

Electronic Supplementary Information (ESI)

Fluoride-Catalyzed Nucleophilic Addition of PhSCF₂SiMe₃ to Anhydride: Synthesis of γ -Difluoromethylated γ -Lactams

Vannapha Pharikronburee, Teerachai Punirun, Darunee Soorukram, Chutima Kuhakarn, Patoomratana Tuchinda, Vichai Reutrakul and Manat Pohmakotr*

Department of Chemistry and Center of Excellence for Innovation in Chemistry, Faculty of Science, Mahidol University, Rama VI Road, Bangkok 10400, Thailand

Fax: +66 2 644 5126

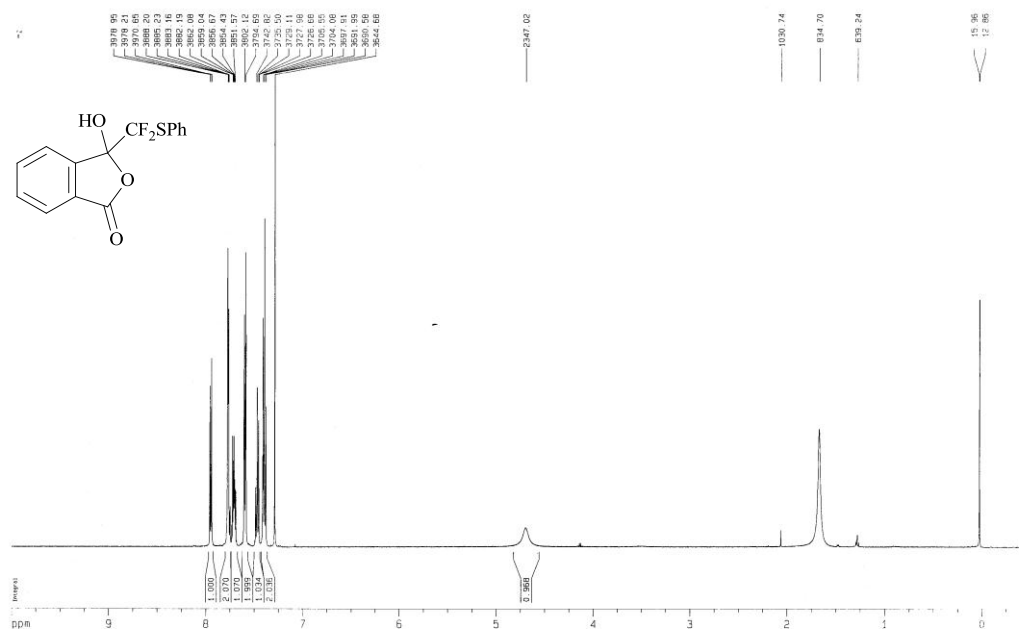
E-mail: manat.poh@mahidol.ac.th

Contents

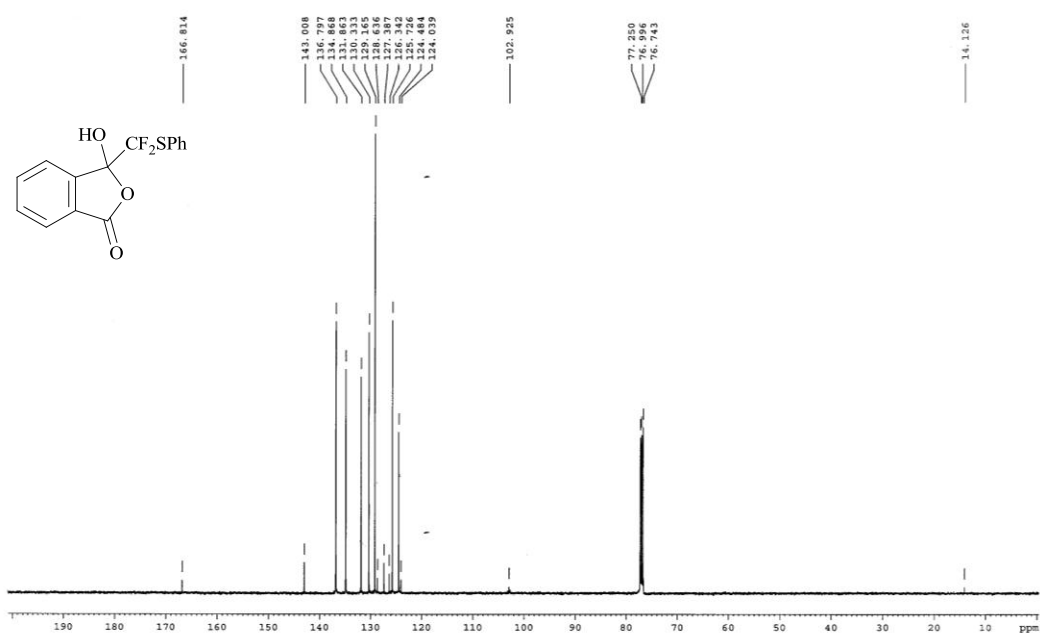
	page
¹ H, ¹³ C and ¹⁹ F NMR spectra of compounds 3 and 4	ESI-1
¹ H, ¹³ C and ¹⁹ F NMR spectra of compounds 5	ESI-12
¹ H, ¹³ C and ¹⁹ F NMR spectra of compounds 6	ESI-20
¹ H, ¹³ C and ¹⁹ F NMR spectra of compounds 7	ESI-29
¹ H, ¹³ C and ¹⁹ F NMR spectra of compounds 8	ESI-36
Observed correlations in HMQC, COSY (¹ H- ¹ H), and HMBC spectra of 3fA	ESI-44
Observed correlations in HMQC, COSY (¹ H- ¹ H), and HMBC spectra of 3fB	ESI-47

^1H , ^{13}C and ^{19}F NMR spectra of compounds 3 and 4

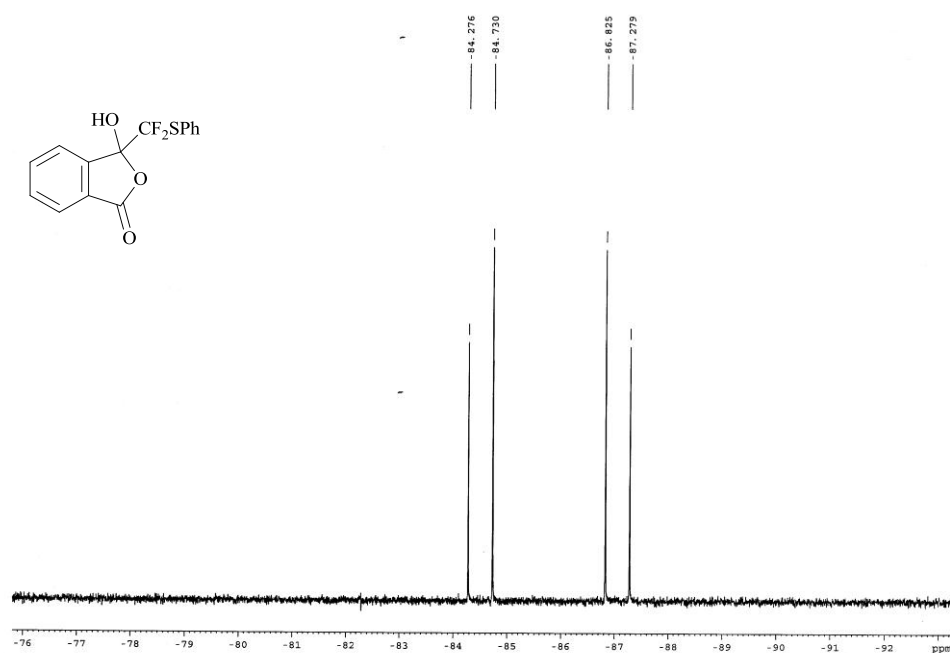
^1H NMR spectrum of 3a (500 MHz, CDCl_3)



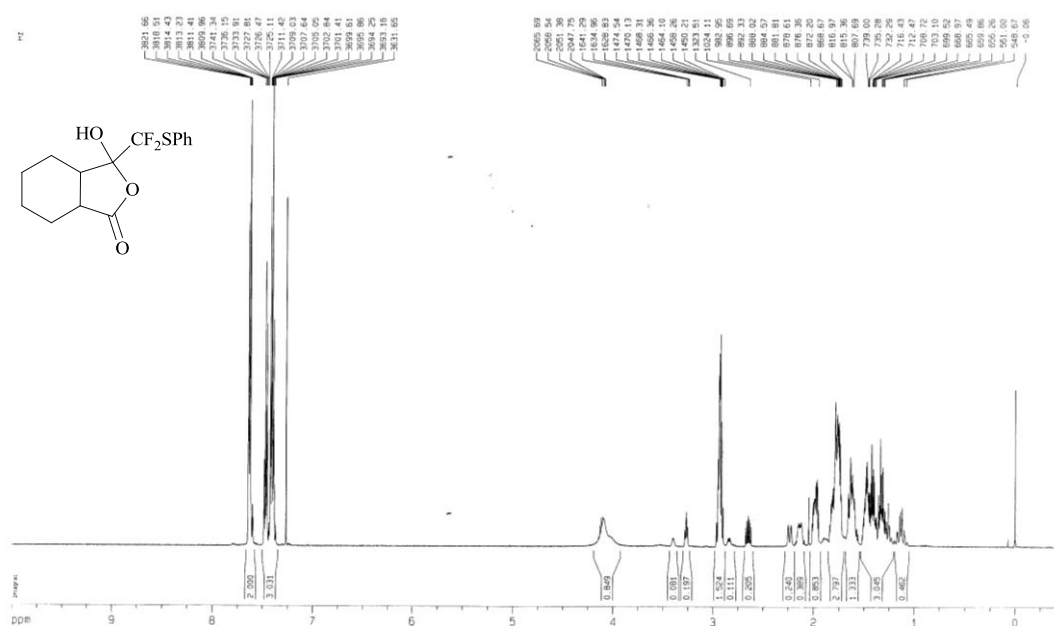
^{13}C NMR spectrum of 3a (125 MHz, CDCl_3)



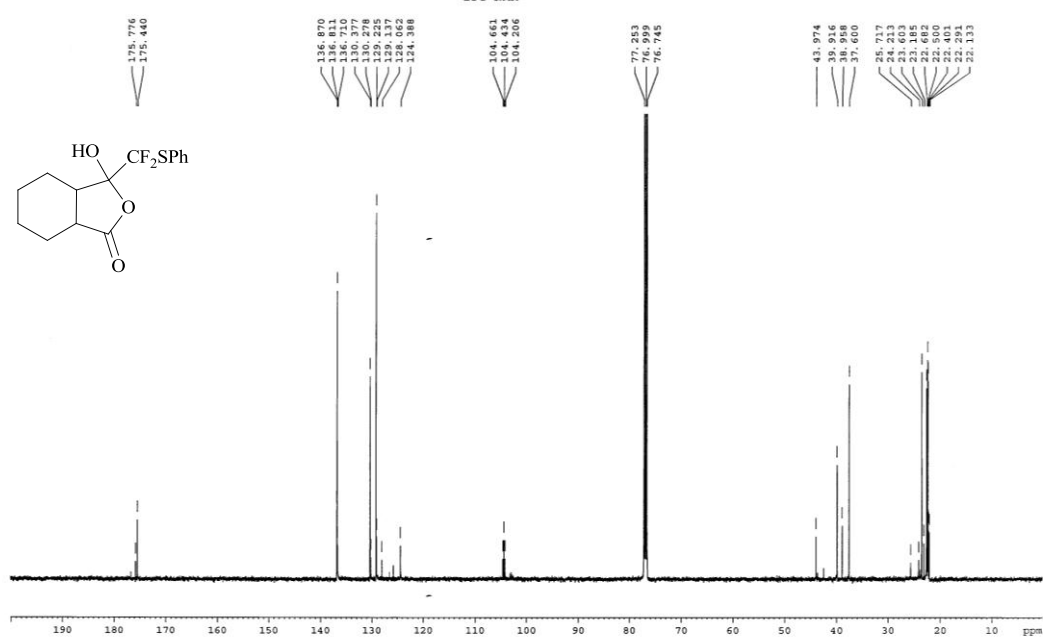
^{19}F NMR spectrum of **3a** (470 MHz, CDCl_3)



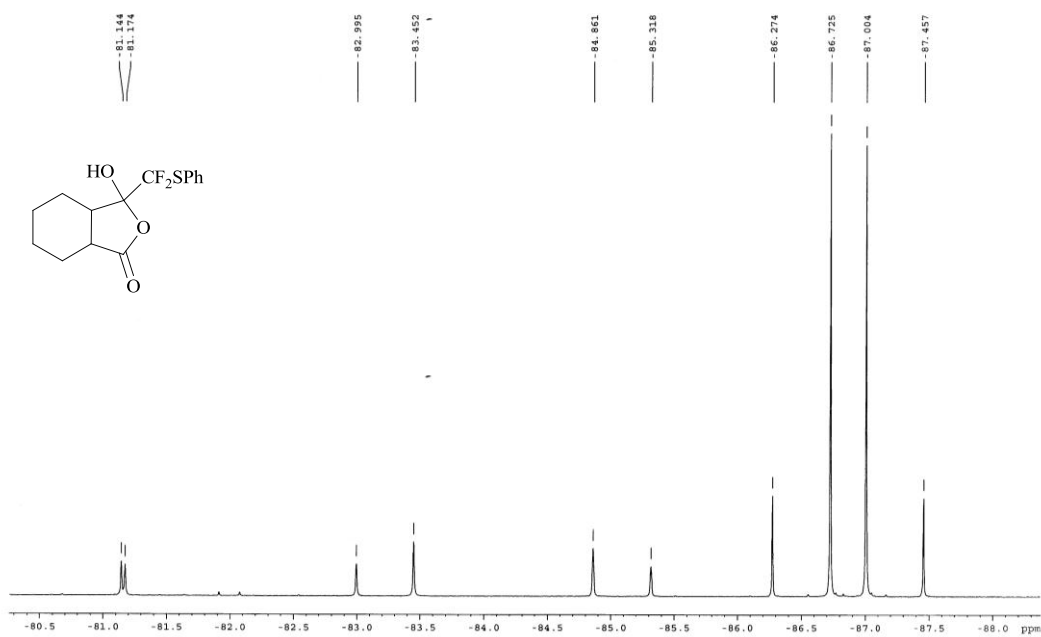
^1H NMR spectrum of **3b** (500 MHz, CDCl_3)



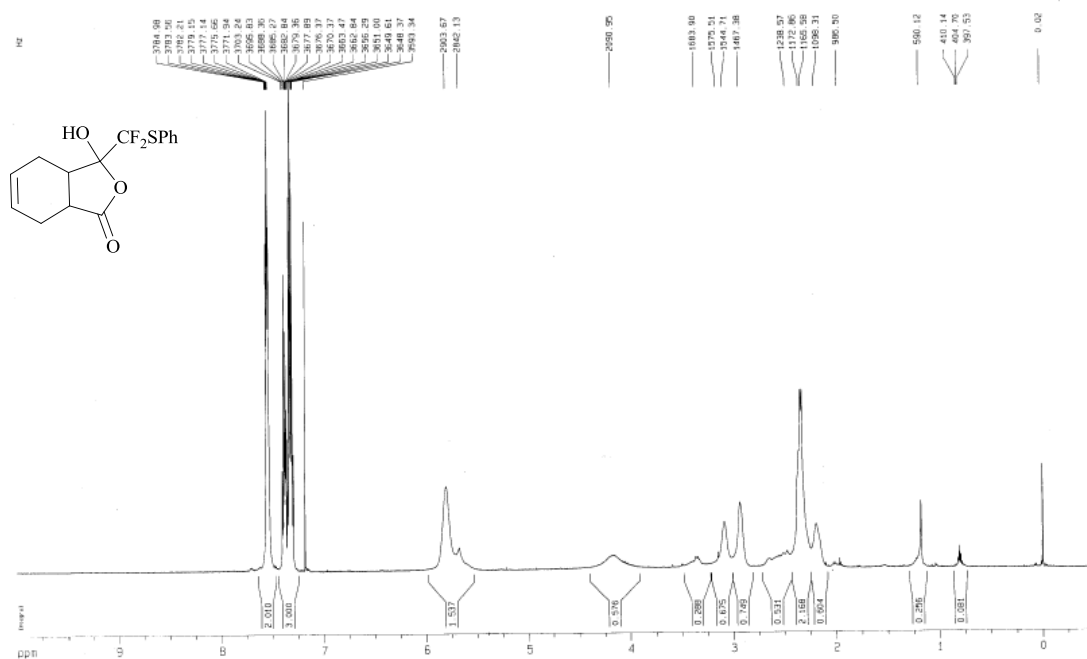
^{13}C NMR spectrum of **3b** (125 MHz, CDCl_3)



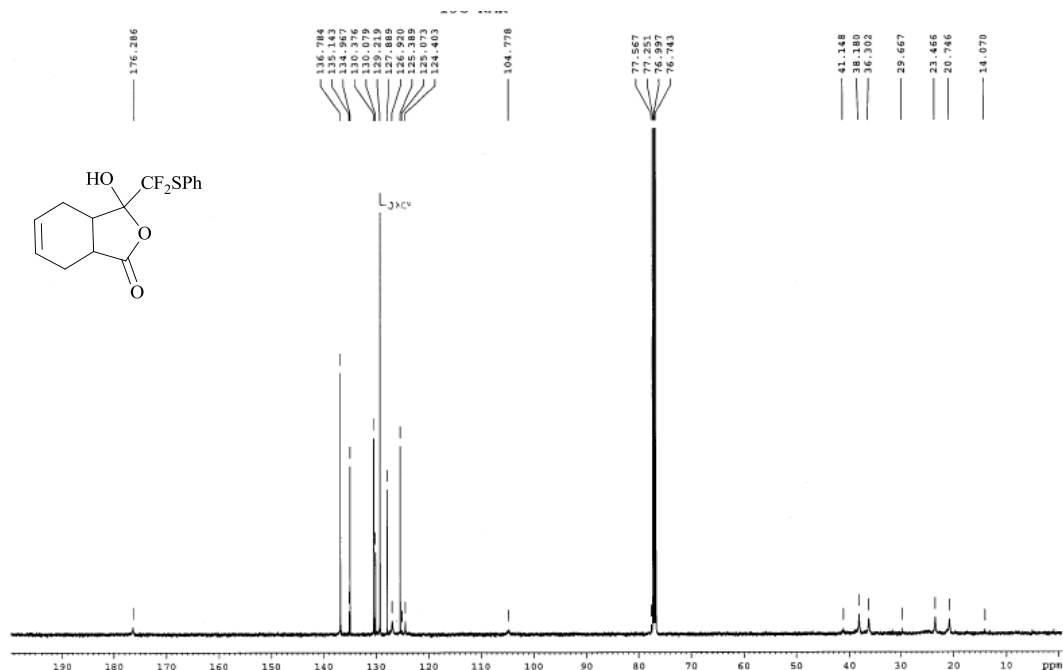
^{19}F NMR spectrum of **3b** (470 MHz, CDCl_3)



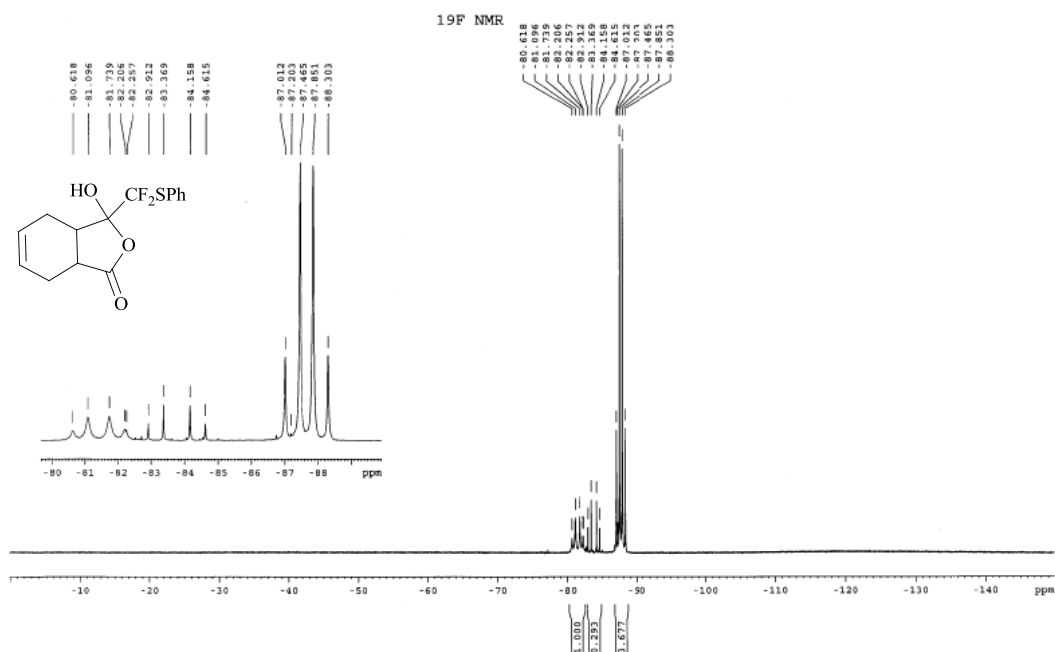
^1H NMR spectrum of **3c** (500 MHz, CDCl_3)



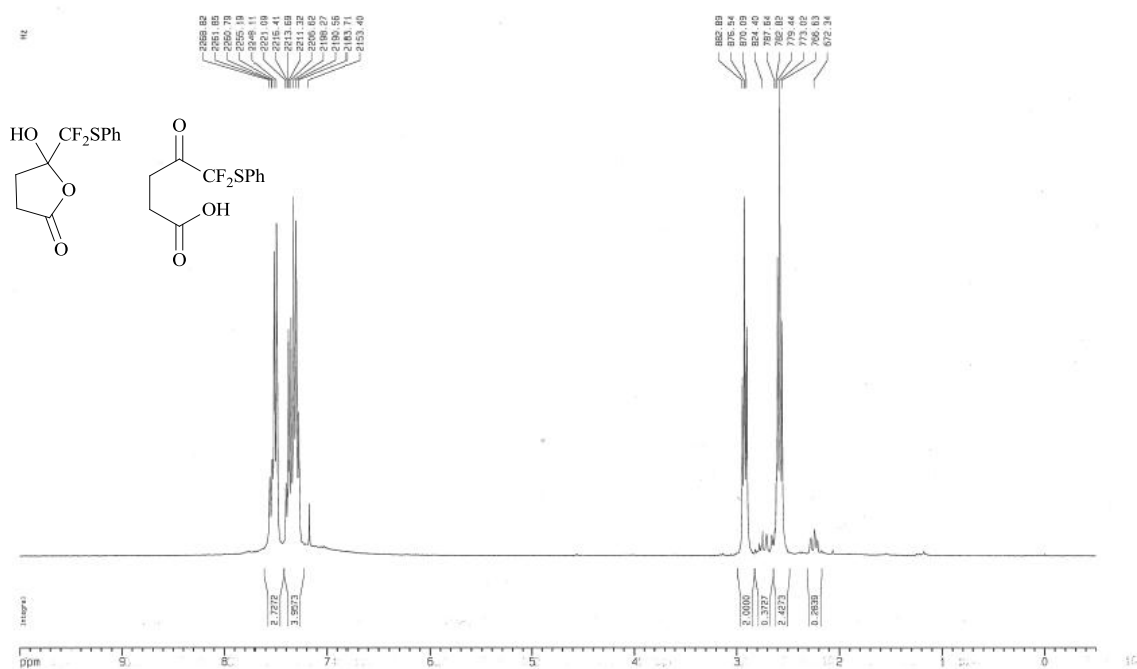
^{13}C NMR spectrum of **3c** (125 MHz, CDCl_3)



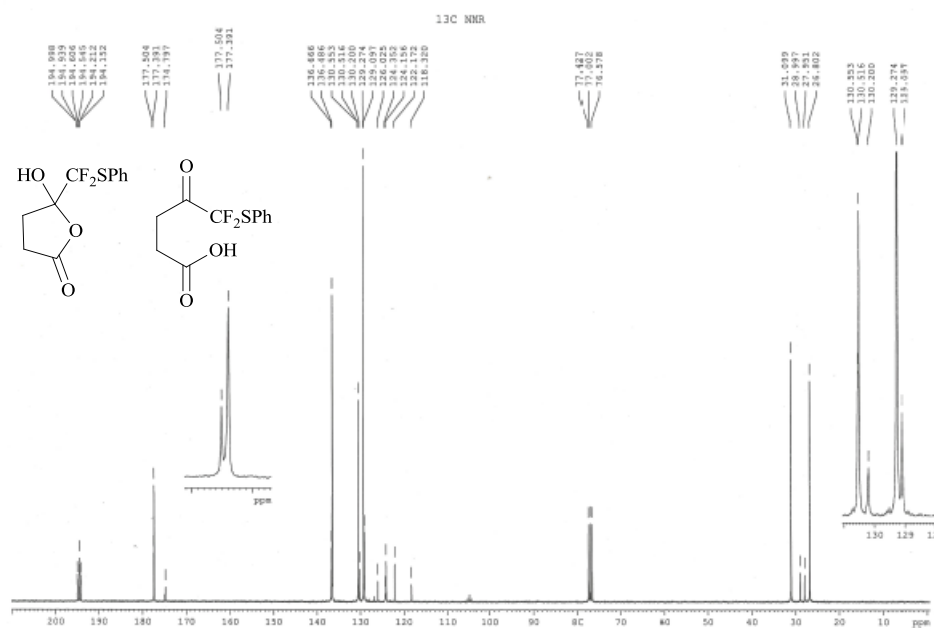
^{19}F NMR spectrum of **3c** (470 MHz, CDCl_3)



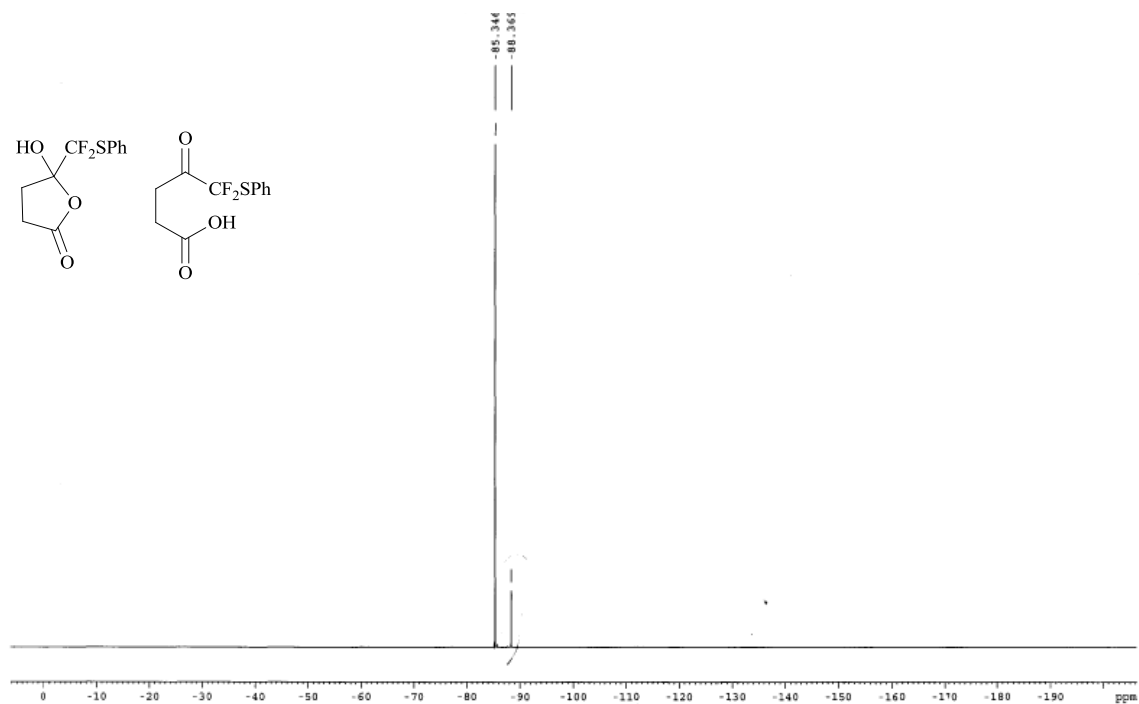
^1H NMR spectrum of **3d** and **4d** (300 MHz, CDCl_3)

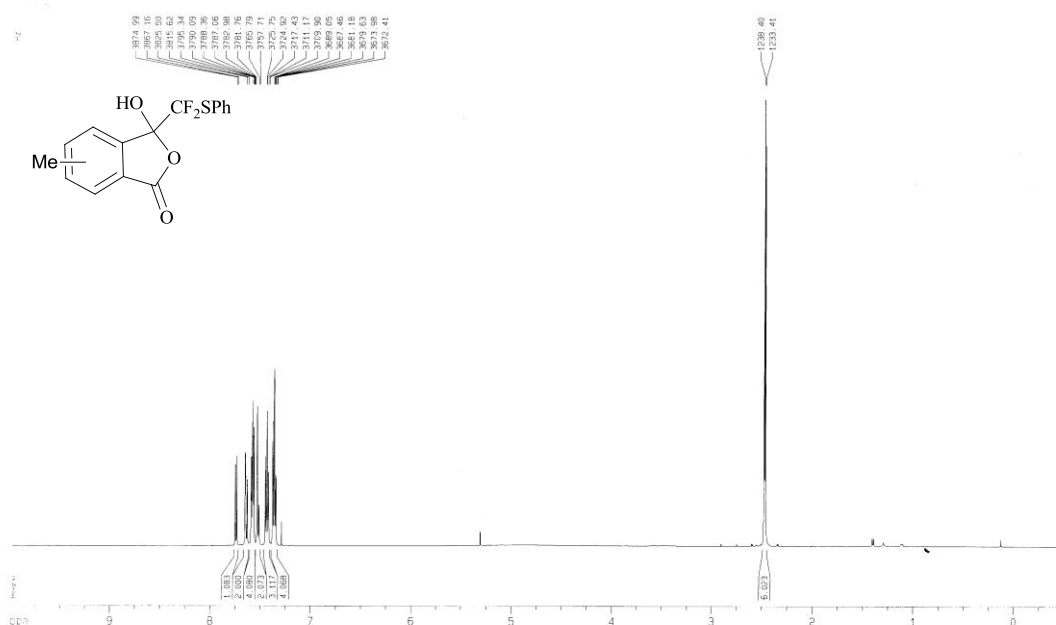
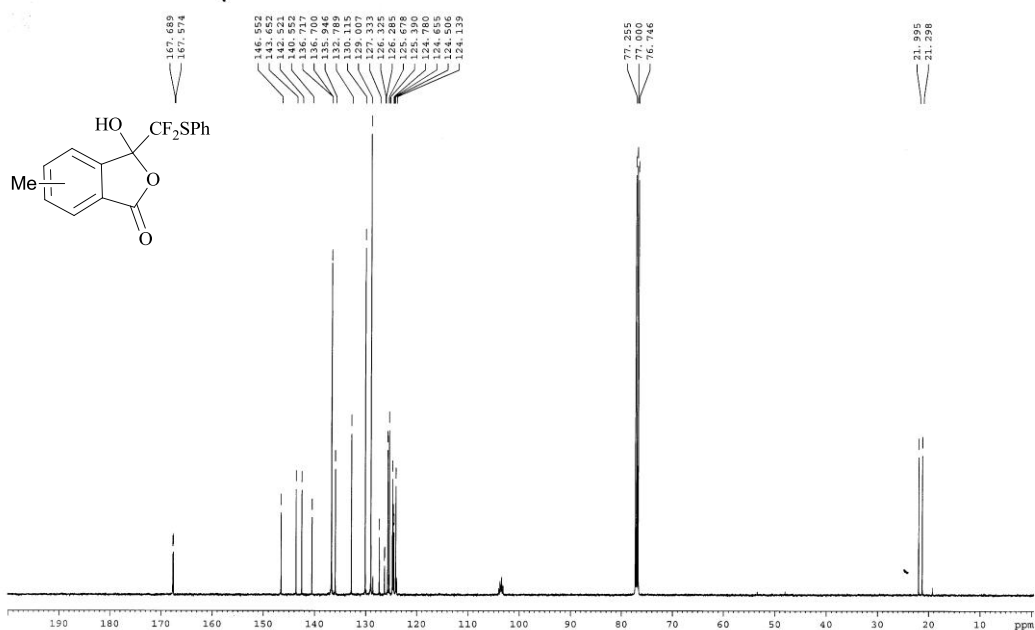


^{13}C NMR spectrum of **3d** and **4d** (125 MHz, CDCl_3)

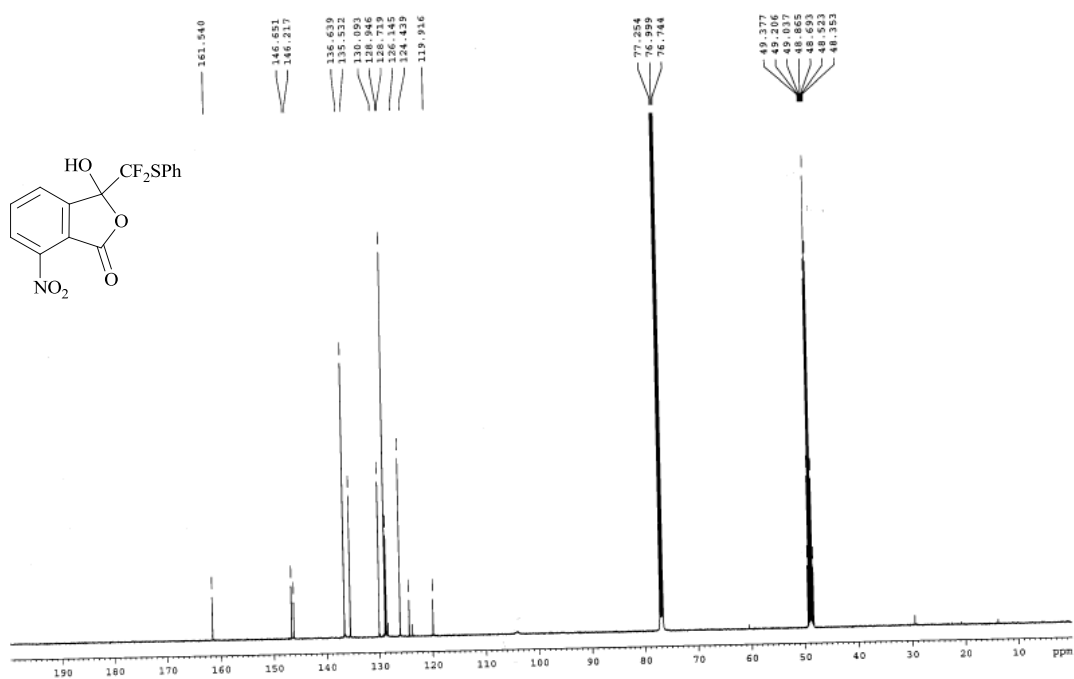


^{19}F NMR spectrum of **3d** and **4d** (470 MHz, CDCl_3)

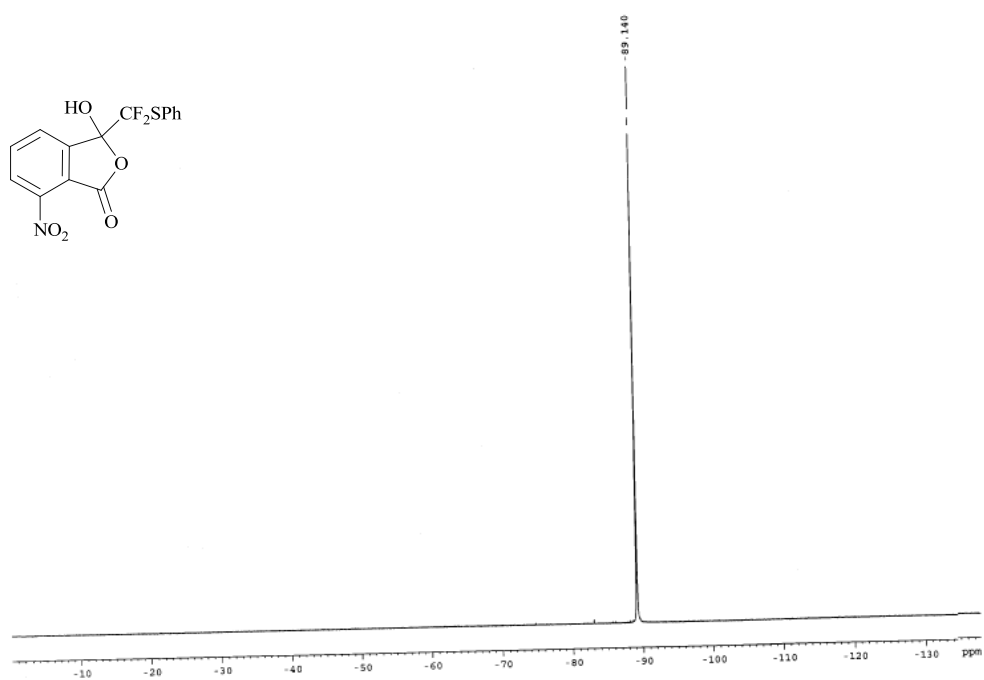


¹H NMR spectrum of **3e** (500 MHz, CDCl₃) ^{13}C NMR spectrum of **3e** (125 MHz, CDCl_3)

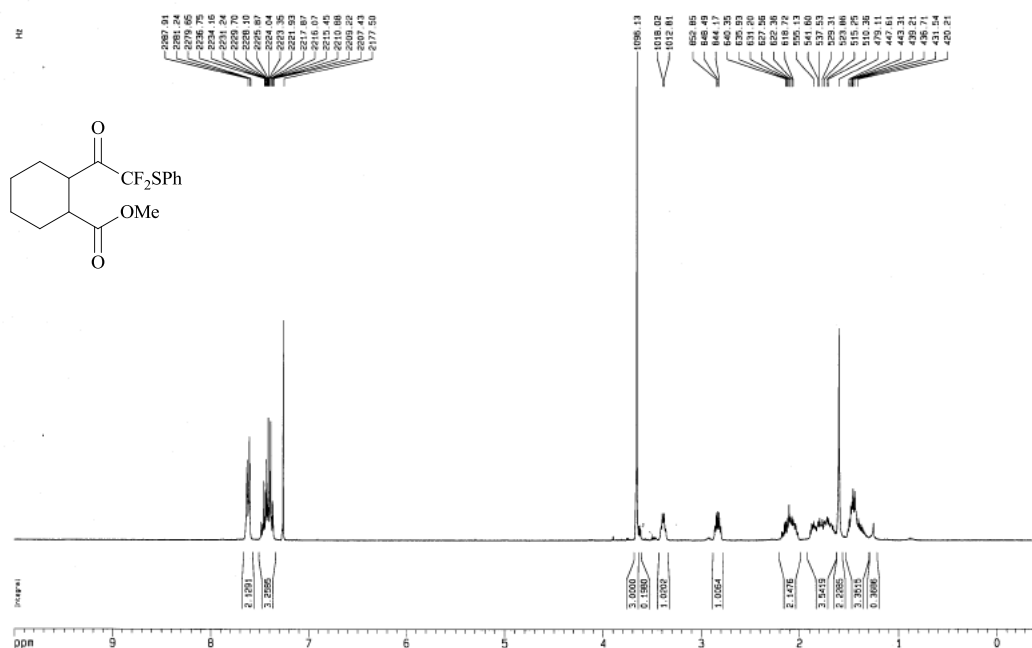
^{13}C NMR spectrum of **3fA** (125 MHz, CDCl_3)



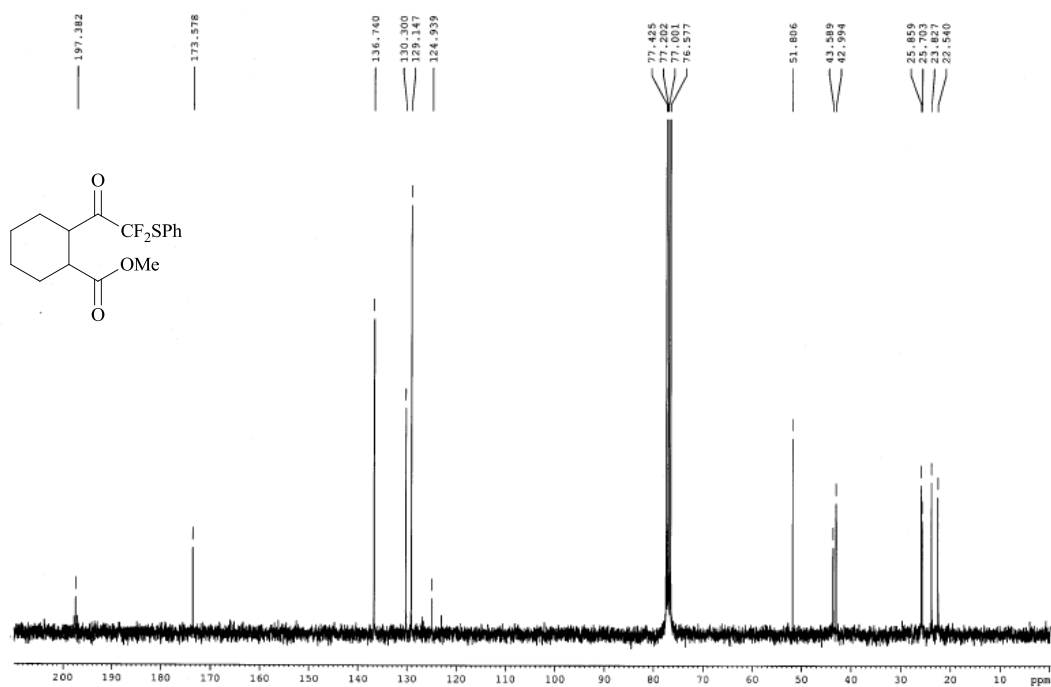
^{19}F NMR spectrum of **3fA** (470 MHz, CDCl_3)



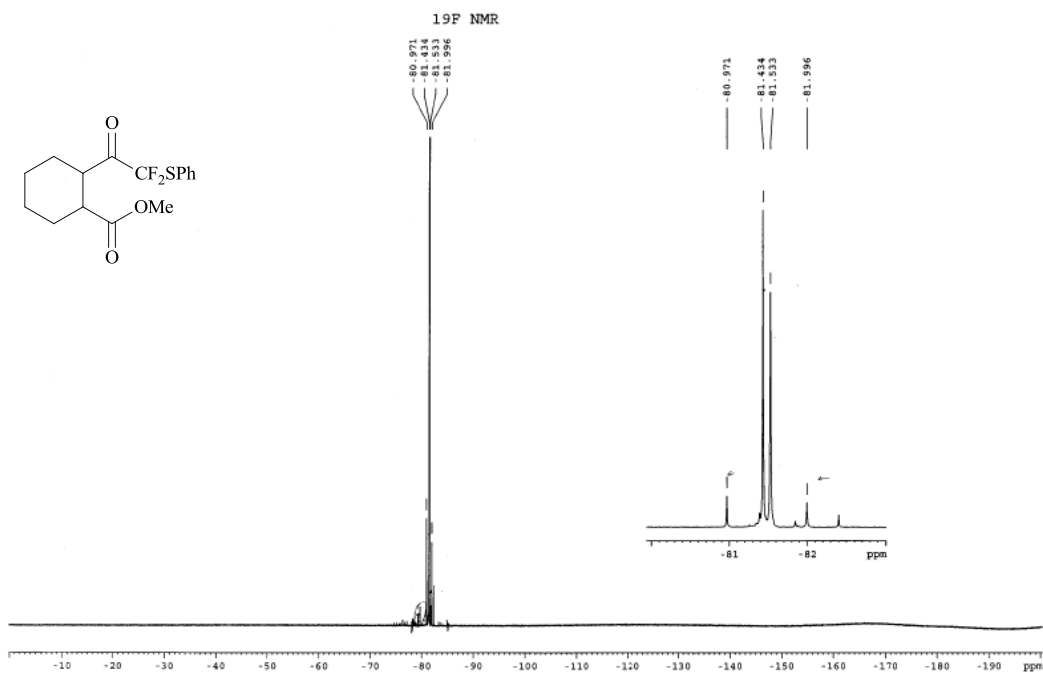
^1H NMR spectrum of **5Bb** (300 MHz, CDCl_3)



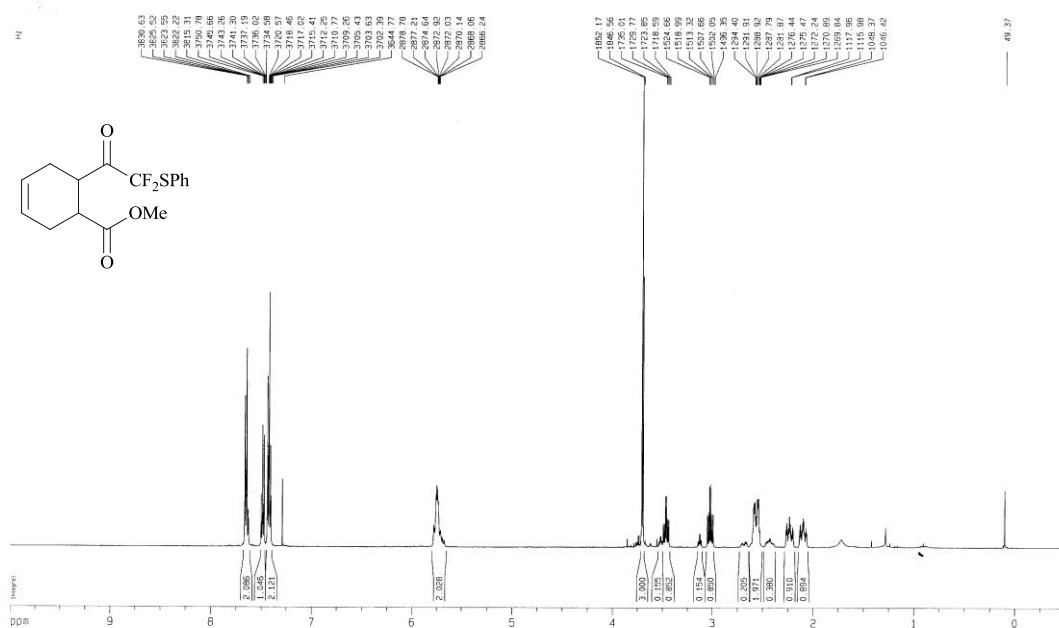
^{13}C NMR spectrum of **5Bb** (75 MHz, CDCl_3)



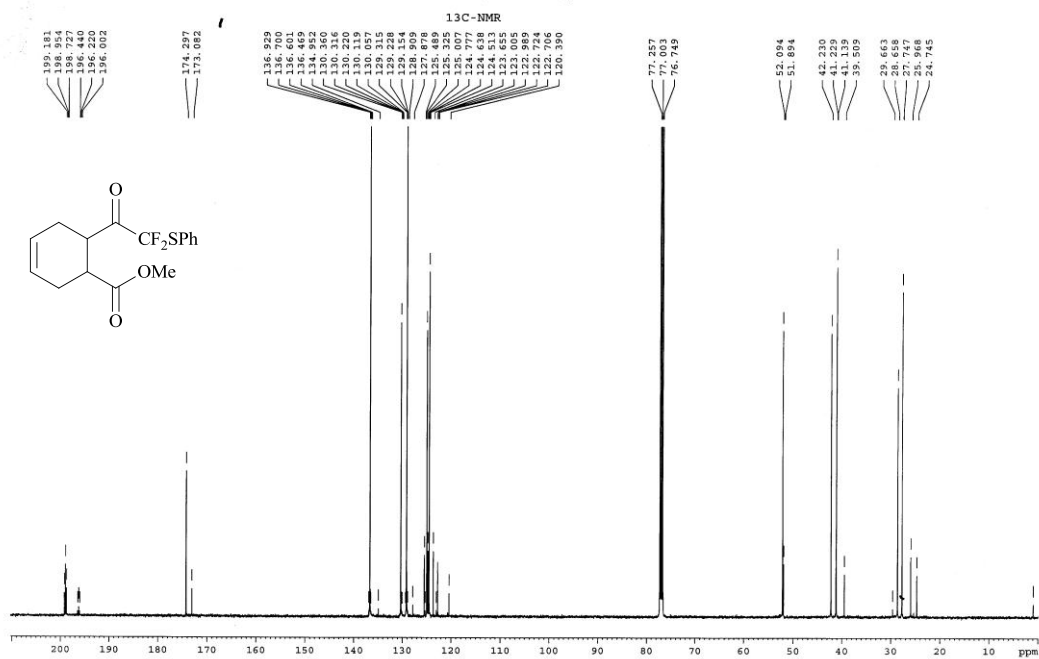
¹⁹F NMR spectrum of **5Bb** (470 MHz, CDCl₃)



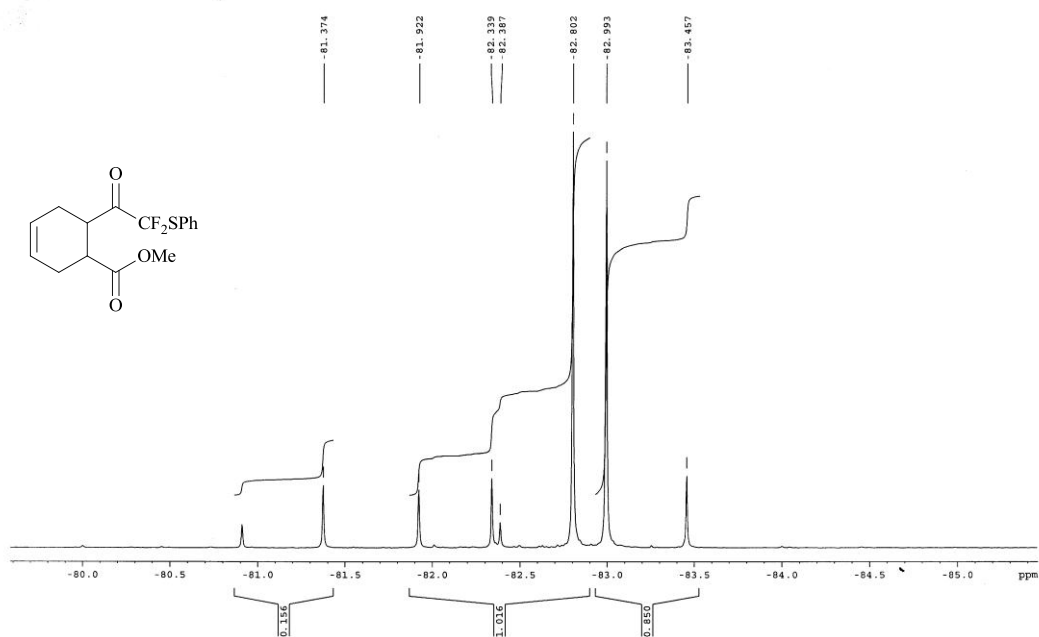
¹H NMR spectrum of **5Bc** (500 MHz, CDCl₃)



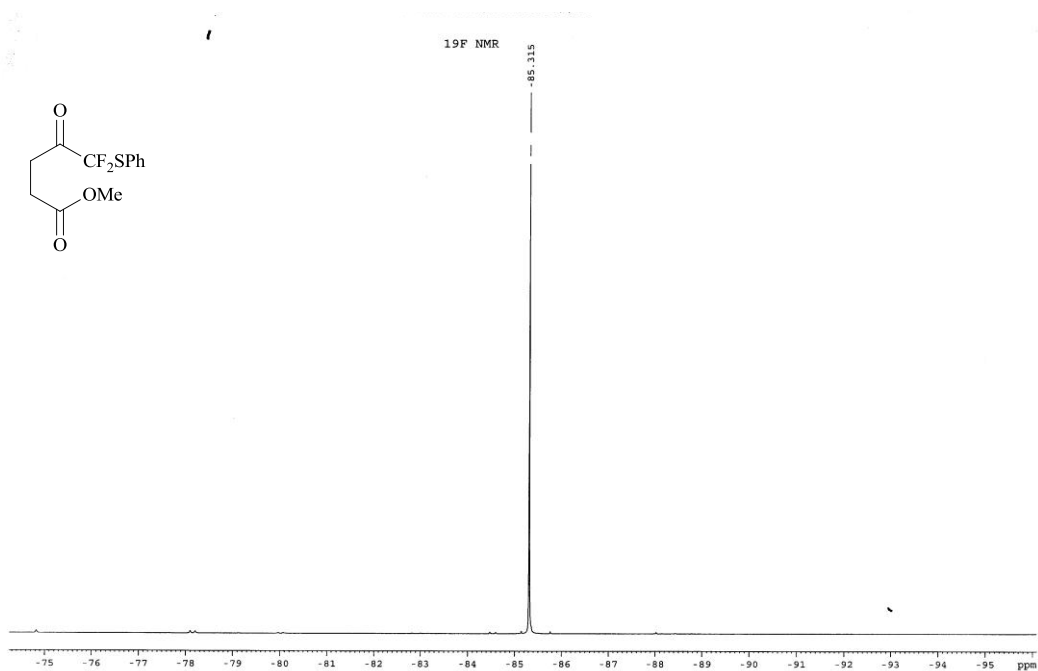
^{13}C NMR spectrum of **5Bc** (125 MHz, CDCl_3)



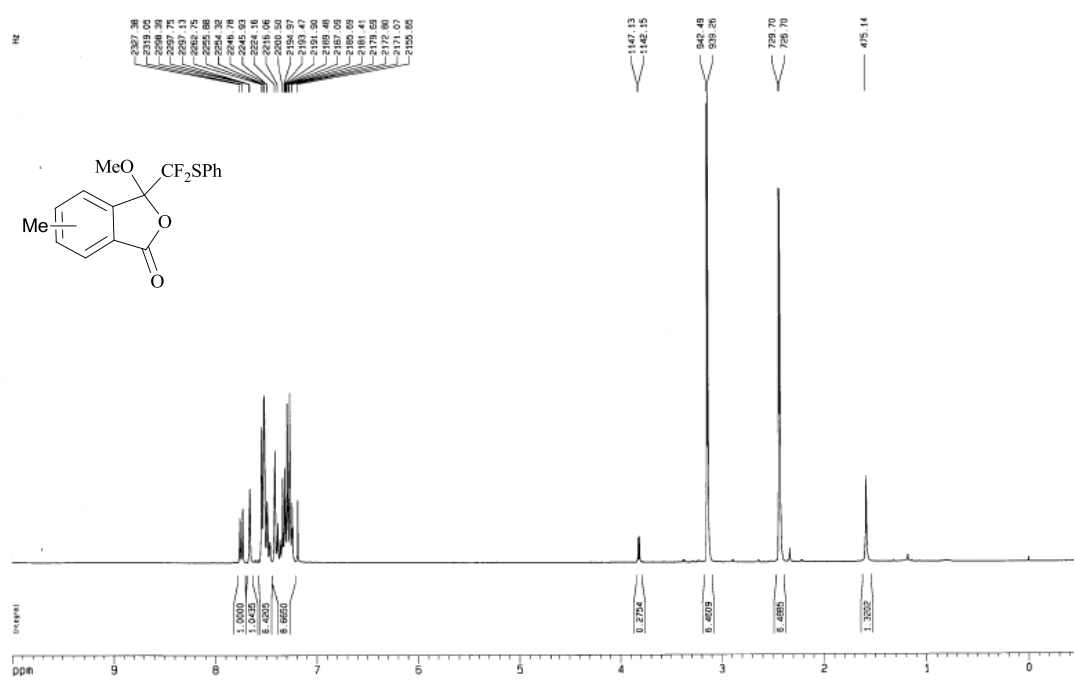
^{19}F NMR spectrum of **5Bc** (470 MHz, CDCl_3)



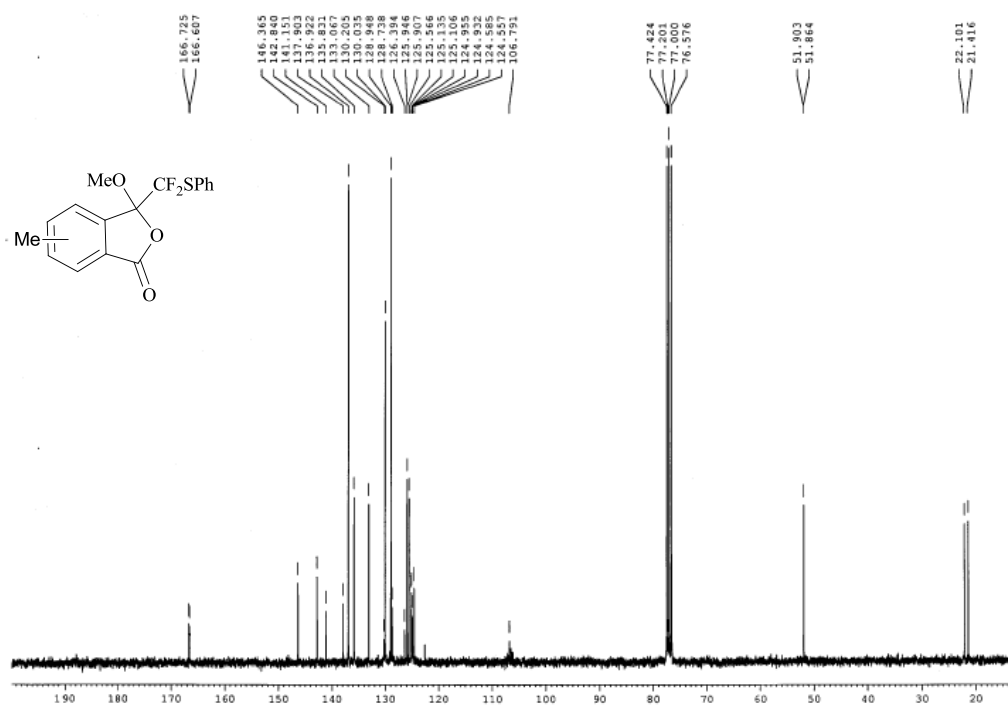
^{19}F NMR spectrum of **5Bd** (470 MHz, CDCl_3)



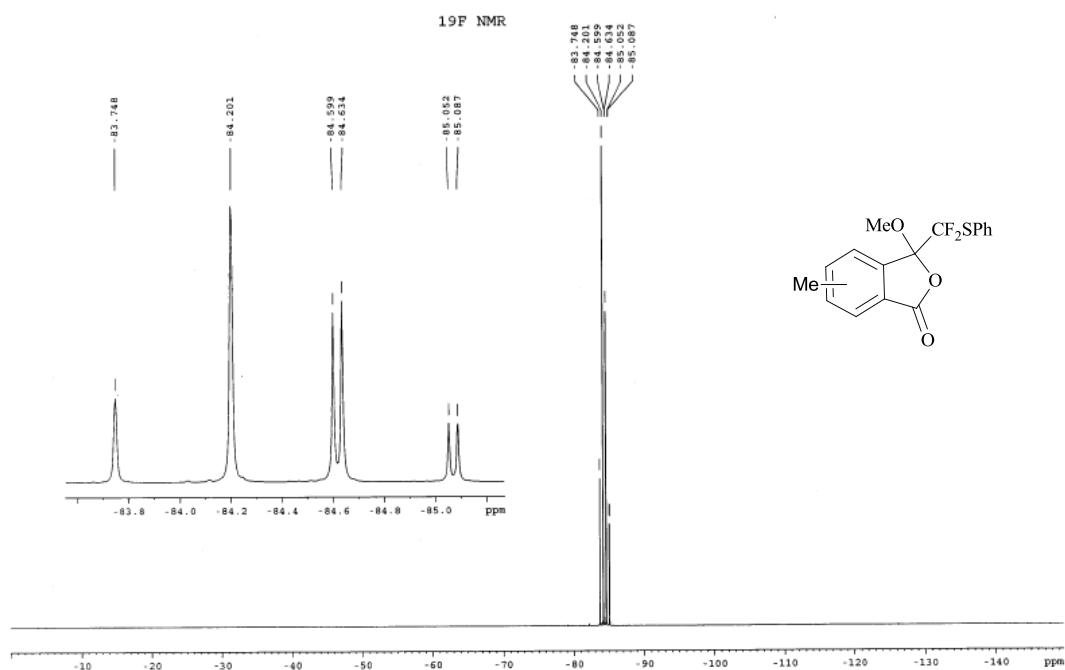
^1H NMR spectrum of **5Ae** (300 MHz, CDCl_3)



¹³C NMR spectrum of **5Ae** (75 MHz, CDCl₃)

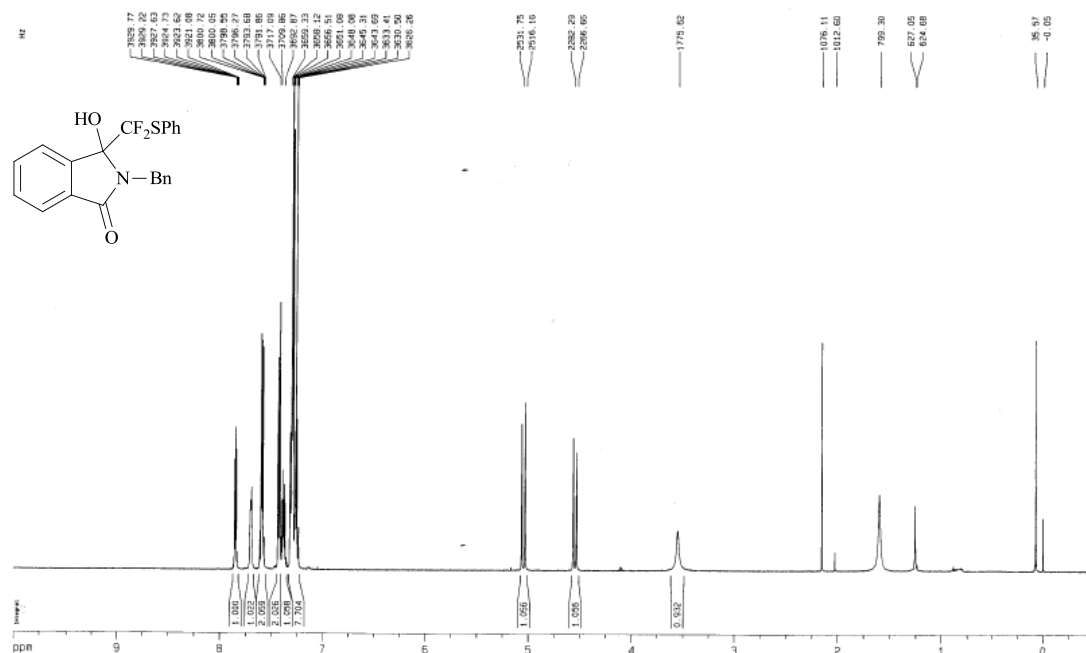


¹⁹F NMR spectrum of **5Ae** (470 MHz, CDCl₃)

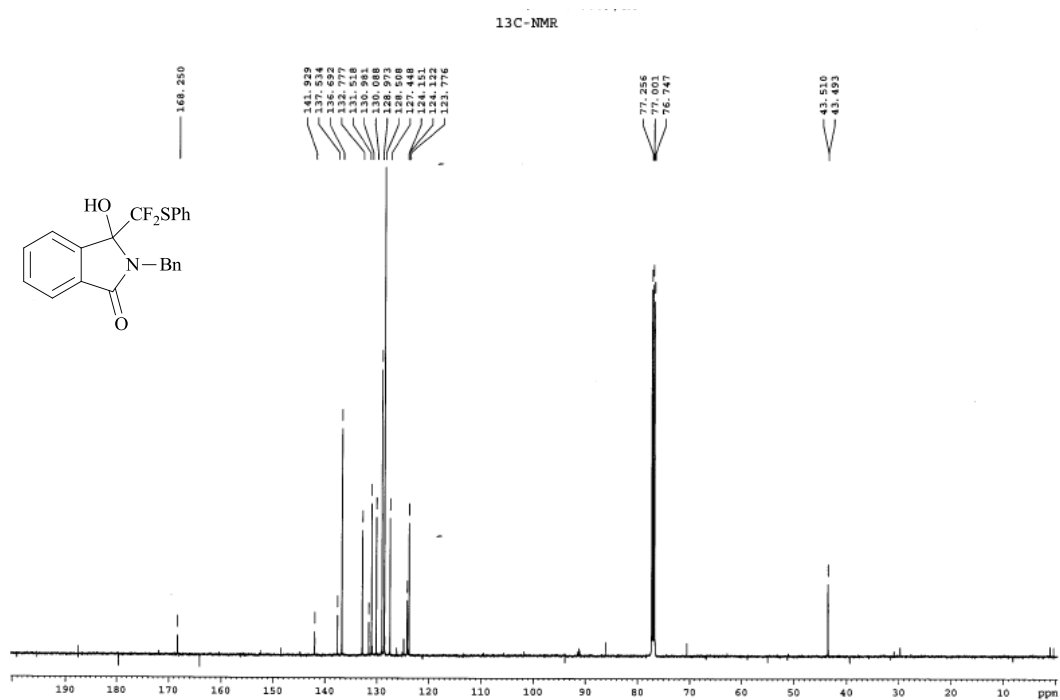


¹H, ¹³C and ¹⁹F NMR spectra of compounds 6

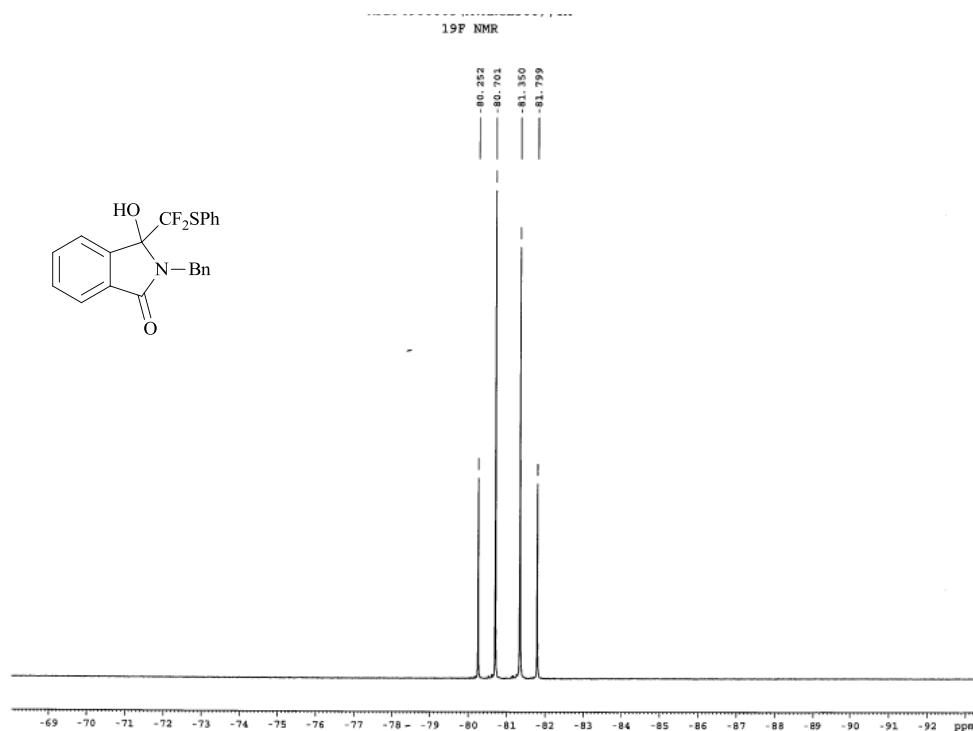
¹H NMR spectrum of **6a** (500 MHz, CDCl₃)



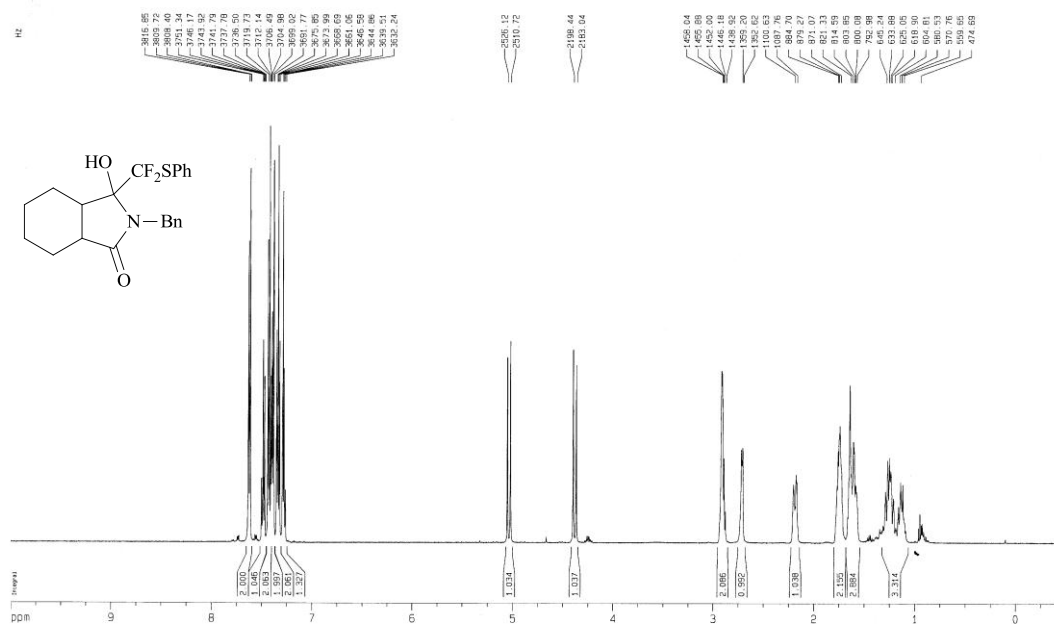
¹³C NMR spectrum of **6a** (125 MHz, CDCl₃)



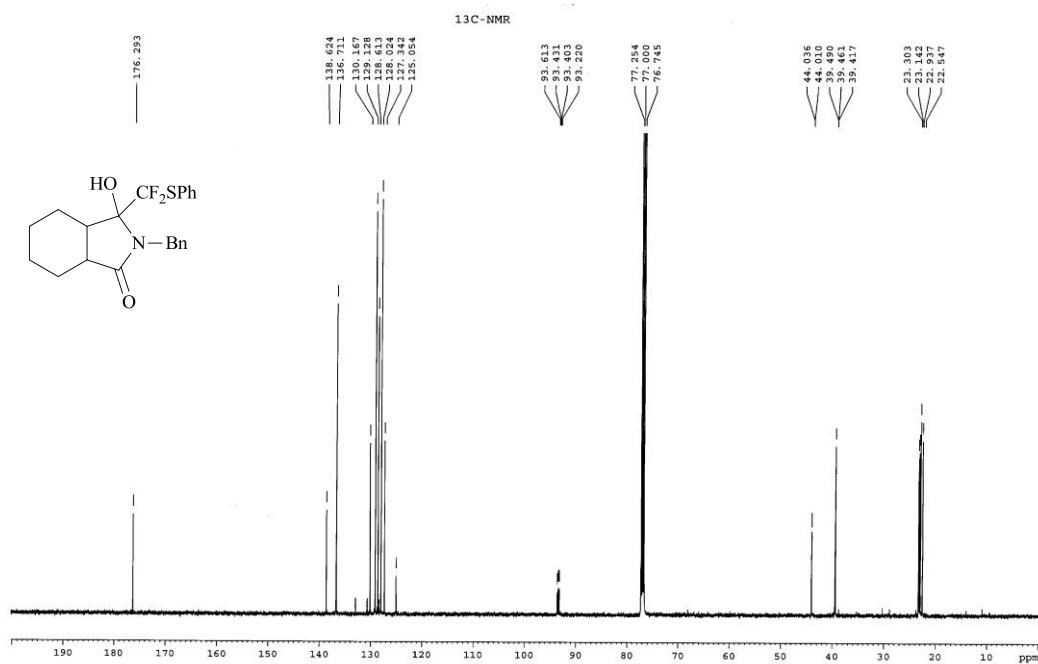
^{19}F NMR spectrum of **6a** (470 MHz, CDCl_3)



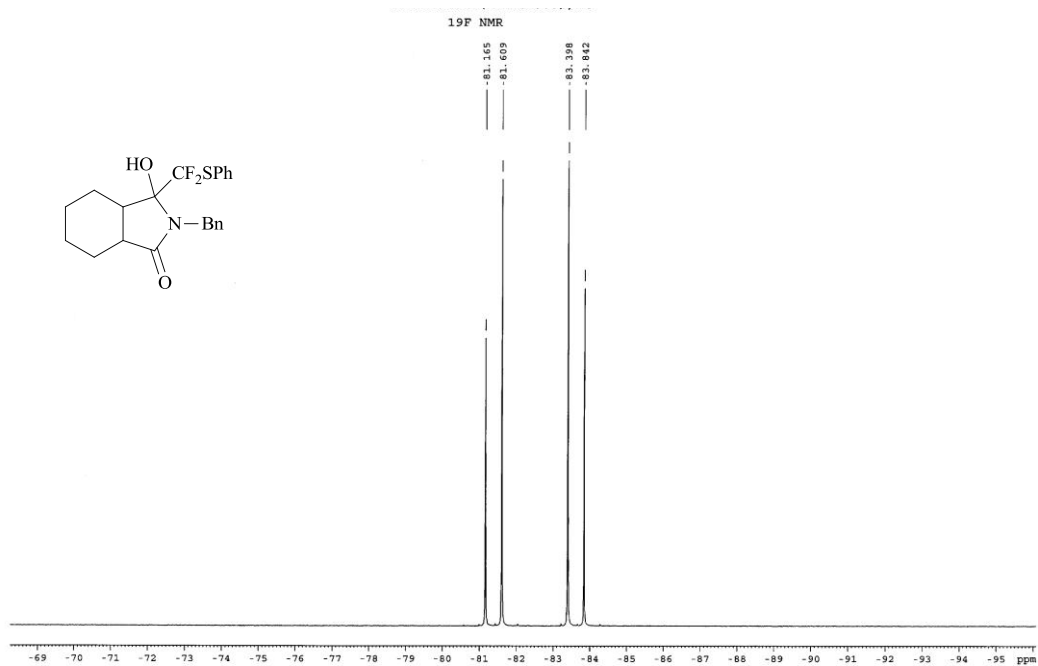
^1H NMR spectrum of **6b** (500 MHz, CDCl_3)



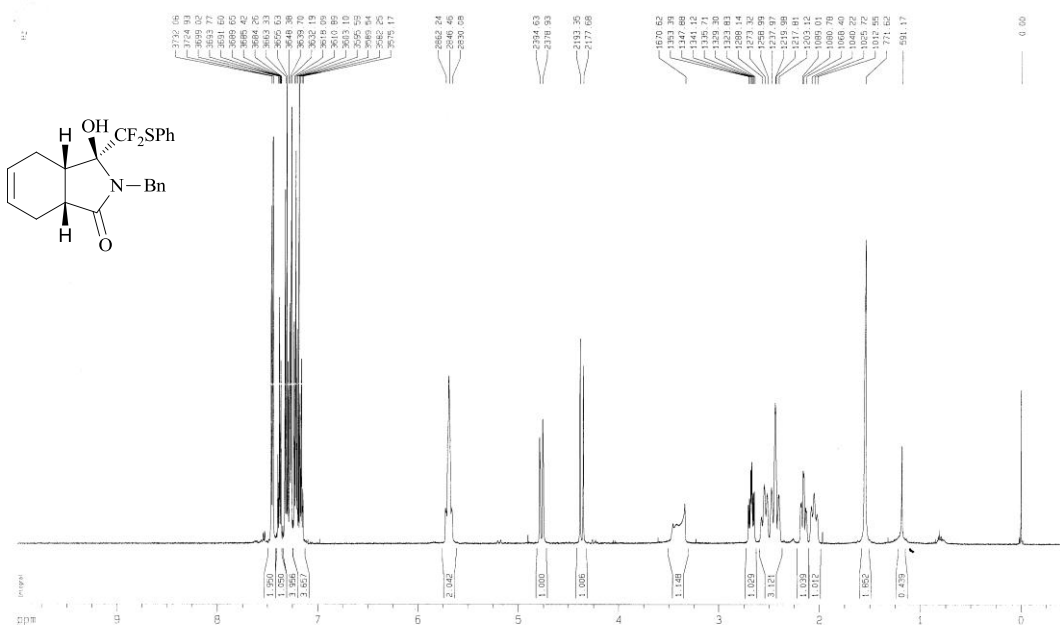
^{13}C NMR spectrum of **6b** (125 MHz, CDCl_3)



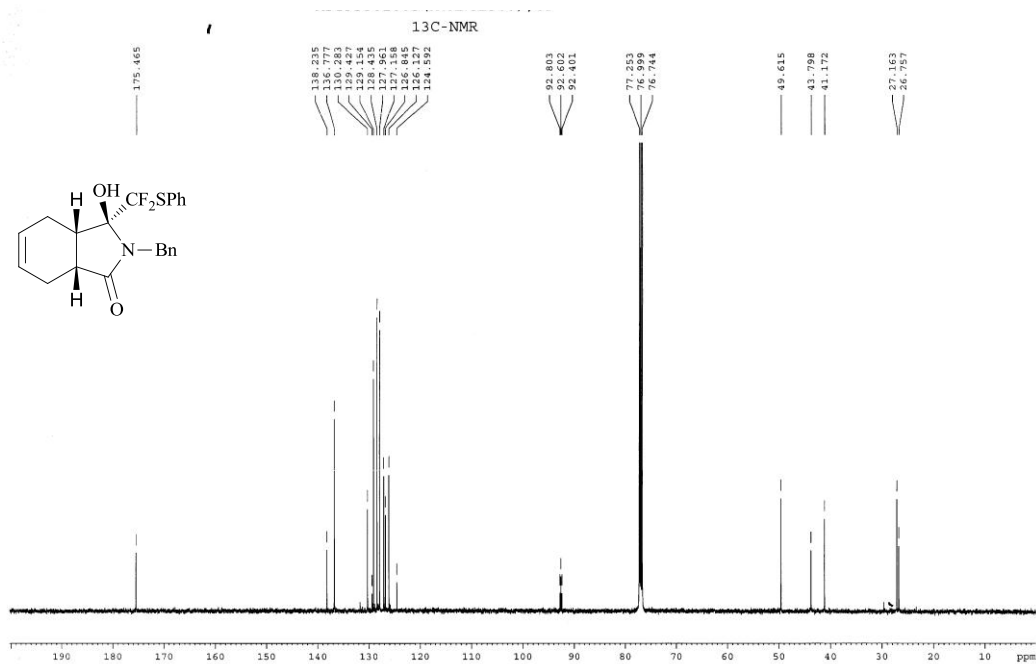
^{19}F NMR spectrum of **6b** (470 MHz, CDCl_3)



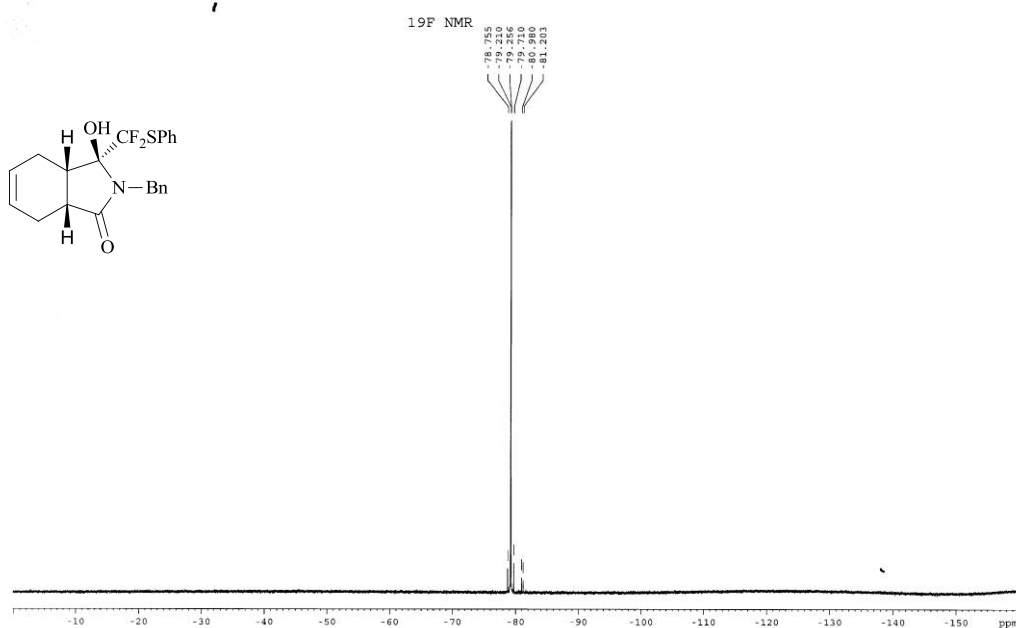
¹H NMR spectrum of **6cA** (500 MHz, CDCl₃)



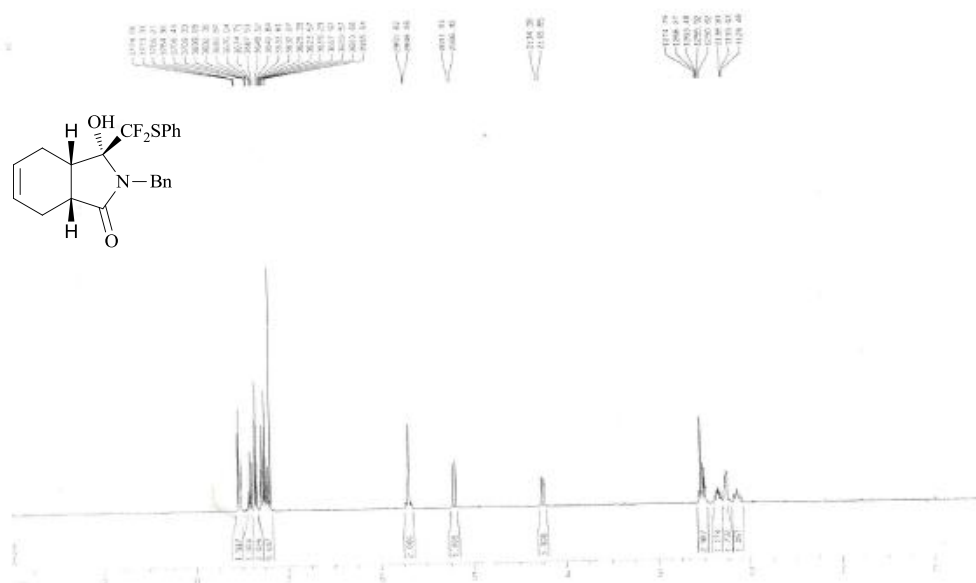
¹³C NMR spectrum of **6cA** (125 MHz, CDCl₃)



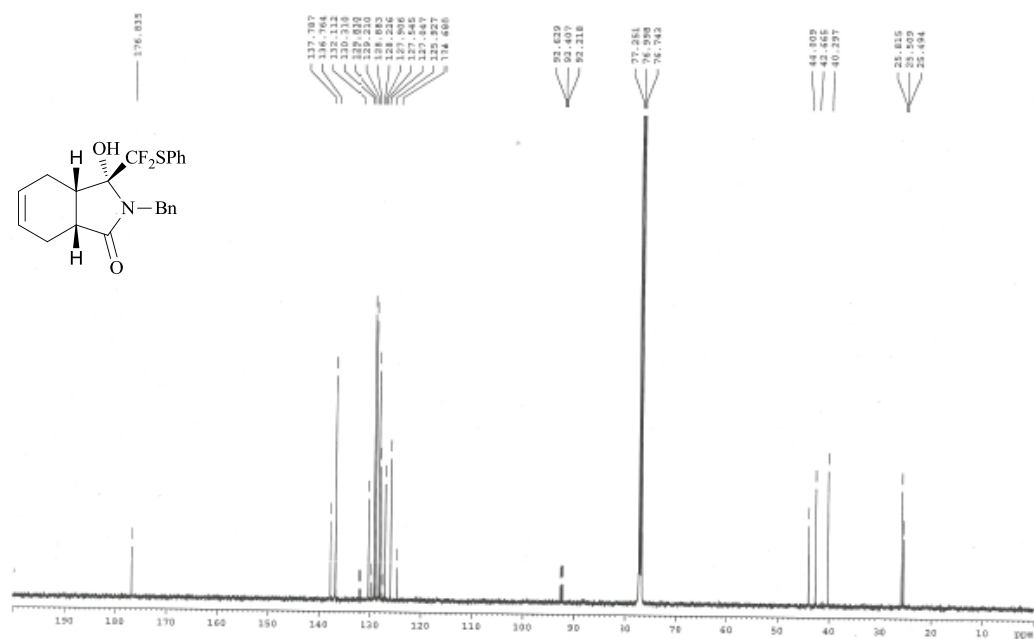
^{19}F NMR spectrum of **6cA** (470 MHz, CDCl_3)



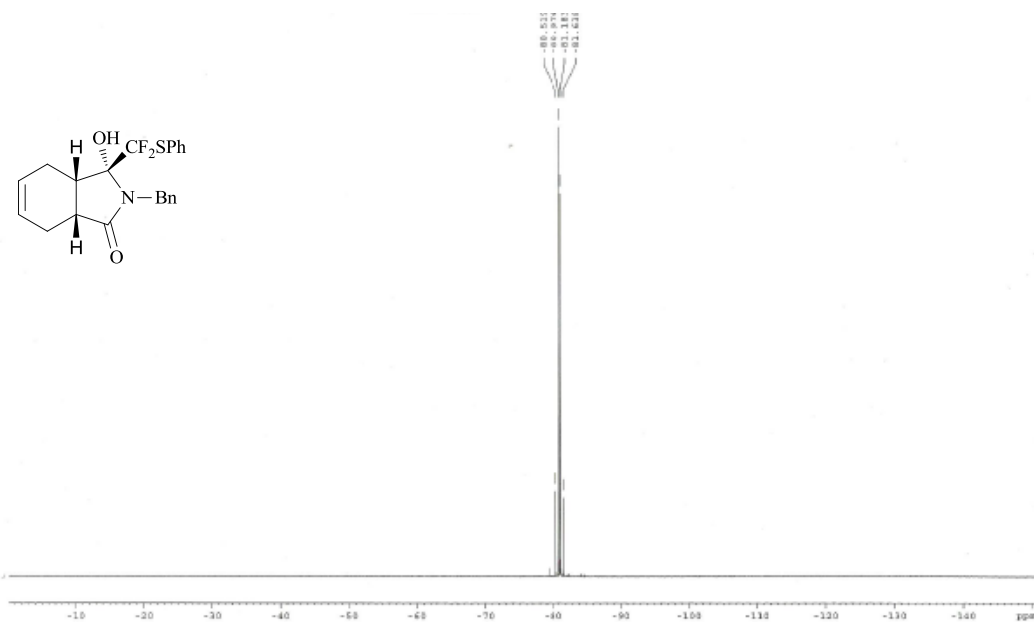
^1H NMR spectrum of **6cB** (500 MHz, CDCl_3)



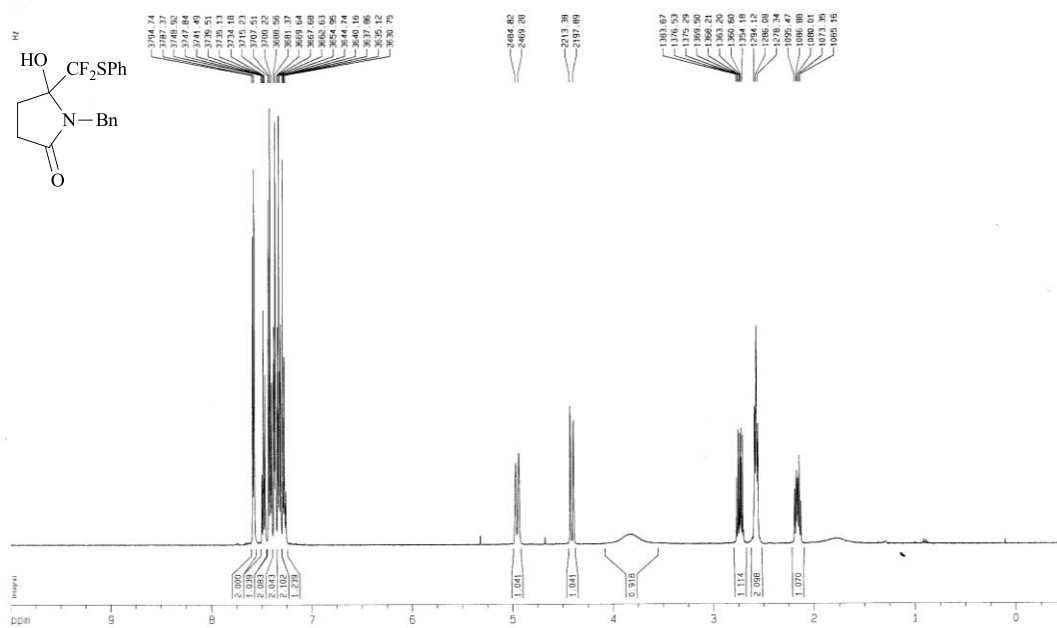
^{13}C NMR spectrum of **6cB** (125 MHz, CDCl_3)



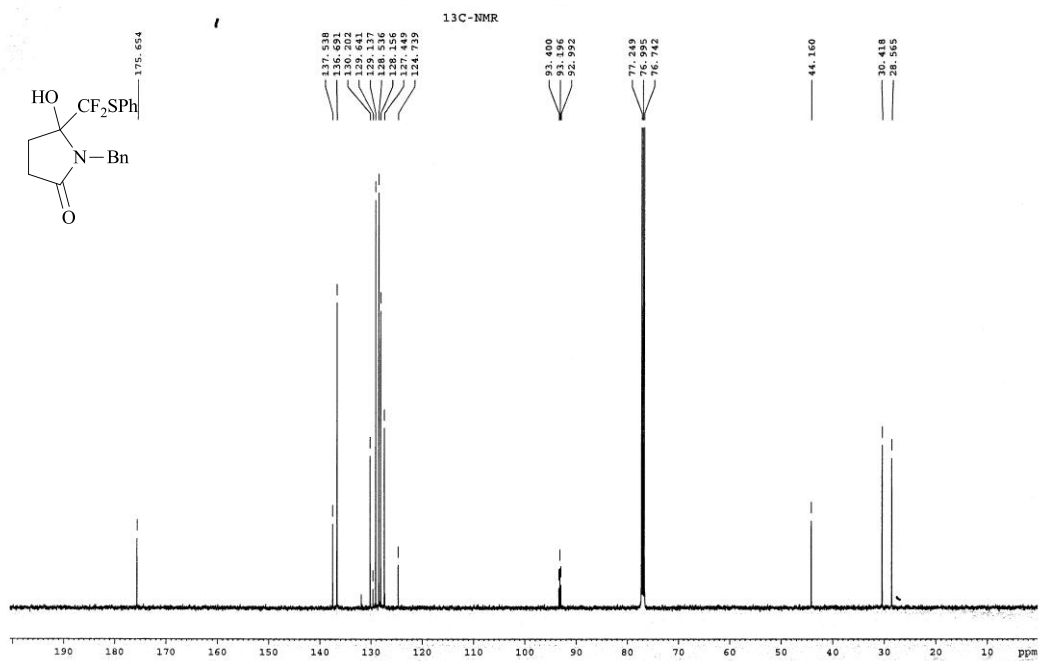
^{19}F NMR spectrum of **6cB** (470 MHz, CDCl_3)



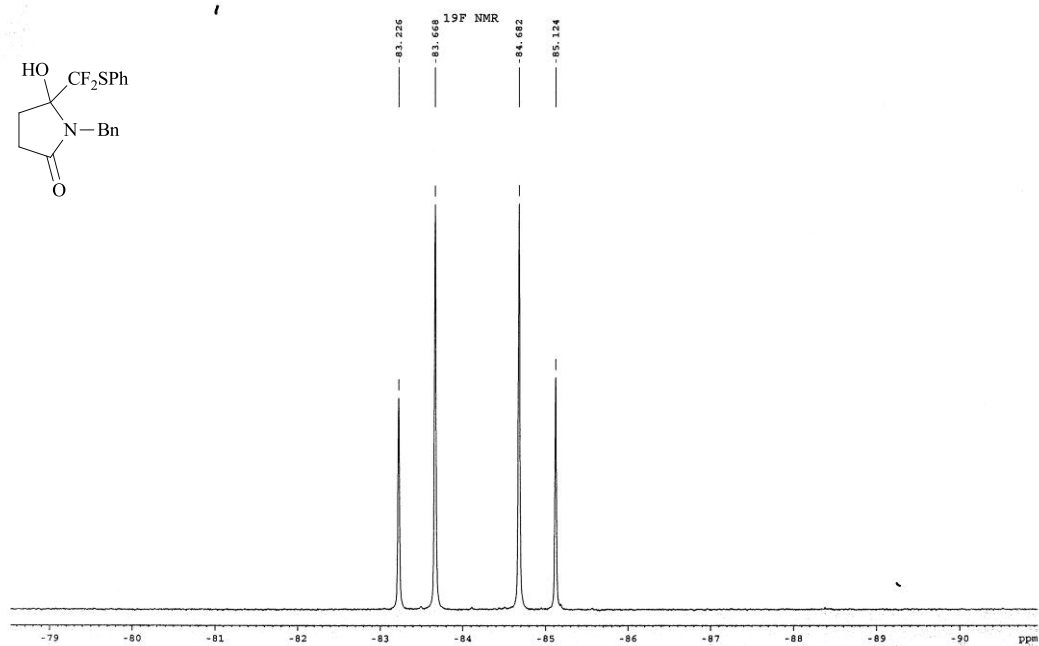
^1H NMR spectrum of **6d** (500 MHz, CDCl_3)



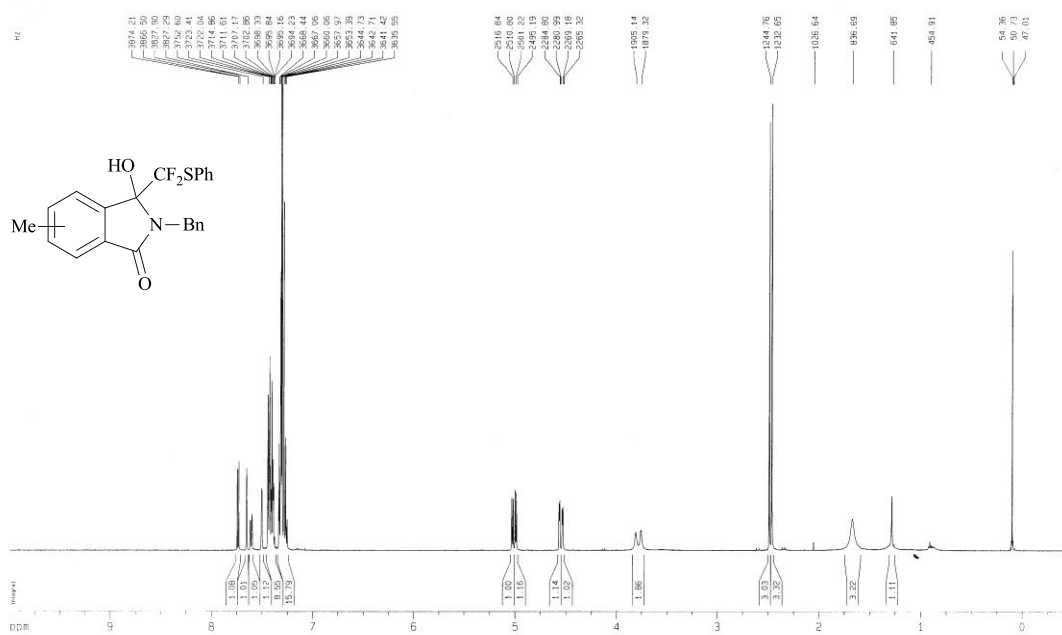
^{13}C NMR spectrum of **6d** (125 MHz, CDCl_3)



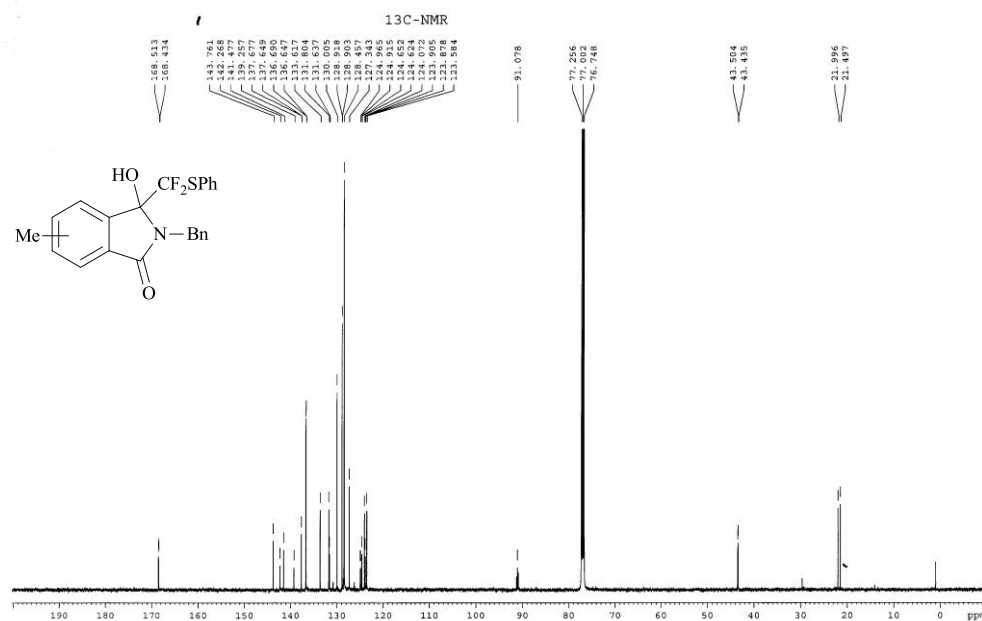
¹⁹F NMR spectrum of **6d** (470 MHz, CDCl₃)



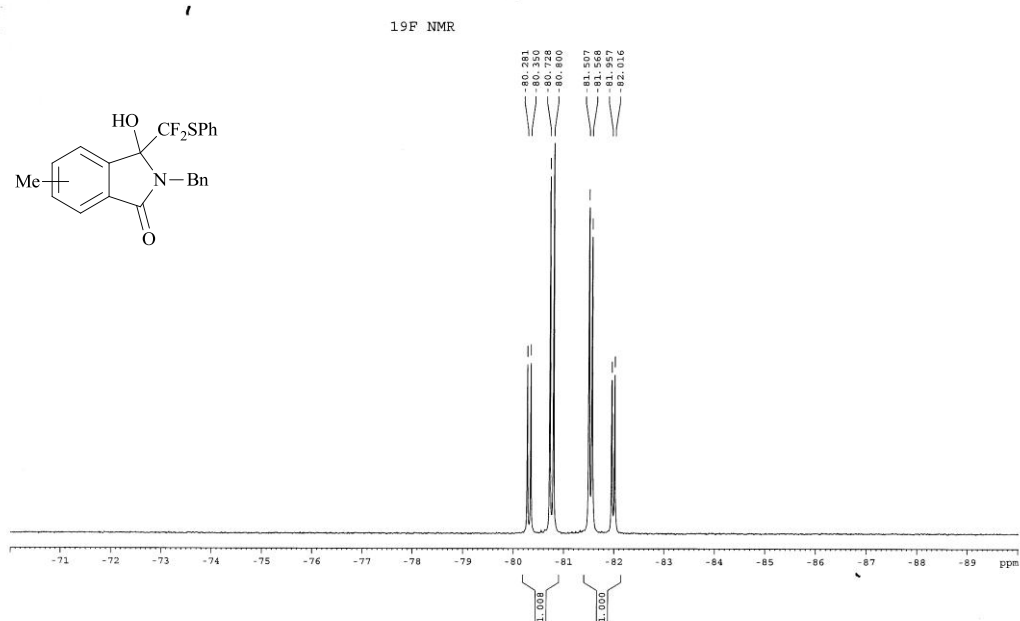
¹H NMR spectrum of **6e** (500 MHz, CDCl₃)



^{13}C NMR spectrum of **6e** (125 MHz, CDCl_3)

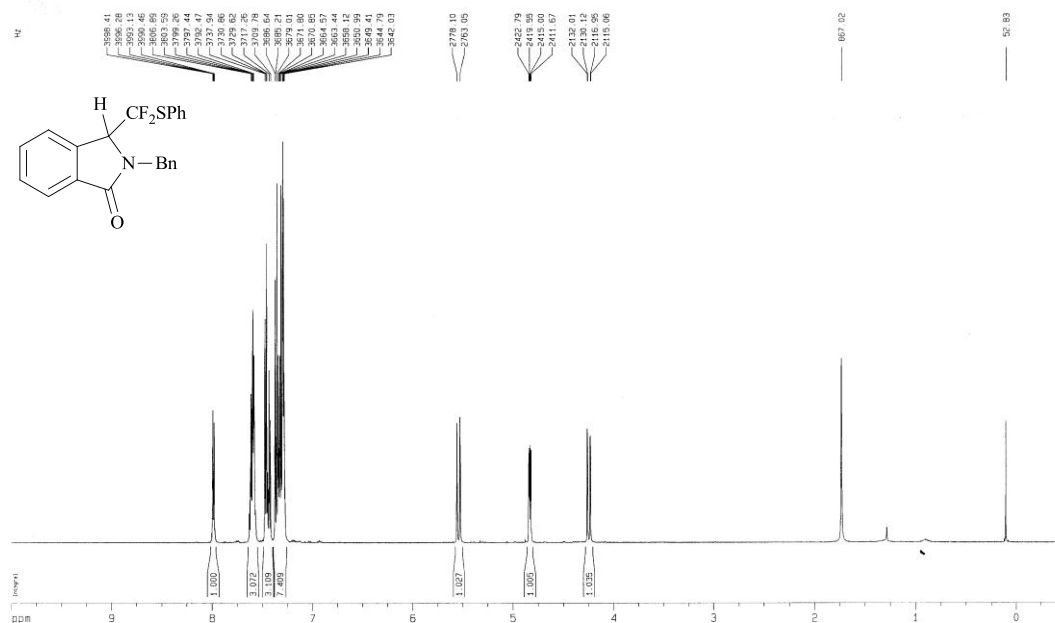


^{19}F NMR spectrum of **6e** (470 MHz, CDCl_3)

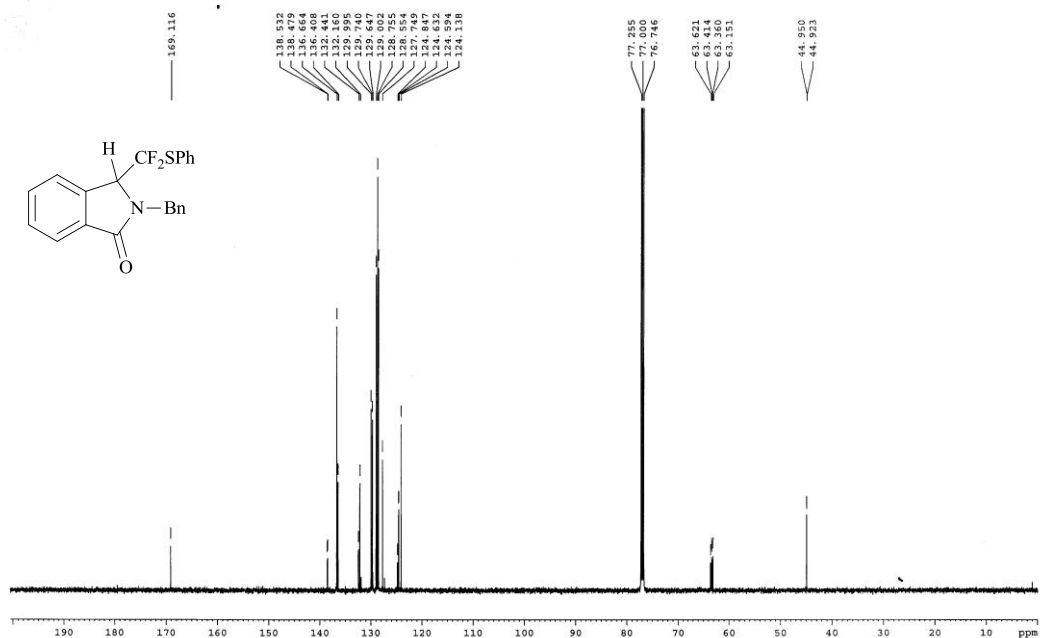


^1H , ^{13}C and ^{19}F NMR spectra of compounds 7

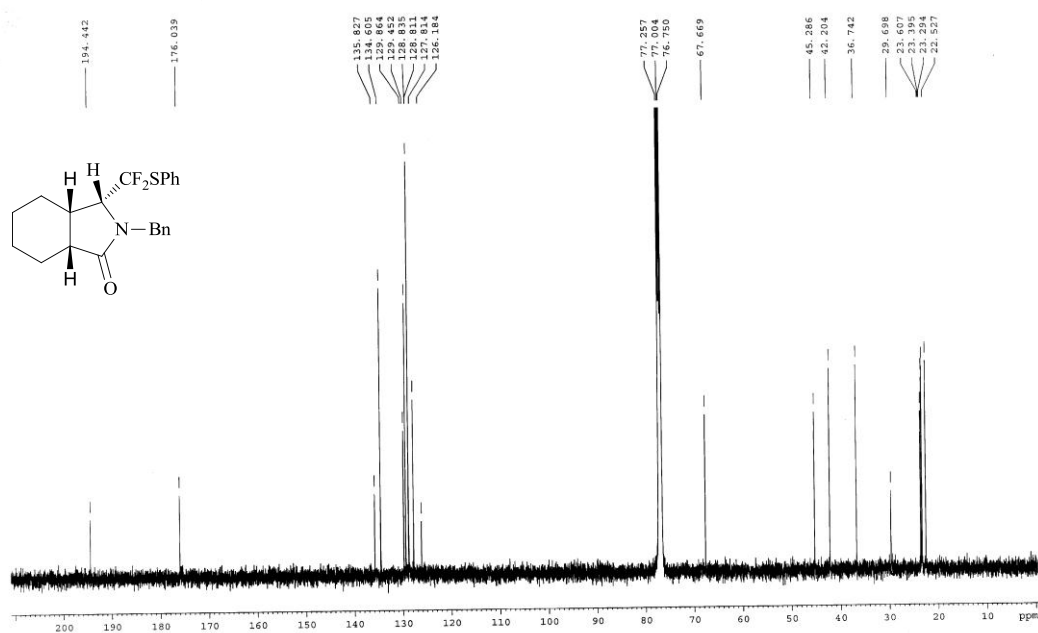
^1H NMR spectrum of 7a (500 MHz, CDCl_3)



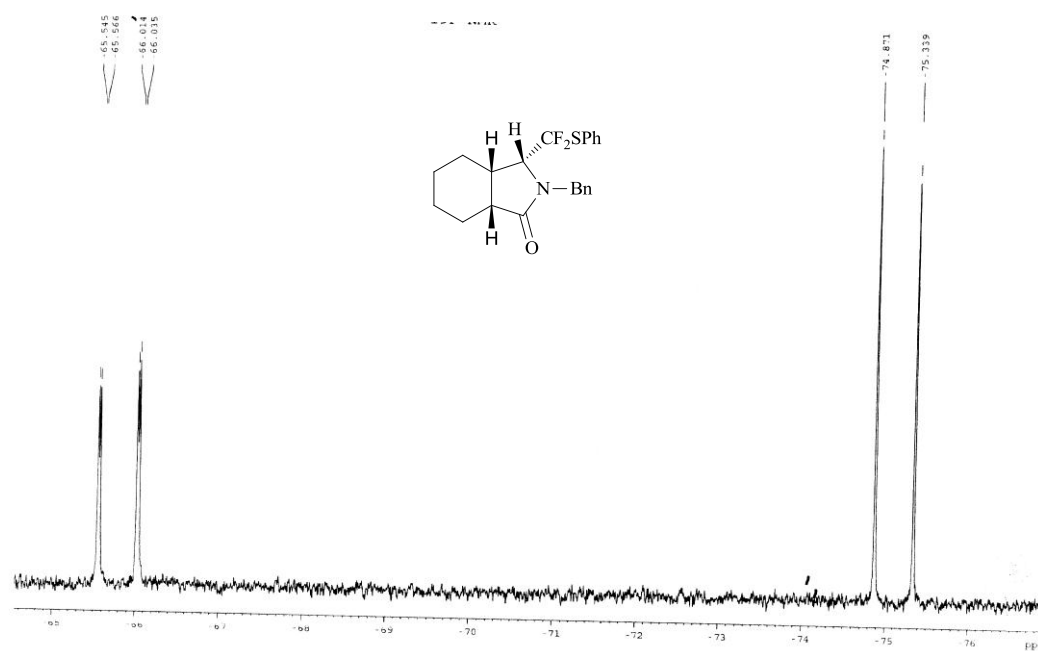
^{13}C NMR spectrum of 7a (125 MHz, CDCl_3)



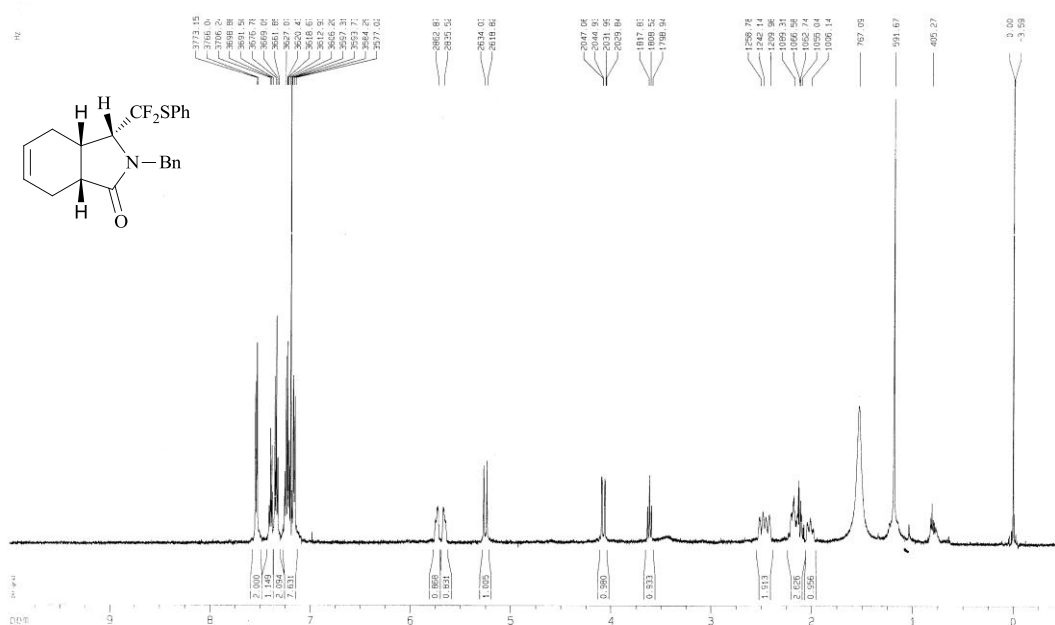
^{13}C NMR spectrum of **7b** (125 MHz, CDCl_3)



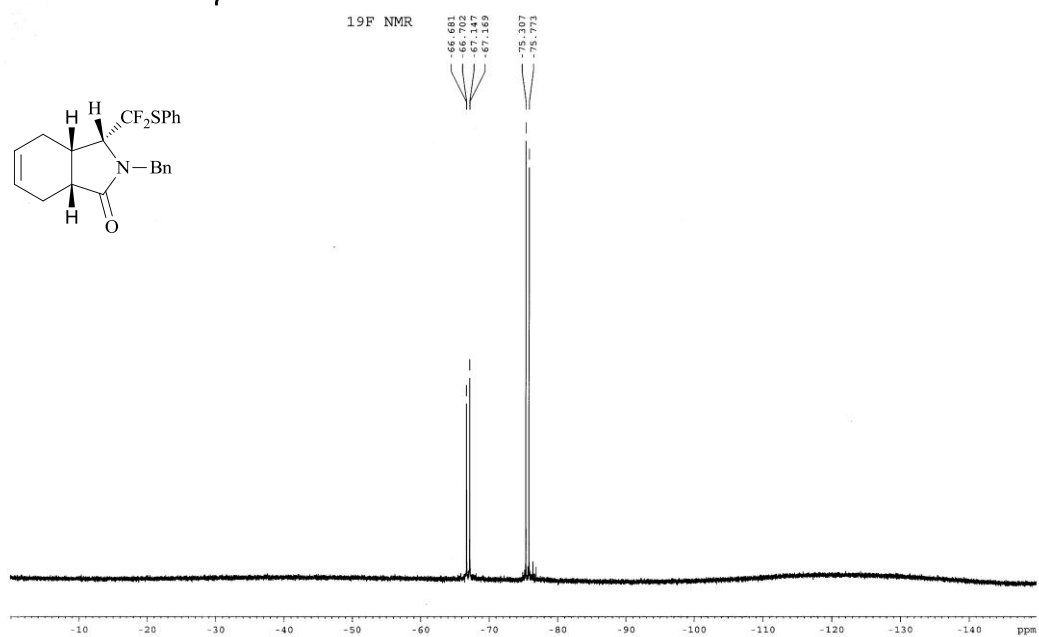
^{19}F NMR spectrum of **7c** (470 MHz, CDCl_3)



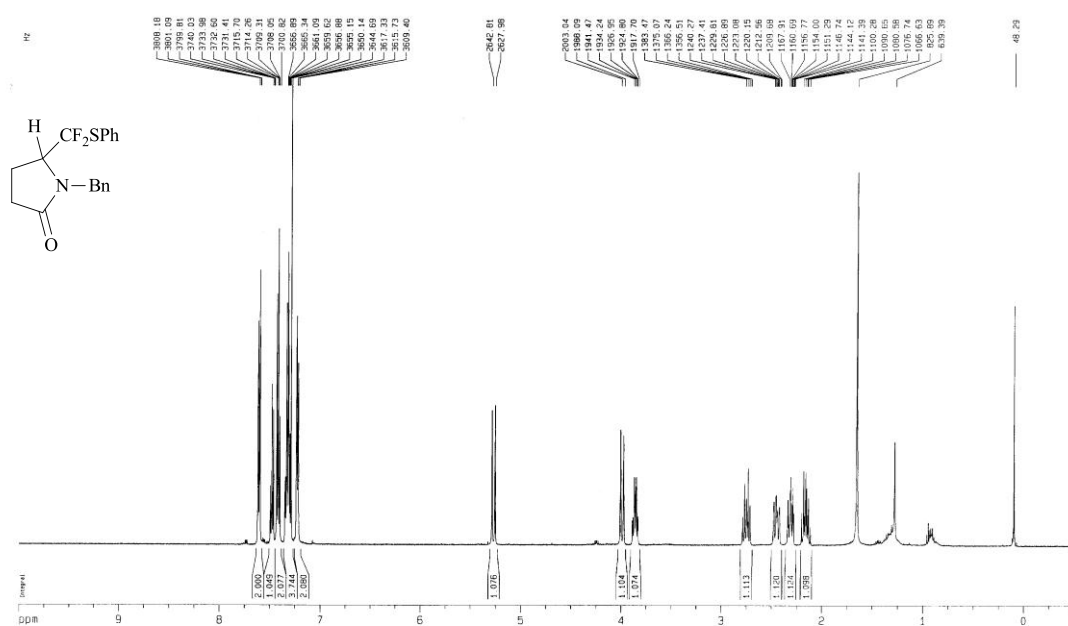
^1H NMR spectrum of 7c (500 MHz, CDCl_3)



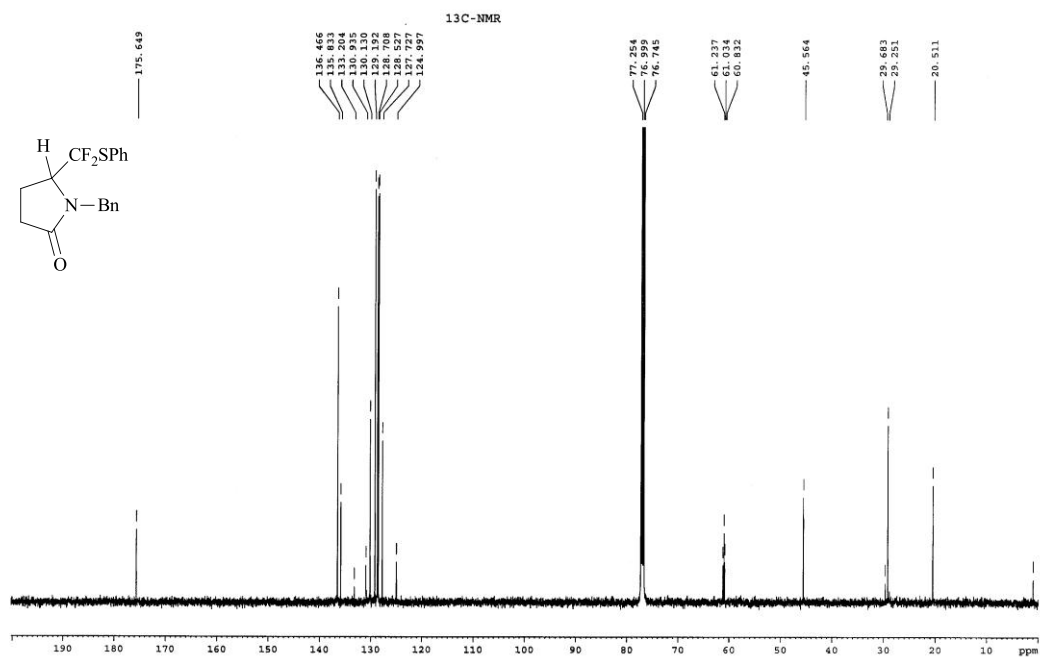
^{19}F NMR spectrum of **7c** (470 MHz, CDCl_3)



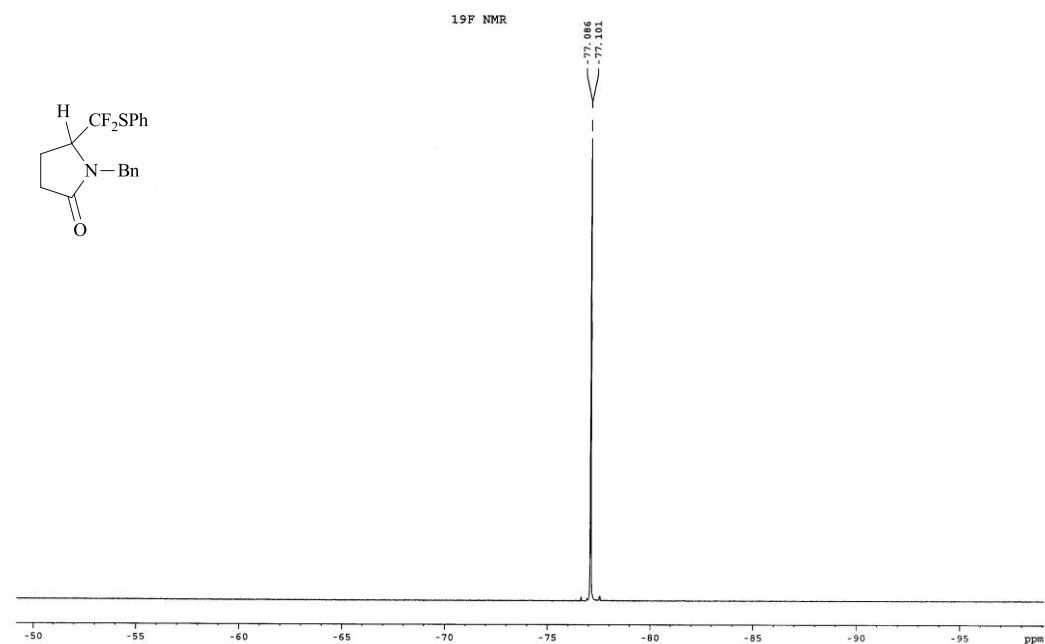
^1H NMR spectrum of **7d** (500 MHz, CDCl_3)



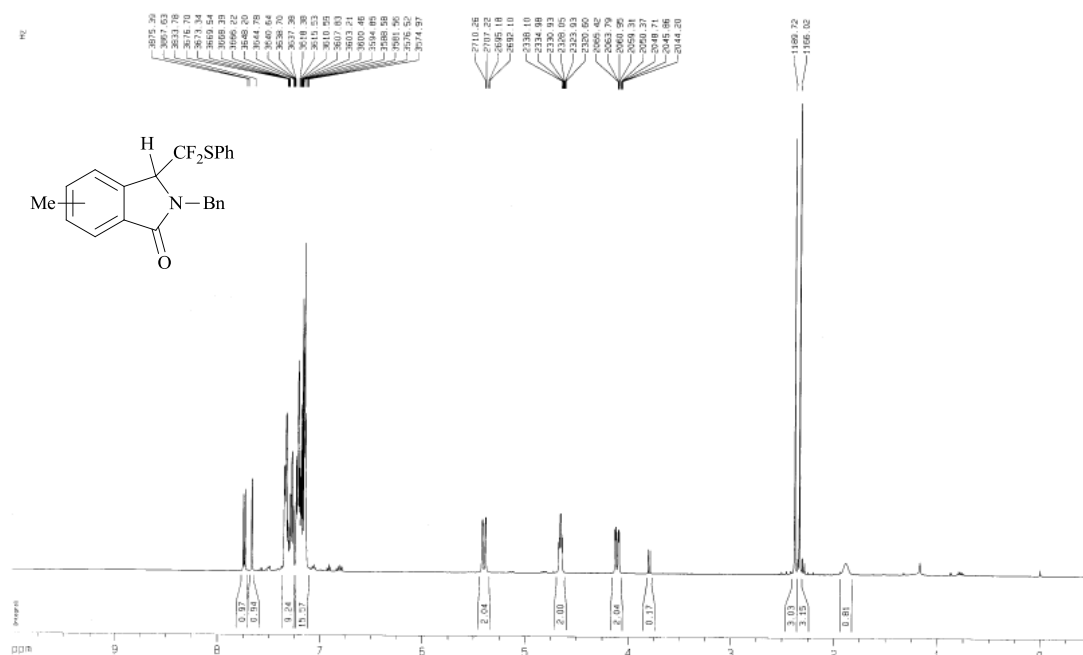
^{13}C NMR spectrum of **7d** (125 MHz, CDCl_3)



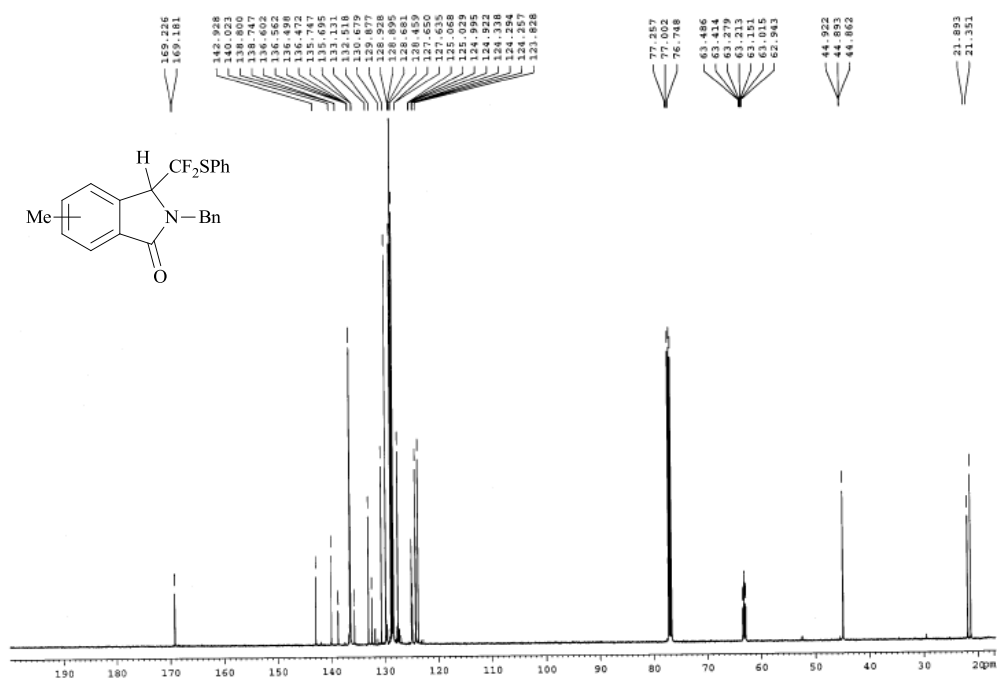
^{19}F NMR spectrum of **7d** (470 MHz, CDCl_3)



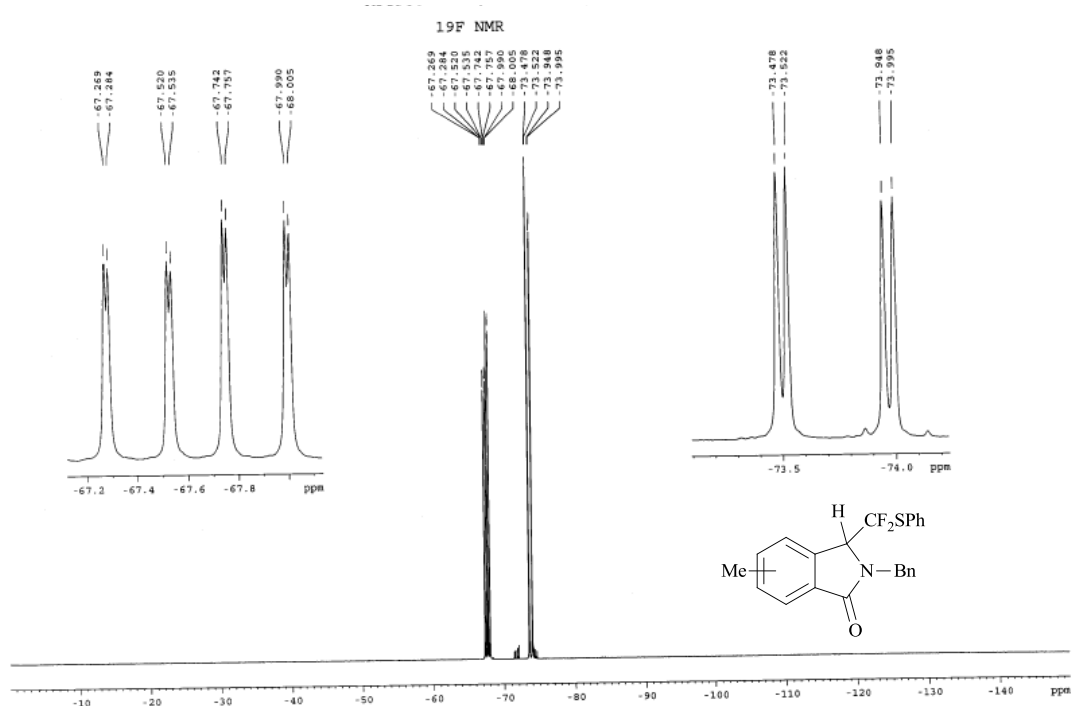
¹H NMR spectrum of 7e (500 MHz, CDCl₃)



¹³C NMR spectrum of 7c (125 MHz, CDCl₃)

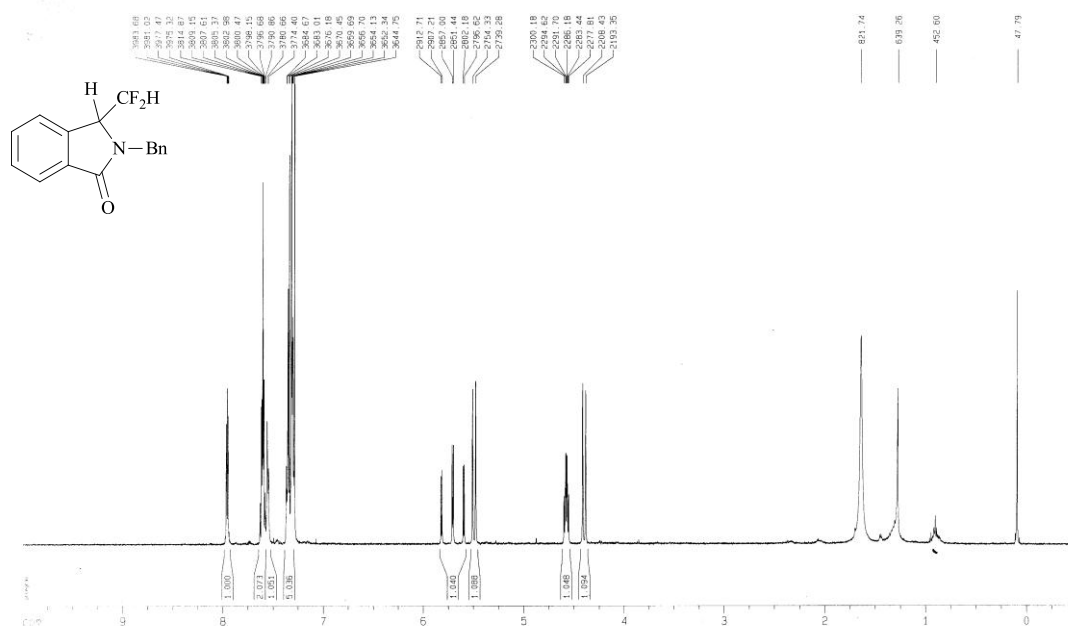


^{19}F NMR spectrum of **7c** (470 MHz, CDCl_3)

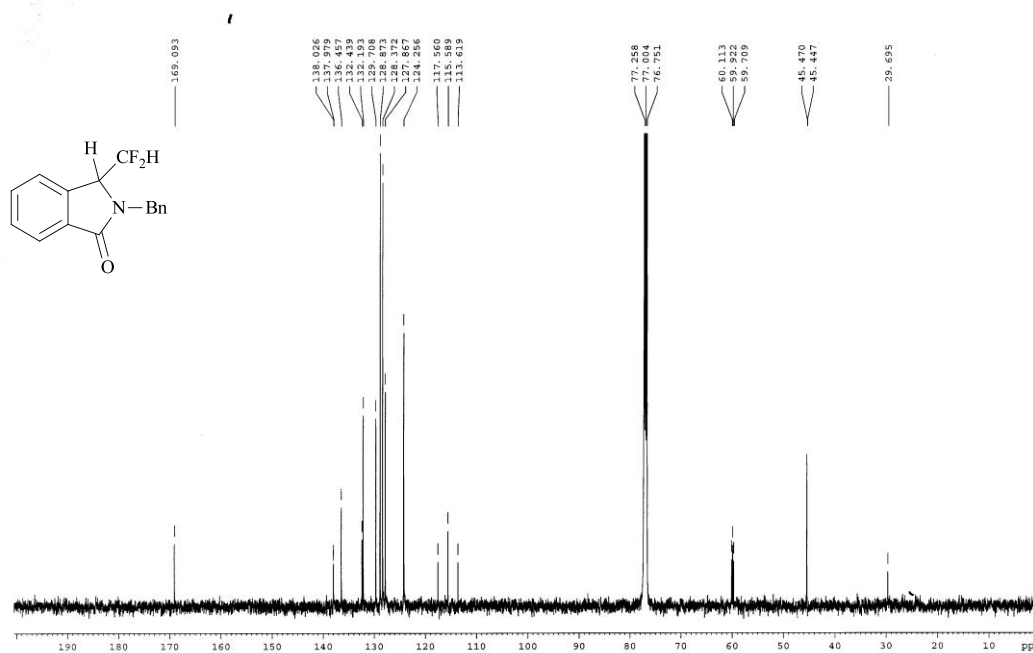


^1H , ^{13}C and ^{19}F NMR spectra of compounds **8**

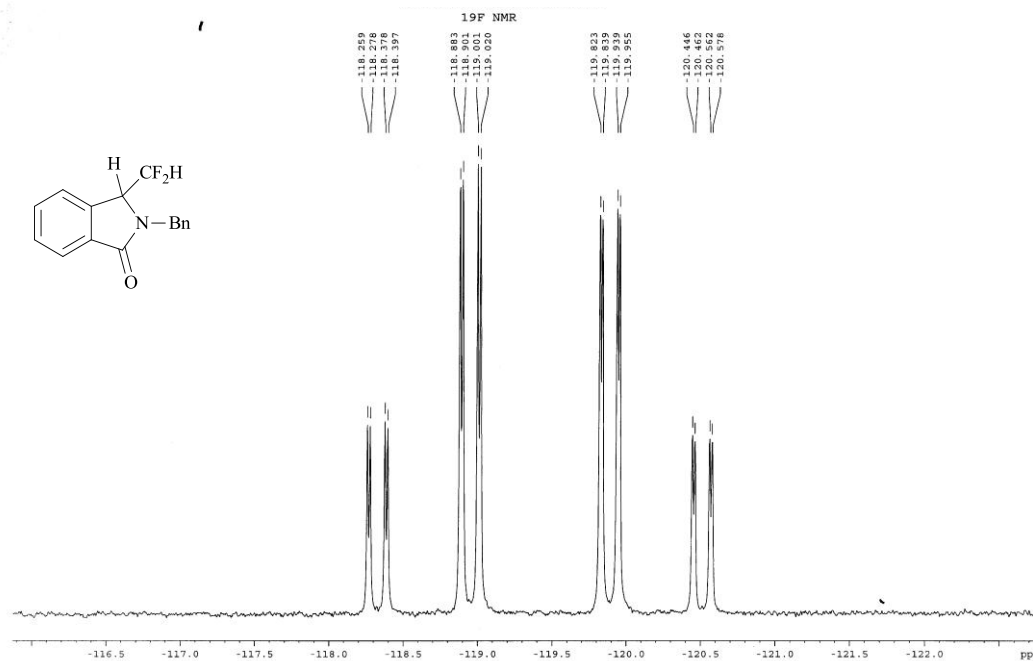
^1H NMR spectrum of **8a** (500 MHz, CDCl_3)



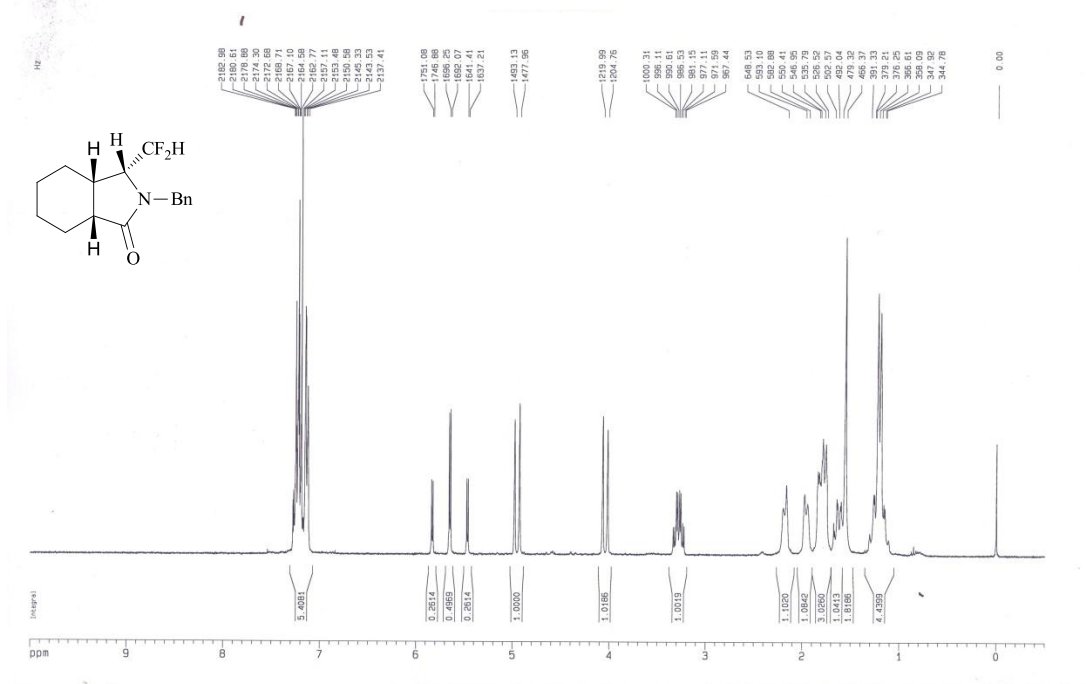
^{13}C NMR spectrum of **8a** (125 MHz, CDCl_3)



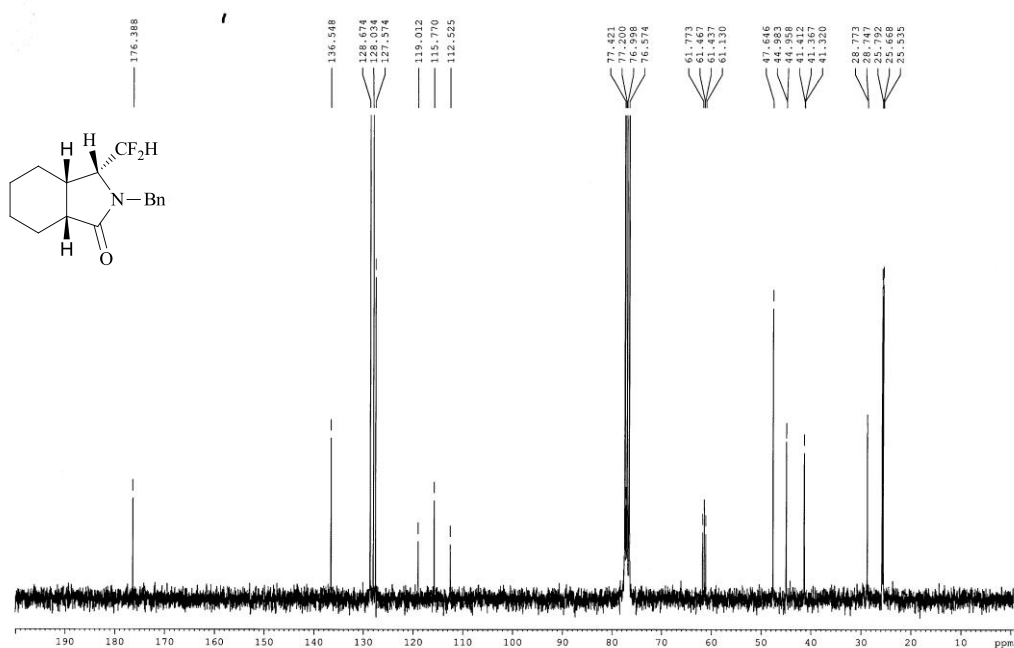
^{19}F NMR spectrum of **8a** (470 MHz, CDCl_3)



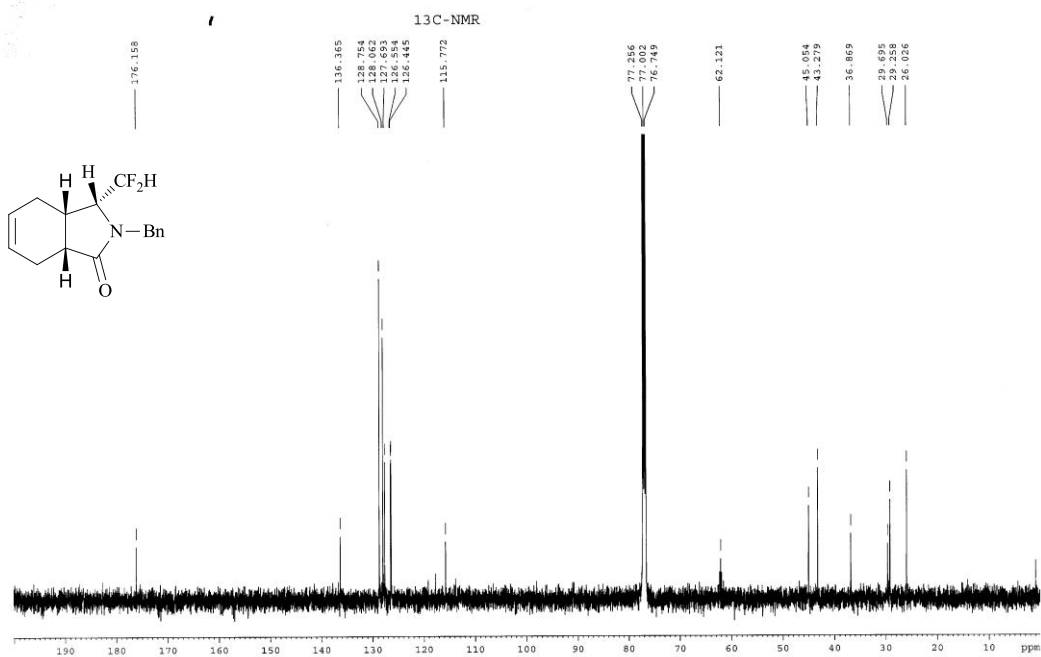
¹H NMR spectrum of **8b** (300 MHz, CDCl₃)



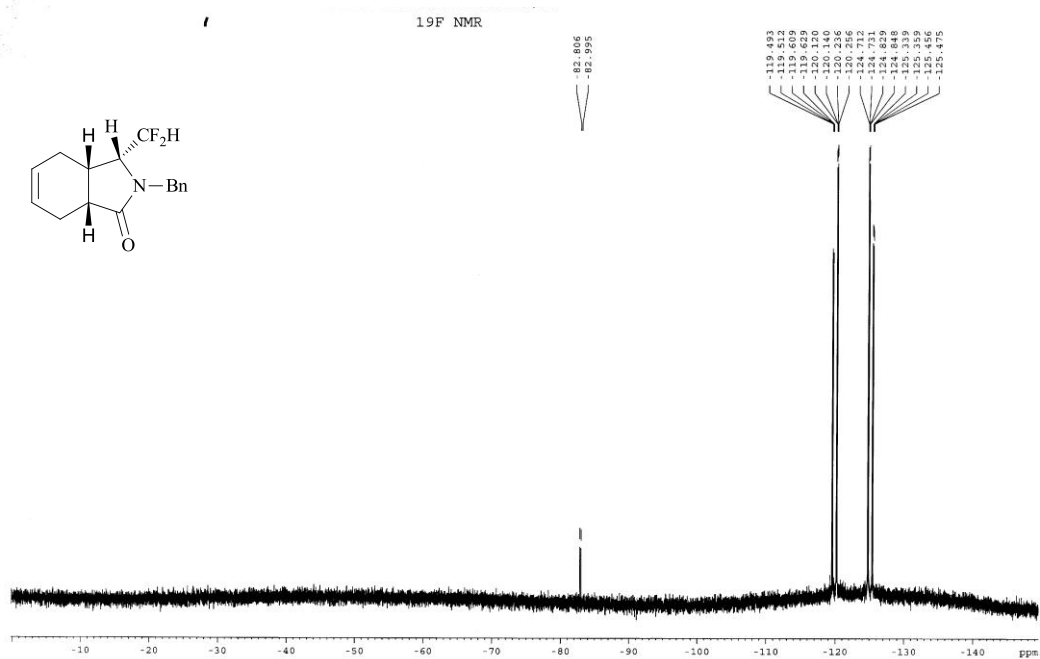
¹³C NMR spectrum of **8b** (75 MHz, CDCl₃)



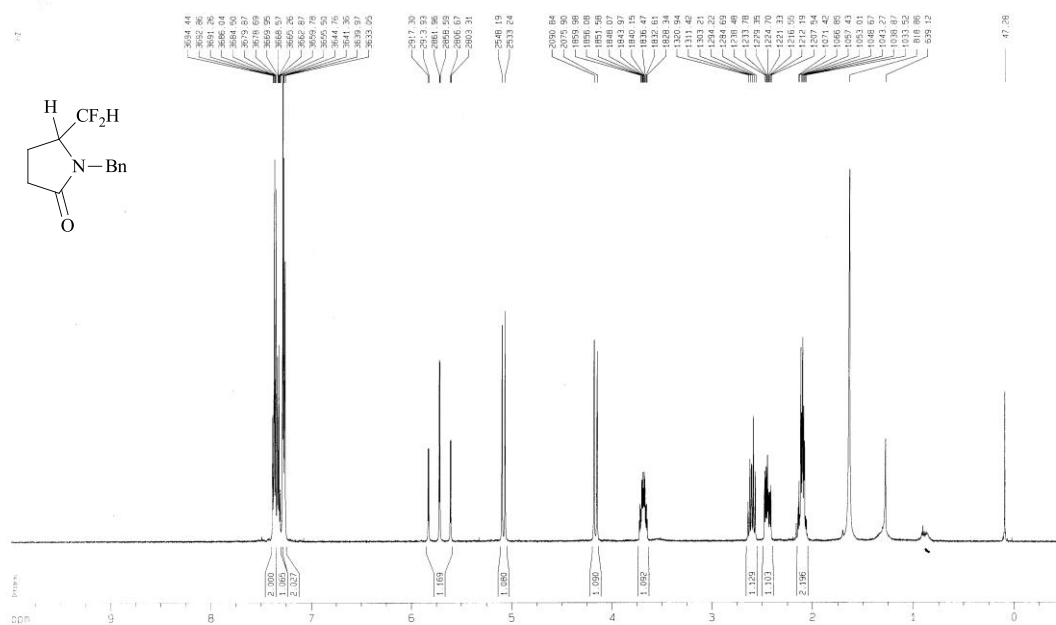
^{13}C NMR spectrum of **8c** (125 MHz, CDCl_3)



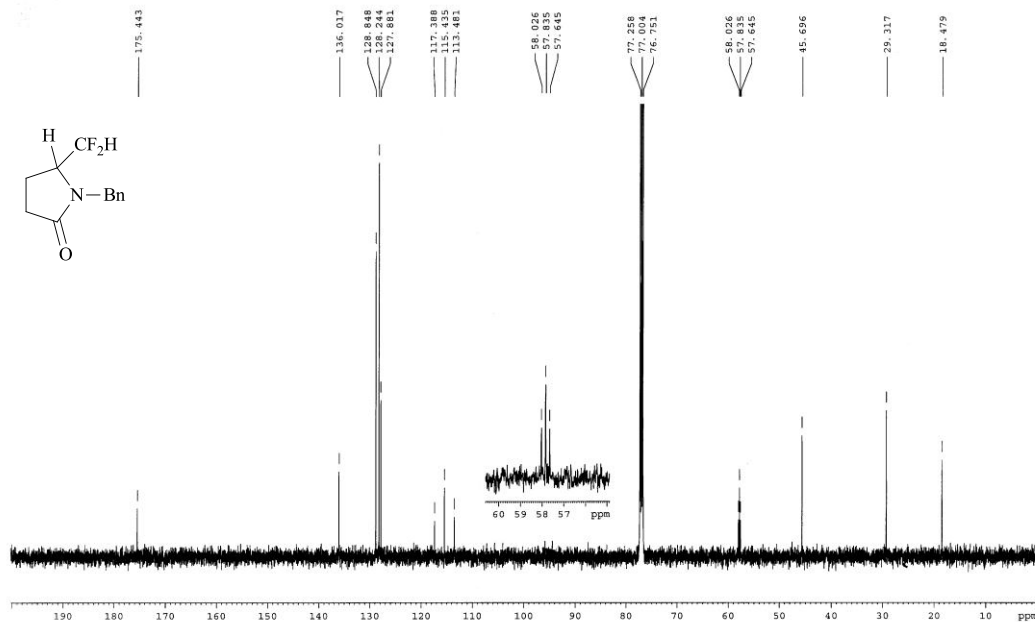
^{19}F NMR spectrum of **8c** (470 MHz, CDCl_3)



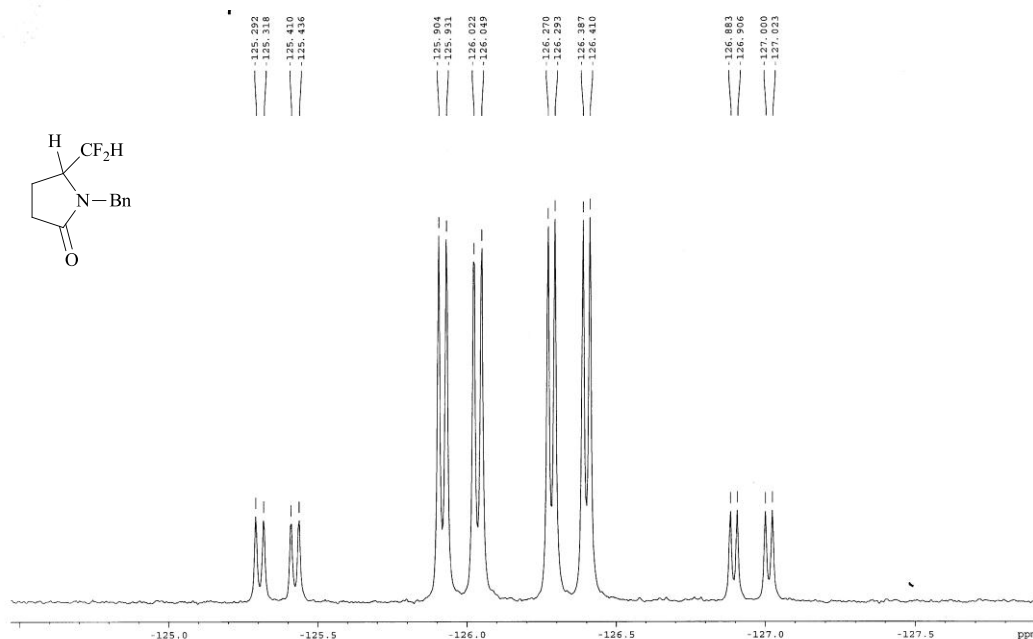
¹H NMR spectrum of **8d** (500 MHz, CDCl₃)



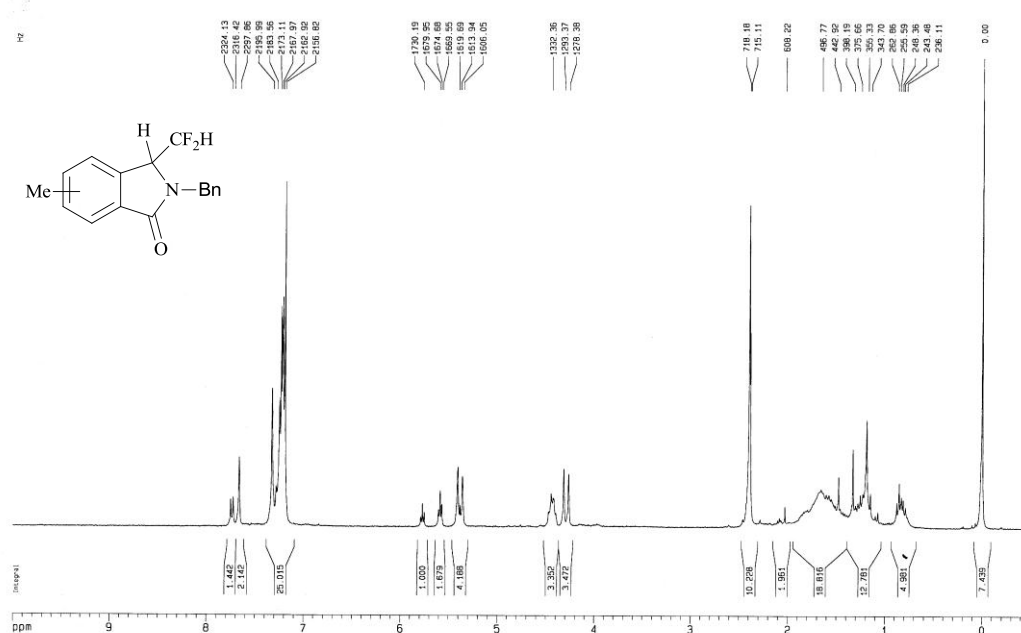
¹³C NMR spectrum of **8d** (125 MHz, CDCl₃)



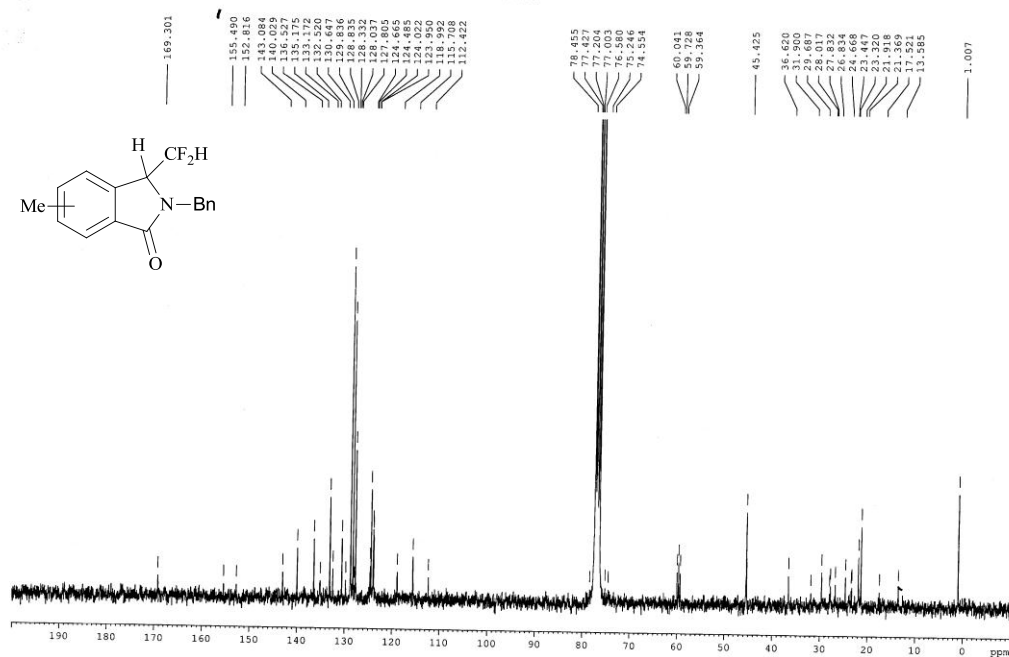
^{19}F NMR spectrum of **8d** (470 MHz, CDCl_3)



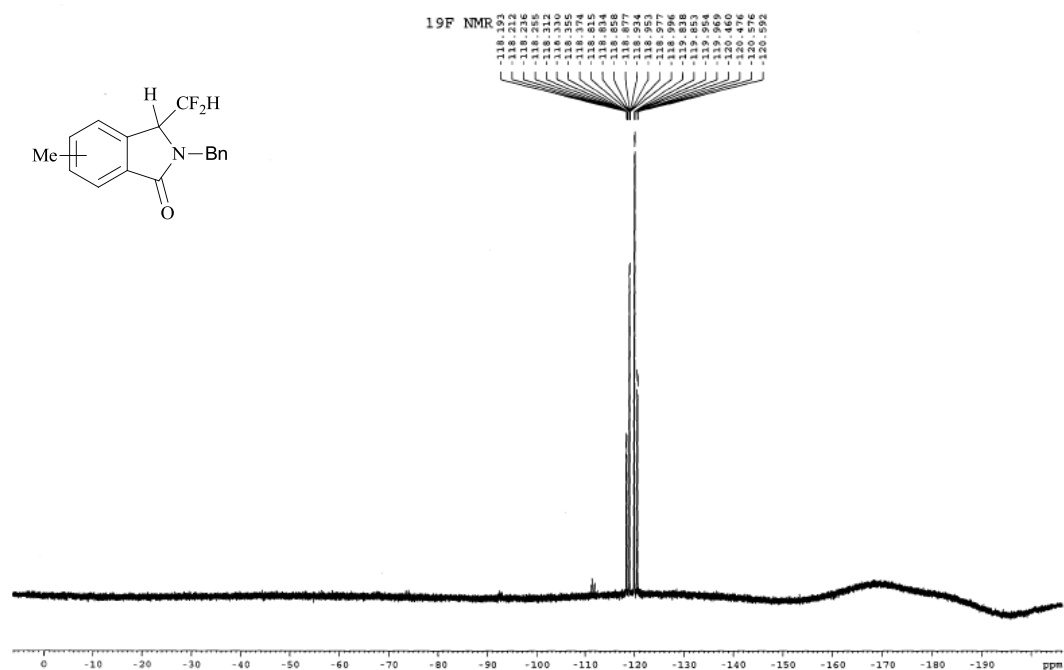
^1H NMR spectrum of **8e** (300 MHz, CDCl_3)



^{13}C NMR spectrum of **8e** (75 MHz, CDCl_3)



^{19}F NMR spectrum of **8e** (470 MHz, CDCl_3)



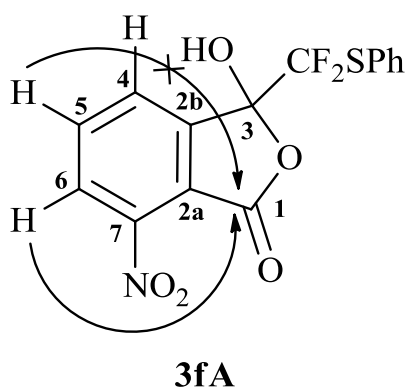


Table 1. Observed correlations in the HMQC spectrum of **3fA** (CD₃OD).

Carbon	δ_C (ppm)	δ_H of correlated proton (ppm)
C-1	163.0	-
C-2a	120.7	-
C-2b	129.9	-
C-3	104.5	-
C-4	129.9	8.06 (dd, $J = 1.0, 7.8$ Hz, H-4)
C-5	137.7	8.00 (dd, $J = 7.8, 7.8$ Hz, H-5)
C-6	127.5	8.81 (dd, $J = 1.0, 7.8$ Hz, H-6)
C-7	147.9	-

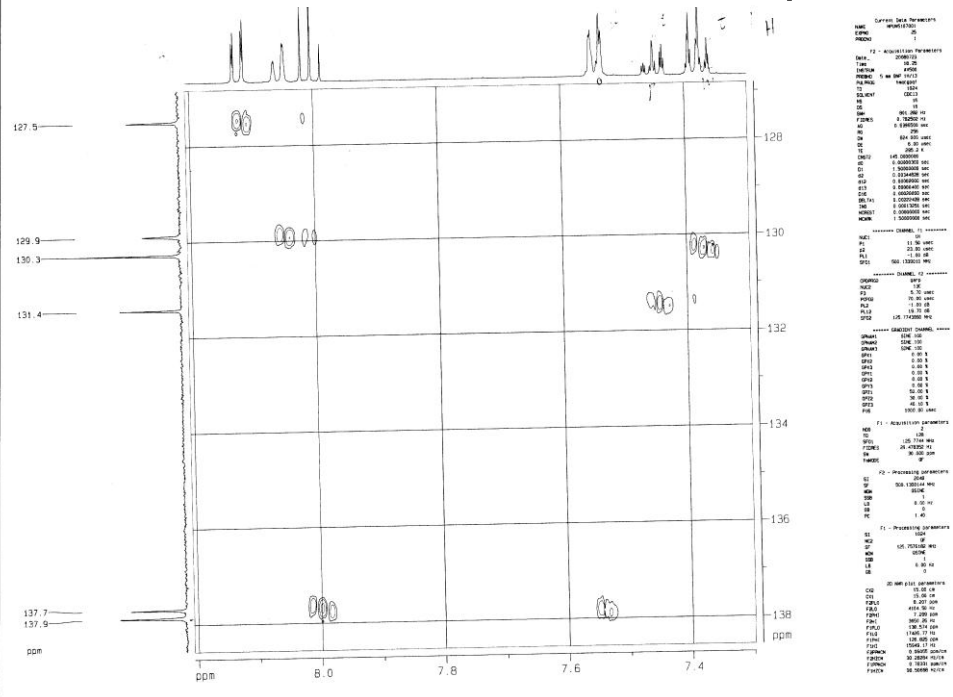
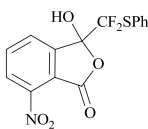
Table 2. Observed correlations in COSY (¹H-¹H) spectrum of **3fA** (CD₃OD).

δ_H (ppm)	δ_H (ppm) of correlated proton
8.00 (H-5)	8.06 (H-4), 8.81 (H-6)
8.06 (H-4)	8.00 (H-5), 8.81 (H-6)
8.81 (H-6)	8.00 (H-5), 8.06 (H-4)

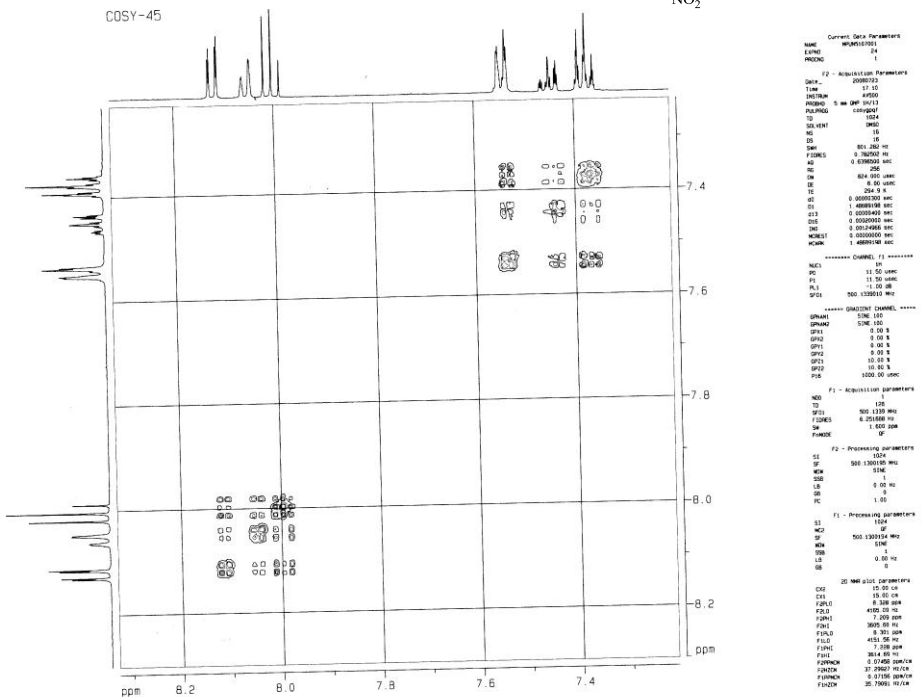
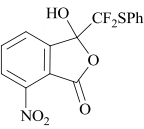
Table 3. Observed long-range C-H correlations in the HMBC spectrum of **3fA** (CD₃OD).

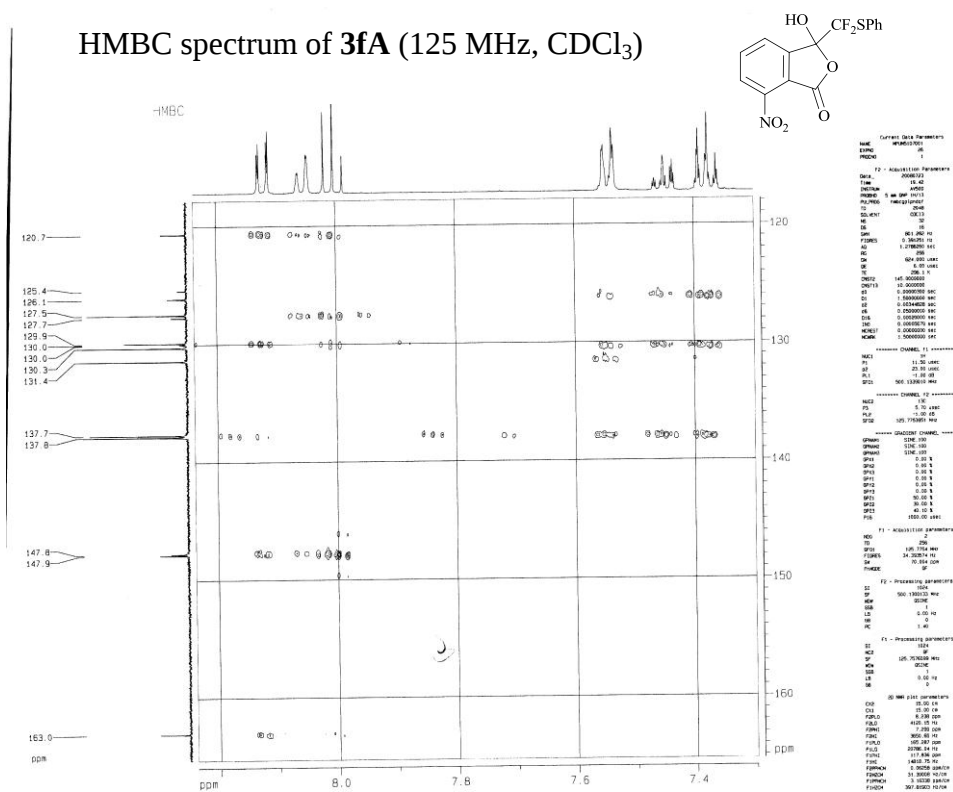
δ_H (ppm)	Carbon correlated with δ_H (ppm)
8.00 (H-5)	120.7 (C-2a), 127.1 (CF ₂), 129.9 (C-2b), 147.9 (C-7)
8.06 (H-4)	120.7 (C-2a), 127.1 (CF ₂), 147.9 (C-7)
8.81 (H-6)	120.7 (C-2a), 129.9 (C-2b), 127.9 (C-2), 163.0 (C-1)

HMQC spectrum of **3fA** (125 MHz, CDCl₃)



COSY-45 spectrum of **3fA** (125 MHz, CDCl₃)





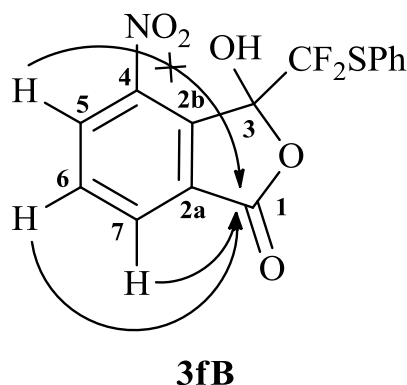


Table 4. Observed correlations in the HMQC spectrum of **3fB** (CD₃OD).

Carbon	δ_C (ppm)	δ_H of correlated proton (ppm)
C-1	166.3	-
C-2a	131.5	-
C-2b	136.1	-
C-3	100.0	-
C-4	148.6	-
C-5	131.5	8.29 (dd, $J = 0.9, 7.9$ Hz, H-5)
C-6	135.0	7.97 (dd, $J = 7.9, 7.9$ Hz, H-6)
C-7	131.1	8.24 (dd, $J = 0.9, 7.9$ Hz, H-7)

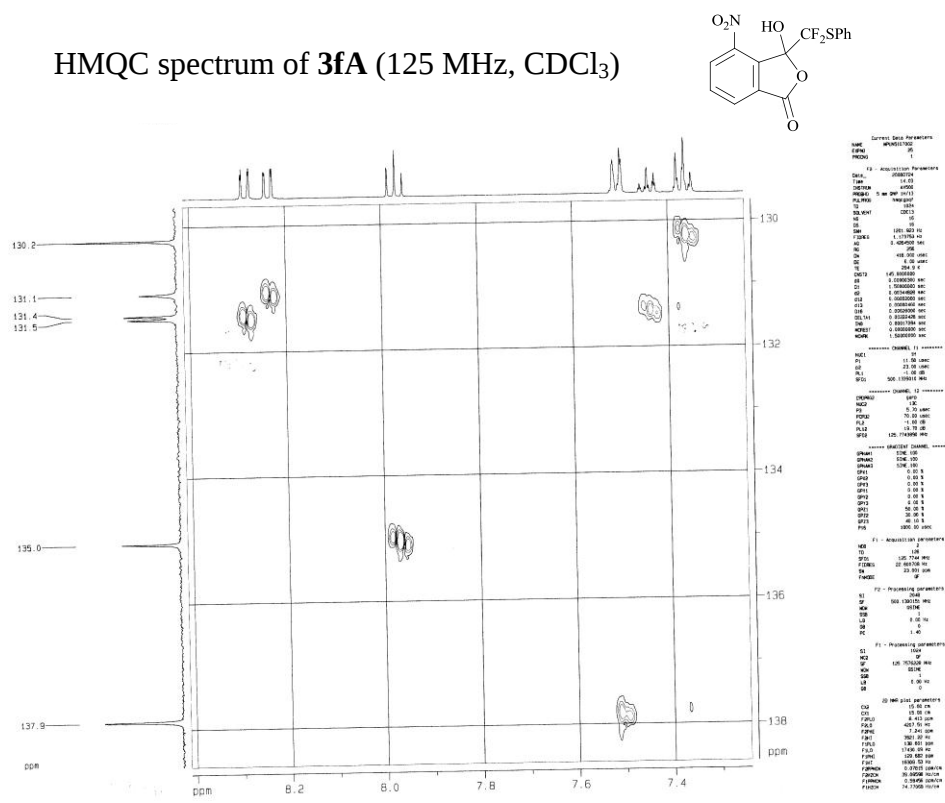
Table 5. Observed correlations in COSY (¹H-¹H) spectrum of **3fB** (CD₃OD).

δ_H (ppm)	δ_H (ppm) of correlated proton
7.97 (H-6)	8.24 (H-7), 8.29 (H-5)
8.24 (H-7)	7.79 (H-6), 8.29 (H-5)
8.29 (H-5)	7.97 (H-6), 8.24 (H-7)

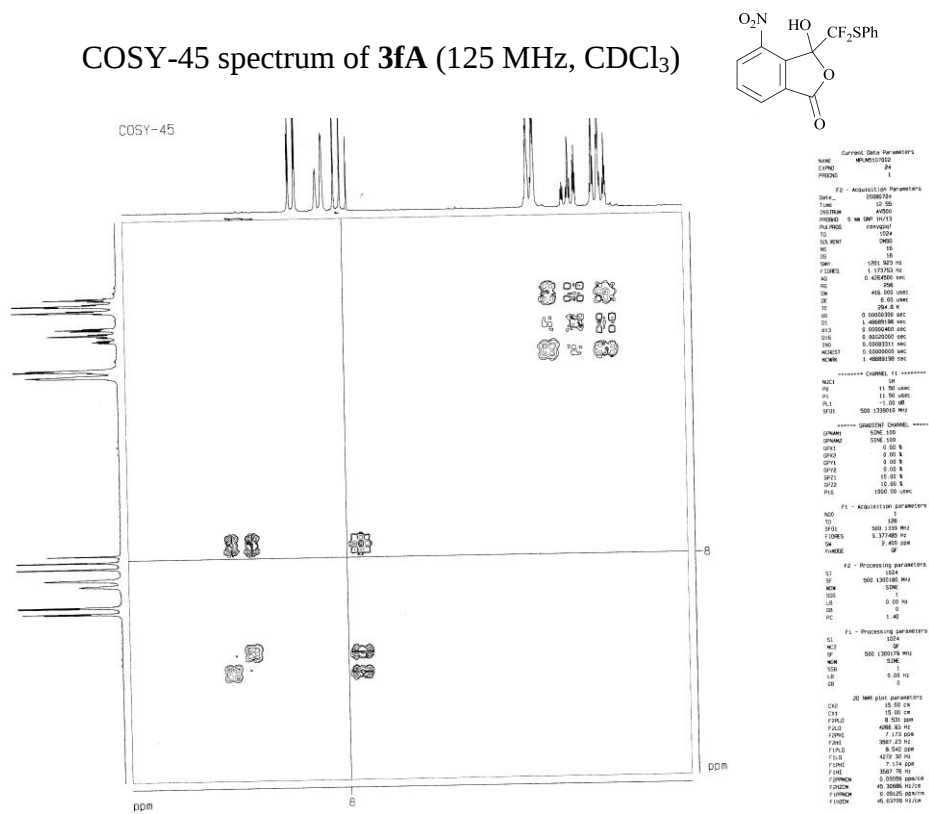
Table 6. Observed long-range C-H correlations in the HMBC spectrum of **3fB** (CD₃OD).

δ_H (ppm)	Carbon correlated with δ_H (ppm)
7.97 (H-6)	131.1 (C-7), 131.5 (C-2a, C-5), 136.1 (C-2b), 148.6 (C-4), 166.3 (C-1)
8.24 (H-7)	131.1 (C-7), 131.5 (C-2a, C-5), 136.1 (C-2b), 148.6 (C-4), 166.3 (C-1)
8.29 (H-5)	131.1 (C-7), 131.5 (C-2a, C-5), 136.1 (C-2b), 148.6 (C-4)

HMQC spectrum of **3fA** (125 MHz, CDCl₃)



COSY-45 spectrum of **3fA** (125 MHz, CDCl₃)



HMBC spectrum of **3fA** (125 MHz, CDCl₃)

