

Intermolecular Diels-Alder Cycloaddition for the Construction of Bicyclo[2.2.2]-diazaoctane Structures: Formal Synthesis of Brevianamide B and Premalbrancheamide

Jacob G. Robins, Kyu J. Kim, Alex J. Chinn, John S. Woo, and Jonathan R. Scheerer*

Department of Chemistry, The College of William & Mary, P. O. Box 8795, Williamsburg, Virginia, 23187, USA

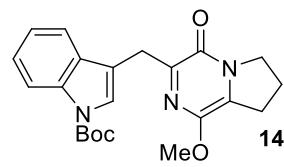
Supporting Information

1. General Information	S1
2. ^1H and ^{13}C NMR Spectra	S2 – S16

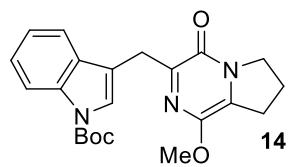
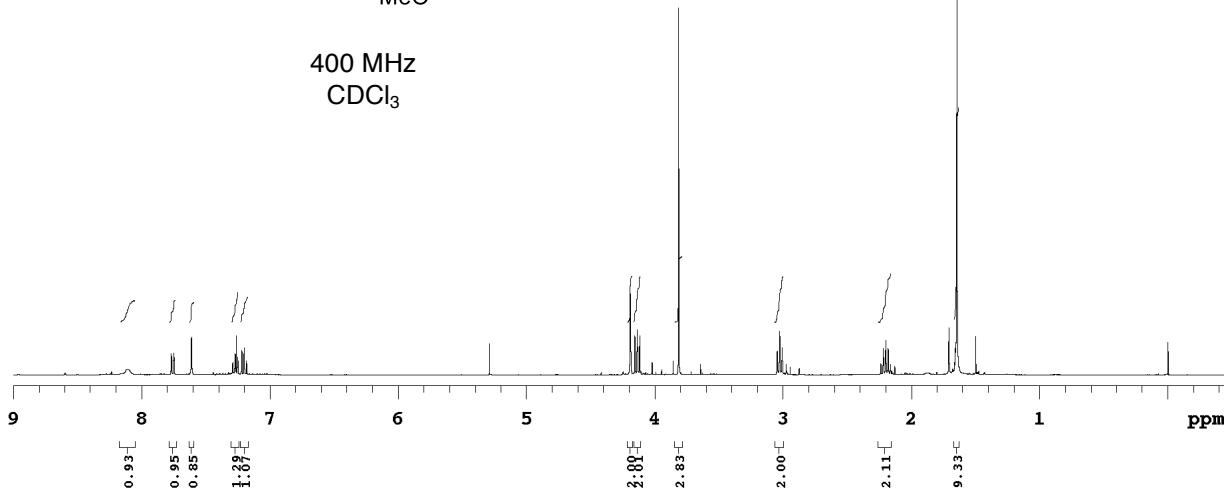
General Information. All reactions were carried out under an atmosphere of nitrogen in flame-dried or oven-dried glassware with magnetic stirring unless otherwise indicated. Acetonitrile, THF, toluene, and Et₂O were degassed with argon and purified by passage through a column of molecular sieves and a bed of activated alumina¹. Dichloromethane was distilled from CaH₂ prior of use. All reagents were used as received unless otherwise noted. Flash column chromatography² was performed using silica gel (230-400 mesh). Analytical thin layer chromatography was performed on 60Å glass plates. Visualization was accomplished with UV light, anisaldehyde, ceric ammonium molybdate (CAM), potassium permanganate, or ninhydrin, followed by heating. Film (or KBR pellet) infrared spectra were recorded using a FTIR spectrophotometer. Optical rotations were determined by a digital polarimeter at 25 °C. ^1H NMR spectra were recorded on a 400 MHz spectrometer and are reported in ppm using solvent as an internal standard (CDCl₃ at 7.26 ppm) or tetramethylsilane (0.00 ppm). Proton-decoupled ^{13}C NMR spectra were recorded on a 400 MHz spectrometer and are reported in ppm using solvent as an internal standard (CDCl₃ at 77.0 ppm). All compounds were judged to be homogeneous (>95% purity) by ^1H and ^{13}C NMR spectroscopy unless otherwise noted as mixtures. Mass spectra data analysis was obtained through positive electrospray ionization (ICR-MS w/NaCl).

¹ Pangborn, A.B.; Giardello, M.A.; Grubbs, R.H.; Rosen, R.K.; Timmers, F. J., *Organometal.* **1996**, *15*, 1518-1520.

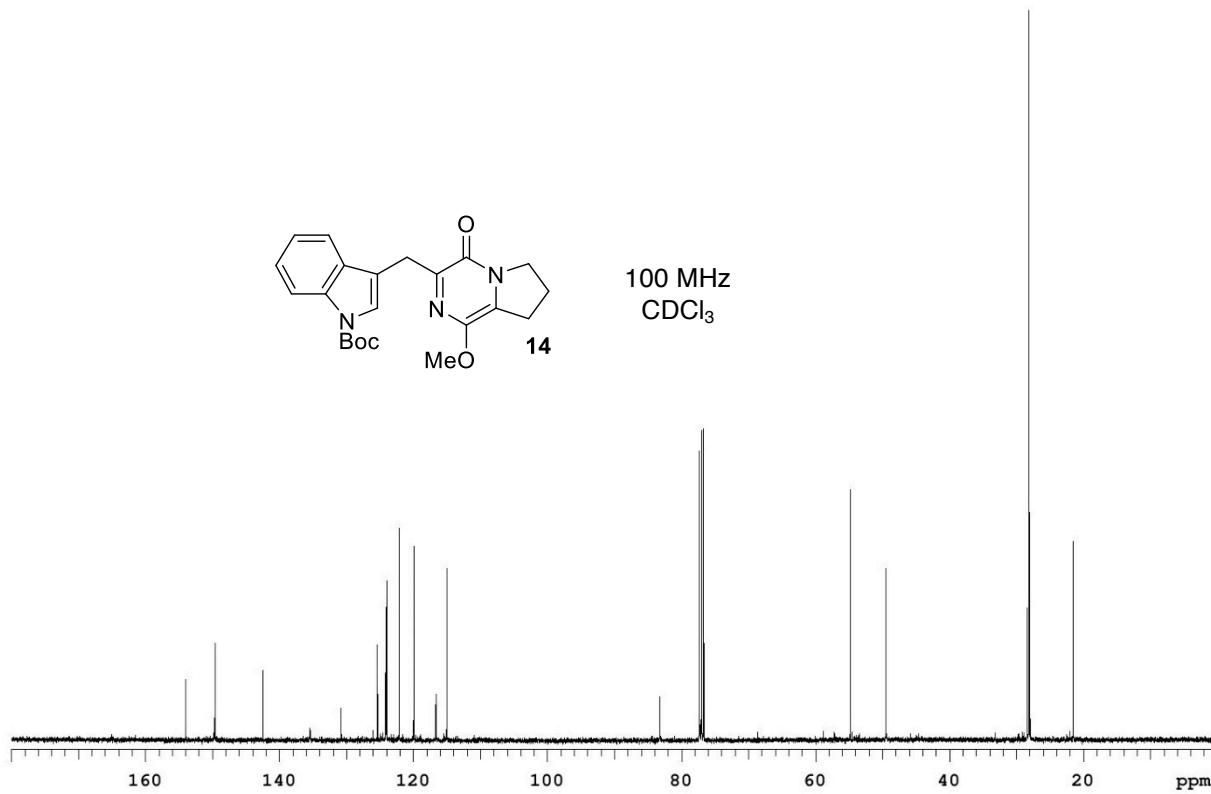
² Still, W.C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923-2925.

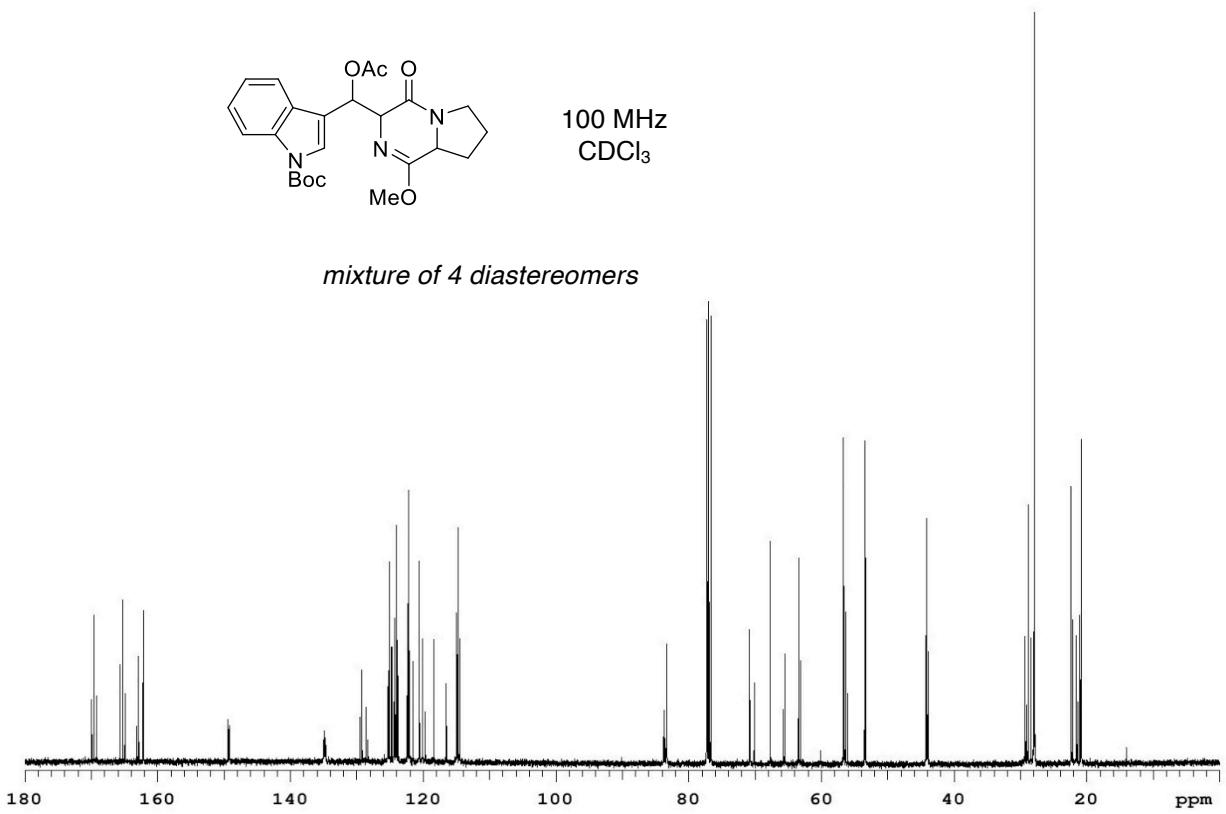
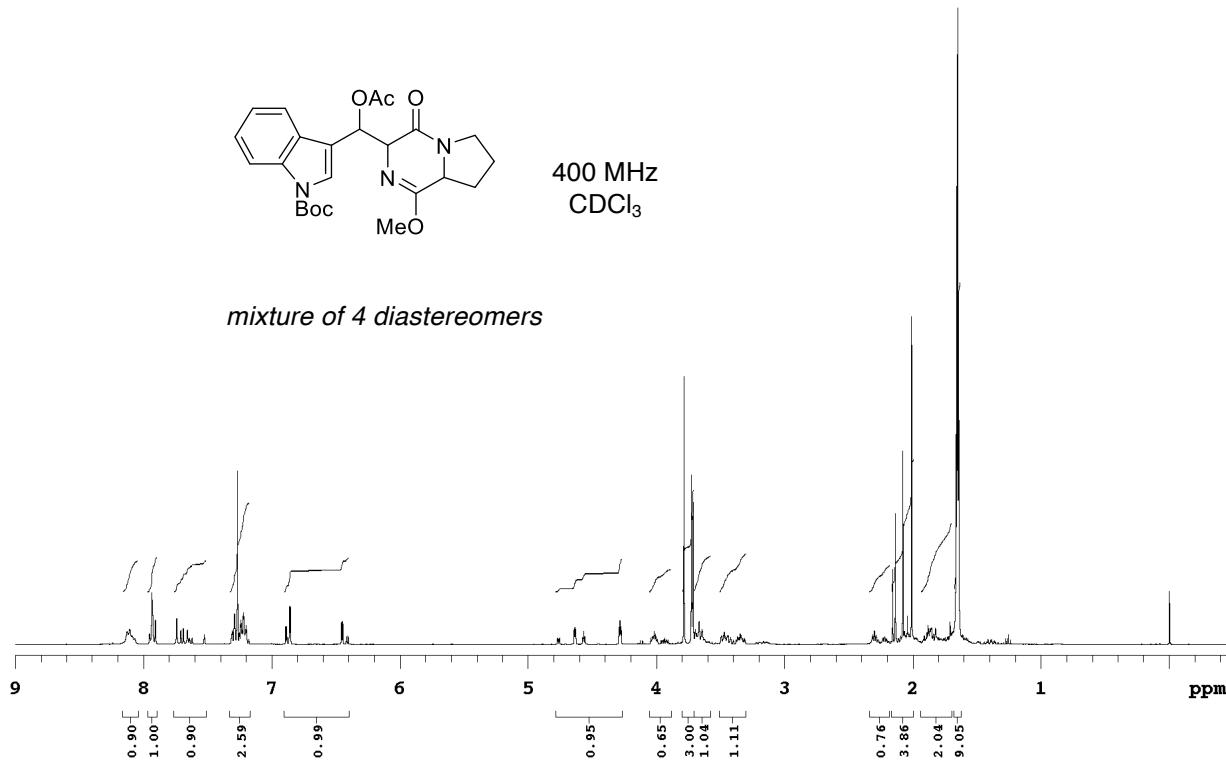


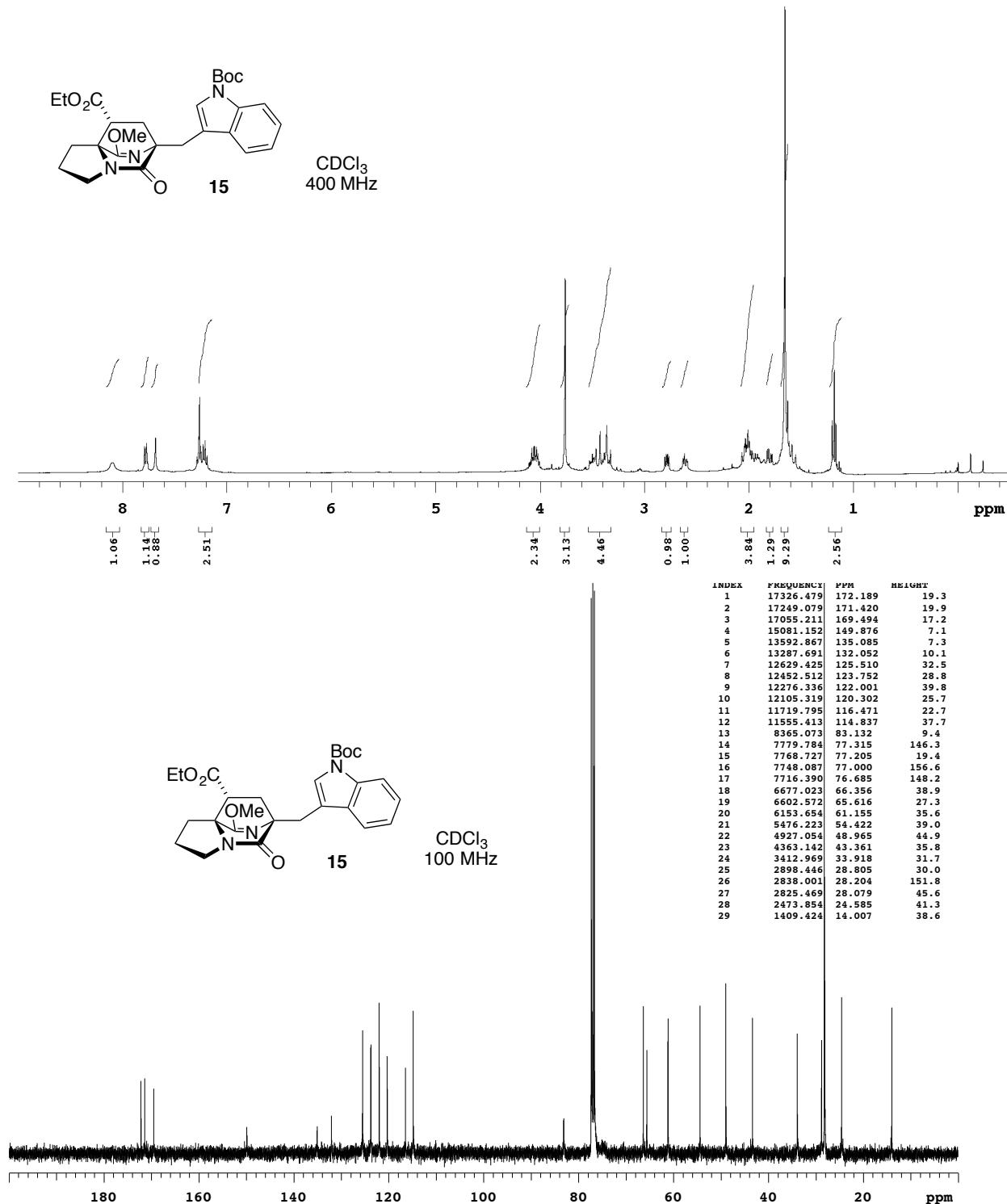
400 MHz
CDCl₃

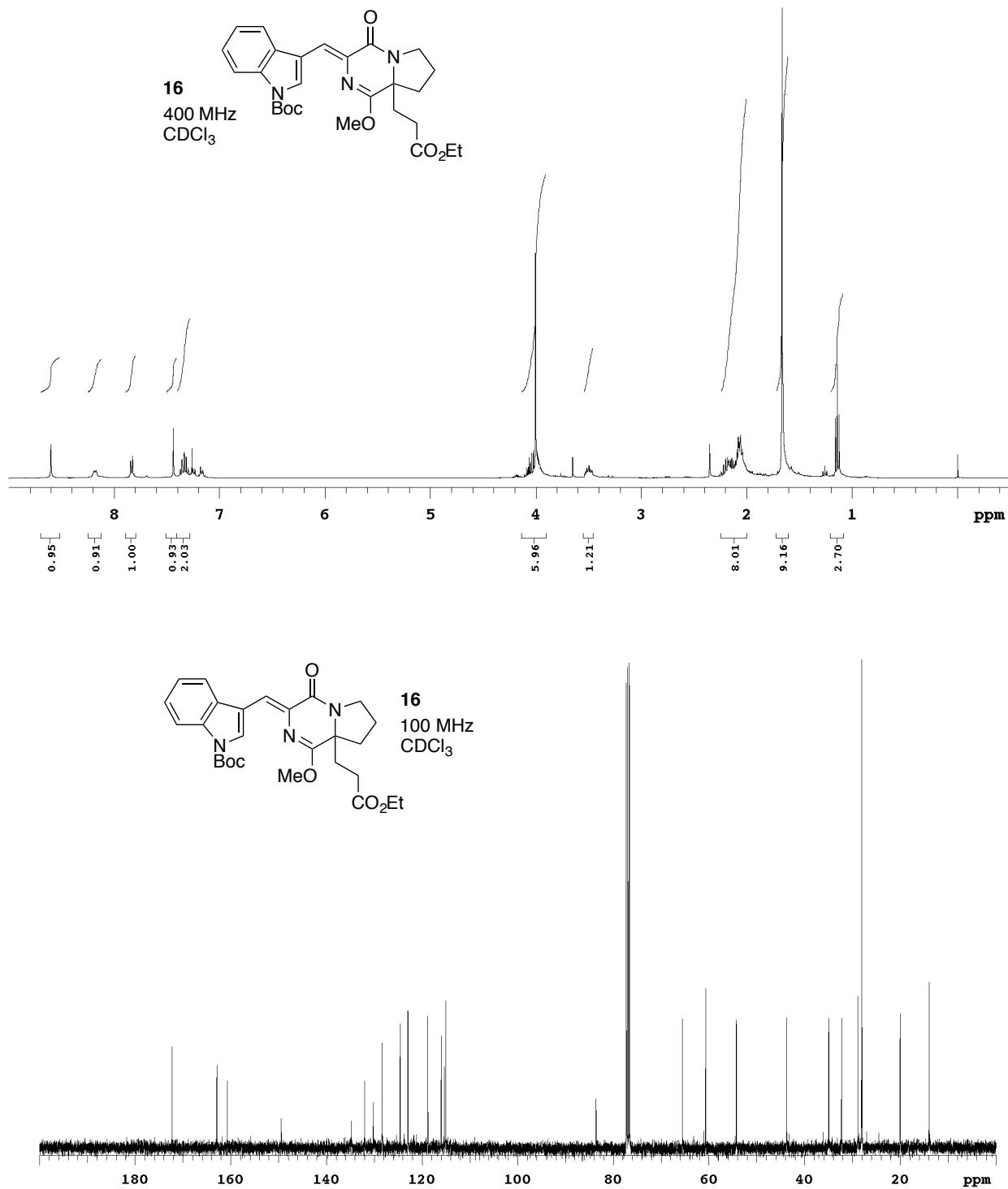


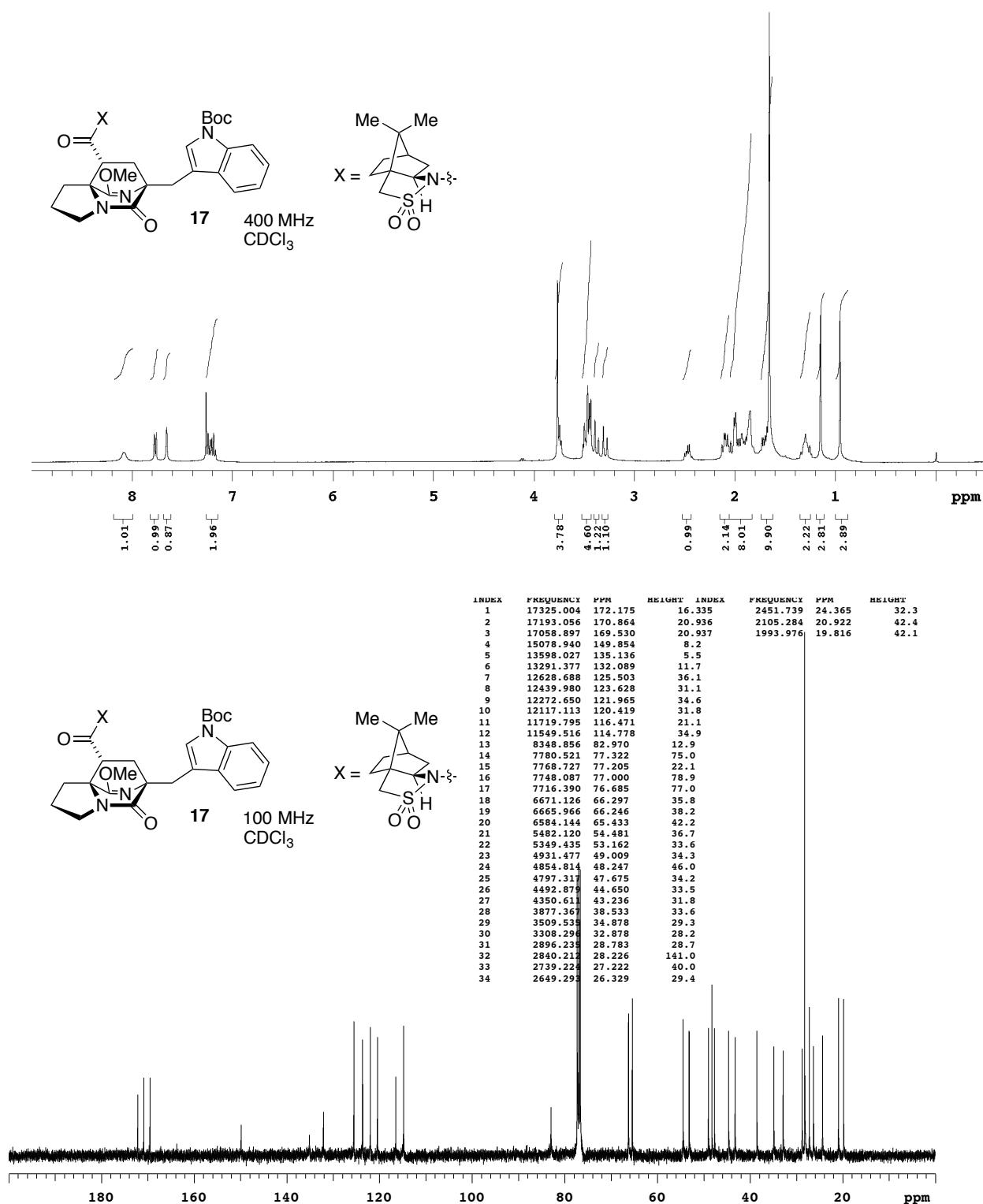
100 MHz
CDCl₃

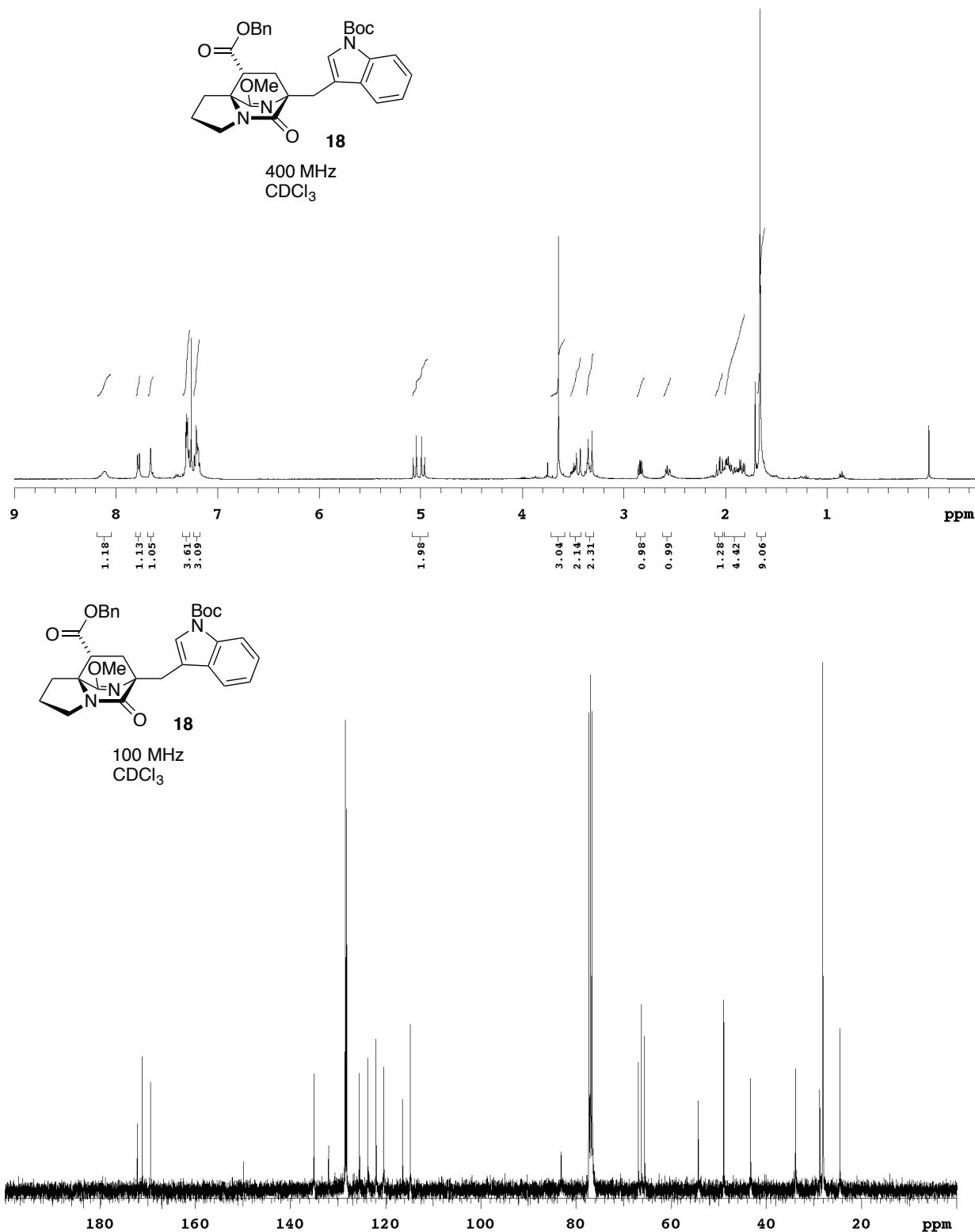


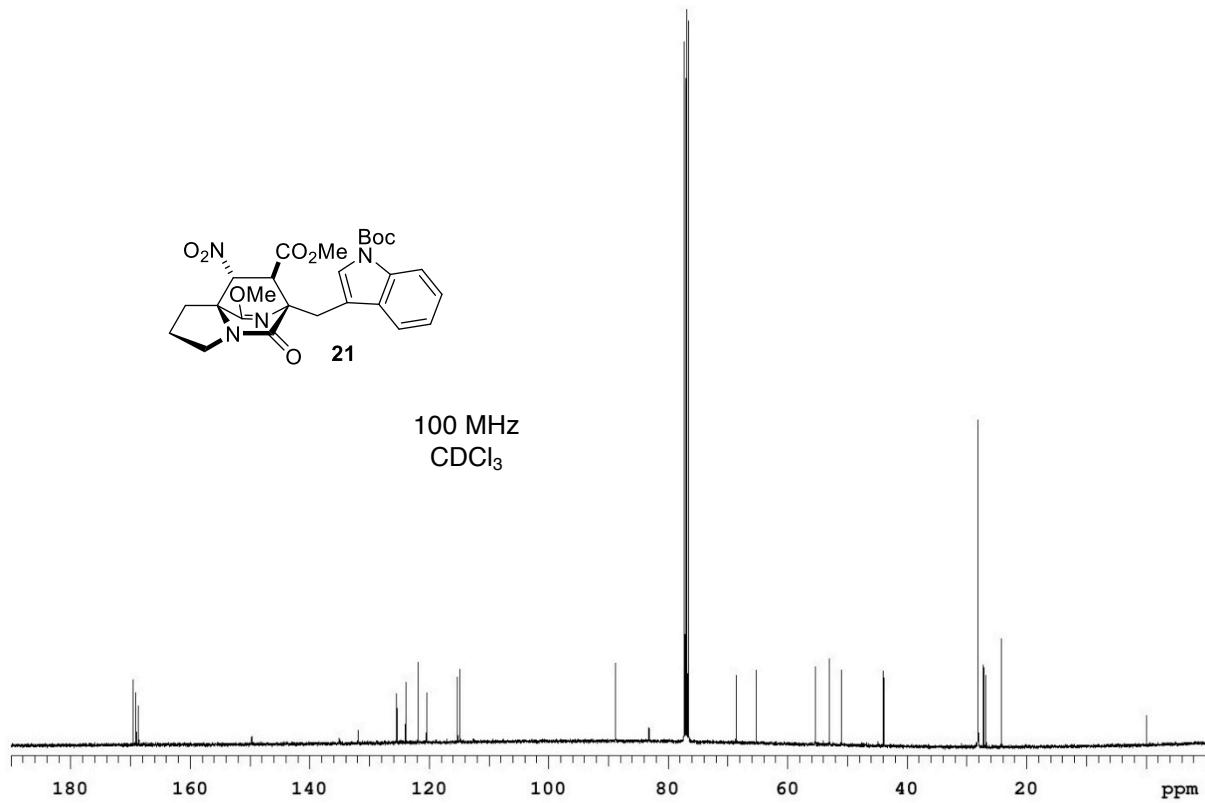
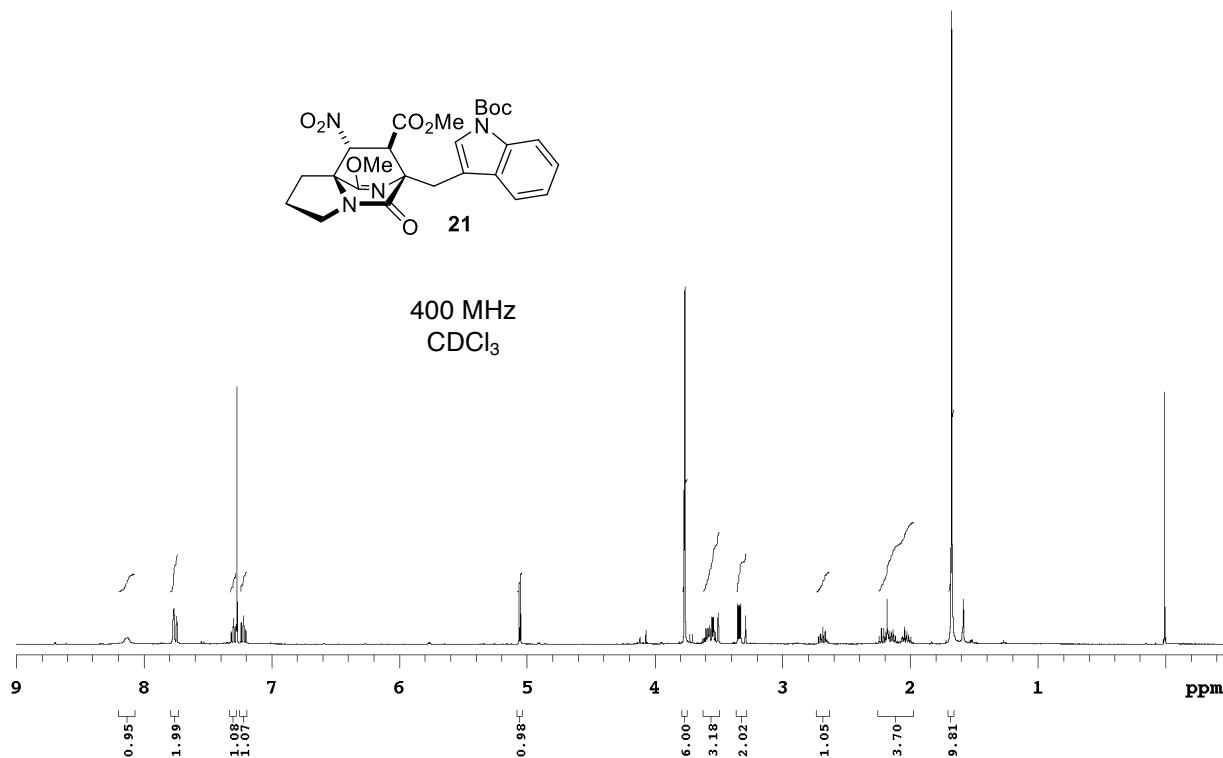


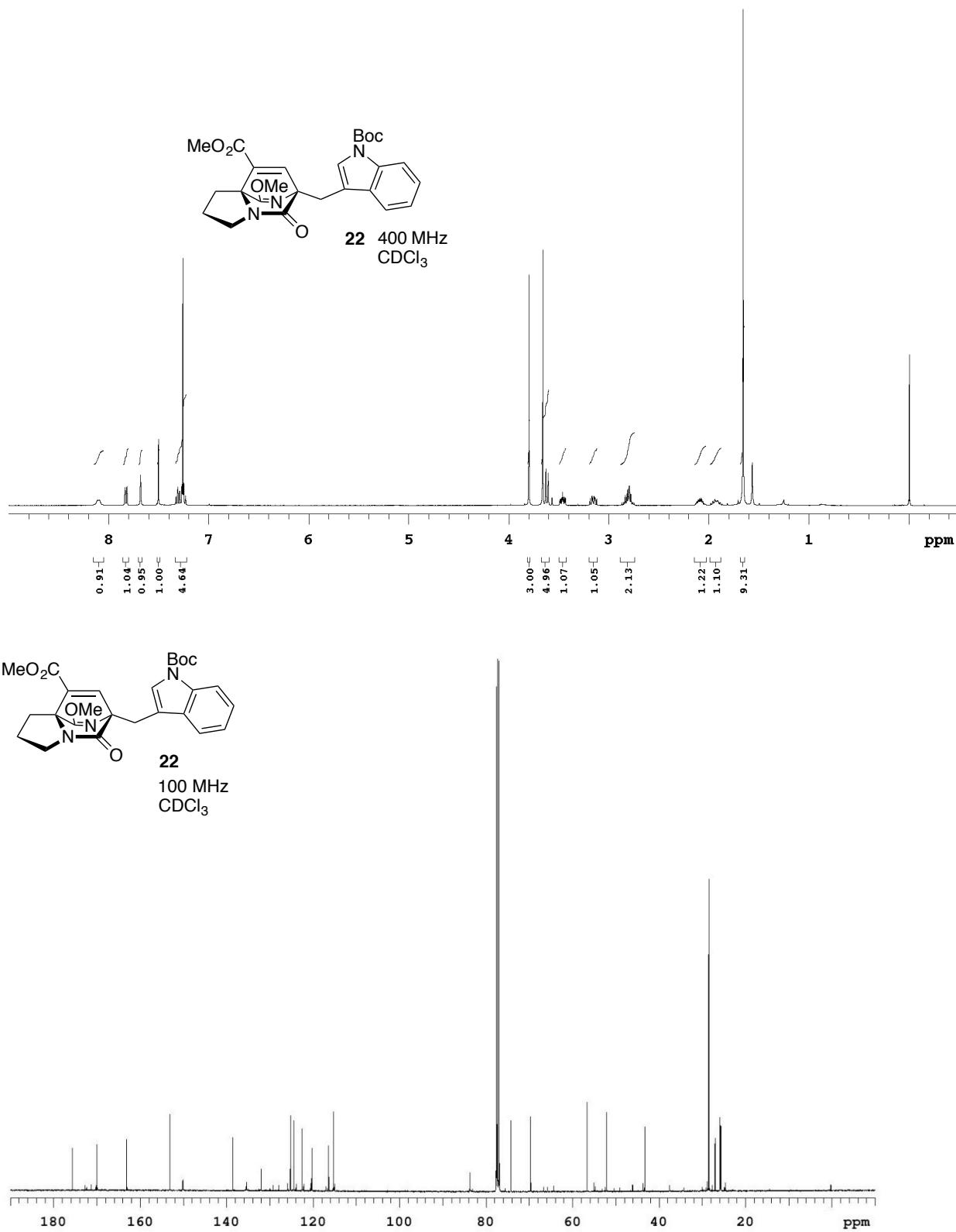


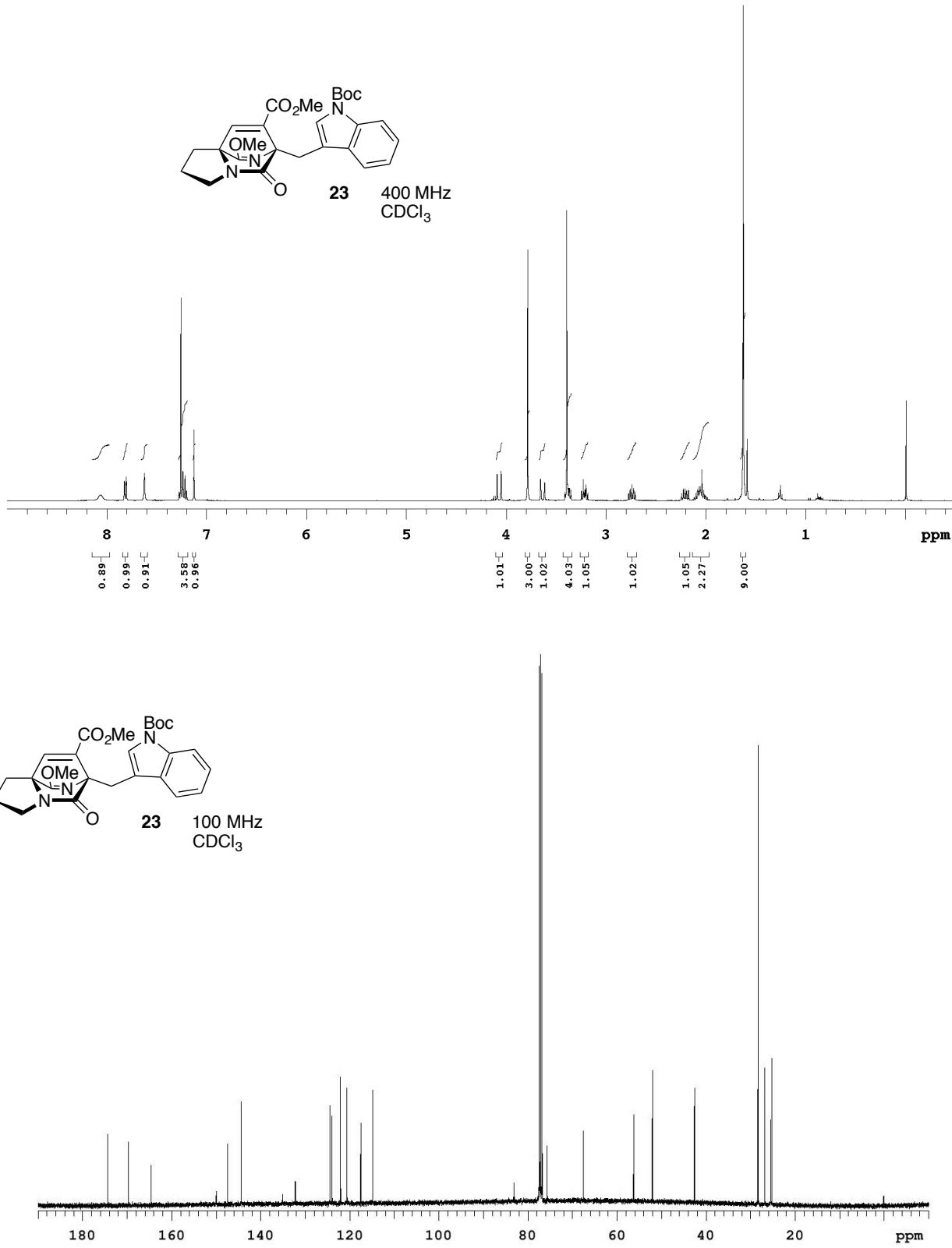


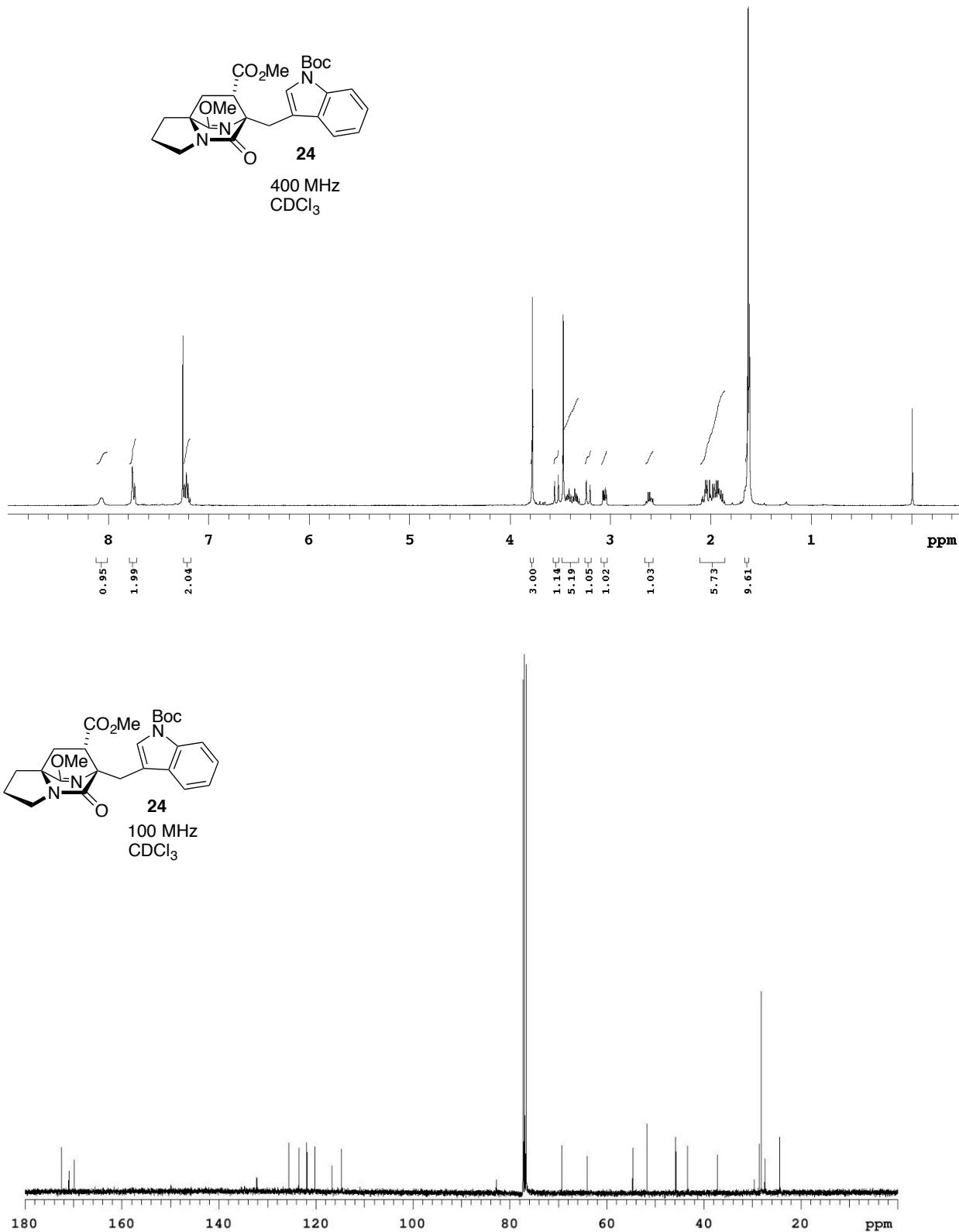


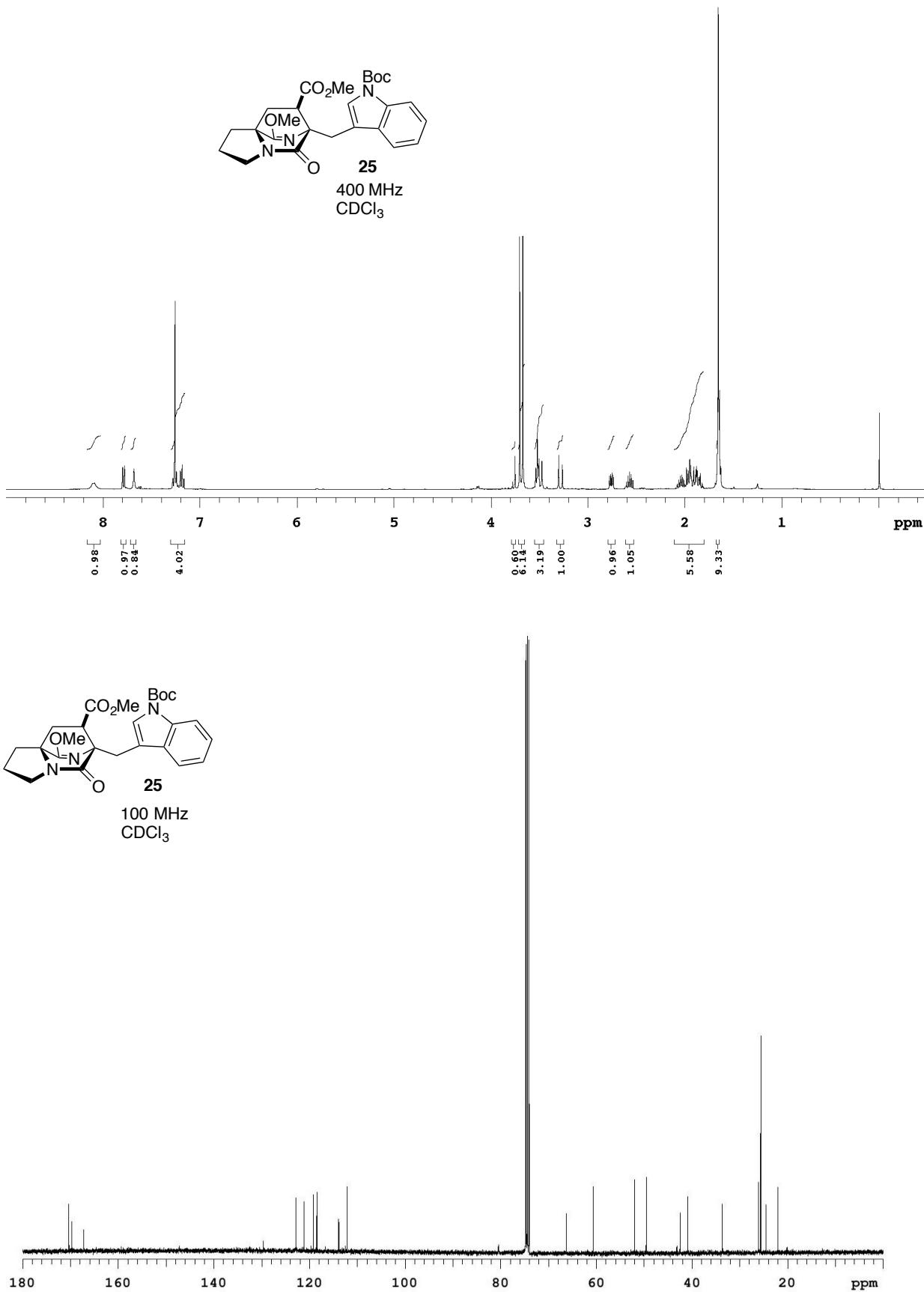


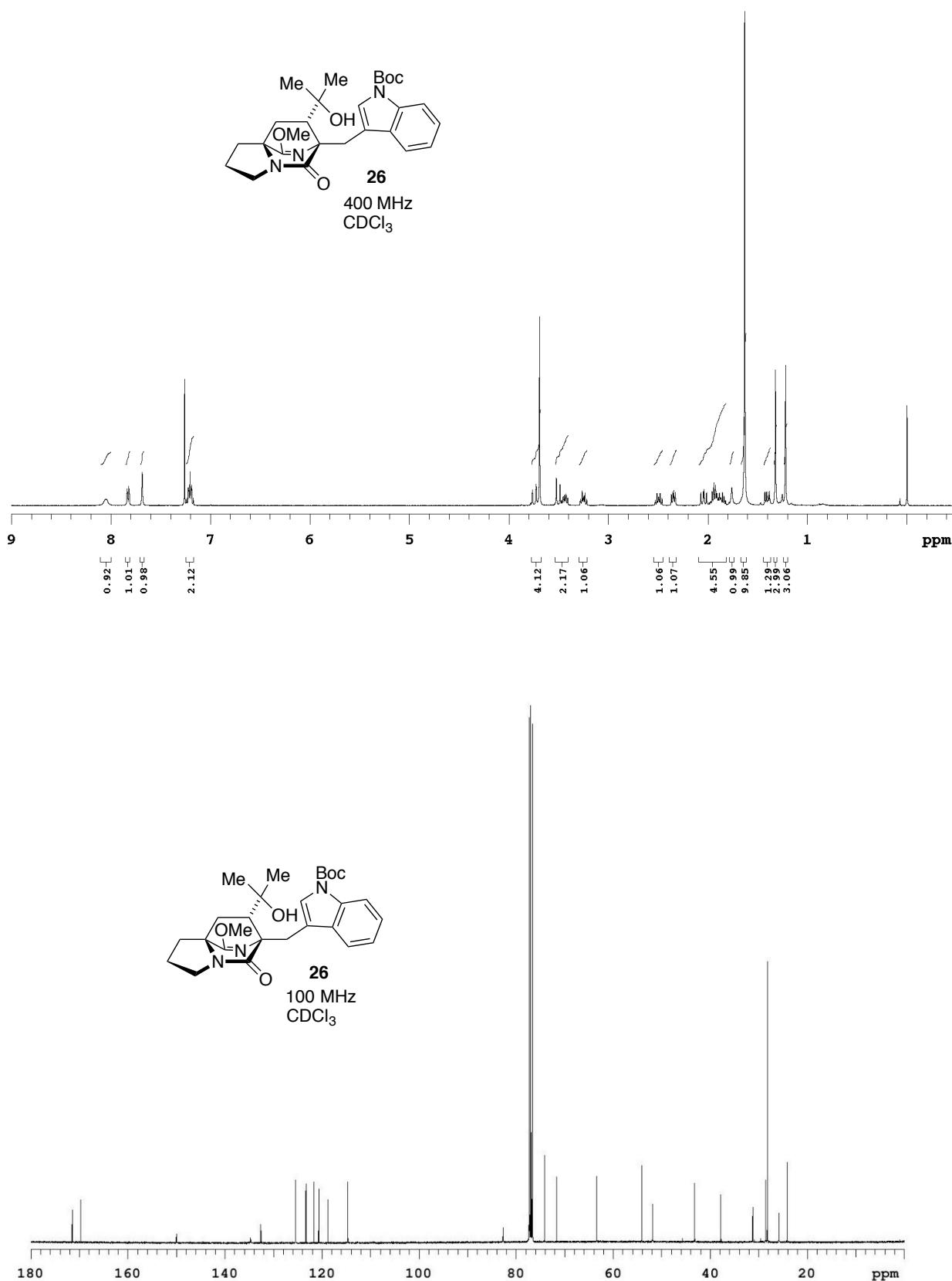


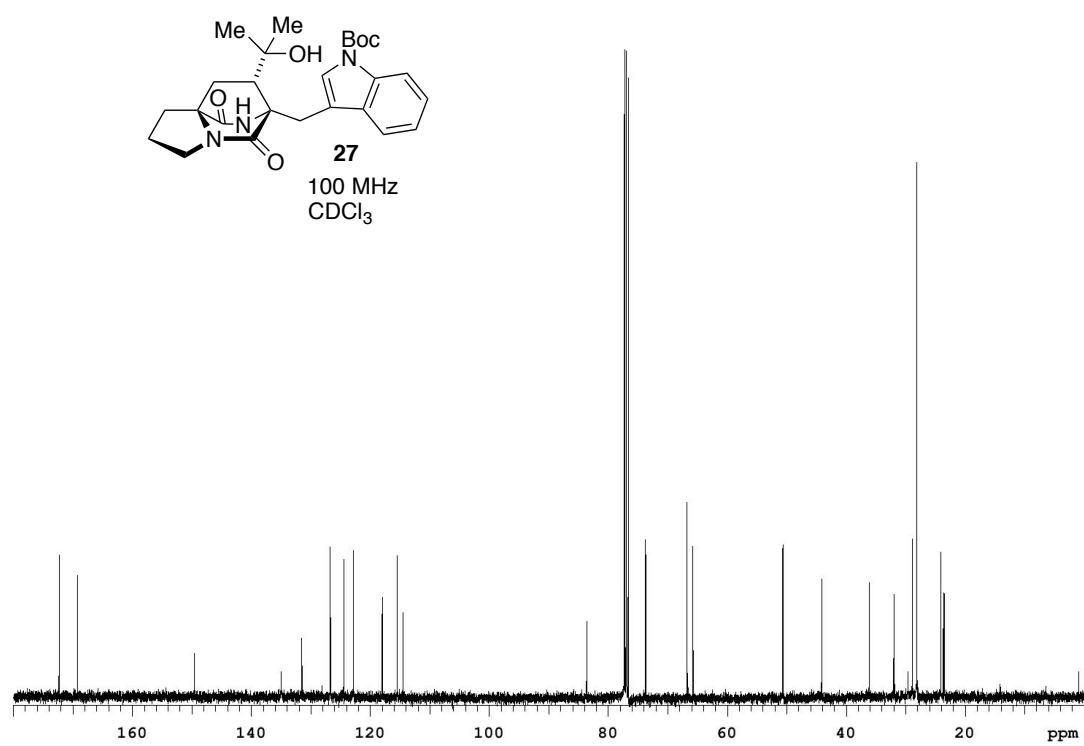
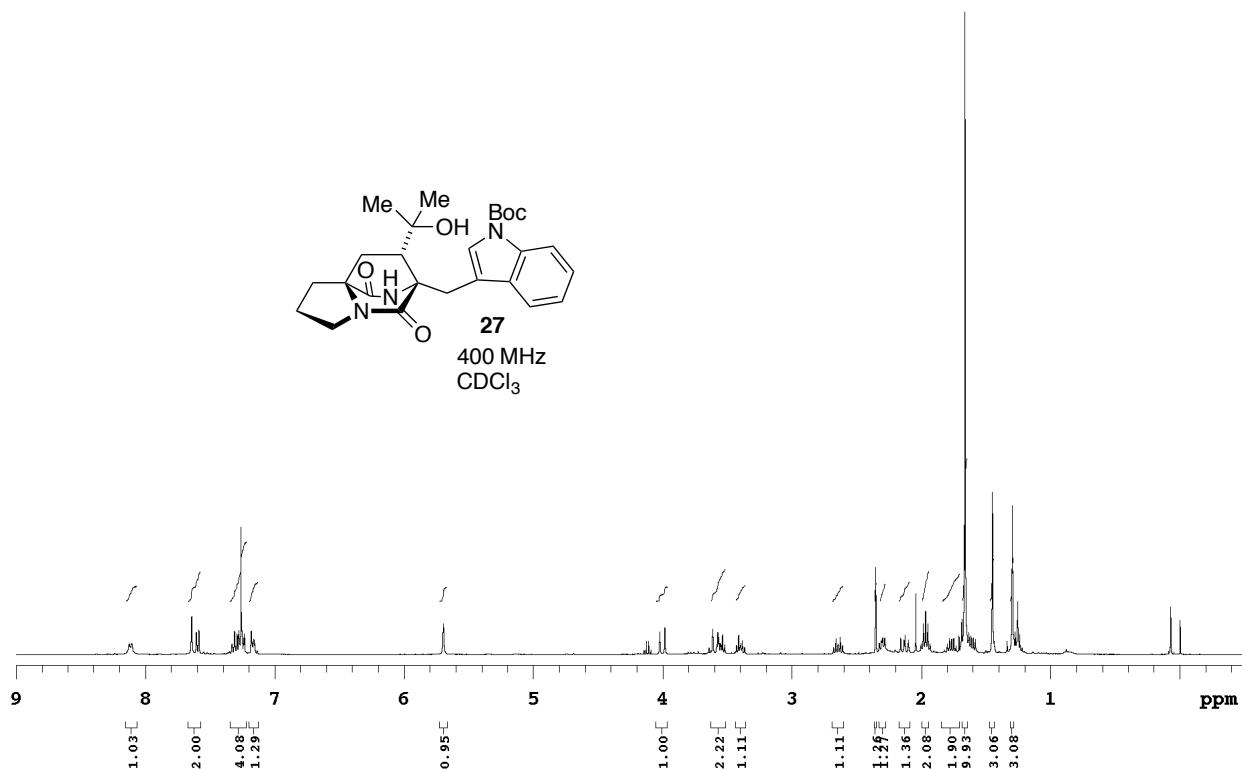


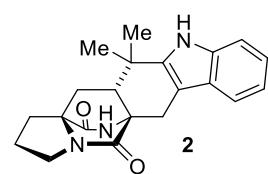
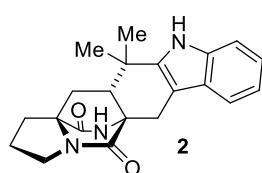
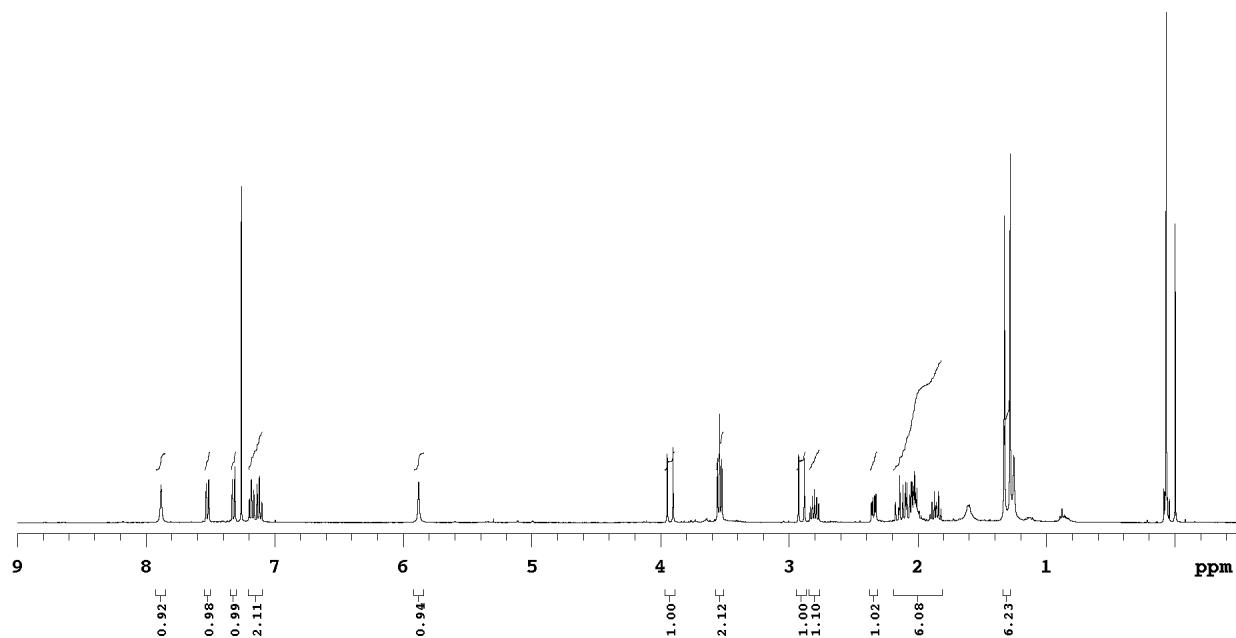










400 MHz
 CDCl_3 100 MHz
 CDCl_3 