

Enantioselective Synthesis of 4,5-dihydropyrroles via Domino Ring Opening Cyclization (DROC) of Aziridines with Malononitrile

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Supporting Information

SL No.	Contents	Page No.
1.	NMR spectra	S 2
2.	NOE Spectra of 3r	S 22
3.	Selected HPLC chromatograms	S 25

3. NMR Spectra:

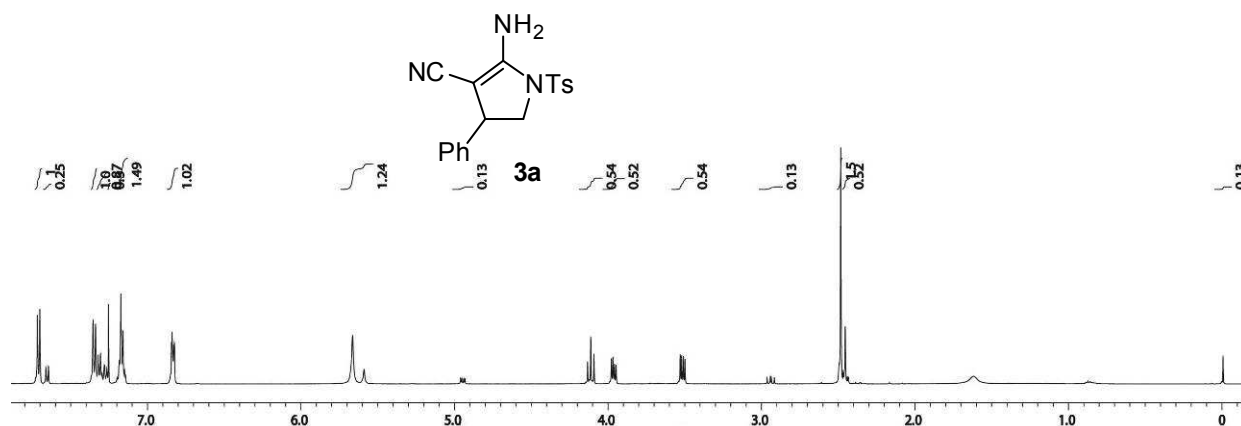


Figure 1b: ^1H NMR spectrum of **3a** (mixture of regioisomers) (CDCl_3 , 500 MHz)

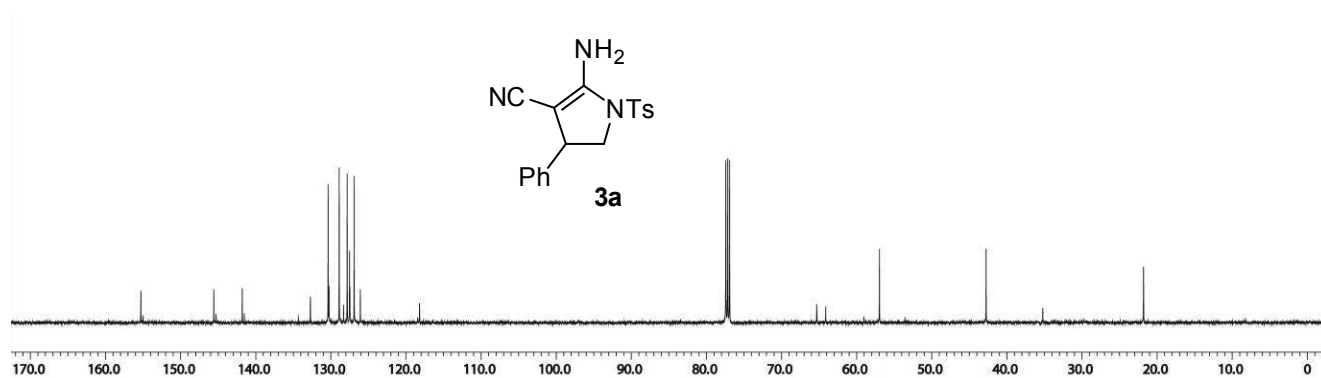


Figure 2: ^{13}C NMR spectrum of **3a** (CDCl_3 , 125 MHz)

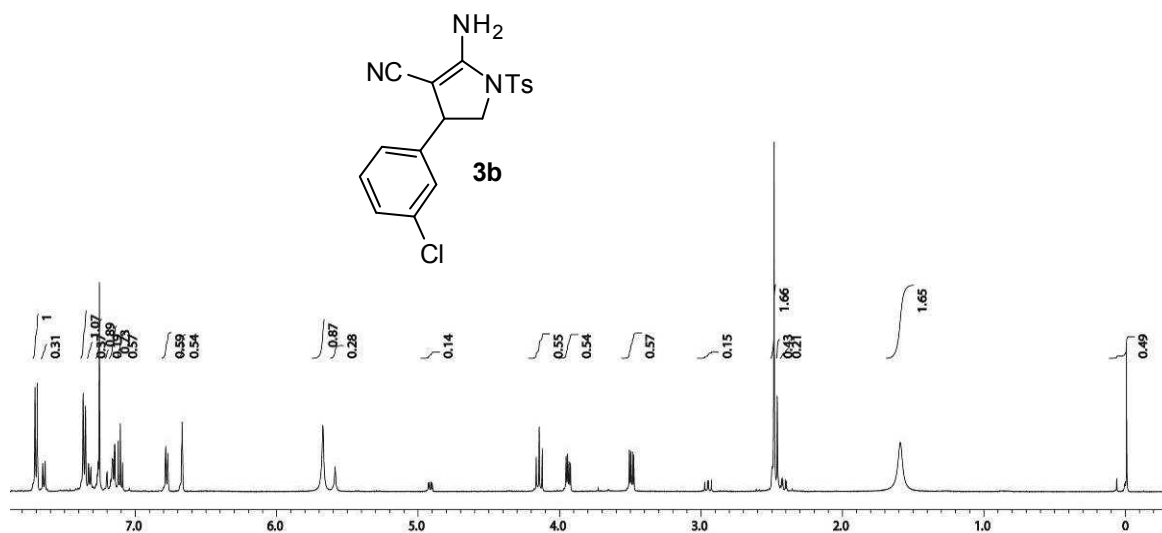


Figure 3: ¹H NMR spectrum of **3b** (mixture of regioisomers) (CDCl₃, 500 MHz)

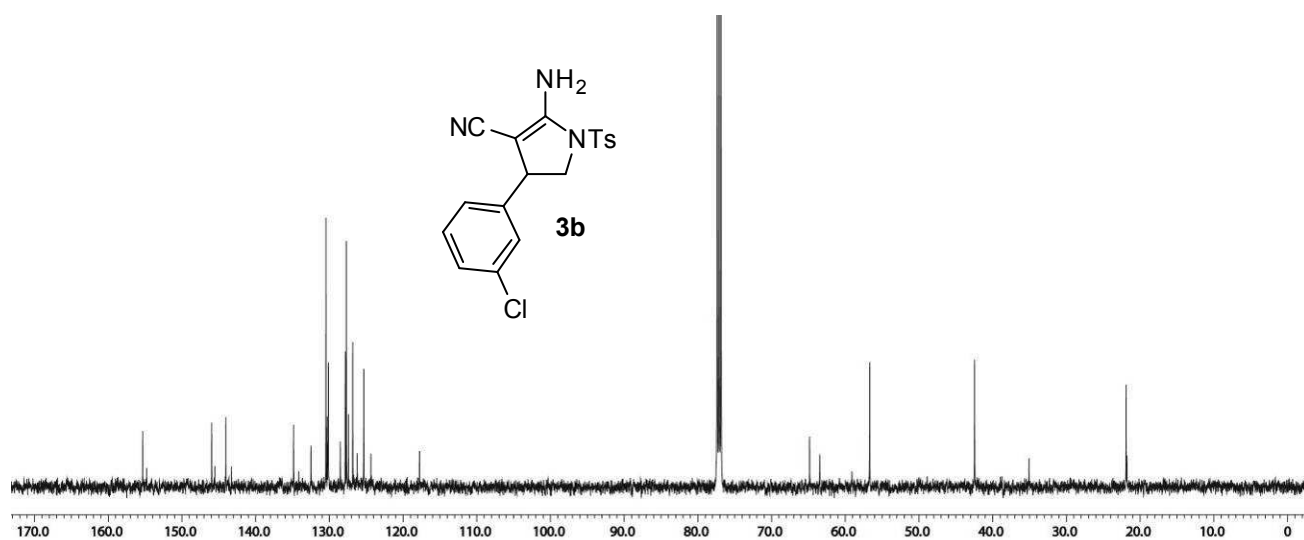


Figure 4: ¹³C NMR spectrum of **3b** (mixture of regioisomers) (CDCl₃, 125 MHz)

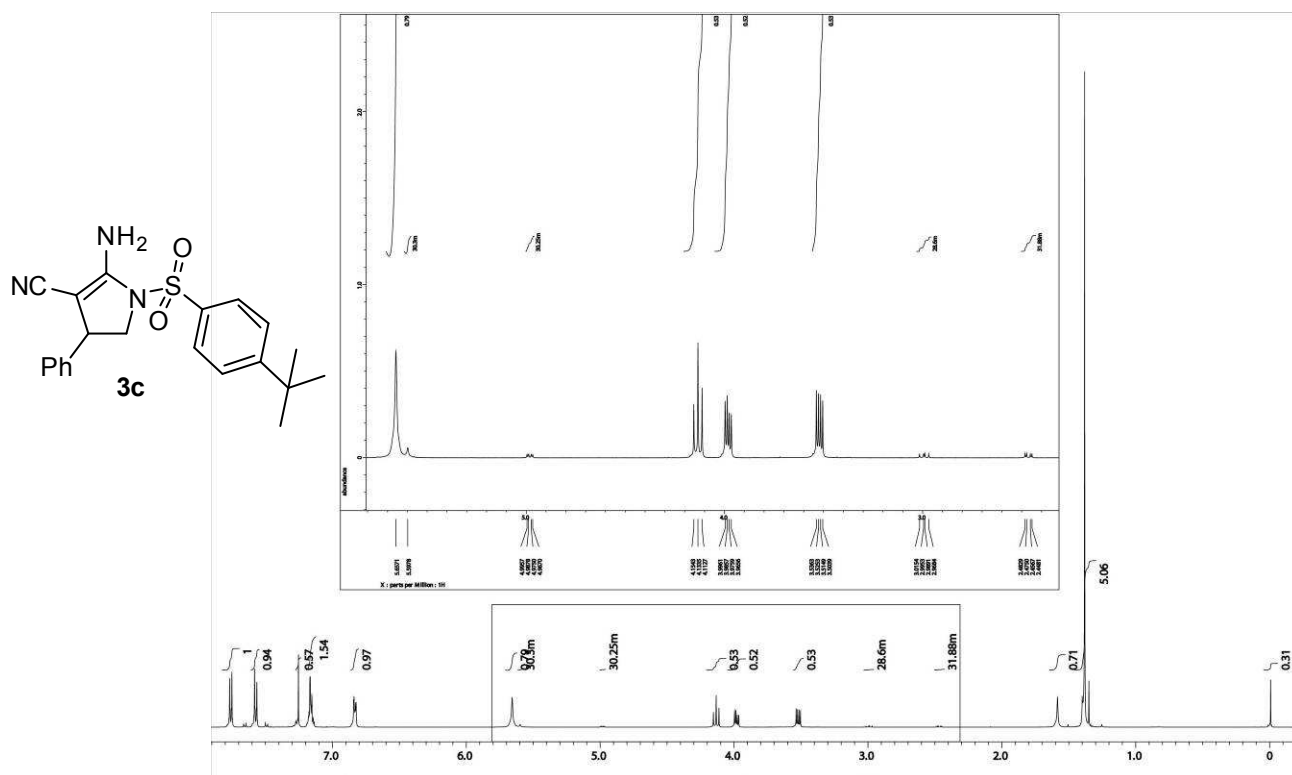


Figure 5: ^1H NMR spectrum of **3c** (mixture of regioisomers) (CDCl_3 , 500 MHz)

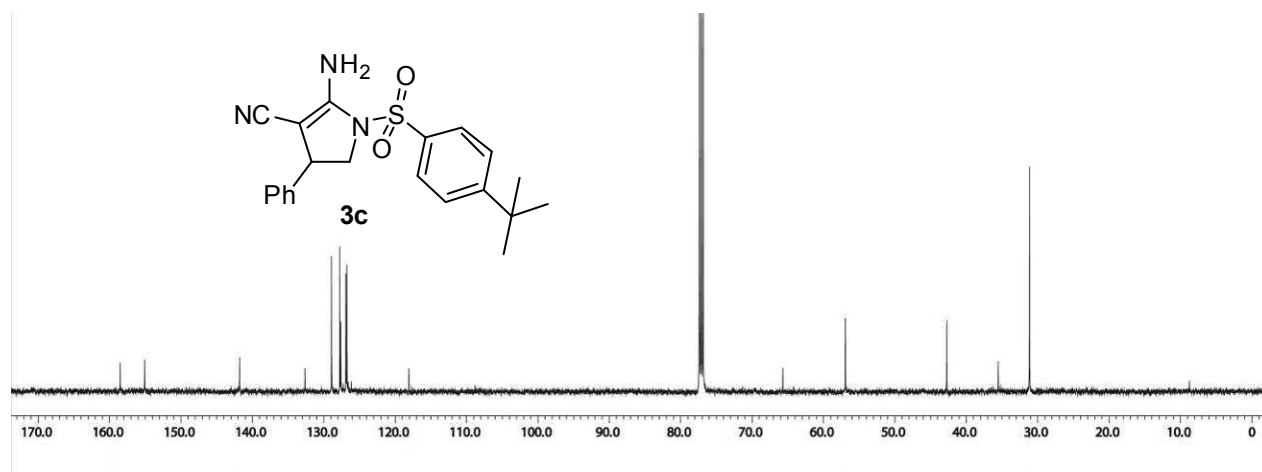


Figure 6: ^{13}C NMR spectrum of **3c** (mixture of regioisomers) (CDCl_3 , 125 MHz)

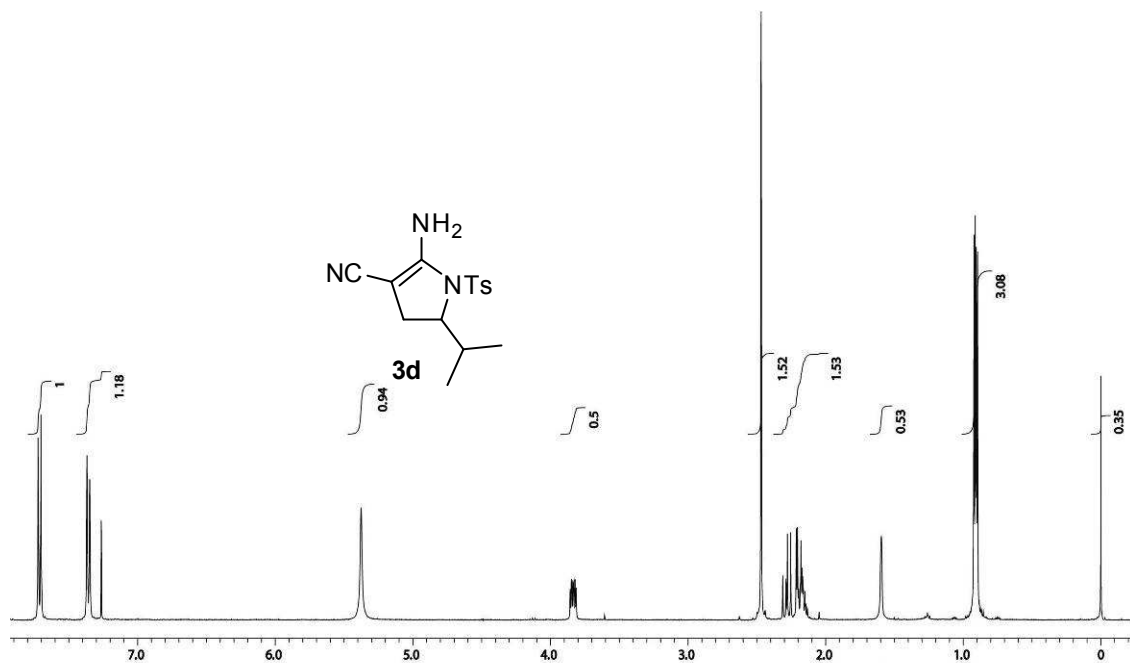


Figure 7: ^1H NMR spectrum of **3d** (CDCl_3 , 400 MHz)

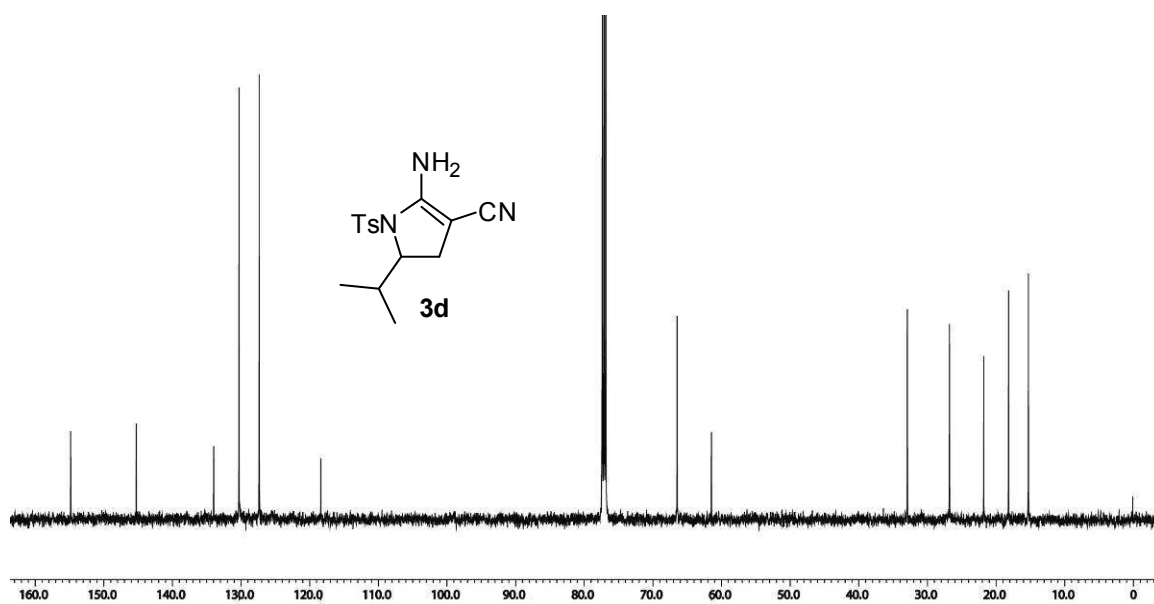


Figure 8: ^{13}C NMR spectrum of **3d** (CDCl_3 , 125 MHz)

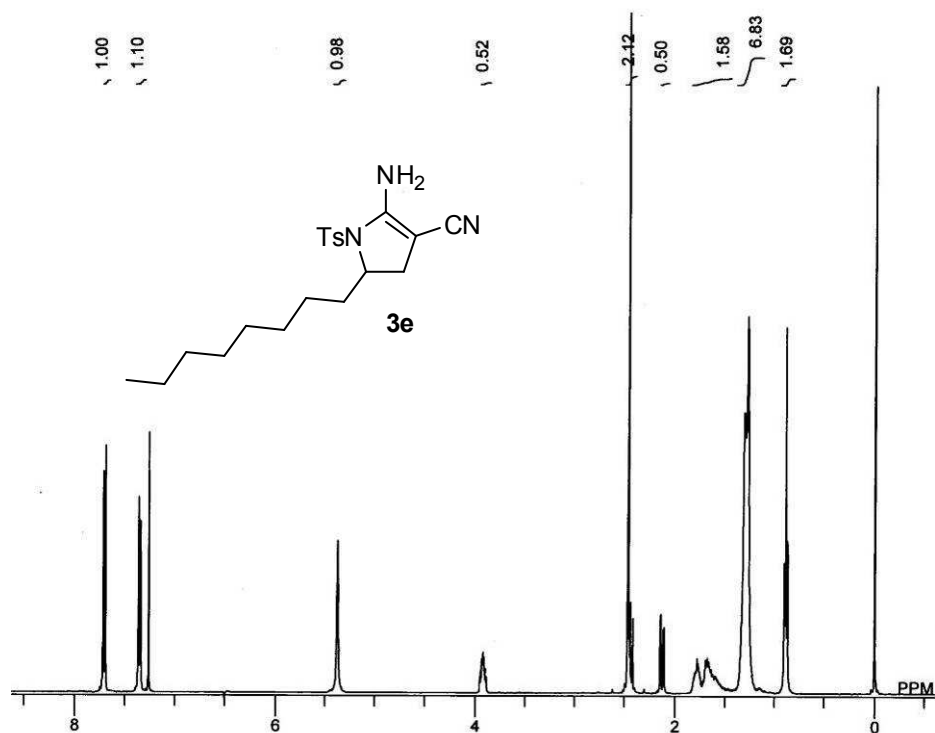


Figure 9: ¹H NMR spectrum of **3e** (CDCl₃, 400 MHz)

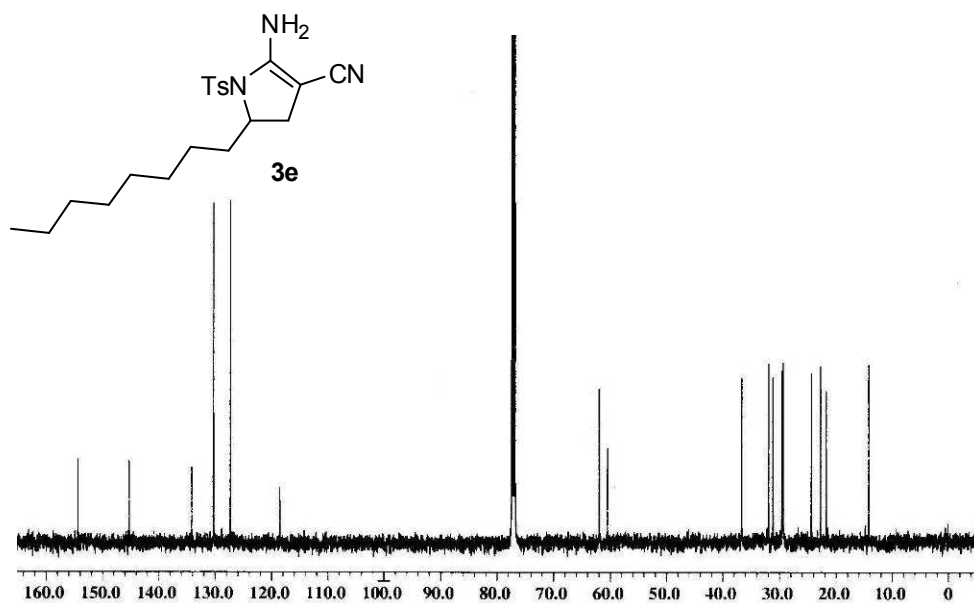


Figure 10: ¹³C NMR spectrum of **3e** (CDCl₃, 125 MHz)

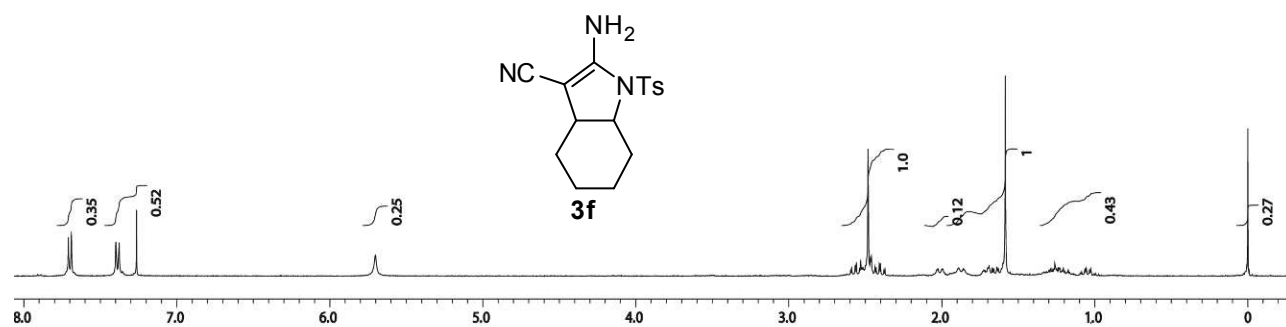


Figure 11: ¹H NMR spectrum of **3f** (CDCl₃, 400 MHz)

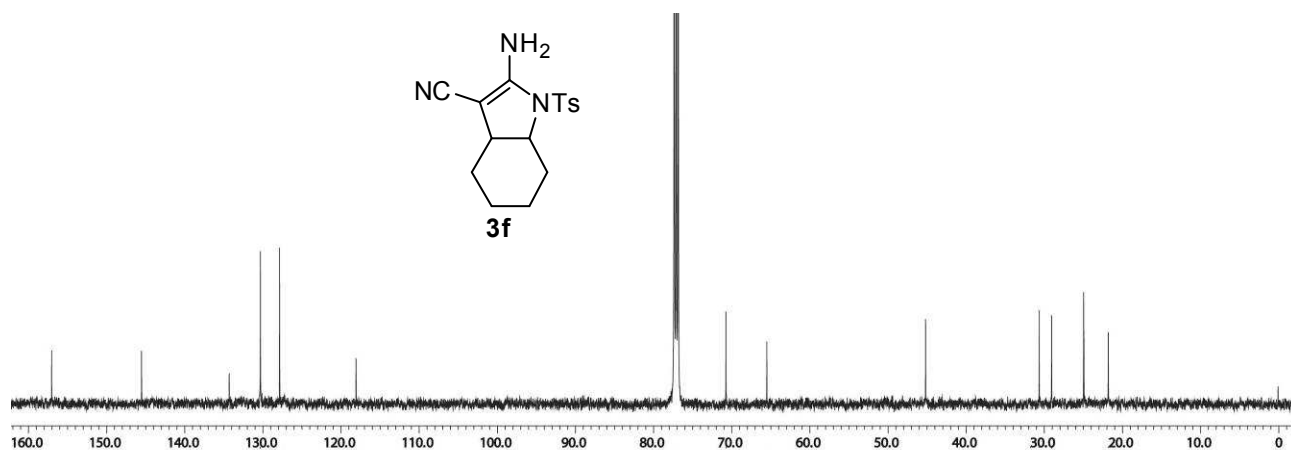


Figure 12: ¹³C NMR spectrum of **3f** (CDCl₃, 125 MHz)

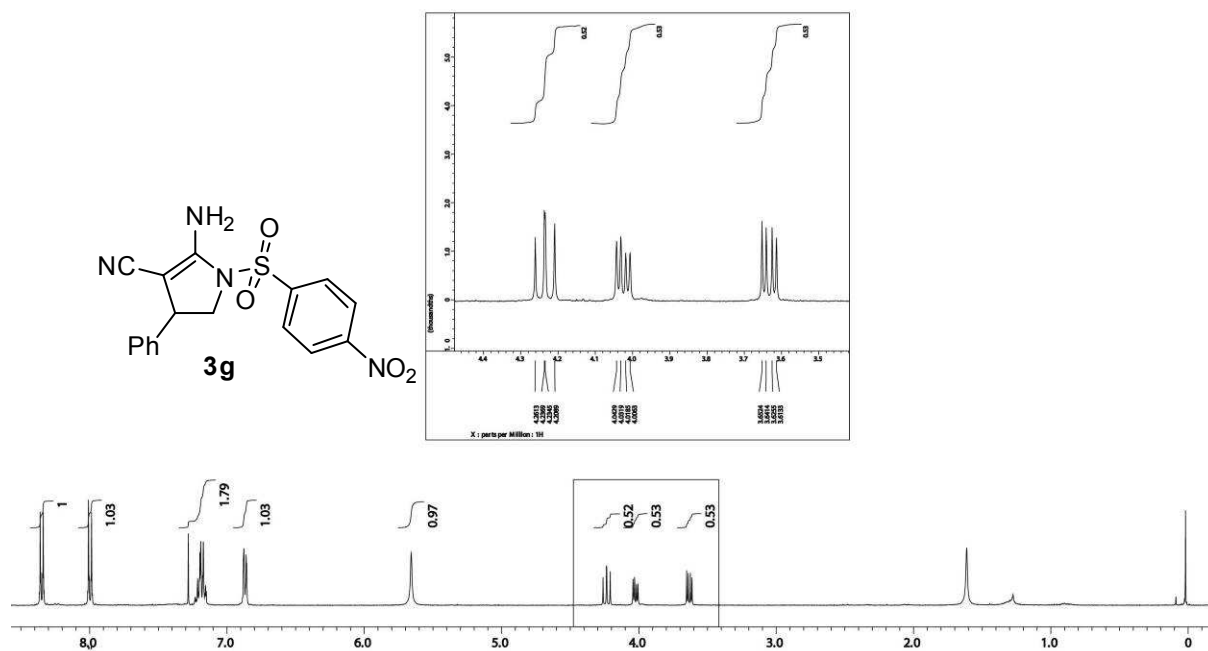


Figure 13: ¹H NMR spectrum of **3g** (CDCl₃, 400 MHz)

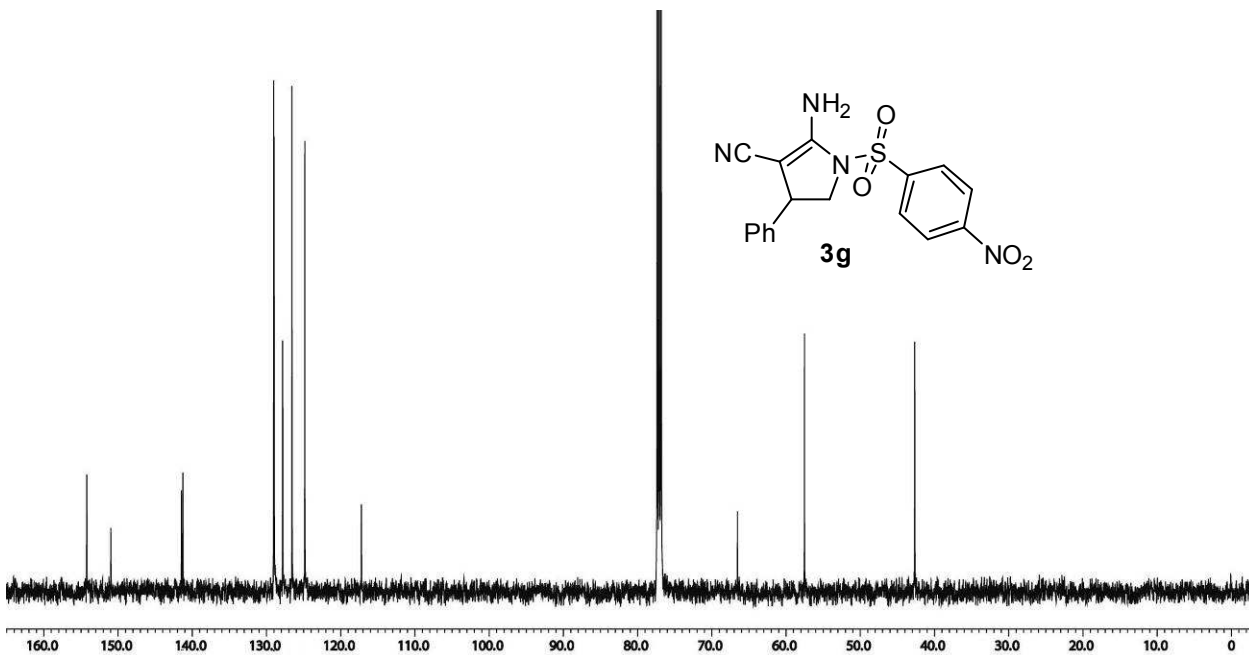


Figure 14: ¹³C NMR spectrum of **3g** (CDCl₃, 125 MHz)

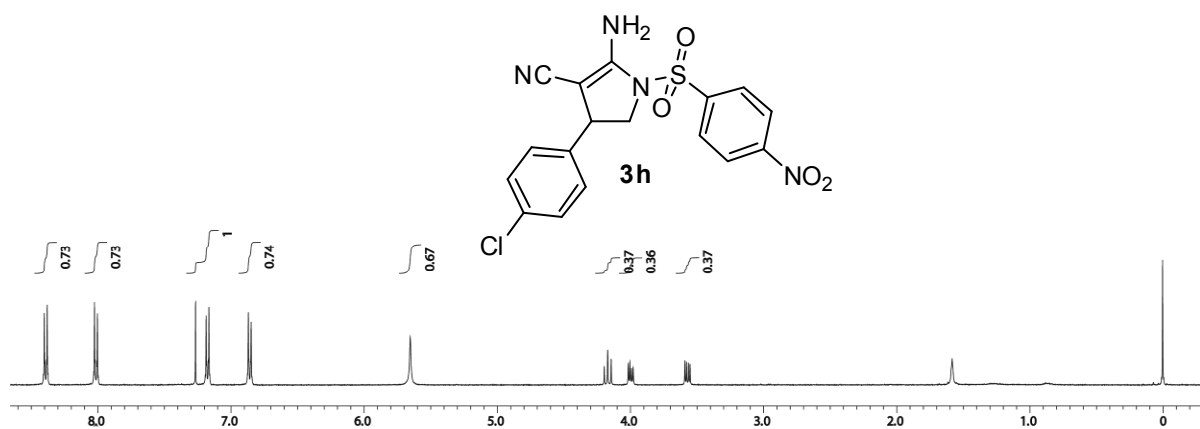


Figure 15: ¹H NMR spectrum of **3h** (CDCl₃, 400 MHz)

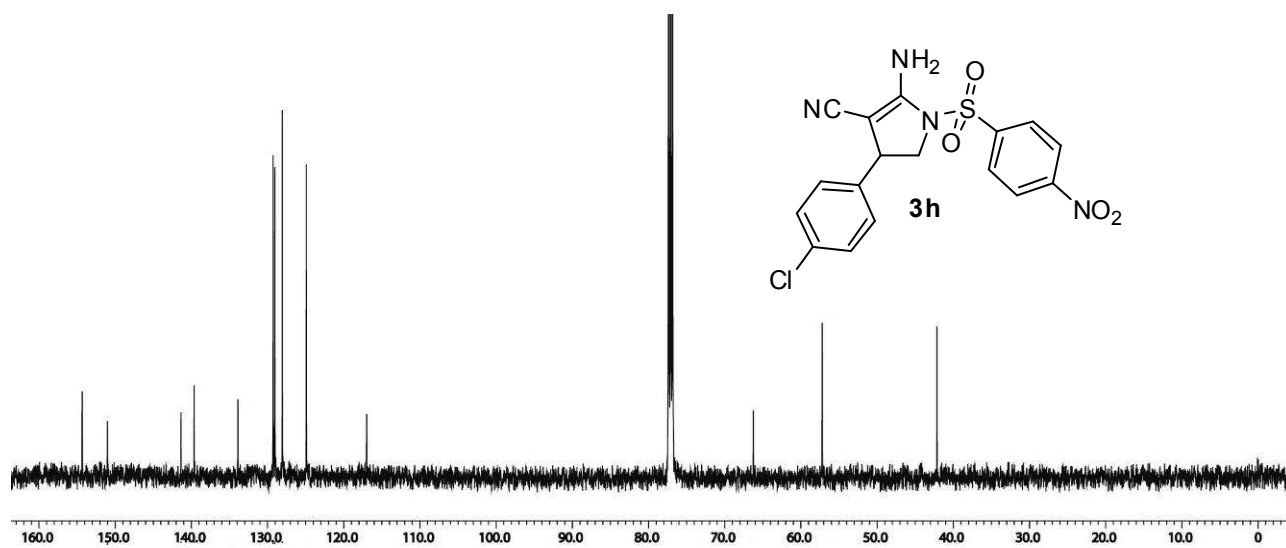


Figure 16: ¹³C NMR spectrum of **3h** (CDCl₃, 125 MHz)

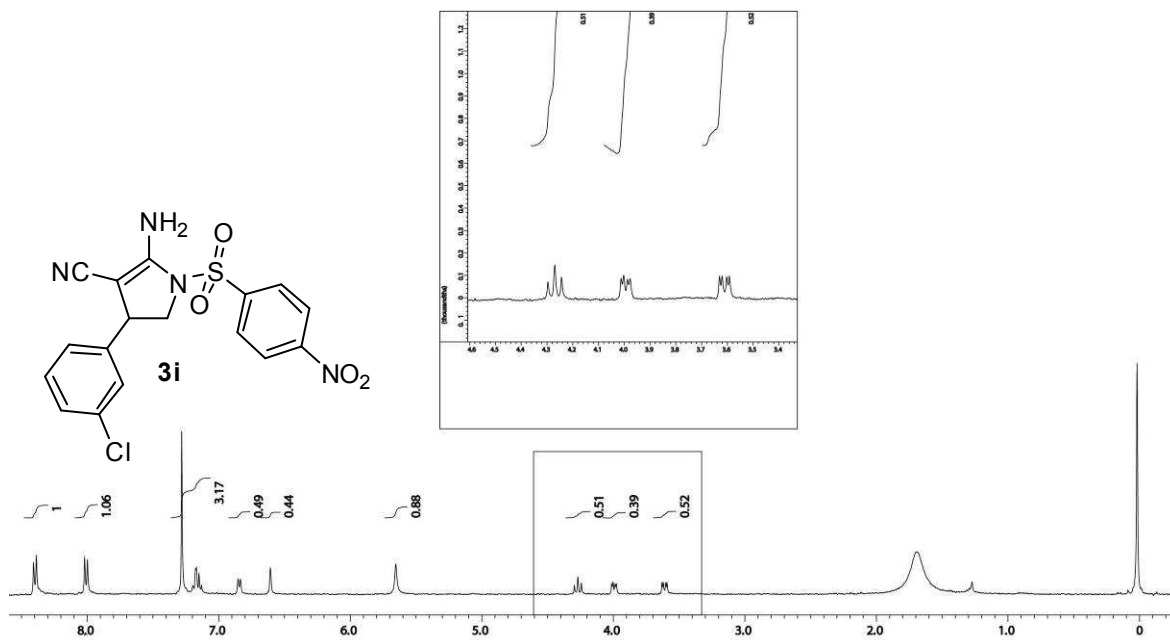


Figure 17: ¹H NMR spectrum of **3i** (CDCl₃, 400 MHz)

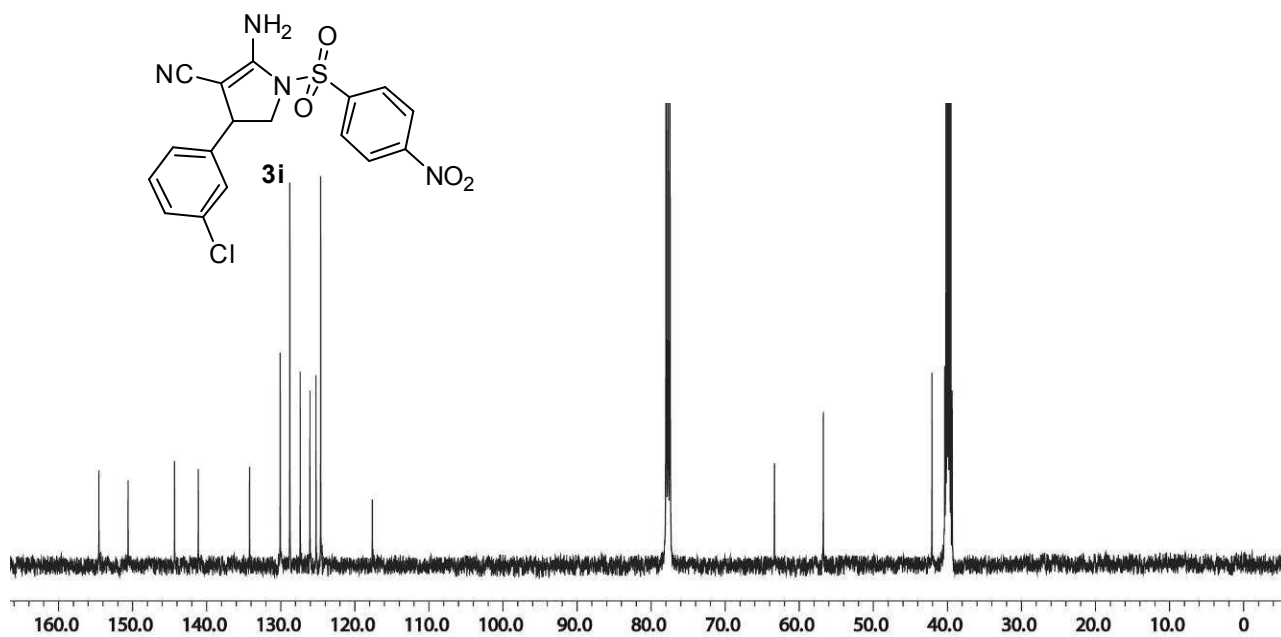


Figure 18: ¹³C NMR spectrum of **3i** (CDCl₃+DMSO-d₆, 125 MHz)

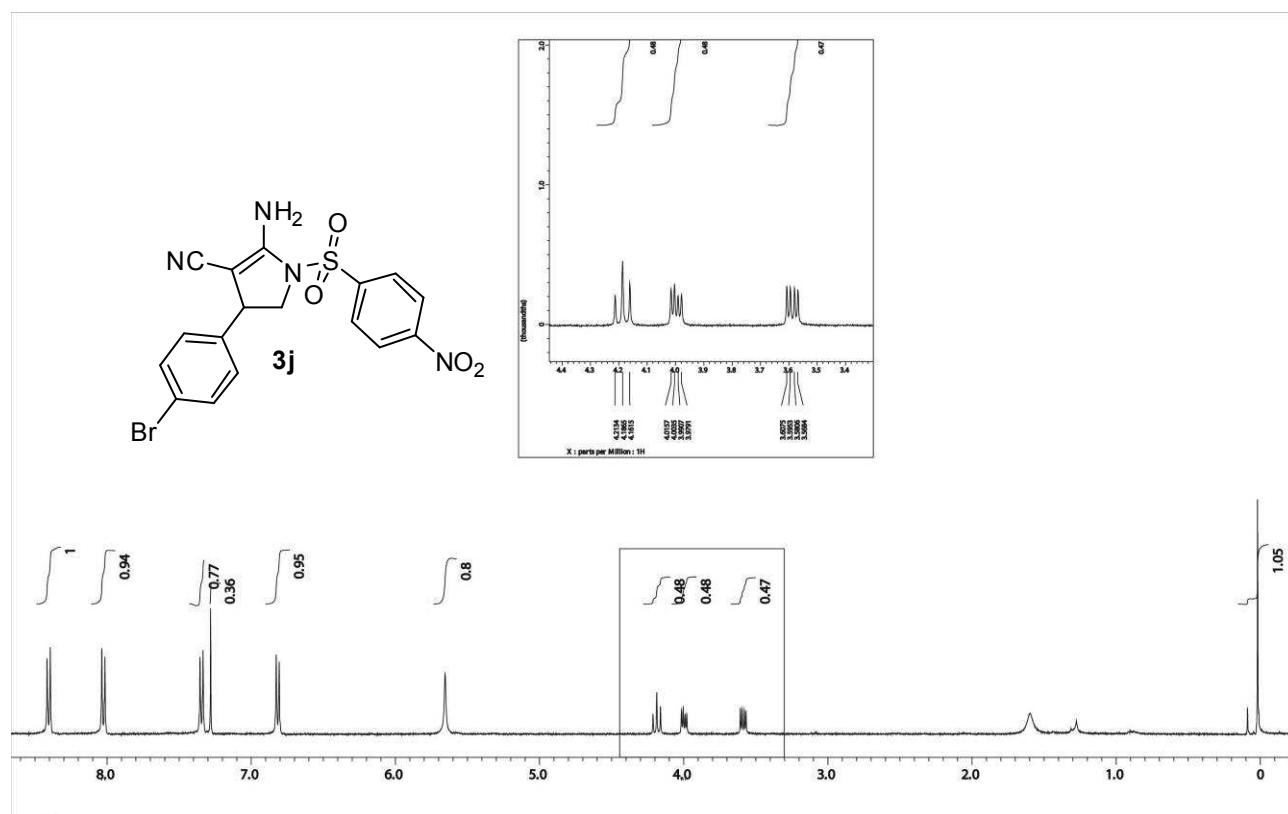


Figure 19: ¹H NMR spectrum of **3j** (CDCl₃, 400 MHz)

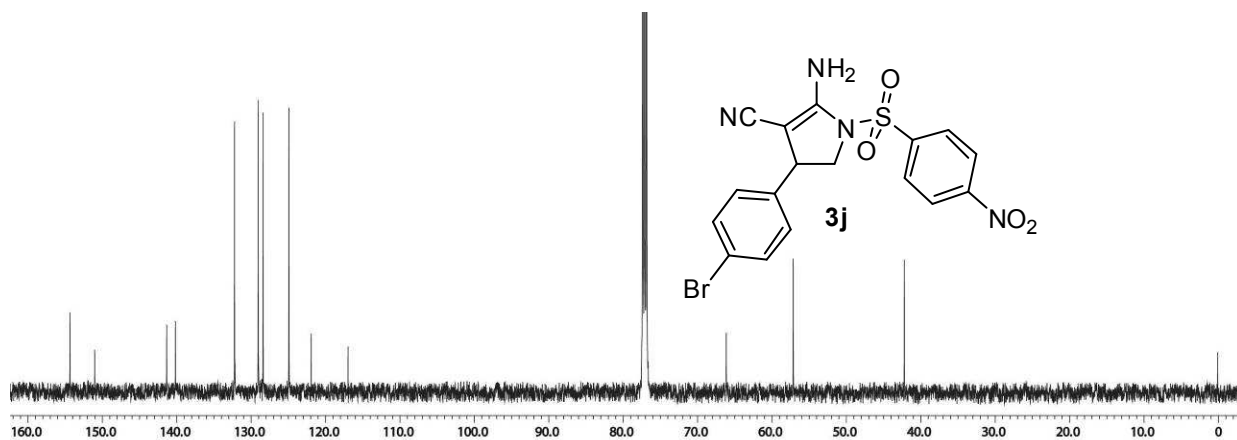


Figure 20: ¹³C NMR spectrum of **3j** (CDCl₃, 125 MHz)

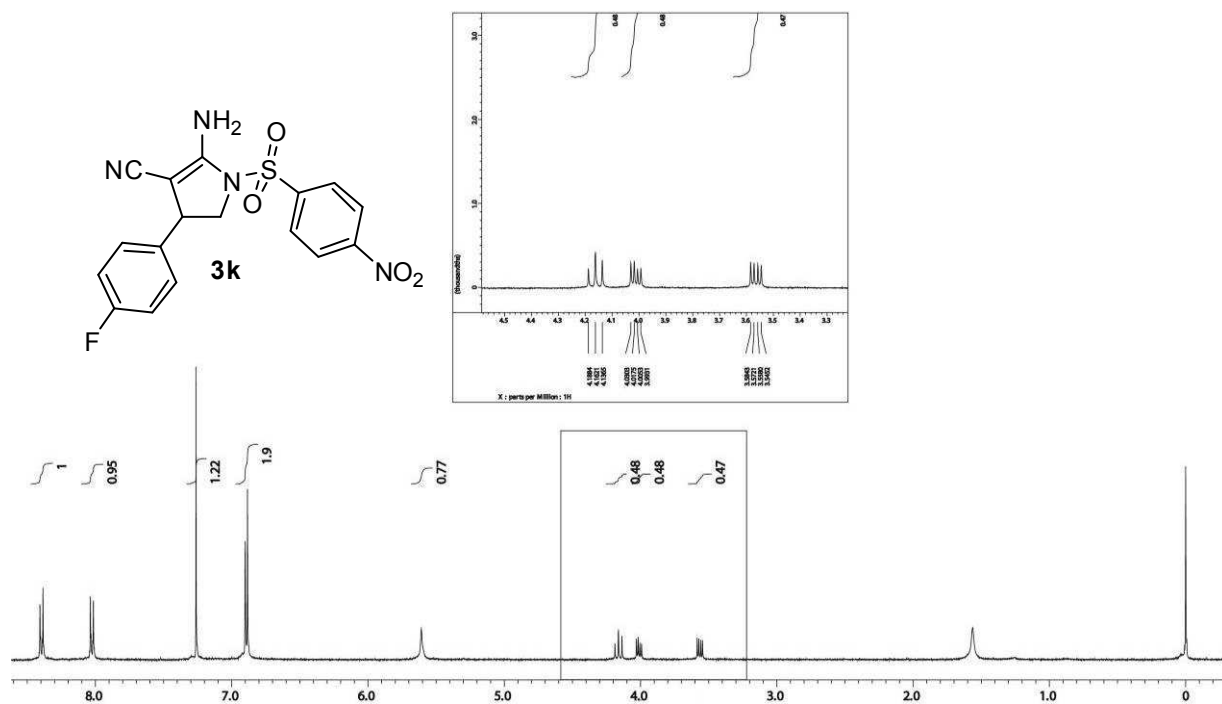


Figure 21: ¹H NMR spectrum of **3k** (CDCl₃, 400 MHz)

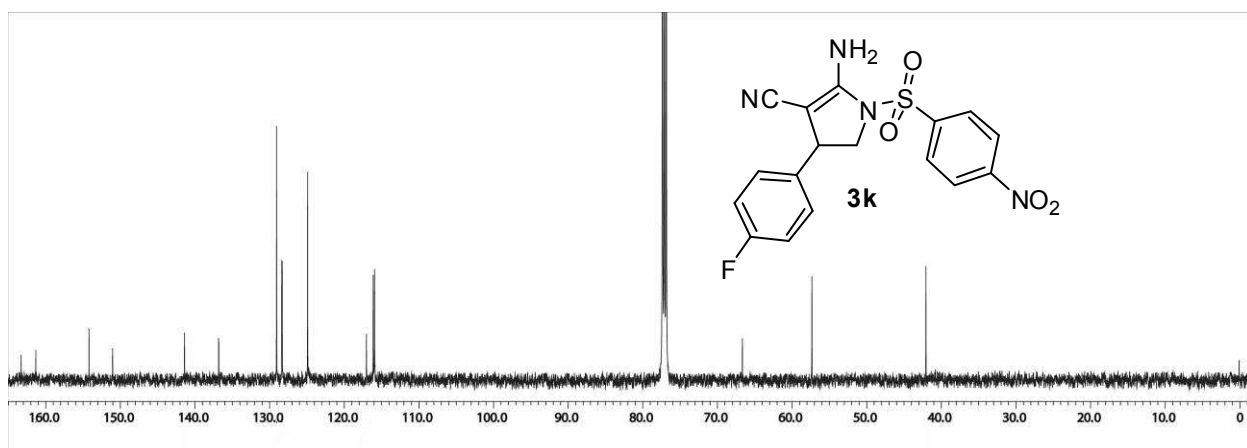


Figure 22: ¹³C NMR spectrum of **3k** (CDCl₃, 125 MHz)

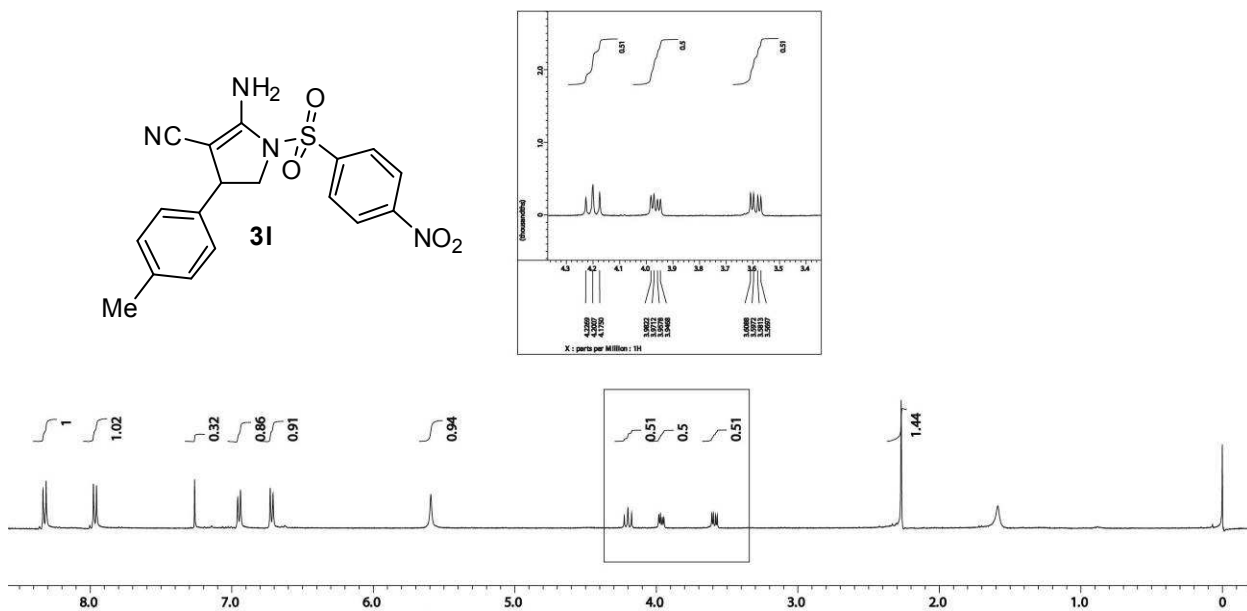


Figure 23: ¹H NMR spectrum of **3I** (CDCl₃, 400 MHz)

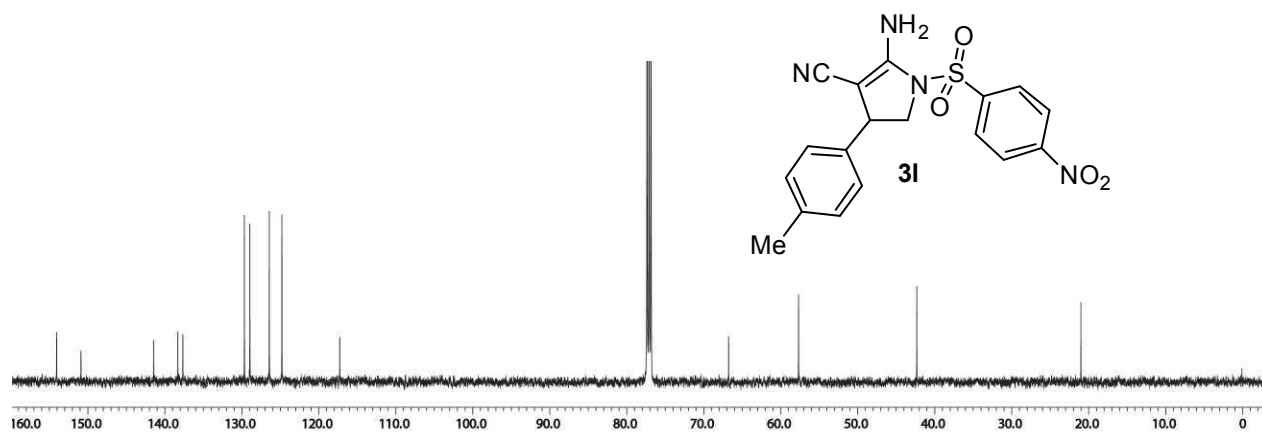


Figure 24: ¹³C NMR spectrum of **3I** (CDCl₃, 125 MHz)

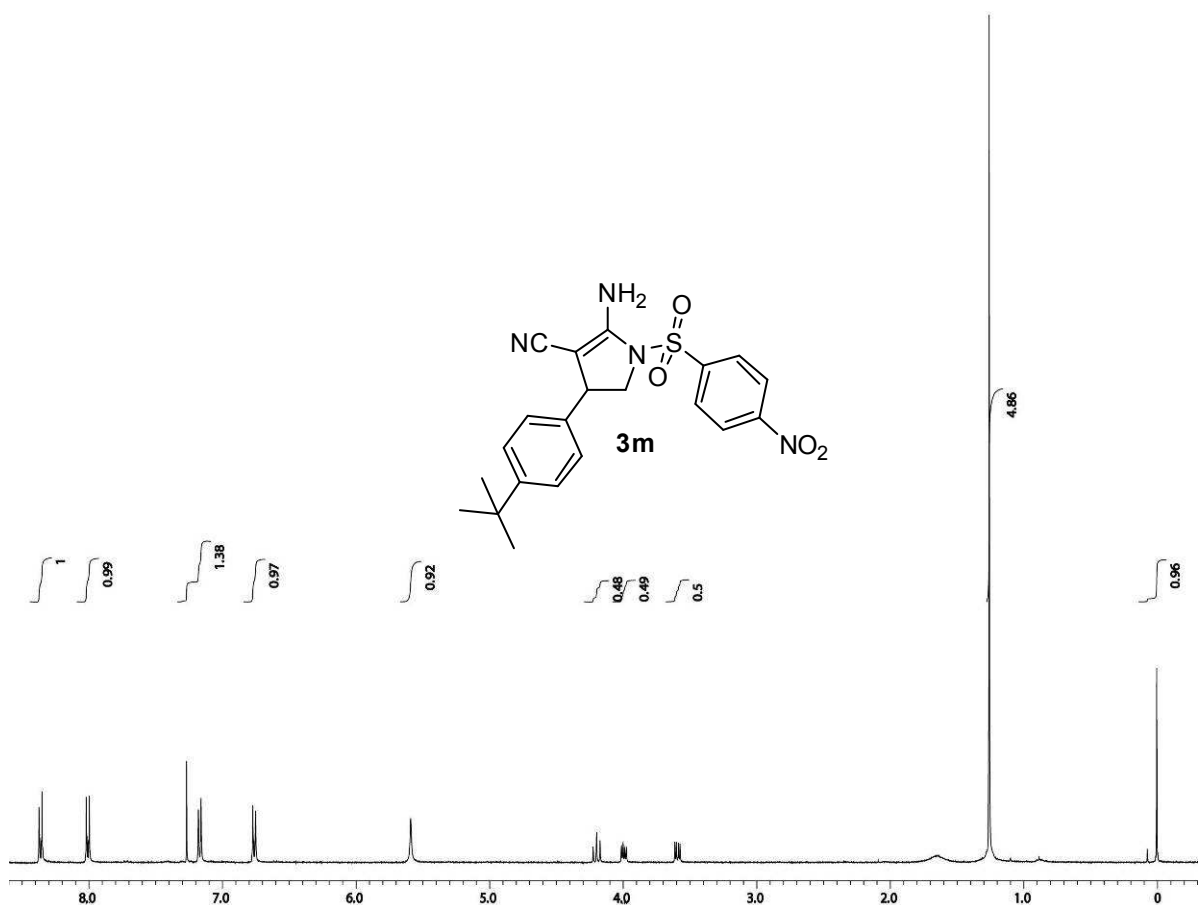


Figure 25: ¹H NMR spectrum of **3m** (CDCl₃, 400 MHz)

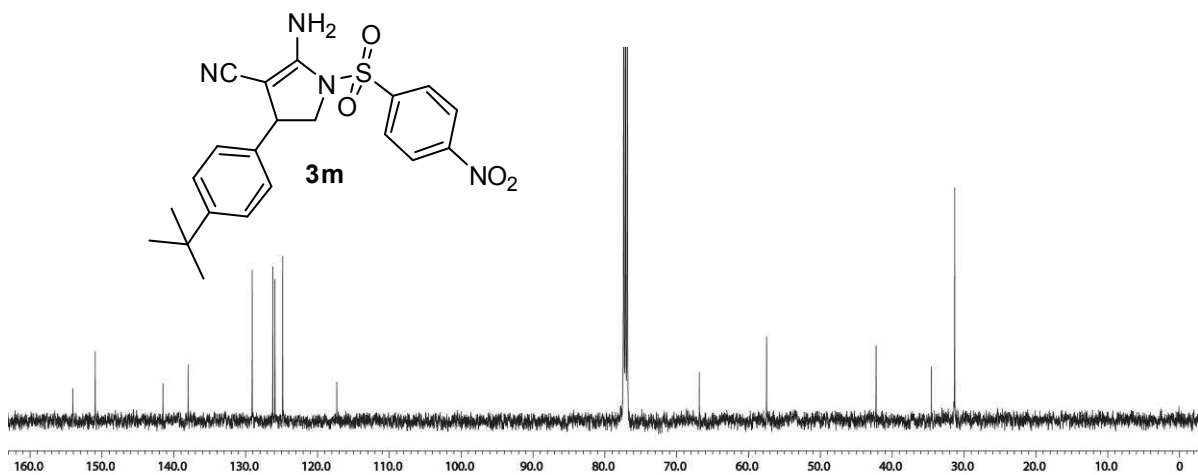


Figure 26: ¹³C NMR spectrum of **3m** (CDCl₃, 125 MHz)

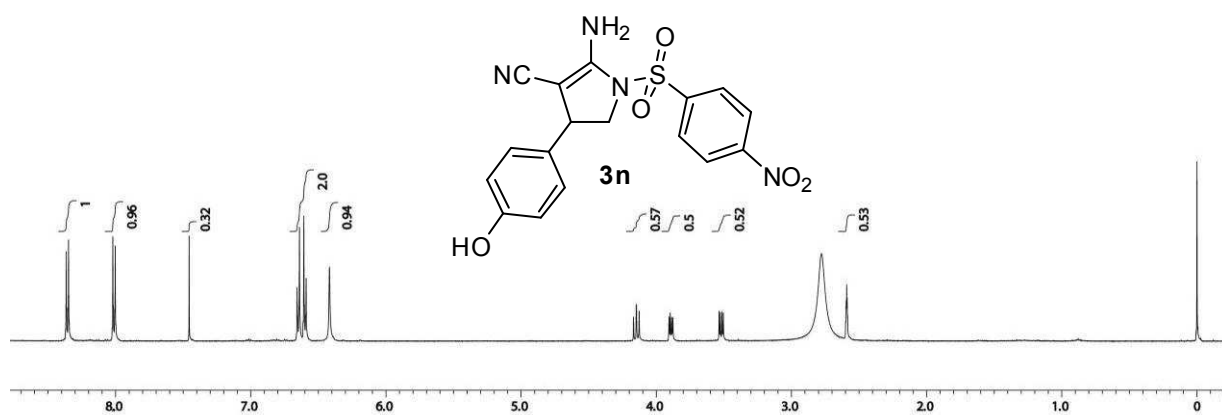


Figure 27: ¹H NMR spectrum of **3n** (CDCl₃+DMSO-d₆, 500 MHz)

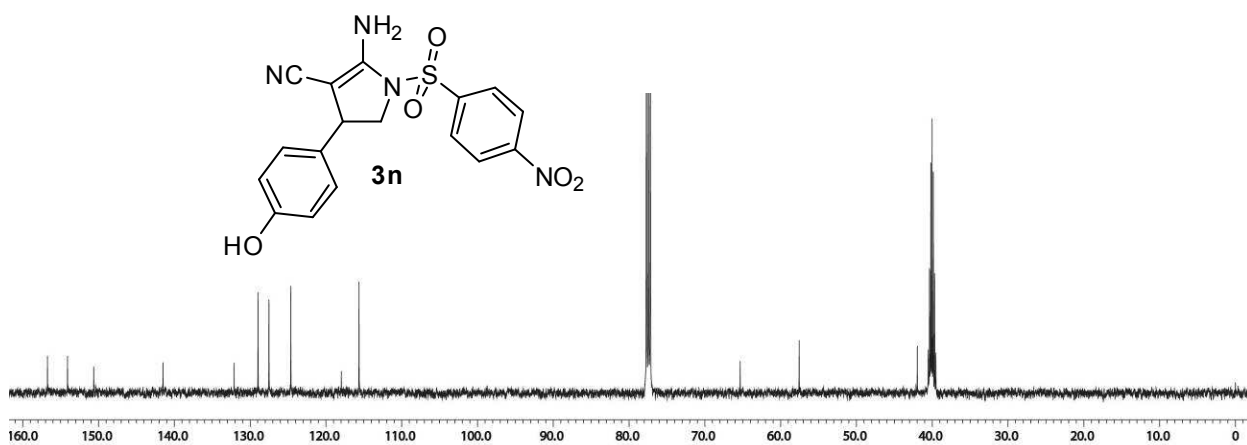


Figure 28: ¹³C NMR spectrum of **3n** (CDCl₃+DMSO-d₆, 125 MHz)

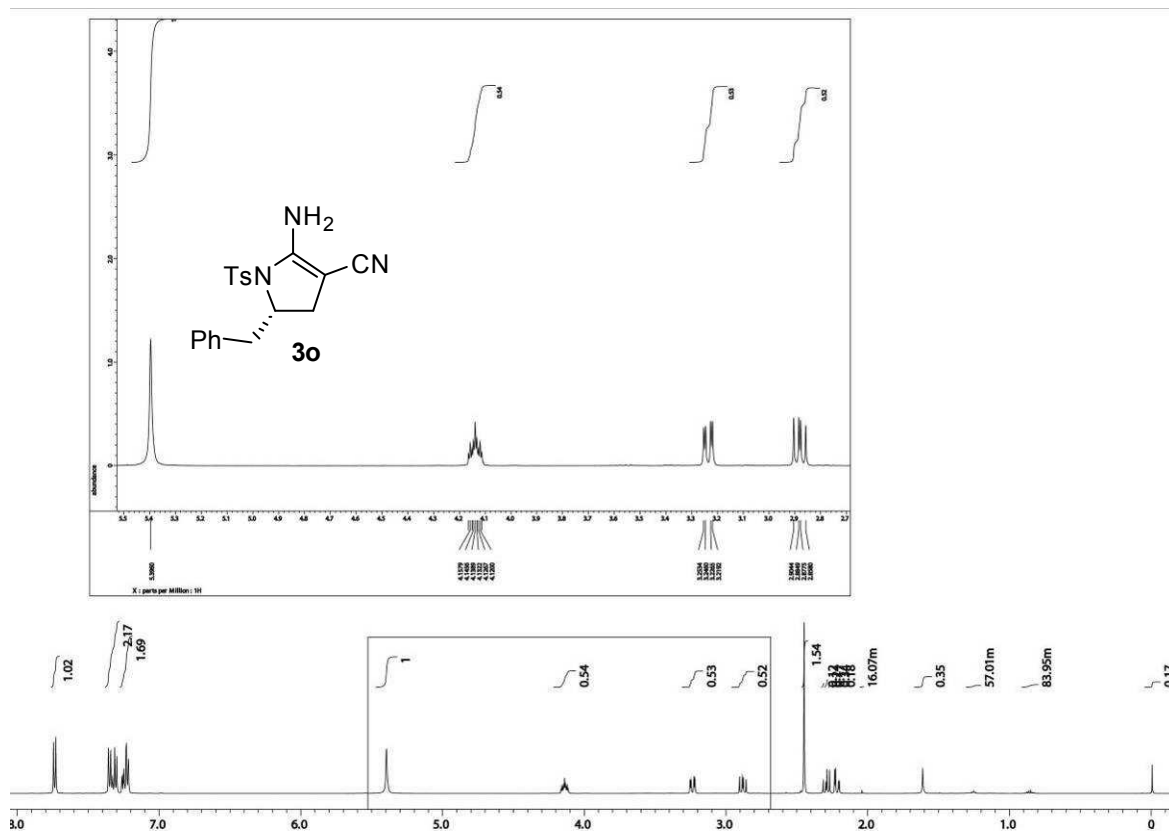


Figure 29: ¹H NMR spectrum of **3o** (CDCl₃, 400 MHz)

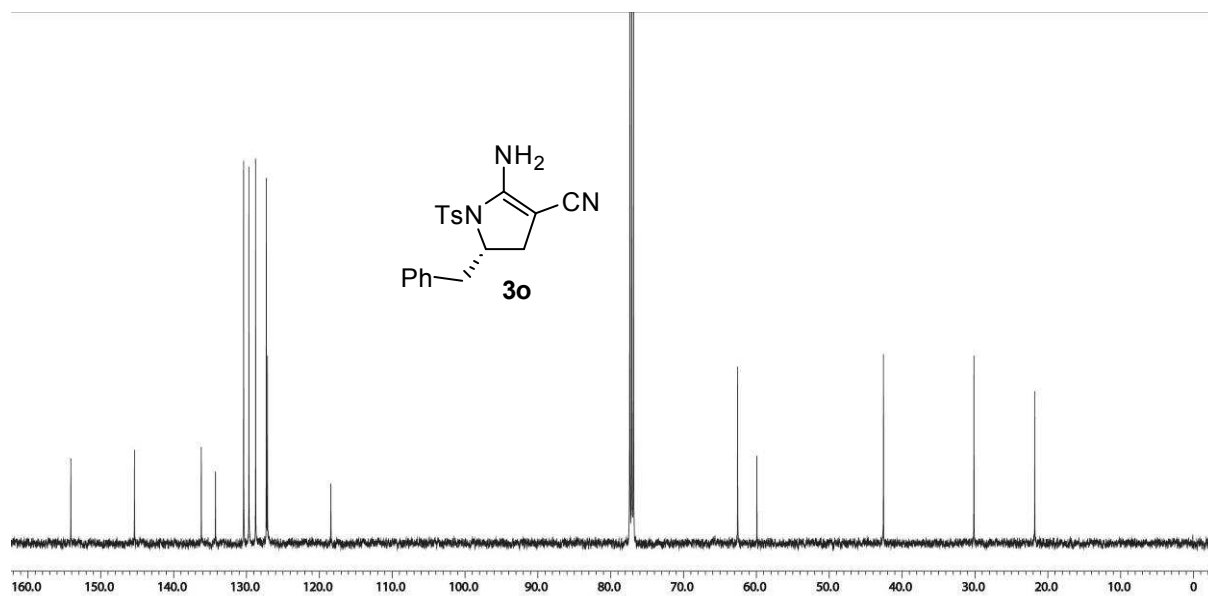


Figure 30: ¹³C NMR spectrum of **3o** (CDCl₃, 125 MHz)

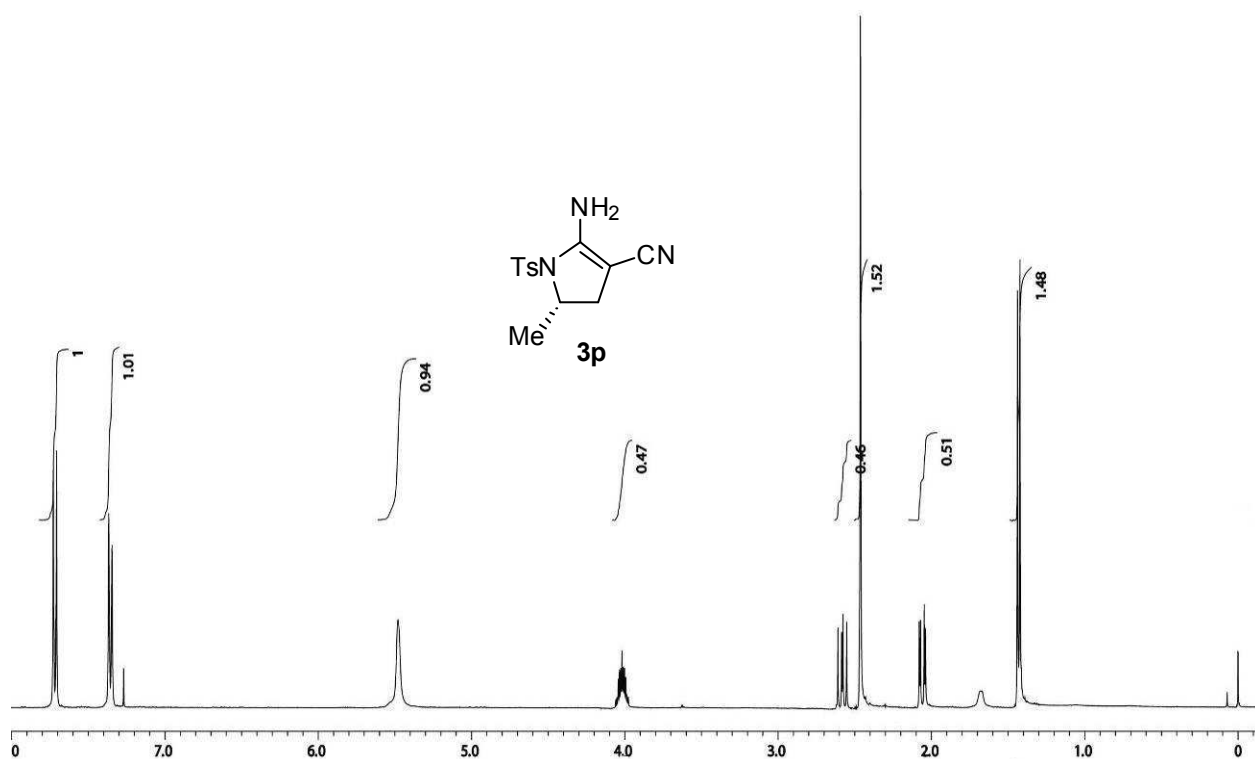


Figure 31: ¹H NMR spectrum of **3p** (CDCl₃, 400 MHz)

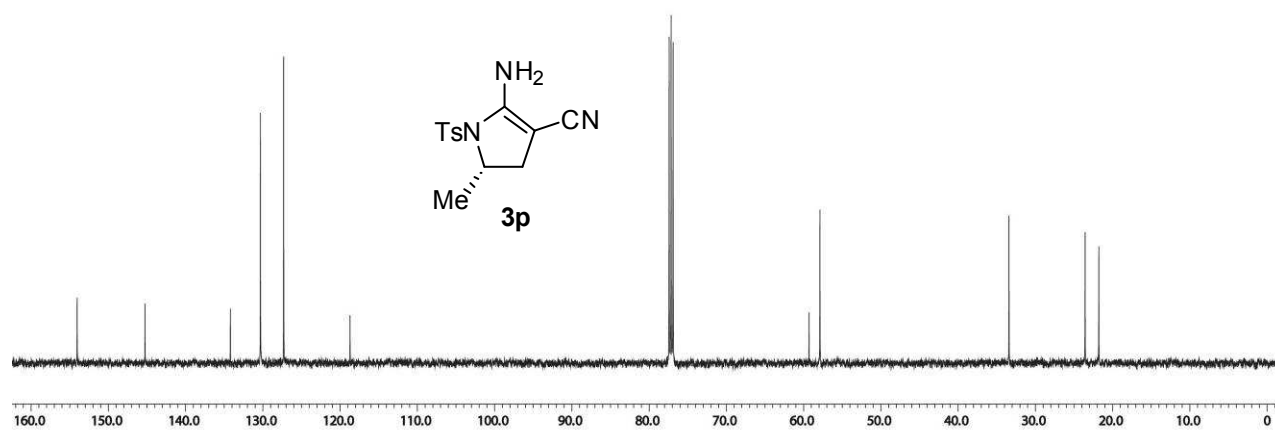


Figure 32: ¹³C NMR spectrum of **3p** (CDCl₃, 125 MHz)

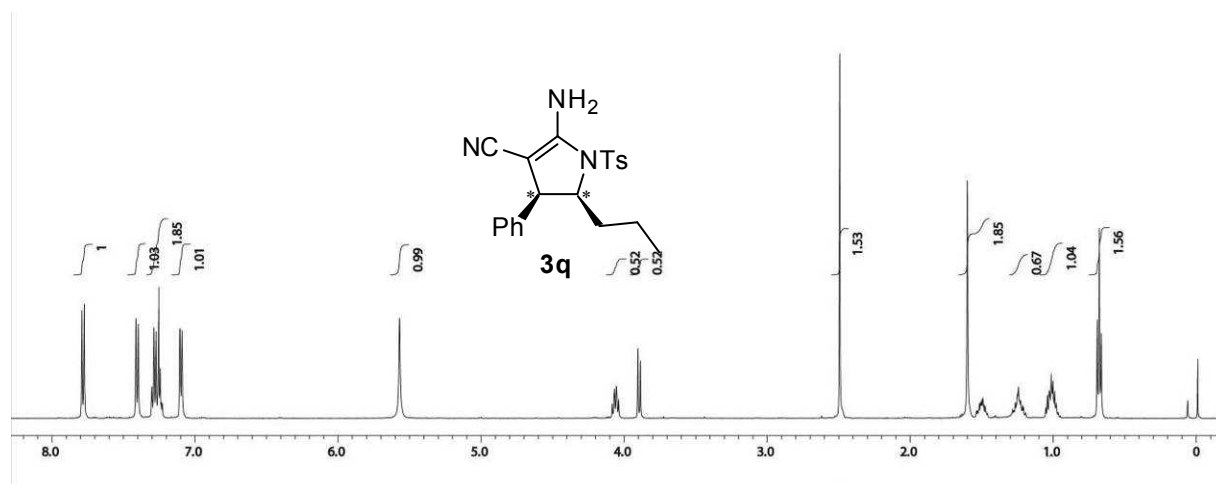


Figure 33: ¹H NMR spectrum of **3q** (CDCl₃, 500 MHz)

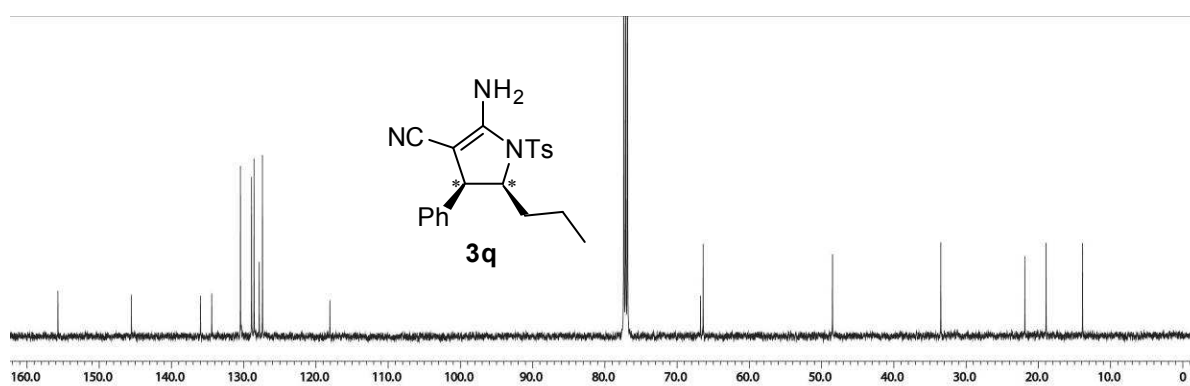


Figure 34: ¹³C NMR spectrum of **3q** (CDCl₃, 125 MHz)

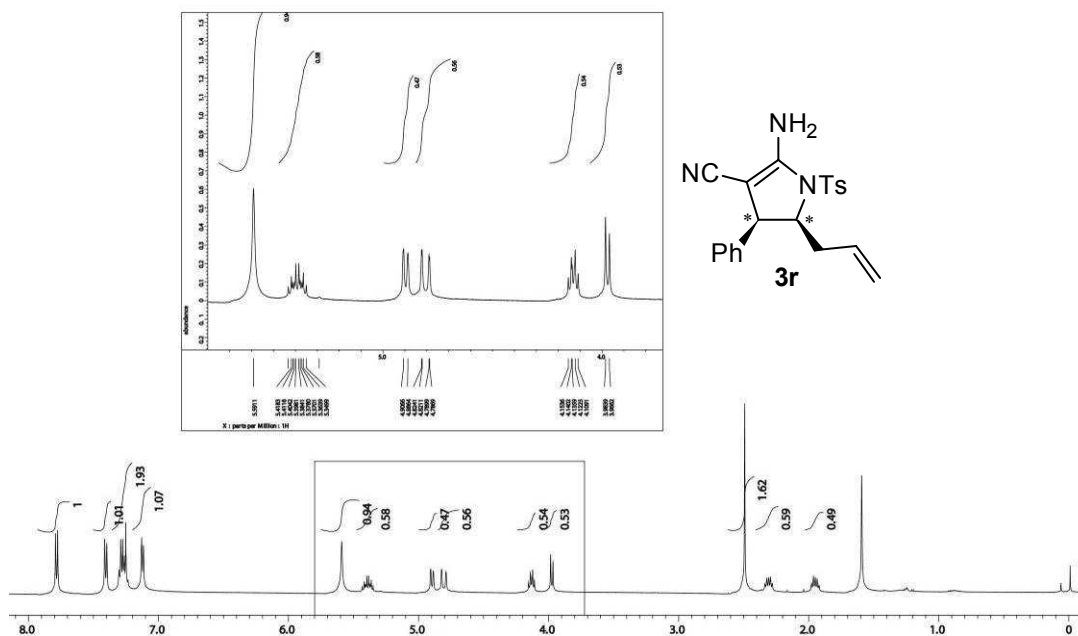


Figure 35: ^1H NMR spectrum of **3r** (CDCl_3 , 500 MHz)

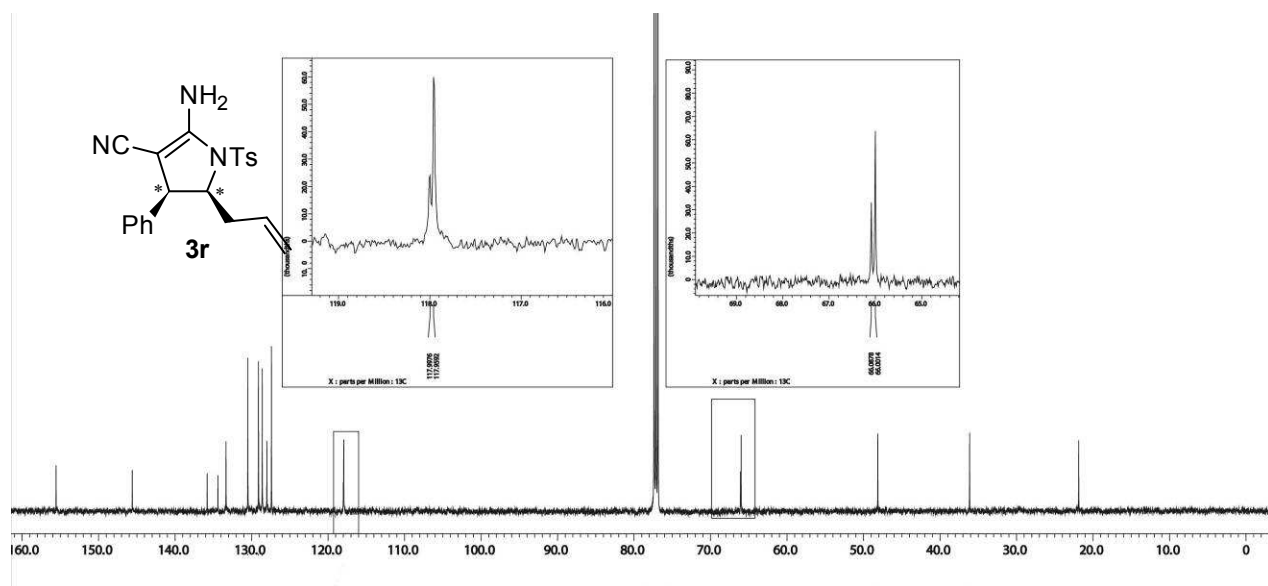


Figure 36: ^{13}C NMR spectrum of **3r** (CDCl_3 , 125 MHz)

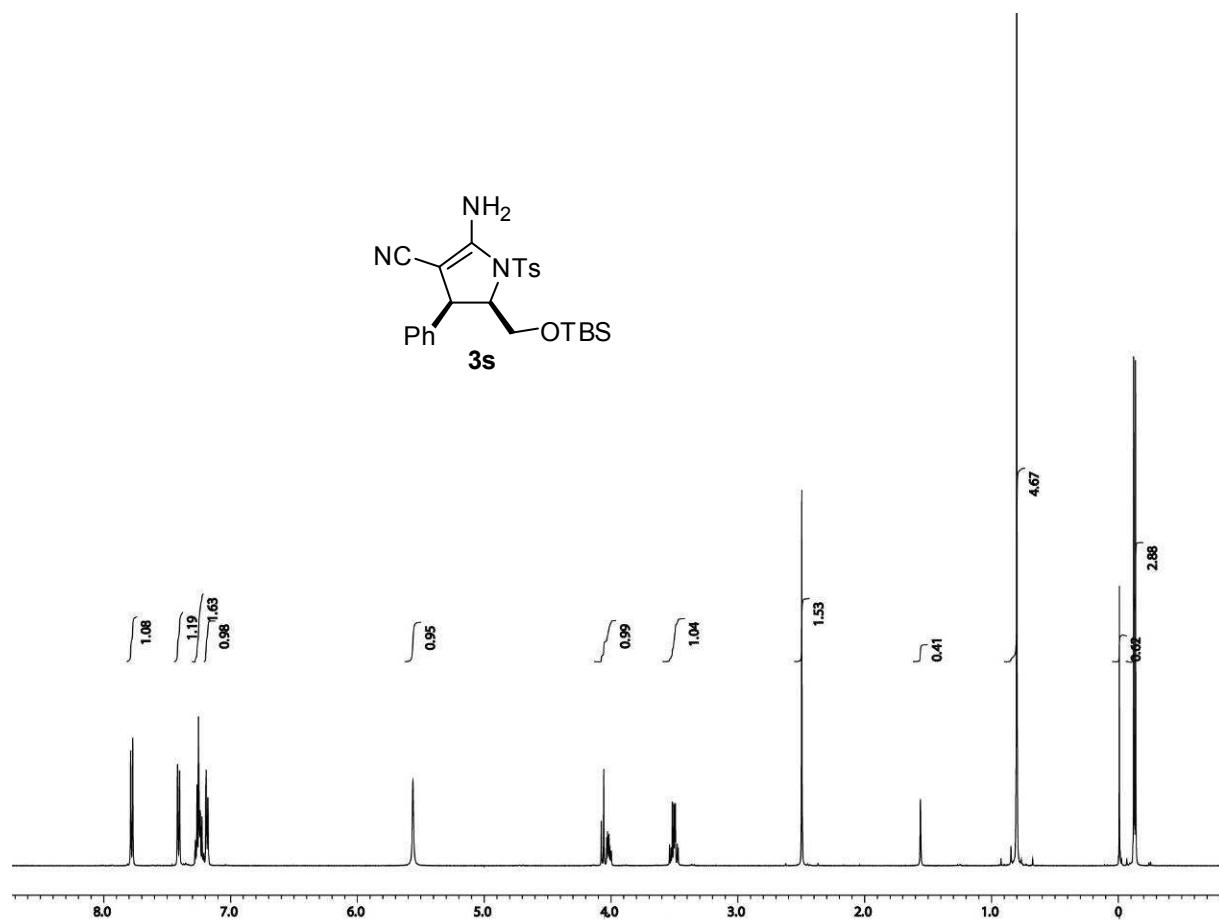


Figure 37: ^1H NMR spectrum of **3s** (CDCl₃, 500 MHz)

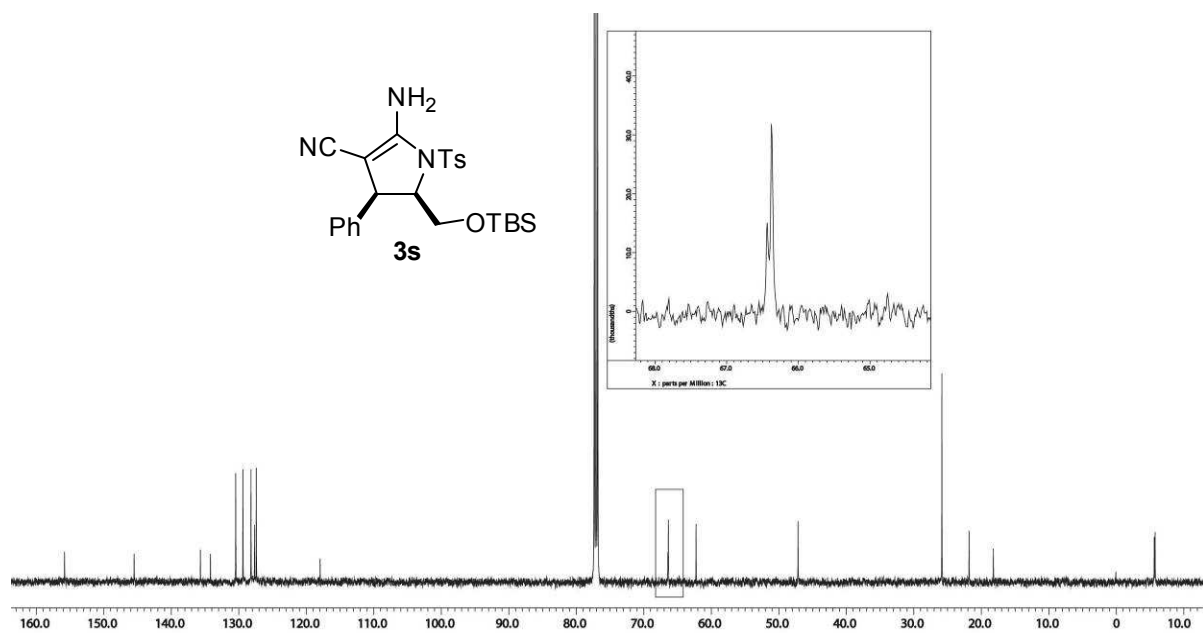


Figure 38: ^{13}C NMR spectrum of **3s** (CDCl_3 , 125 MHz)

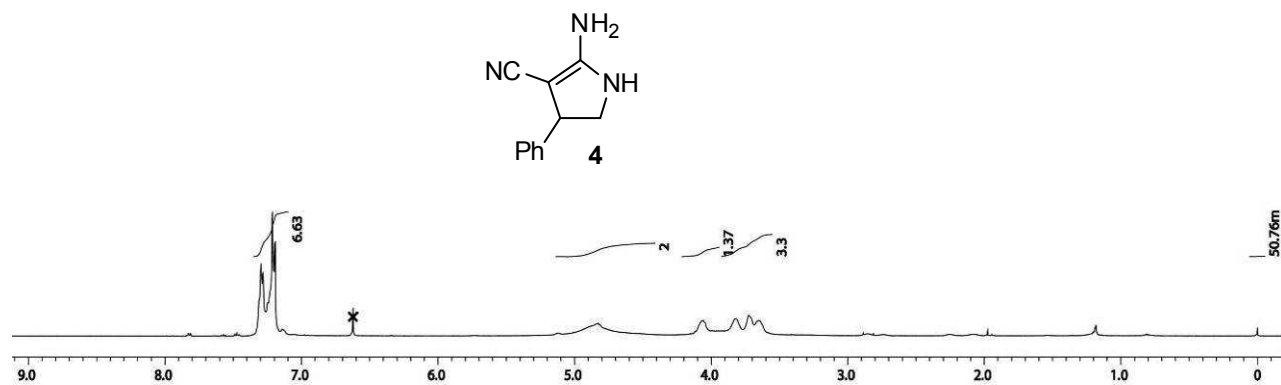


Figure 39: ^1H NMR spectrum of **4** (CDCl_3 , 500 MHz)

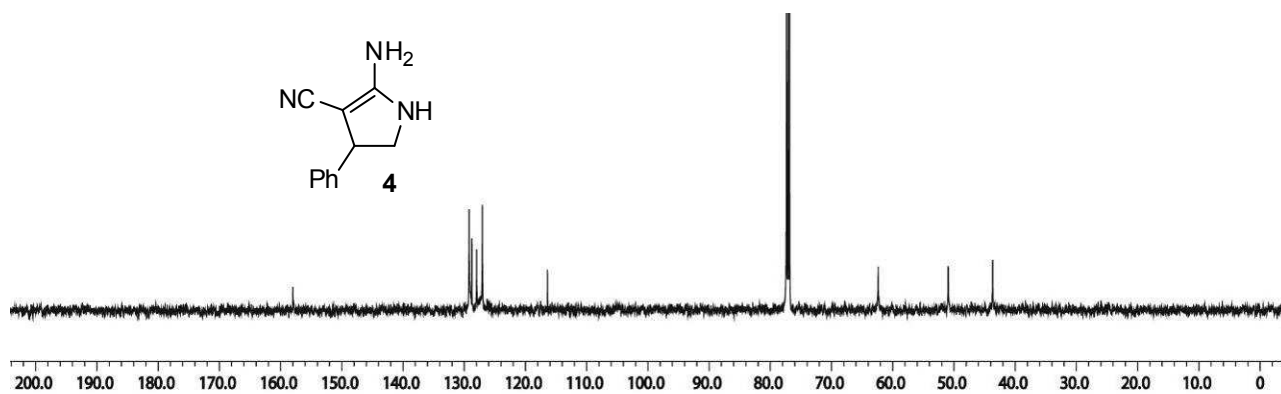


Figure 40: ^{13}C NMR spectrum of **4** (CDCl_3 , 125 MHz)

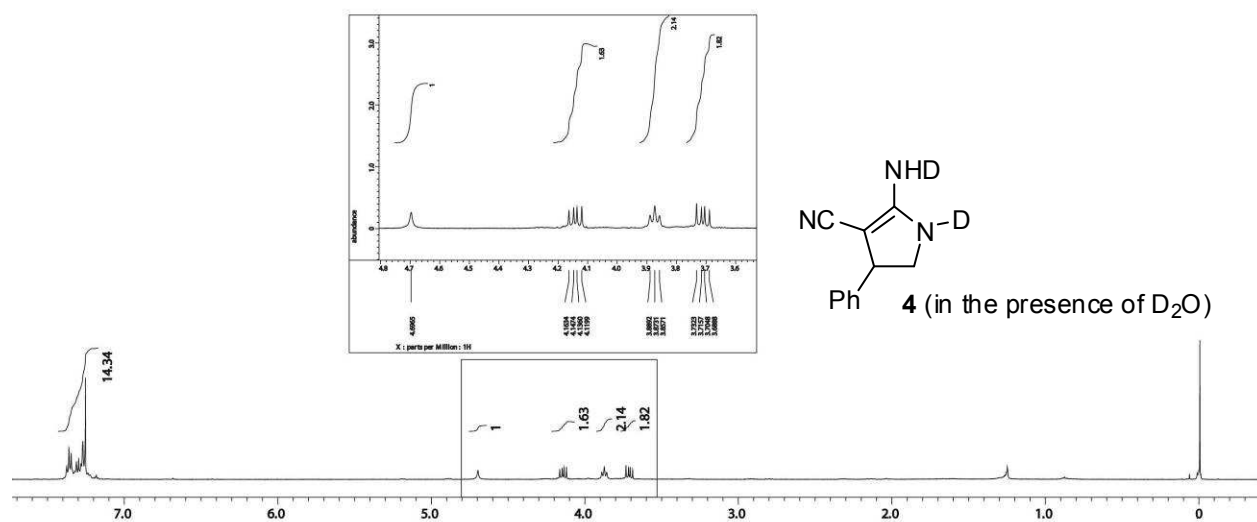


Figure 41: ¹H NMR spectrum of **4** (CDCl₃+D₂O, 500 MHz)

7. NOE spectra of **3r**.

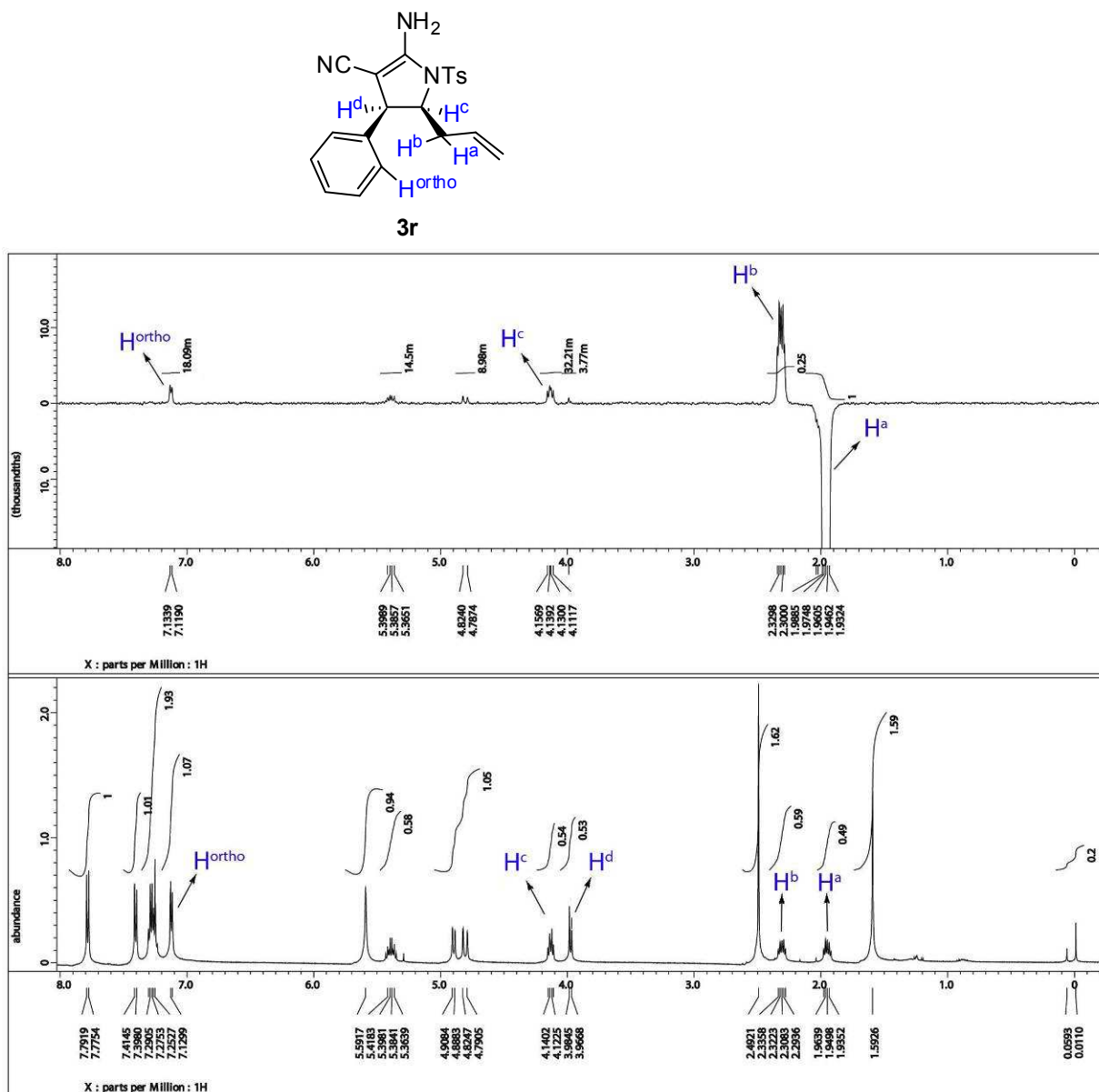
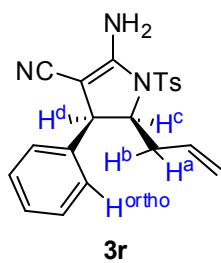


Figure 42. NOE spectrum of **3r** showing *trans* orientation of between H^a and H^d
 (when H^a is irradiated, intensity of H^d does not enhance)



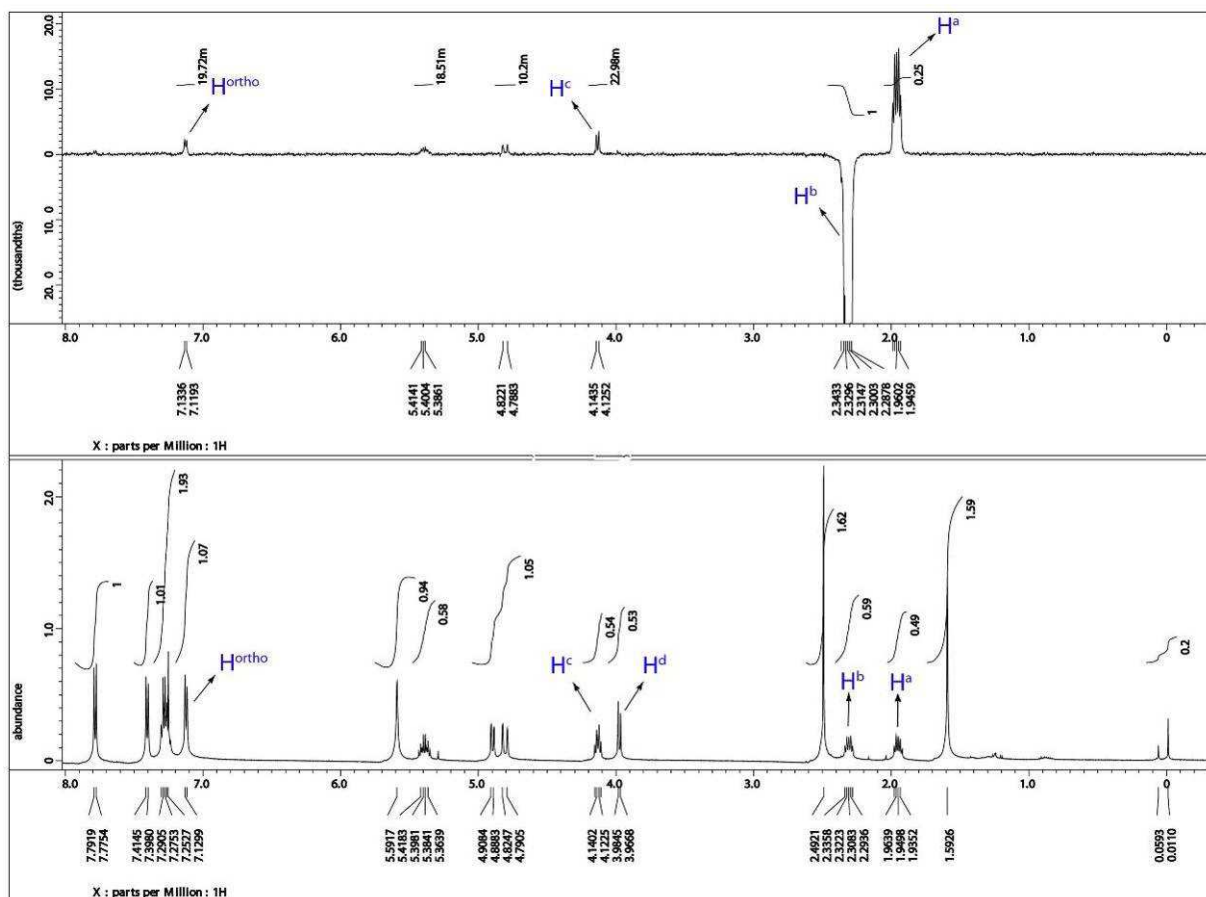
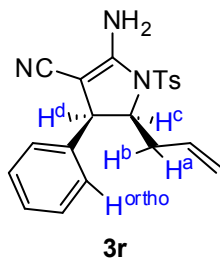


Figure 43. NOE spectrum of **3r** showing *trans* orientation of between H^b and H^d
(when H^b is irradiated, intensity of H^d does not enhance)



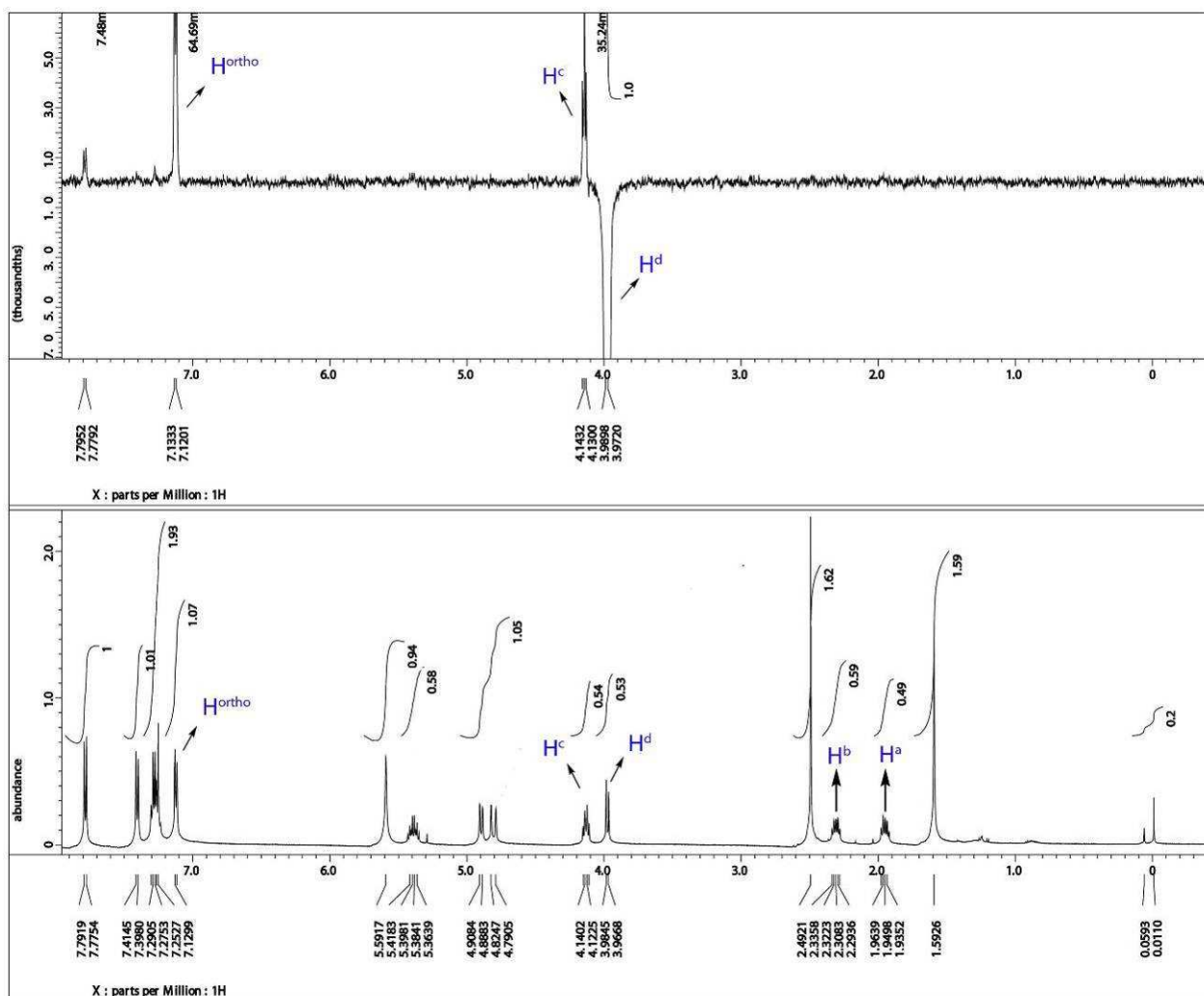


Figure 44. NOE spectrum of **3r** showing *trans* orientation of between H^d and H^a or H^b (when H^d is irradiated, intensities of H^a and H^b does not enhance)

8. Selected HPLC chromatograms.

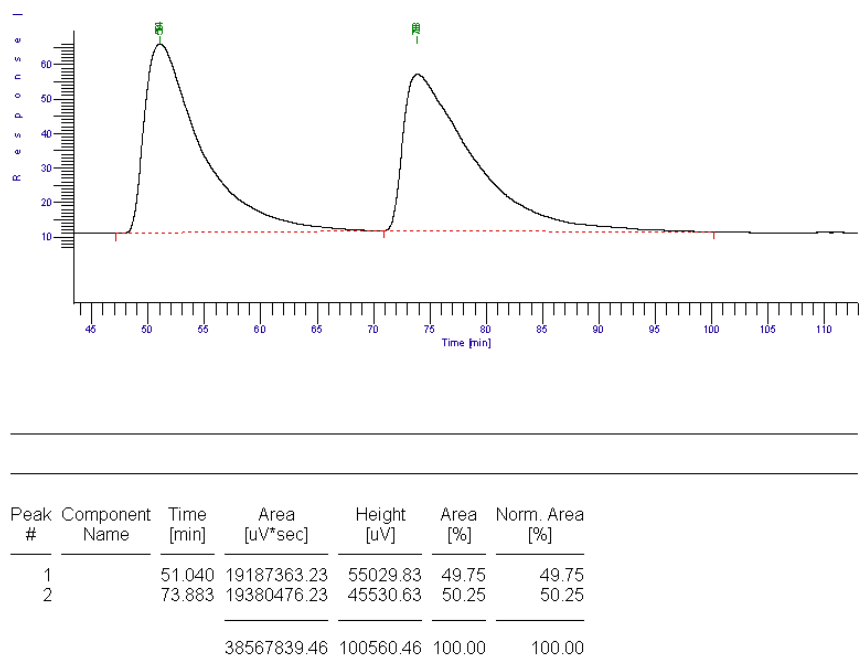


Figure 45. HPLC chromatogram of racemic compound **3d** (AS-H column; 95:5 Hexane-Isopropanol; 1.0 mL min⁻¹).

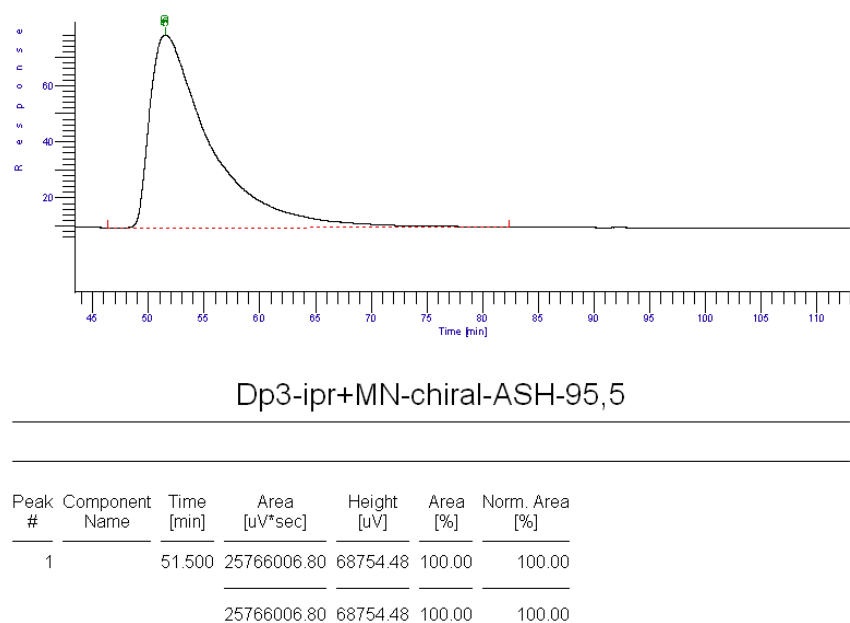
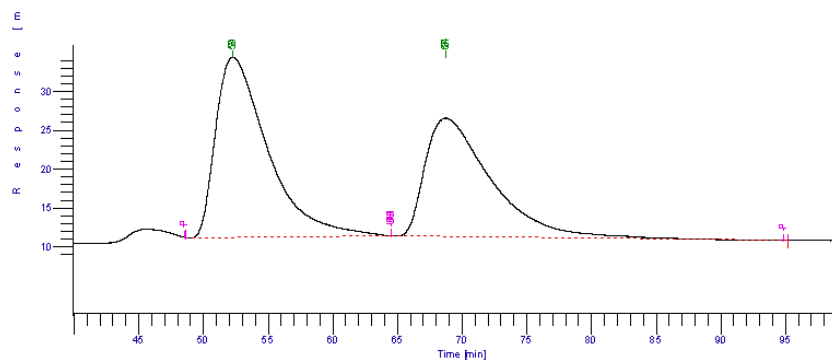


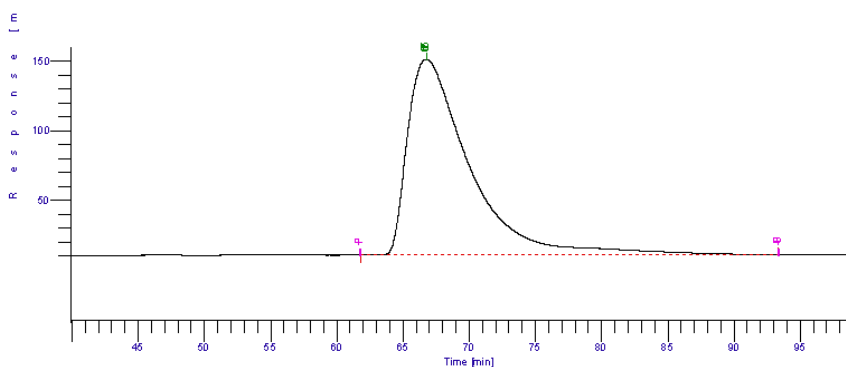
Figure 46. HPLC chromatogram of non-racemic compound (*S*)-**3d** (AS-H column; 95:5 Hexane-Isopropanol; 1.0 mL min⁻¹).



DP3-MN-Ns-rac-OD-H-90,10

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]
1		52.292	6481506.30	23220.78	54.47	54.47
2		68.737	5416833.30	15268.19	45.52	45.52
3		111.095	1893.10	7621.00	0.02	0.02
			11900232.70	46109.97	100.00	100.00

Figure 47. HPLC chromatogram of racemic compound **3g** (OD-H column; 90:10 Hexane-Isopropanol; 1.0 mL min⁻¹)



DP3-MN-Ns-chiral-OD-H-90,10

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]
1		66.747	45813900.38	140740.28	100.00	100.00
			45813900.38	140740.28	100.00	100.00

Figure 48. HPLC chromatogram of non-racemic compound (*S*)-**3g** (OD-H column; 90:10 Hexane-Isopropanol; 1.0 mL min⁻¹).