Potassium *tert*-Butoxide-Catalyzed Synthesis of Benzofuroazepines via Cyclization of 2-Alkynyl Benzyloxy Nitriles

Rafaela Gai,[†] *Davi F. Back*[‡] *and Gilson Zeni**[,†]

[†]Laboratório de Síntese, Reatividade, Avaliação Farmacológica e Toxicológica de Organocalcogênios and [‡]Laboratório de Materiais Inorgânicos, CCNE, UFSM, Santa Maria, Rio Grande do Sul, Brazil 97105-900

[*]*gzeni@ufsm.br*

SUPPORTING INFORMATION

Table of Contents

Materials and Methods

Proton nuclear magnetic resonance spectra (^{1}H NMR) were obtained on a NMR spectrometer at 400 MHz. Spectra were recorded in $CDCl_3$ solutions. Chemical shifts are reported in ppm, referenced to the solvent peak of $CDCl_3$ or tetramethylsilane (TMS) as the external reference. Data are reported as follows: chemical shift (δ), multiplicity, coupling constant *(J)* in Hertz and integrated intensity. Carbon-13 nuclear magnetic resonance spectra (^{13}C NMR) were obtained on a 400 NMR spectrometer at 100 MHz. Spectra were recorded in $CDCl_3$ solutions. Chemical shifts are reported in ppm, referenced to the solvent peak of $CDCl_3$. Abbreviations to denote the multiplicity of a particular signal are s (singlet), d (doublet), t (triplet), q (quartet), quint (quintet), sex (sextet), dt (double triplet), td (triple doublet) and m (multiplet). High resolution mass spectra were recorded on a mass spectrometer using electrospray ionization (ESI). Column chromatography was performed using Silica Gel (230-400 mesh) following the methods described by Still.[1] Thin layer chromatography (TLC) was performed using Gel GF_{254}, 0.25 mm thickness. For visualization, TLC plates were either placed under ultraviolet light, or stained with iodine vapor, or acidic vanillin. Most reactions were monitored by TLC for disappearance of starting material. The following solvents were dried and purified by distillation from the reagents indicated: tetrahydrofuran from sodium with a benzophenone ketyl indicator. All other solvents were ACS or HPLC grade unless otherwise noted. Air- and moisture-sensitive reactions were conducted in flame-dried or oven dried

glassware equipped with tightly fitted rubber septa and under a positive atmosphere of dry nitrogen or argon. Reagents and solvents were handled using standard syringe techniques.

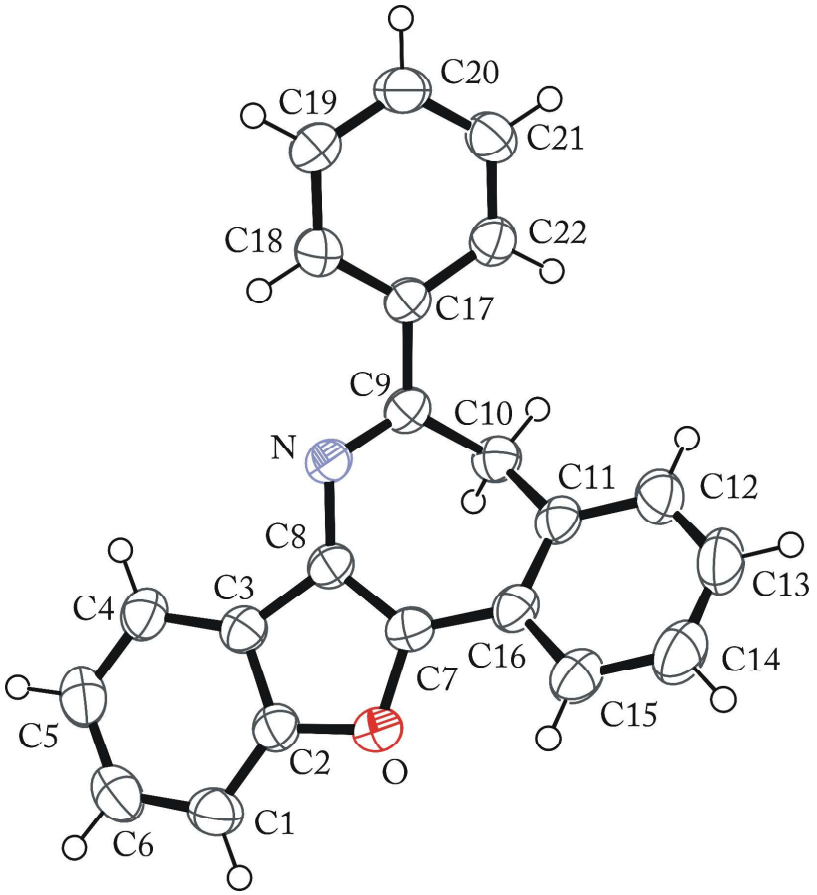


Figure S1. The molecular structure with the atom-labeling scheme of the compound 2a with 50% thermal ellipsoids (using ORTEP software[2]) (CCDC 1413101).

Description of the Solution of the X-ray Structure

Data were collected with a CCD area-detector diffractometer and graphite–monochromatized Mo–K_{α} radiation. The structure was solved by direct methods using SHELXS.[1] Subsequent Fourier-difference map analyses yielded the positions of the non-hydrogen atoms. Refinements were carried out with the SHELXL package.[1] All refinements were made by full-matrix least squares on F^2 with anisotropic displacement parameters for all non–hydrogen atoms. Hydrogen atoms were included in the refinement in calculated positions but the atoms (of

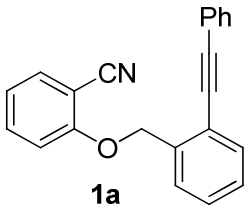
hydrogens) that are commenting performing special bond were located in the Fourier map. Illustration of complexes was done using *ORTEP3* for Windows.[2]

References

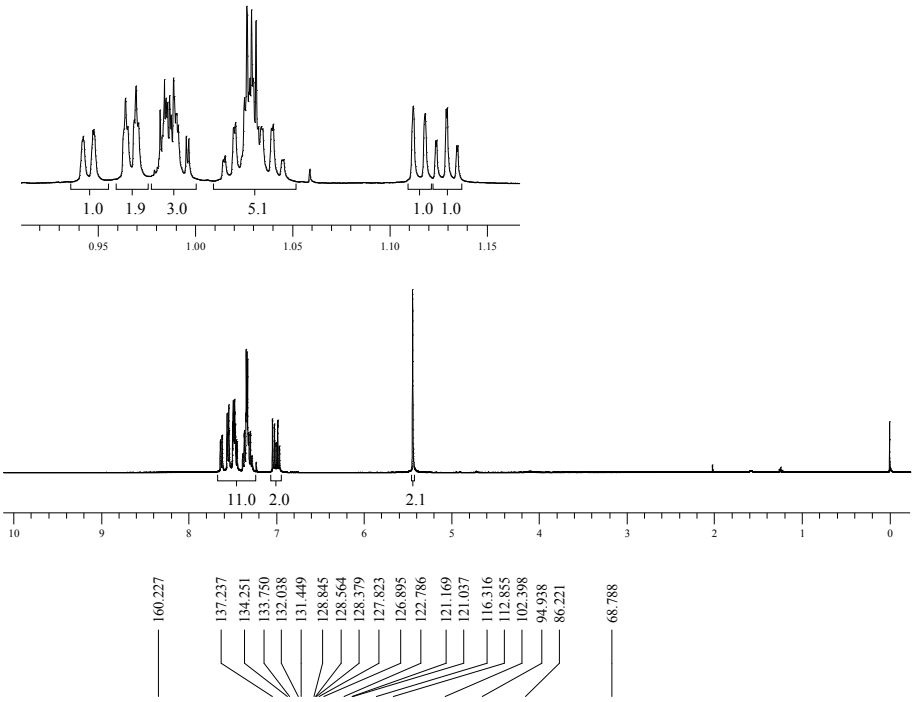
(1). Sheldrick, G. M. *Acta Cryst*. **2008**, *A64*, 111.

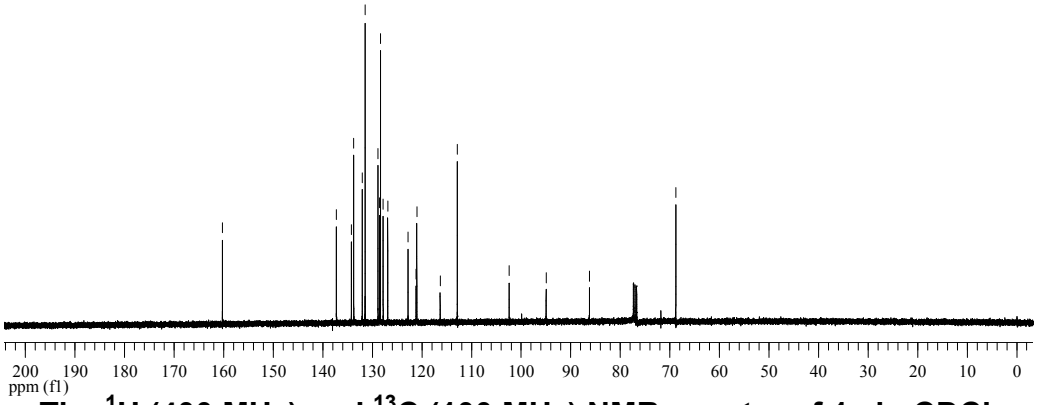
(2). ORTEP3 for Windows - *Farrugia*, *L. J. J. Appl. Crystallogr*. **1997**, *30*, *565*.

SELECTED SPECTRA

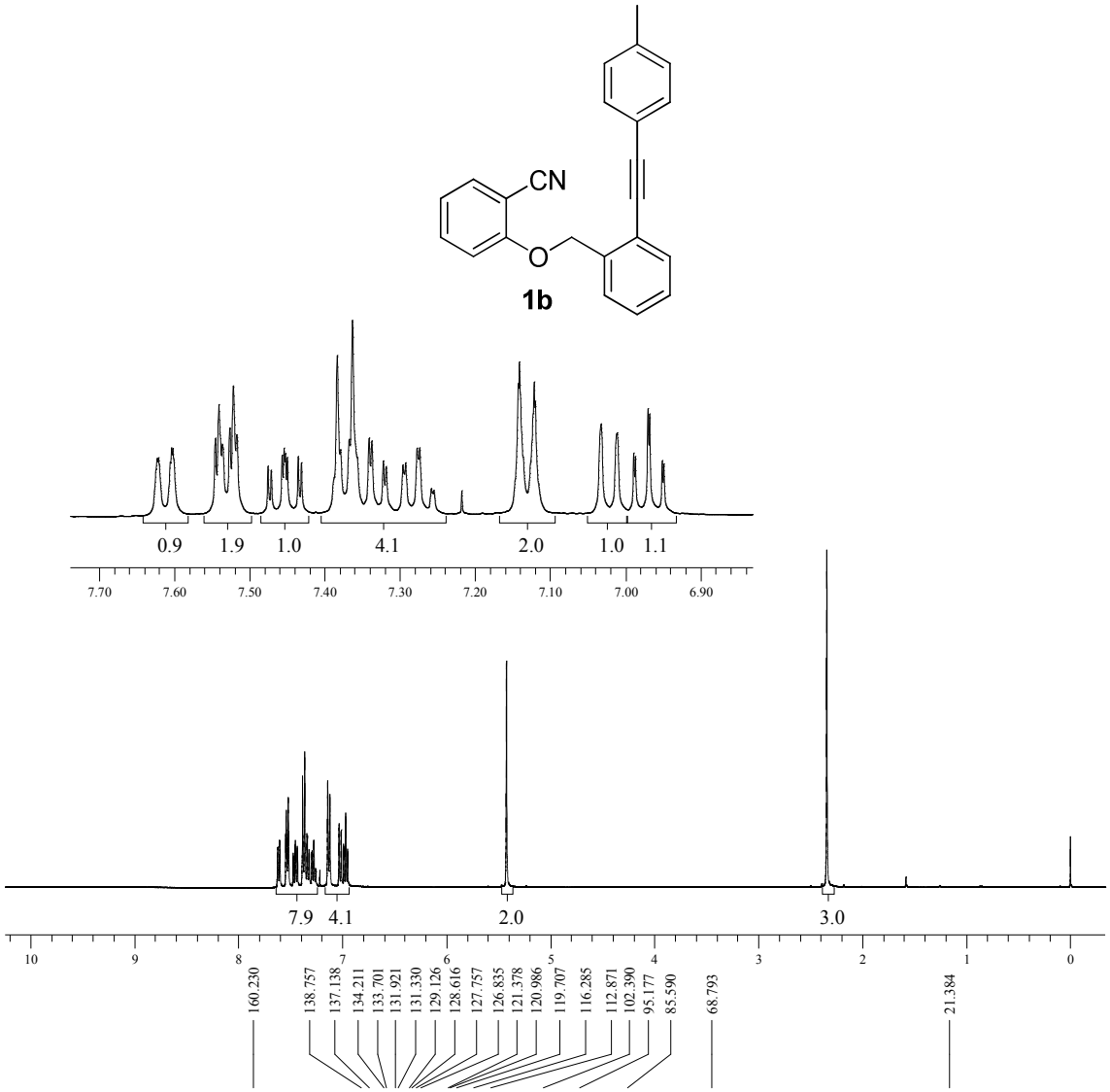


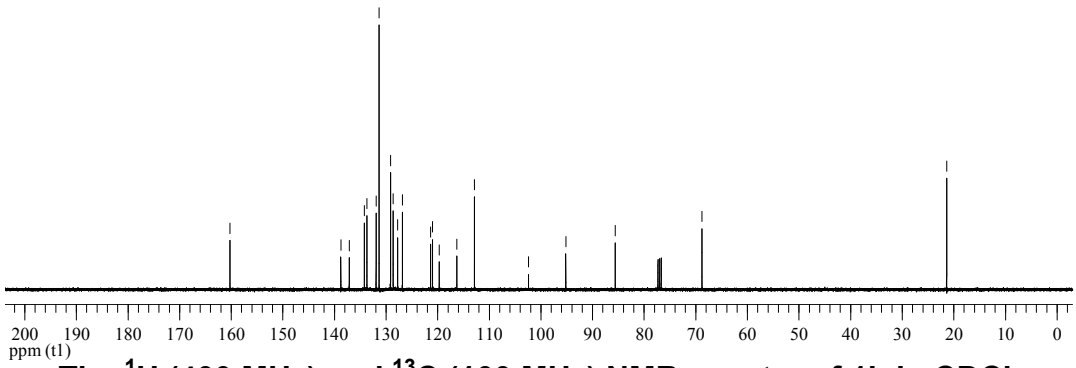
1a



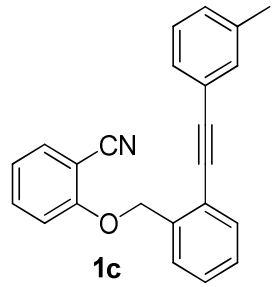


The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 1a in $CDCl_3$

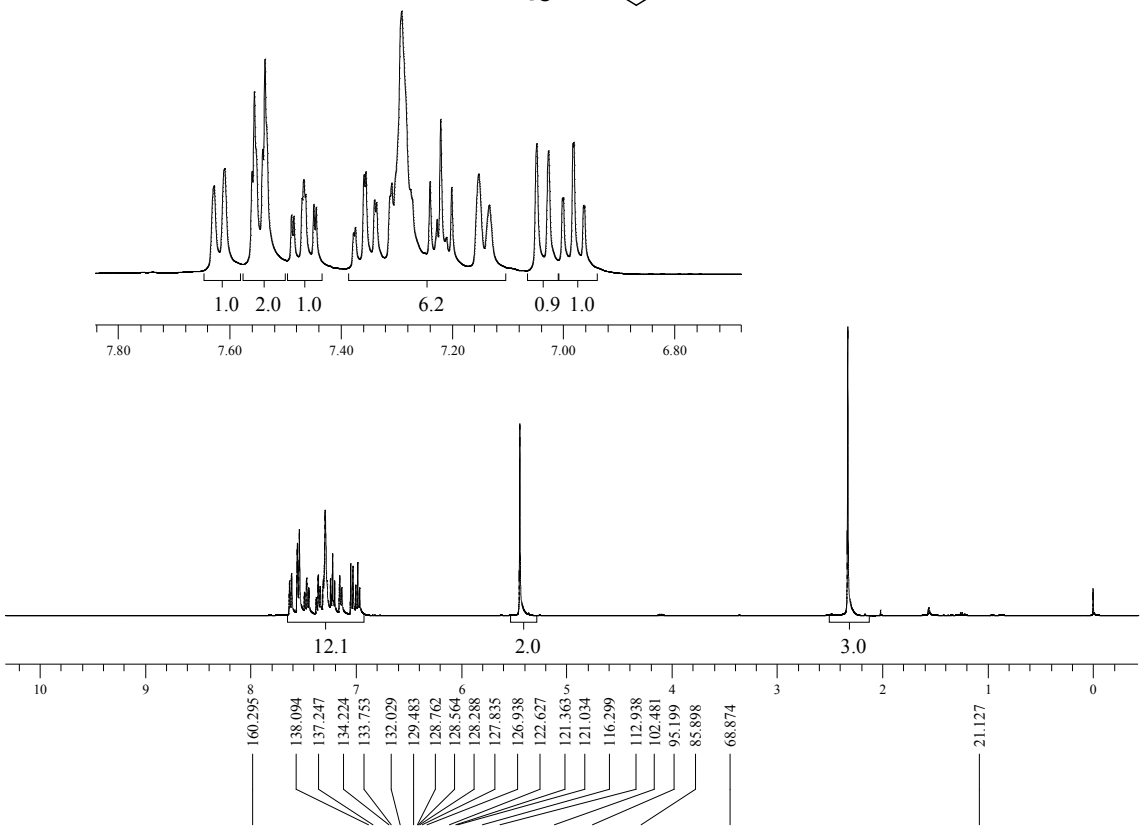


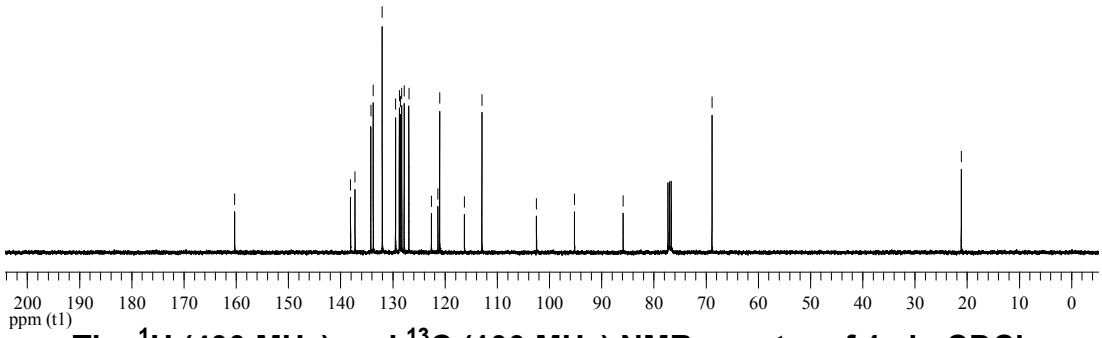


The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 1b in $CDCl_3$

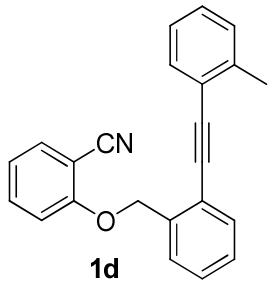


1c

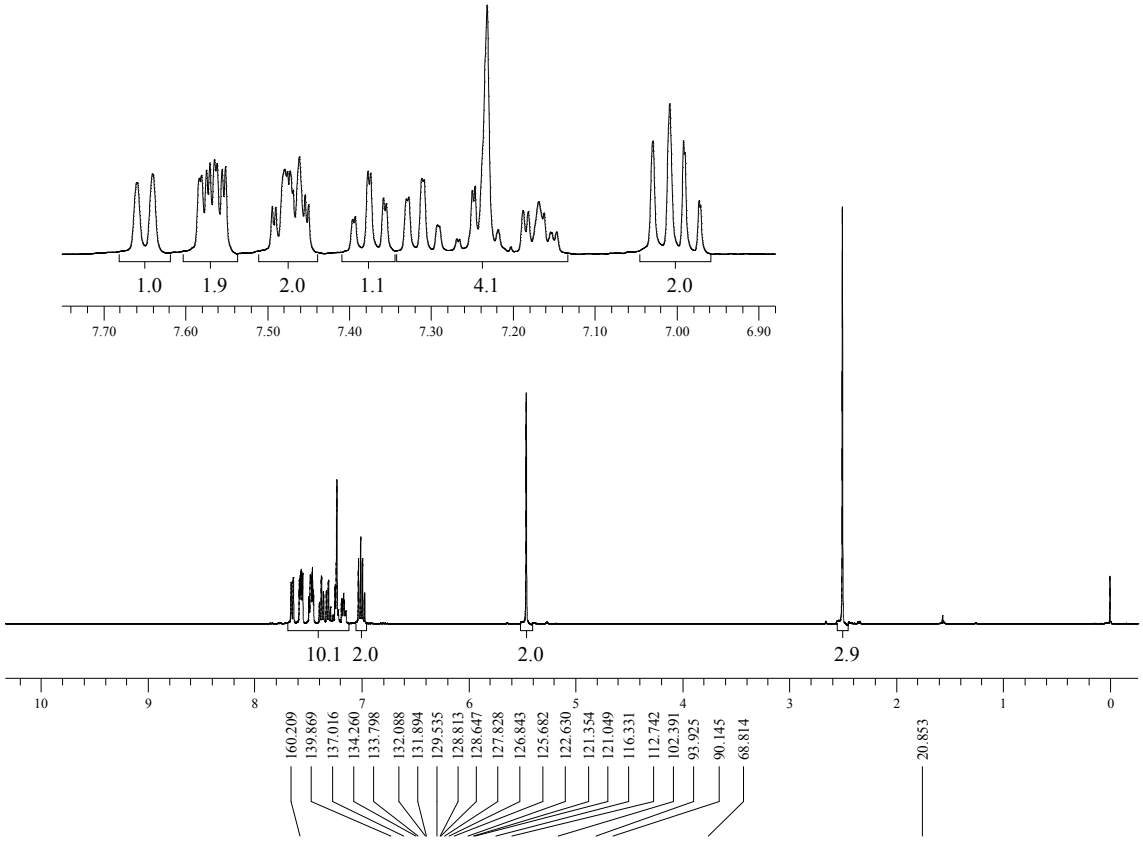


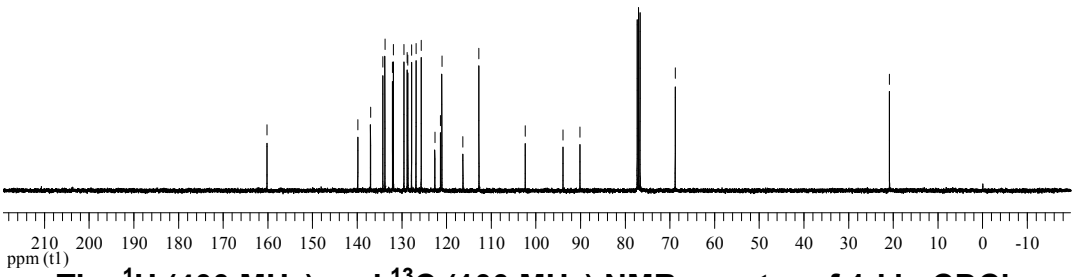


The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 1c in $CDCl_3$

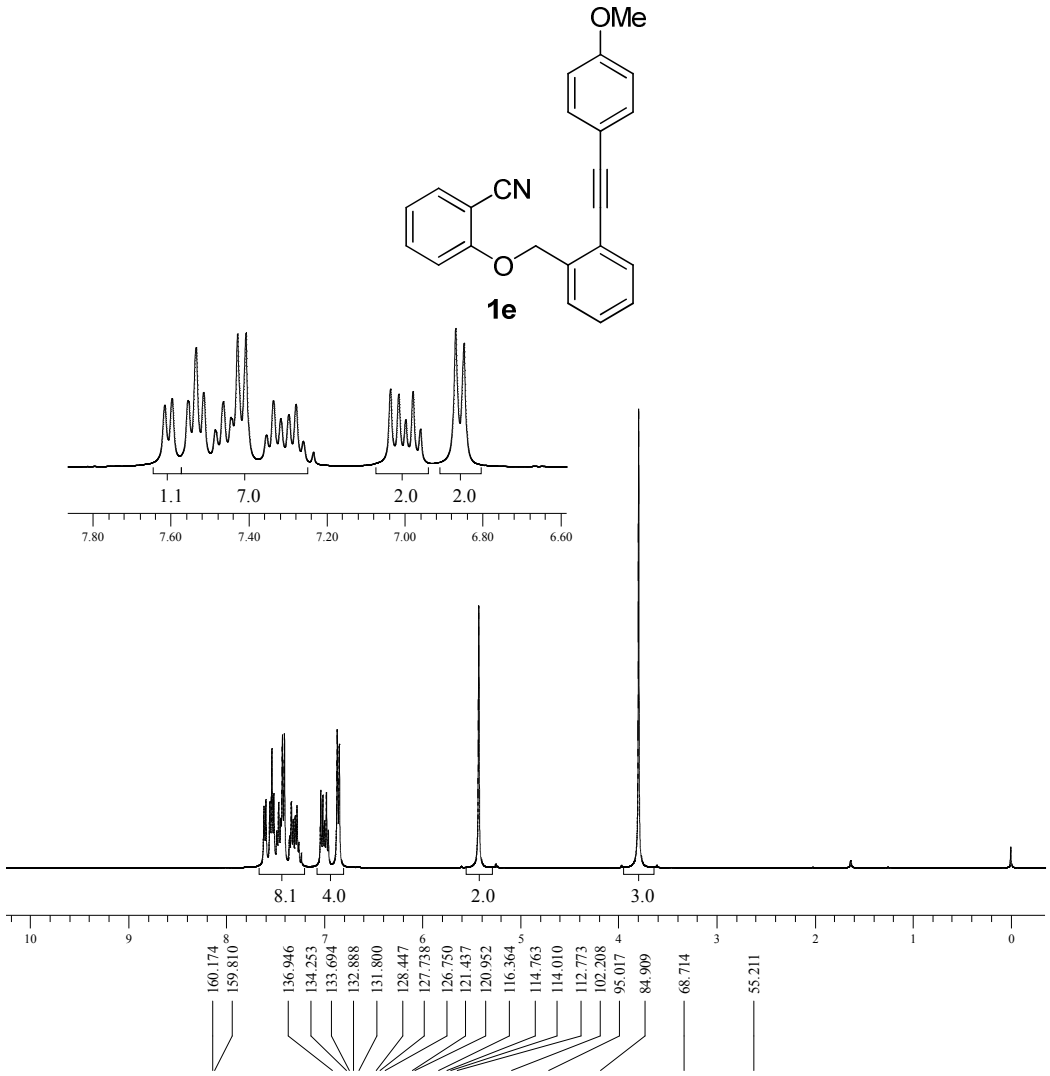


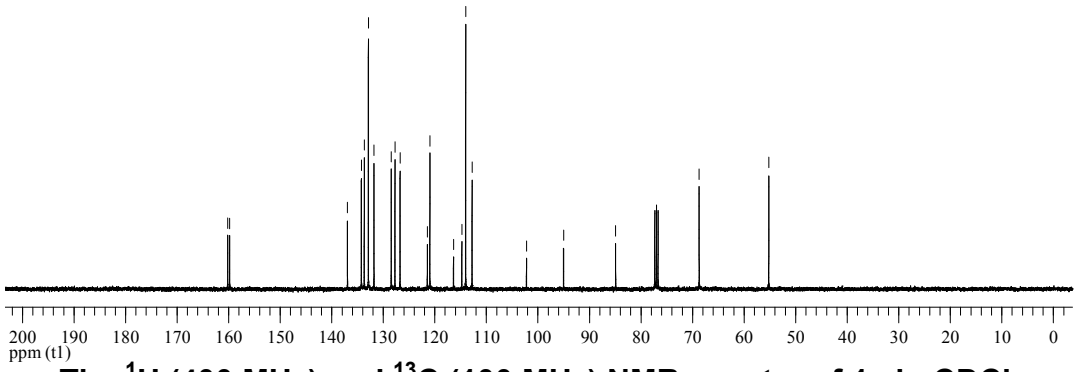
1d



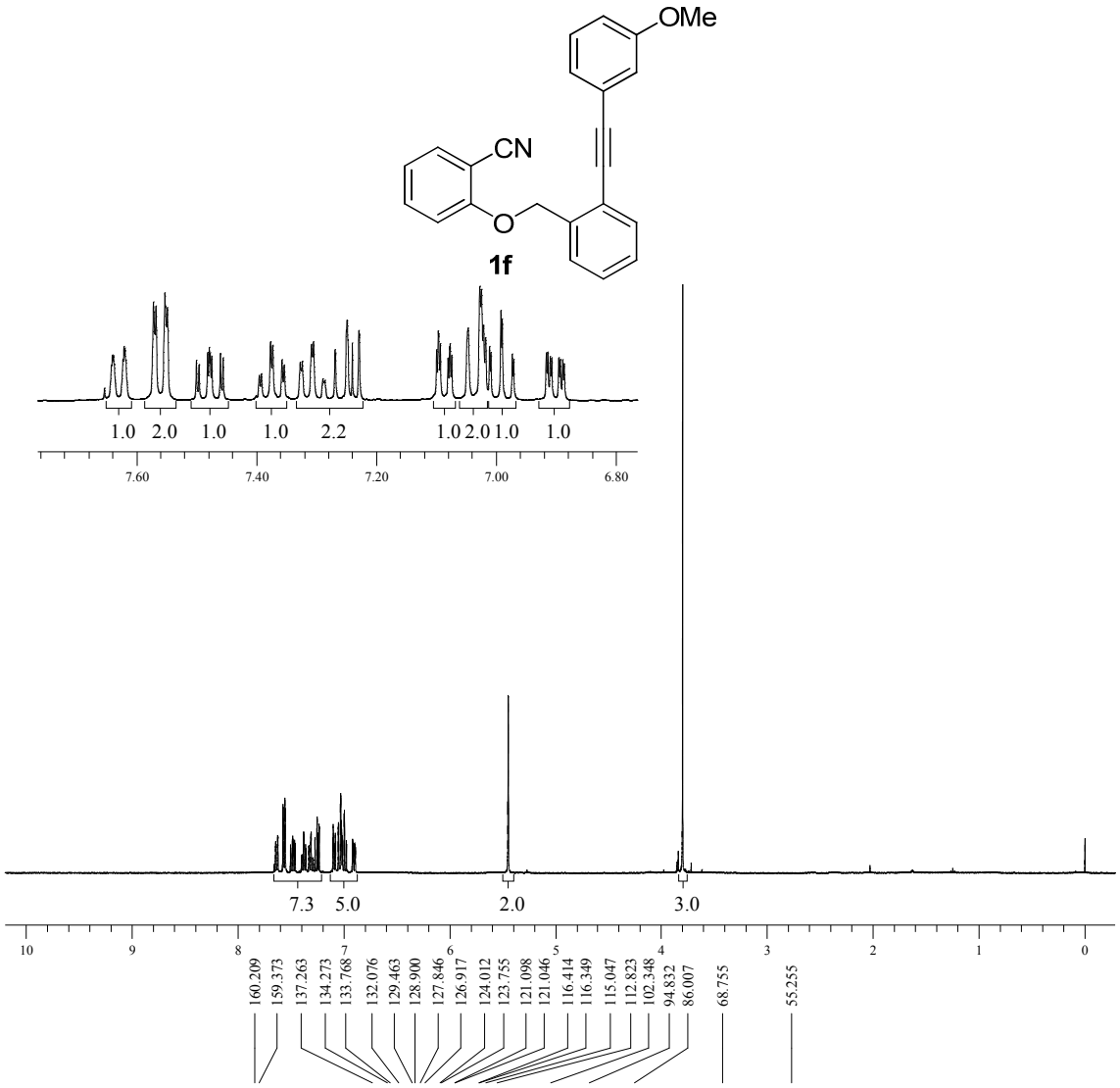


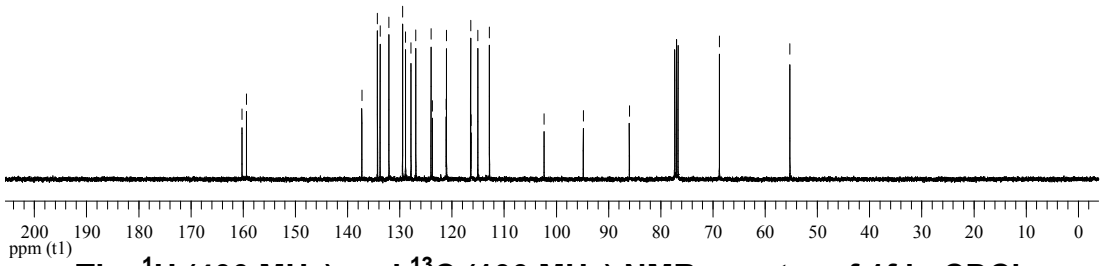
The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 1d in $CDCl_3$



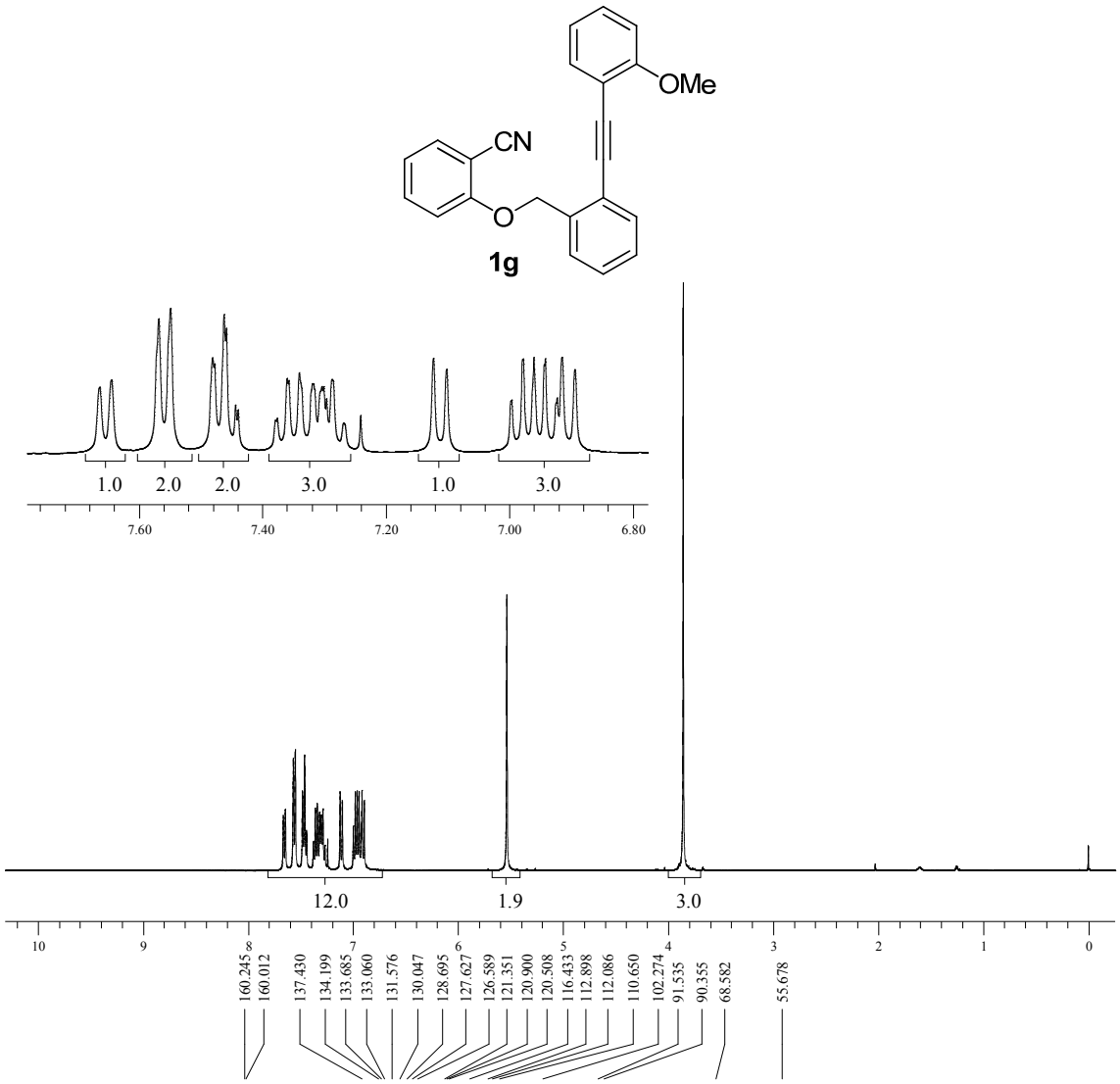


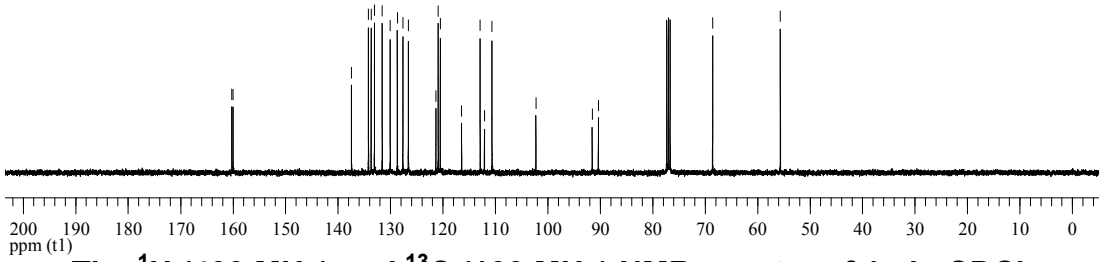
The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 1e in $CDCl_3$



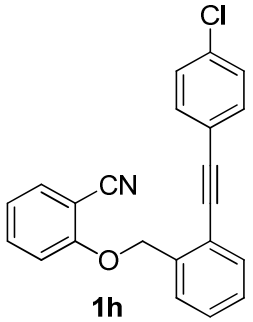


The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 1f in $CDCl_3$

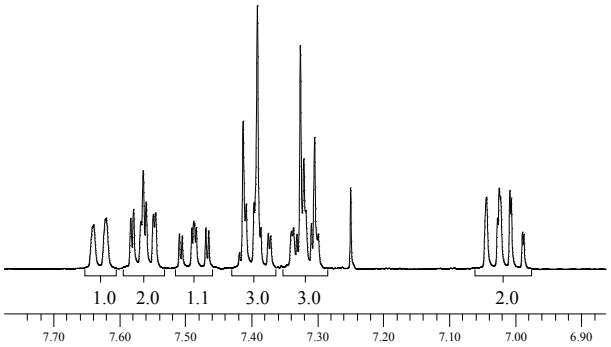


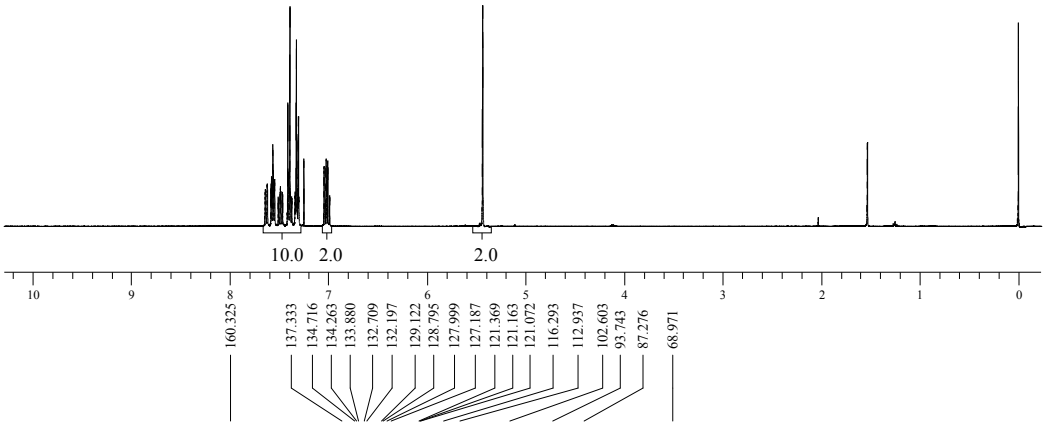


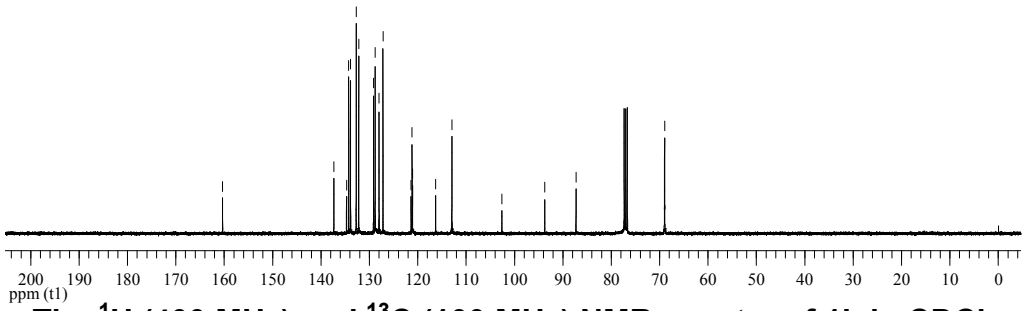
The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 1g in $CDCl_3$



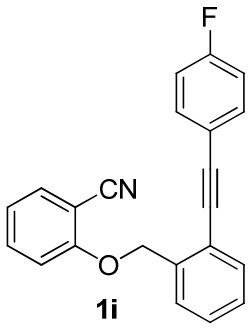
1h



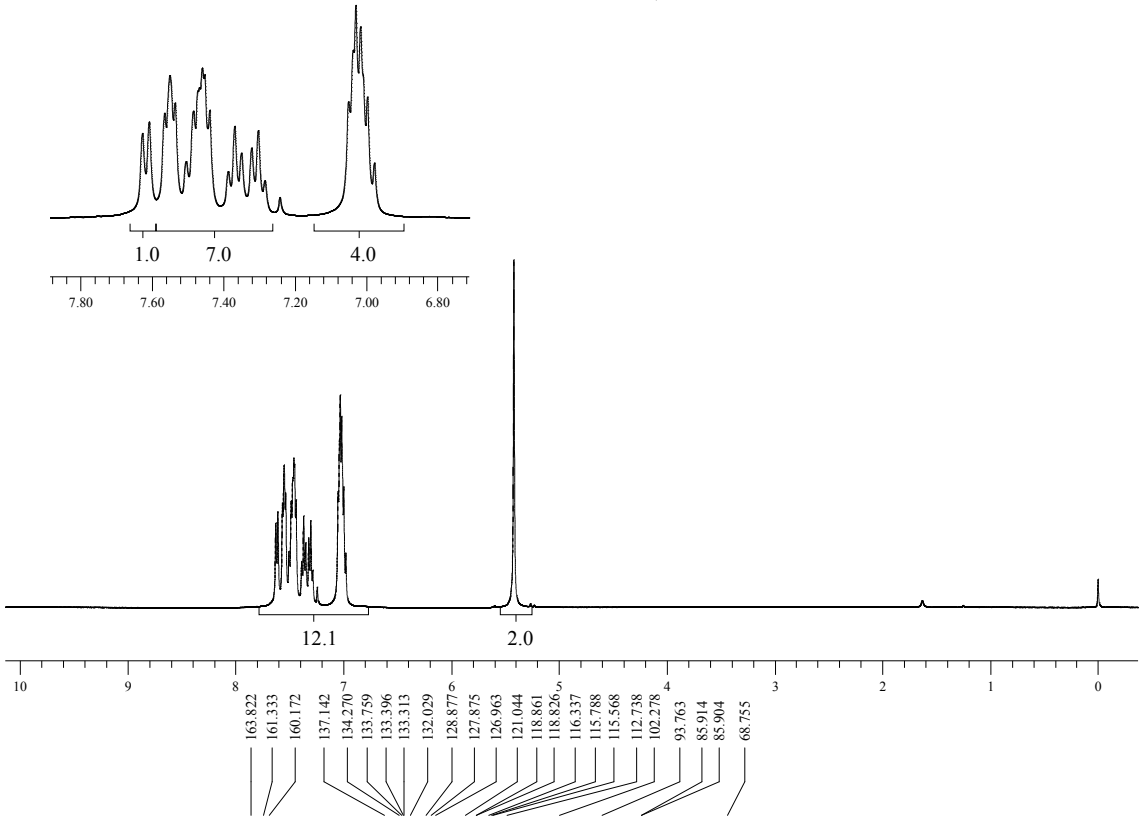


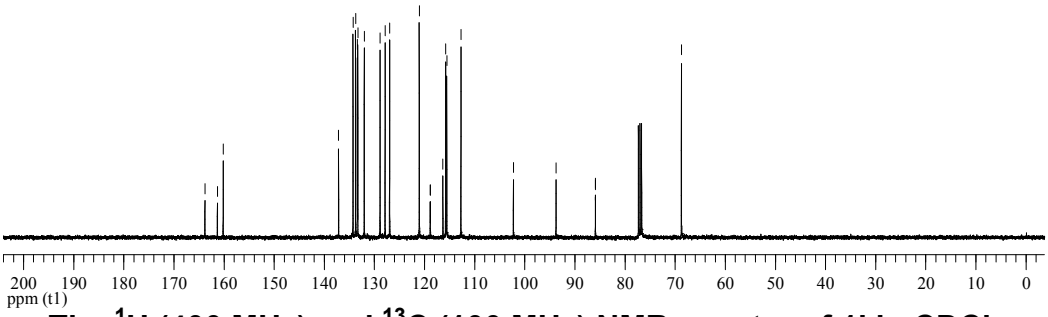


The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 1h in $CDCl_3$

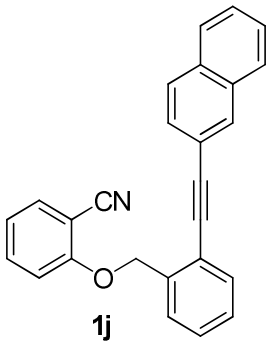


1i

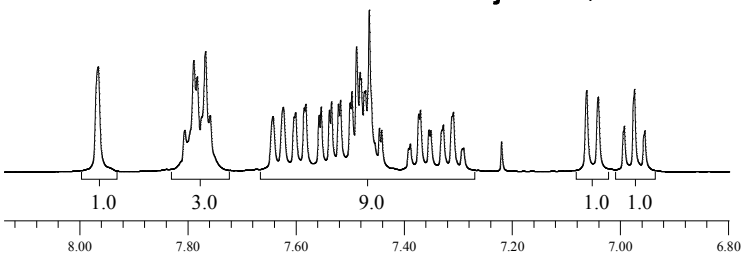


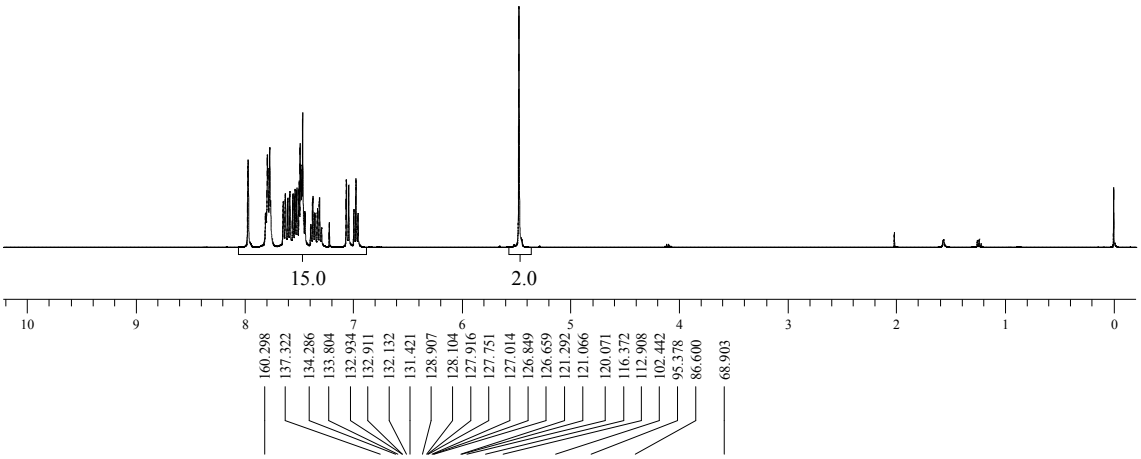


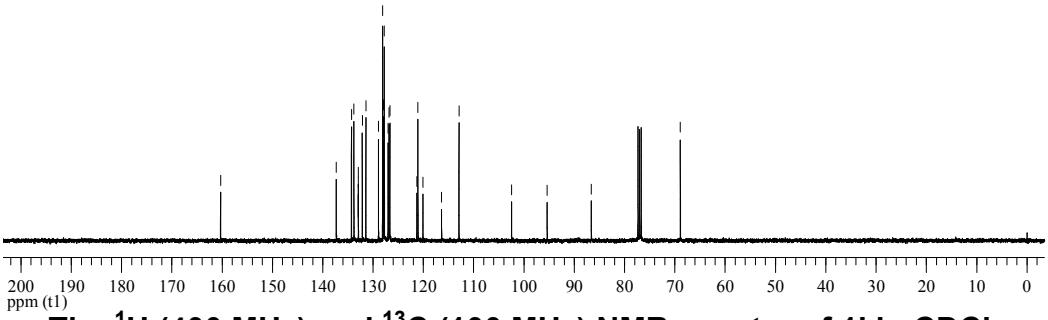
The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 1i in $CDCl_3$



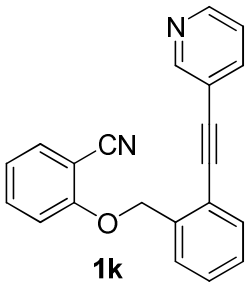
1j



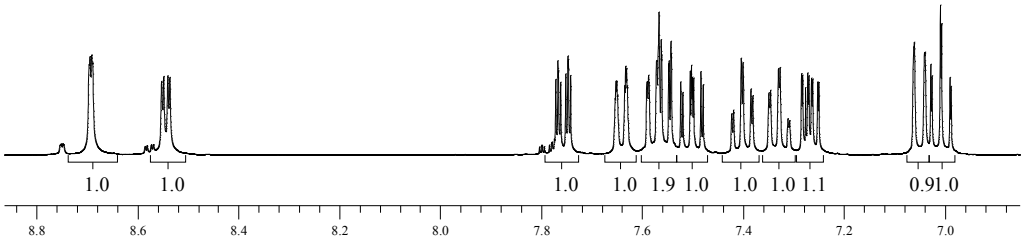


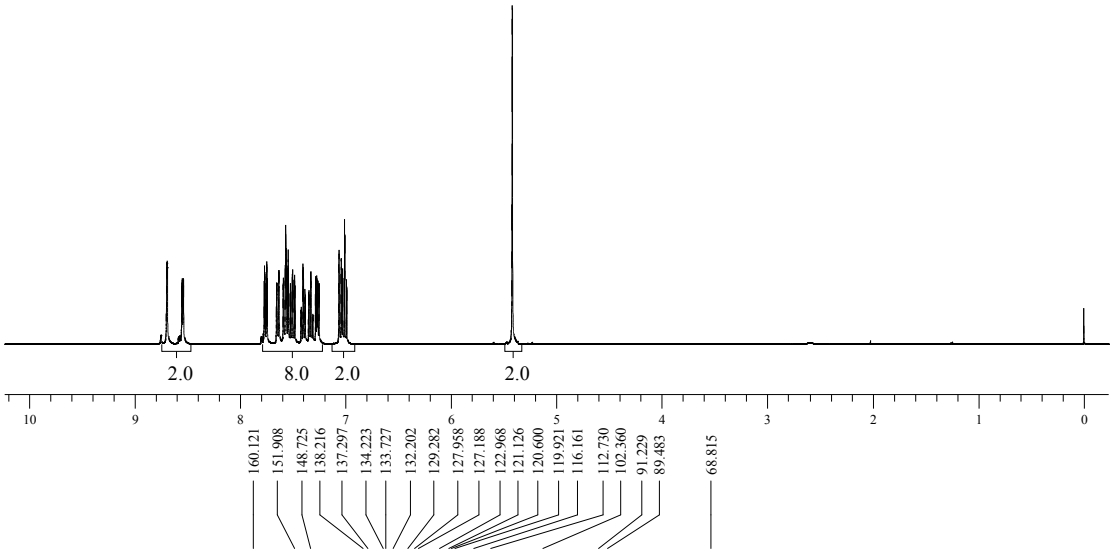


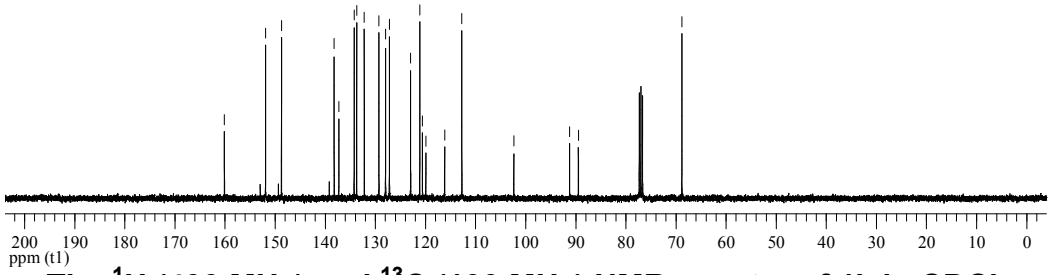
The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 1j in $CDCl_3$



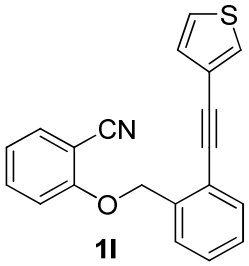
1k



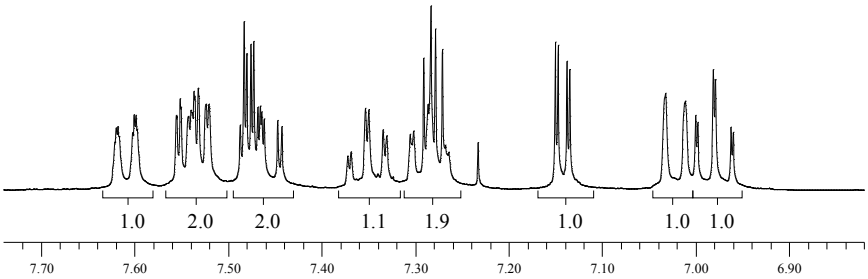


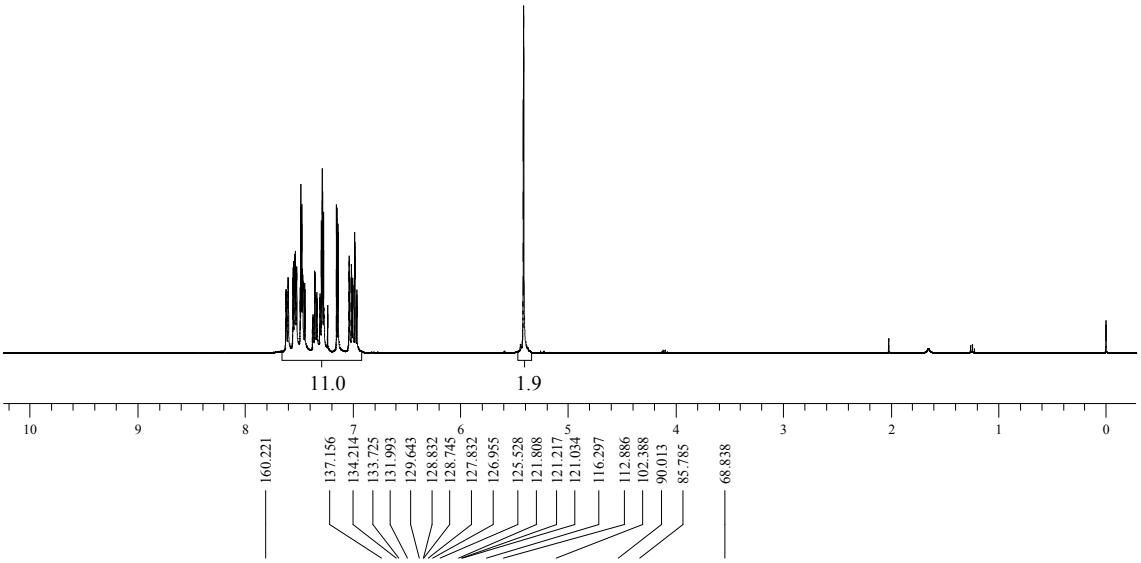


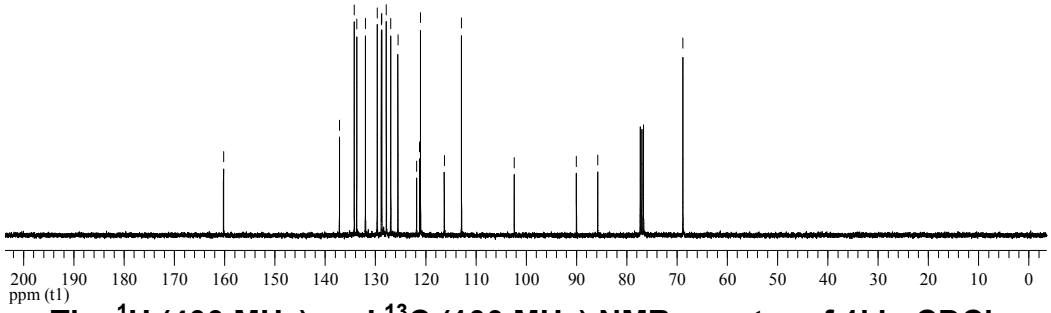
The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 1k in $CDCl_3$



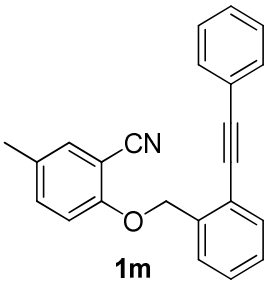
1l



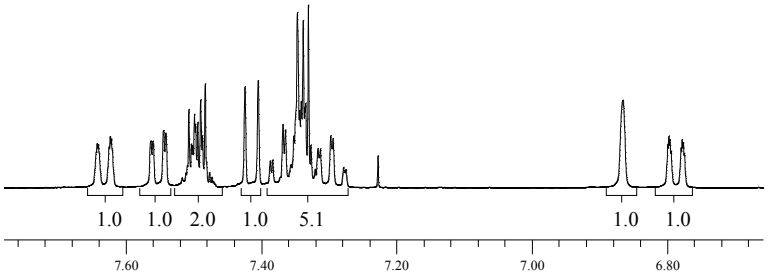


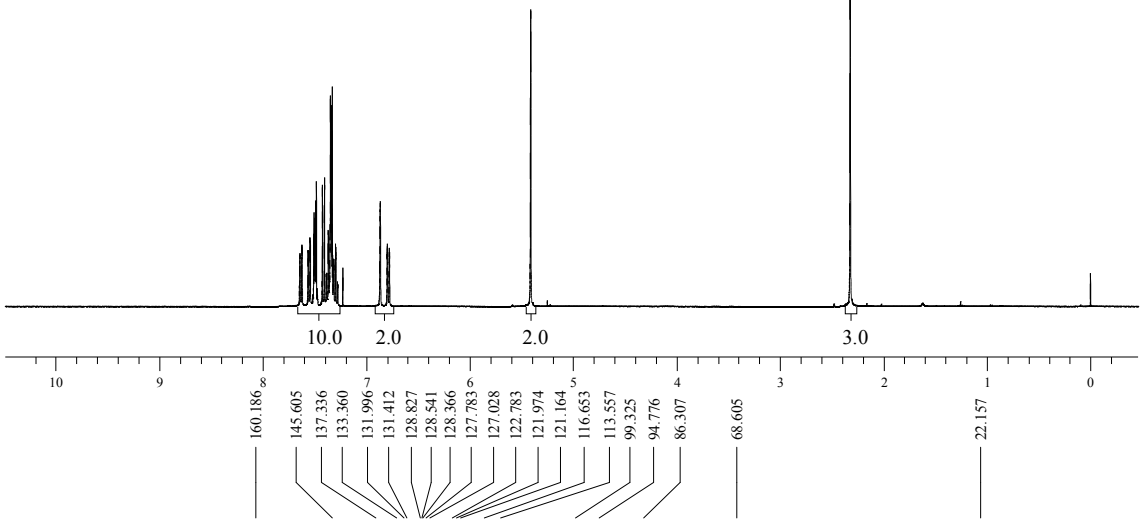


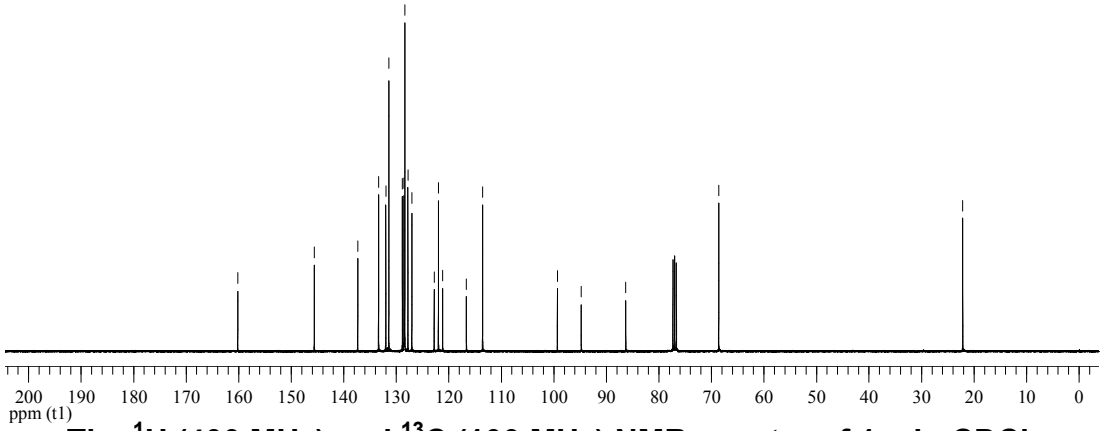
The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 1l in $CDCl_3$



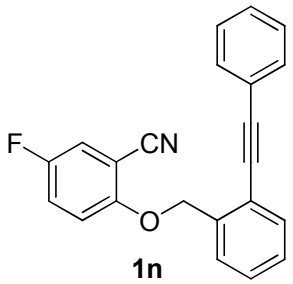
1m



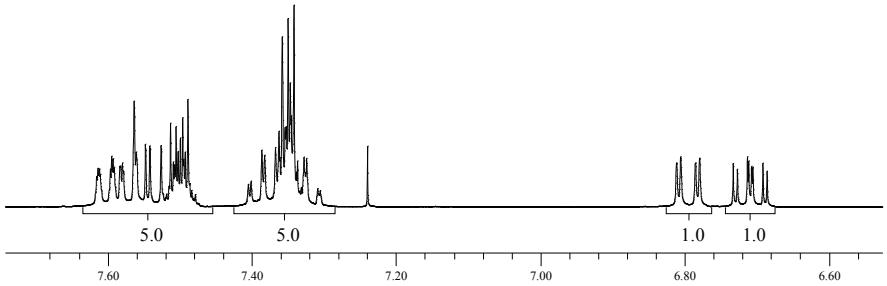


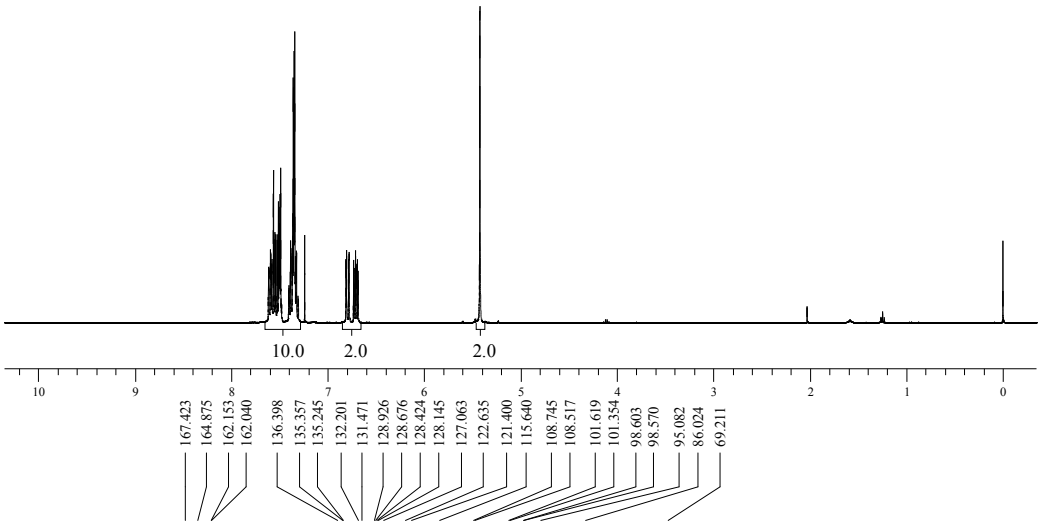


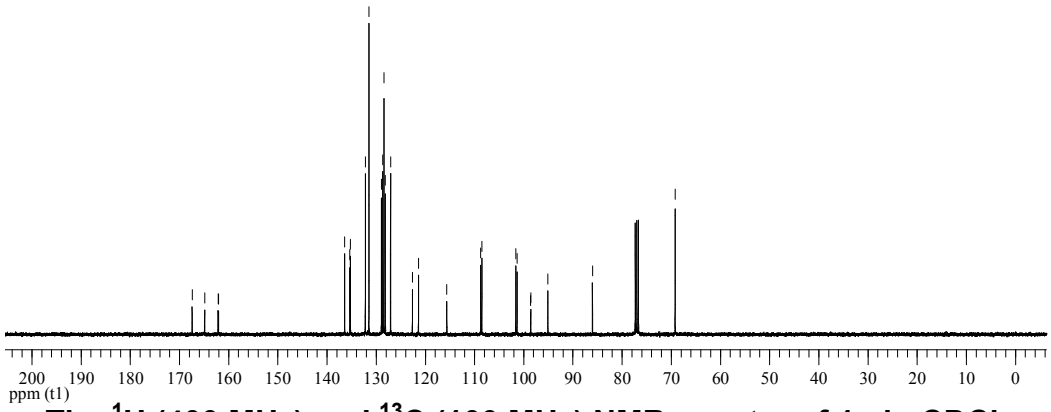
The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 1m in $CDCl_3$



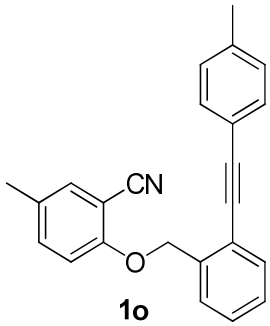
1n



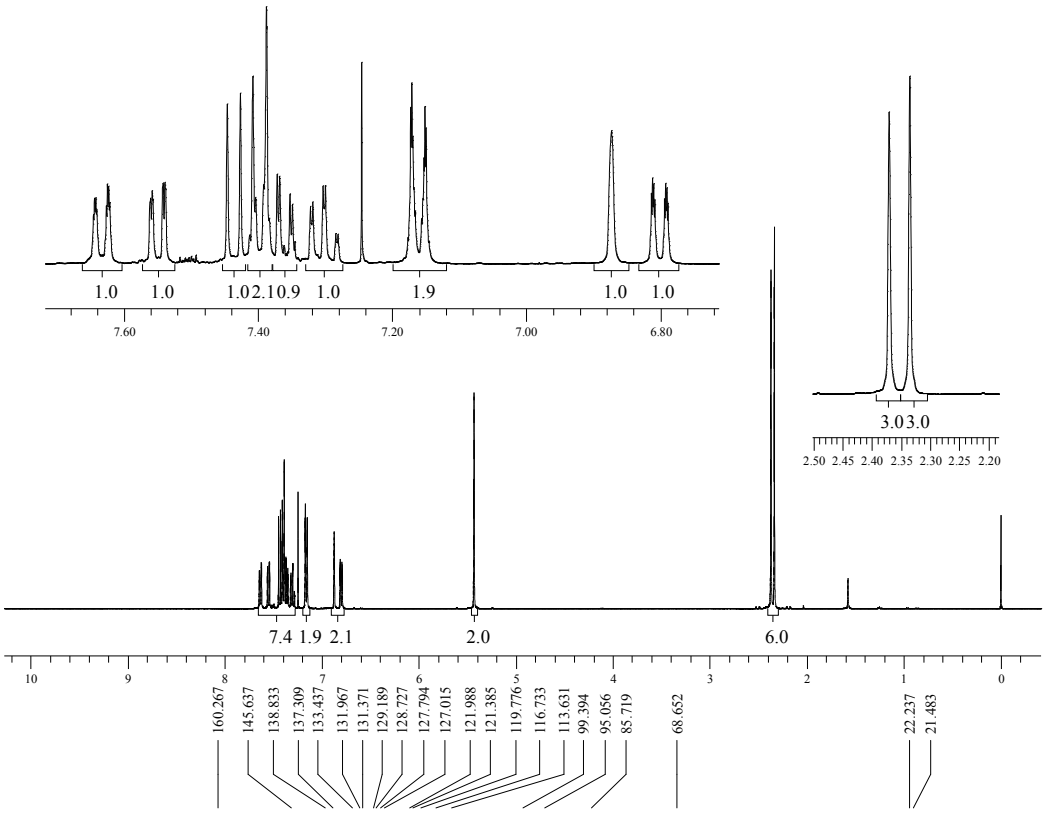


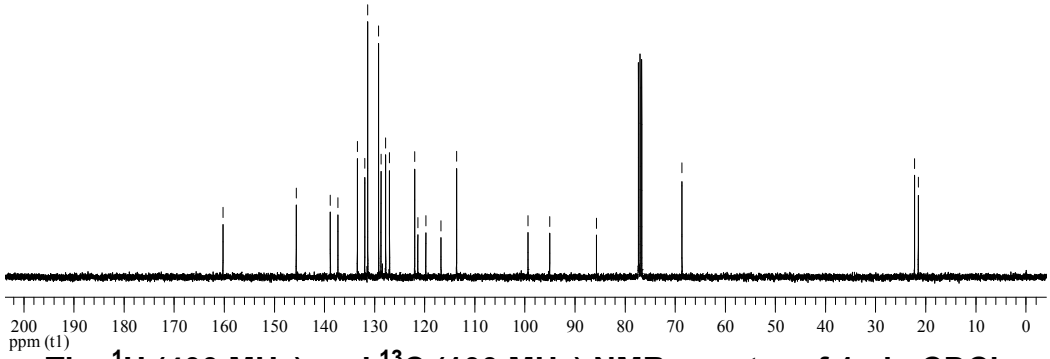


The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 1n in $CDCl_3$

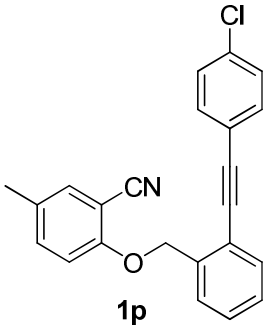


1o

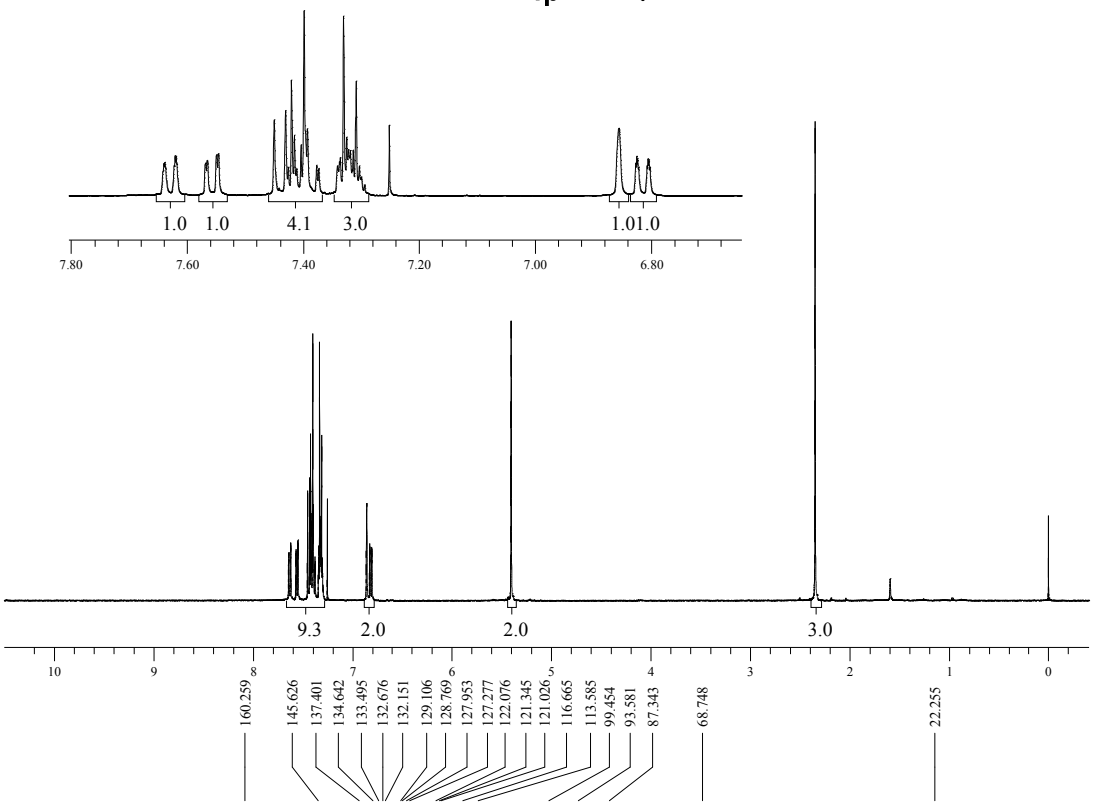


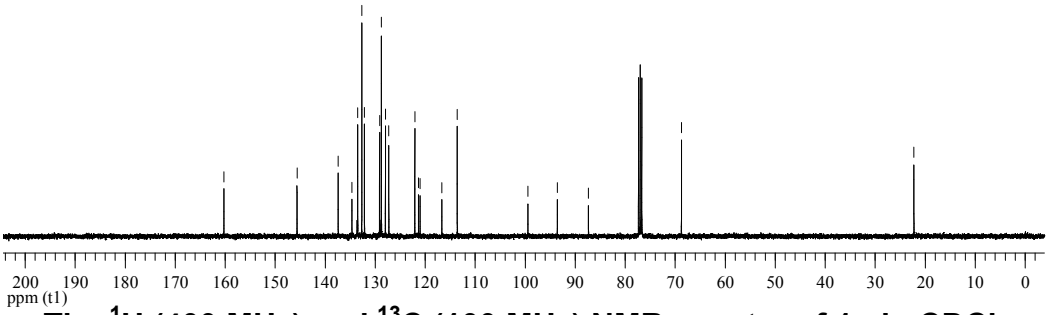


The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 1o in $CDCl_3$

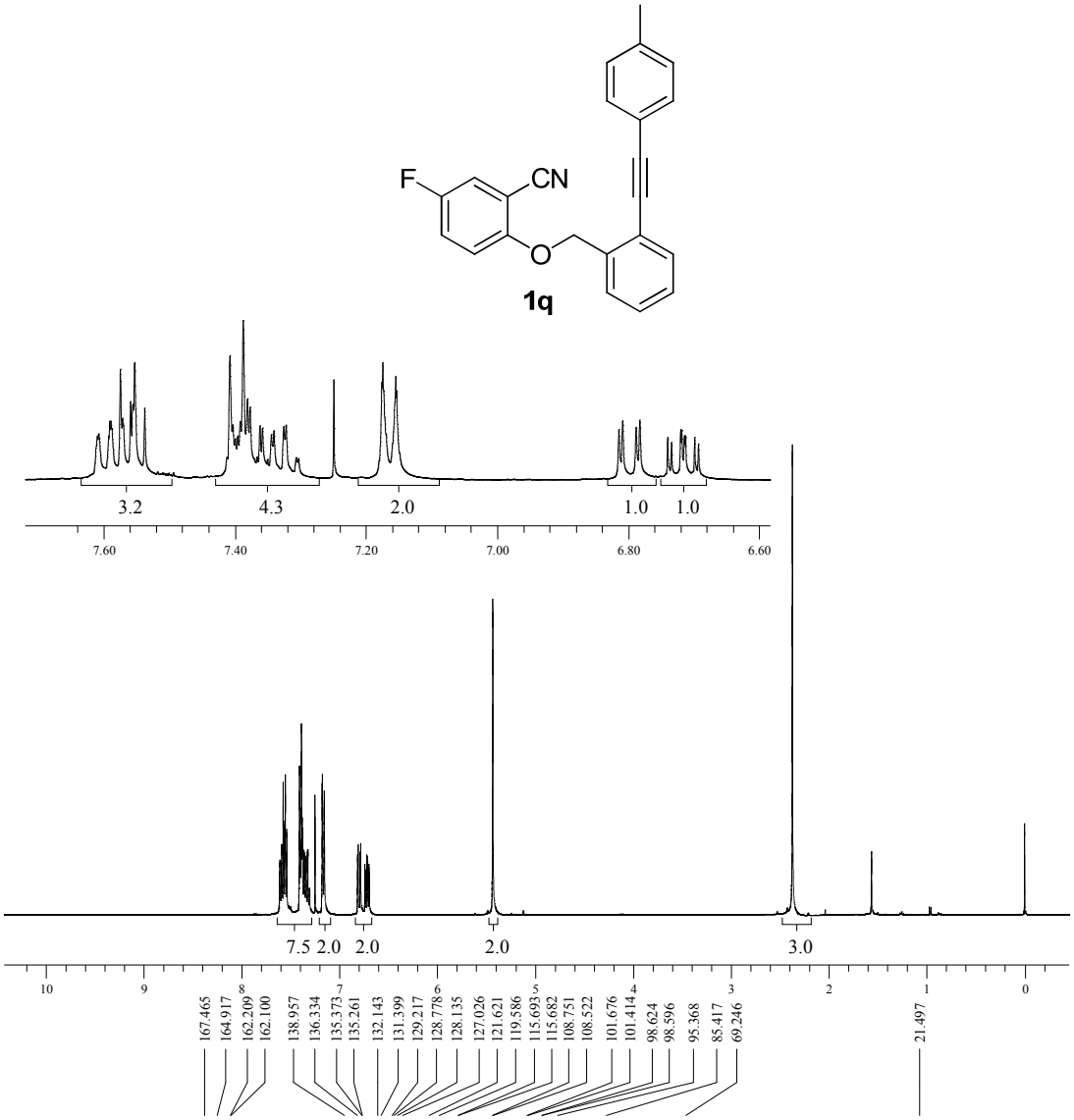


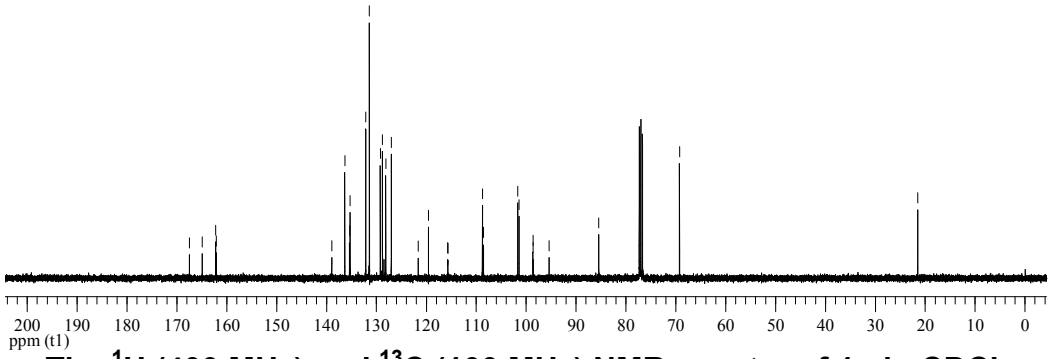
1p





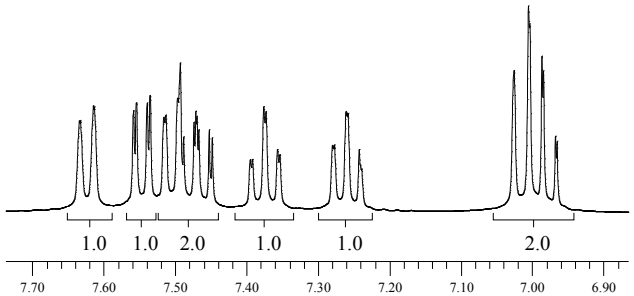
The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 1p in $CDCl_3$

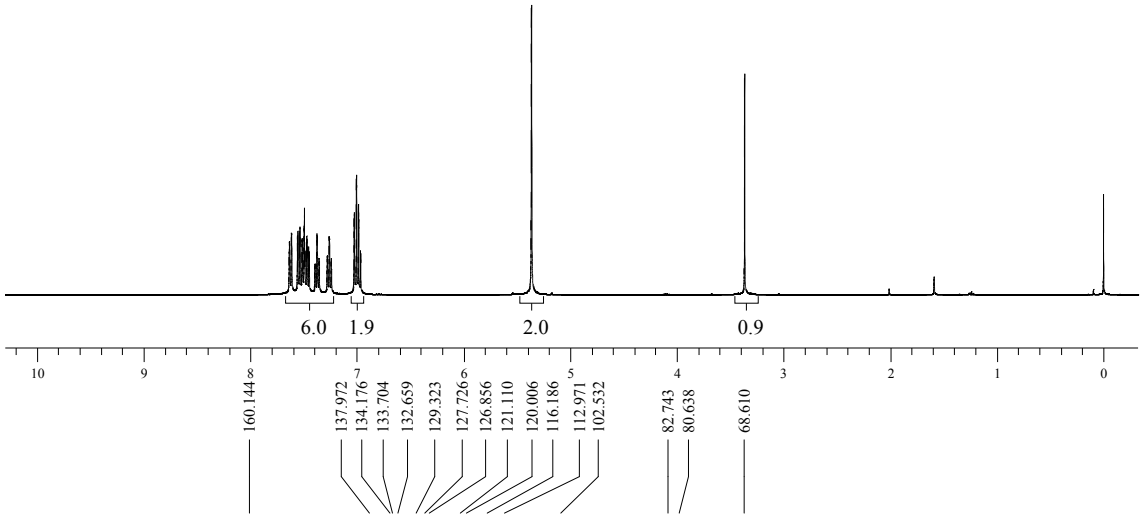


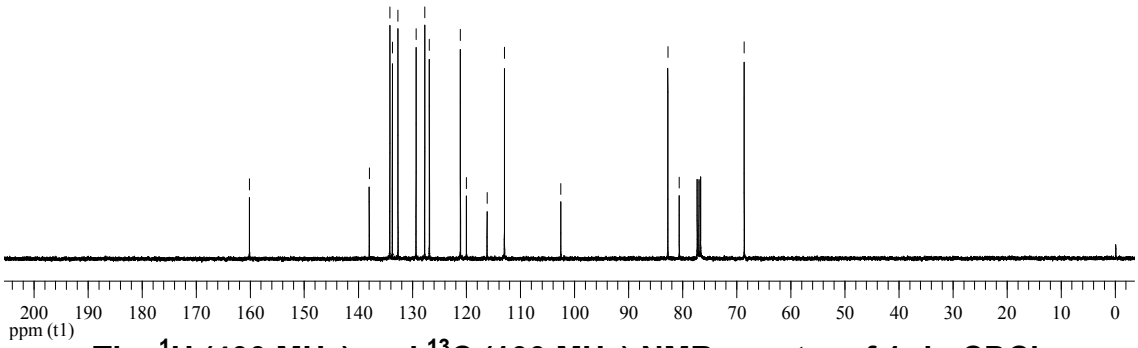


The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 1q in $CDCl_3$

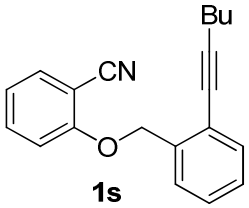
1r



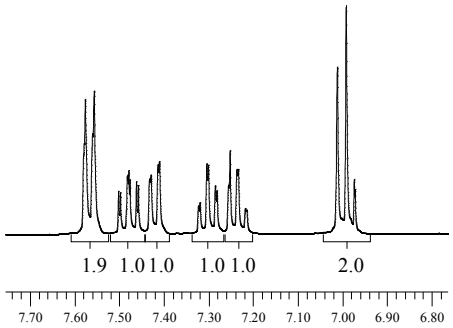


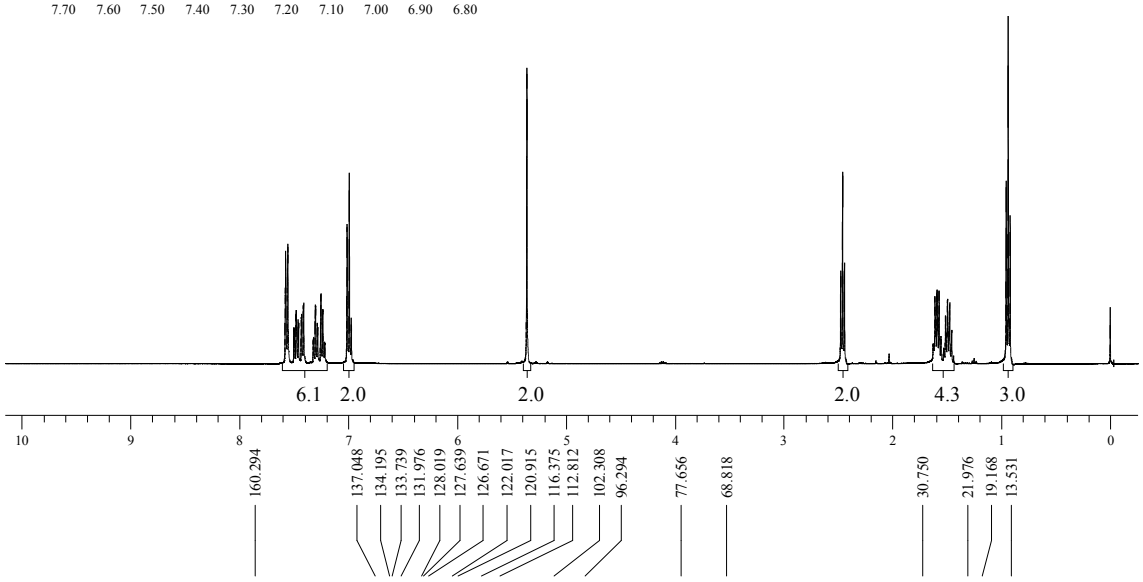


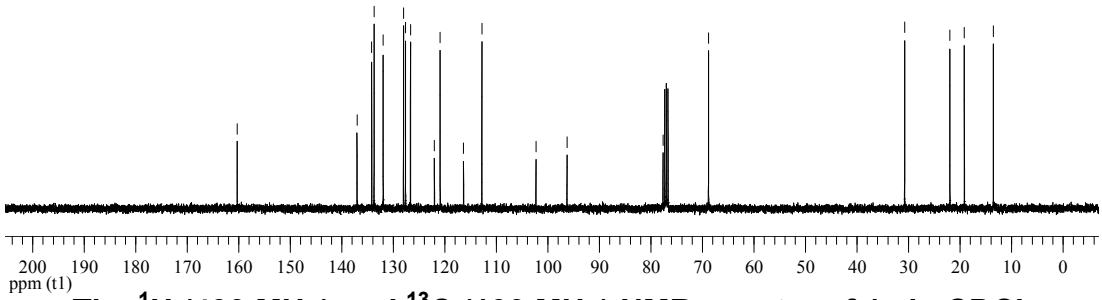
The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 1r in $CDCl_3$



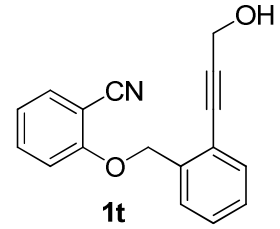
1s



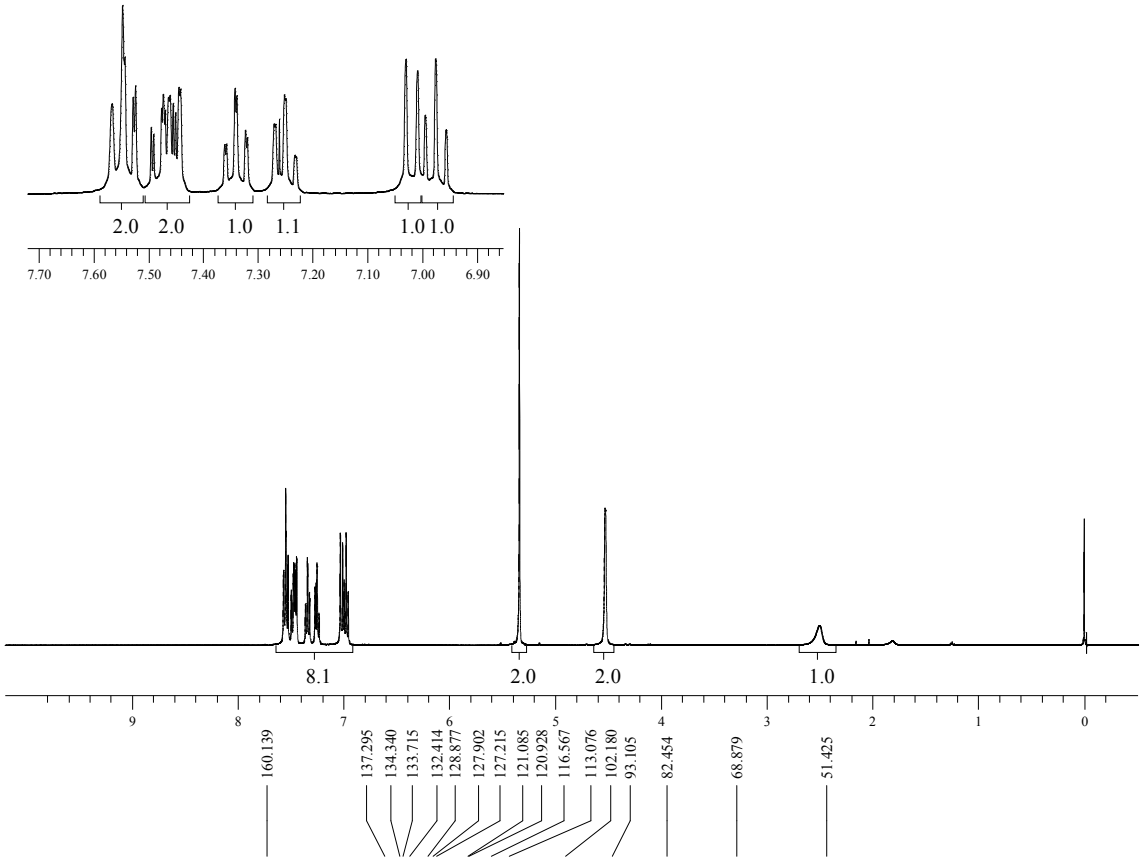


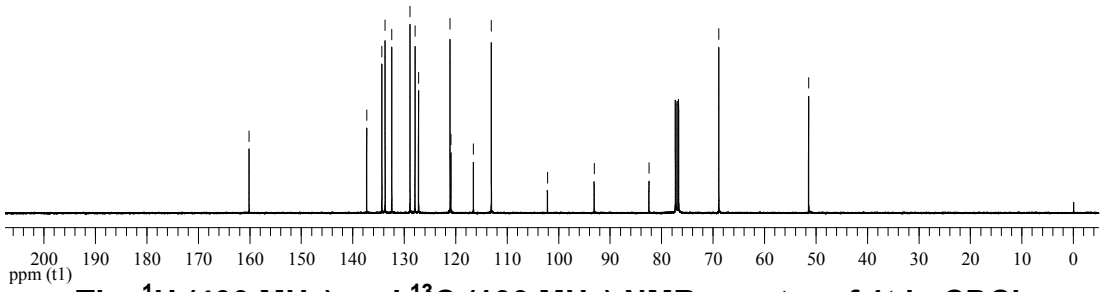


The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 1s in $CDCl_3$

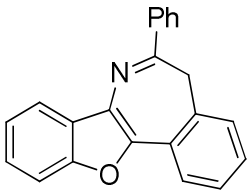


1t

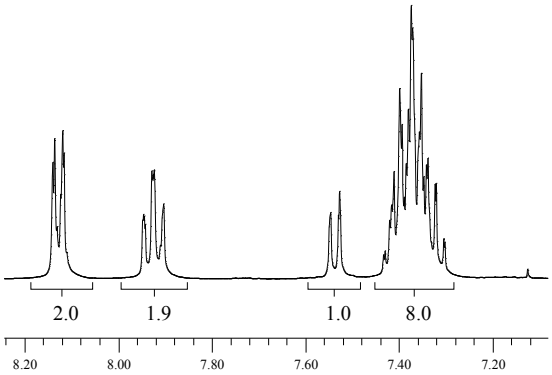


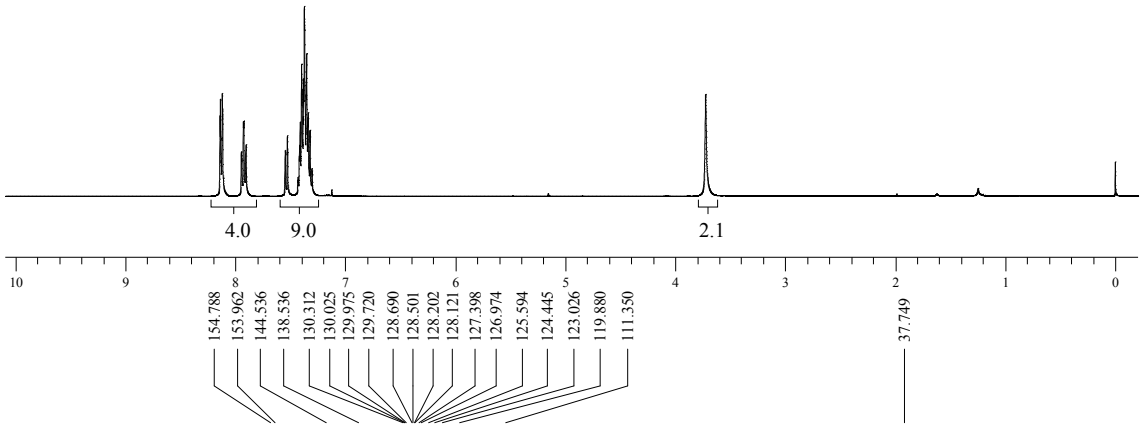


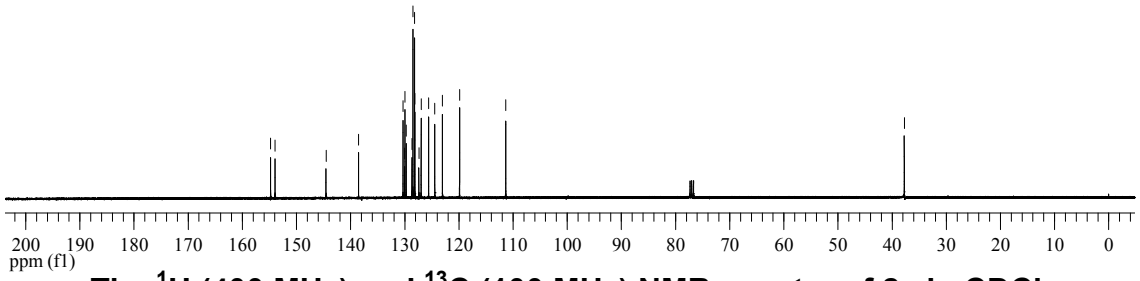
The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 1t in $CDCl_3$



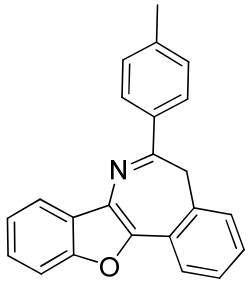
2a



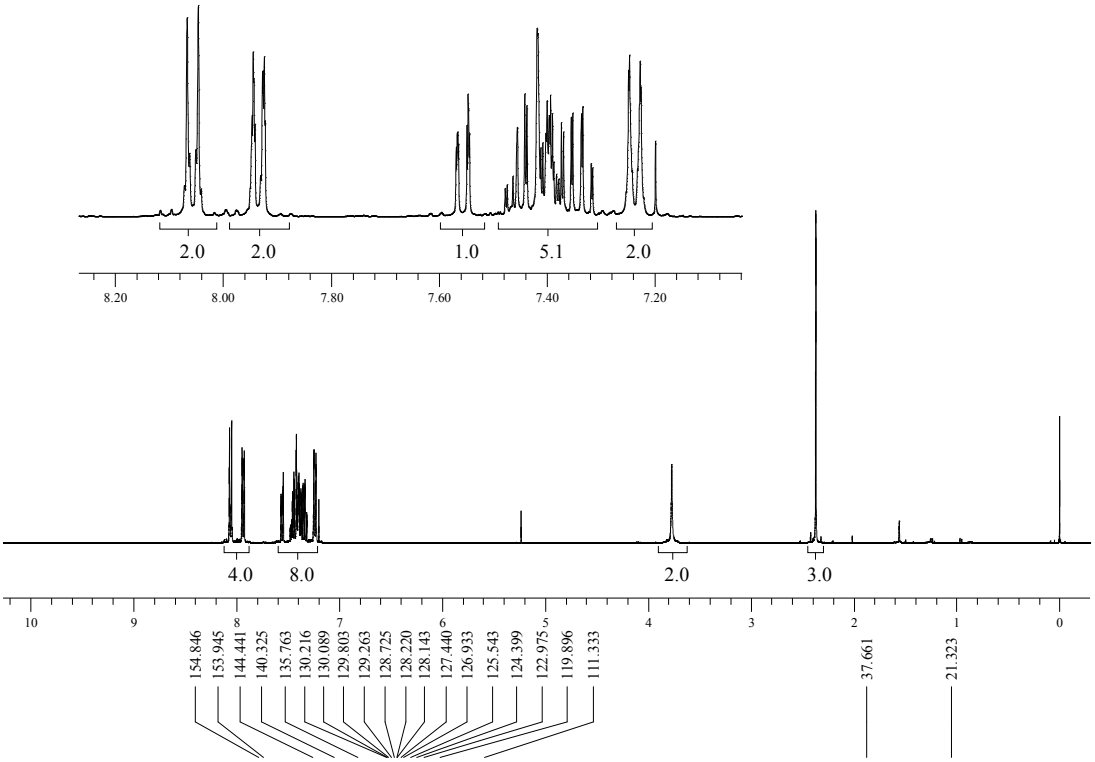


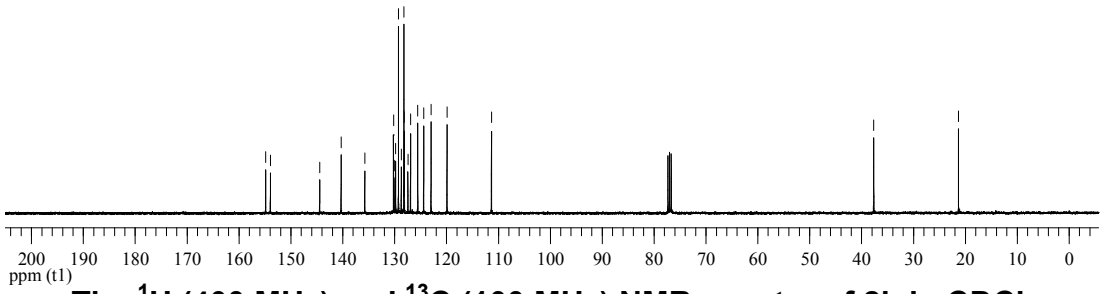


The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 2a in $CDCl_3$

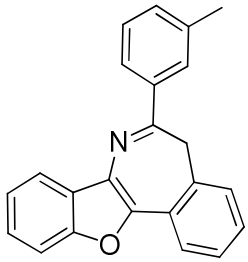


2b

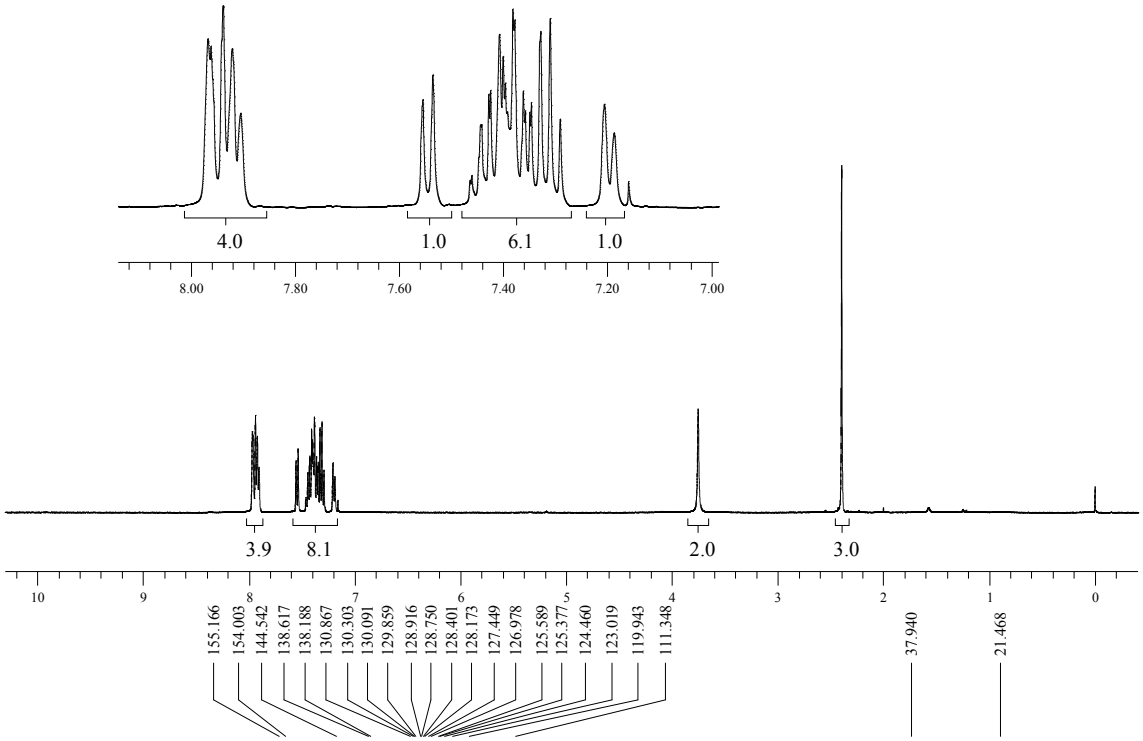


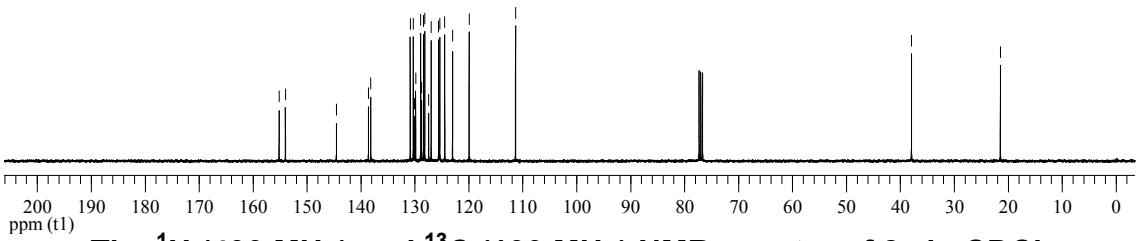


The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 2b in $CDCl_3$

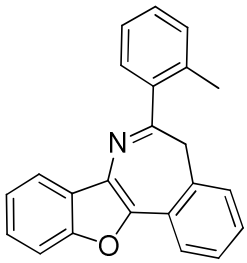


2c

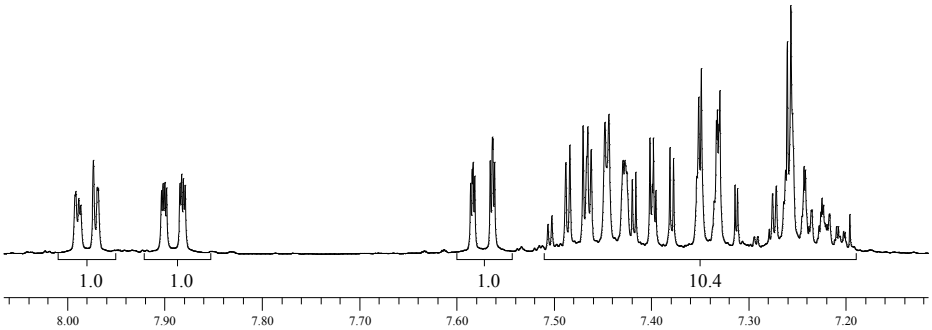


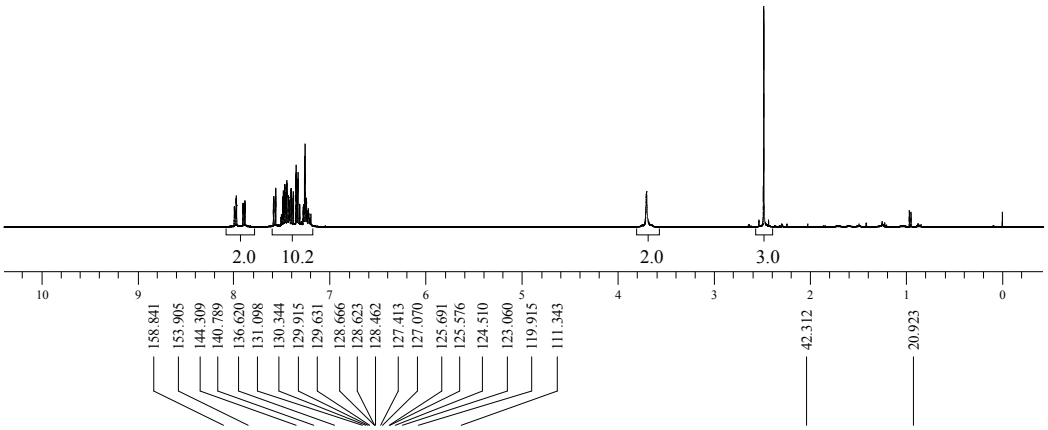


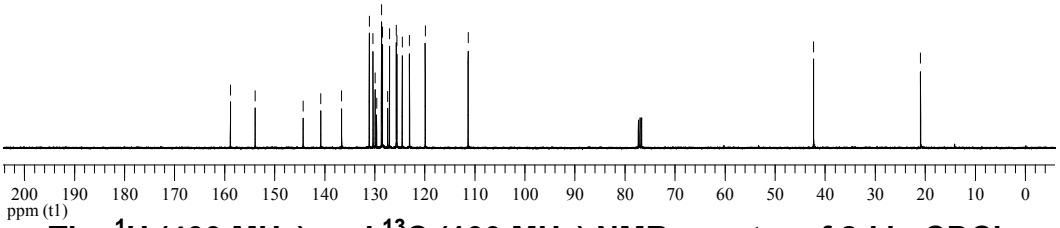
The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 2c in $CDCl_3$



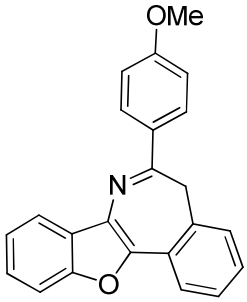
2d



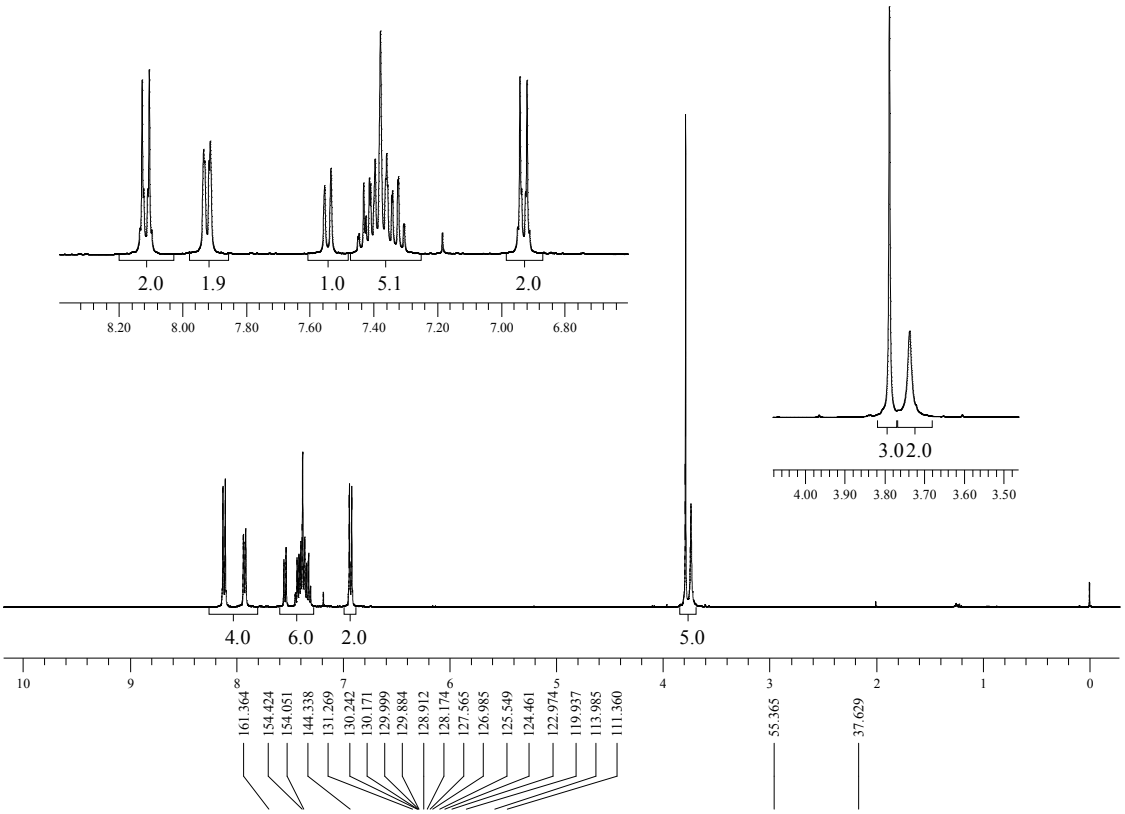


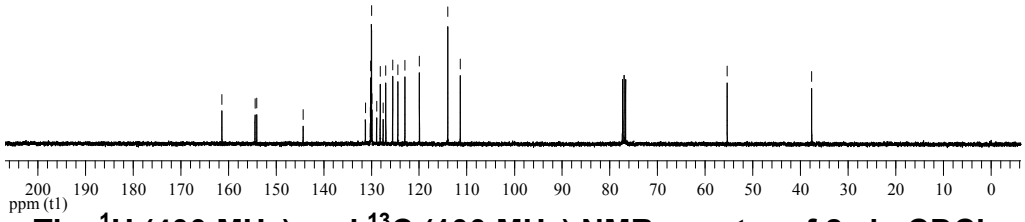


The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 2d in $CDCl_3$

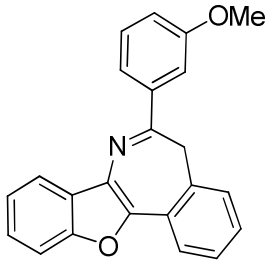


2e

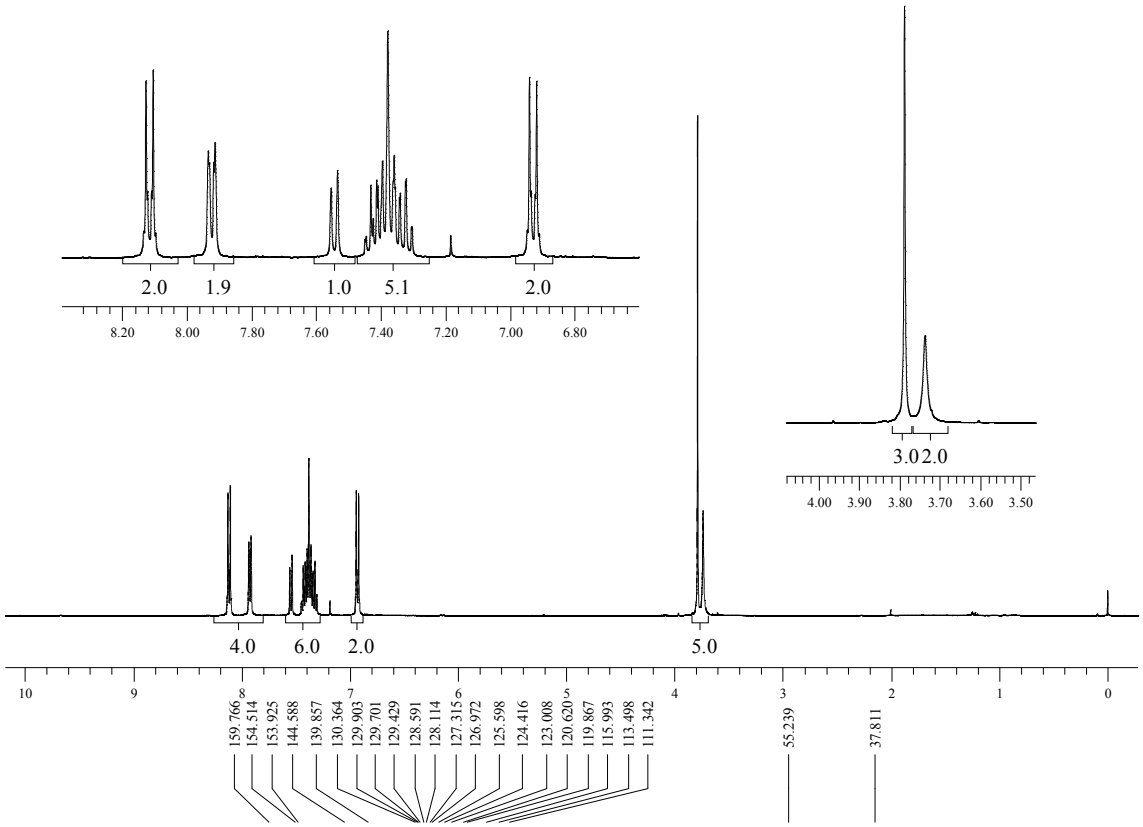


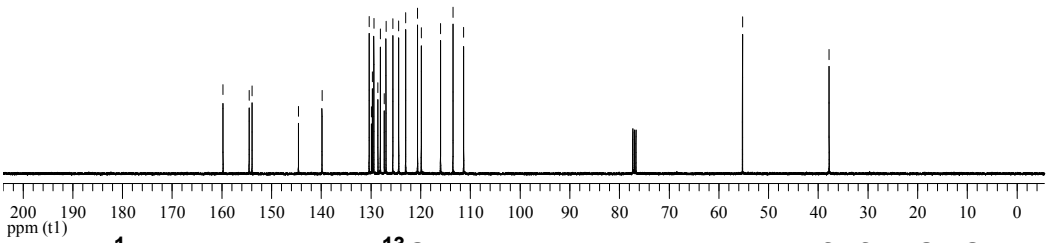


The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 2e in $CDCl_3$

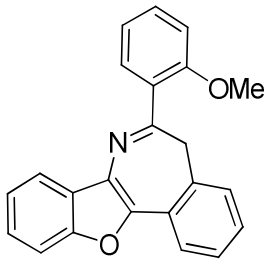


2f

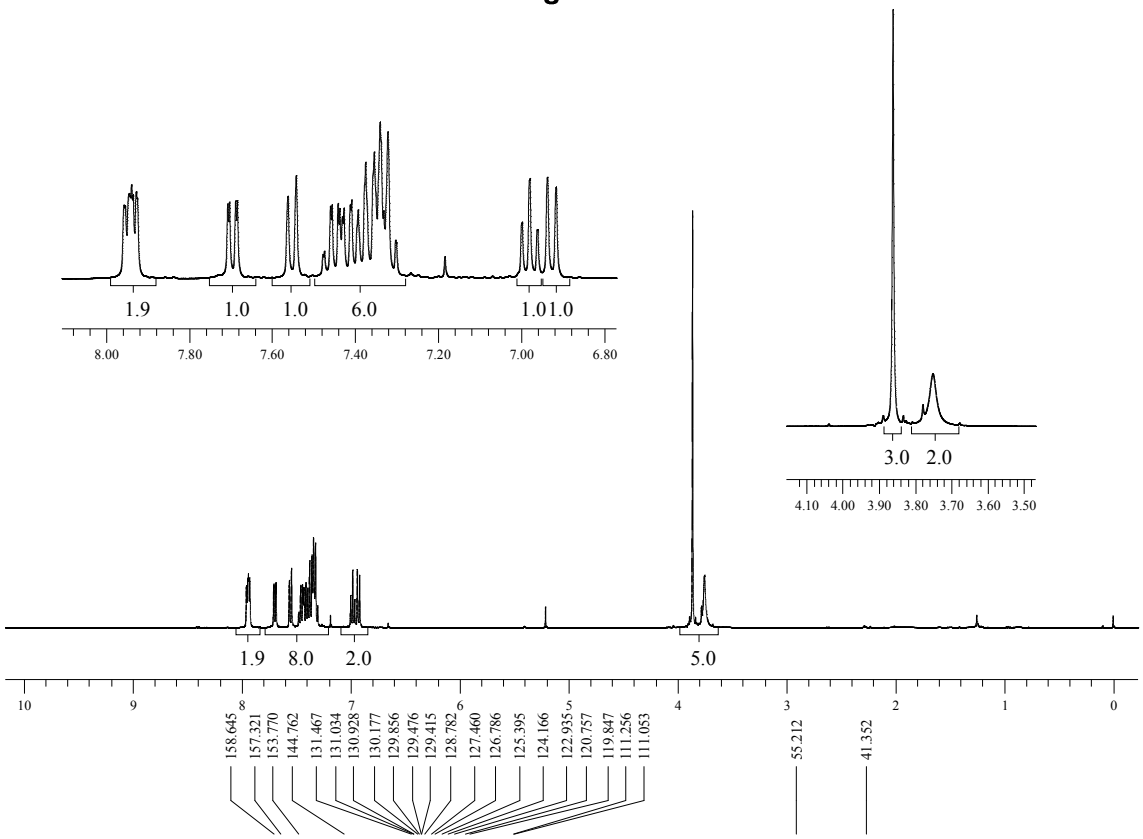


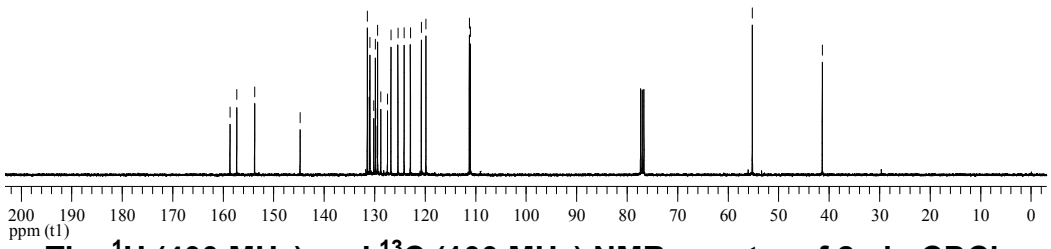


The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 2f in $CDCl_3$

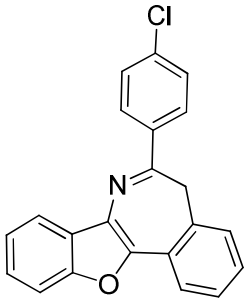


2g

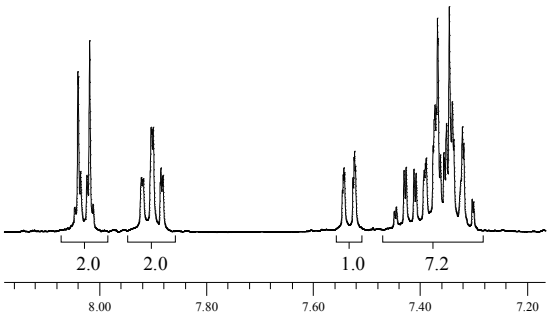


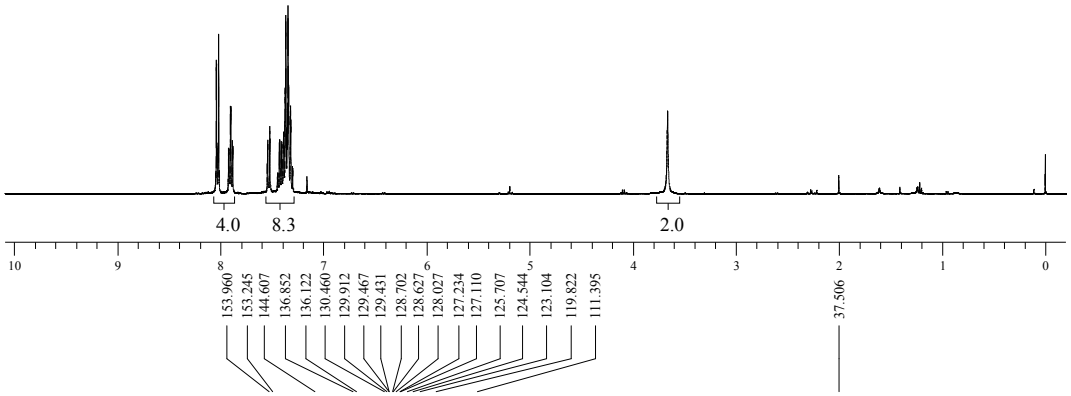


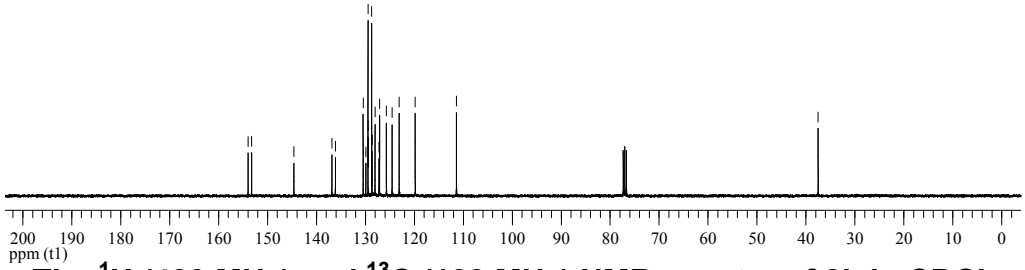
The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 2g in $CDCl_3$



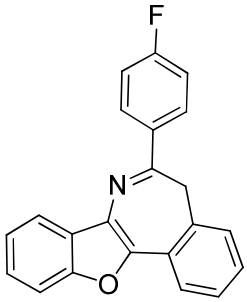
2h



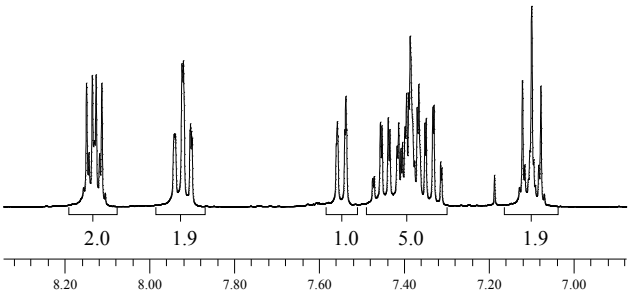


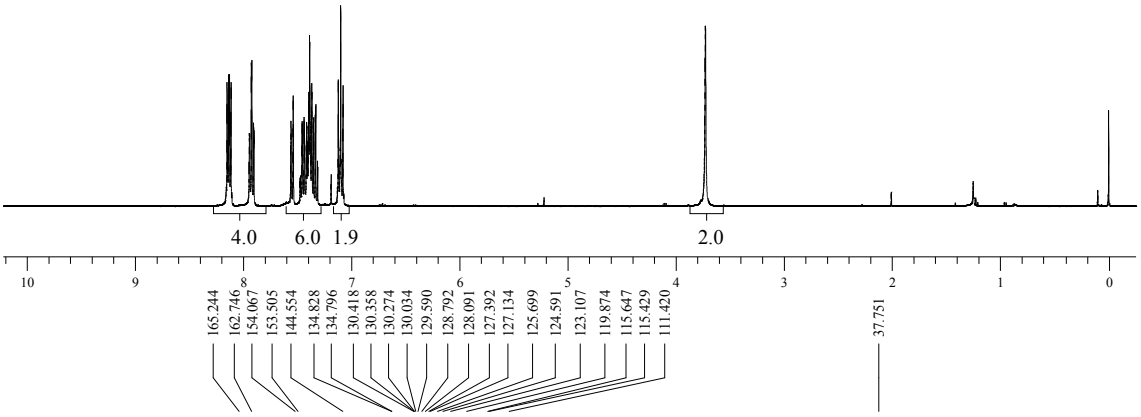


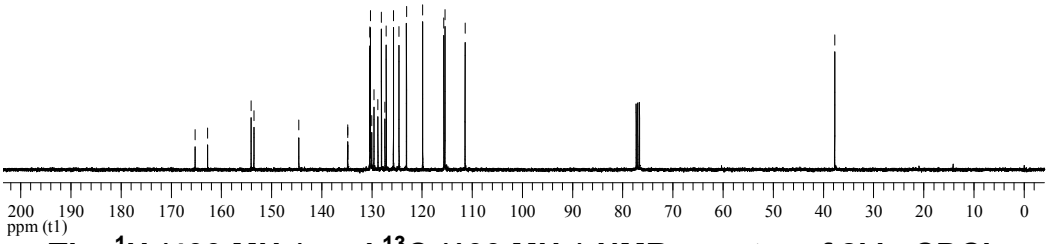
The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 2h in $CDCl_3$



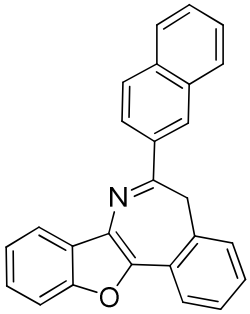
2i



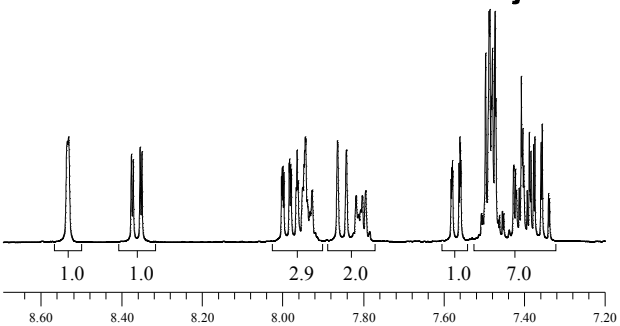


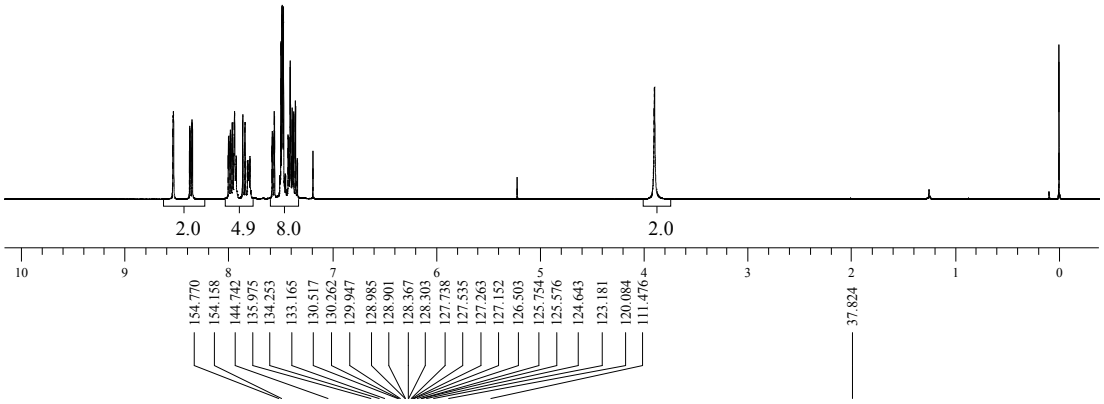


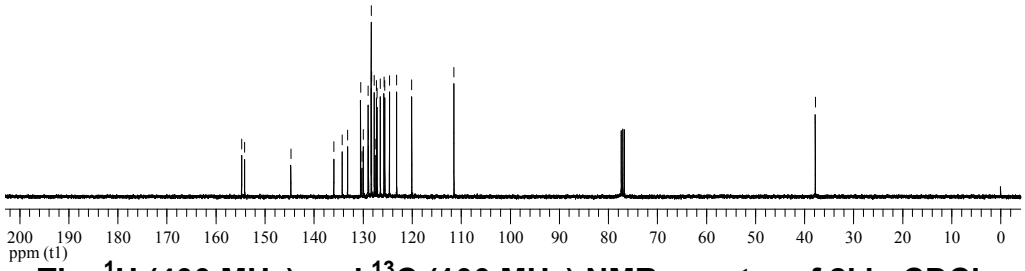
The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 2i in $CDCl_3$



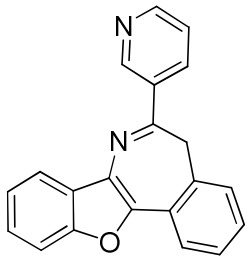
2j



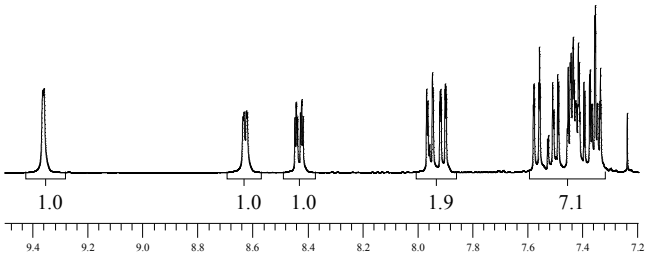


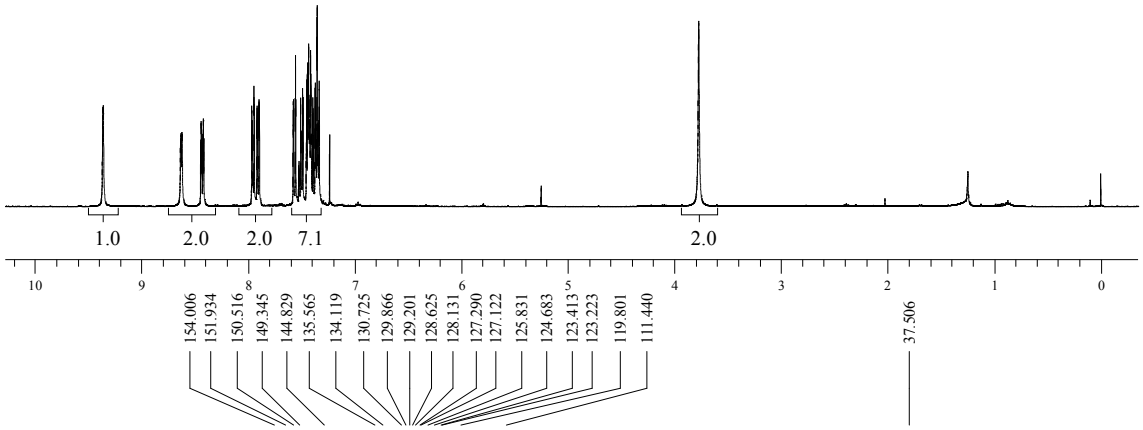


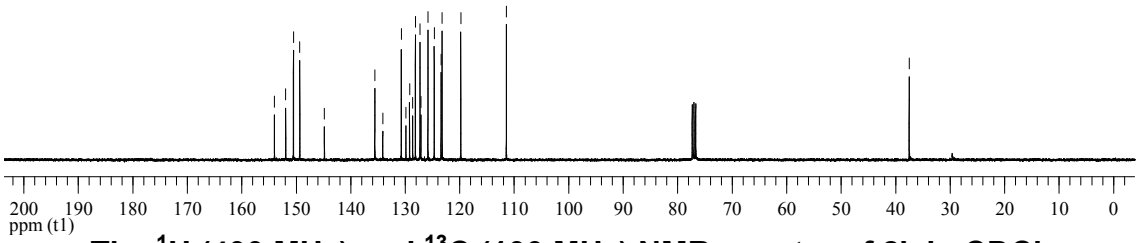
The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 2j in $CDCl_3$



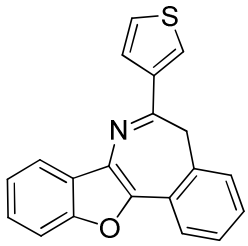
2k



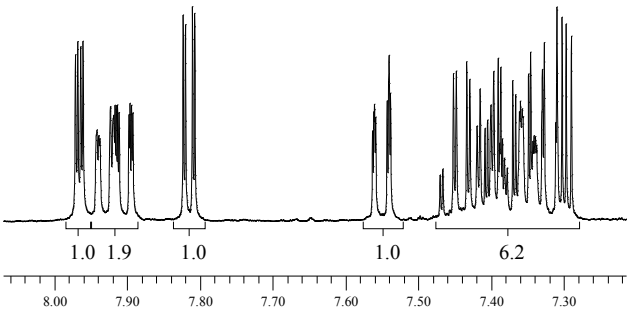


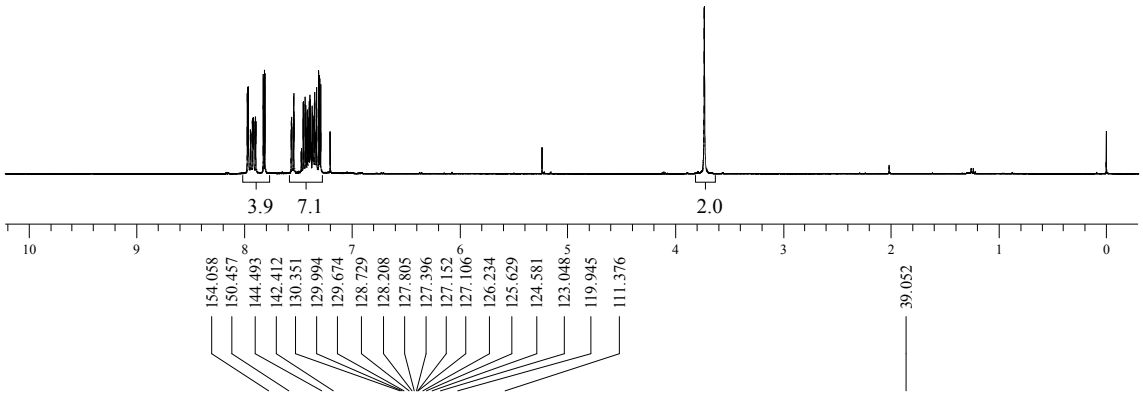


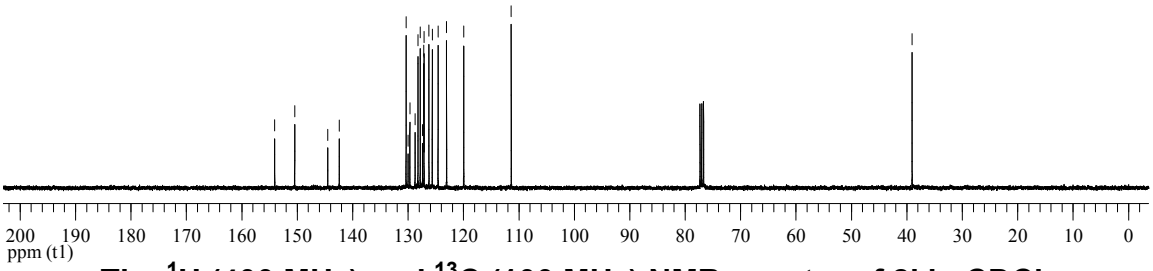
The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 2k in $CDCl_3$



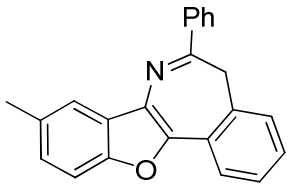
2l



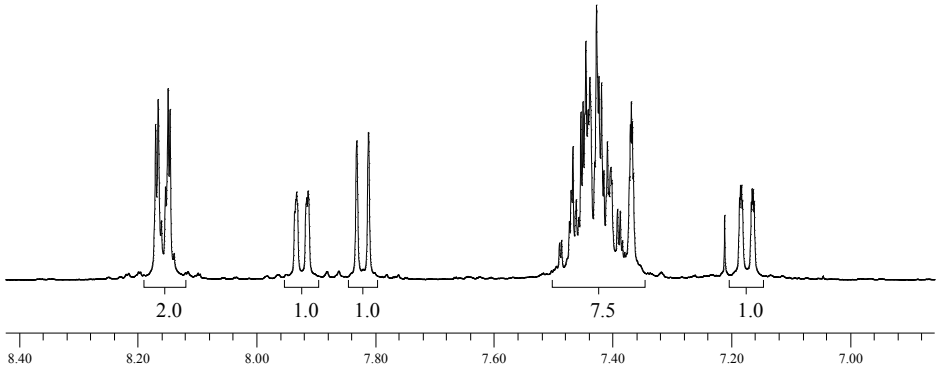


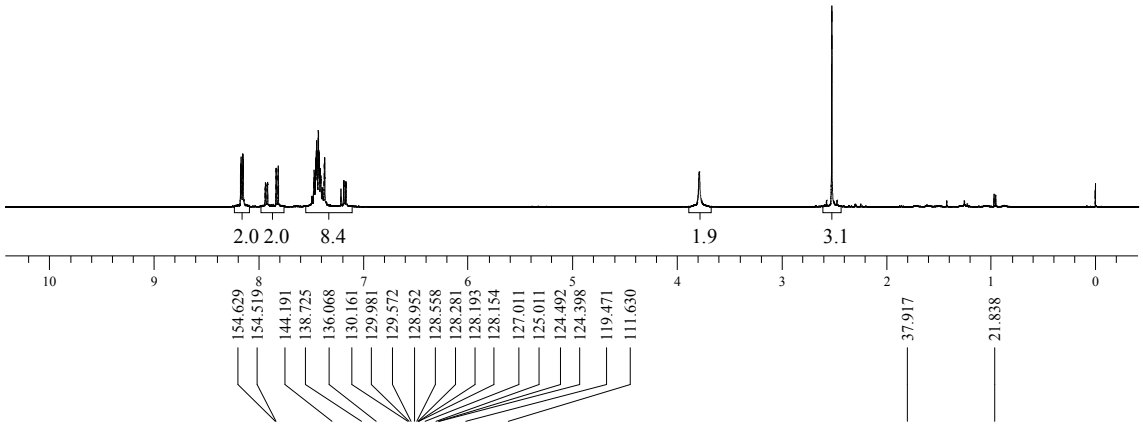


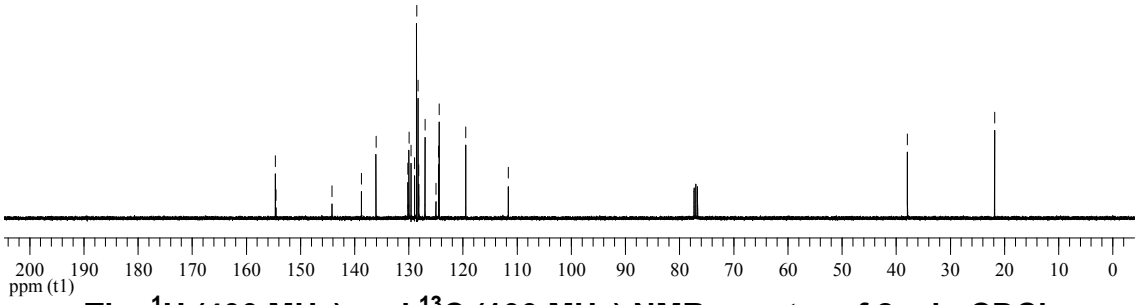
The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 2l in $CDCl_3$



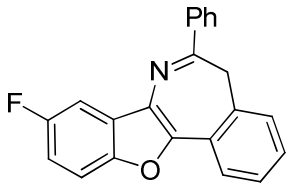
2m



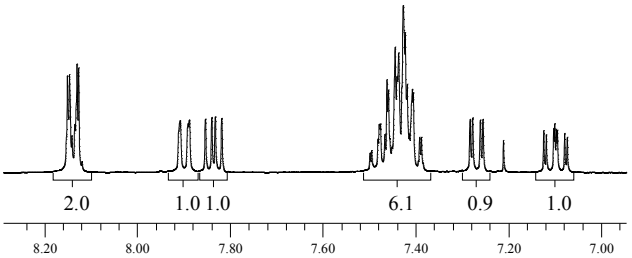


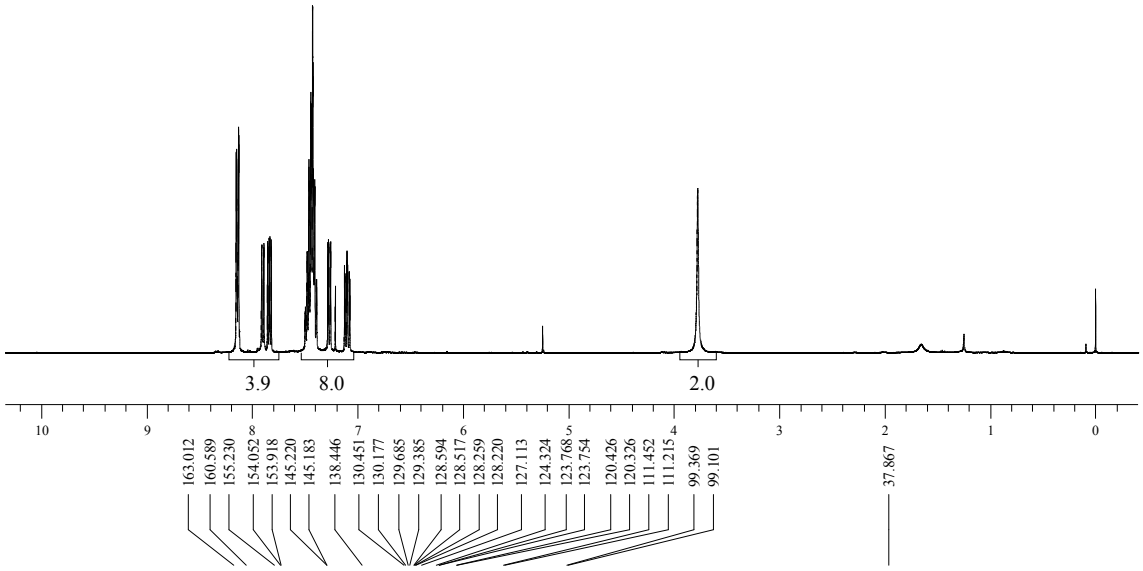


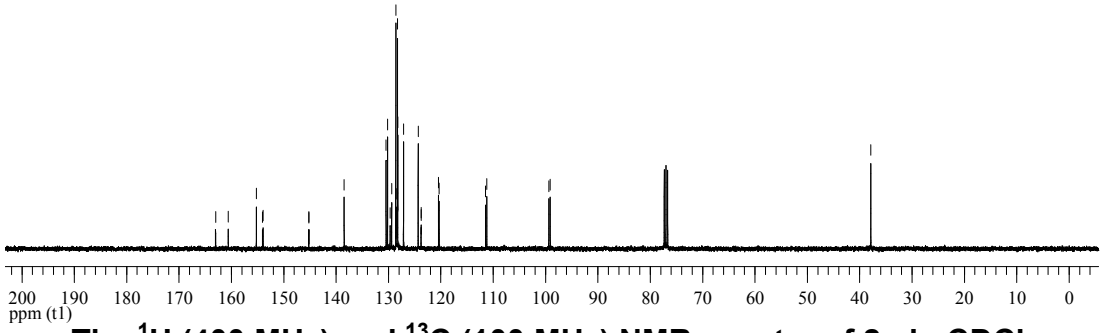
The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 2m in $CDCl_3$



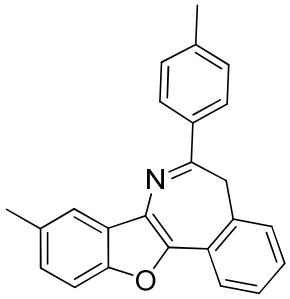
2n



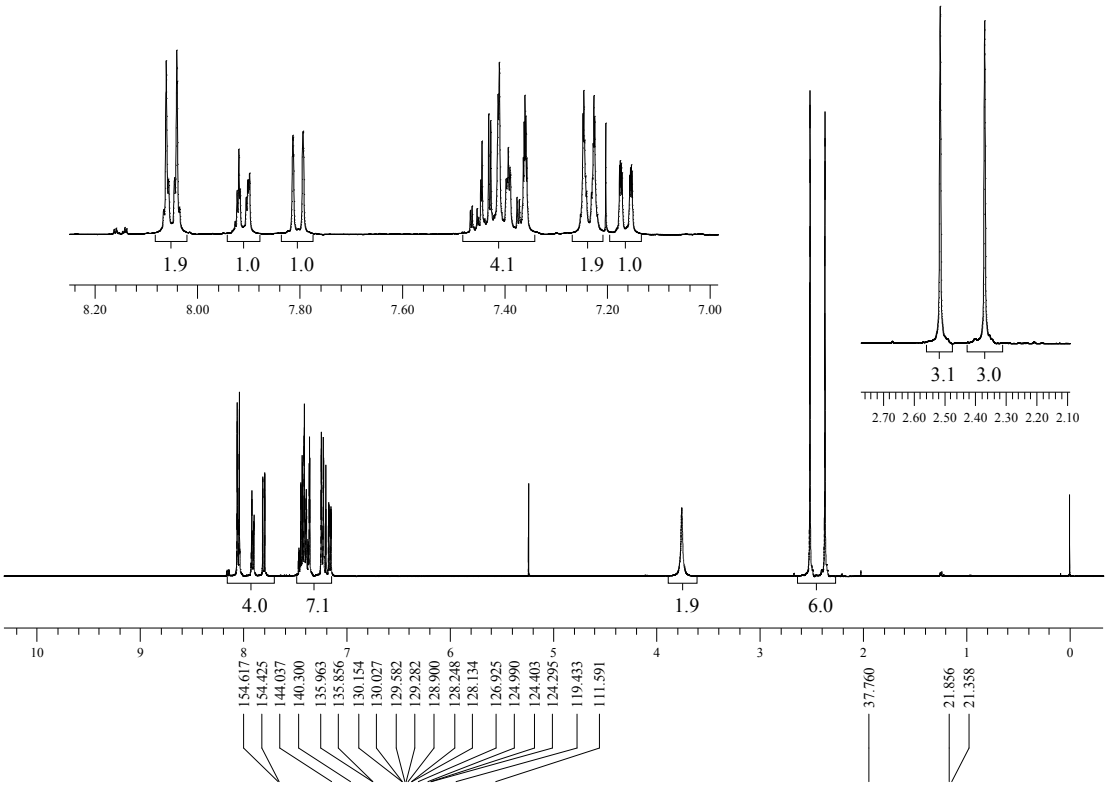


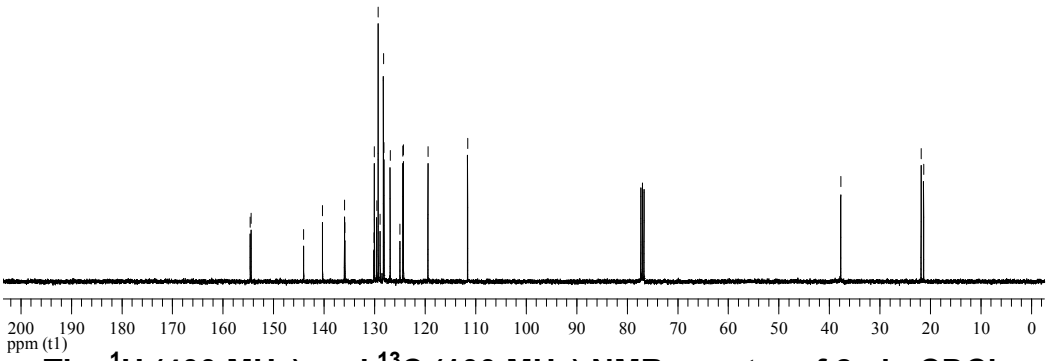


The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 2n in $CDCl_3$

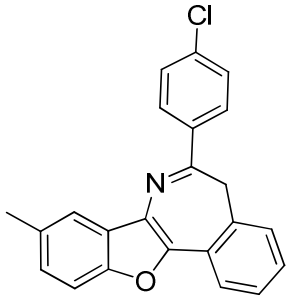


2o

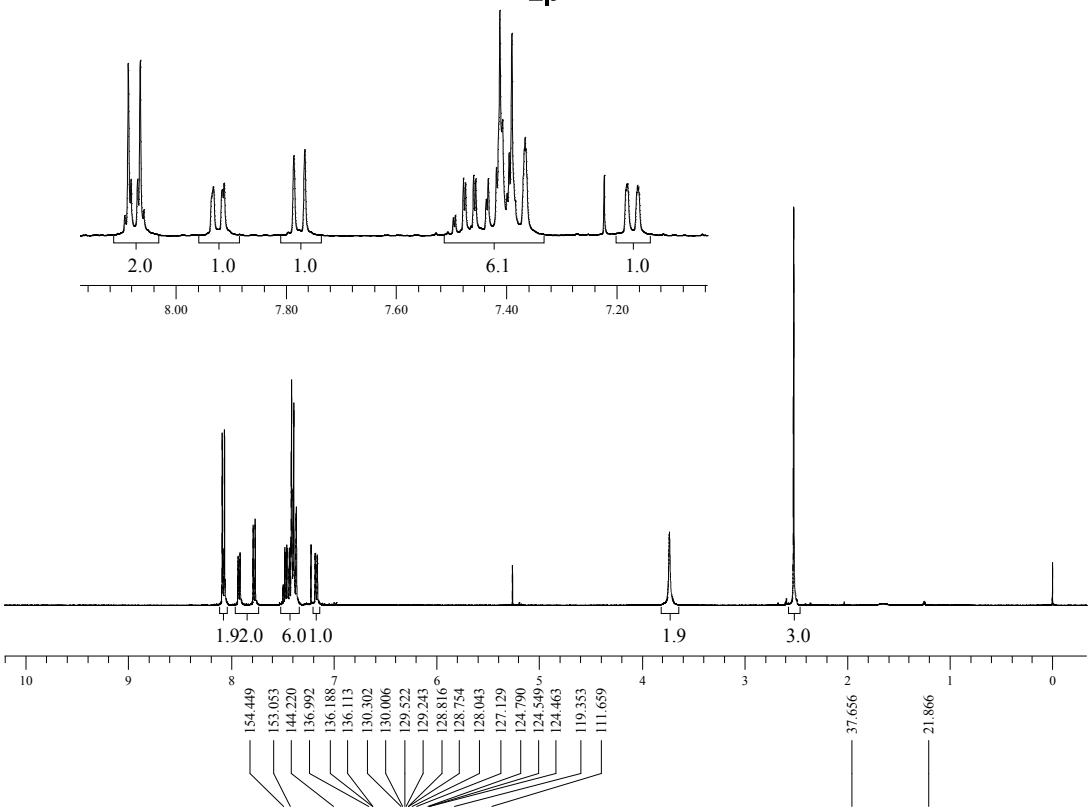


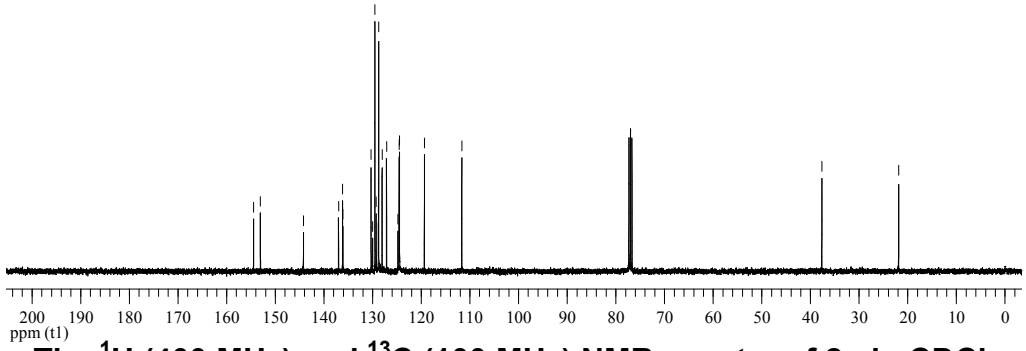


The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 2o in $CDCl_3$

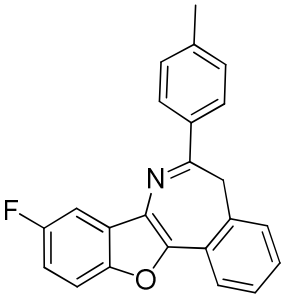


2p

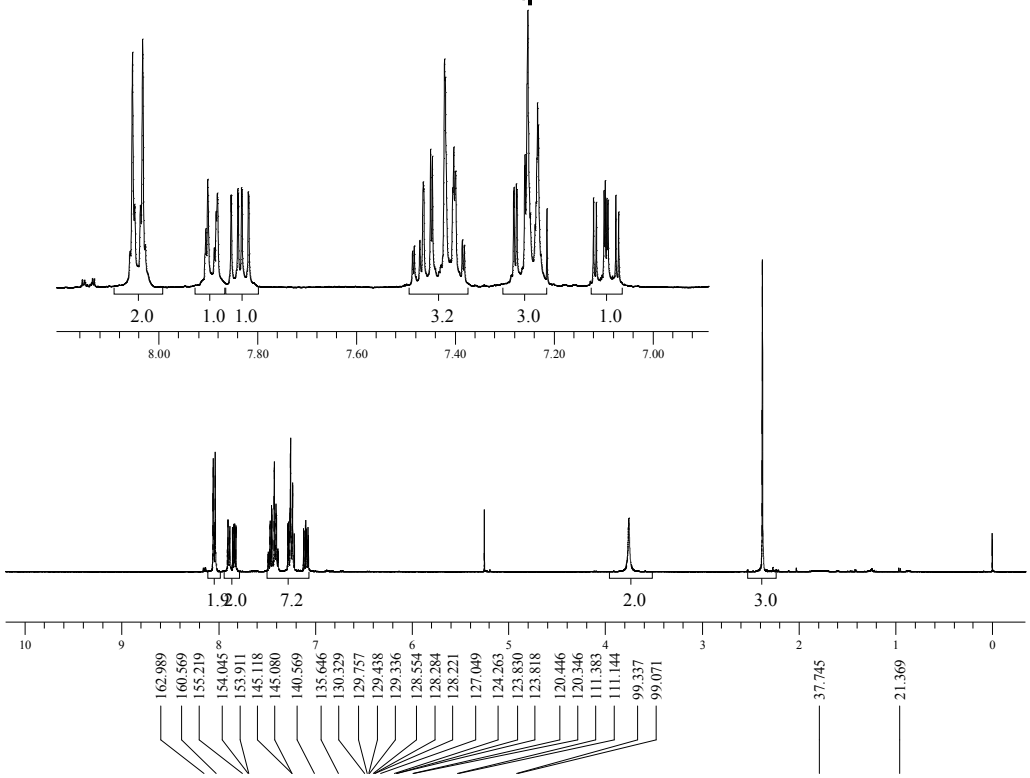


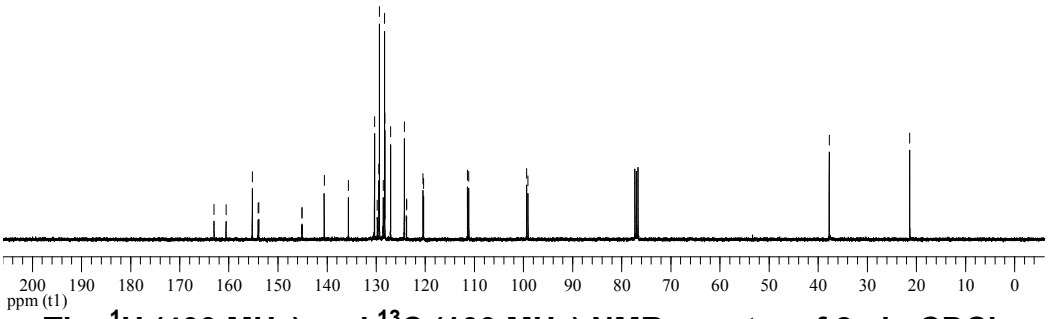


The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 2p in $CDCl_3$



2q





The ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 2q in $CDCl_3$