Journal of Organic Chemistry, Note

SUPPORTING INFORMATION

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"Expedient five-step synthesis of SIB-1508Y from natural nicotine"

I. General Experimental Methods----------(S2)

II. ^{1}H and/or ^{13}C NMR spectra for **6-8** and **9-12**.

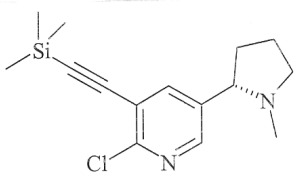
6, ^{1}H, ^{13}C----------(S3, S4)
7, ^{1}H, ^{13}C----------(S5, S6)
8, ^{1}H ----------(S7)
9, ^{1}H, ^{13}C----------(S8, S9)
10, ^{1}H, ^{13}C----------(S10, S11)
1, ^{1}H, ^{13}C----------(S12, S13)
11, ^{1}H, ^{13}C----------(S14, S15)
12, ^{1}H, ^{13}C----------(S16, S17)

Total pages of supporting information: (17).

General Experimental Methods

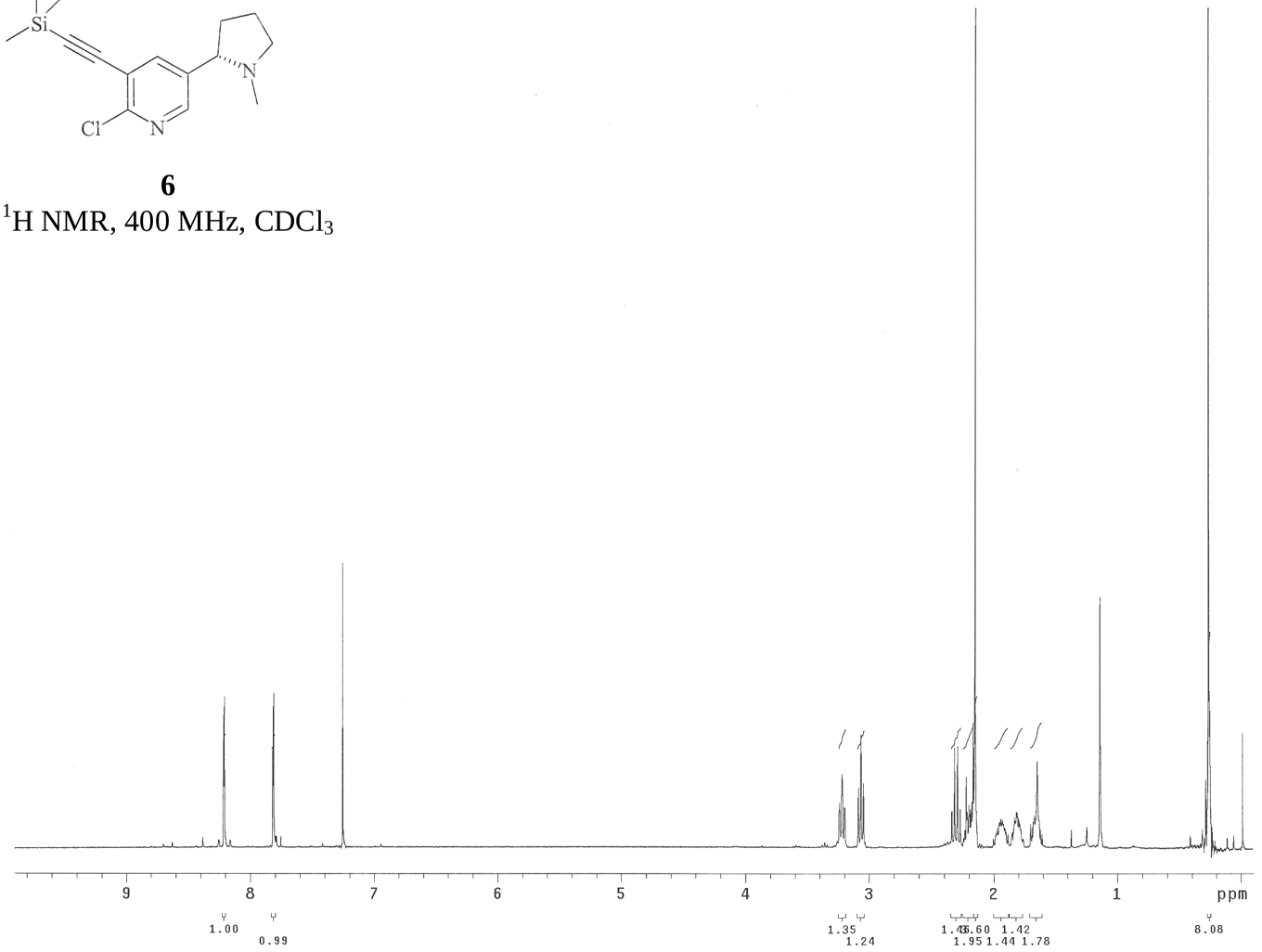
All reactions were performed in oven or flame-dried glassware under a argon atmosphere and stirred magnetically. THF, Et_2O and toluene were distilled from sodium/benzophenone ketyl prior to use. Triethylamine, diisopropylamine and methylene chloride were distilled from calcium hydride and stored under argon over 4Å molecular sieves. *n*-Butyllithium was titrated against diphenylacetic acid according to the procedure of Kofron and Baclawski.[i] Other reagents and solvents from commercial sources were stored under argon and used directly. Melting points were measured on a capillary melting point apparatus and are uncorrected. Radial preparative layer chromatography (radial PLC) was performed using glass plates coated with 1, 2 or 4 mm layers of Kieselgel 60 PF254 containing gypsum. High-resolution mass spectral analysis (HRMS) was performed at North Carolina State University. Optical rotations were measured on a precision automated polarimeter. NMR spectra were recorded on a 300 or 400 MHz spectrometer. *Chemical shifts* are reported in ppm. Coupling constants (*J* values) are reported in Hertz. IR spectra were recorded on a FT-IR spectrometer. Chemical shifts are in ppm units with TMS (0.0 ppm) used as the internal standard for ^{1}H NMR spectra and the $CDCl_3$ absorption of 77.23 ppm for ^{13}C NMR.

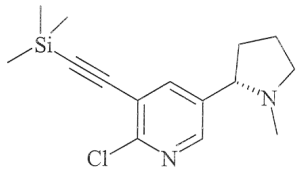
(i) Kofron, W. G.; Baclawski, L. M. *J. Org. Chem.* **1976**, *41*, 1879.



6

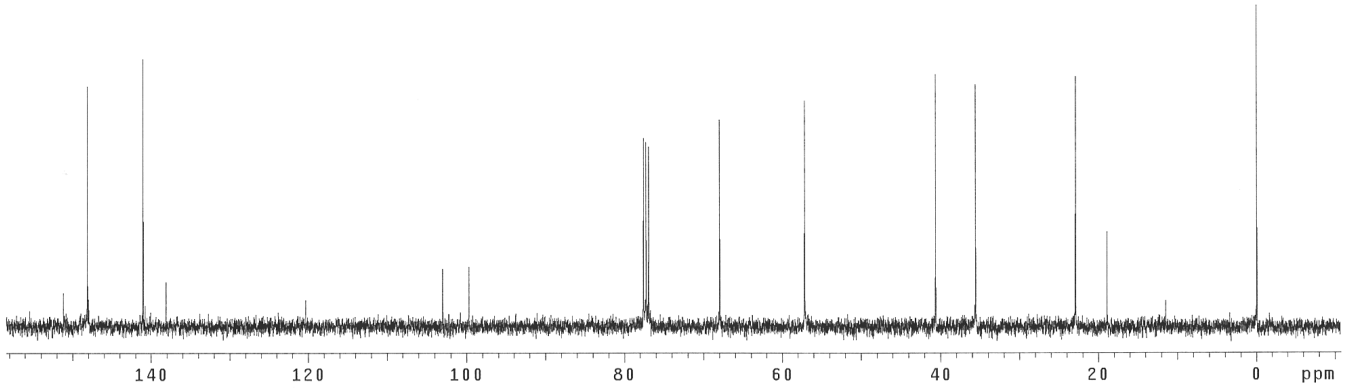
^{1}H NMR, 400 MHz, $CDCl_3$

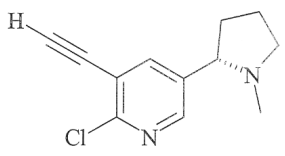




6

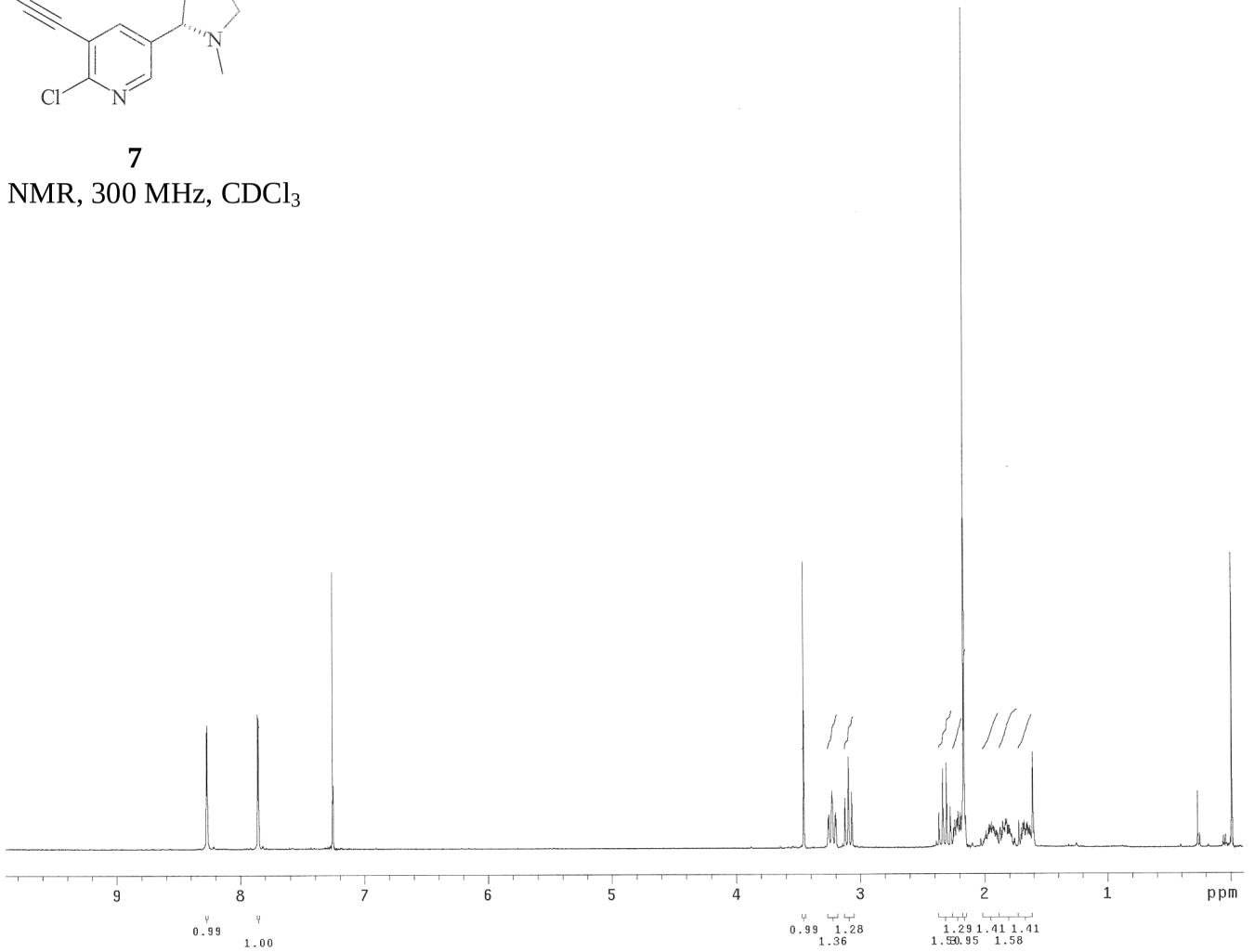
^{13}C NMR, 100 MHz, $CDCl_3$

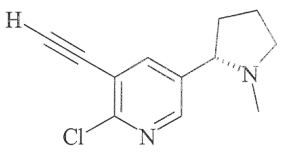




7

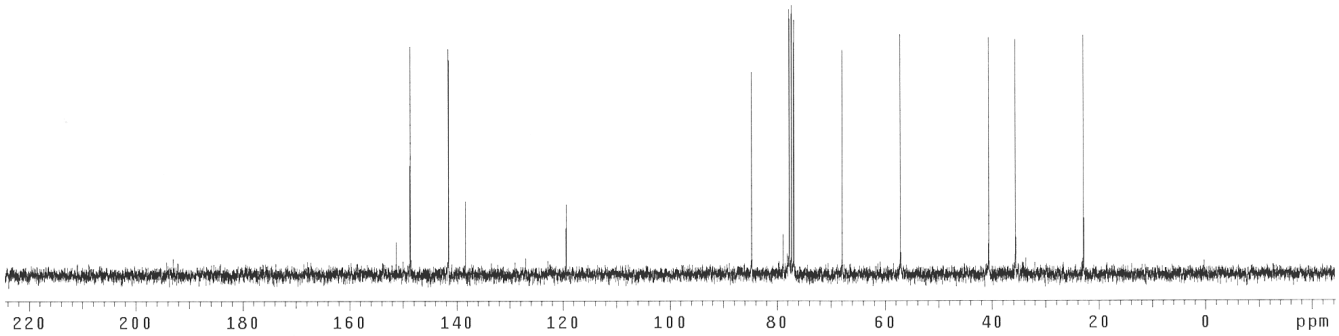
^{1}H NMR, 300 MHz, $CDCl_3$





7

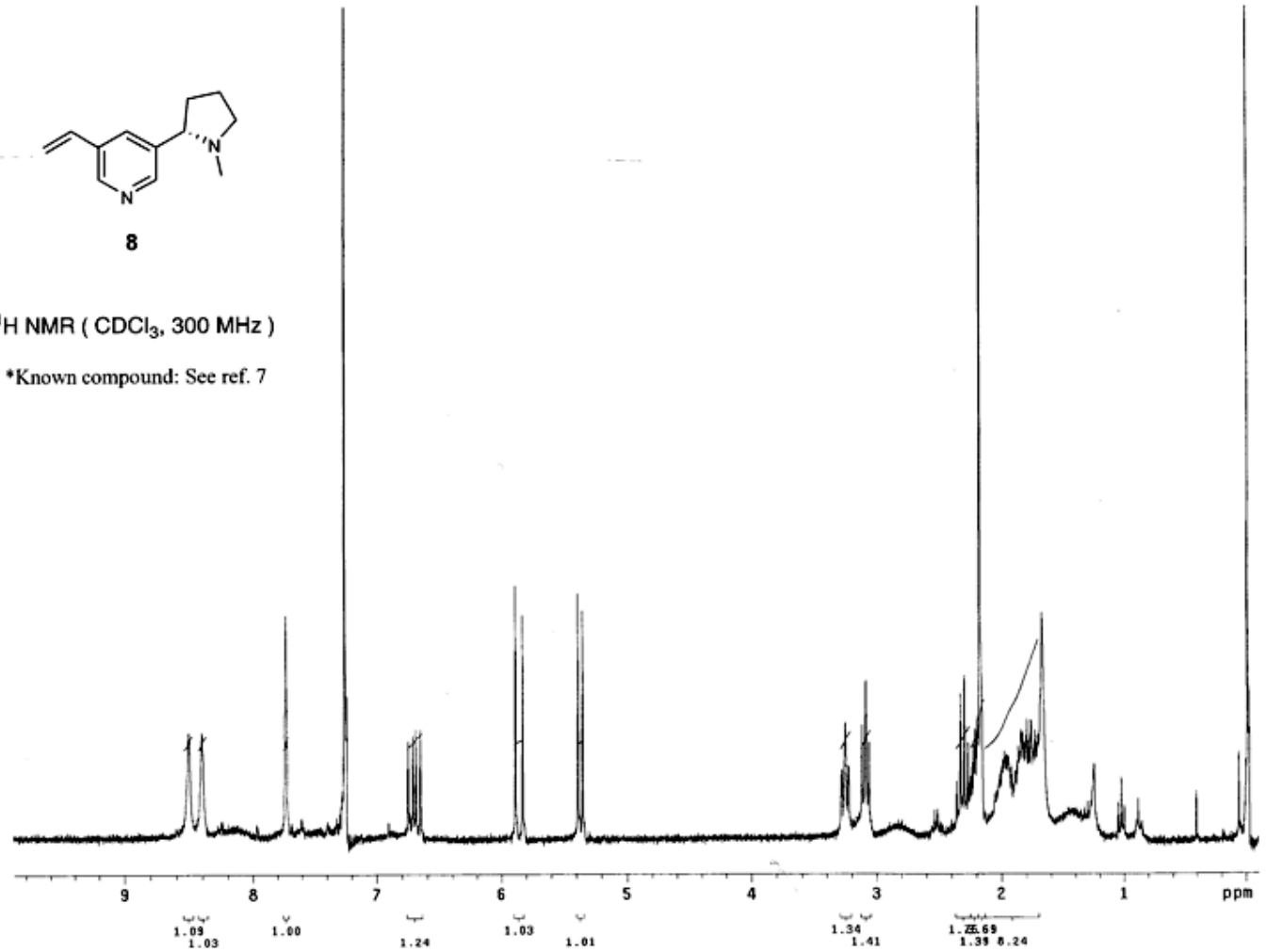
^{13}C NMR, 75 MHz, $CDCl_3$

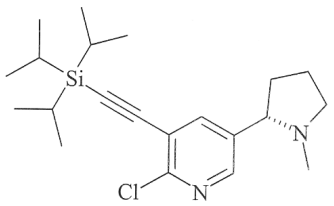


8

^{1}H NMR ($CDCl_3$, 300 MHz)

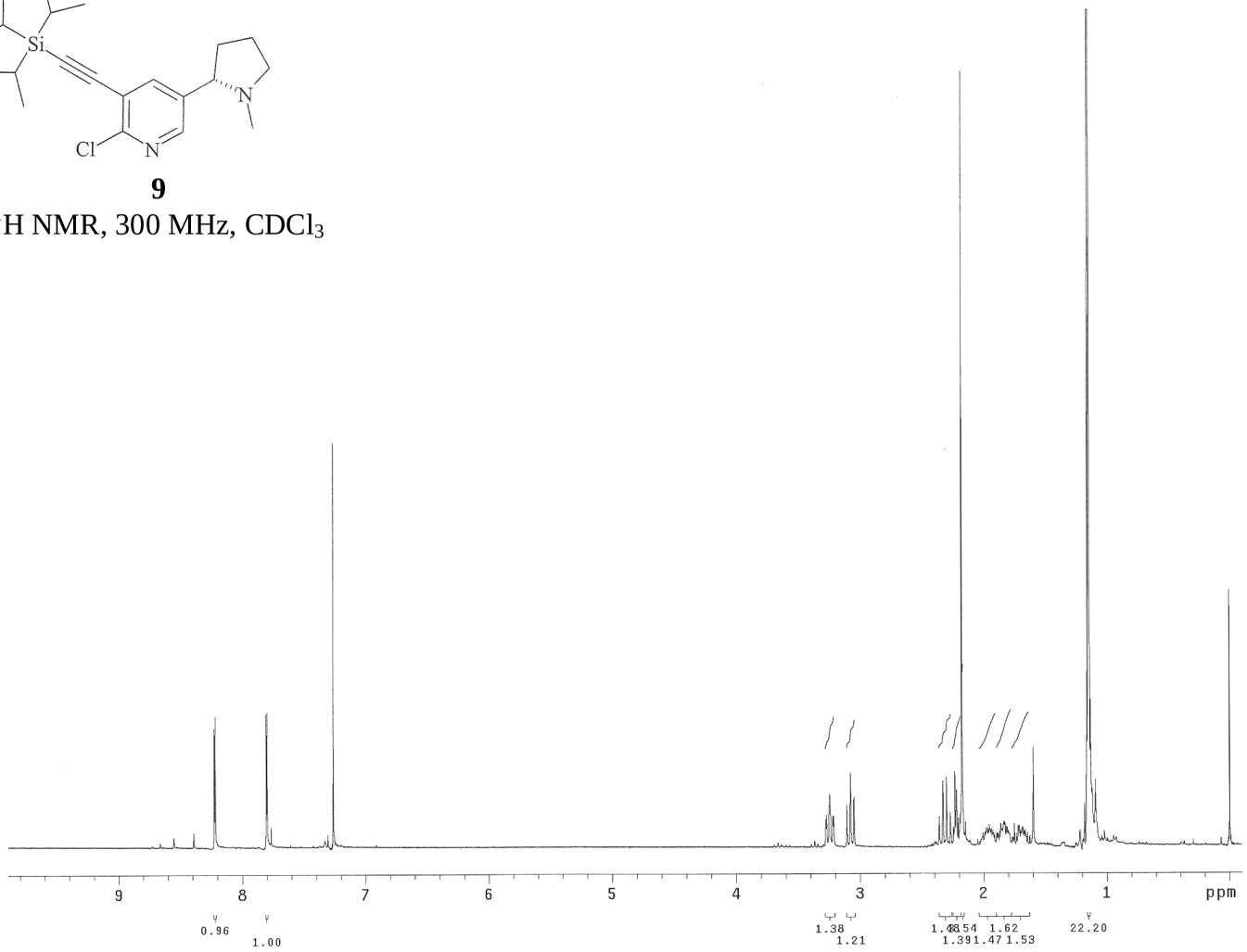
*Known compound: See ref. 7

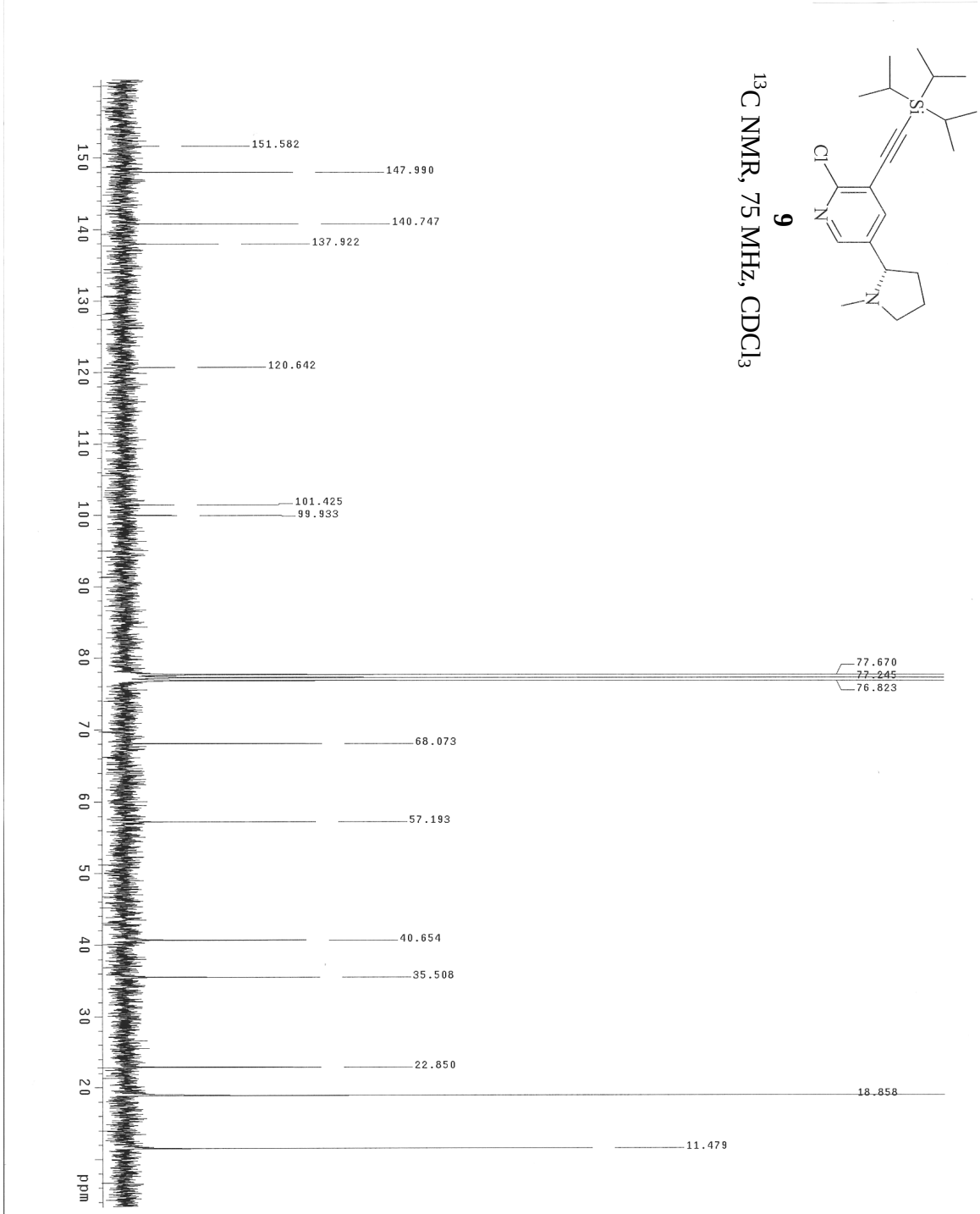




9

^{1}H NMR, 300 MHz, $CDCl_3$



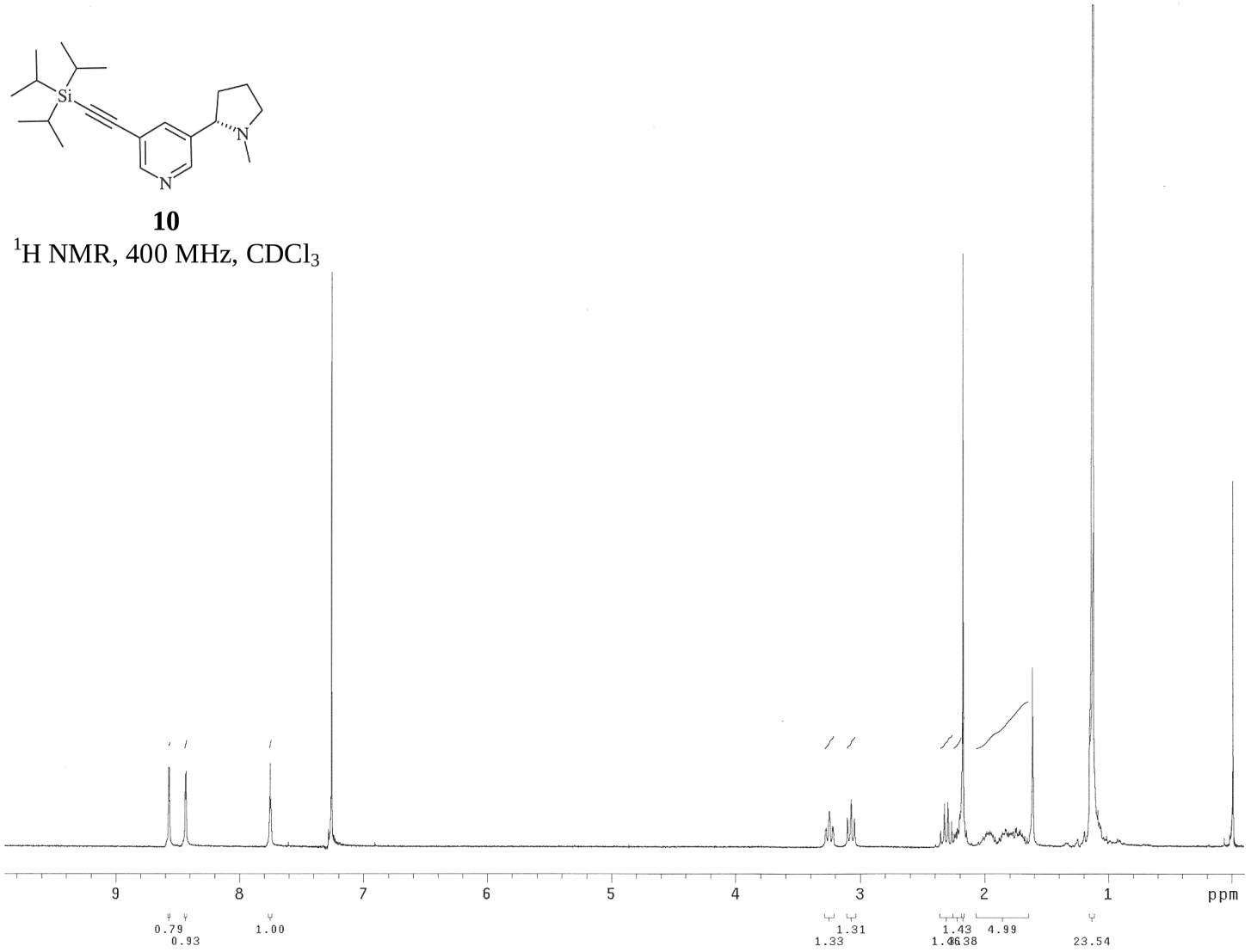


9

^{13}C NMR, 75 MHz, $CDCl_3$

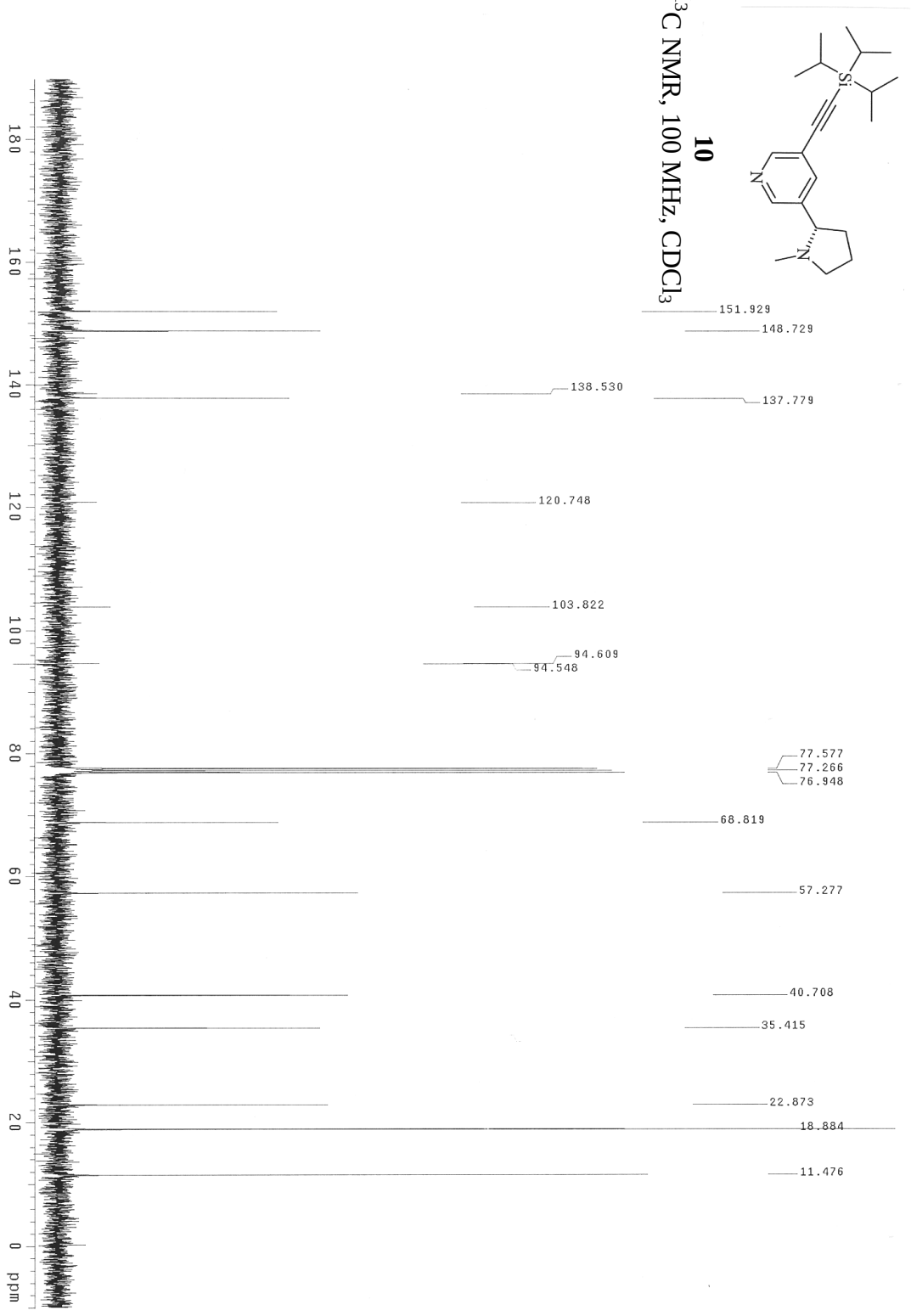
10

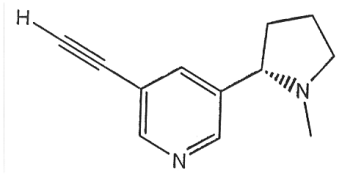
^{1}H NMR, 400 MHz, $CDCl_3$



10

^{13}C NMR, 100 MHz, $CDCl_3$

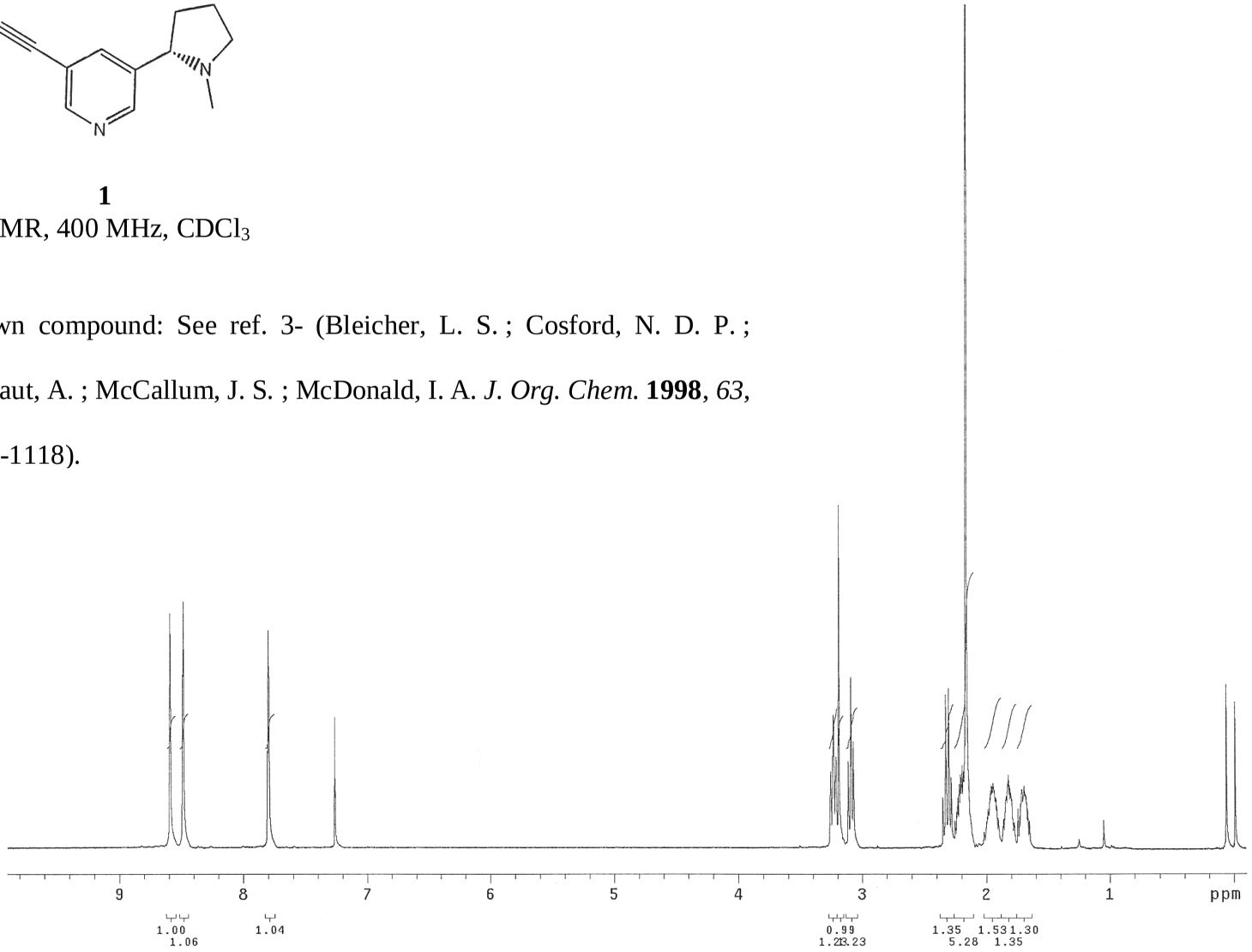


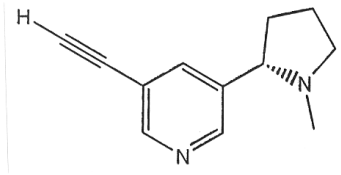


1

^{1}H NMR, 400 MHz, $CDCl_3$

Known compound: See ref. 3- (Bleicher, L. S. ; Cosford, N. D. P. ; Herbaut, A. ; McCallum, J. S. ; McDonald, I. A. *J. Org. Chem.* **1998**, *63*, 1109-1118).

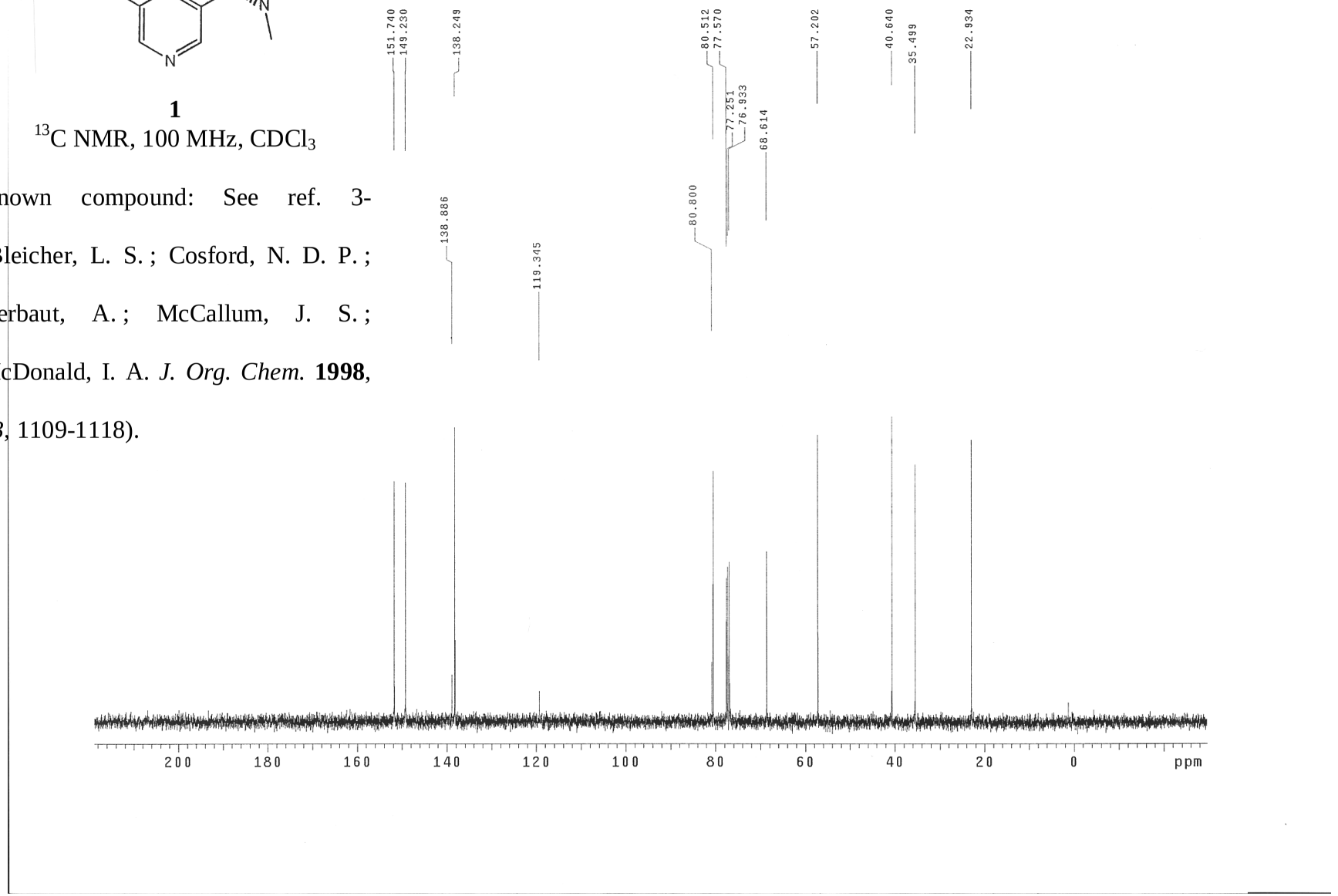


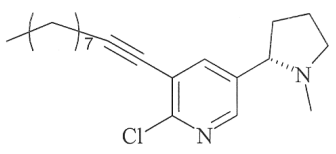


1

^{13}C NMR, 100 MHz, $CDCl_3$

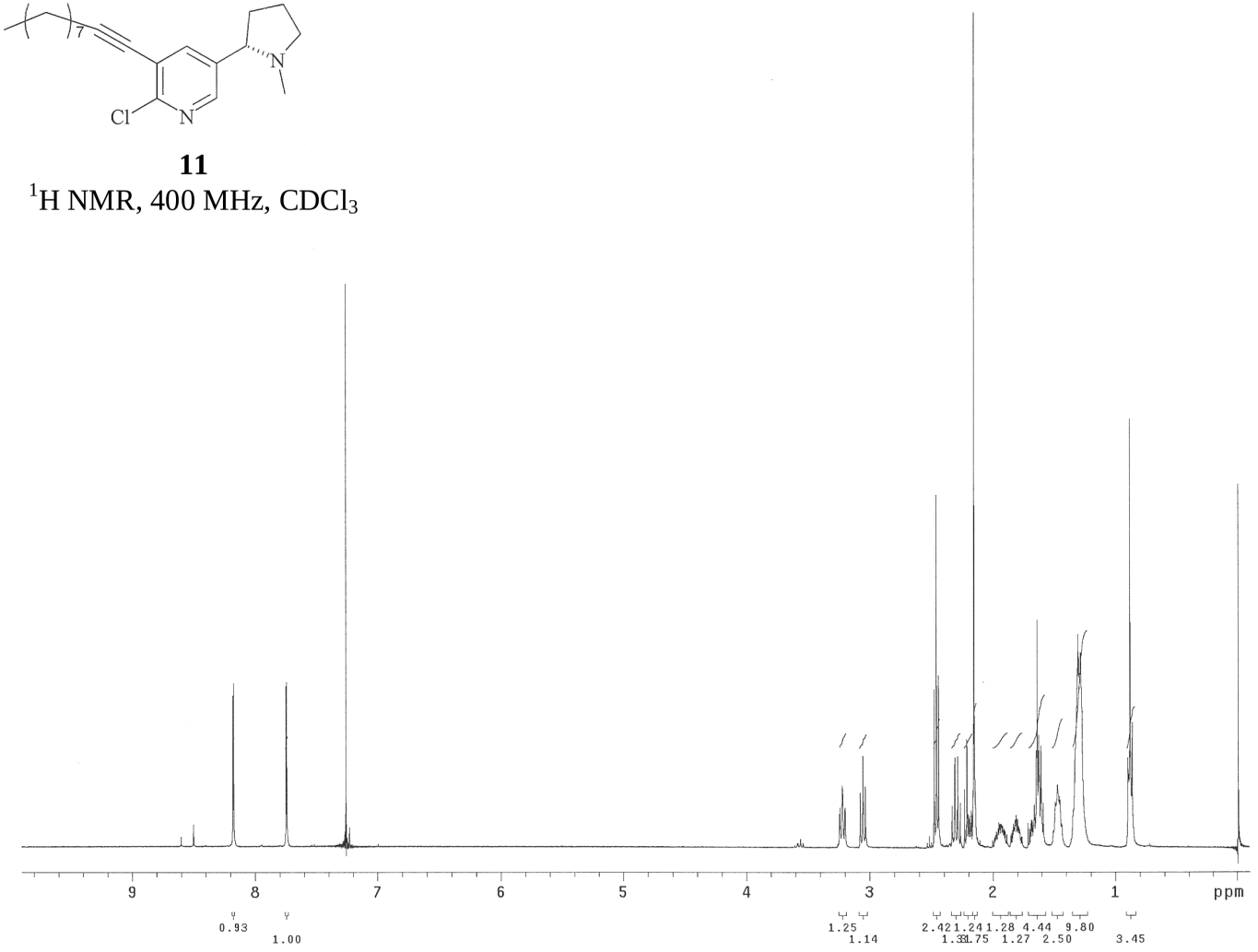
Known compound: See ref. 3-(Bleicher, L. S. ; Cosford, N. D. P. ; Herbaut, A. ; McCallum, J. S. ; McDonald, I. A. *J. Org. Chem.* **1998**, *63*, 1109-1118).





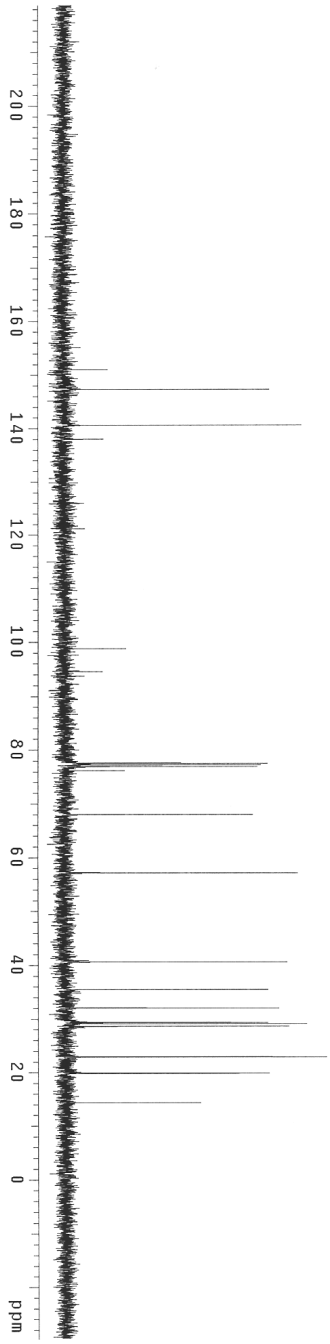
11

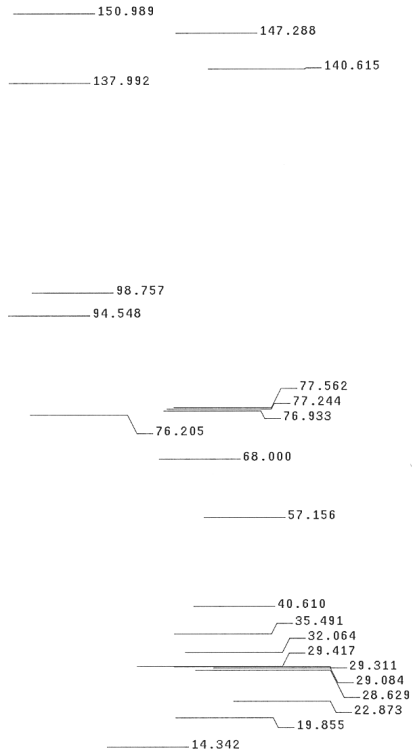
^{1}H NMR, 400 MHz, $CDCl_3$

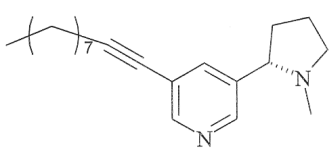


11

^{13}C NMR, 100 MHz, $CDCl_3$

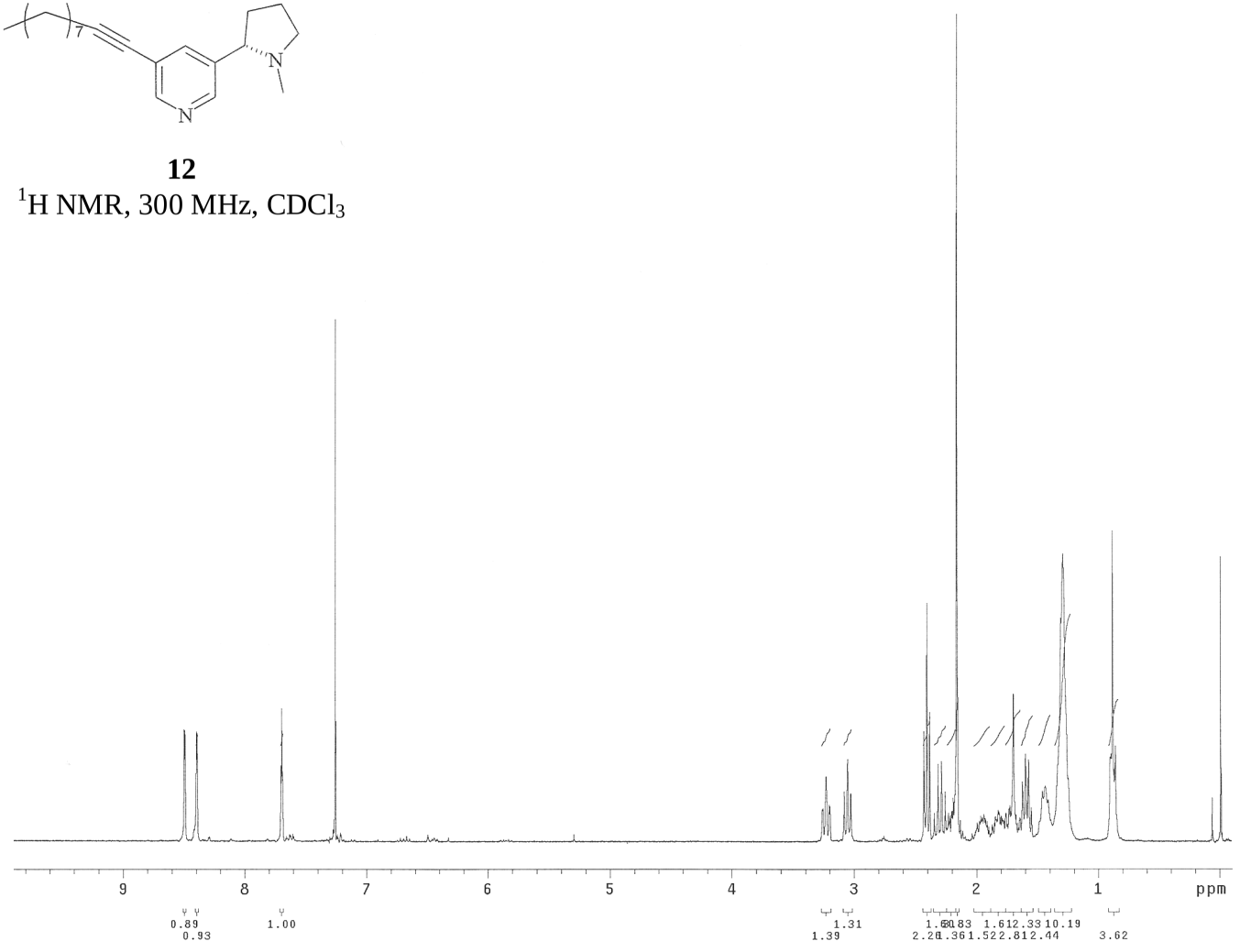


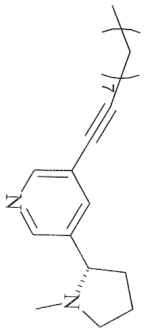
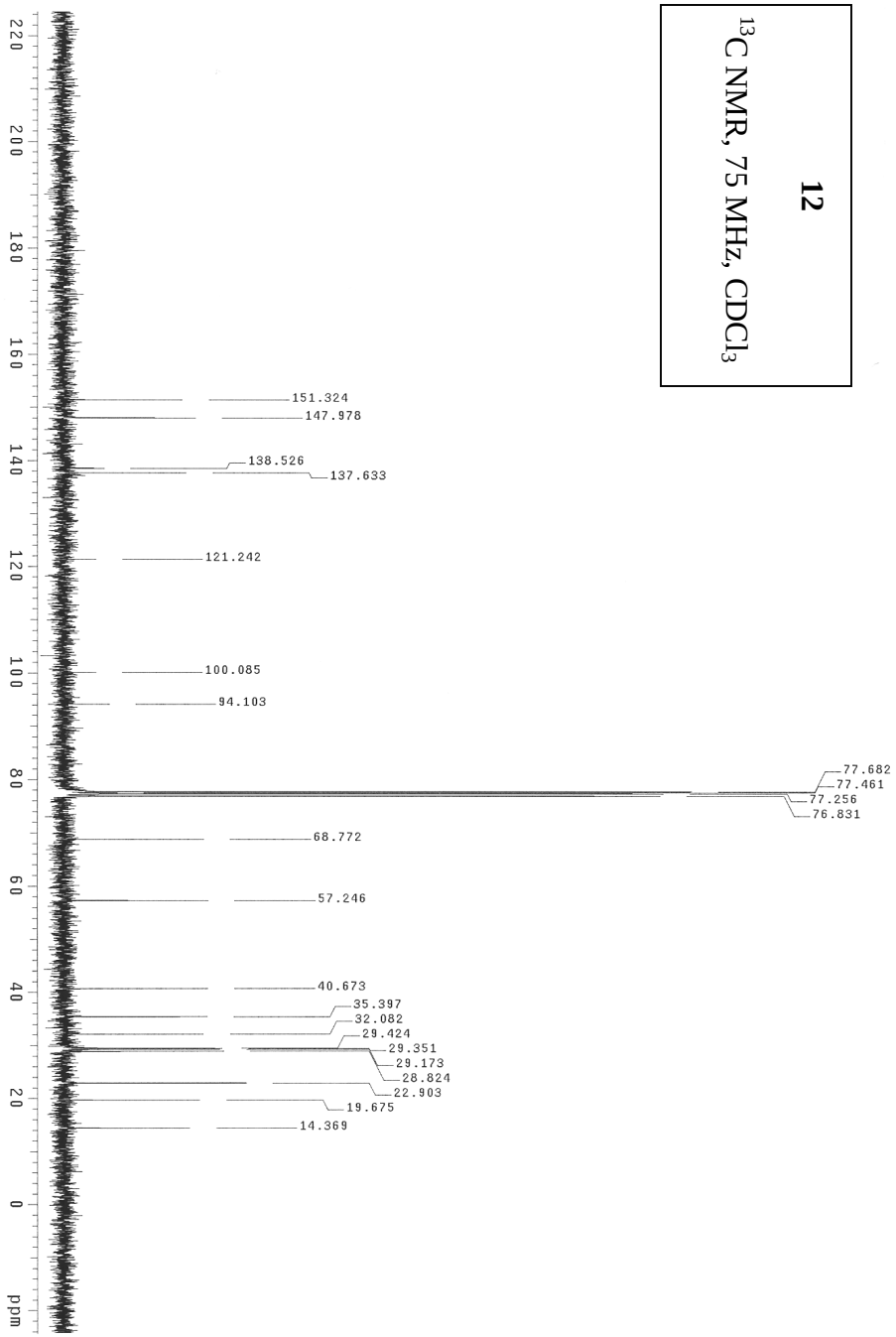




12

^{1}H NMR, 300 MHz, $CDCl_3$



12

^{13}C NMR, 75 MHz, $CDCl_3$