Electronic Supporting Information

A synthesis of γ-trifluoromethyl α,β-unsaturated γ-butyrolactones using CF_3SiMe_3 as a trifluoromethylating agent

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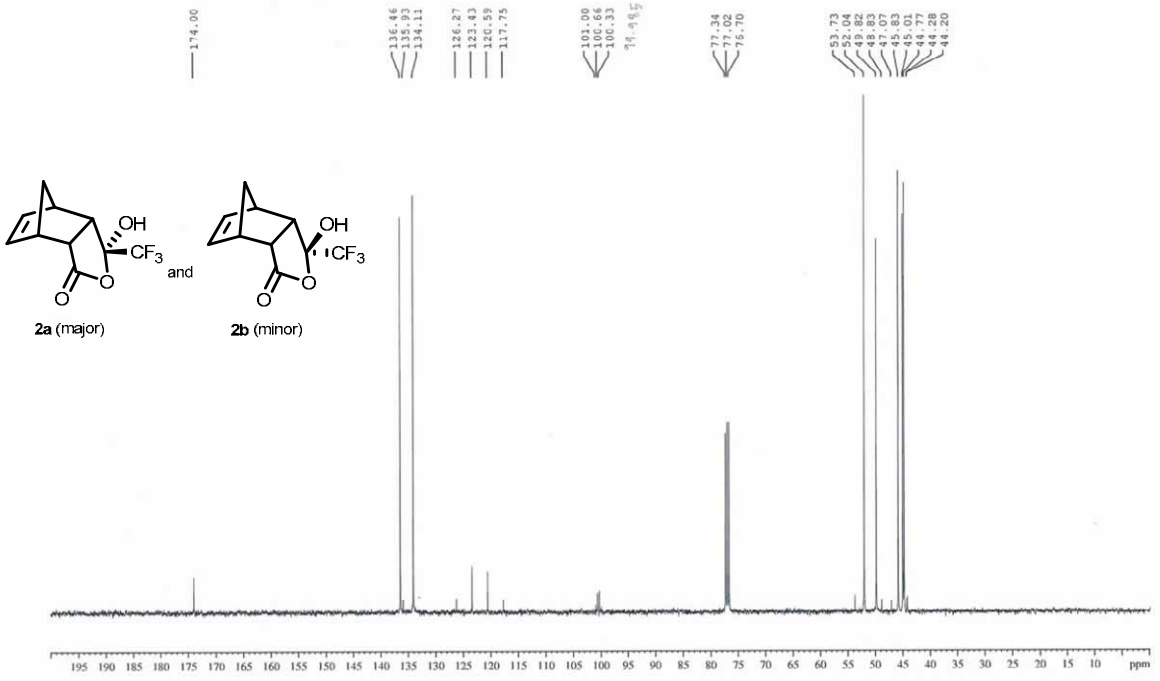
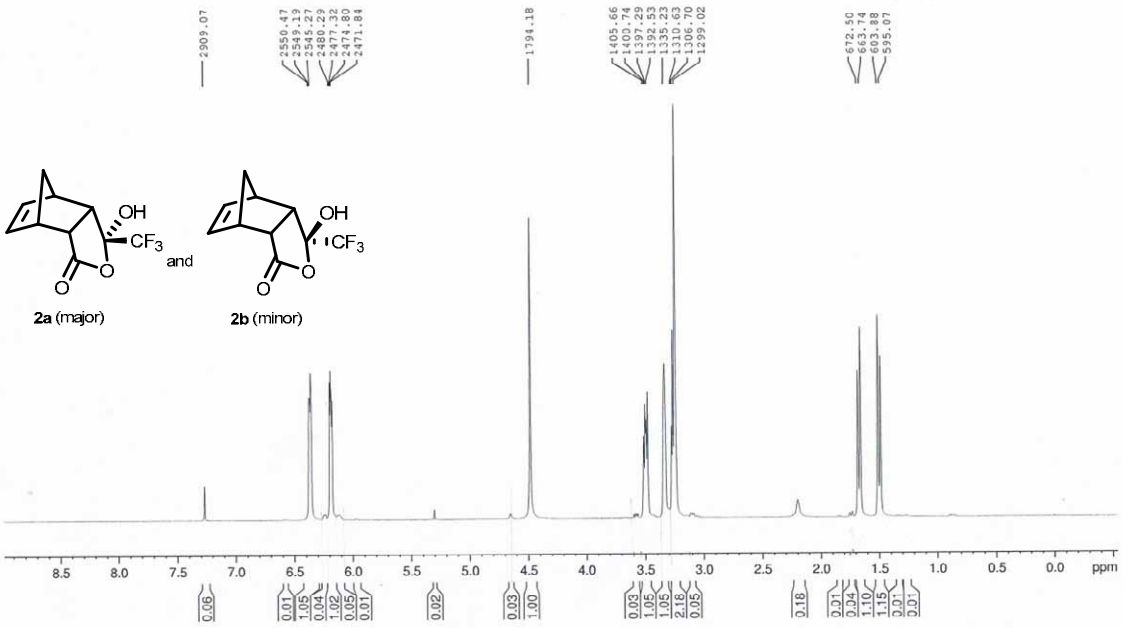
Contents

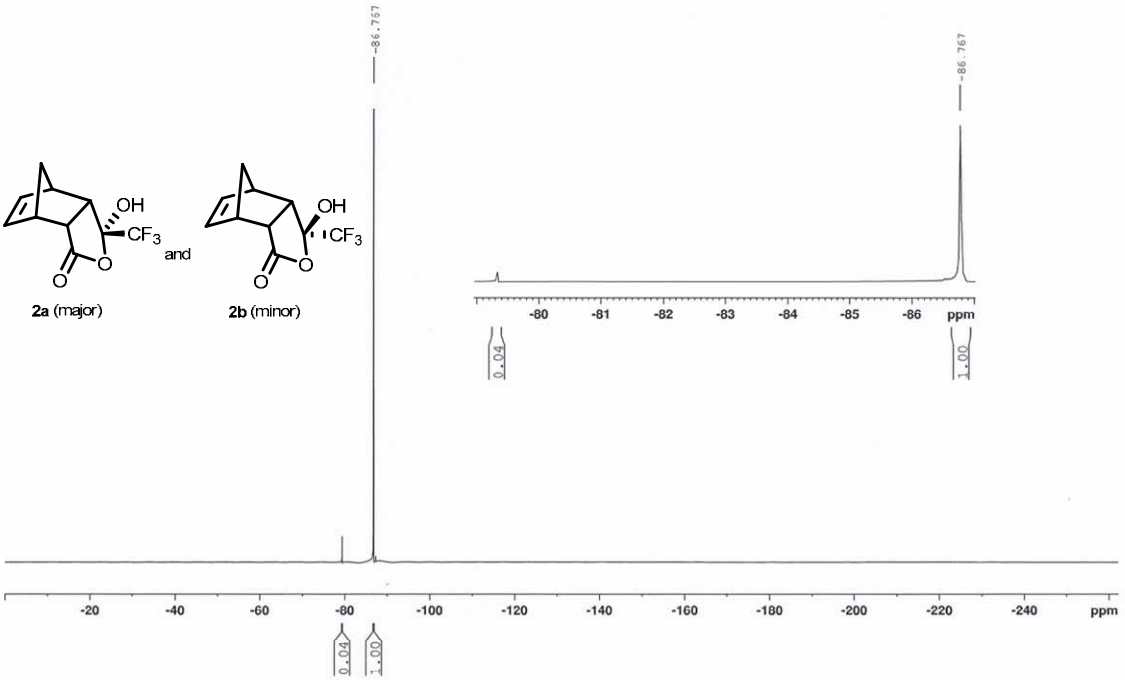
^{1}H NMR Spectrum of **2a** and **2b** (400 MHz, $CDCl_3$)



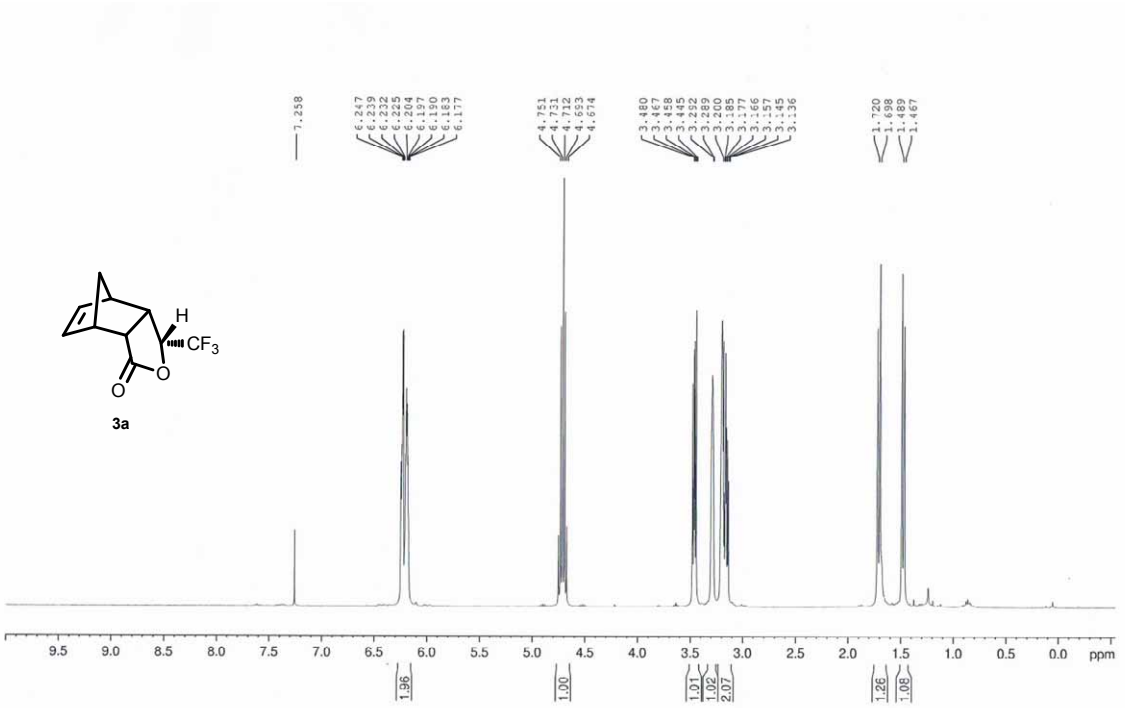
^{13}C NMR Spectrum of **2a** and **2b** (100 MHz, $CDCl_3$)



^{19}F NMR Spectrum of **2a** and **2b** (376 MHz, $CDCl_3$)



^{1}H NMR Spectrum of **3a** (400 MHz, $CDCl_3$)



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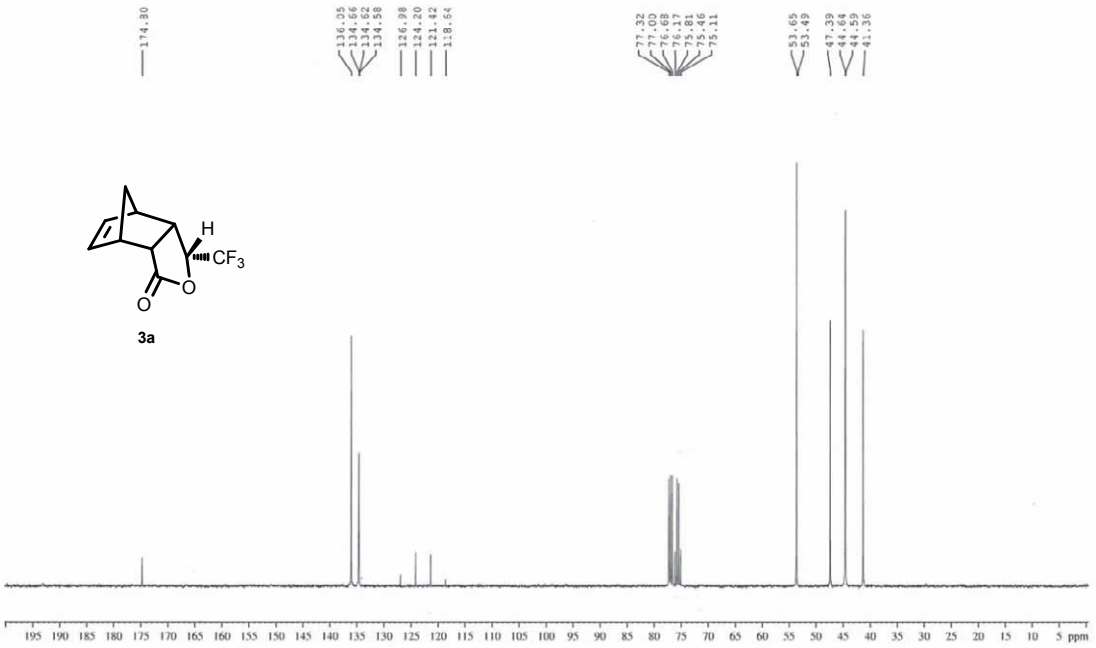

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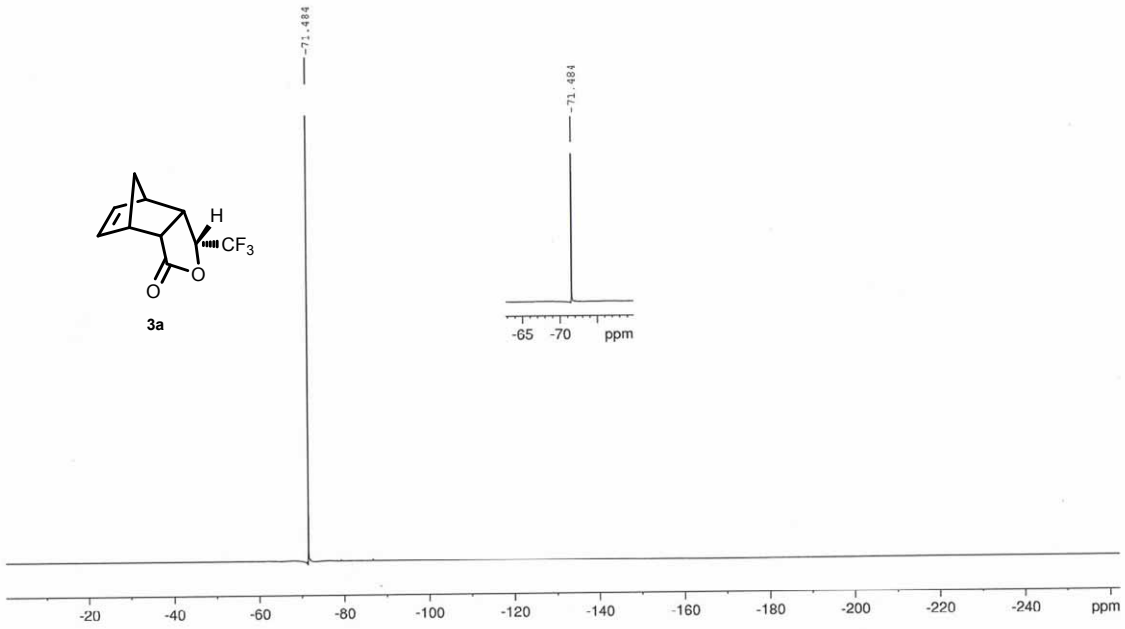
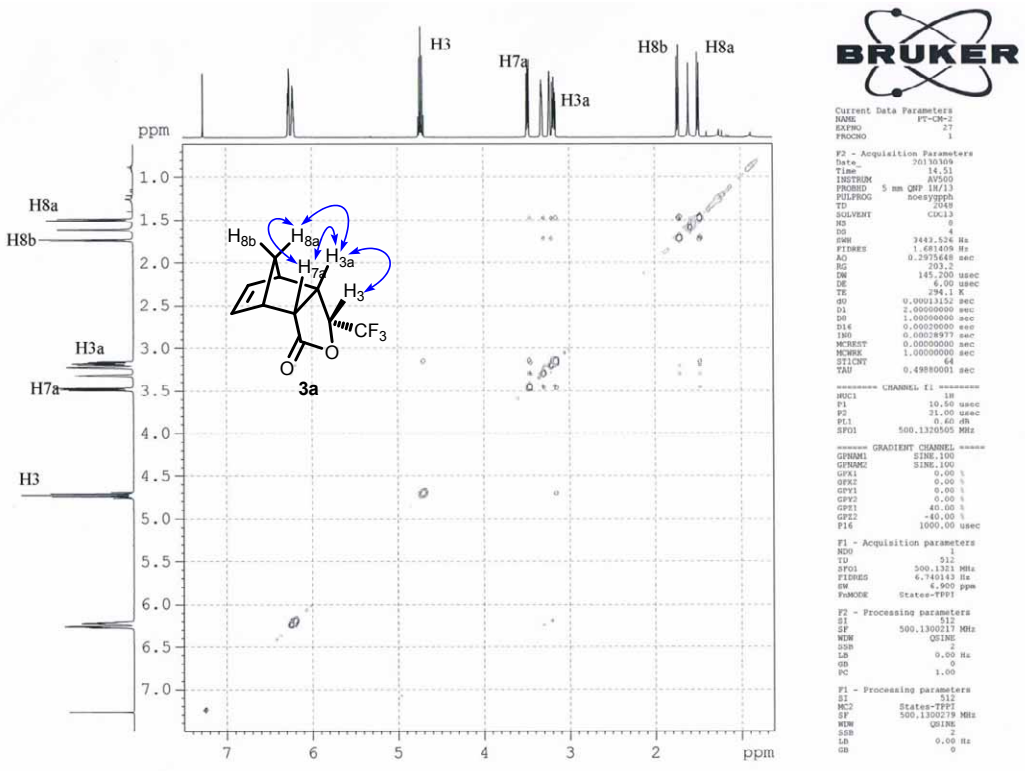


NOESY Spectrum of **3a** (500 MHz, $CDCl_3$)



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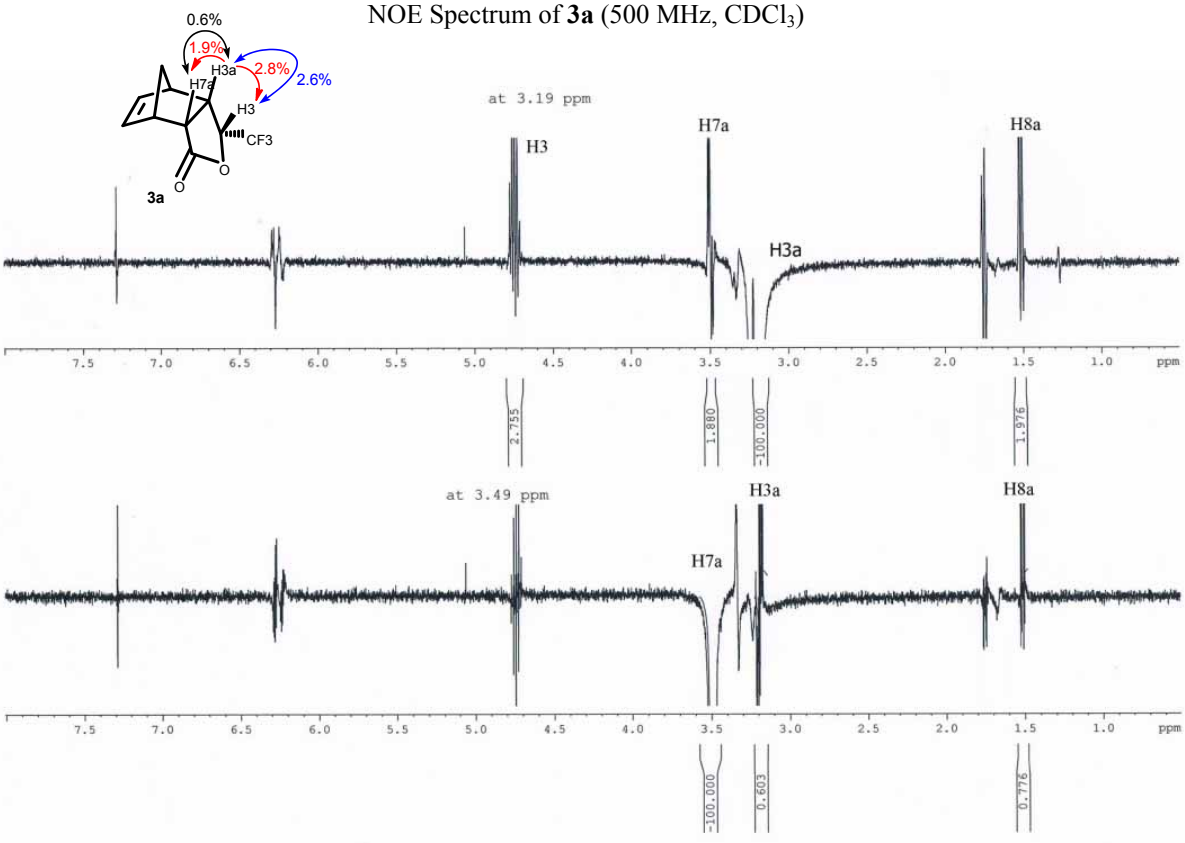

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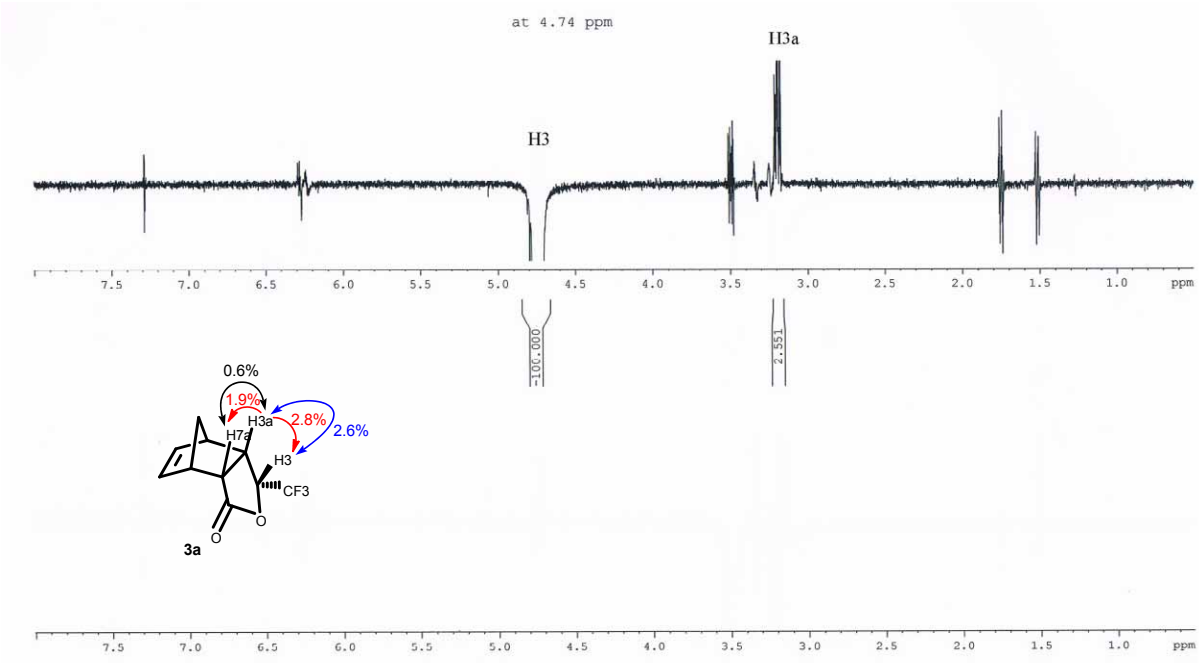
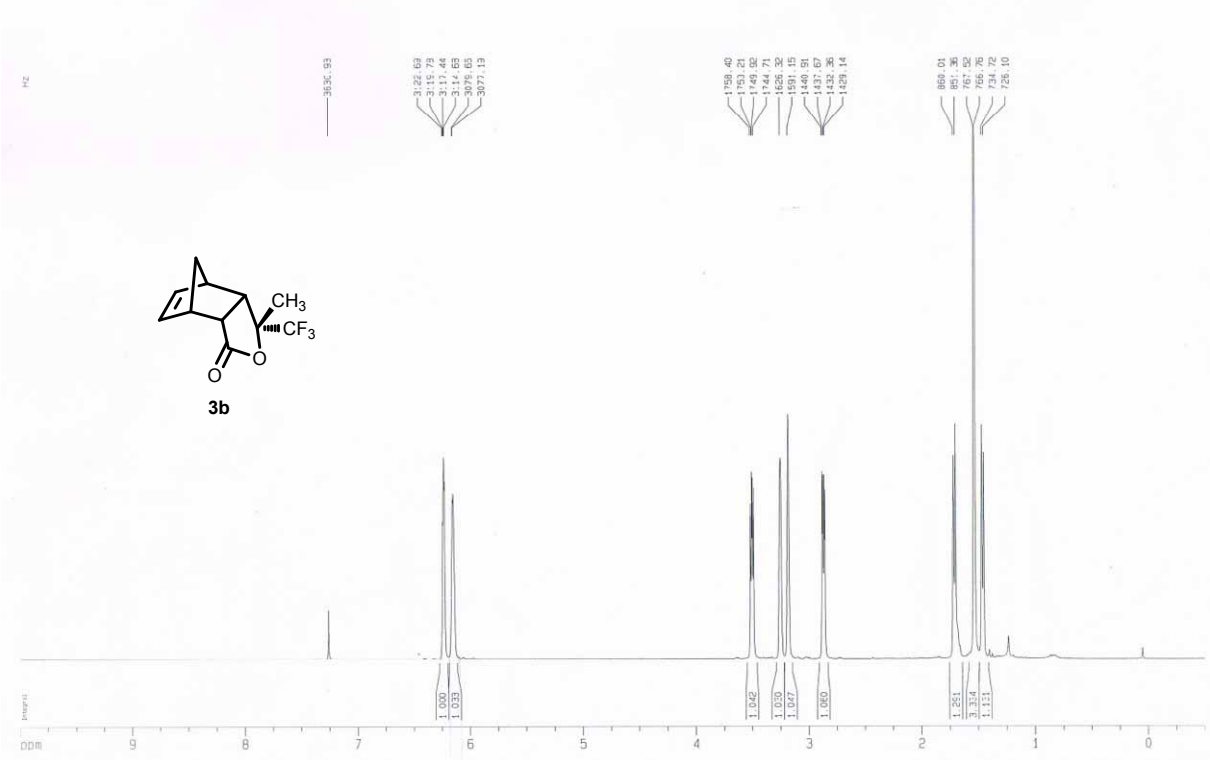
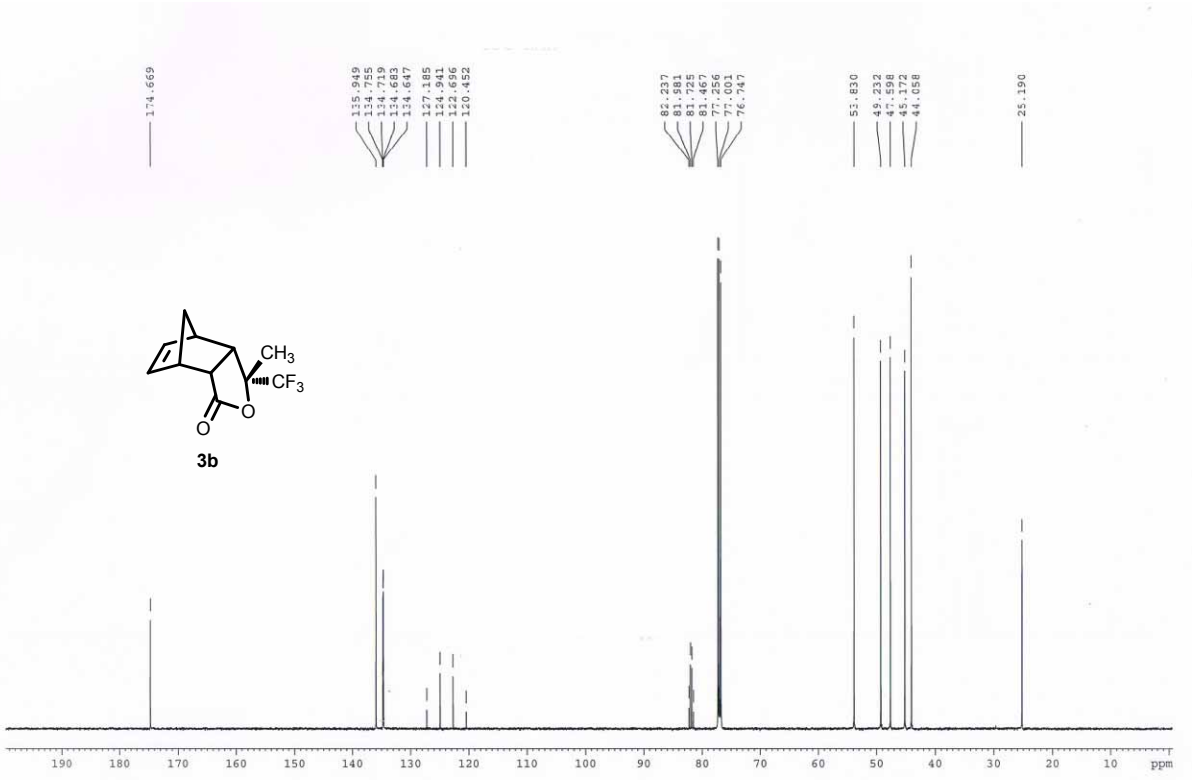


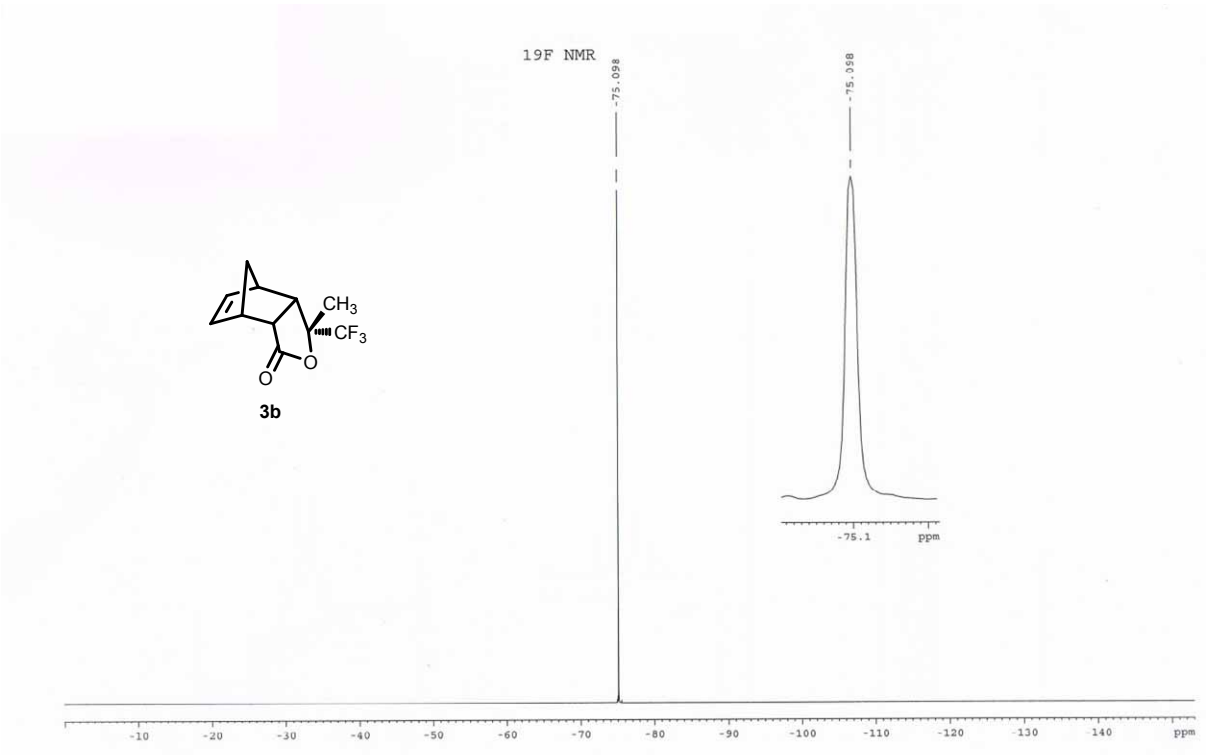
^{1}H NMR Spectrum of **3b** (500 MHz, $CDCl_3$)



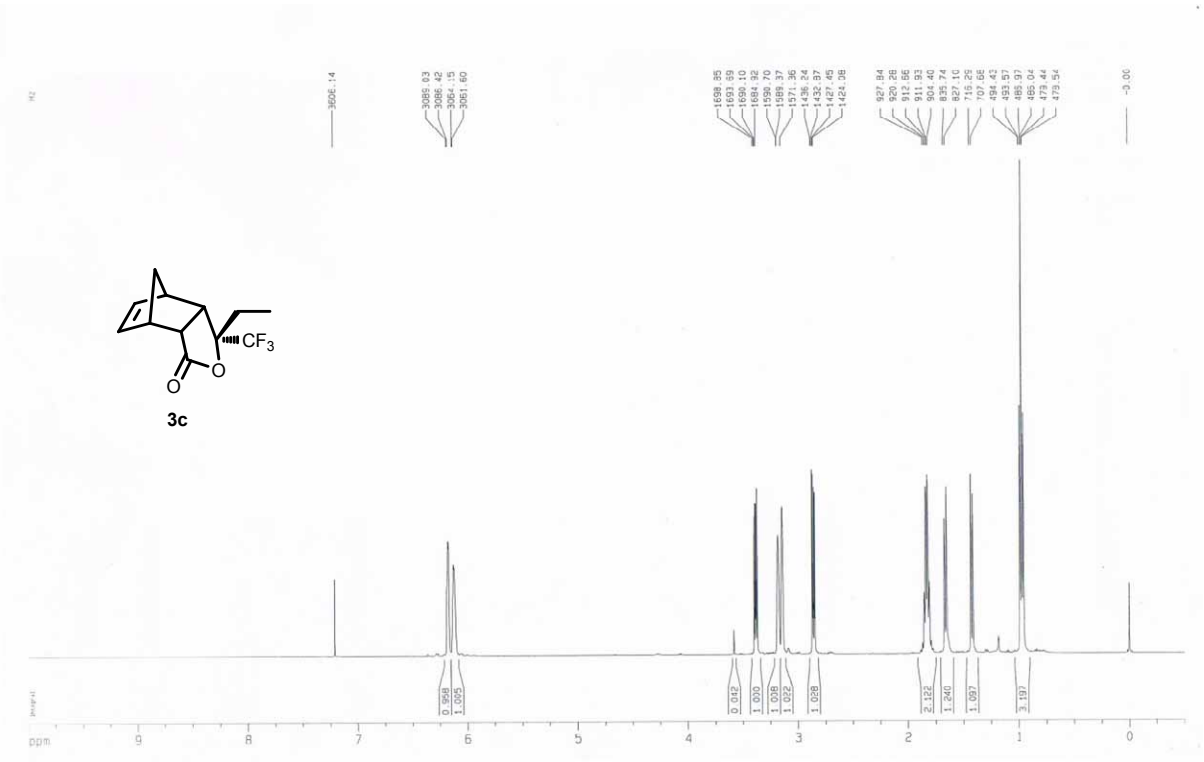
^{13}C NMR Spectrum of **3b** (125 MHz, $CDCl_3$)



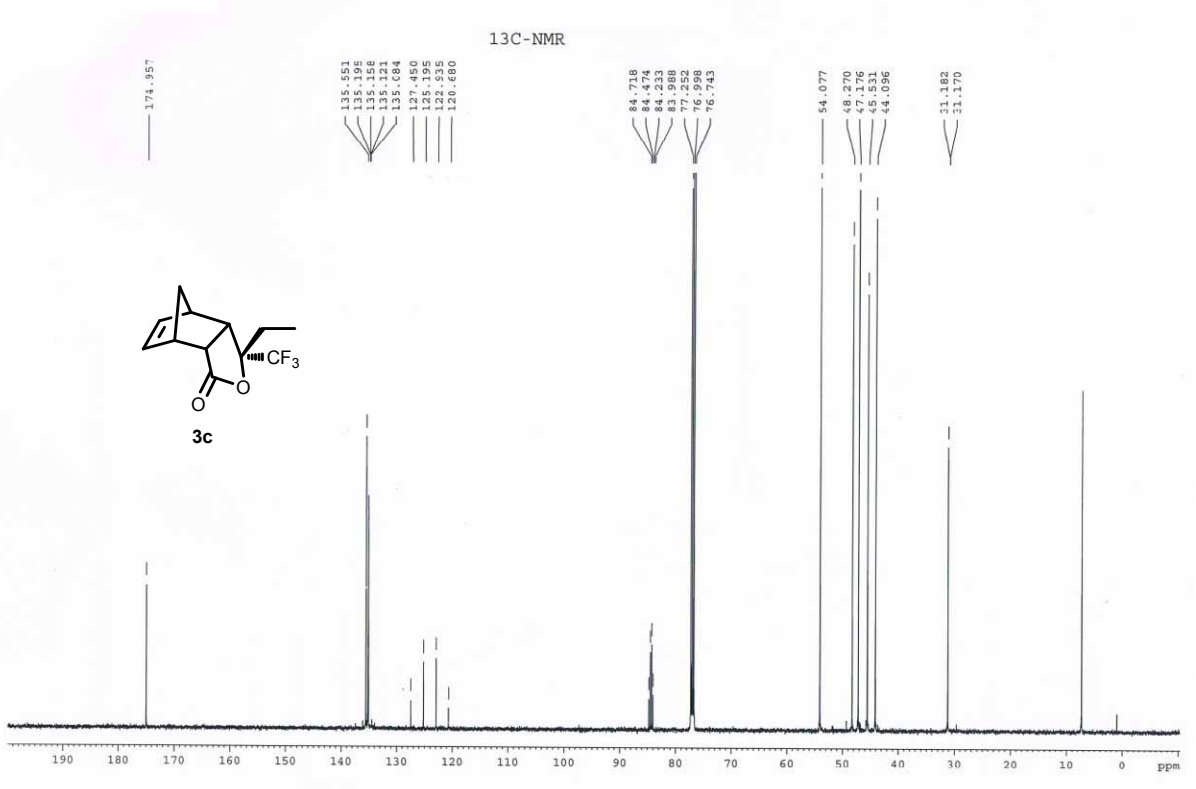
^{19}F NMR Spectrum of **3b** (470 MHz, $CDCl_3$)



^{1}H NMR Spectrum of **3c** (500 MHz, $CDCl_3$)



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



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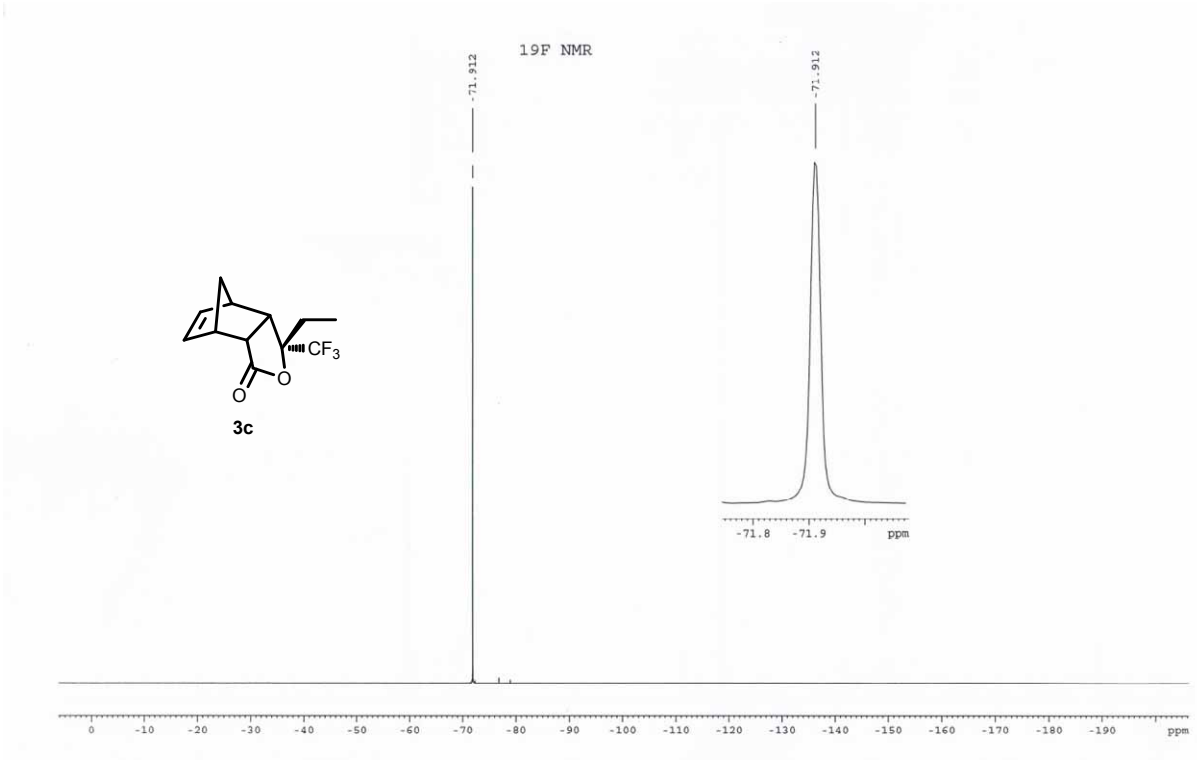

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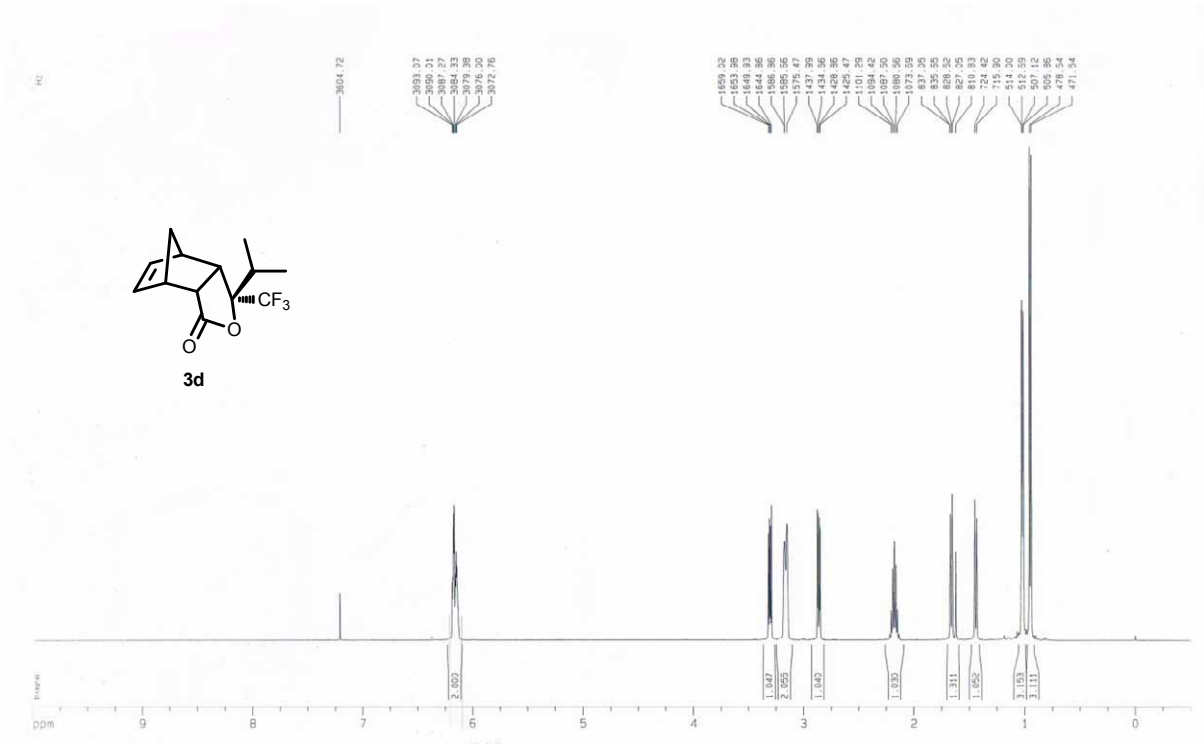

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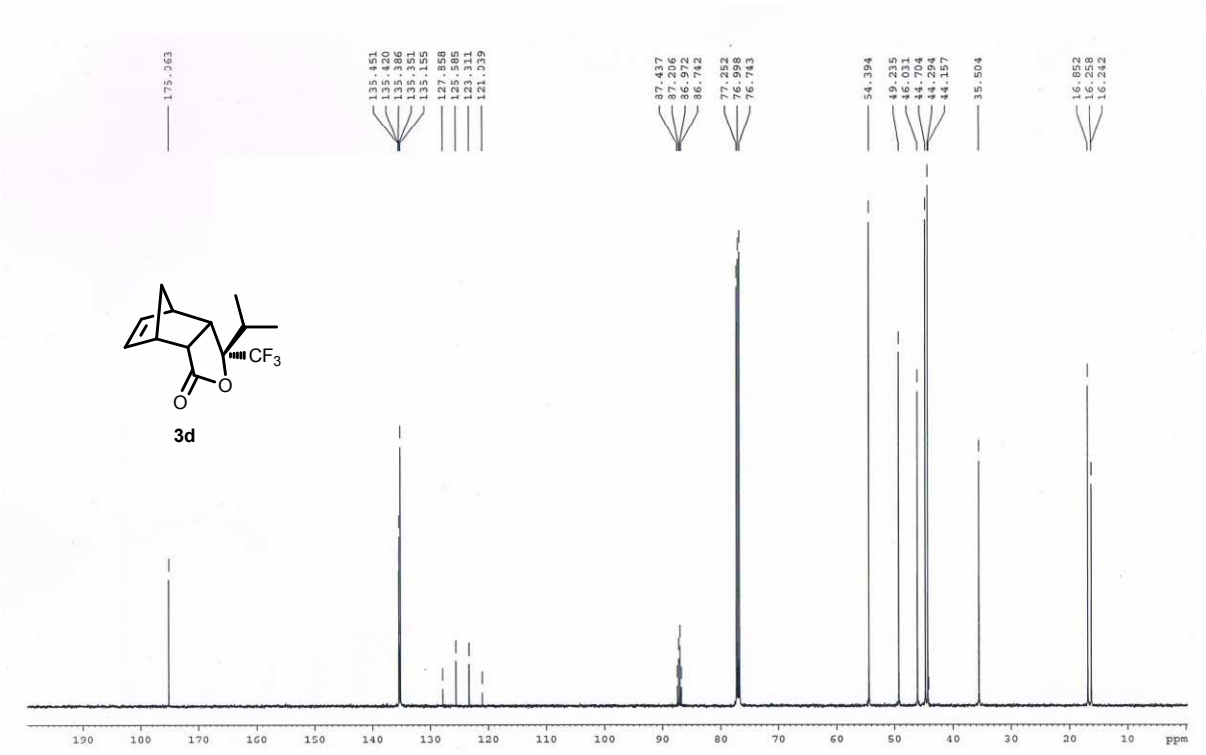

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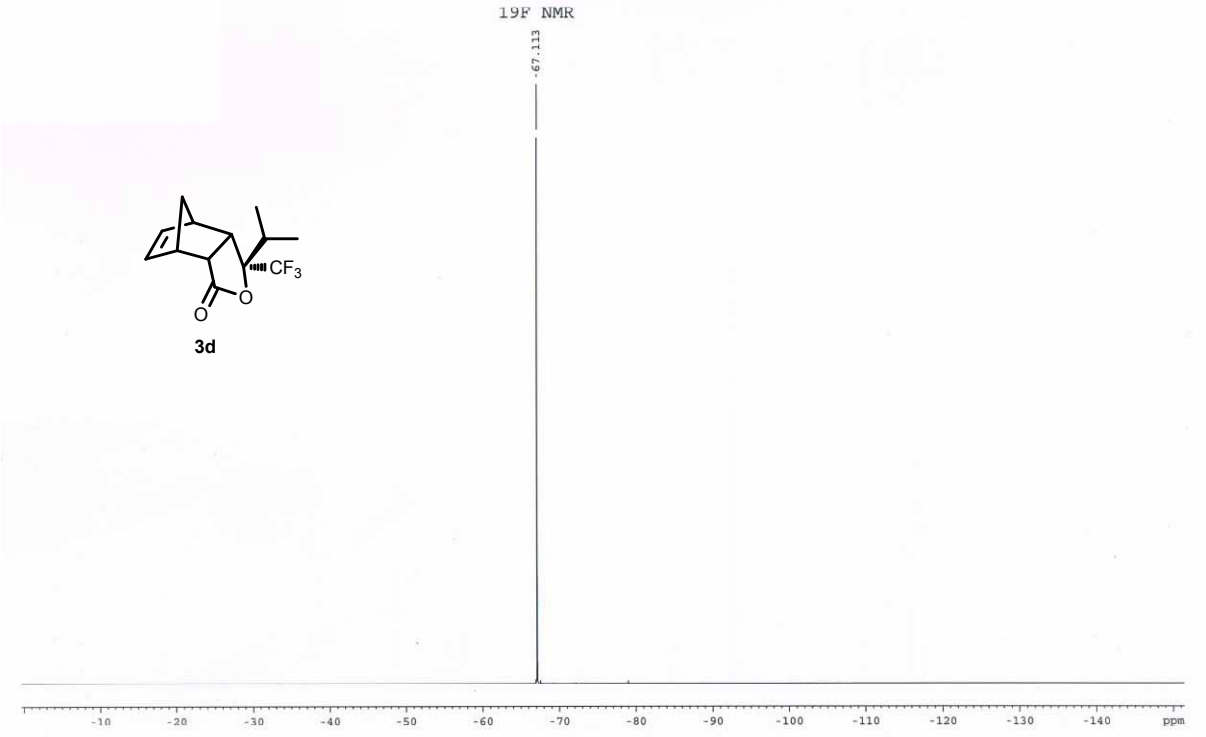

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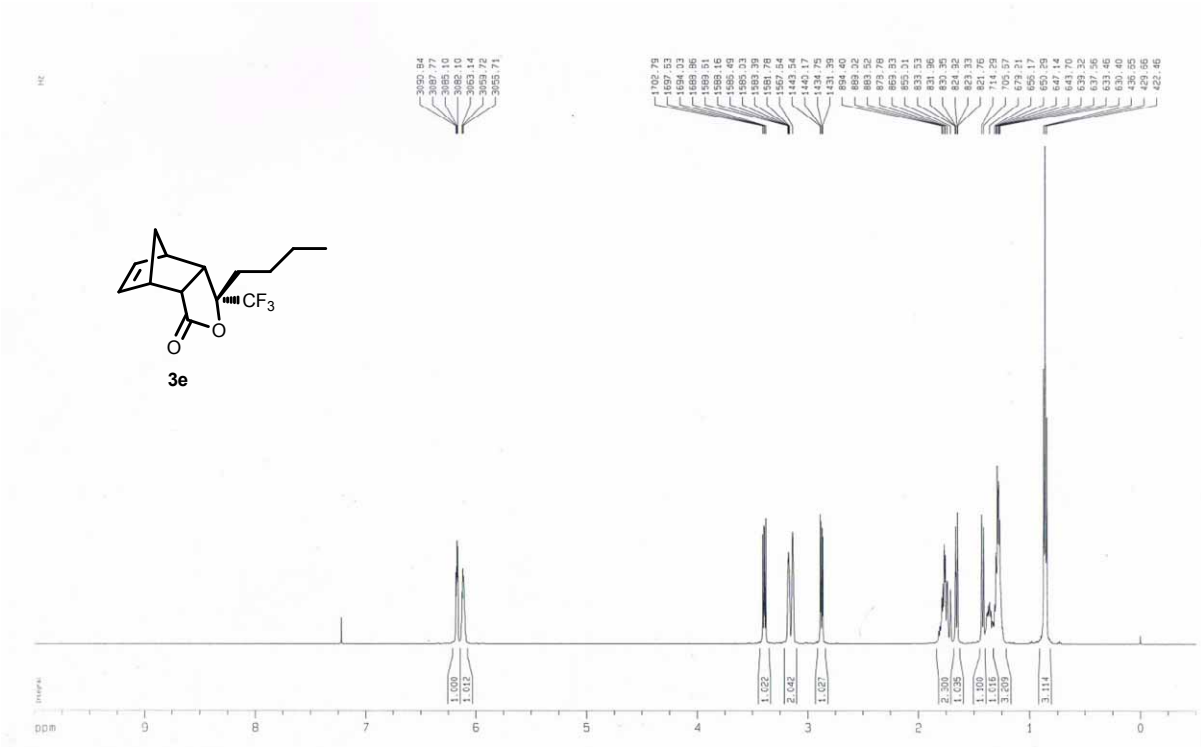

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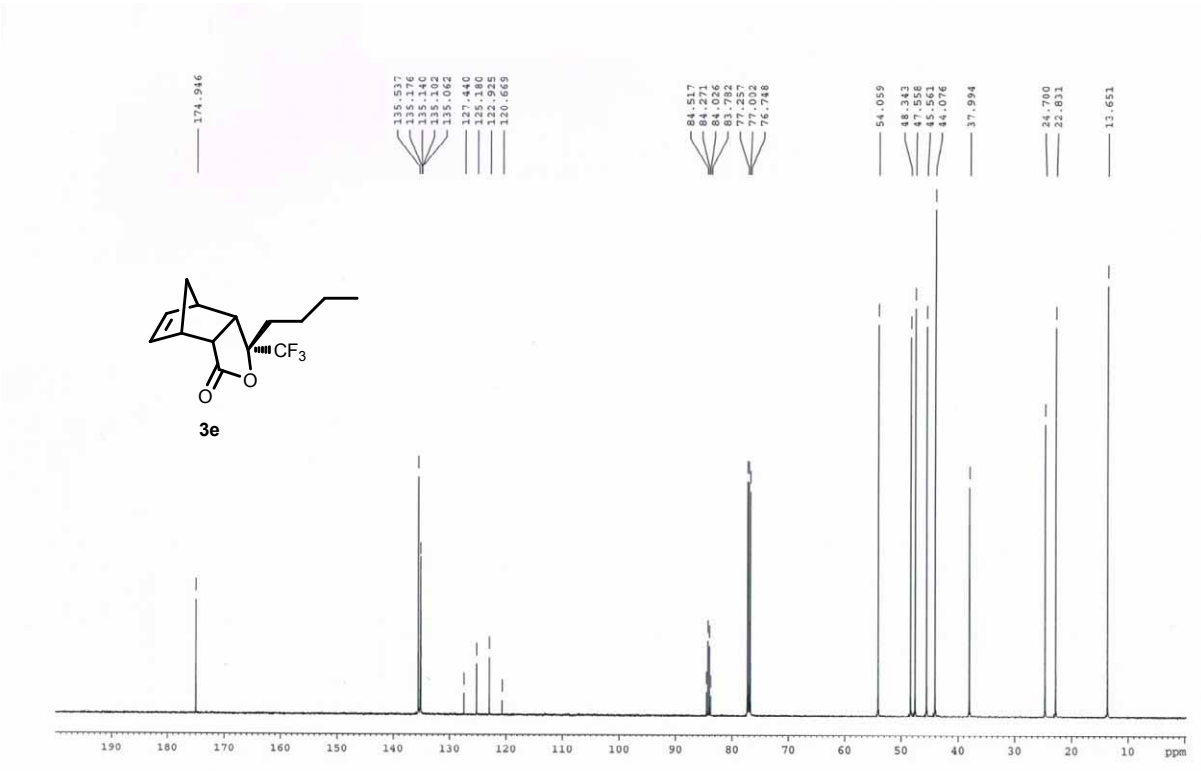
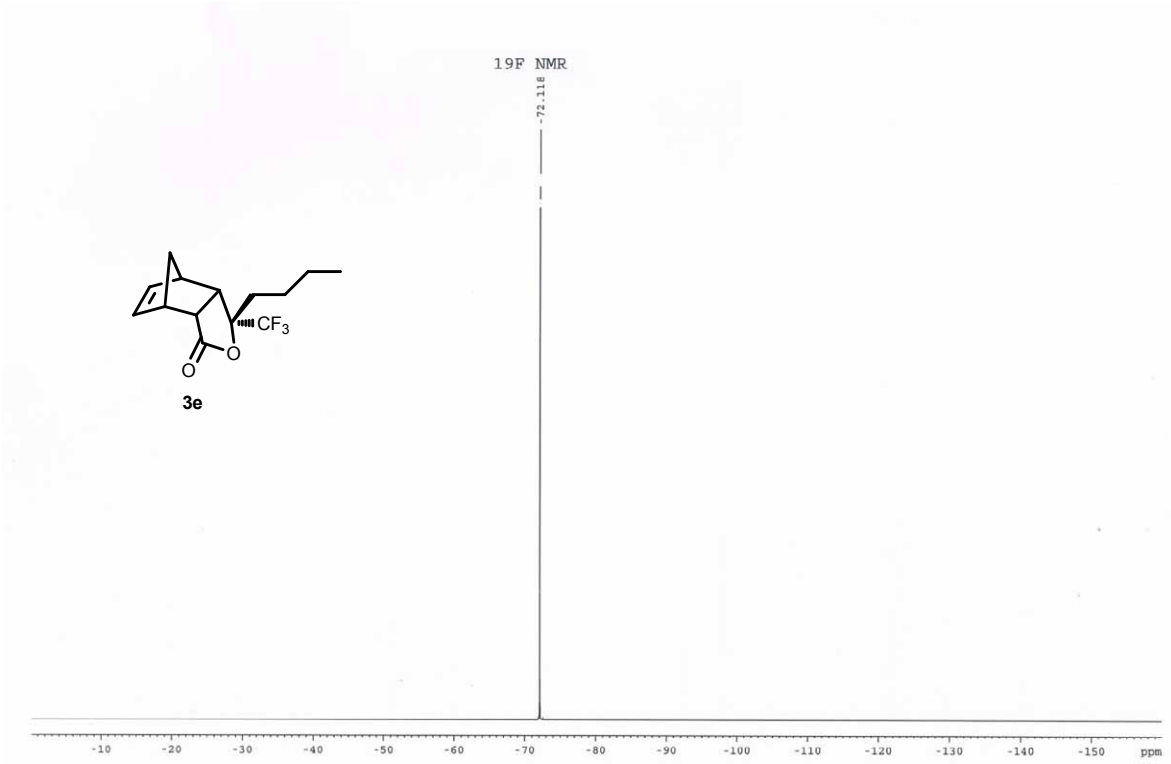


^{19}F NMR Spectrum of **3e** (470 MHz, $CDCl_3$)



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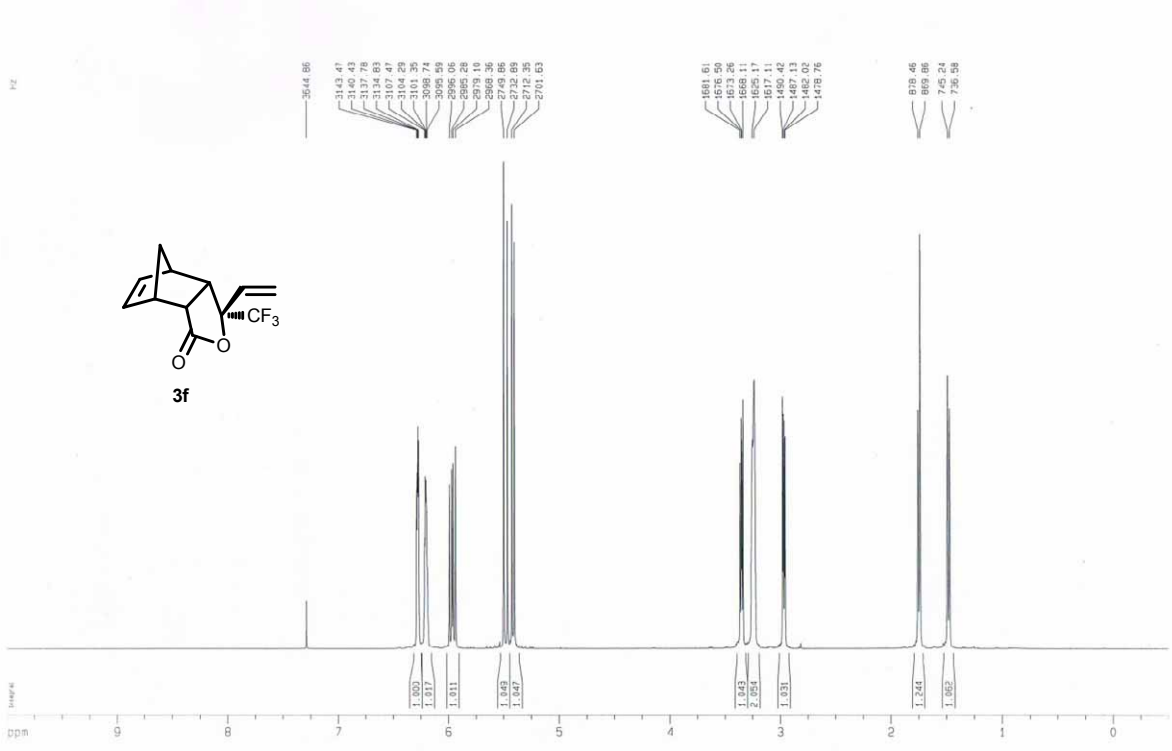

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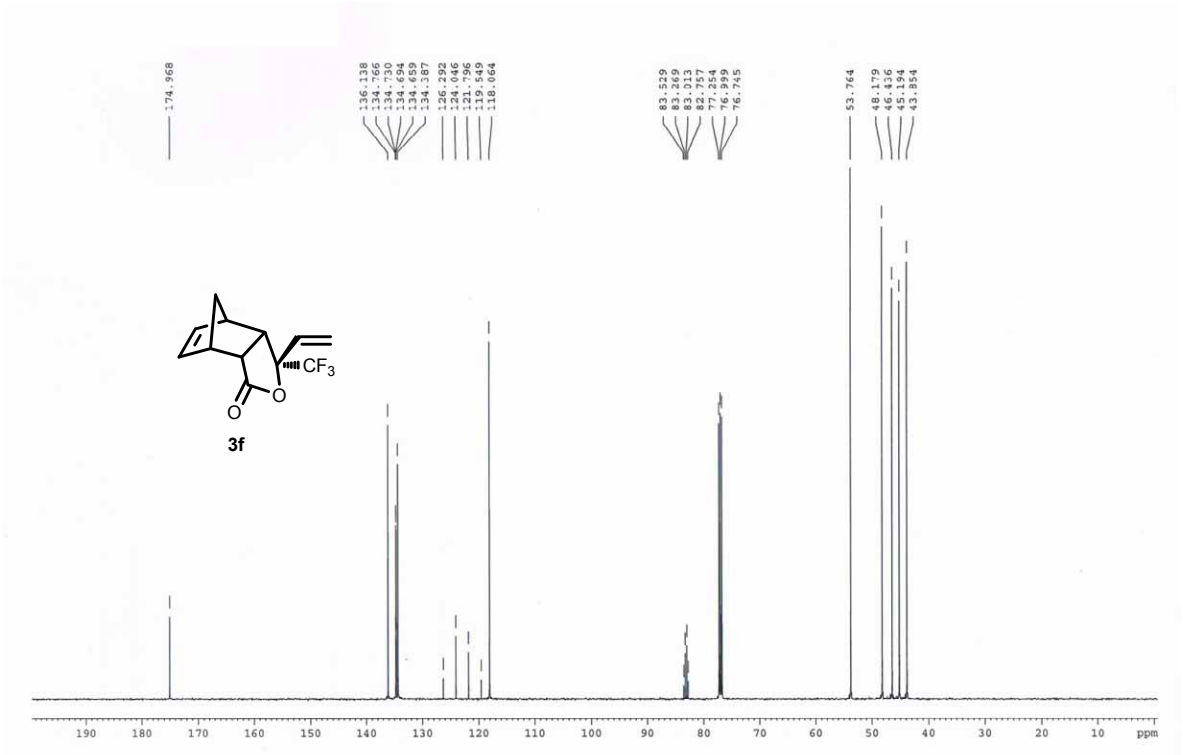

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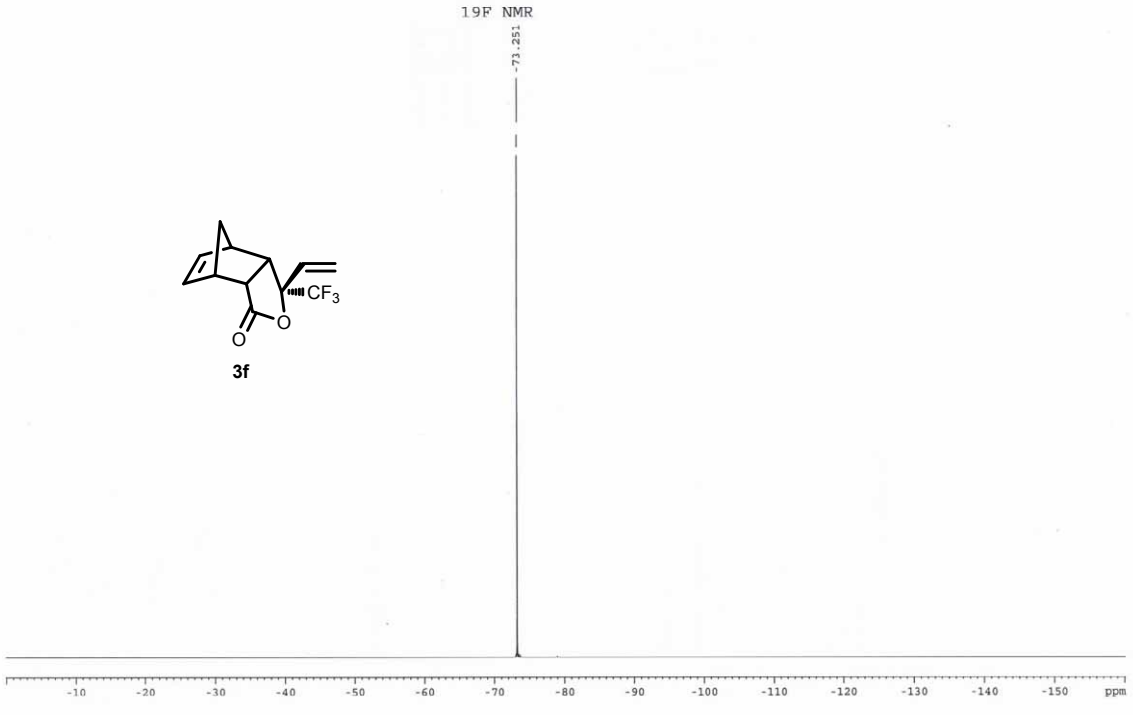
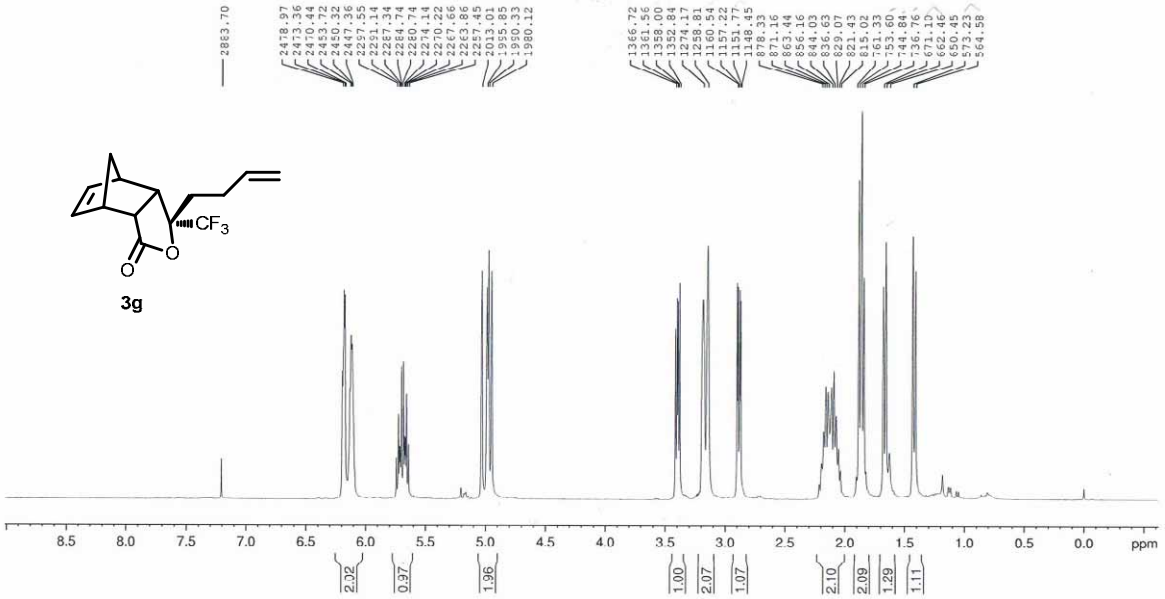
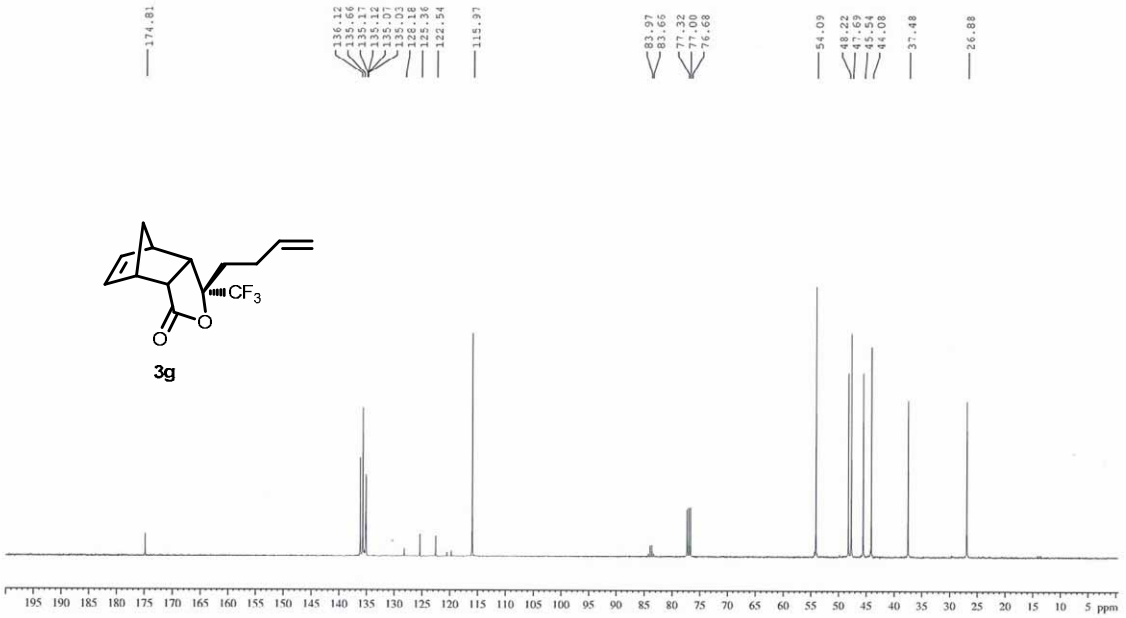


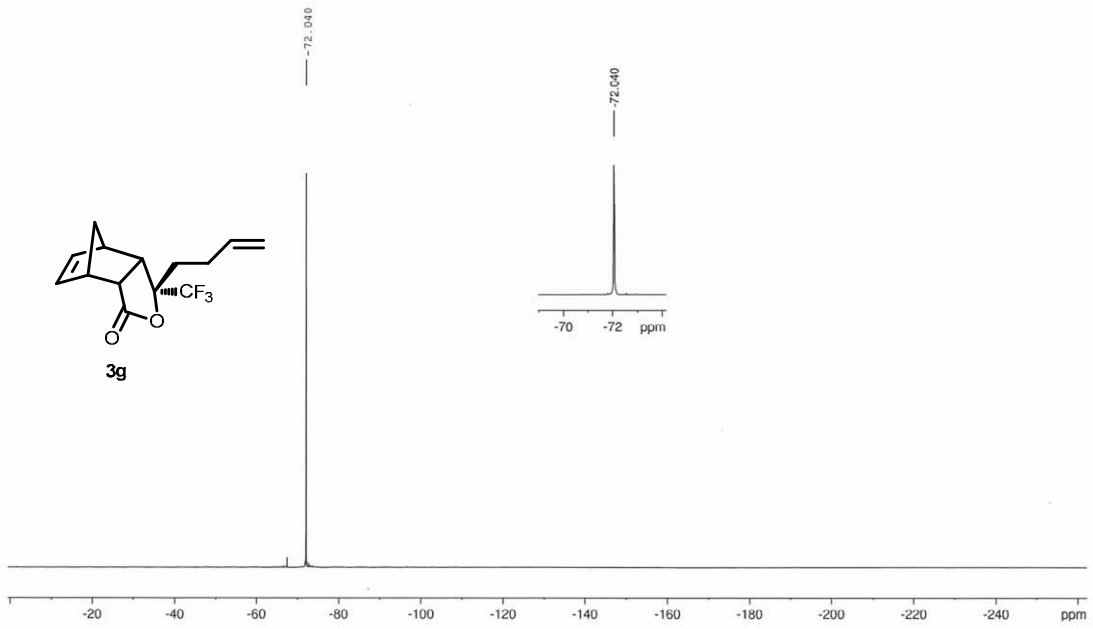
^{1}H NMR Spectrum of **3g** (400 MHz, $CDCl_3$)



^{13}C NMR Spectrum of **3g** (100 MHz, $CDCl_3$)



^{19}F NMR Spectrum of **3g** (376 MHz, $CDCl_3$)



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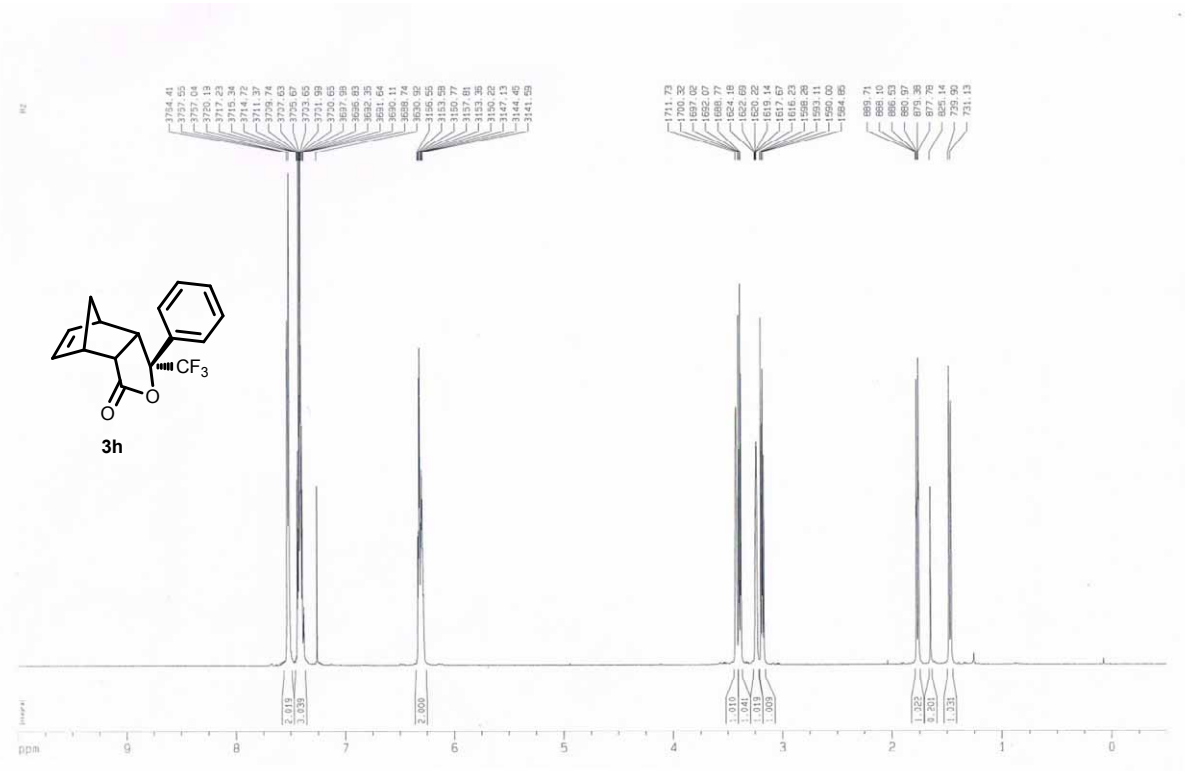
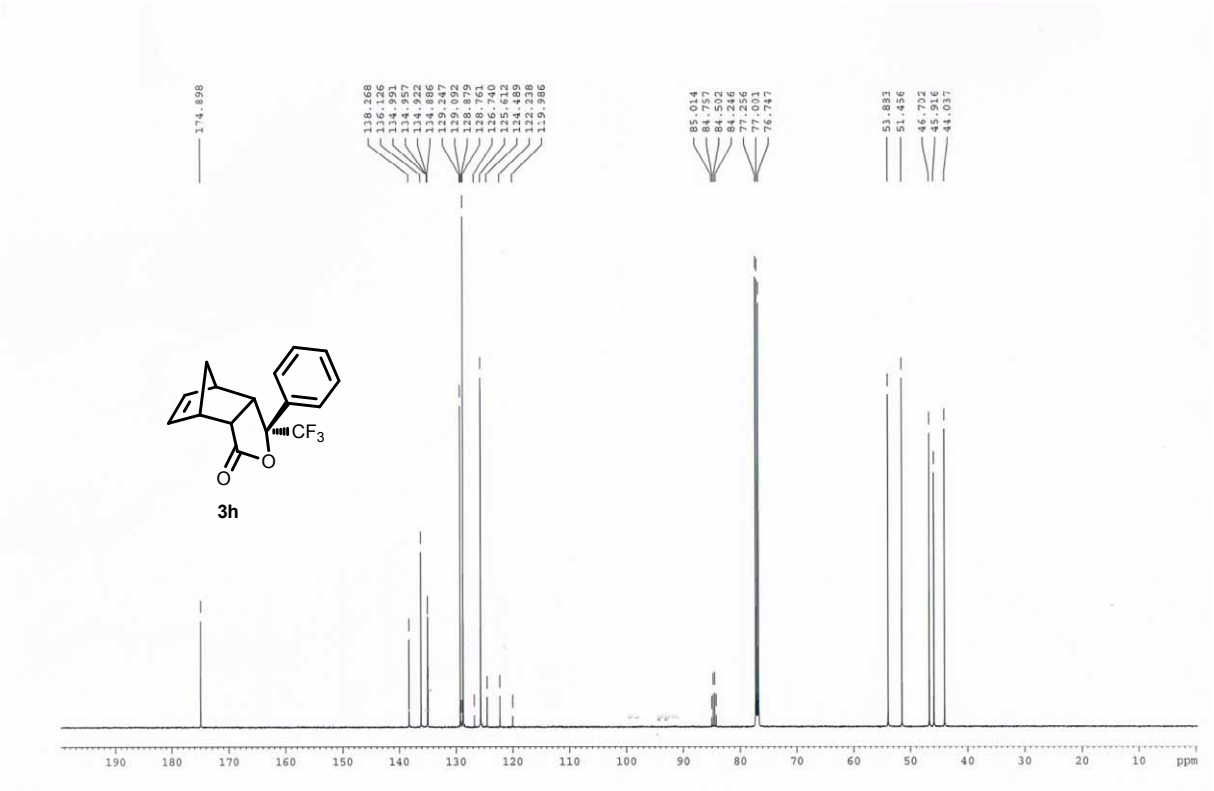
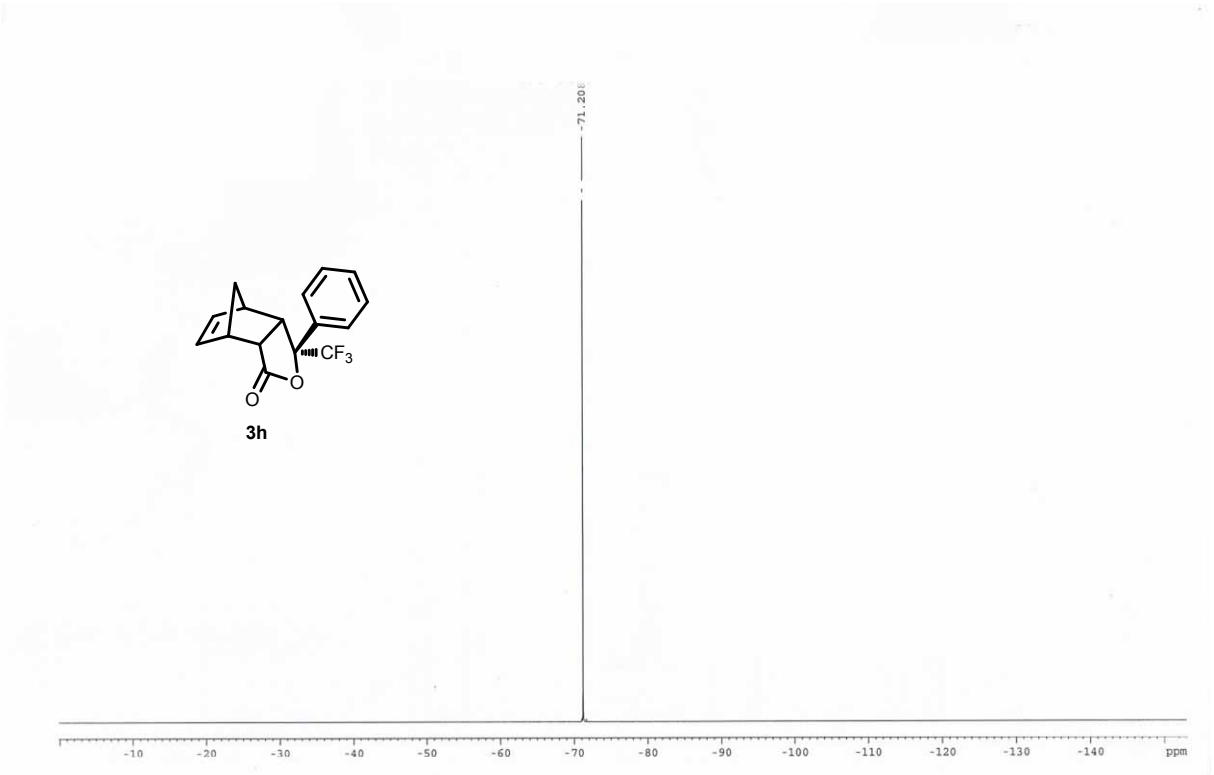


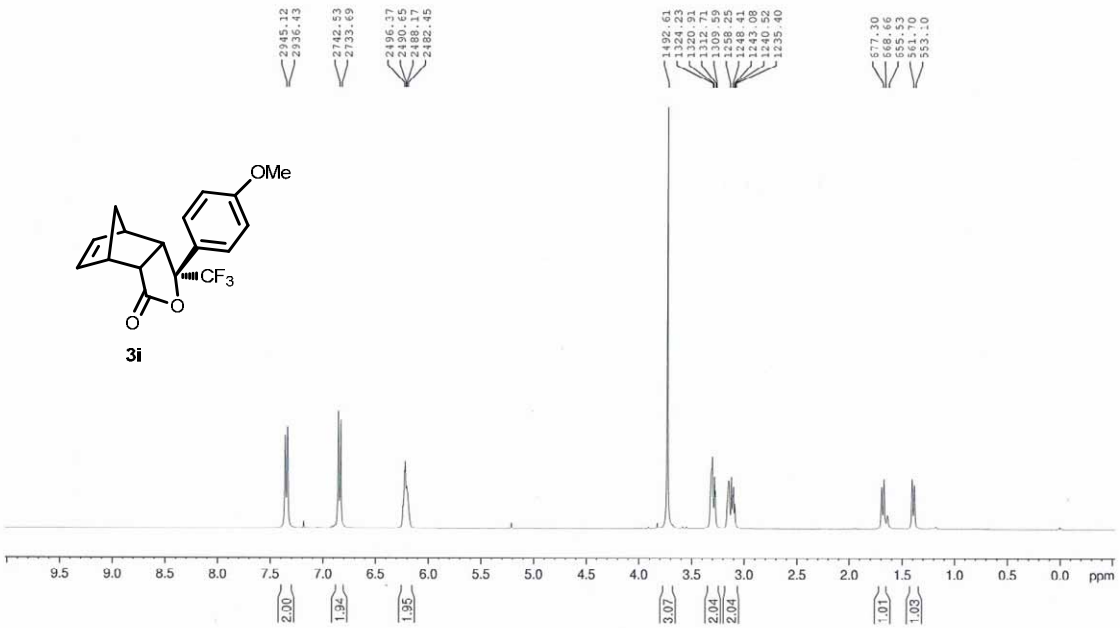
^{13}C NMR Spectrum of **3h** (125 MHz, $CDCl_3$)



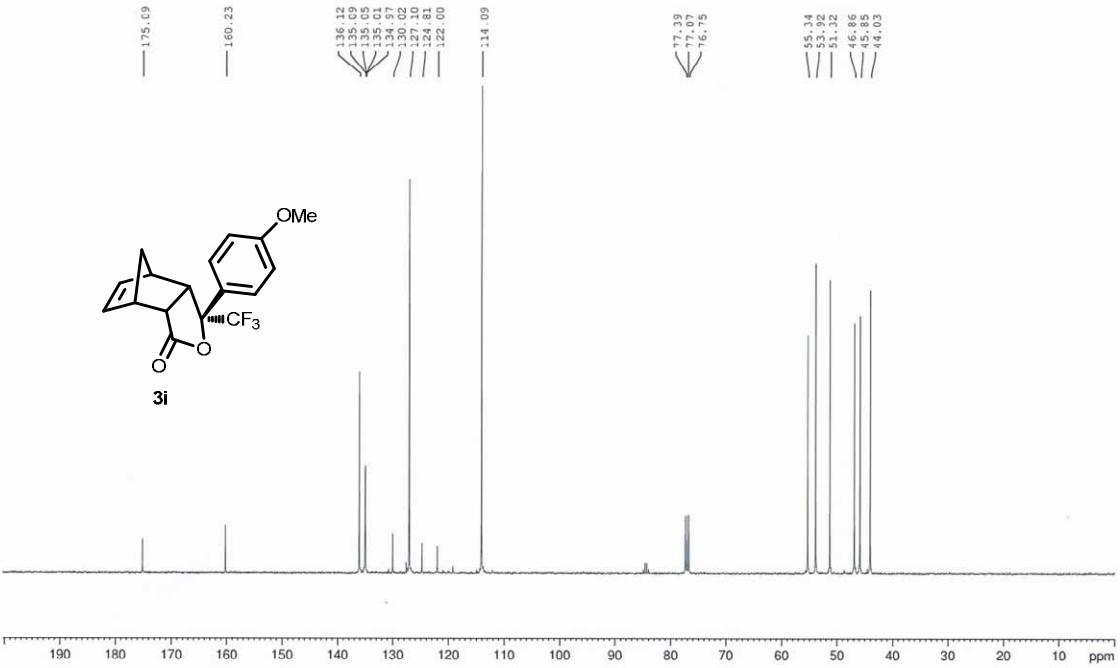
^{19}F NMR Spectrum of **3h** (470 MHz, $CDCl_3$)



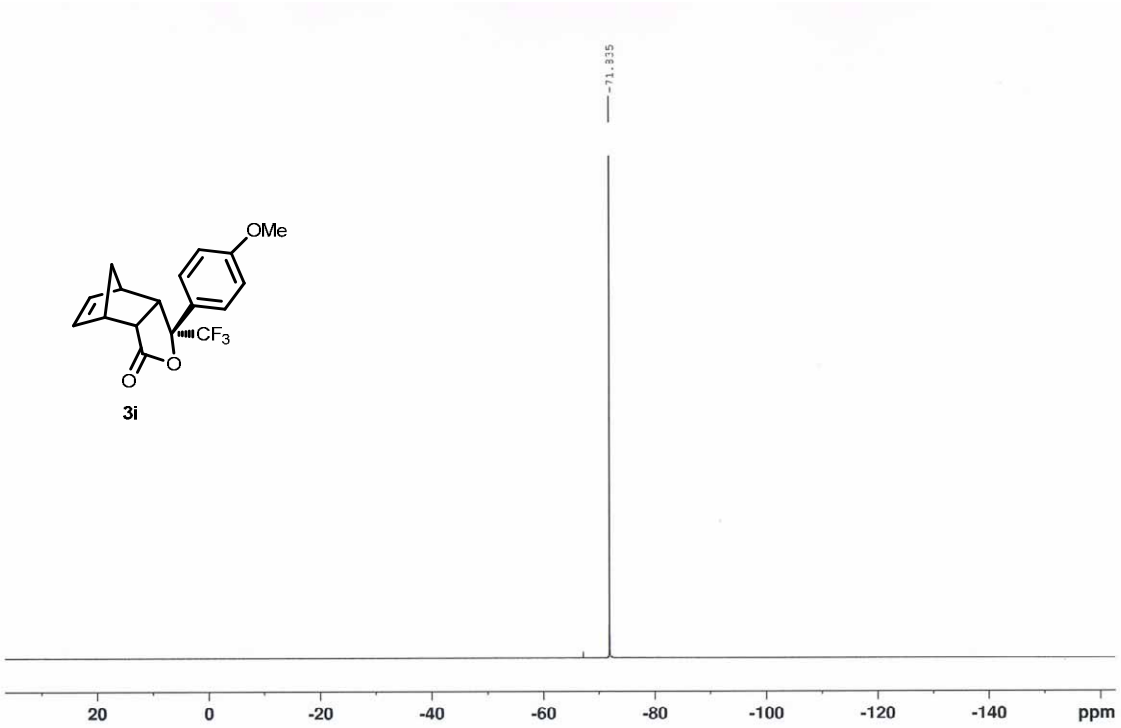
^{1}H NMR Spectrum of **3i** (400 MHz, $CDCl_3$)



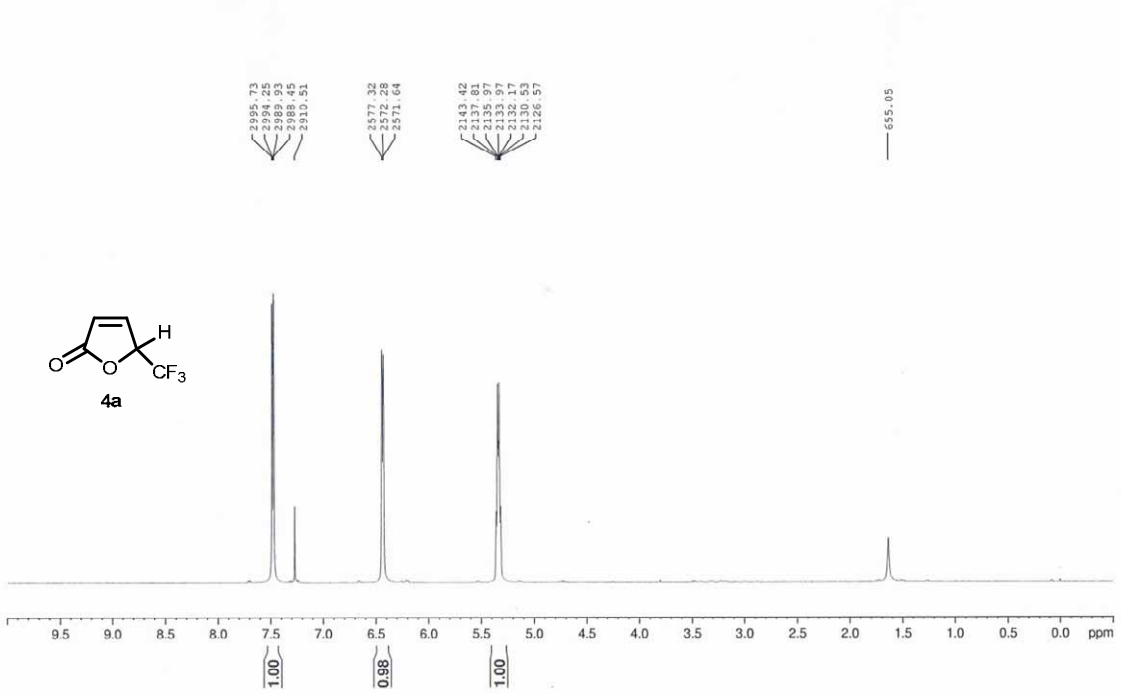
^{13}C NMR Spectrum of **3i** (100 MHz, $CDCl_3$)



^{19}F NMR Spectrum of **3i** (376 MHz, $CDCl_3$)



^{1}H NMR Spectrum of **4a** (400 MHz, $CDCl_3$)



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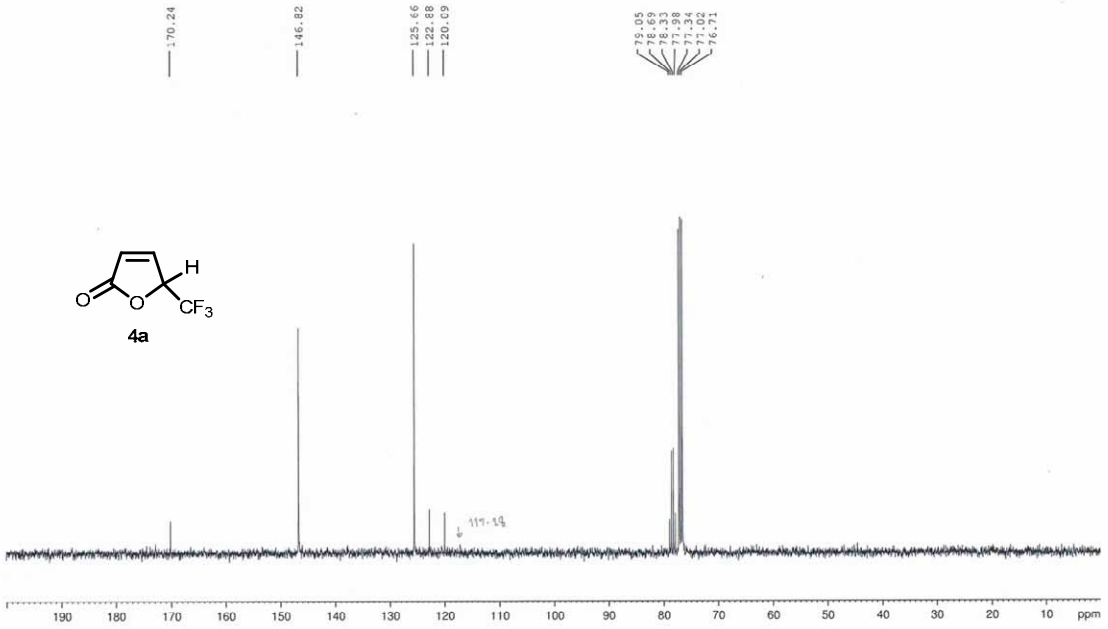

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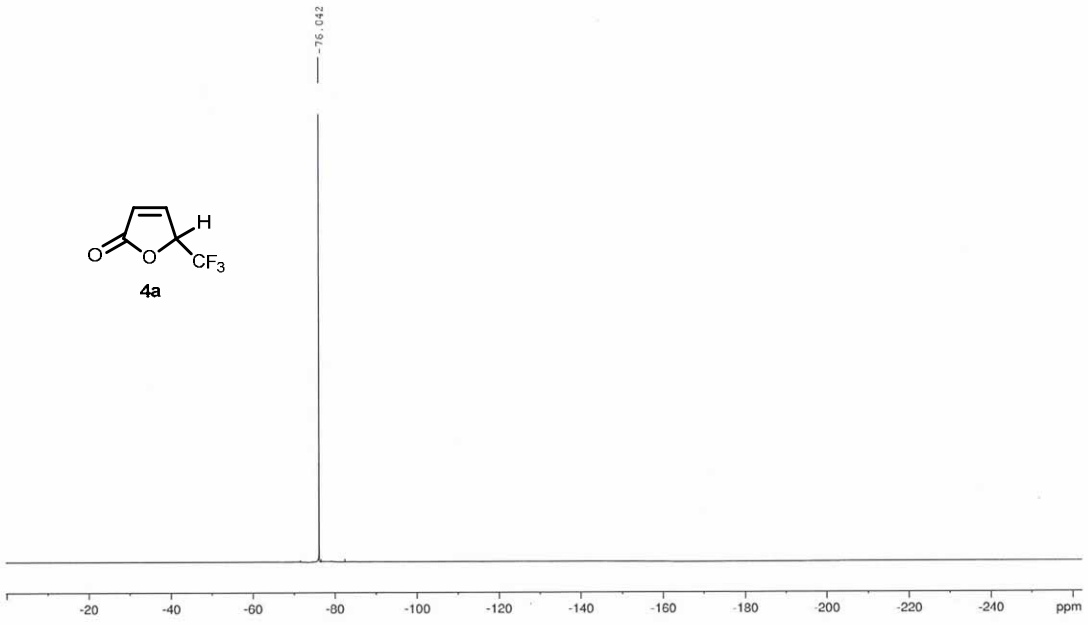

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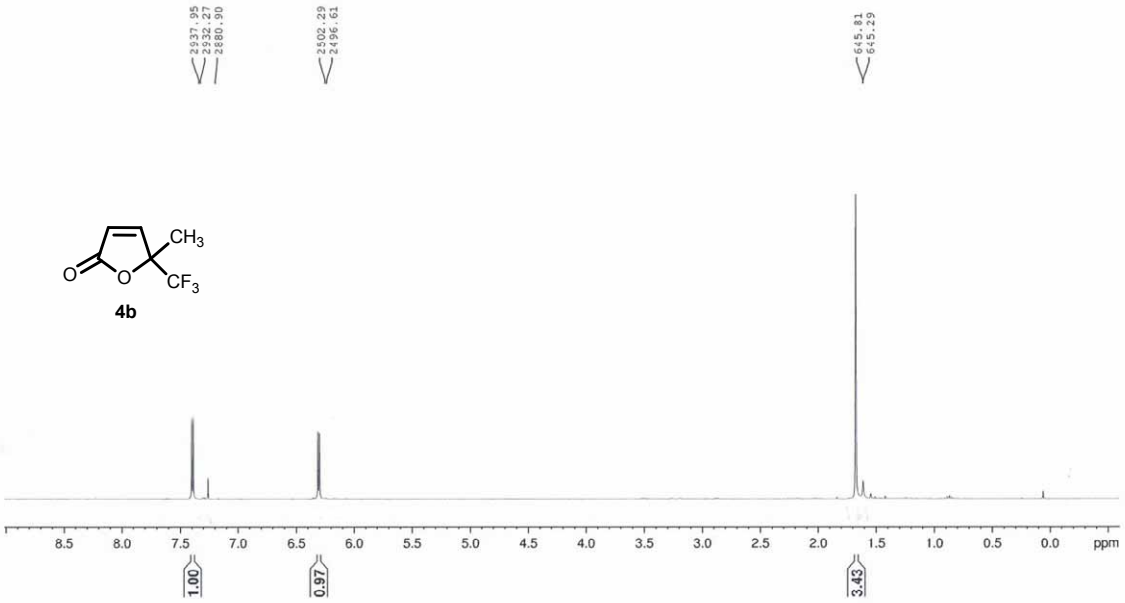

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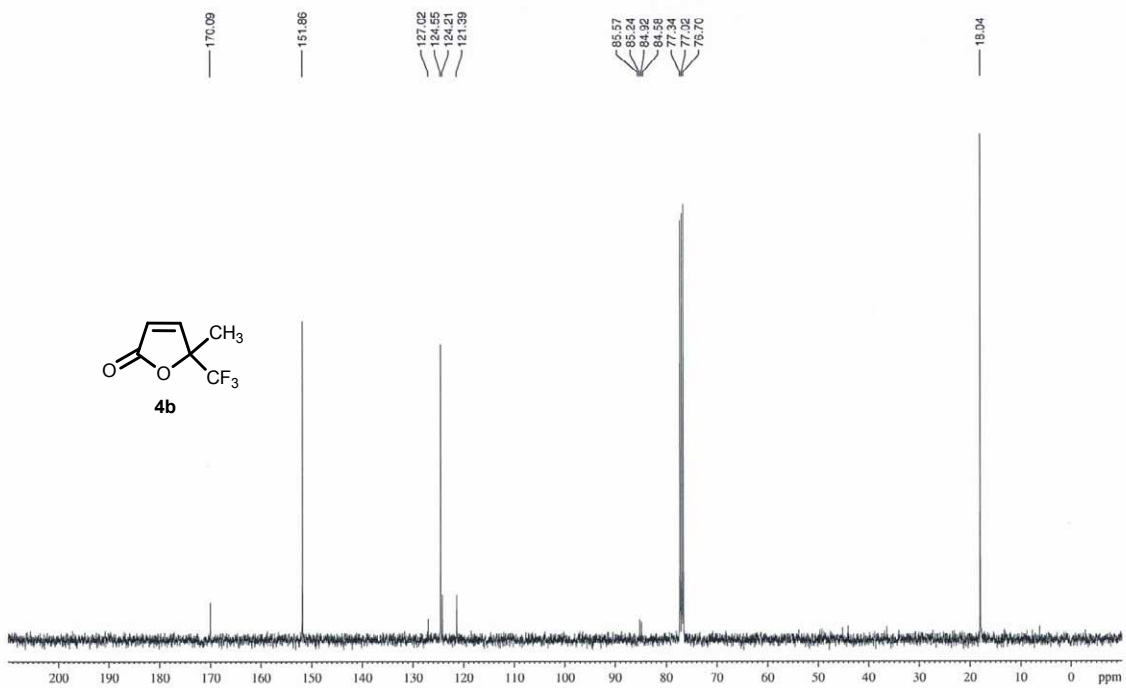
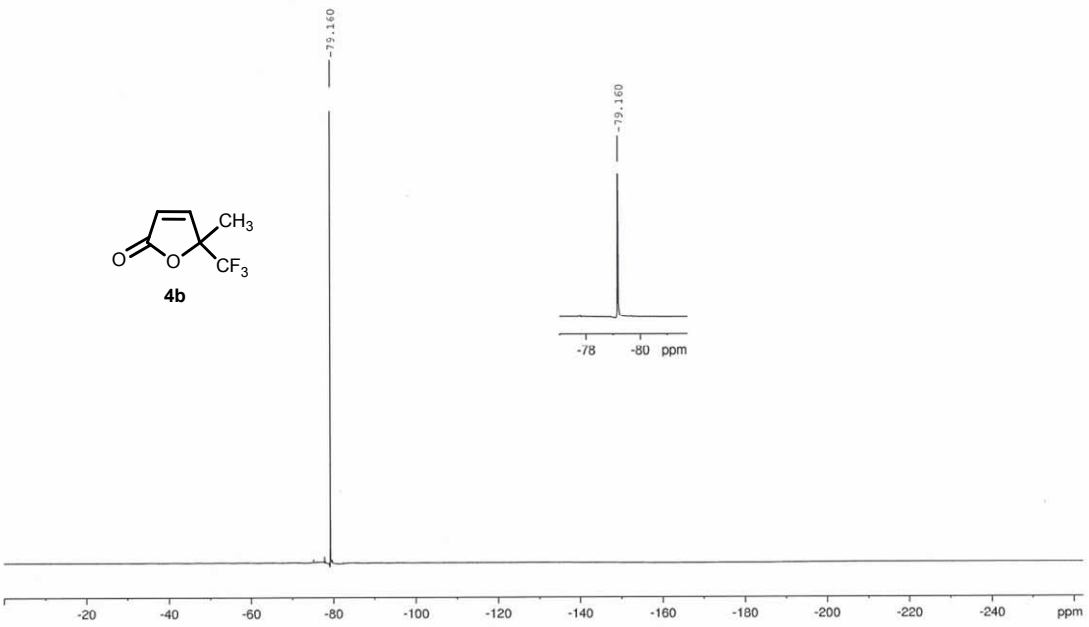


^{19}F NMR Spectrum of **4b** (376 MHz, $CDCl_3$)



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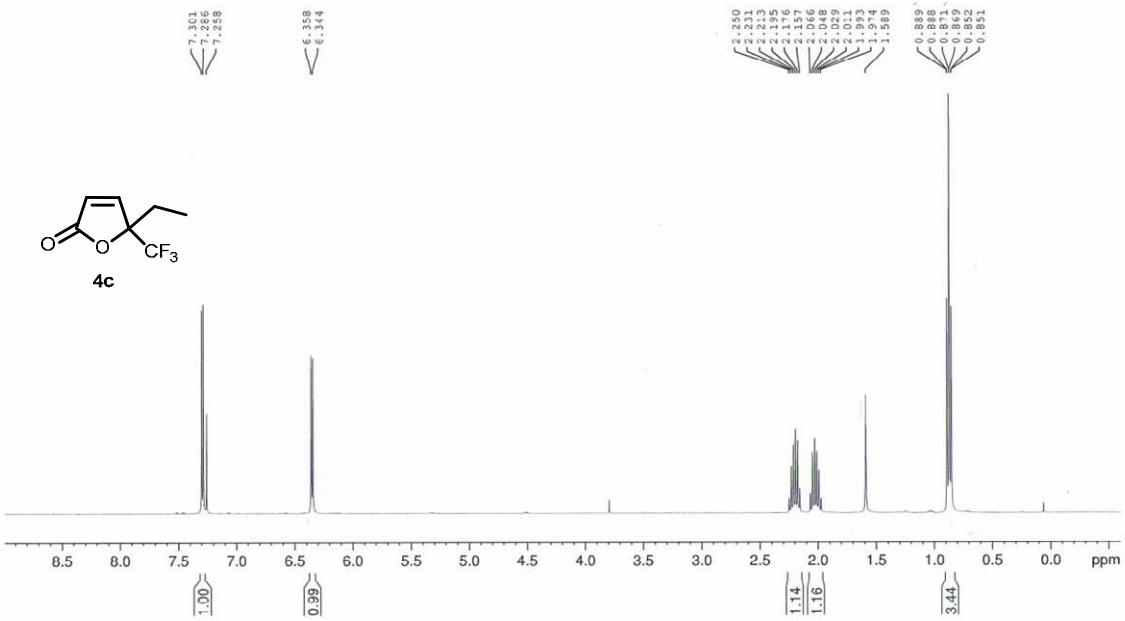
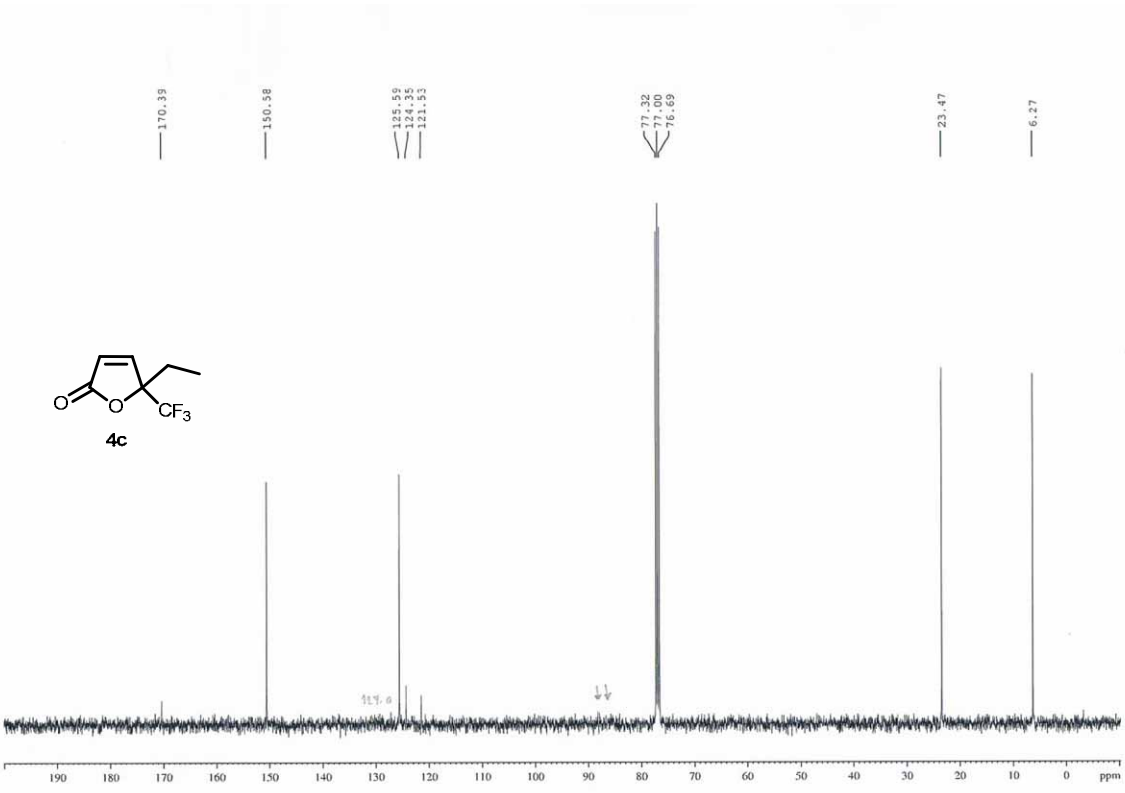


^{13}C NMR Spectrum of **4c** (100 MHz, $CDCl_3$)



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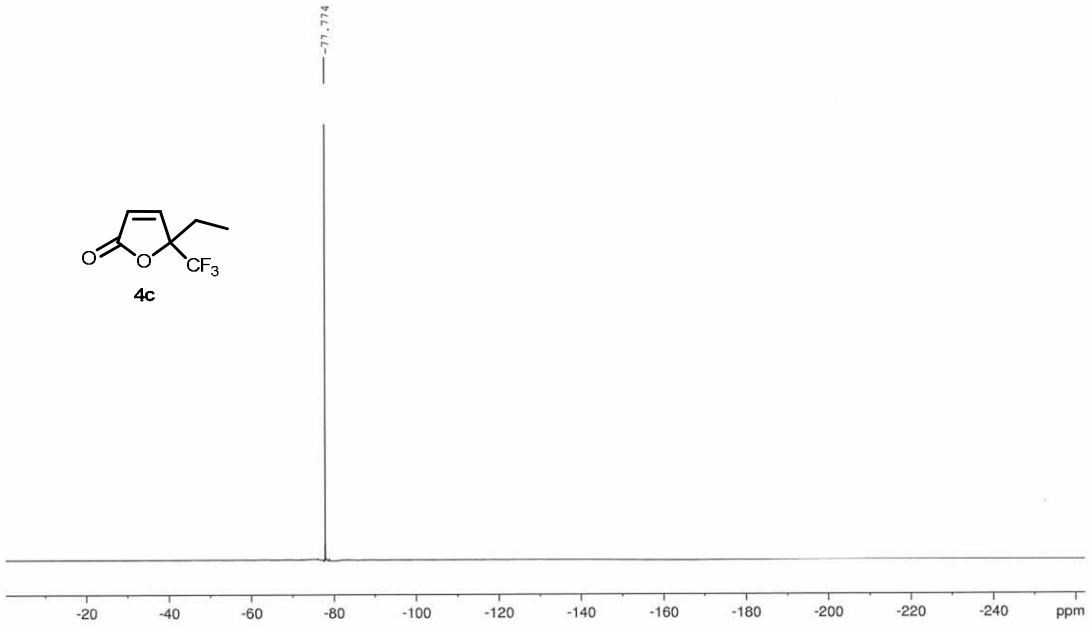

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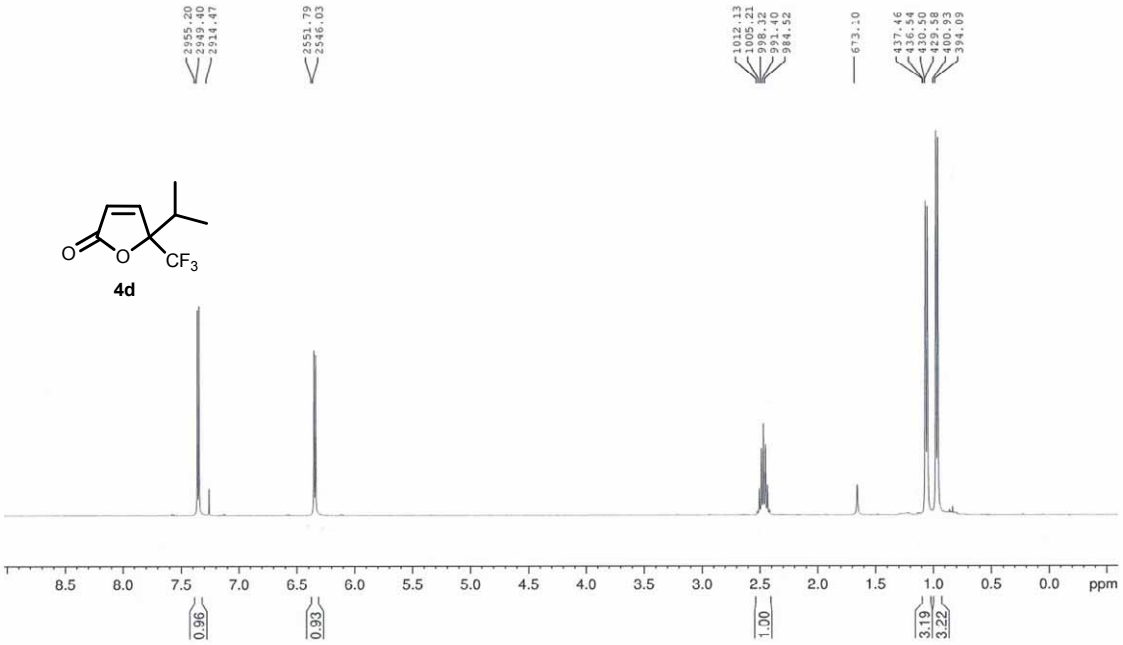

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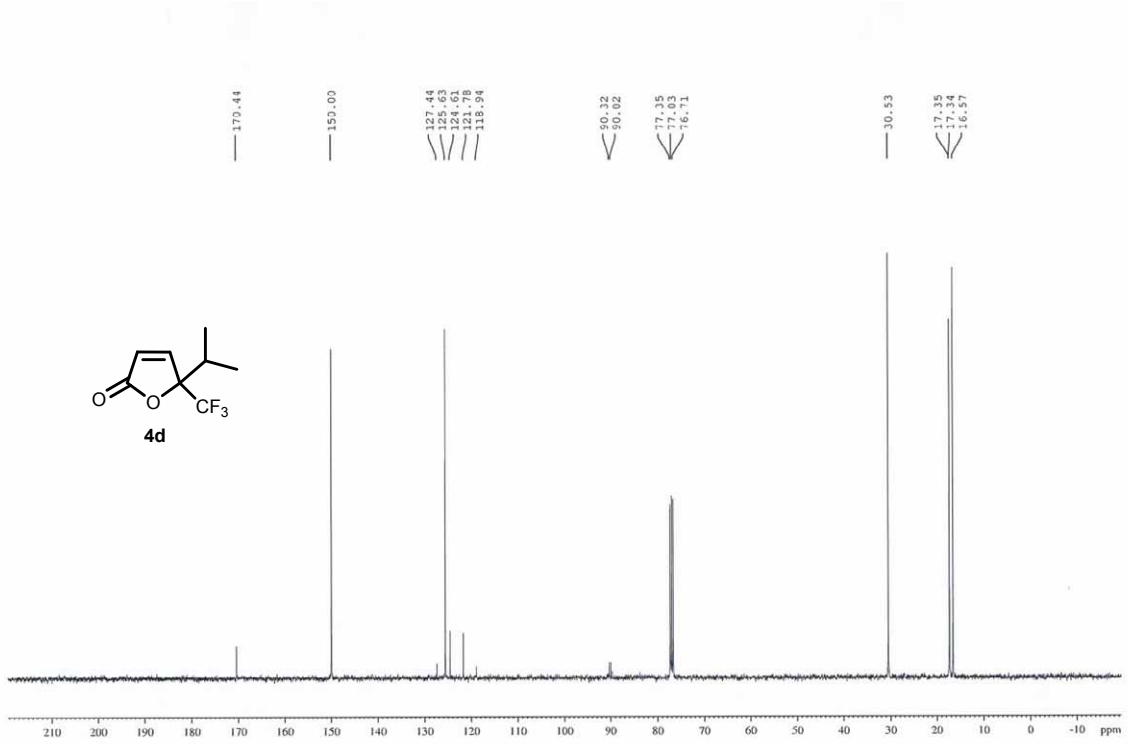

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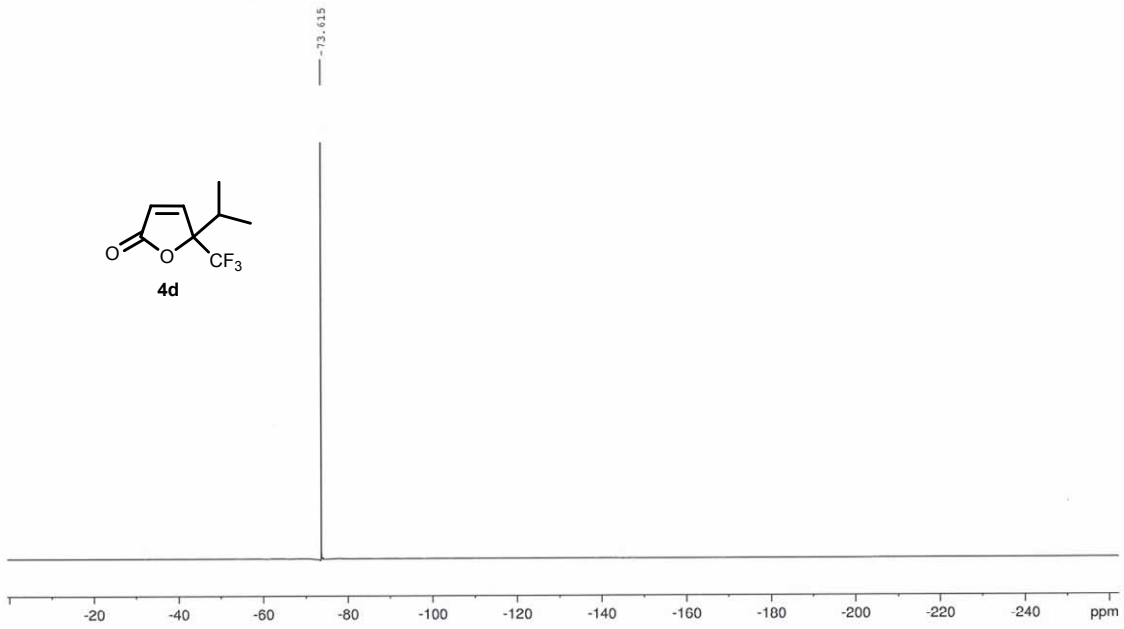

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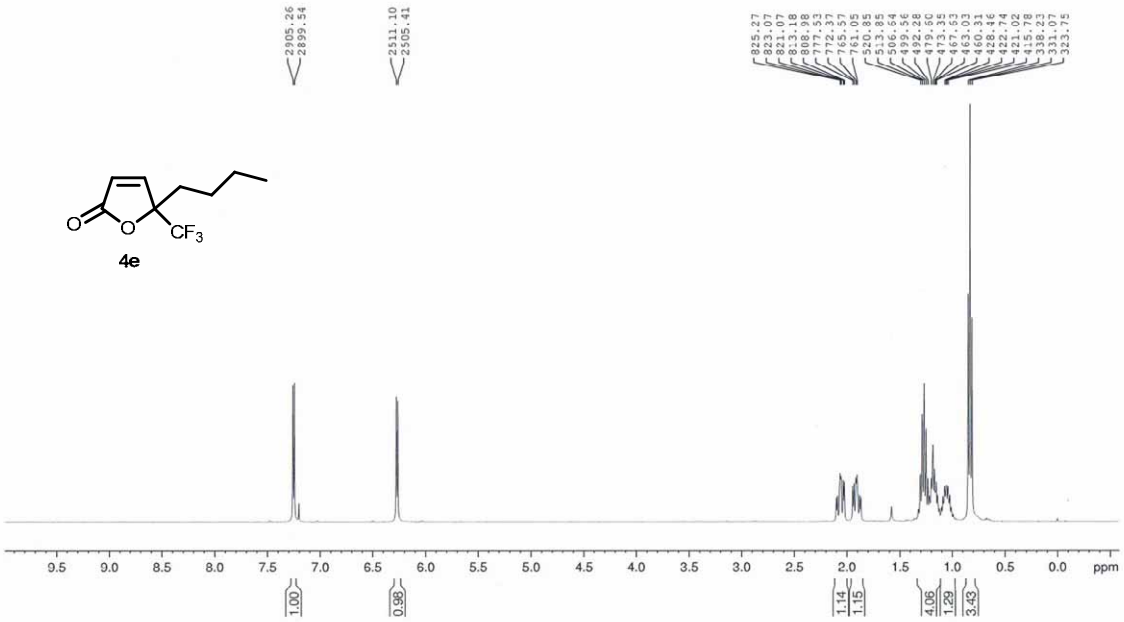

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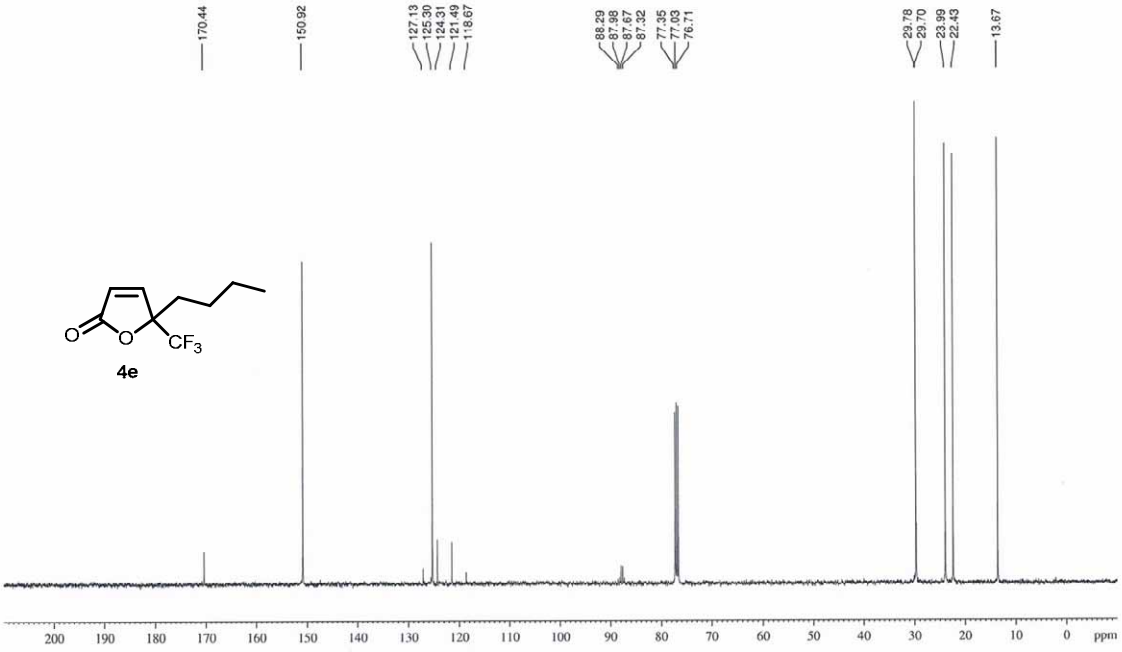

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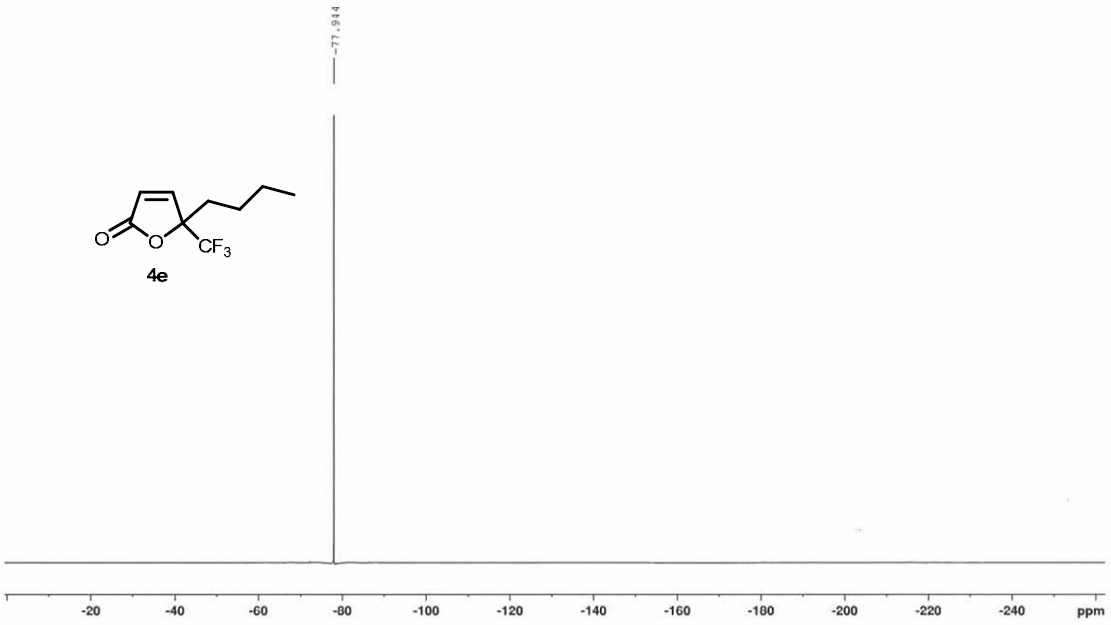

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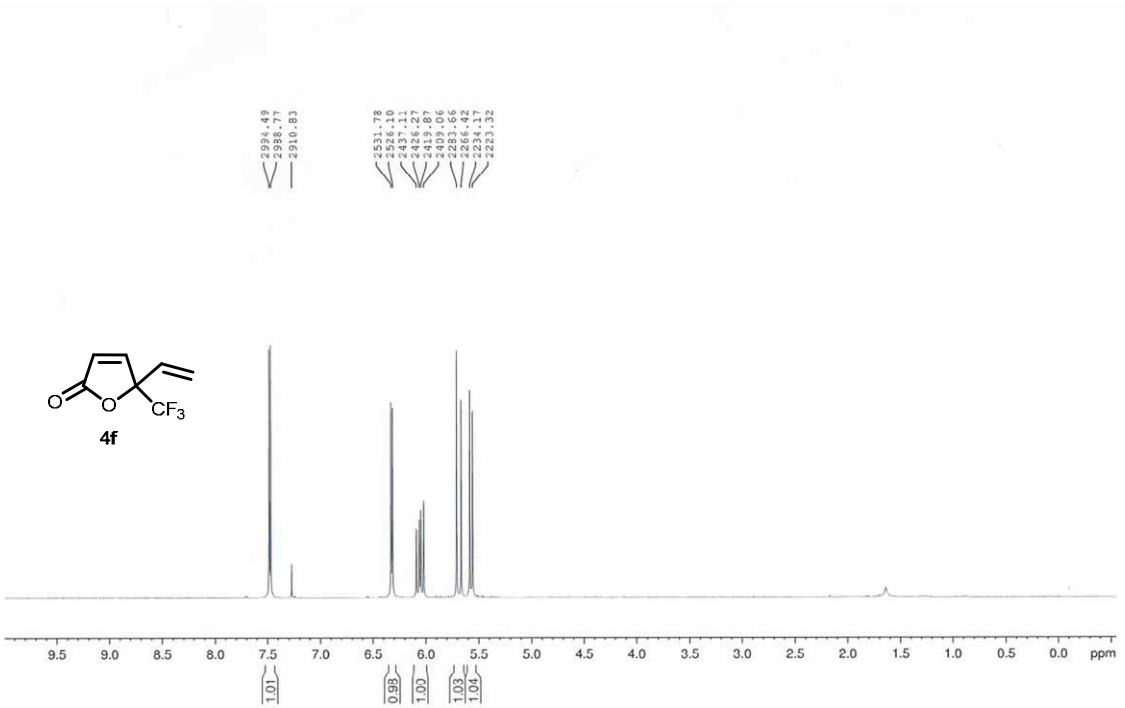

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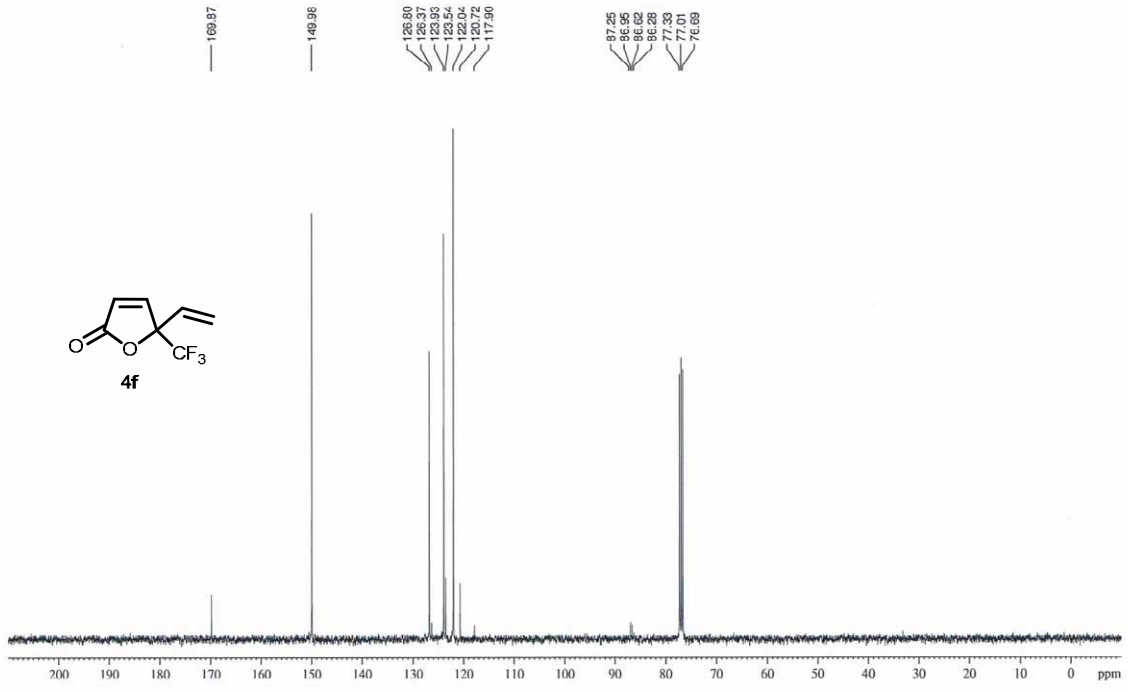
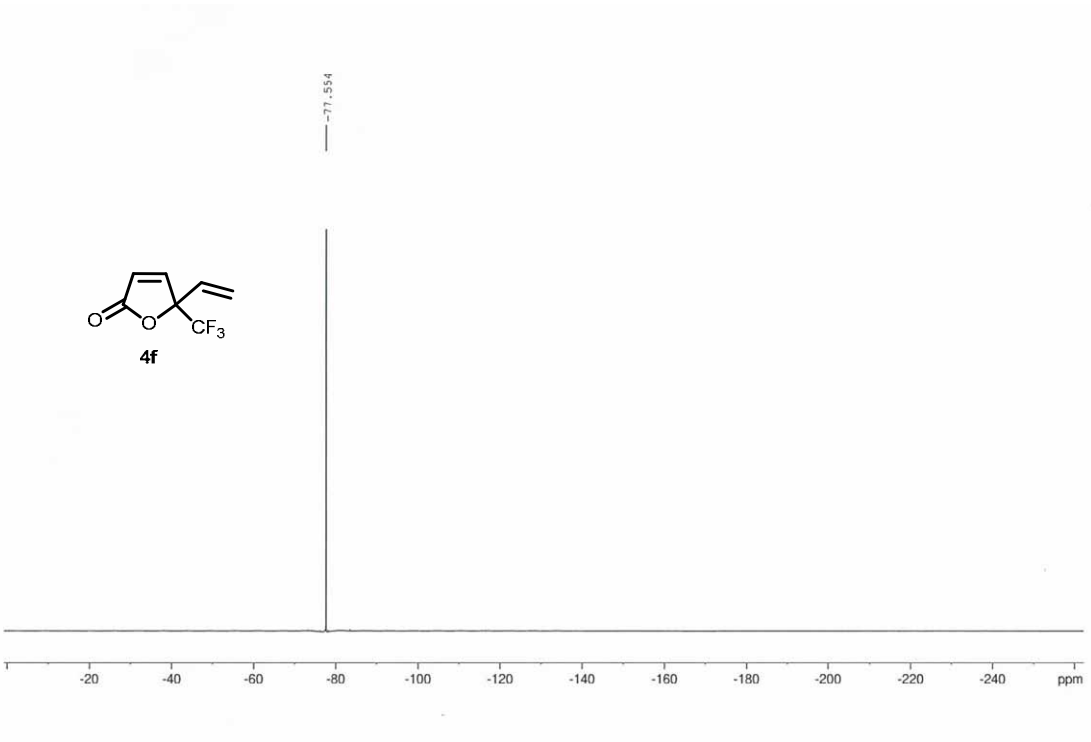
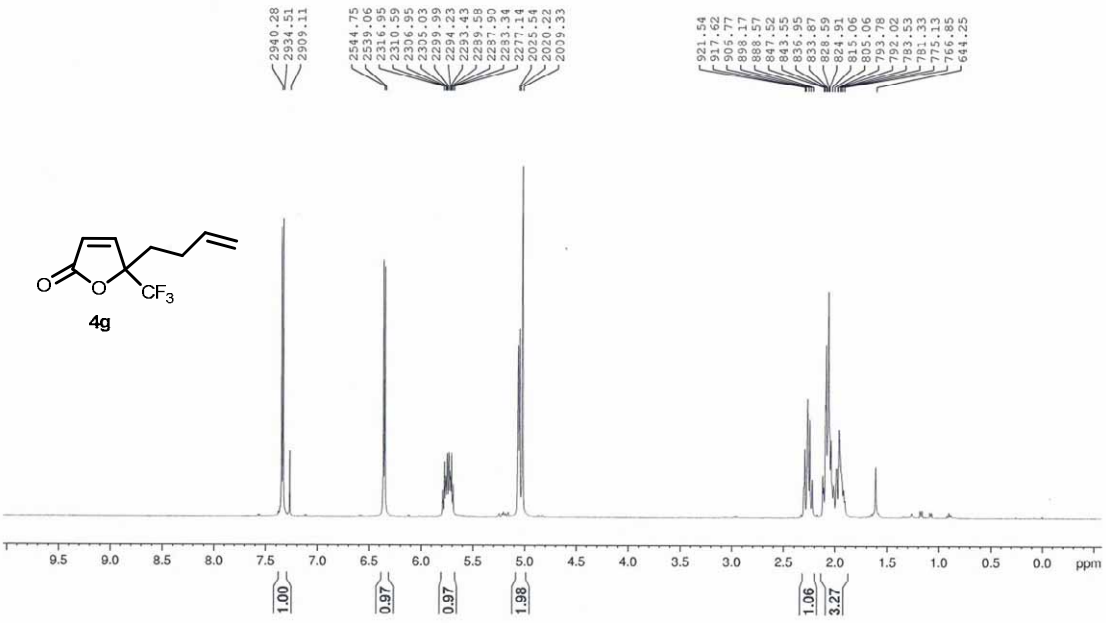


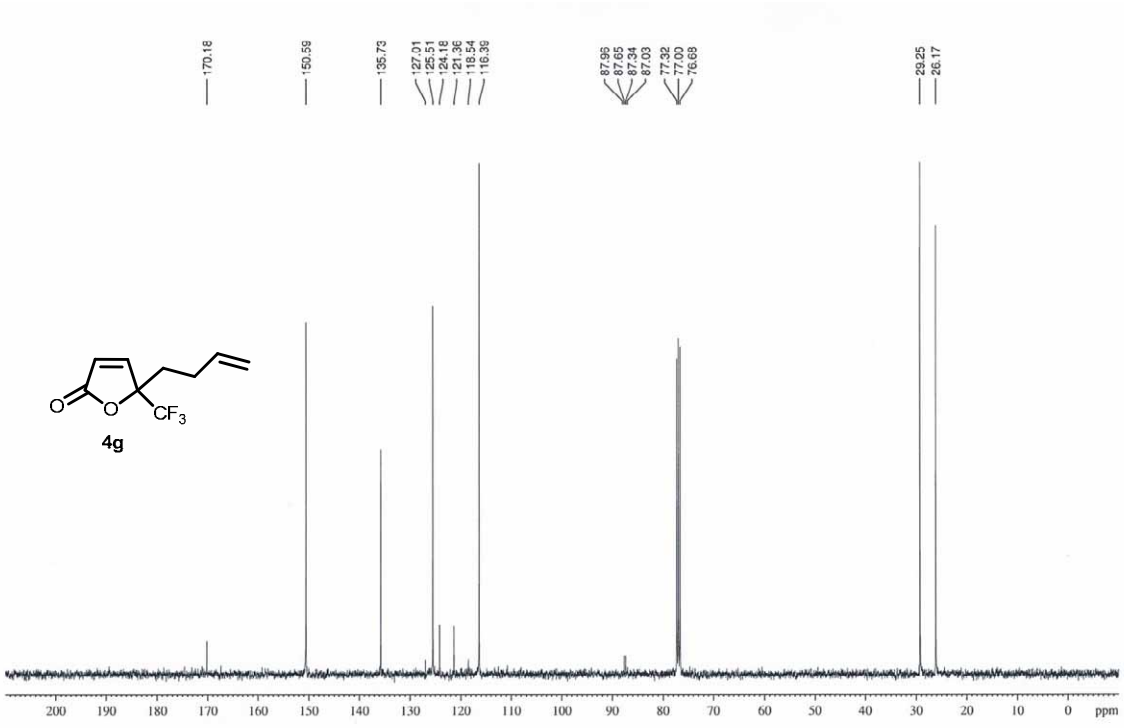
^{19}F NMR Spectrum of **4f** (376 MHz, $CDCl_3$)



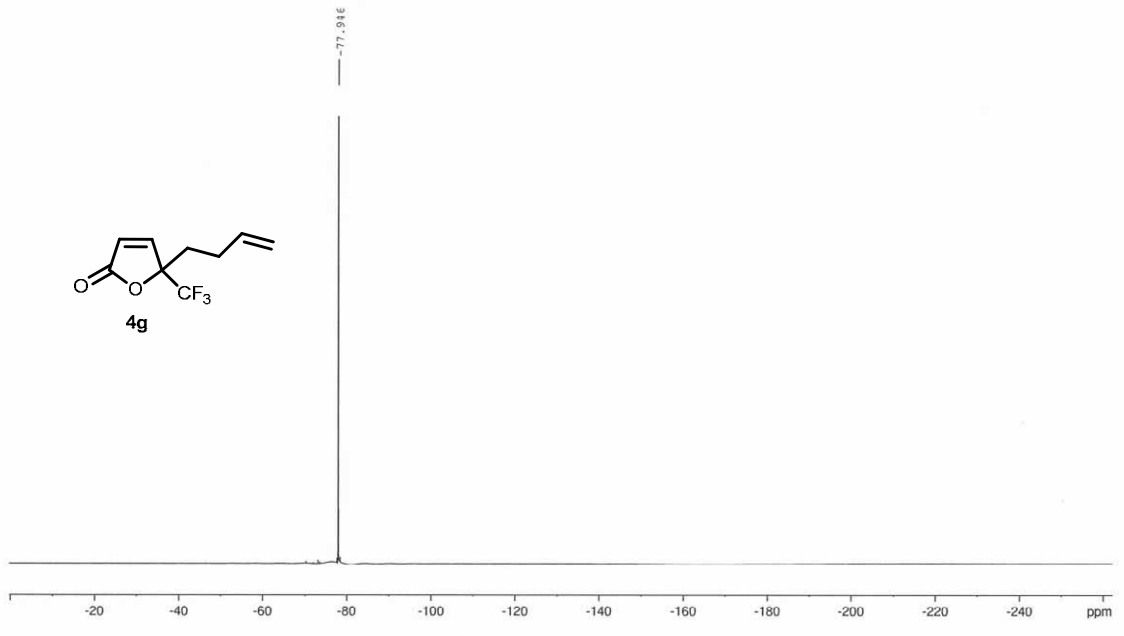
^{1}H NMR Spectrum of **4g** (400 MHz, $CDCl_3$)



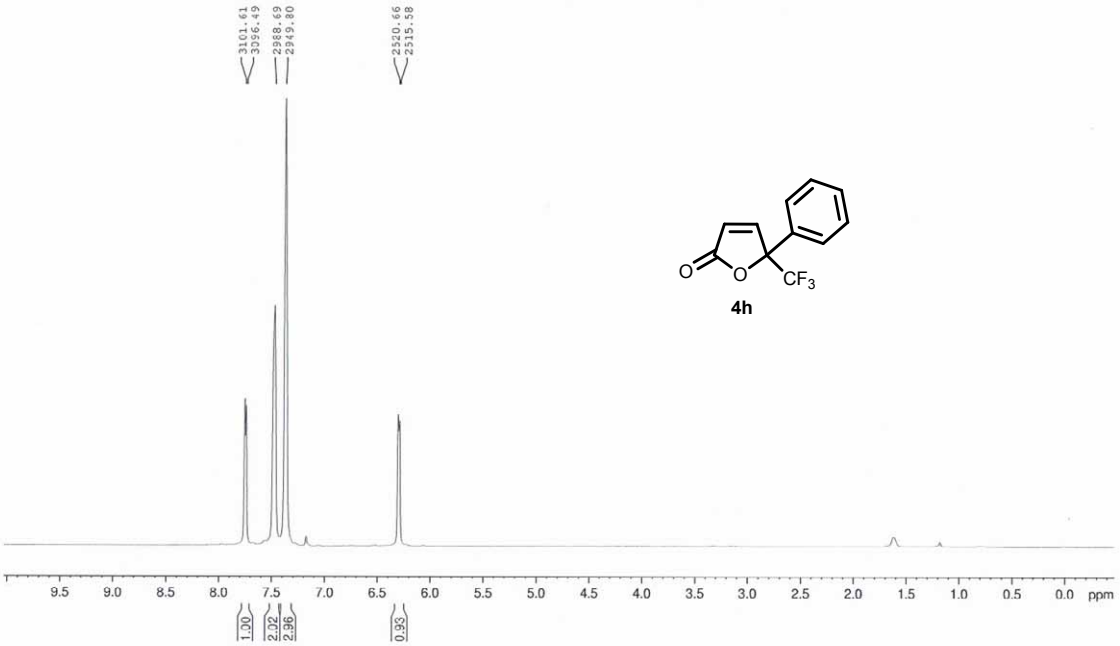
^{13}C NMR Spectrum of **4g** (100 MHz, $CDCl_3$)



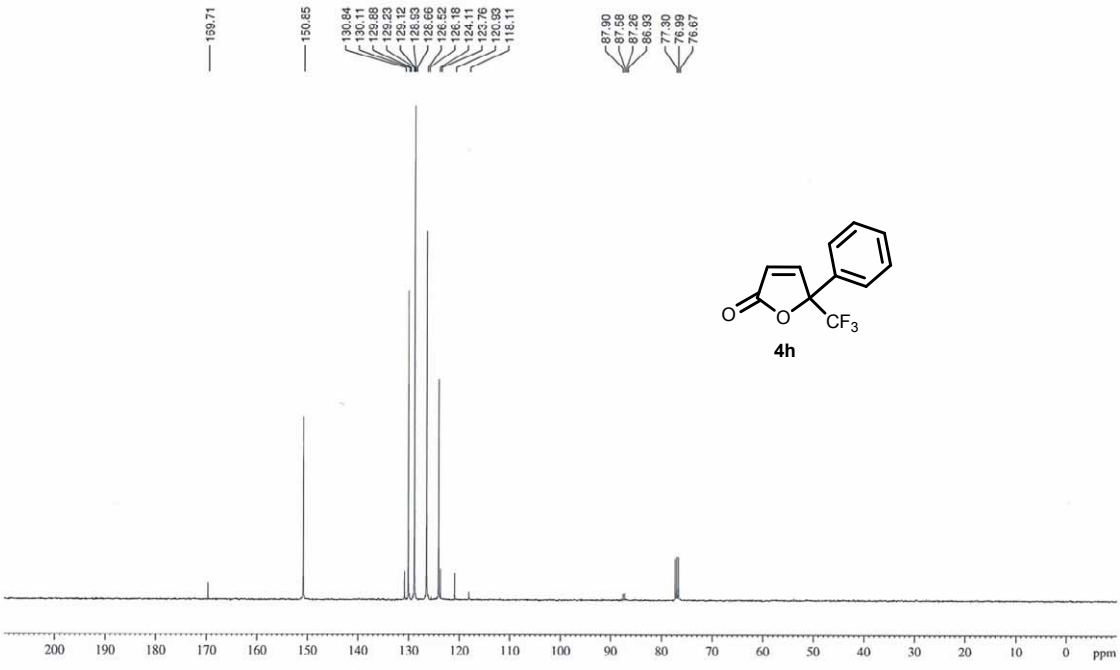
^{19}F NMR Spectrum of **4g** (376 MHz, $CDCl_3$)



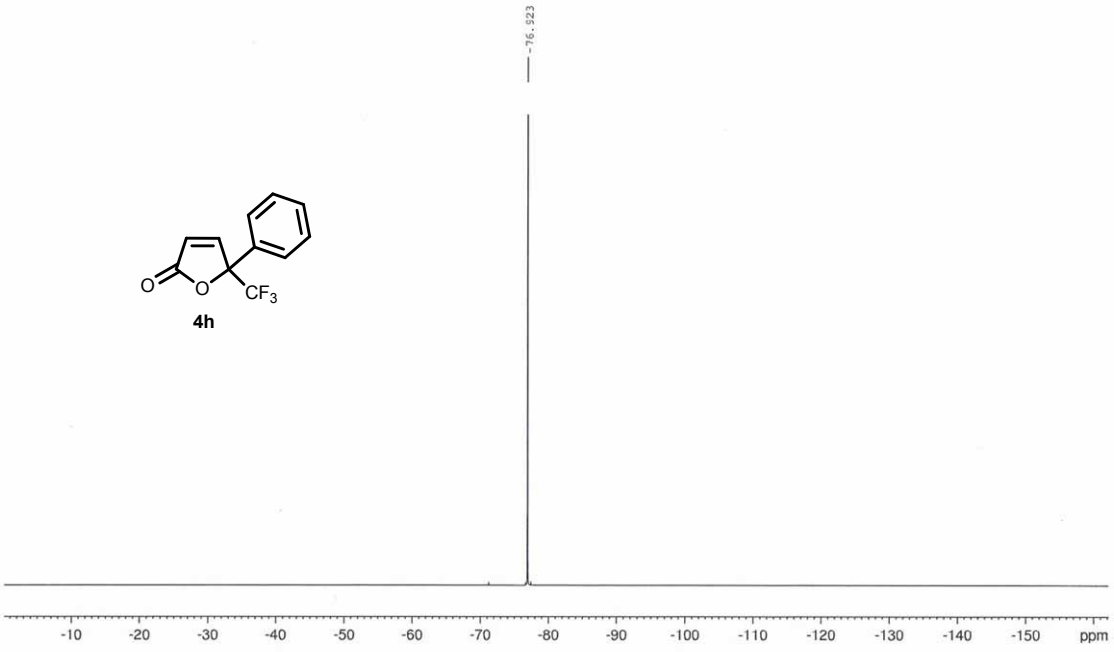
^{1}H NMR Spectrum of **4h** (400 MHz, $CDCl_3$)



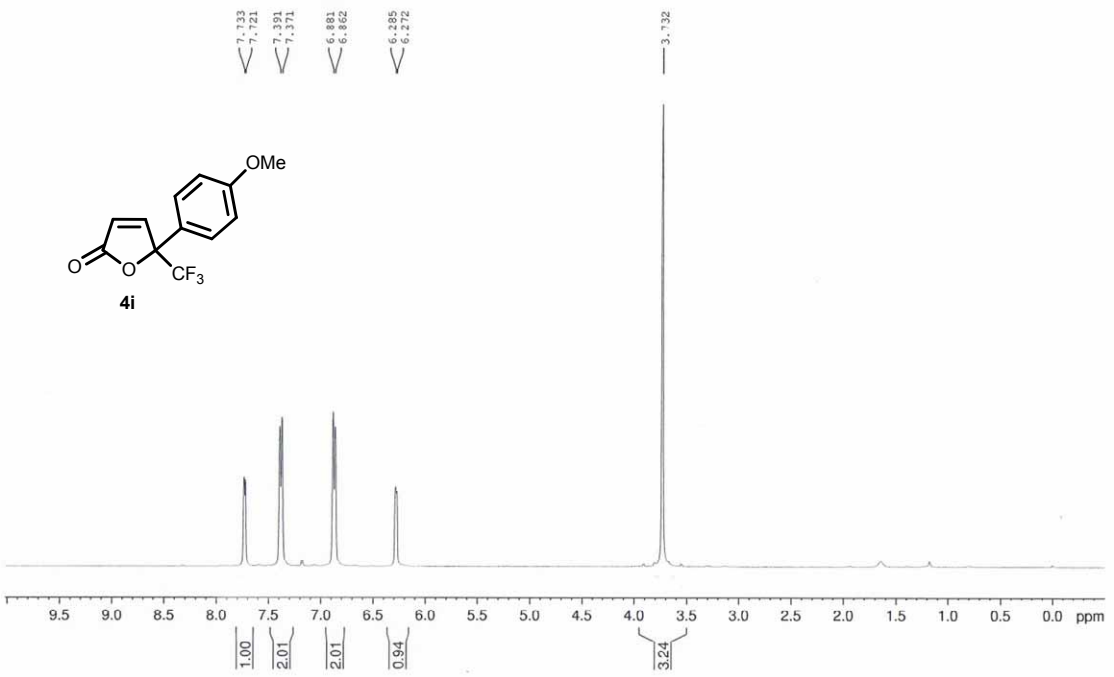
^{13}C NMR Spectrum of **4h** (100 MHz, $CDCl_3$)



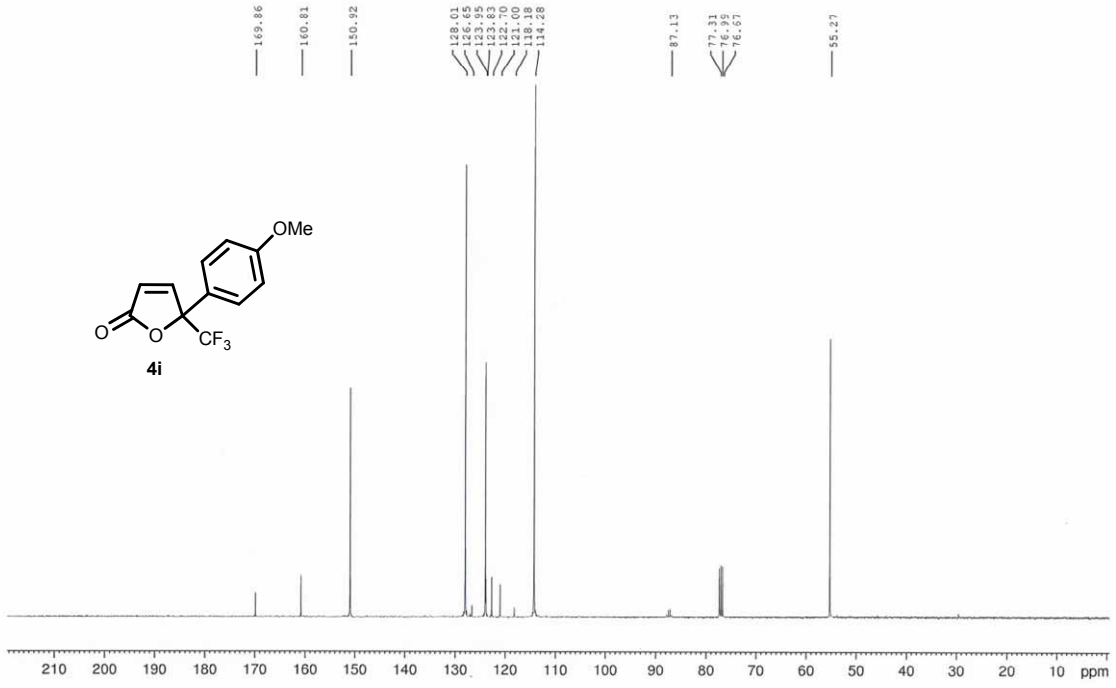
^{19}F NMR Spectrum of **4h** (376 MHz, $CDCl_3$)



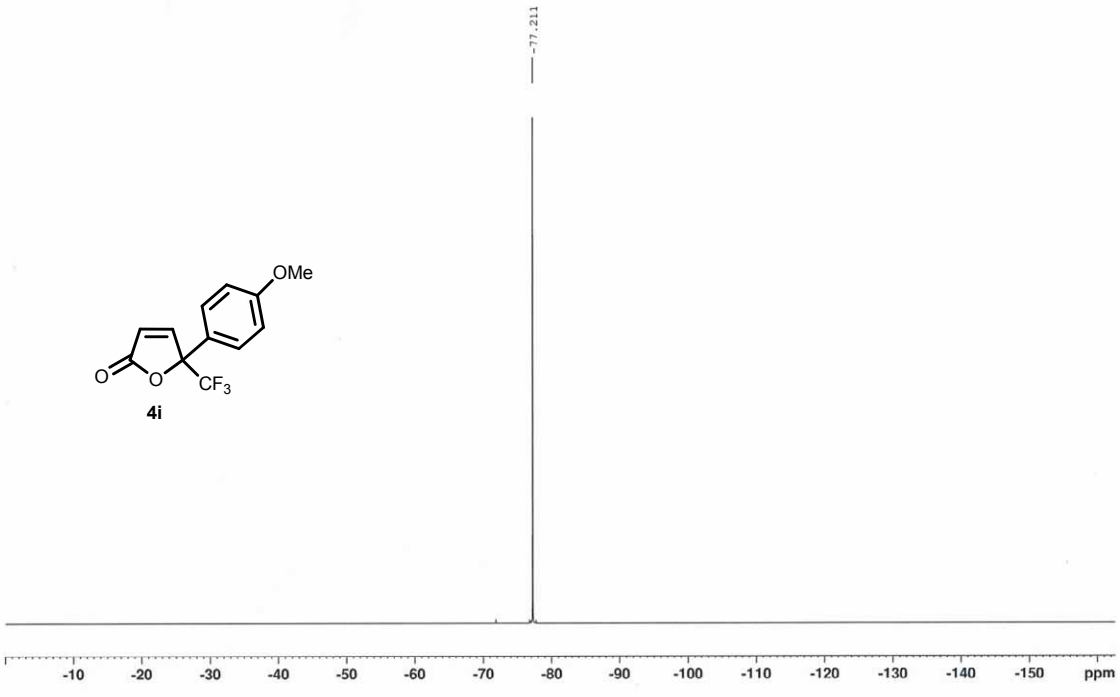
^{1}H NMR Spectrum of **4i** (400 MHz, $CDCl_3$)



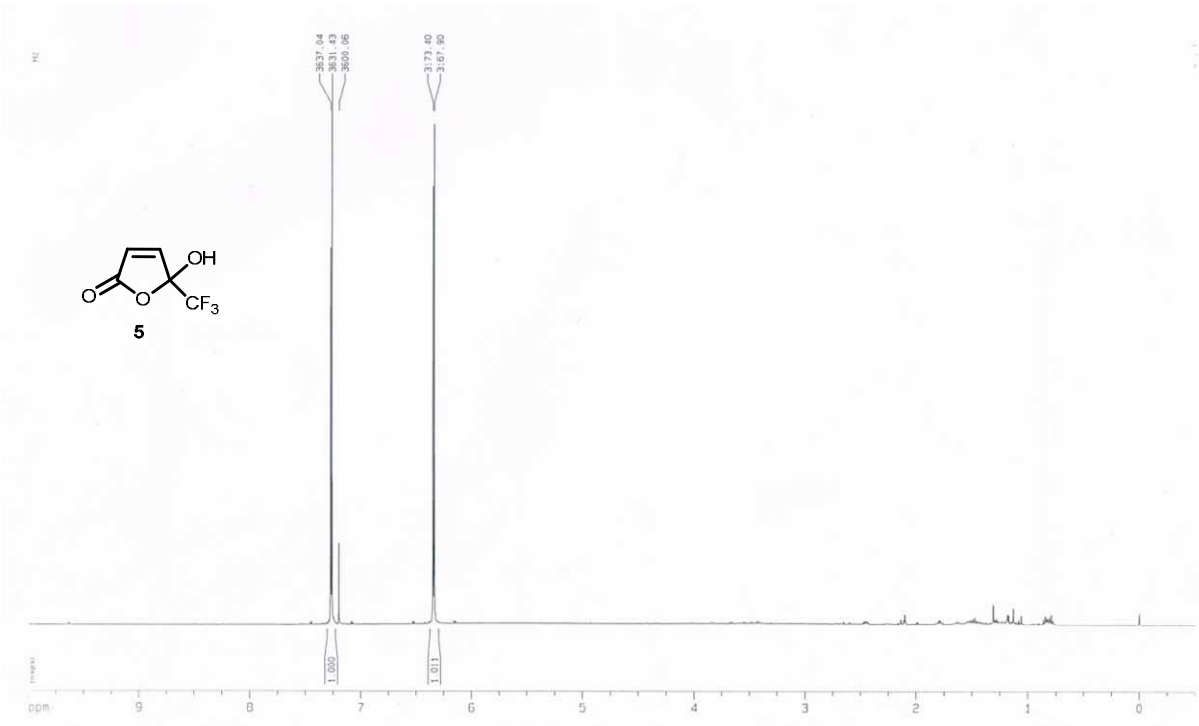
^{13}C NMR Spectrum of **4i** (100 MHz, $CDCl_3$)



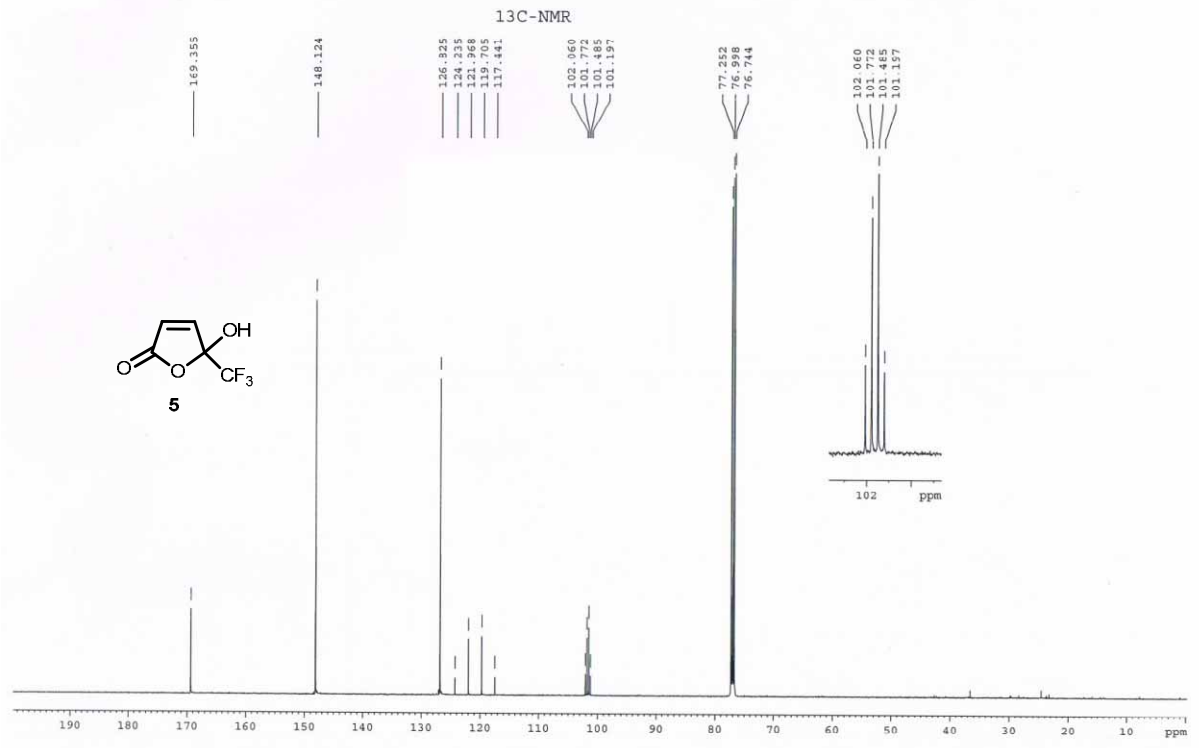
^{19}F NMR Spectrum of **4i** (376 MHz, $CDCl_3$)



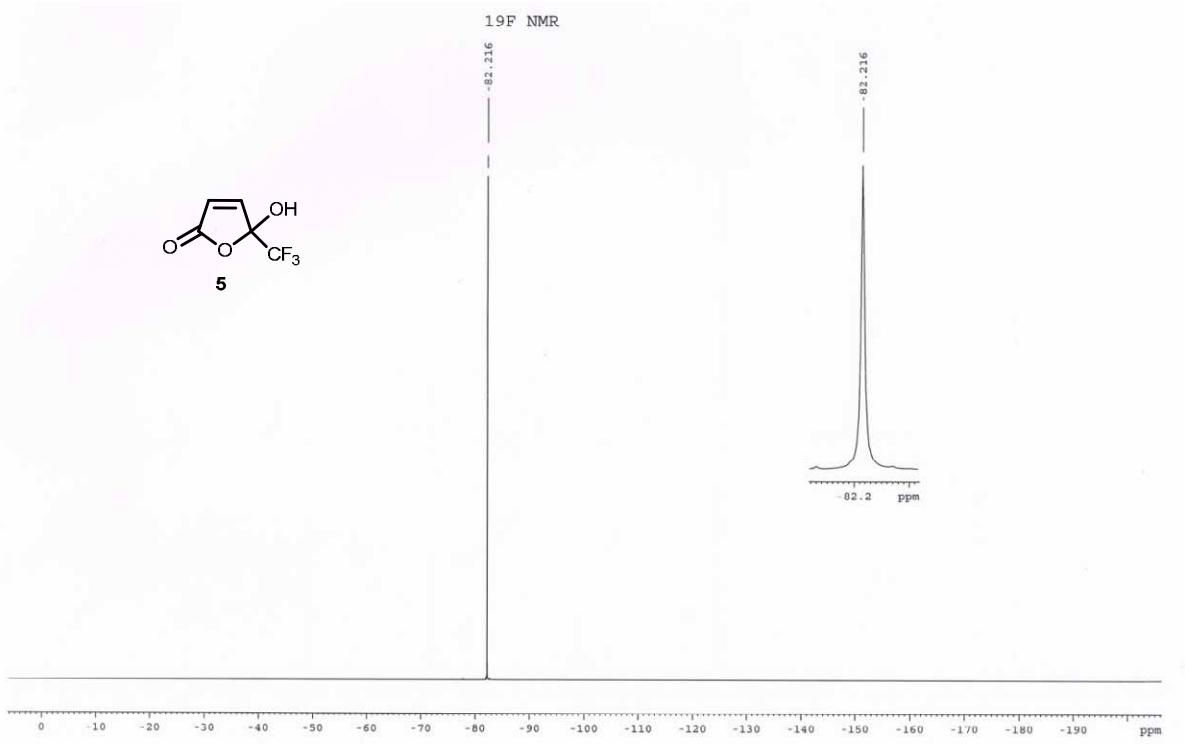
^{1}H NMR Spectrum of **5** (500 MHz, $CDCl_3$)



^{13}C NMR Spectrum of **5** (125 MHz, $CDCl_3$)



^{19}F NMR Spectrum of **5** (470 MHz, $CDCl_3$)



X-ray crystallographic analysis

X-ray diffraction data were measured on a Brucker-Nonius kappa CCD diffractometer with graphite monochromated MoKα radiation (λ = 0.71073 Å) at 293 (2) K. The structure were solved by direct methods by SIR97,[1] and refined with full-matrix least-squares calculations on F^2 using SHELXL-97.[2] Crystalographic data have been deposited at the Cambridge Crystallographic data Cantre under the reference number CCDC 933154 and 933155. Copies of the data can be obtained, free of charge, on application to the Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (e-mail: deposite@ccdc.cam.ac.th).

Crystal data and structure refinement for **2a**.

Identification code	cm002-rt-c2-c	
Empirical formula	C10 H9 F3 O3	
Formula weight	234.17	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 17.239(11) Å	alpha= 90deg.
	b = 12.975(8) Å	beta= 134.379(10)deg.
	c = 12.221(7) Å	gamma = 90deg.
Volume	1954(2) $Å^3$	
Z	8	
Density (calculated)	1.592 Mg/m^3	
Absorption coefficient	0.153 mm^{-1}	
F(000)	960	
Crystal size	0.163 x 0.136 x 0.072 mm^3	
Theta range for data collection	2.29 to 27.88deg.	
Index ranges	-22<=h<=22, -17<=k<=16, -15<=l<=16	
Reflections collected	7440	
Independent reflections	2304 [R(int) = 0.7145]	
Completeness to theta = 27.88	99.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.000 and 0.082	

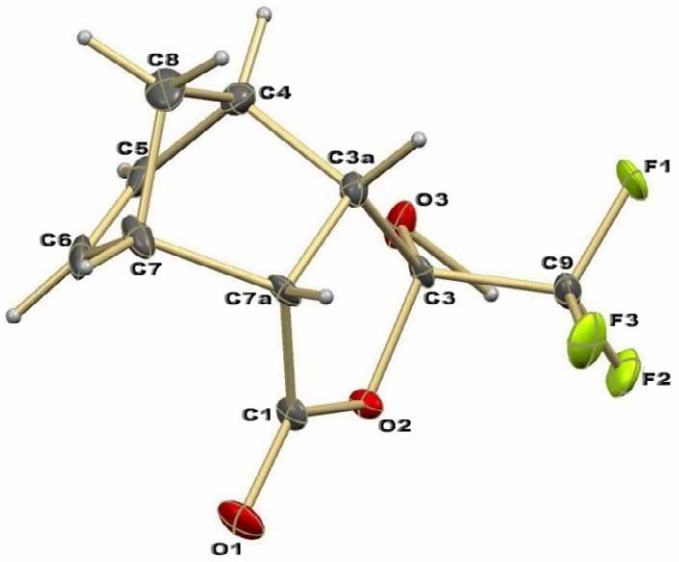
1. A. Altomare, M. C. Burla, M. Camalli, G. L. Cascarano, C. Giacovazzo, A. Guagliardi, A. G. G. Moliterni, G. Polidori, R. Spagna, *J. Appl. Cryst*., 1999, **32**, 115.
2. G. M. Sheldrick, *ActaCryst*., 2008, **A64**, 112.

Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2304 / 0 / 148
Goodness-of-fit on F^2	0.960
Final R indices [I>2sigma(I)]	R1 = 0.1265, wR2 = 0.2795
R indices (all data)	R1 = 0.1909, wR2 = 0.2964
Largest diff. peak and hole	0.638 and -0.629 e.A^{-3}

Crystal data and structure refinement for **3i**.

Identification code	cm001	
Empirical formula	C17 H15 F3 O3	
Formula weight	324.29	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	*P* 21/*n*	
Unit cell dimensions	*a* = 5.8968(7) Å	alpha= 90deg.
	b = 21.674(2) Å	beta= 103.342(2)deg.
	c = 12.1696(13) Å	gamma= 90deg.
Volume	1513.4(3) $Å^3$	
Z	4	
Density (calculated)	1.423 Mg/m^3	
Absorption coefficient	0.121 mm^{-1}	
F(000)	672	
Crystal size	0.302 x 0.089 x 0.02 mm^3	
Theta range for data collection	1.88 to 25.00deg.	
Index ranges	-7<=h<=7, -25<=k<=25, -14<=l<=14	
Reflections collected	14149	
Independent reflections	2662 [*R*(int) = 0.0950]	
Completeness to theta = 25.00	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.998 and 0.087	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2662 / 1 / 209	
Goodness-of-fit on F^2	1.091	
Final *R* indices [*I*>2□(I)]	*R*1 = 0.1027, *wR*2 = 0.1932	
R indices (all data)	*R*1 = 0.1709, *wR*2 = 0.2251	
Largest diff. peak and hole	0.234 and -0.306 e. $Å^{-3}$	

ORTEP plot of **2a (**CCDC 933155)



ORTEP plot of **3i** (CCDC 933154)