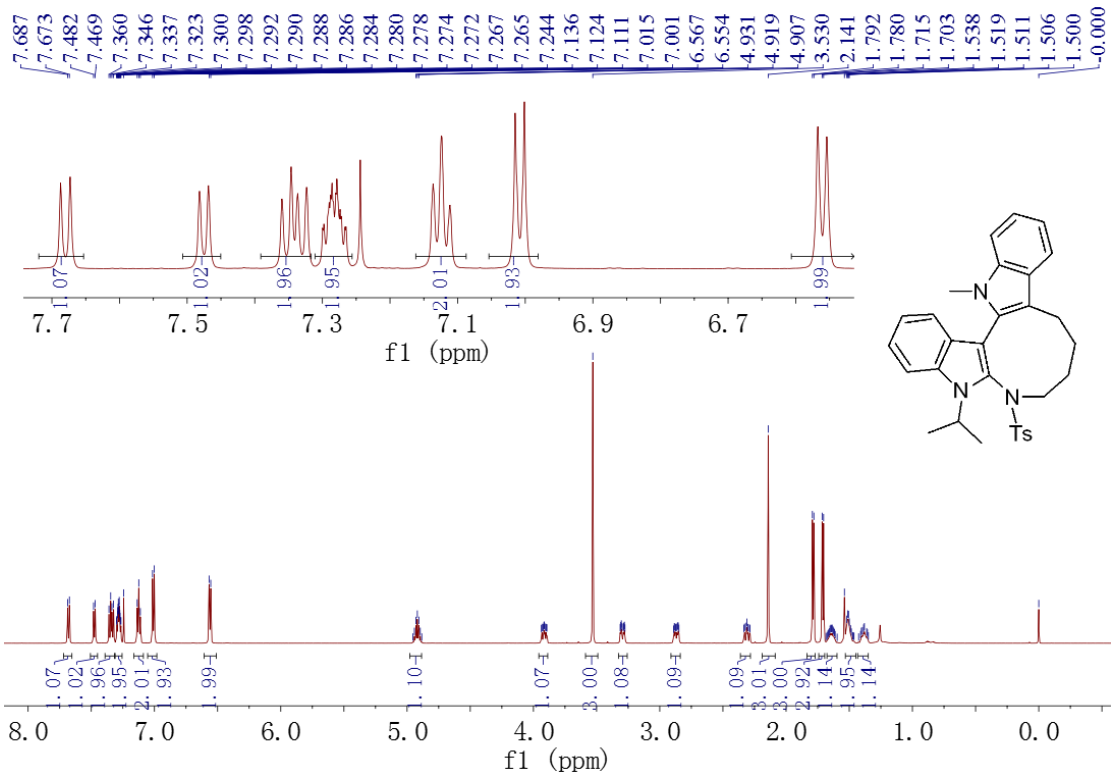
^{13}C NMR Spectrum for 7d ($CDCl_3$, 150 MHz)



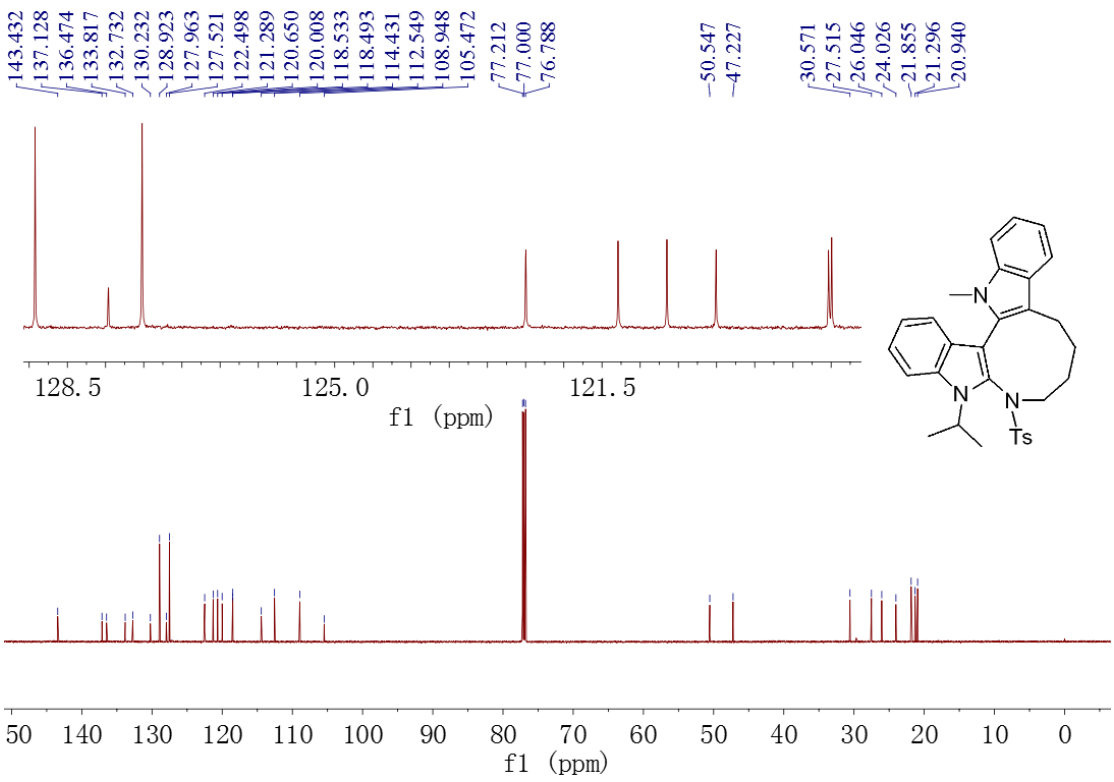
^{1}H NMR Spectrum for 7e ($CDCl_3$, 600 MHz)



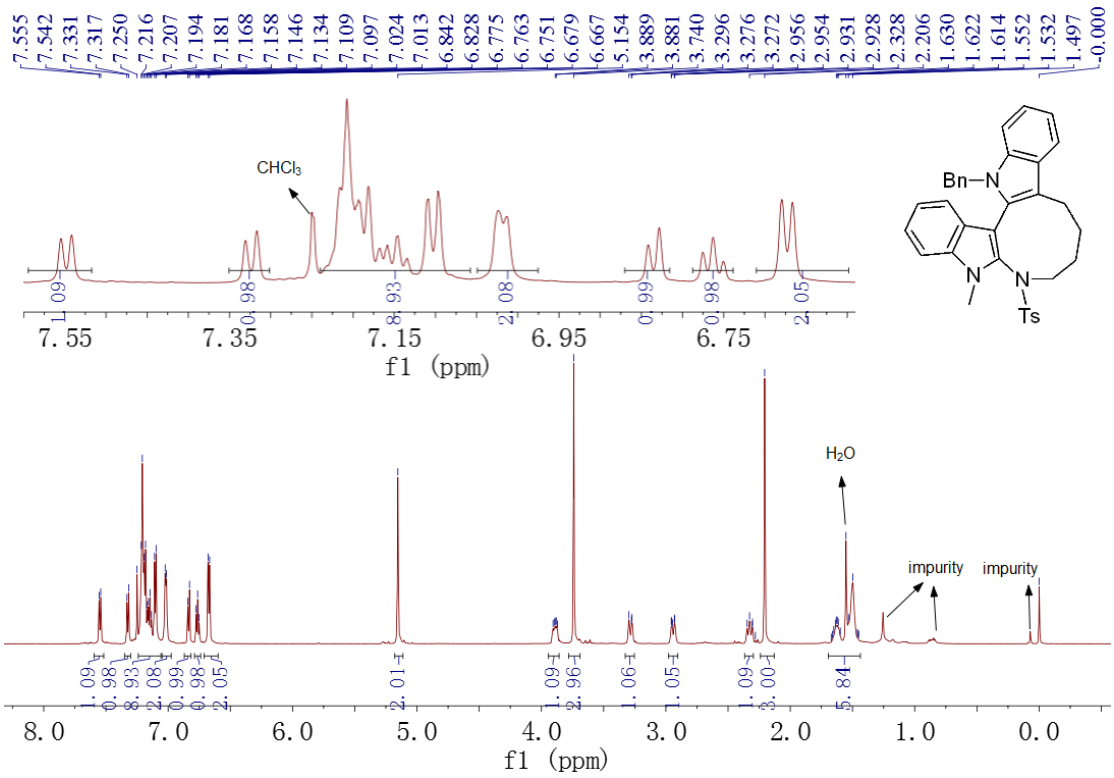
^{13}C NMR Spectrum for 7e ($CDCl_3$, 150 MHz)



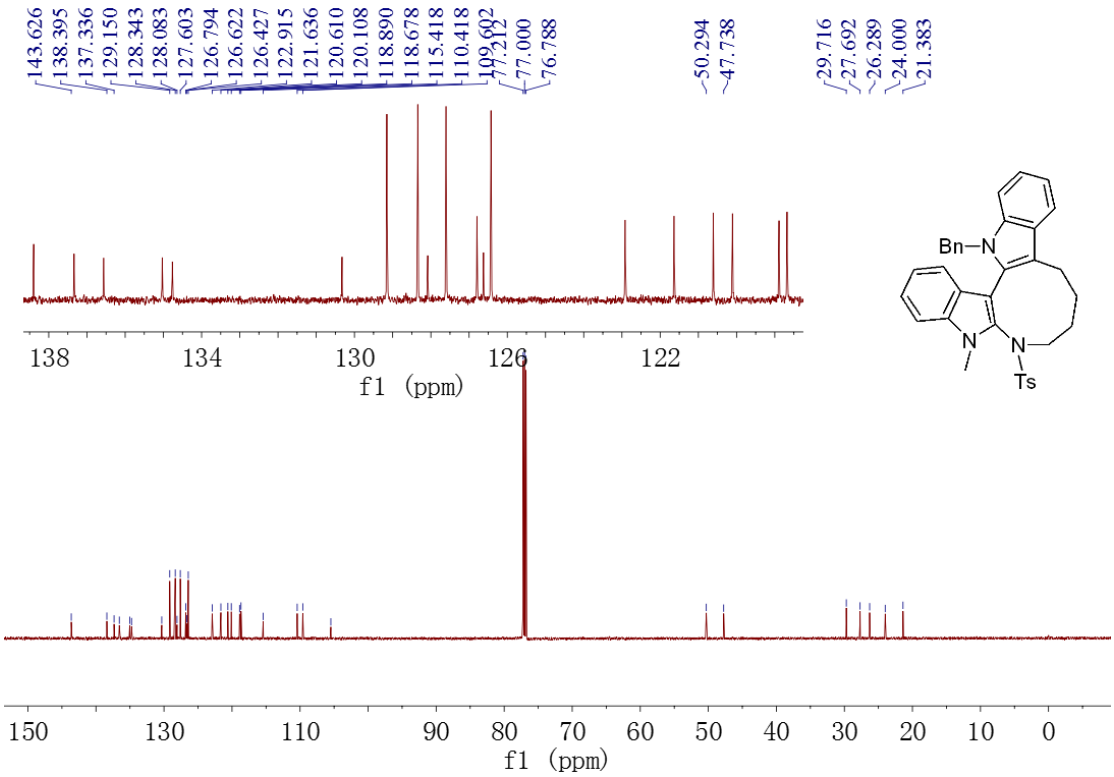
^{1}H NMR Spectrum for 7f ($CDCl_3$, 600 MHz)



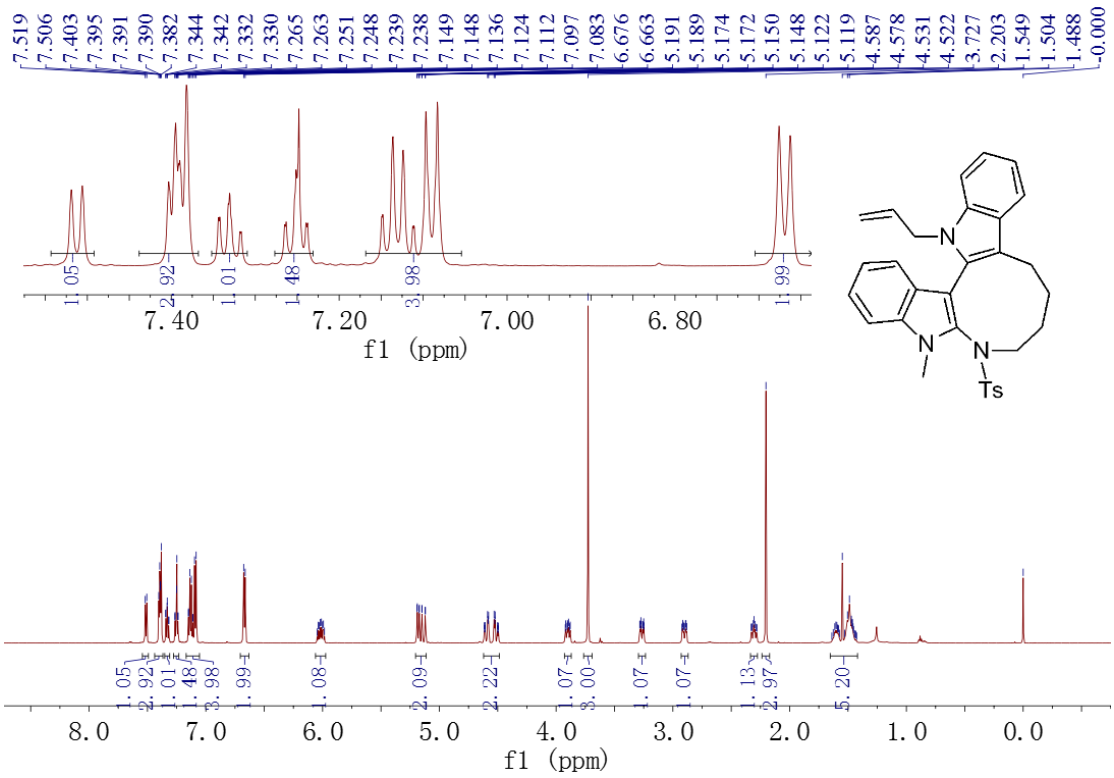
^{13}C NMR Spectrum for 7f ($CDCl_3$, 150 MHz)



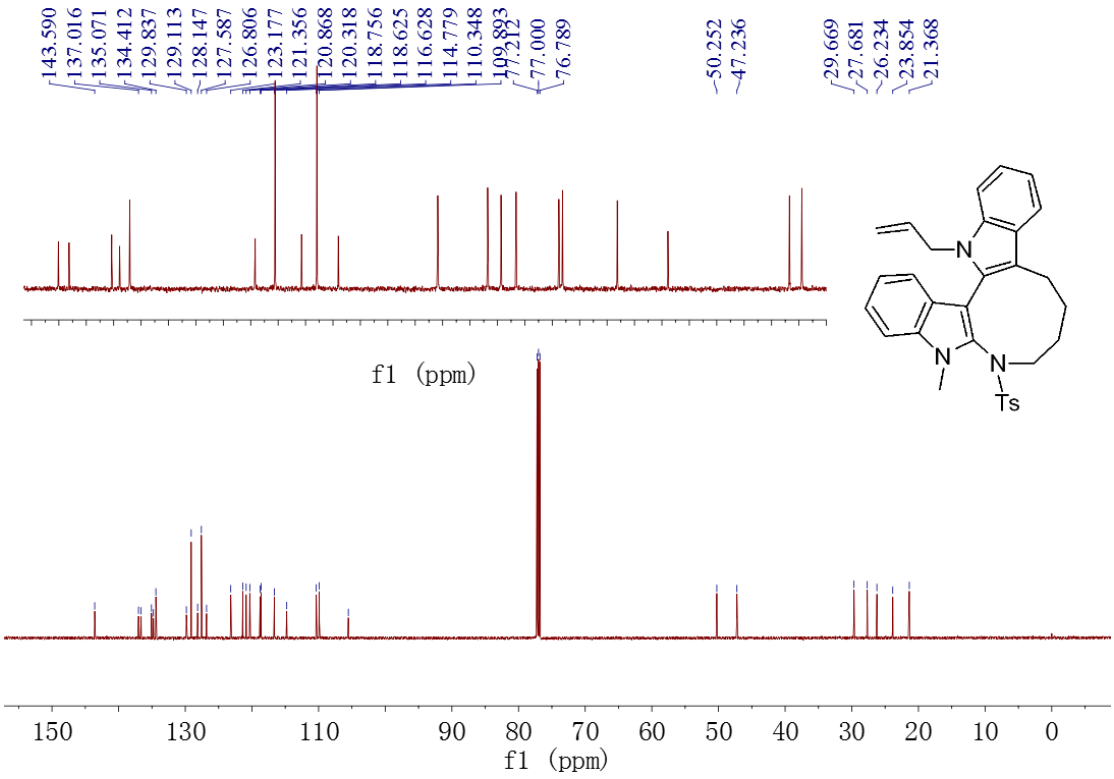
^{1}H NMR Spectrum for 7g ($CDCl_3$, 600 MHz)



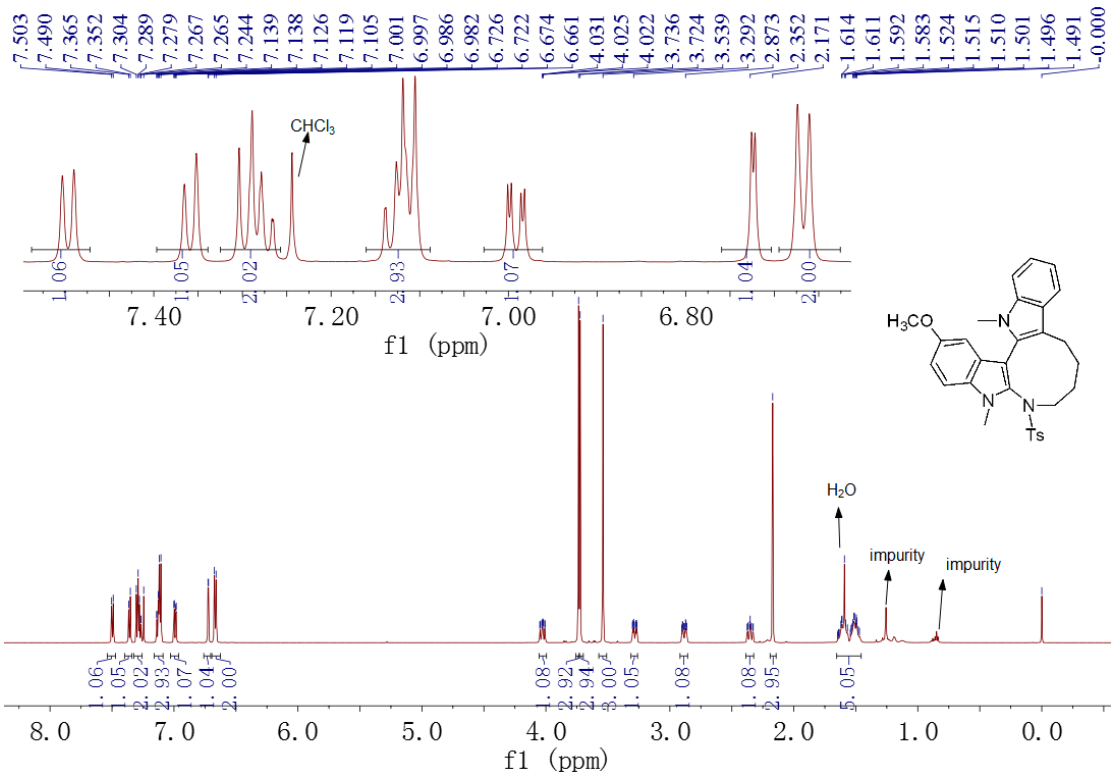
^{13}C NMR Spectrum for 7g ($CDCl_3$, 150 MHz)



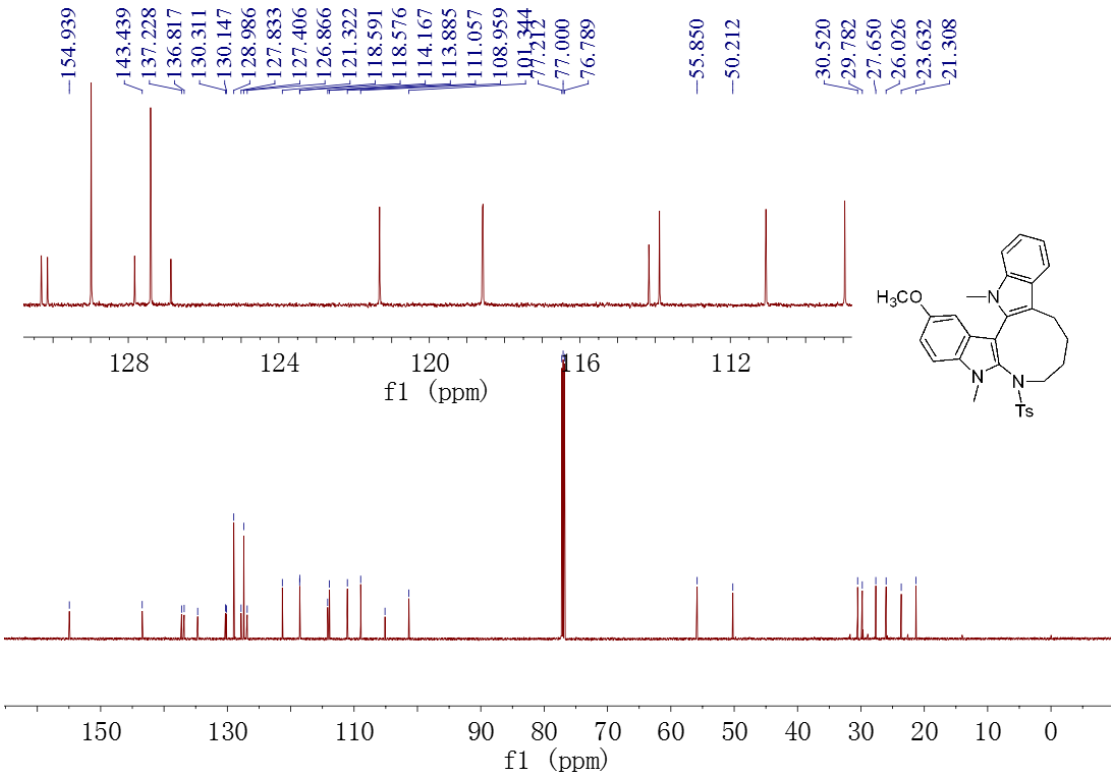
^{1}H NMR Spectrum for 7h ($CDCl_3$, 600 MHz)



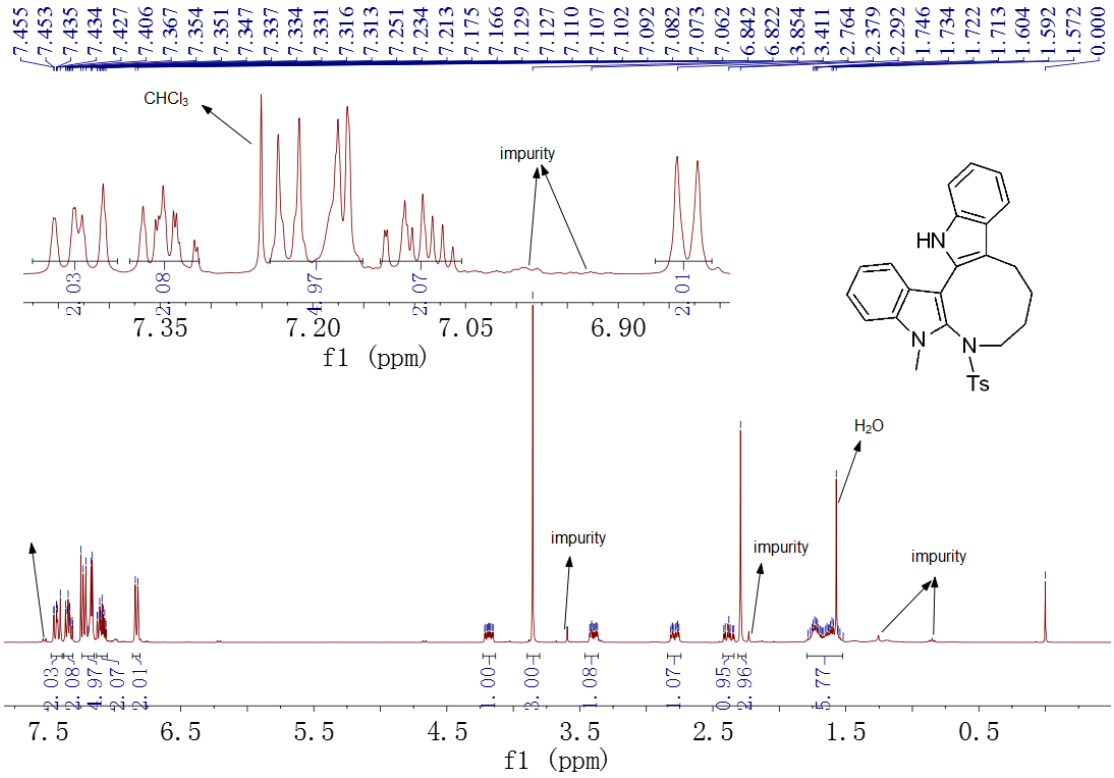
^{13}C NMR Spectrum for 7h ($CDCl_3$, 150 MHz)



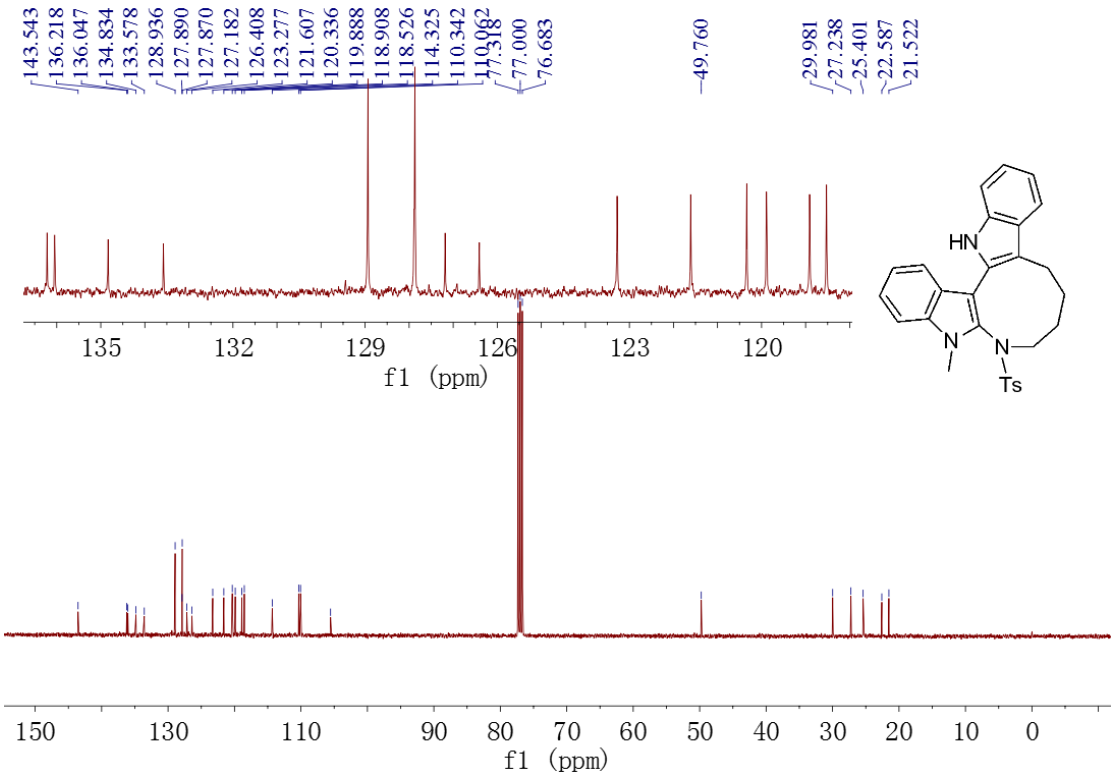
^{1}H NMR Spectrum for 7i ($CDCl_3$, 600 MHz)



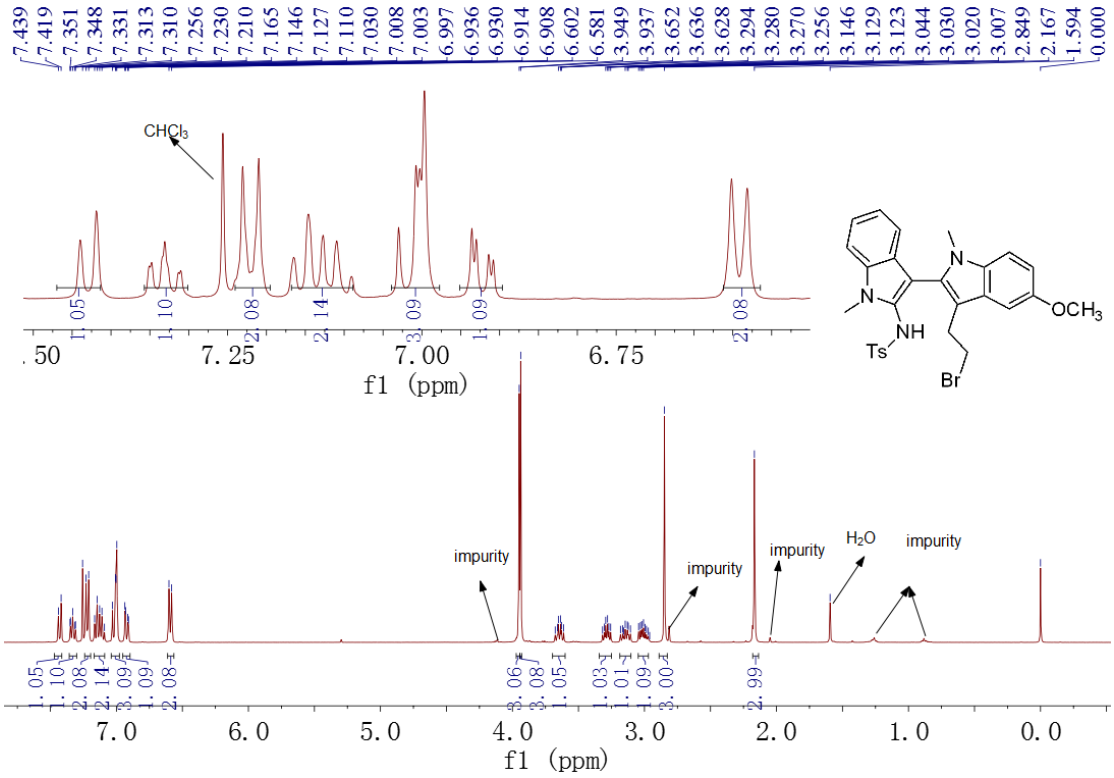
^{13}C NMR Spectrum for 7i ($CDCl_3$, 150 MHz)



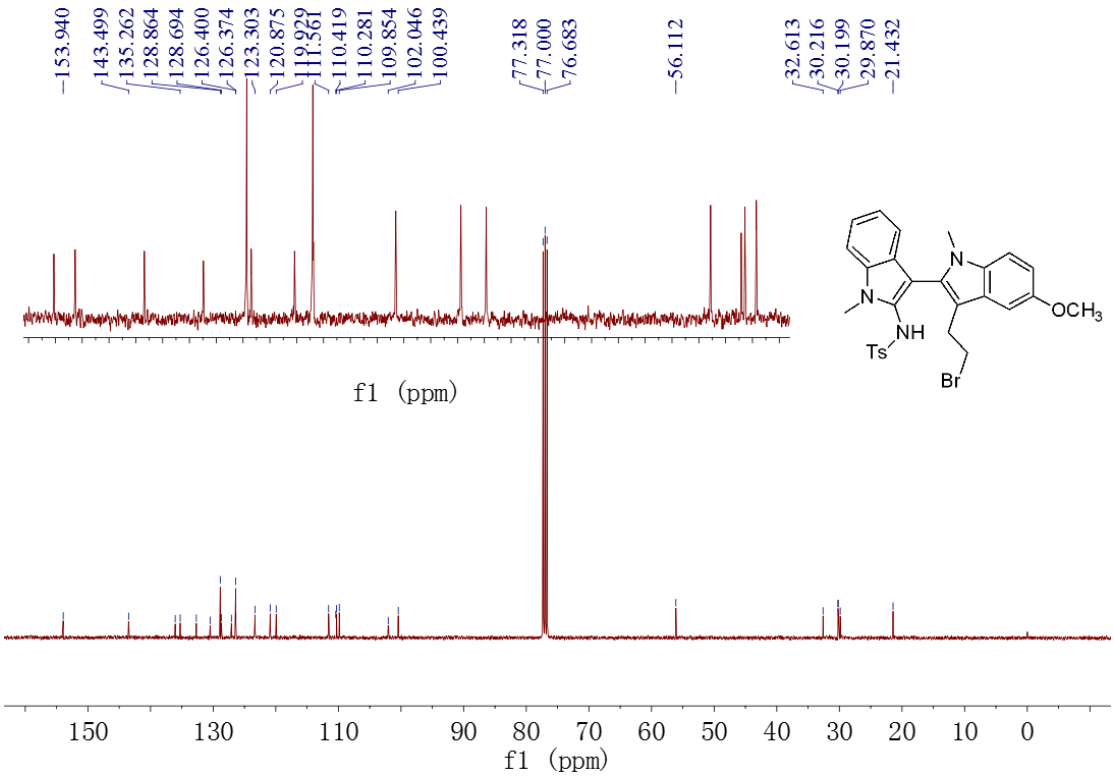
^{1}H NMR Spectrum for 7j ($CDCl_3$, 400 MHz)



^{13}C NMR Spectrum for 7j ($CDCl_3$, 100 MHz)



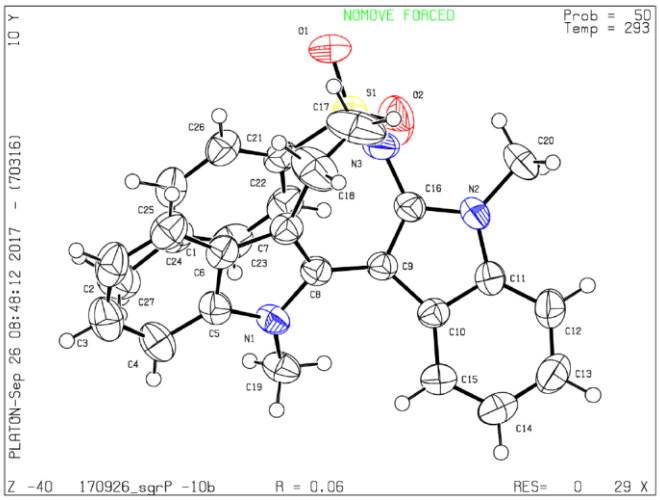
^{1}H NMR Spectrum for 8 ($CDCl_3$, 400 MHz)



^{13}C NMR Spectrum for 8 ($CDCl_3$, 100 MHz)

Crystal Structures of 3a

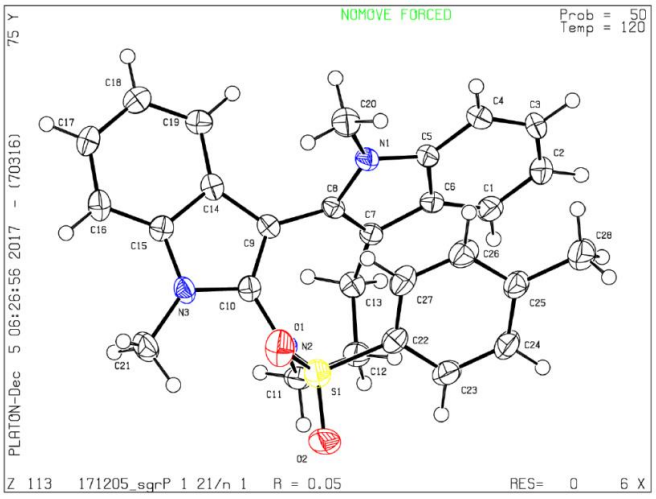
The ORTEP diagram and Crystal Parameters of **3a** wherein thermal ellipsoids are drawn at 30% probability level. The crystals of suitable quality were obtained from EA/n-hexane, and were analyzed by single crystal diffractometer (Varian, Gemini A Ultra). Atomic coordinates, bond lengths, bond angles, and thermal parameters have been deposited at the Cambridge Crystallographic Data Centre. CCDC deposit number is 1857861.



```
Bond precision:  C-C = 0.0045 A          Wavelength=0.71073

Cell:          a=10.2015(9)       b=10.5115(10)     c=12.4818(14)
               alpha=66.676(10)   beta=66.523(10)   gamma=89.163(7)
Temperature:   293 K

                 Calculated                Reported
Volume           1110.9(2)                 1110.9(2)
Space group      P -1                      P -1
Hall group       -P 1                      -P 1
Moiety formula   C27 H25 N3 O2 S           C27 H25 N3 O2 S
Sum formula      C27 H25 N3 O2 S           C27 H25 N3 O2 S
Mr               455.56                    455.56
Dx,g cm-3        1.362                     1.362
Z                2                         2
Mu (mm-1)        0.177                     0.177
F000             480.0                     480.0
F000'            480.43
h,k,lmax         12,12,15                  12,12,15
Nref             4060                      4049
Tmin,Tmax        0.917,0.952               0.873,1.000
Tmin'            0.917

Correction method= # Reported T Limits: Tmin=0.873 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.997          Theta(max)= 25.348

R(reflections)= 0.0579( 3076)     wR2(reflections)= 0.1719( 4049)

S = 1.045                 Npar= 301
```

Crystal Structures of 5a

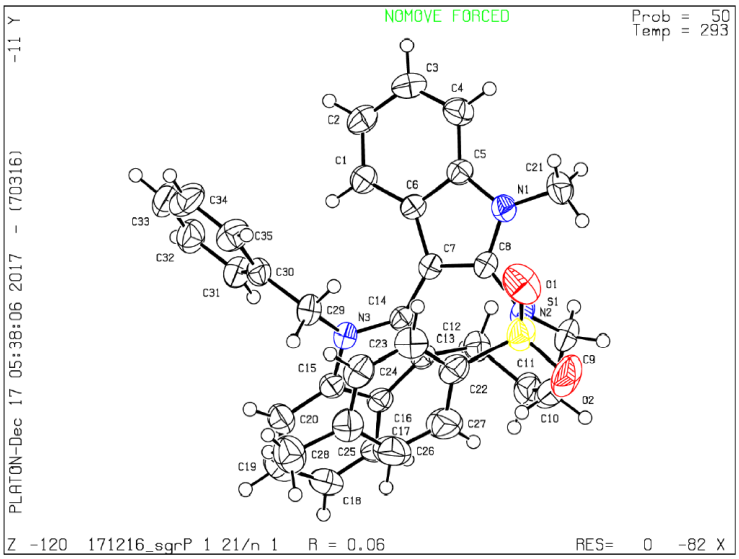
The ORTEP diagram and Crystal Parameters of **5a** wherein thermal ellipsoids are drawn at 30% probability level. The crystals of suitable quality were obtained from EA/n-hexane, and were analyzed by single crystal diffractometer (Varian, Gemini A Ultra). Atomic coordinates, bond lengths, bond angles, and thermal parameters have been deposited at the Cambridge Crystallographic Data Centre. CCDC deposit number is 1857867.



```
Bond precision:  C-C = 0.0033 A          Wavelength=0.71073

Cell:            a=8.6570(7)     b=26.7995(18)       c=10.5139(8)
                 alpha=90        beta=102.303(8)     gamma=90
Temperature:     120 K

                 Calculated                Reported
Volume           2383.2(3)                 2383.2(3)
Space group      P 21/n                    P 1 21/n 1
Hall group       -P 2yn                    -P 2yn
Moiety formula   C28 H27 N3 O2 S           C28 H27 N3 O2 S
Sum formula      C28 H27 N3 O2 S           C28 H27 N3 O2 S
Mr               469.59                    469.58
Dx,g cm-3        1.309                     1.309
Z                4                         4
Mu (mm-1)        0.167                     0.167
F000             992.0                     992.0
F000'            992.86
h,k,lmax         10,32,12                  10,32,12
Nref             4368                      4353
Tmin,Tmax        0.931,0.970               0.792,1.000
Tmin'            0.931

Correction method= # Reported T Limits: Tmin=0.792 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.997          Theta(max)= 25.350

R(reflections)= 0.0510( 3293)     wR2(reflections)= 0.1335( 4353)

S = 1.017                 Npar= 310
```

Crystal Structures of 7g

The ORTEP diagram and Crystal Parameters of **7g** wherein thermal ellipsoids are drawn at 30% probability level. The crystals of suitable quality were obtained from EA/n-hexane, and were analyzed by single crystal diffractometer (Varian, Gemini A Ultra). Atomic coordinates, bond lengths, bond angles, and thermal parameters have been deposited at the Cambridge Crystallographic Data Centre. CCDC deposit number is 1857866.



Bond precision:	C-C = 0.0038 A		Wavelength=0.71073
Cell:	a=14.9128(9)	b=9.6989(5)	c=19.5579(10)
	alpha=90	beta=96.549(6)	gamma=90
Temperature:	293 K		

	Calculated	Reported
Volume	2810.4(3)	2810.4(3)
Space group	P 21/n	P 1 21/n 1
Hall group	-P 2yn	-P 2yn
Moiety formula	C35 H33 N3 O2 S	C35 H33 N3 O2 S
Sum formula	C35 H33 N3 O2 S	C35 H33 N3 O2 S
Mr	559.70	559.70
Dx,g cm-3	1.323	1.323
Z	4	4
Mu (mm-1)	0.154	0.154
F000	1184.0	1184.0
F000'	1184.93	
h,k,lmax	17,11,23	17,11,23
Nref	5148	5137
Tmin,Tmax	0.943,0.958	0.447,1.000
Tmin'	0.942	

Correction method= # Reported T Limits: Tmin=0.447 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.998 Theta(max)= 25.349

R(reflections)= 0.0640(3696) wR2(reflections)= 0.1878(5137)

S = 1.041 Npar= 372