

Supporting information

Visible Light Photoredox Atom Transfer Ueno-Stork

Reaction

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General Information:

All reactions were carried out in glassware protected by N₂. Chemicals without special descriptions were obtained from commercial sources, and were used without further purification. Column chromatography was generally performed on silica gel (300-400 mesh). Thin layer chromatography (TLC) was visualized using UV light. All new compounds were characterized by ¹H-NMR, ¹³C-NMR and Mass Spectra. NMR spectra were recorded on Varian mercury 300 and Varian mercury 400 spectrometers using CDCl₃ as the solvent unless noted. Chemical shifts are reported in parts per million (δ) relative to CDCl₃ (7.27 ppm) for ¹H-NMR data and CDCl₃ (77.16 ppm) for ¹³C-NMR data. The abbreviations are used: s = singlet, d = doublet, t = triplet, q = quartet, dd = double doublet, m = multiple, and br = broad signal. High resolution mass spectra were measured on Agilent LC-MS 1200/6220 (ESI) and Bruker Apex IV FTMS (ESI) mass spectrometers. Cyclic voltammetry was recorded on Suzhou Ruisite Instrument RST5200 electrochemical workstation. X-Ray crystallographic structure data were collected with a Rigaku Mercury CCD area detector in ω scan mode using Mo-Kα radiation (λ = 0.71070 Å).

Experimental Procedures:

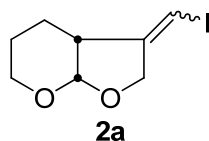
General Procedure for Photoredox Atom Transfer Ueno-Stork Reaction:

To a solution of 3-iodo-2-(prop-2-ynyloxy)tetrahydro-2H-pyran (**1a**) (1 mmol) in MeCN (4 ml), catalyst Ir(ppy)₂(dtb-bpy)PF₆ (1 mol %) and DIEA (0.5 eq) were added successively. The reaction was placed at a distance of about 5 cm from a 14 W fluorescent lamp, and stirred at room temperature for 9 h, the reaction mixture was diluted with H₂O (20 ml) and extracted with EtOAc (3×20 ml). The combined organic layers were washed with brine (20 ml) and dried over MgSO₄. The material was purified by silica gel column chromatography using petroleum ether/ethylacetate to give the desired cyclization product.

For the reactions in **Table 3**, a mixture of MeCN (4 ml) and H₂O (1.0 ml) was used as the solvent.

Characterizations:

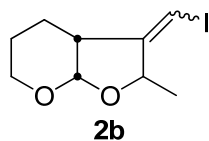
3-(iodomethylene)hexahydro-2H-furo[2,3-b]pyran, **2a**, (Table 2, entry 1)



The material was purified by silica gel column chromatography to give a mixture of isomers in a combined yield of 80%. One of the isomers was isolated by careful column chromatography and used to obtain NMR spectra.

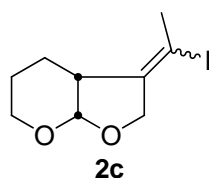
^1H NMR (300 MHz, CDCl_3) δ 5.92 (s, 1H), 5.29 (s, 1H), 4.37 (ABq, $\Delta\delta_{\text{AB}} = 0.05$, $J_{\text{AB}} = 14.2$, 2H), 3.83 (d, $J = 10.5$ Hz, 1H), 3.41 (t, $J = 11.2$ Hz, 1H), 2.66 (s, 1H), 2.05-1.77 (m, 2H), 1.65-1.45 (m, 1H), 1.30 (d, $J = 12.5$ Hz, 1H). ^{13}C NMR (75 MHz, CDCl_3) δ 150.56, 102.26, 75.38, 67.20, 64.37, 44.92, 22.61, 20.43. HRMS (ESI) m/z calculated for $\text{C}_8\text{H}_{11}\text{I}\text{NaO}_2^+$ ($[\text{M}+\text{Na}]^+$) 288.9696, found 288.96982.

3-(iodomethylene)-2-methylhexahydro-2H-furo[2,3-b]pyran, **2b**, (Table 2, entry 2)



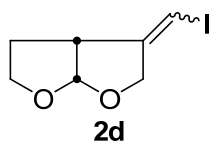
The material was purified by silica gel column chromatography to give a mixture of isomers in a combined yield of 67%. See NMR spectra section for the actual ^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra. HRMS (ESI) m/z calculated for $\text{C}_9\text{H}_{14}\text{IO}_2^+$ ($[\text{M}+\text{H}]^+$) 281.0033, found 281.00328.

3-(1-iodoethylidene)hexahydro-2H-furo[2,3-b]pyran, **2c**^[1], (Table 2, entry 3)



The material was purified by silica gel column chromatography to give a mixture of isomers in a combined yield of 87%. See NMR spectra section for the actual ^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra. HRMS (ESI) m/z calculated for $\text{C}_9\text{H}_{14}\text{IO}_2^+$ ($[\text{M}+\text{H}]^+$) 281.0033, found 281.00350.

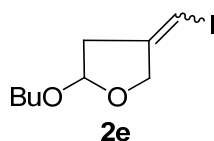
3-(iodomethylene)hexahydrofuro[2,3-b]furan, 2d^[21], (Table 2, entry 4)



The material was purified by silica gel column chromatography to give a mixture of isomers in a combined yield of 72%. One of the isomers was isolated by careful column chromatography and used to obtain NMR spectra.

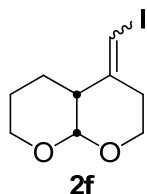
¹H NMR (400 MHz, CDCl₃) δ 6.18-6.01 (m, 2H), 5.90 (d, *J* = 4.7 Hz, 1H), 5.76 (d, *J* = 4.8 Hz, 1H), 4.45-4.22 (m, 4H), 3.97-3.82 (m, 2H), 3.81-3.68 (m, 2H), 3.32-3.25 (m, 1H), 3.24-3.12 (m, 1H), 2.23-2.04 (m, 2H), 2.04-1.94 (m, 1H), 1.90 (dd, *J* = 12.3, 5.0 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 153.56, 153.22, 110.81, 108.82, 76.28, 72.31, 70.64, 69.21, 68.10, 67.42, 51.24, 49.15, 34.06, 32.22. HRMS (ESI) *m/z* calculated for C₇H₁₀IO₂⁺ ([M+H]⁺) 252.9720, found. 252.97192.

2-butoxy-4-(iodomethylene)tetrahydrofuran, 2e, (Table 2, entry 5)



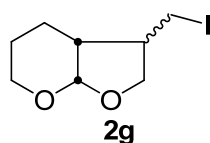
The material was purified by silica gel column chromatography to give a mixture of isomers in a combined yield of 79%. See NMR spectra section for the actual ¹H NMR (400 MHz, CDCl₃) and ¹³C NMR (101 MHz, CDCl₃) spectra. HRMS (ESI) *m/z* calculated for C₉H₁₆IO₂⁺ ([M+H]⁺) 283.0189, found 283.01925.

4-(iodomethylene)octahydropyrano[2,3-b]pyran, 2f, (Table 2, entry 6)



The material was purified by silica gel column chromatography to give a mixture of isomers in a combined yield of 22%. See NMR spectra section for the actual ¹H NMR (400 MHz, CDCl₃) and ¹³C NMR (101 MHz, CDCl₃) spectra. HRMS (ESI) *m/z* calculated for C₉H₁₄IO₂⁺ ([M+H]⁺) 281.0033, found 281.00380.

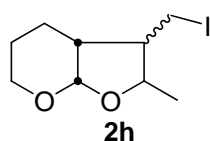
3-(iodomethyl)hexahydro-2H-furo[2,3-b]pyran, 2g^[3], (Table 2, entry 7)



The material was purified by silica gel column chromatography to give a mixture of isomers in a combined yield of 74%. One of the isomers was isolated by careful column chromatography and used to obtain NMR spectra.

¹H NMR (400 MHz, CDCl₃) δ 5.27 (d, *J* = 3.0 Hz, 1H), 4.01 (t, *J* = 8.1 Hz, 1H), 3.80-3.71 (m, 1H), 3.71-3.56 (m, 2H), 3.14 (d, *J* = 8.1 Hz, 2H), 2.88-2.74 (m, 1H), 2.13-2.03 (m, 1H), 1.81-1.71 (m, 1H), 1.64-1.52 (m, 2H), 1.48-1.35 (m, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 101.73, 70.21, 61.32, 44.35, 38.38, 22.79, 18.75, 2.34. HRMS (ESI) *m/z* calculated for C₈H₁₄IO₂⁺ ([M+H]⁺) 269.0033, found 269.00384.

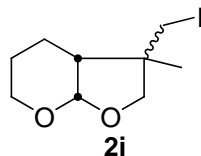
3-(iodomethyl)-2-methylhexahydro-2H-furo[2,3-b]pyran, 2h, (Table 2, entry 8)



The material was purified by silica gel column chromatography to give a mixture of isomers in a combined yield of 70%. One of the isomers was isolated by careful column chromatography and used to obtain NMR spectra.

¹H NMR (400 MHz, CDCl₃) δ 4.93 (d, *J* = 3.3 Hz, 1H), 3.97-3.81 (m, 2H), 3.39 (td, *J* = 11.5, 1.9 Hz, 1H), 3.31 (dd, *J* = 10.4, 3.8 Hz, 1H), 3.21 (dd, *J* = 10.4, 6.2 Hz, 1H), 1.96 (dd, *J* = 10.1, 3.8 Hz, 1H), 1.88-1.76 (m, 3H), 1.70-1.54 (m, 1H), 1.46-1.32 (m, 4H). ¹³C NMR (101 MHz, CDCl₃) δ 101.37, 82.11, 64.43, 45.76, 43.56, 22.34, 22.27, 20.77, 7.64. HRMS (ESI) *m/z* calculated for C₉H₁₆IO₂⁺ ([M+H]⁺) 283.0189, found 283.01901.

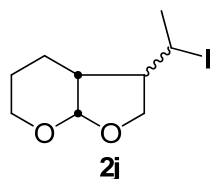
3-(iodomethyl)-3-methylhexahydro-2H-furo[2,3-b]pyran, 2i, (Table 2, entry 9)



The material was purified by silica gel column chromatography to give a mixture of isomers in a combined yield of 66%. One of the isomers was isolated by careful column chromatography and used to obtain NMR spectra.

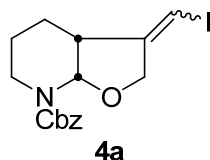
^1H NMR (400 MHz, CDCl_3) δ 5.33 (s, 1H), 3.90 (d, $J = 7.7$ Hz, 1H), 3.73-3.61 (m, 1H), 3.52 (d, $J = 5.0$ Hz, 2H), 3.31 (d, $J = 9.6$ Hz, 1H), 3.13 (d, $J = 9.6$ Hz, 1H), 1.91-1.79 (m, 1H), 1.74 (s, 1H), 1.60-1.34 (m, 3H), 1.20 (s, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 101.59, 75.61, 61.30, 44.30, 43.62, 26.34, 22.60, 20.38, 14.78. HRMS (ESI) m/z calculated for $\text{C}_9\text{H}_{16}\text{IO}_2^+$ ($[\text{M}+\text{H}]^+$) 283.0189, found 283.01906.

3-(1-iodoethyl)hexahydro-2H-furo[2,3-b]pyran, 2j, (Table 2, entry 10)



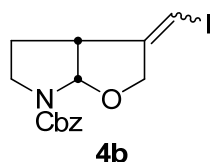
The material was purified by silica gel column chromatography to give a mixture of isomers in a combined yield of 58%. See NMR spectra section for the actual ^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra. HRMS (ESI) m/z calculated for $\text{C}_9\text{H}_{16}\text{IO}_2^+$ ($[\text{M}+\text{H}]^+$) 283.0189, found 283.01891.

benzyl 3-(iodomethylene)hexahydrofuro[2,3-b]pyridine-7(7aH)-carboxylate, 4a, (Table 3, entry 1)



The material was purified by silica gel column chromatography to give a mixture of isomers in a combined yield of 85%. See NMR spectra section for the actual ^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra. HRMS (ESI) m/z calculated for $\text{C}_{16}\text{H}_{19}\text{INO}_3^+$ ($[\text{M}+\text{H}]^+$) 400.0404, found 400.04150.

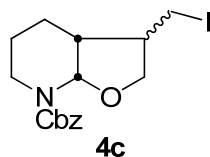
benzyl 3-(iodomethylene)tetrahydro-2H-furo[2,3-b]pyrrole-6(6aH)-carboxylate, 4b, (Table 3, entry 2)



The material was purified by silica gel column chromatography to give a mixture of isomers in a combined yield of 82%. See NMR spectra section for the actual ^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra. HRMS (ESI) m/z calculated for $\text{C}_{15}\text{H}_{17}\text{INO}_3^+$ ($[\text{M}+\text{H}]^+$)

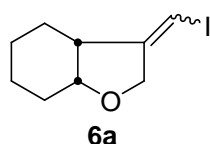
386.0248, found 386.02543.

benzyl 3-(iodomethyl)hexahydrofuro[2,3-b]pyridine-7(7aH)-carboxylate, 4c, (Table 3, entry 3)



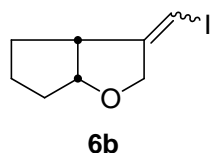
The material was purified by silica gel column chromatography to give a mixture of isomers in a combined yield of 94%. See NMR spectra section for the actual ^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra. HRMS (ESI) m/z calculated for $\text{C}_{16}\text{H}_{21}\text{INO}_3^+$ ($[\text{M}+\text{H}]^+$) 402.0561, found 402.05649.

3-(iodomethylene)octahydrobenzofuran, 6a^[4], (Table 3, entry 4)



The material was purified by silica gel column chromatography to give a mixture of isomers in a combined yield of 80%. See NMR spectra section for the actual ^1H NMR (400 MHz, CDCl_3) and ^{13}C NMR (101 MHz, CDCl_3) spectra. HRMS (ESI) m/z calculated for $\text{C}_9\text{H}_{14}\text{IO}^+$ ($[\text{M}+\text{H}]^+$) 265.0084, found 265.00888.

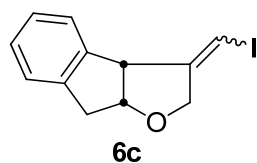
3-(iodomethylene)hexahydro-2H-cyclopenta[b]furan, 6b, (Table 3, entry 5)



The material was purified by silica gel column chromatography to give a mixture of isomers in a combined yield of 74%. One of the isomers was isolated by careful column chromatography and used to obtain NMR spectra.

^1H NMR (400 MHz, CDCl_3) δ 5.99-5.80 (m, 1H), 4.67-4.46 (m, 1H), 4.39-4.25 (d, $J = 13.3$ Hz, 1H), 4.22-4.05 (m, 1H), 3.08-2.92 (m, 1H), 2.01-1.71 (m, 2H), 1.70-1.44 (m, 4H). ^{13}C NMR (101 MHz, CDCl_3) δ 157.64, 157.48, 87.66, 85.86, 76.29, 72.36, 67.47, 66.84, 51.97, 49.94, 34.18, 33.47, 31.40, 24.52, 24.22. HRMS (ESI) m/z calculated for $\text{C}_8\text{H}_{12}\text{IO}^+$ ($[\text{M}+\text{H}]^+$) 250.9927, found 250.99335.

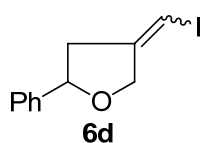
3-(iodomethylene)-3,3a,8,8a-tetrahydro-2H-indeno[2,1-b]furan, 6c, (Table 3, entry 6)



The material was purified by silica gel column chromatography to give a mixture of isomers in a combined yield of 69%. One of the isomers was isolated by careful column chromatography and used to obtain NMR spectra.

^1H NMR (400 MHz, CDCl_3) δ 7.36 (d, $J = 6.0$ Hz, 1H), 7.27-7.04 (m, 3H), 6.02 (s, 1H), 5.74 (d, $J = 6.5$ Hz, 1H), 4.35 (d, $J = 14.4$ Hz, 1H), 4.00 (d, $J = 14.4$ Hz, 1H), 3.44 (s, 1H), 3.20 (dd, $J = 16.1$, 8.0 Hz, 1H), 2.93 (d, $J = 16.2$ Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 156.77, 142.18, 141.15, 129.07, 127.33, 125.37, 124.99, 89.74, 77.48, 77.16, 76.84, 75.54, 68.25, 48.76, 38.06. HRMS (ESI) m/z calculated for $\text{C}_{12}\text{H}_{11}\text{I}\text{NaO}^+$ ($[\text{M}+\text{Na}]^+$) 320.9747, found 320.97539.

4-(iodomethylene)-2-phenyltetrahydrofuran, 6d^[5], (Table 3, entry 7)



The material was purified by silica gel column chromatography to give a mixture of isomers in a combined yield of 78%. One of the isomers was isolated by careful column chromatography and used to obtain NMR spectra.

^1H NMR (400 MHz, CDCl_3) δ 7.53-7.29 (m, 5H), 6.08 (d, $J = 14.3$ Hz, 1H), 5.10 (dd, $J = 16.2$, 8.0 Hz, 1H), 4.62 (d, $J = 13.9$ Hz, 1H), 4.40 (t, $J = 15.4$ Hz, 1H), 3.15 – 2.92 (m, 1H), 2.59 (dd, $J = 24.7$, 12.4 Hz, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 152.08, 151.11, 140.80, 128.40, 127.77, 127.75, 125.81, 125.73, 82.23, 80.14, 75.55, 71.44, 67.63, 67.15, 45.54, 43.13. HRMS (ESI) m/z calculated for $\text{C}_{11}\text{H}_{12}\text{IO}^+$ ($[\text{M}+\text{H}]^+$) 308.9747, found 308.97481.

References:

- [1] R. Yanada, Y. Koh, N. Nishimori, A. Matsumura, S. Obika, H. Mitsuya, N. Fujii and Y. Takemoto, *The Journal of Organic Chemistry* **2004**, *69*, 2417-2422.
- [2] S. A. Eastham, S. P. Ingham, M. R. Hallett, J. Herbert, A. Modi, T. Morley, J. E. Painter, P. Patel, P. Quayle, D. C. Ricketts and J. Raftery, *Tetrahedron* **2008**, *64*, 936-948.
- [3] H. Yorimitsu, T. Nakamura, H. Shinokubo, K. Oshima, K. Omoto and H. Fujimoto, *Journal of the American Chemical Society* **2000**, *122*, 11041-11047.
- [4] G. Haaima, M.-J. Lynch, A. Routledge and R. T. Weavers, *Tetrahedron* **1993**, *49*, 4229-4252.

[5] a) T. Harada, K. Muramatsu, T. Fujiwara, H. Kataoka and A. Oku, *Organic Letters* **2005**, *7*, 779-781; b) T. Harada, K. Muramatsu, K. Mizunashi, C. Kitano, D. Imaoka, T. Fujiwara and H. Kataoka, *The Journal of Organic Chemistry* **2007**, *73*, 249-258.

Cyclic Voltammetry for compound 1a:

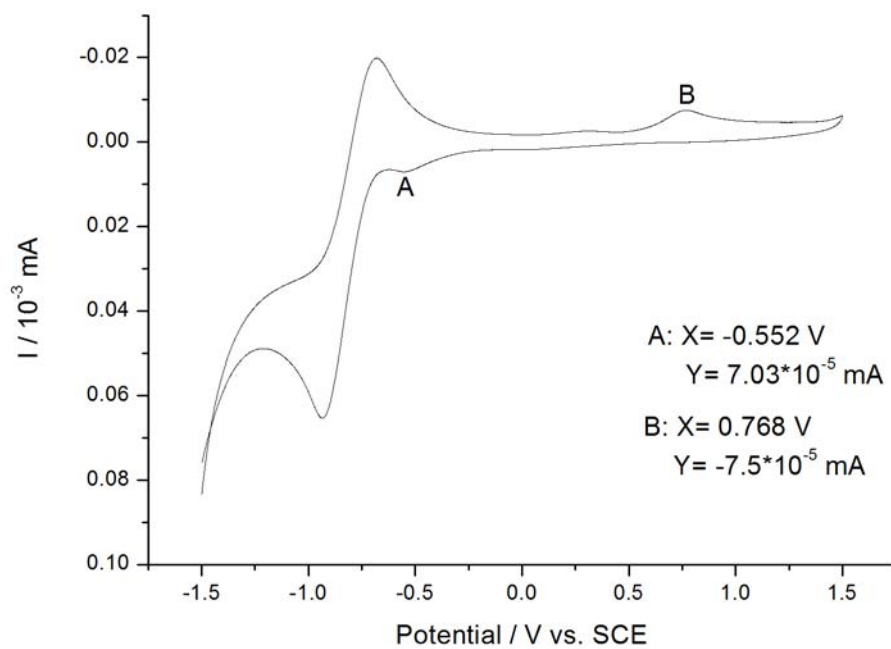


Figure S1. Cyclic voltammetry for 3-iodo-2-(prop-2-ynyloxy)tetrahydro-2H-pyran (**1a**)

Concentration of compound 6 is 0.25 M, determined in 0.1 M TBAPF₆/acetonitrile solution.

Frequency: 50 mv/s. RST5200 Potentiostat, and 3 electrode electrochemical cell consisting of 3 mm

Glassy Carbon electrode, SCE reference electrode, Pt Aux electrode.

Cyclic Voltammetry for compound 2g:

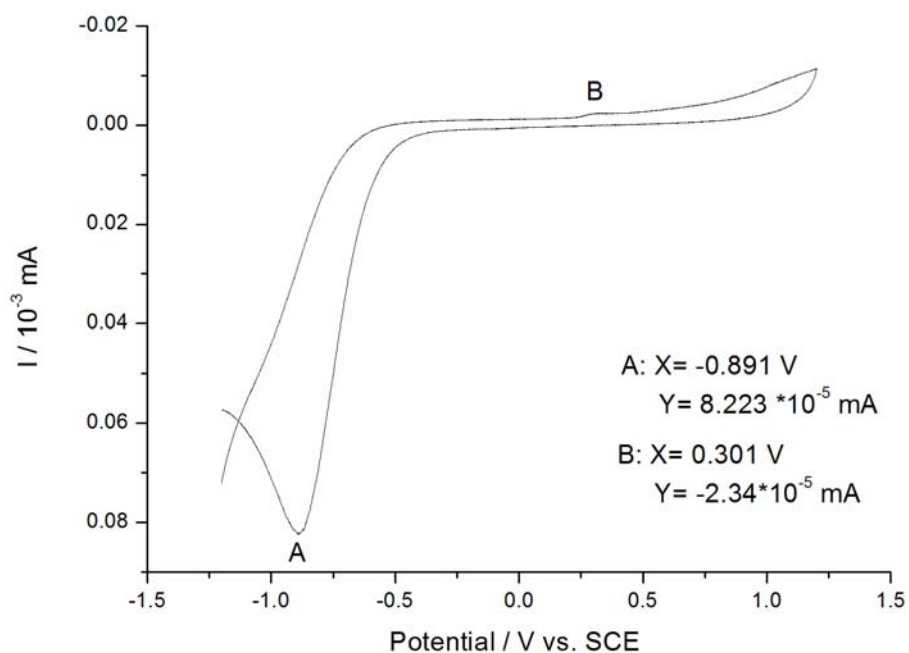


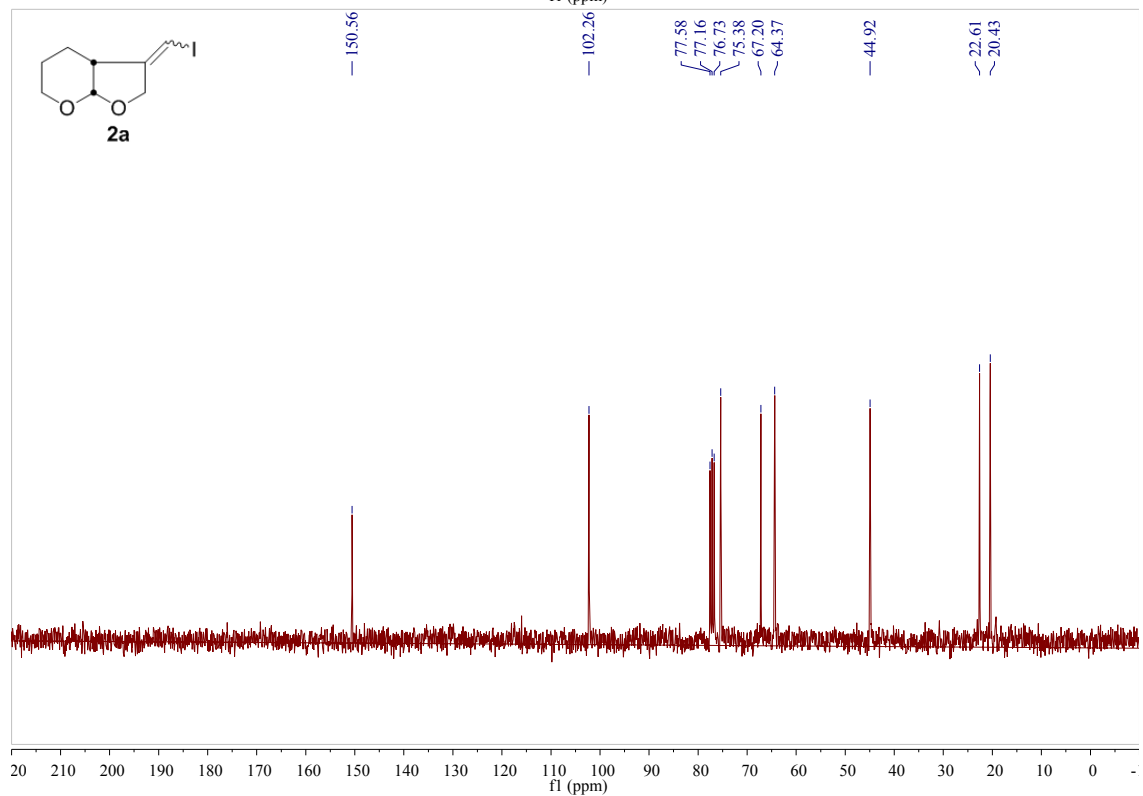
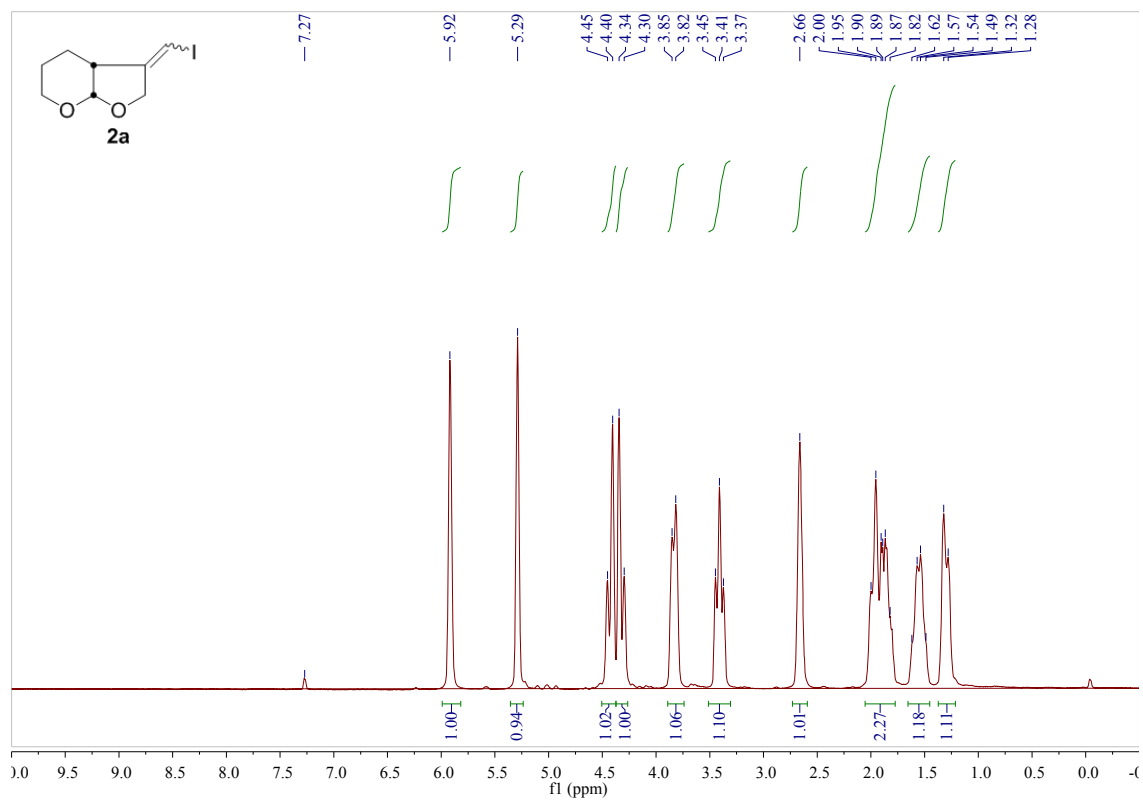
Figure S2. Cyclic voltammetry for 3-(iodomethyl)hexahydro-2H-furo[2,3-b]pyran (**2g**)

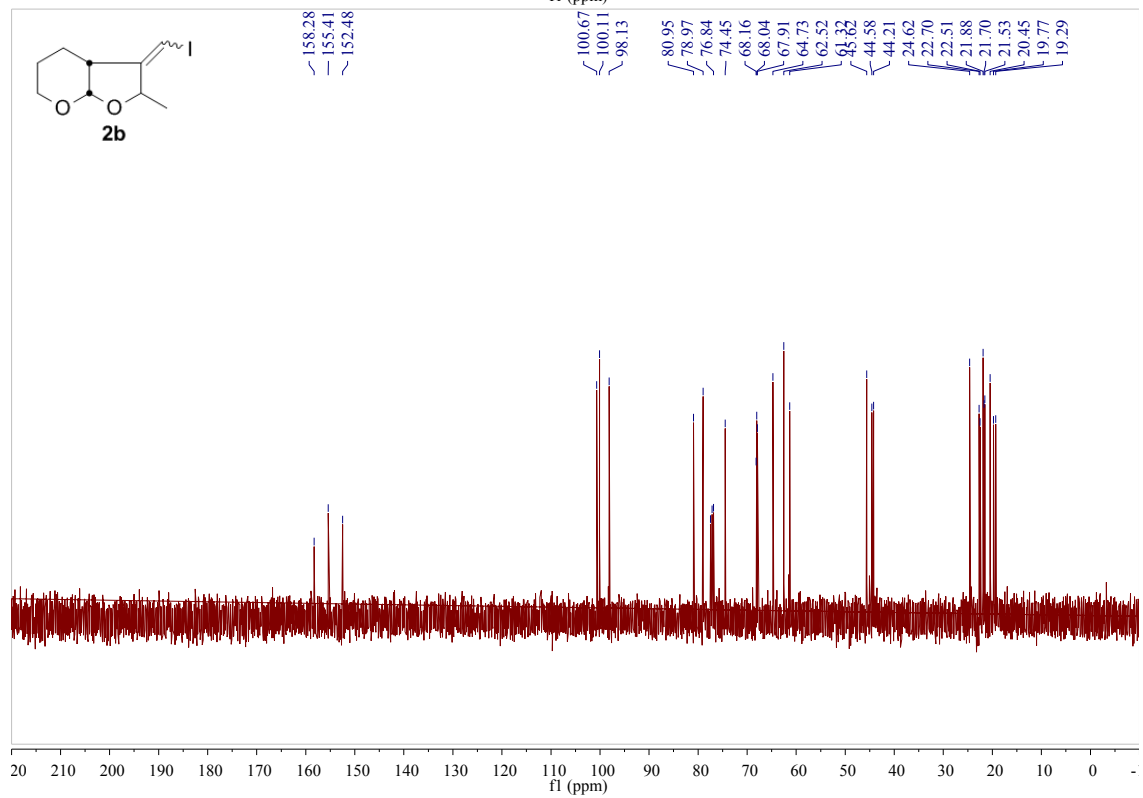
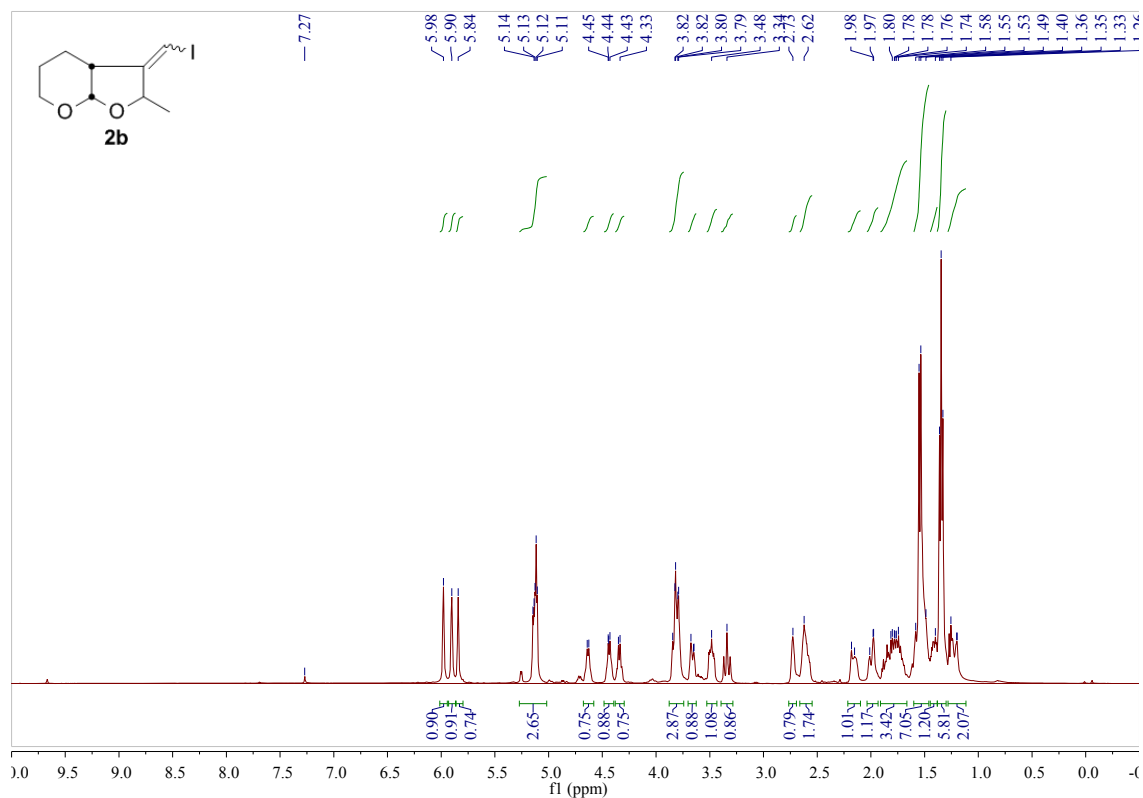
Concentration of compound 6 is 0.12 M, determined in 0.1 M TBAPF₆/acetonitrile solution.

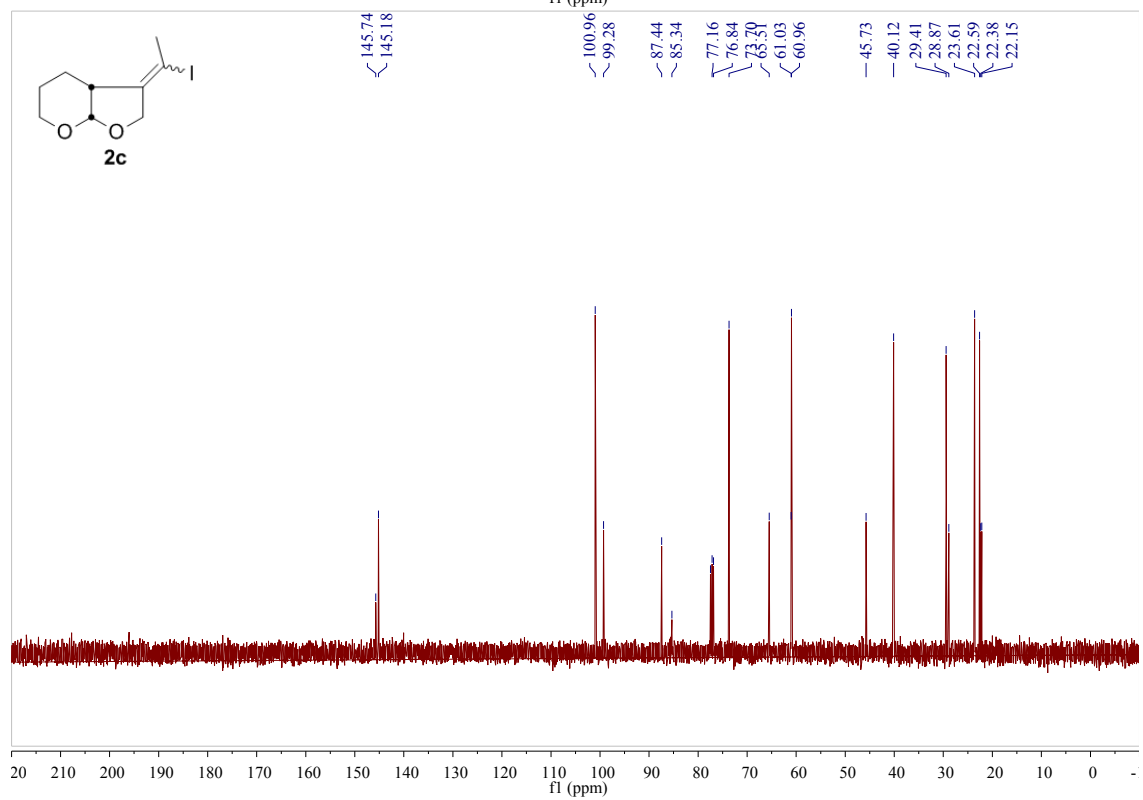
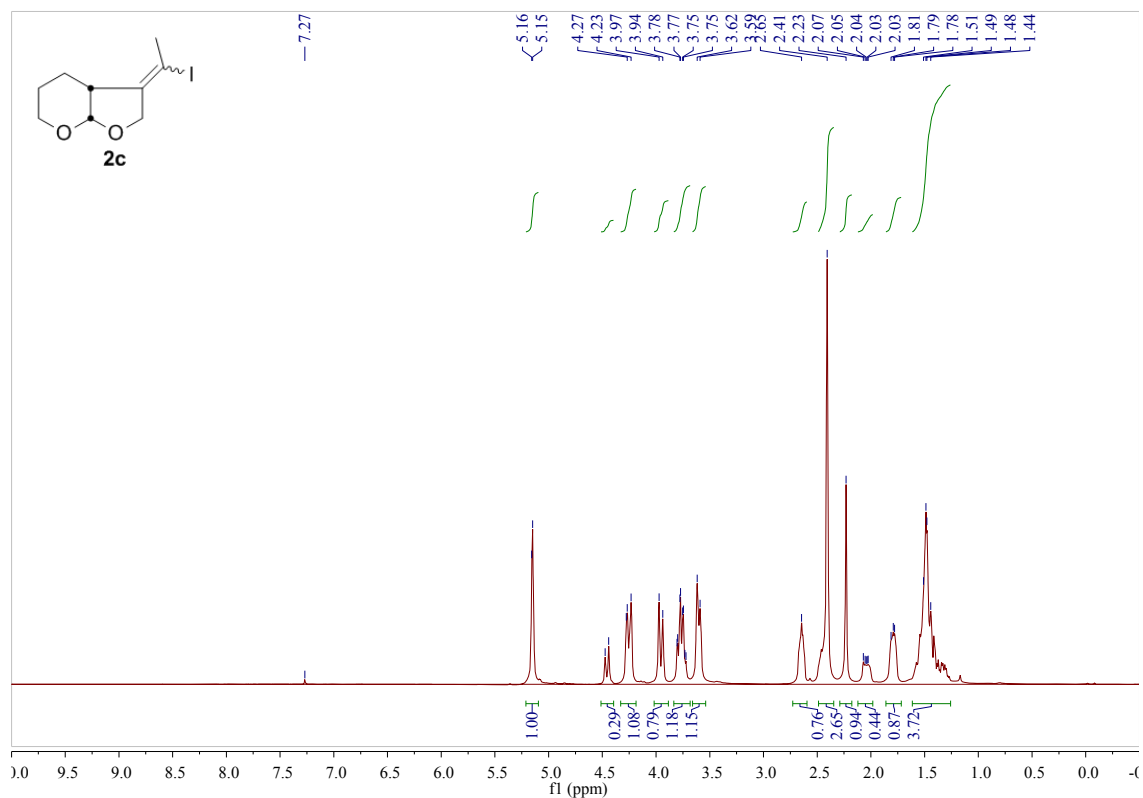
Frequency: 50 mv/s. RST5200 Potentiostat, and 3 electrode electrochemical cell consisting of 3 mm

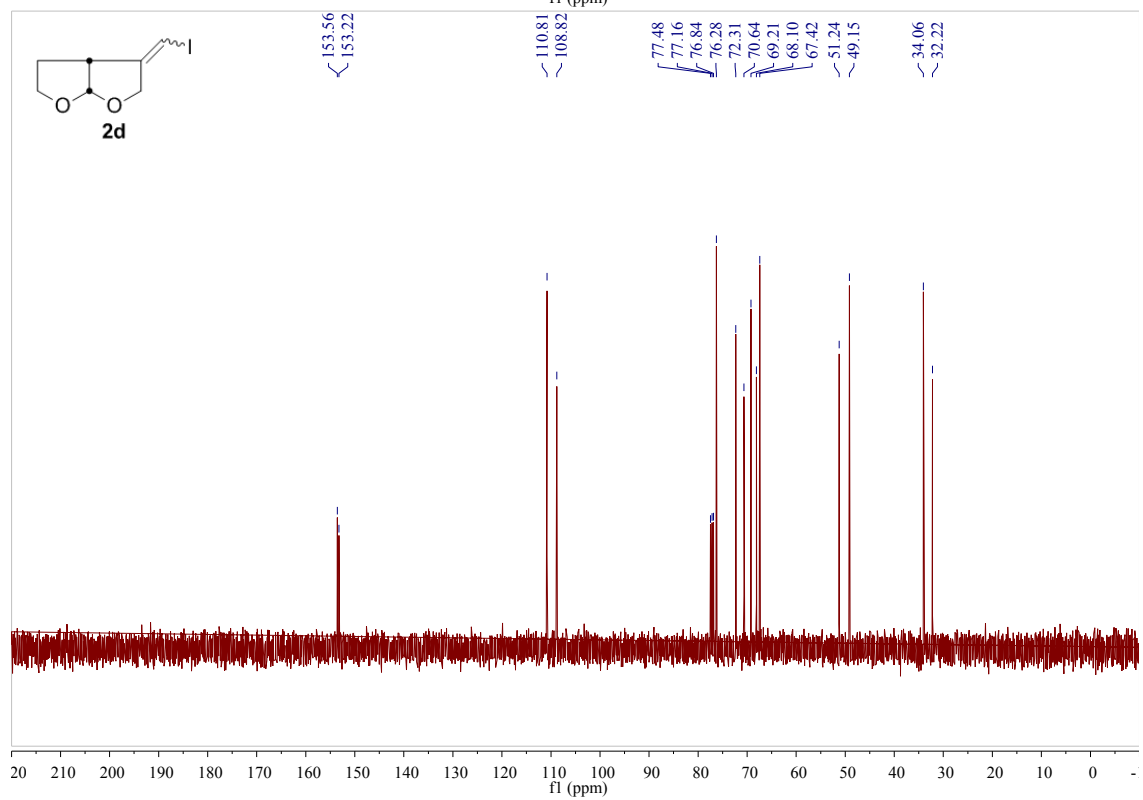
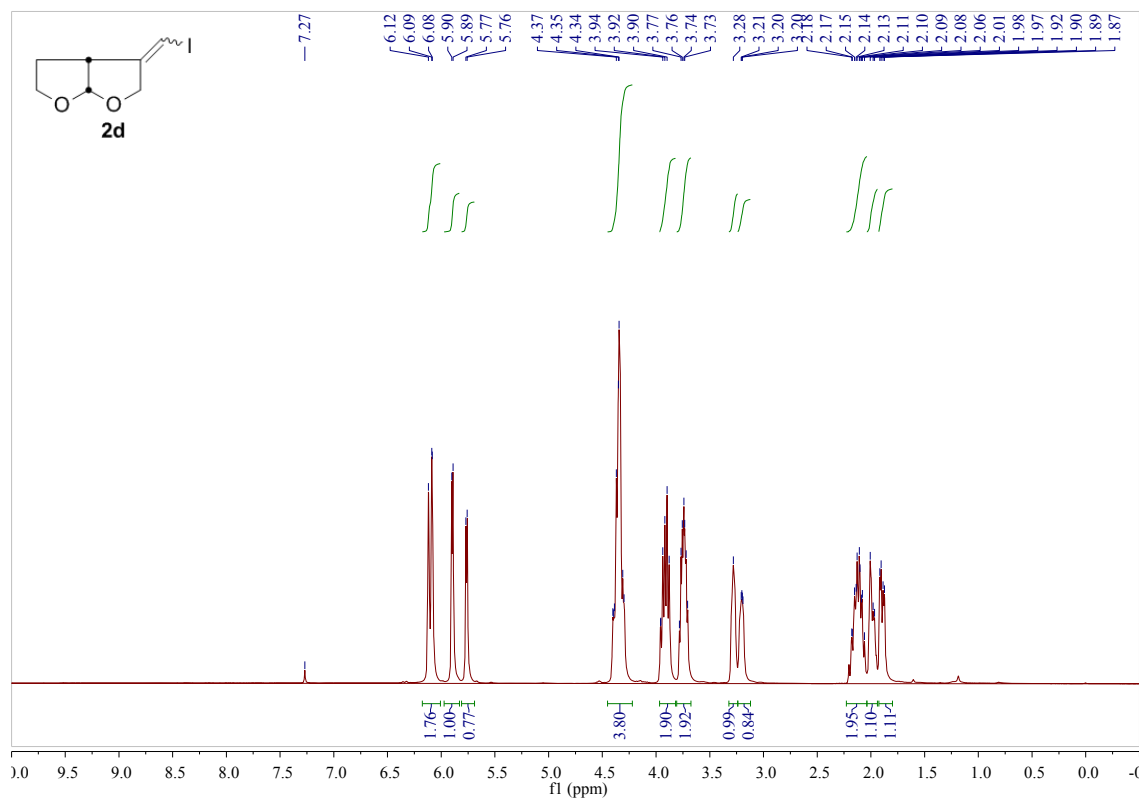
Glassy Carbon electrode, SCE reference electrode, Pt Aux electrode.

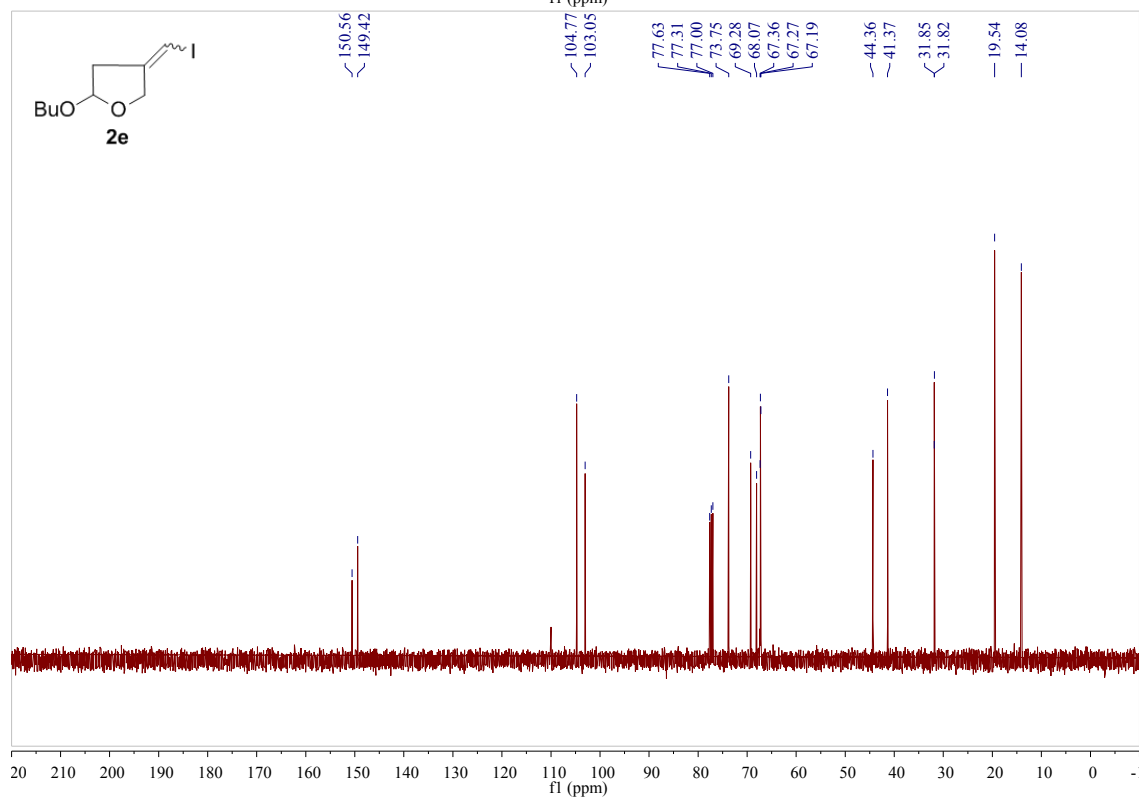
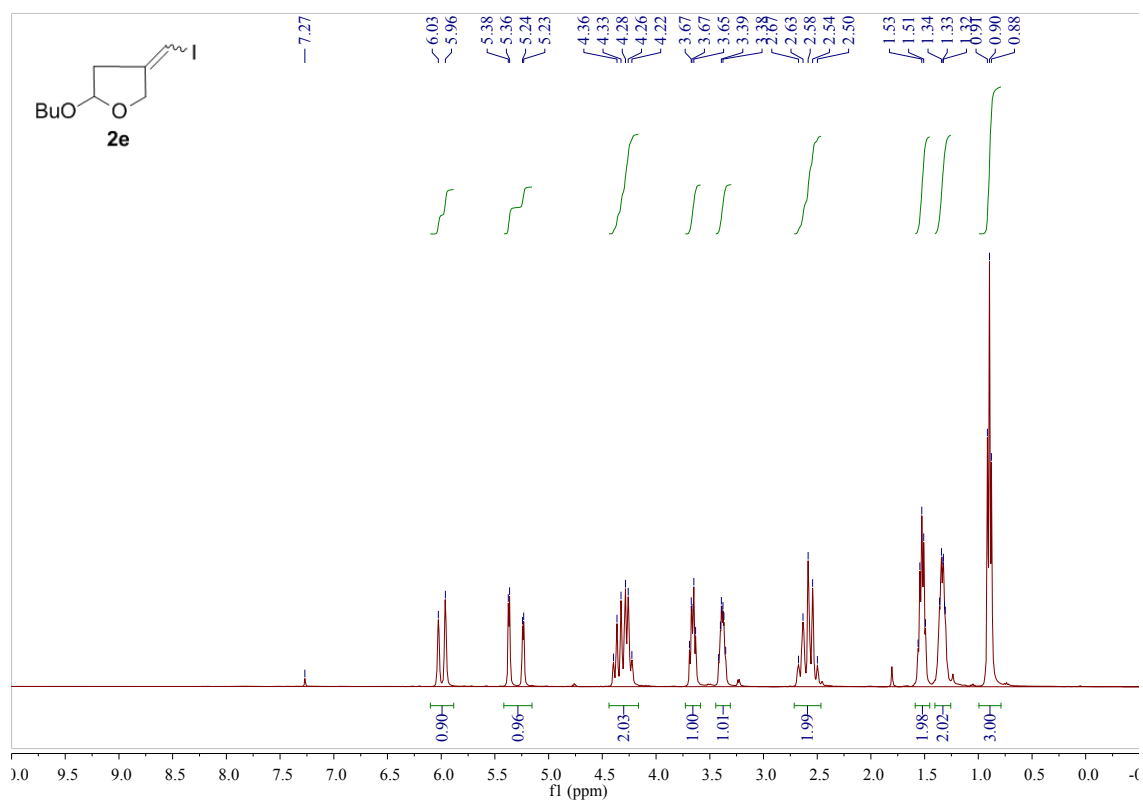
NMR spectra:

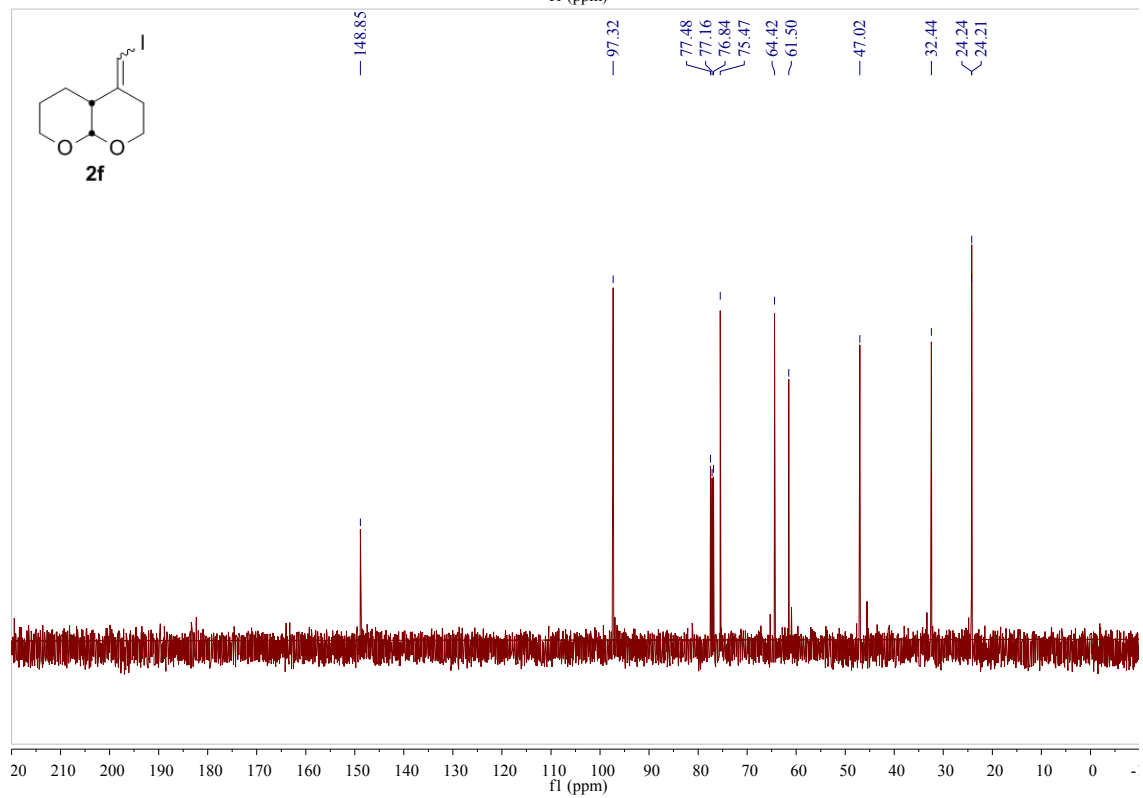
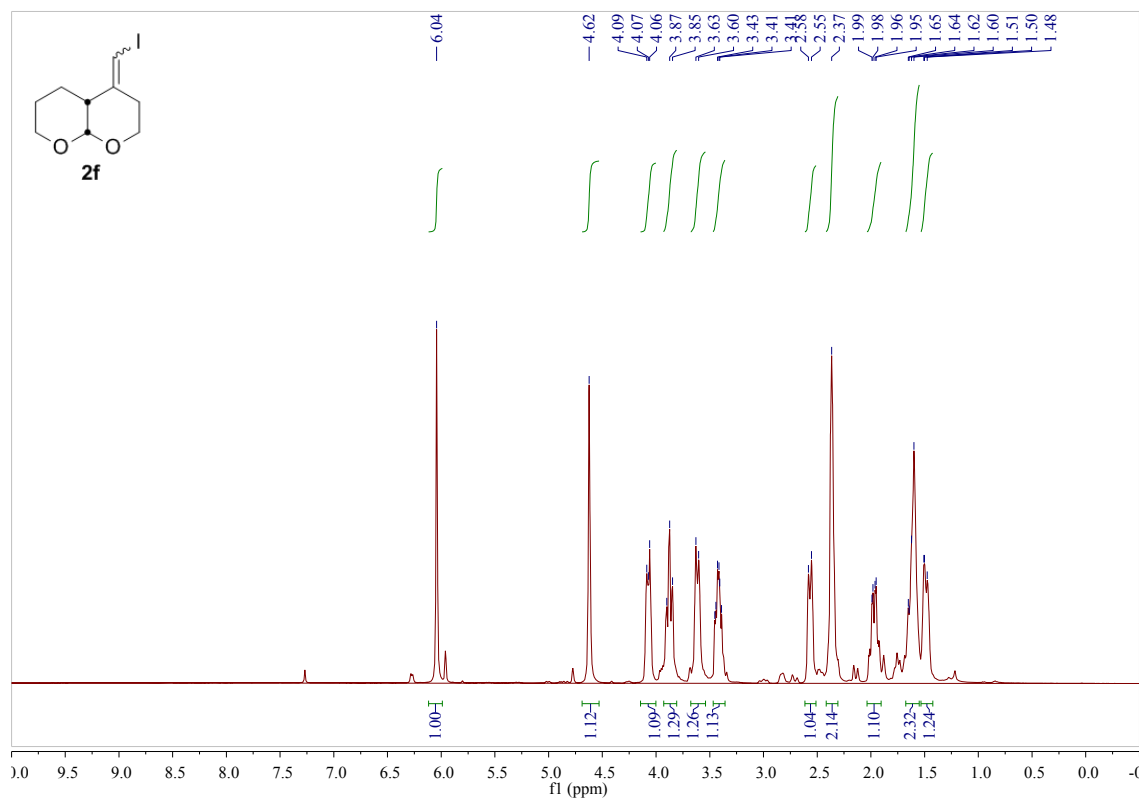


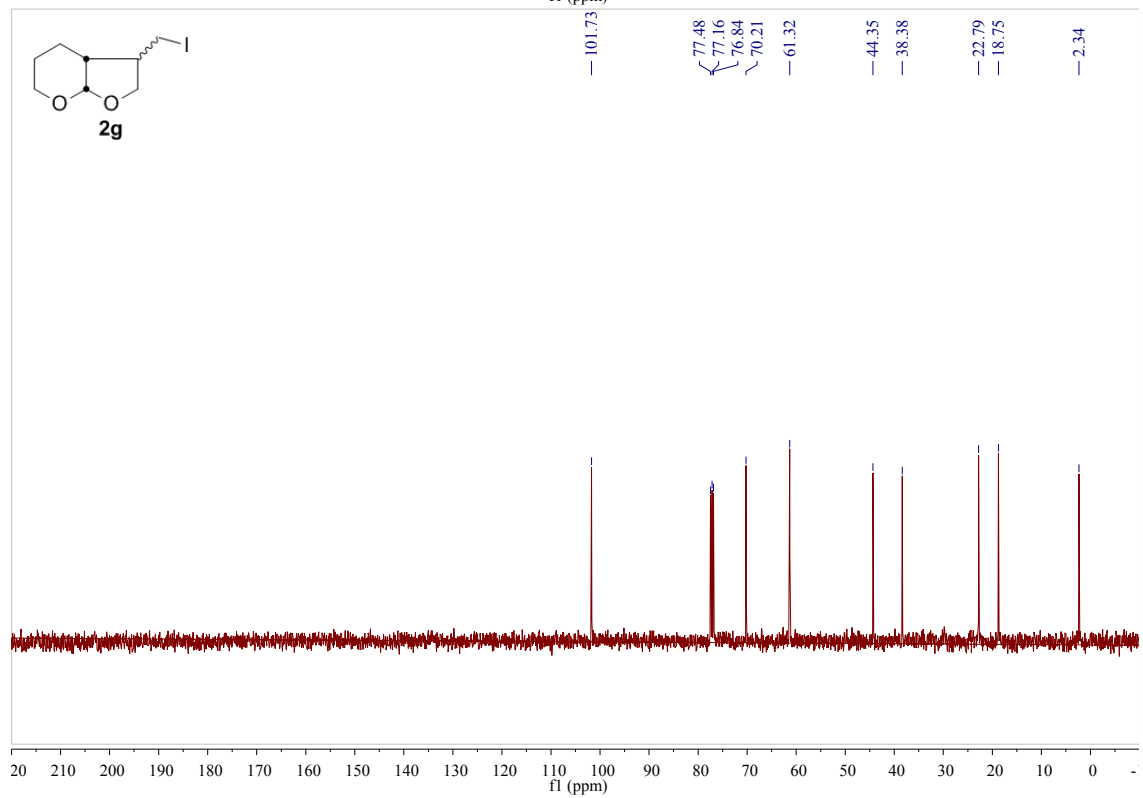
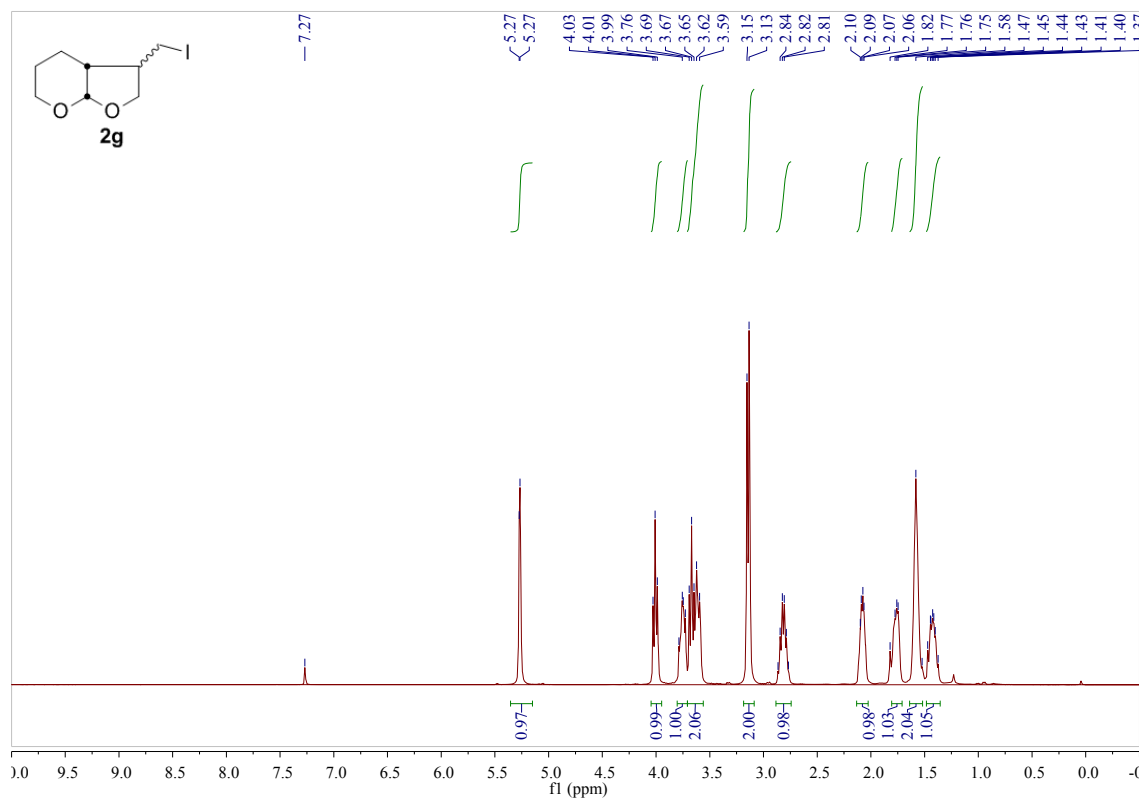


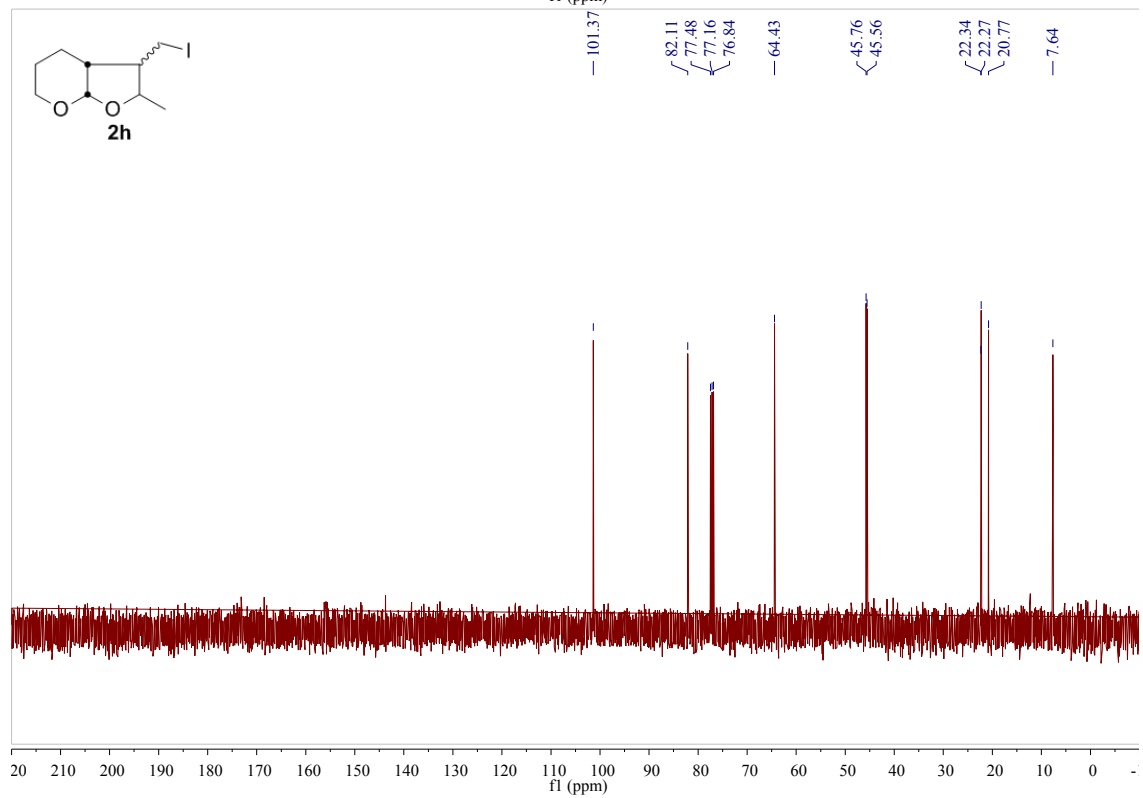
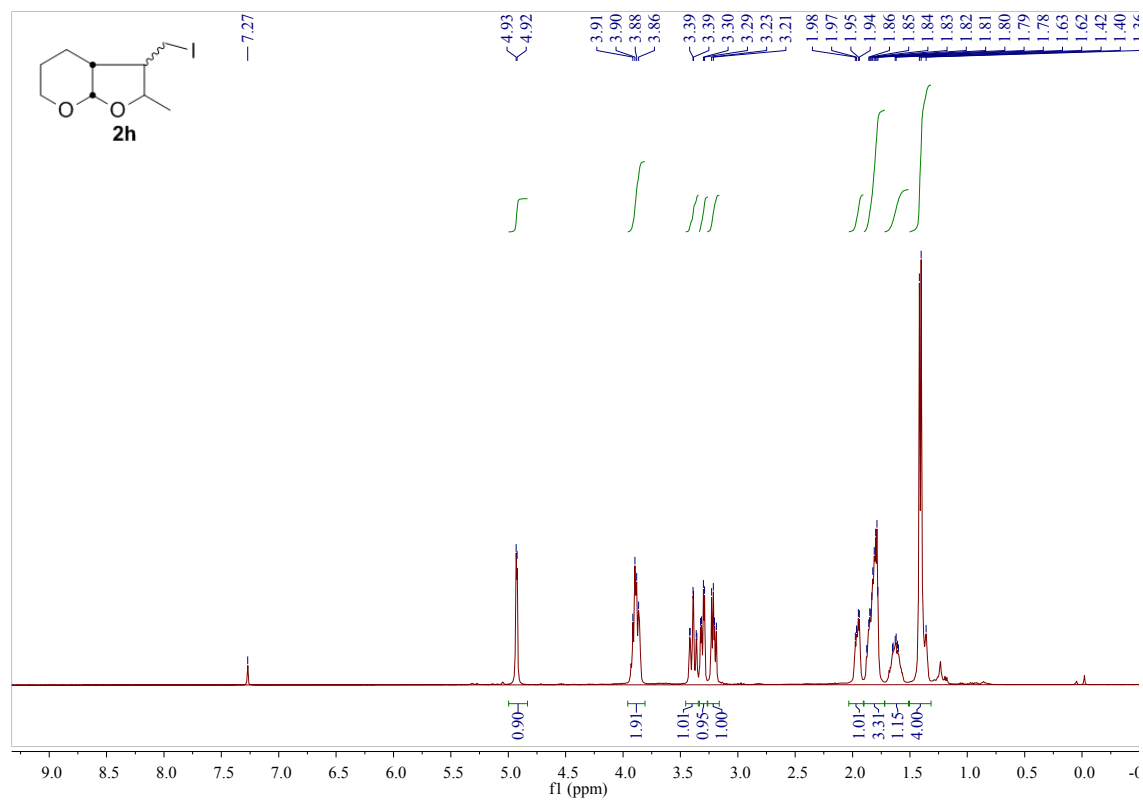


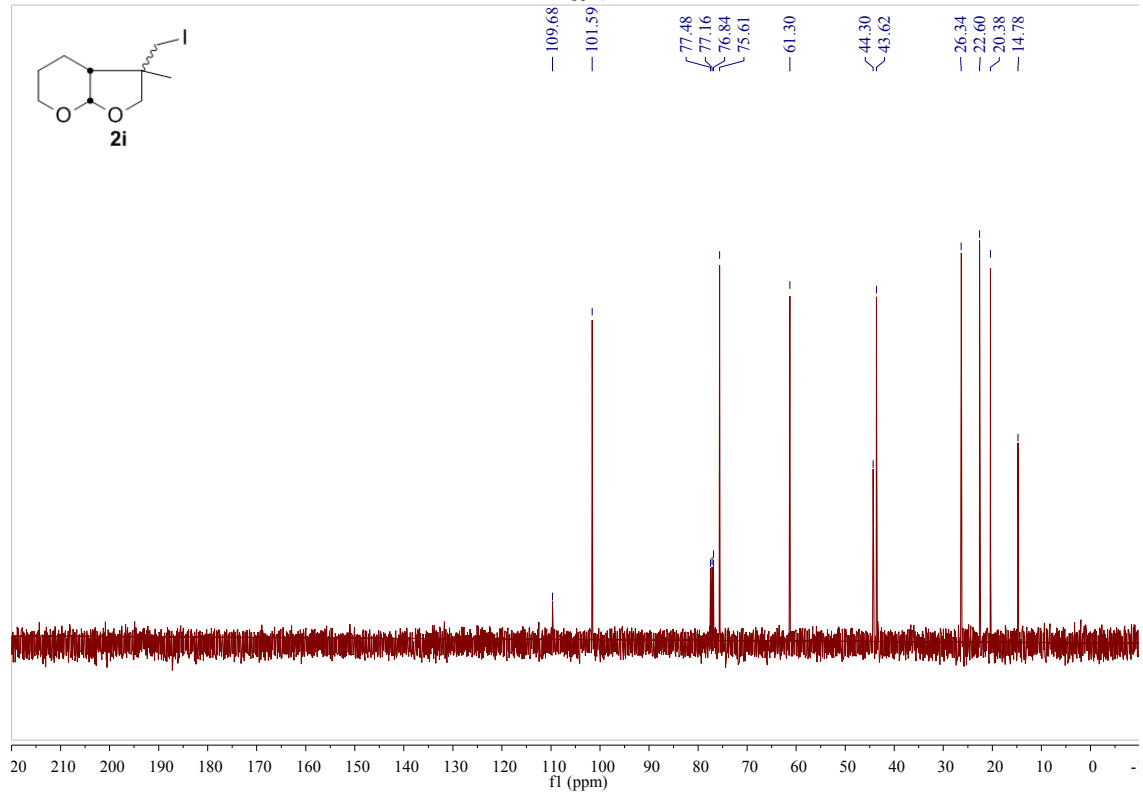
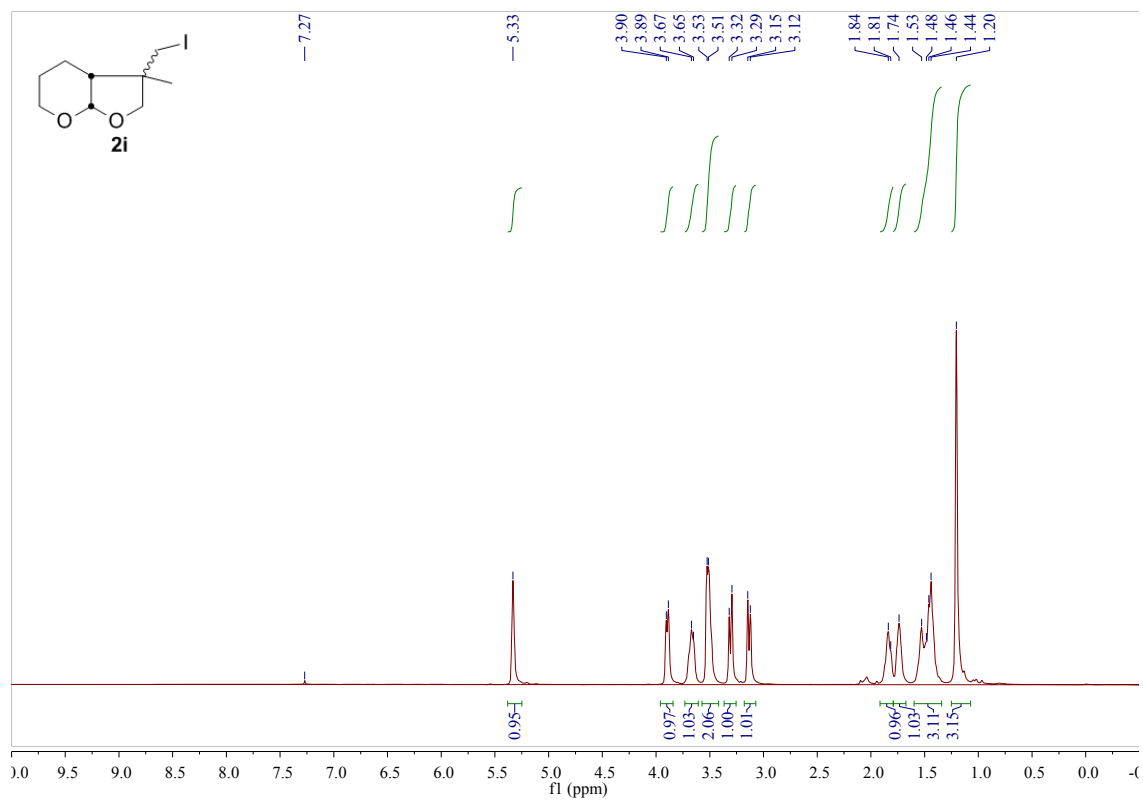


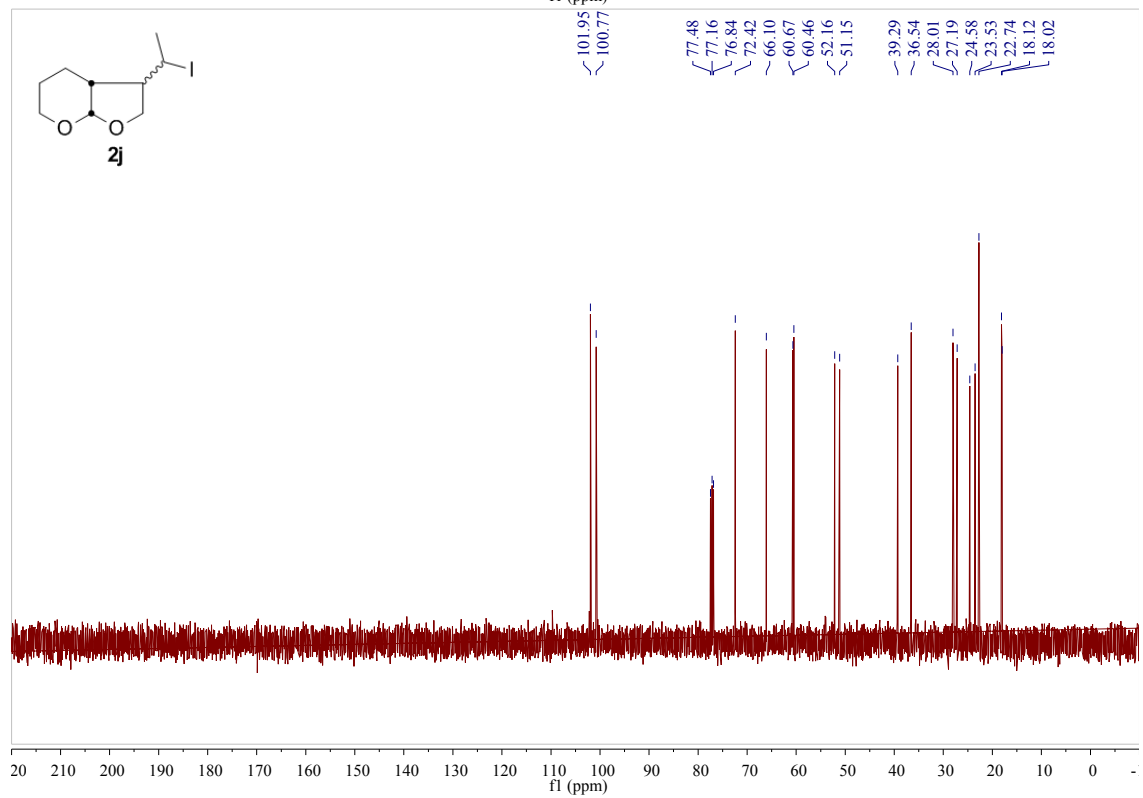
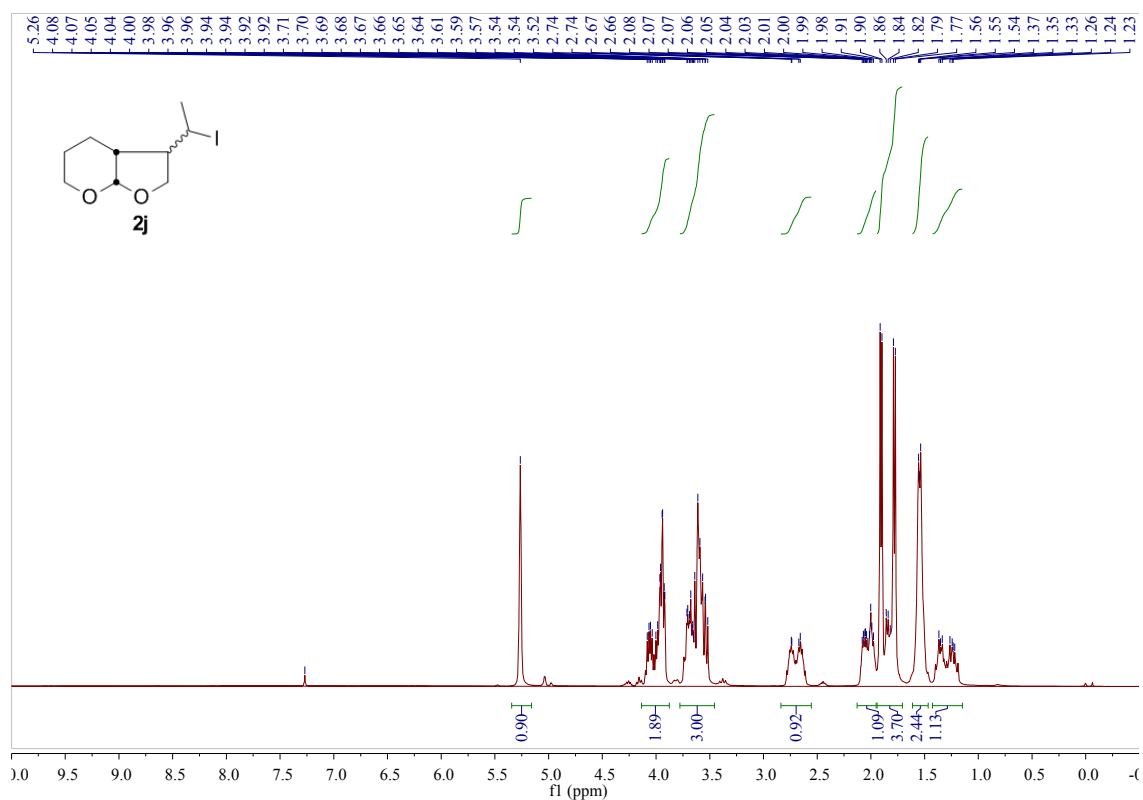


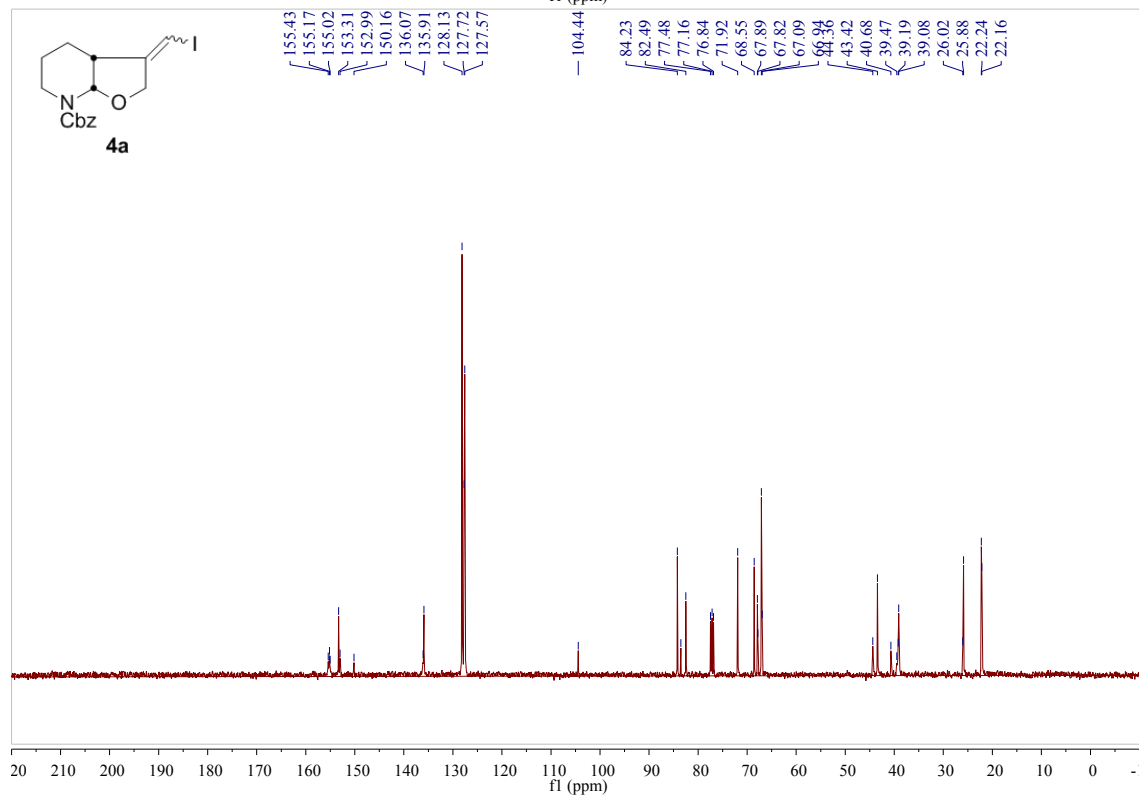
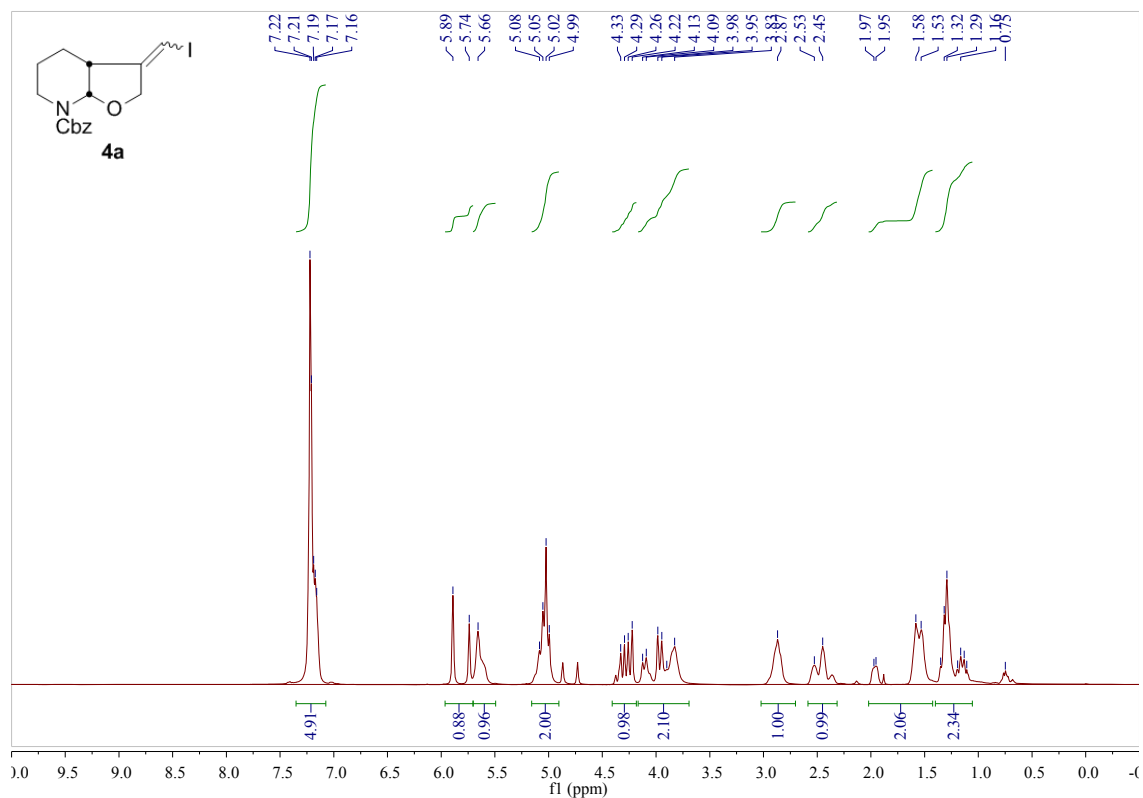


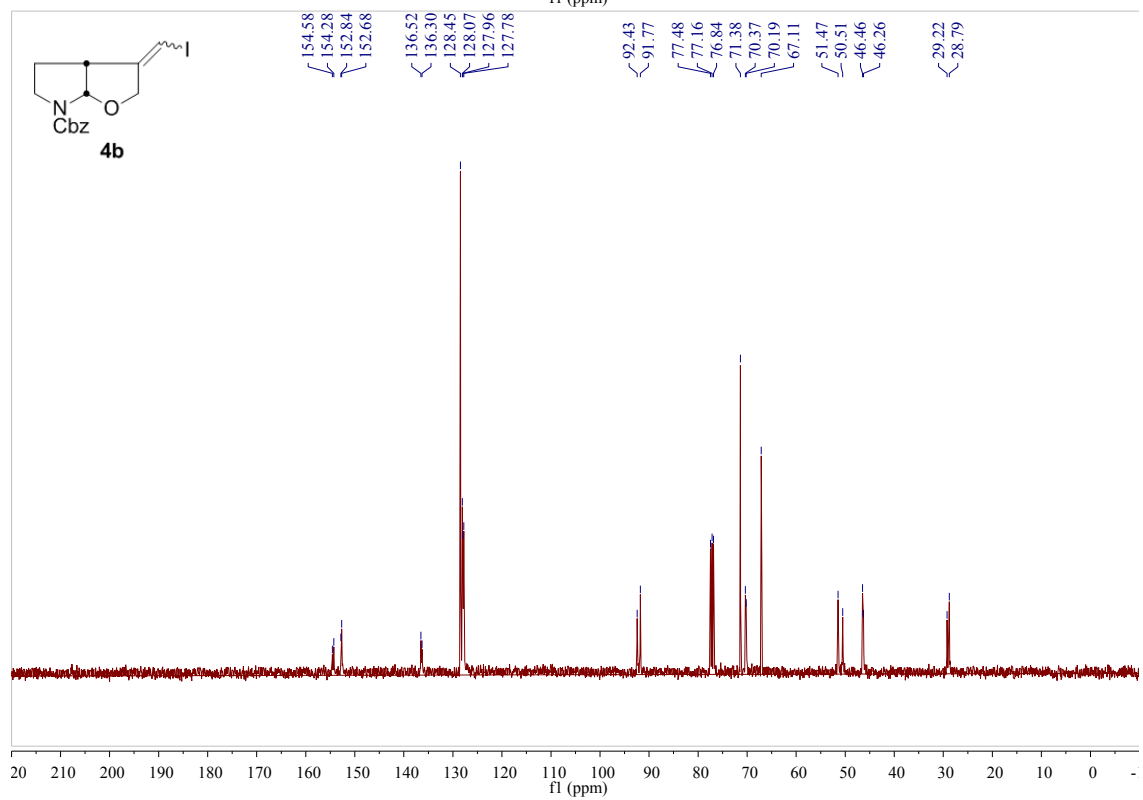
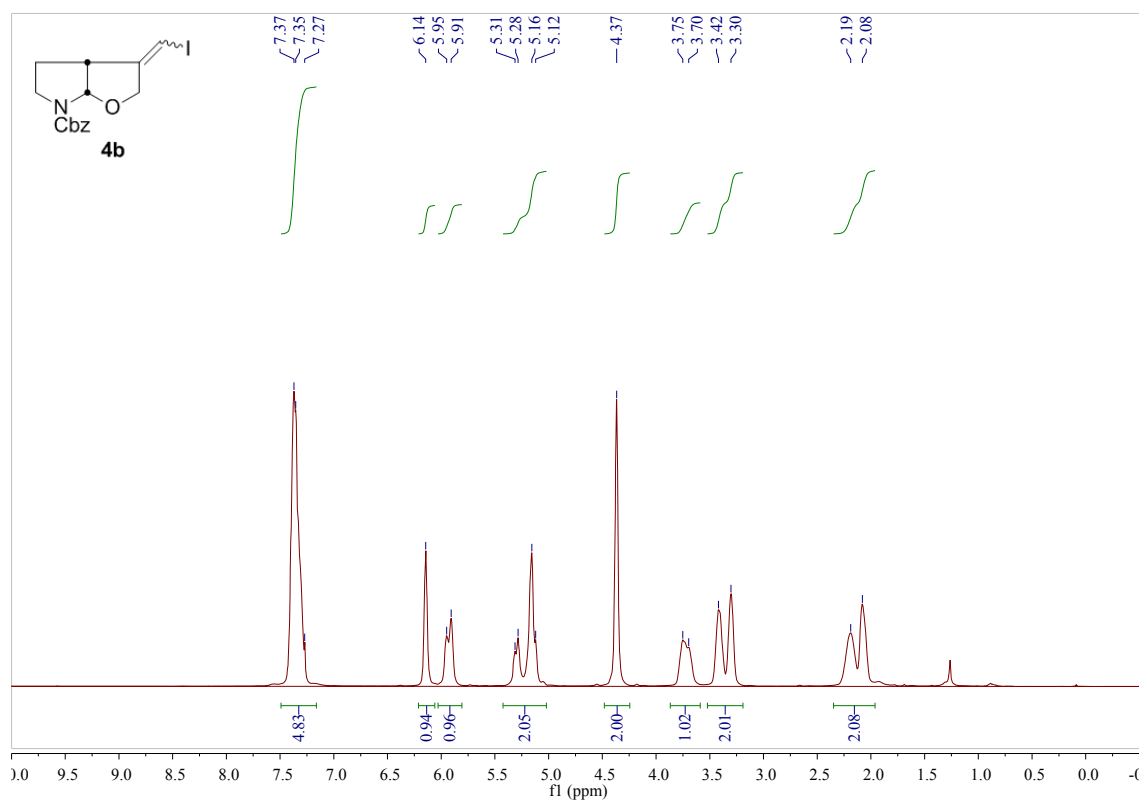


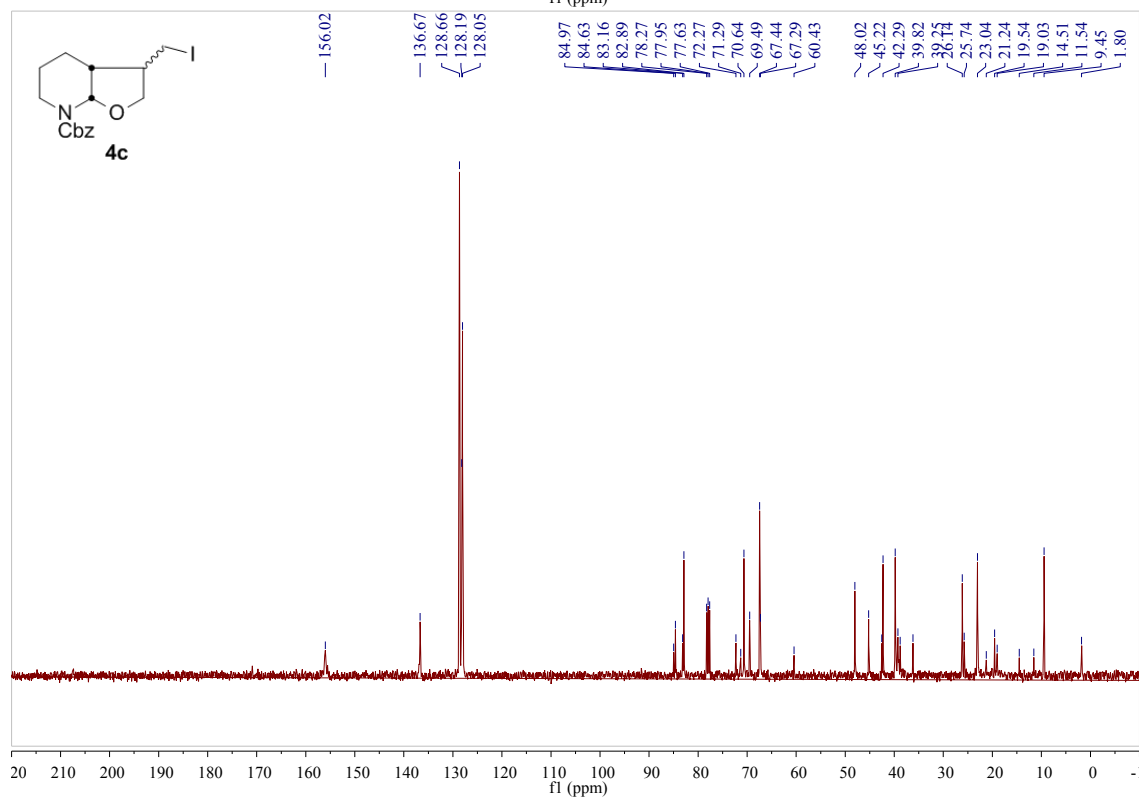
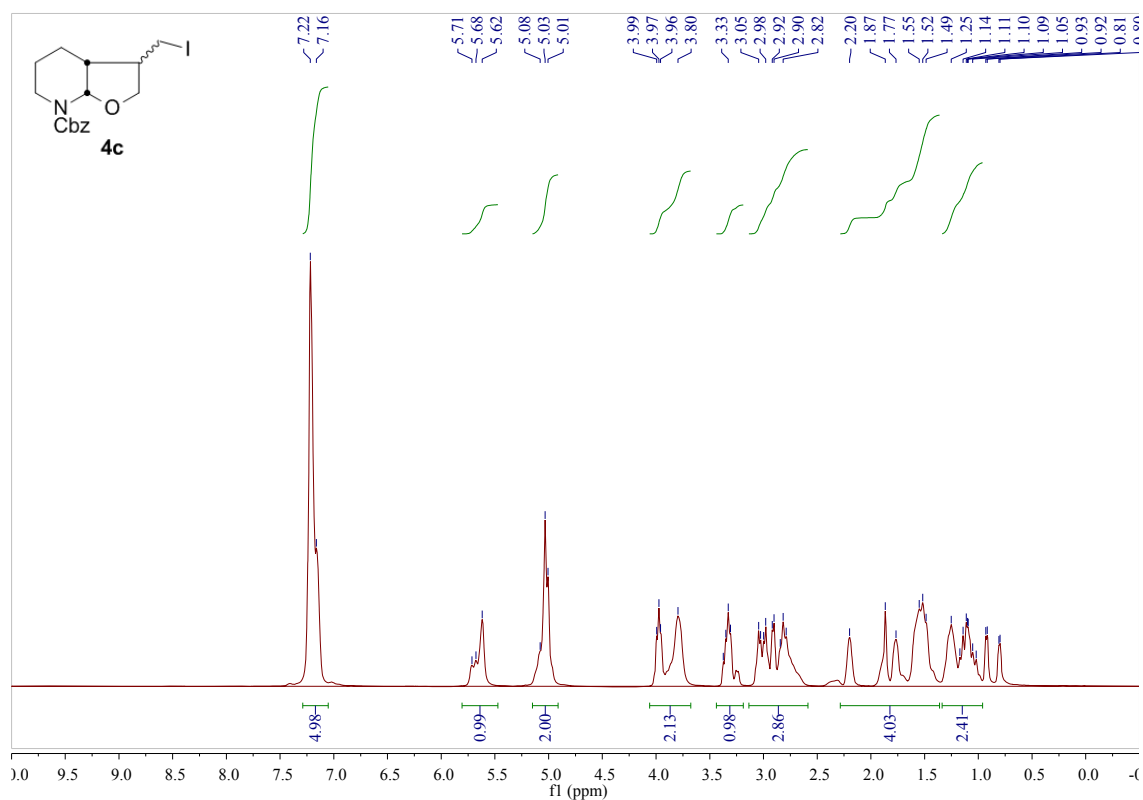


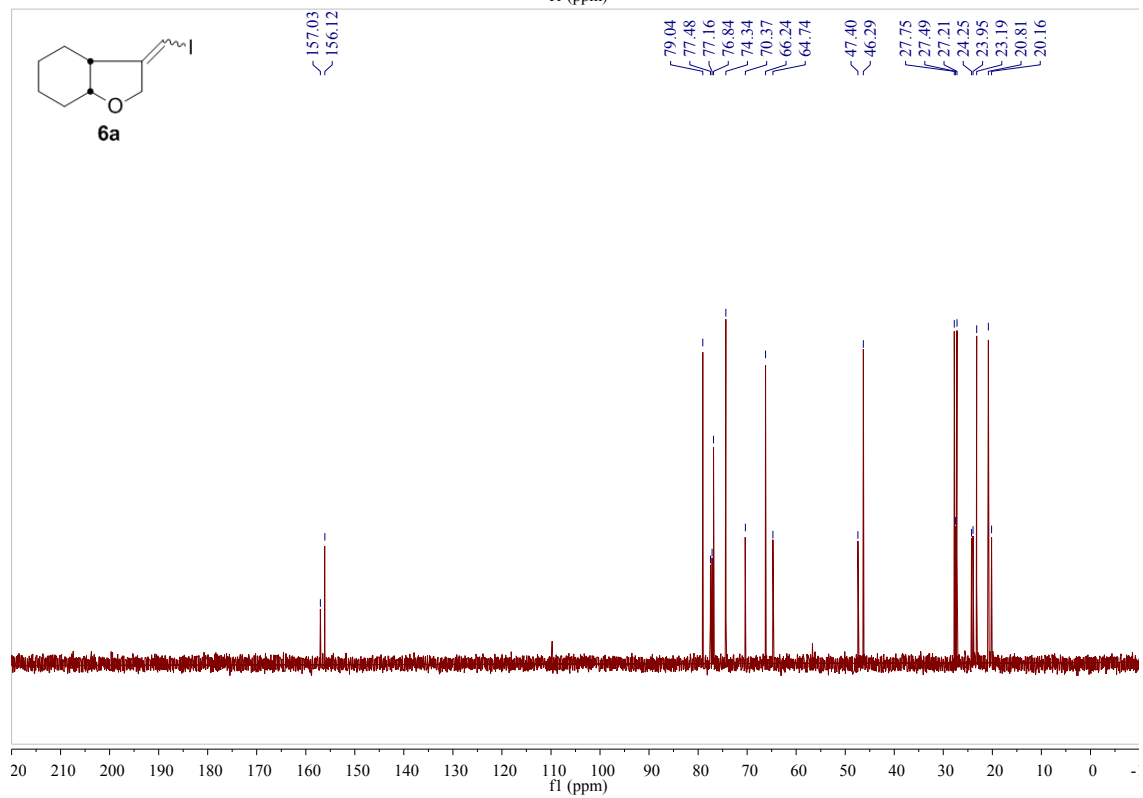
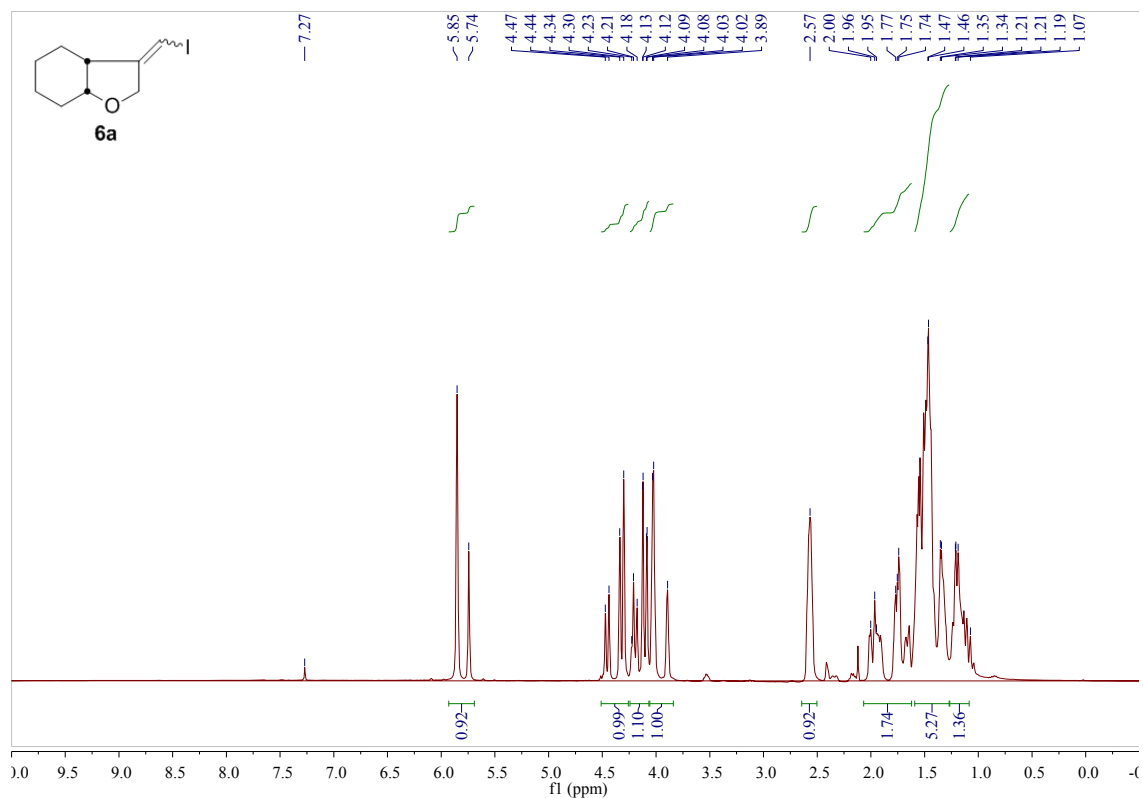


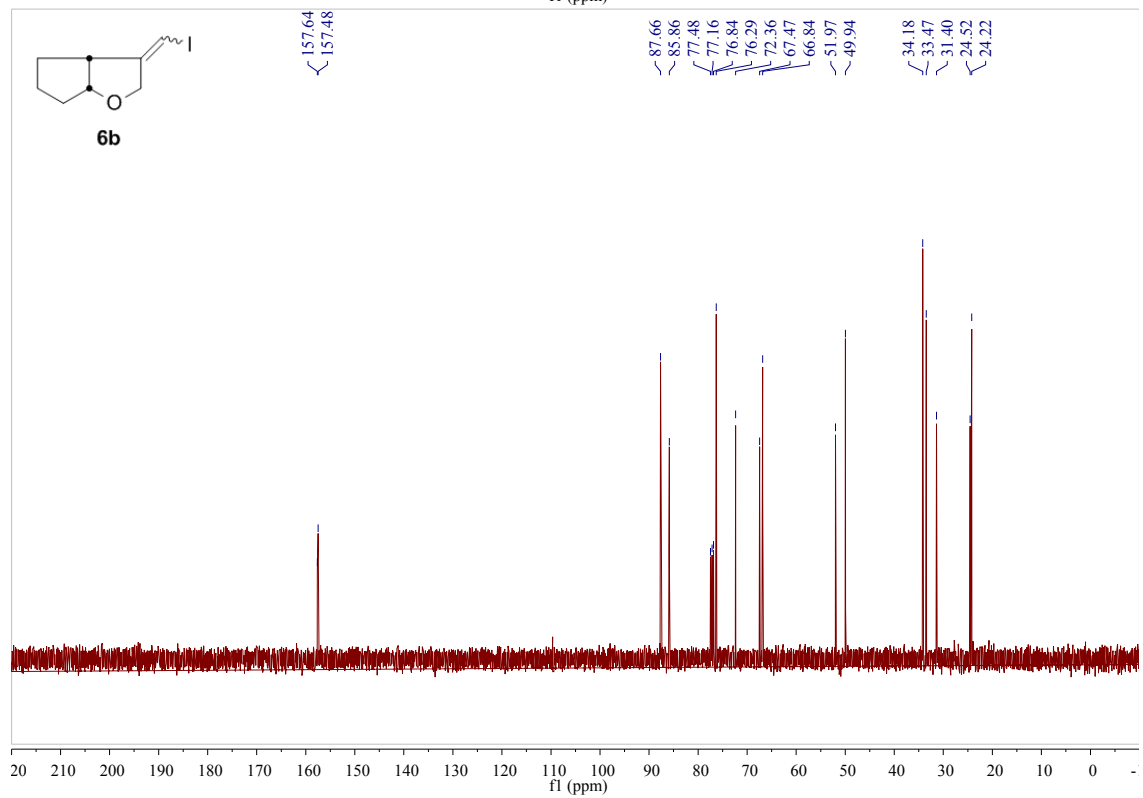
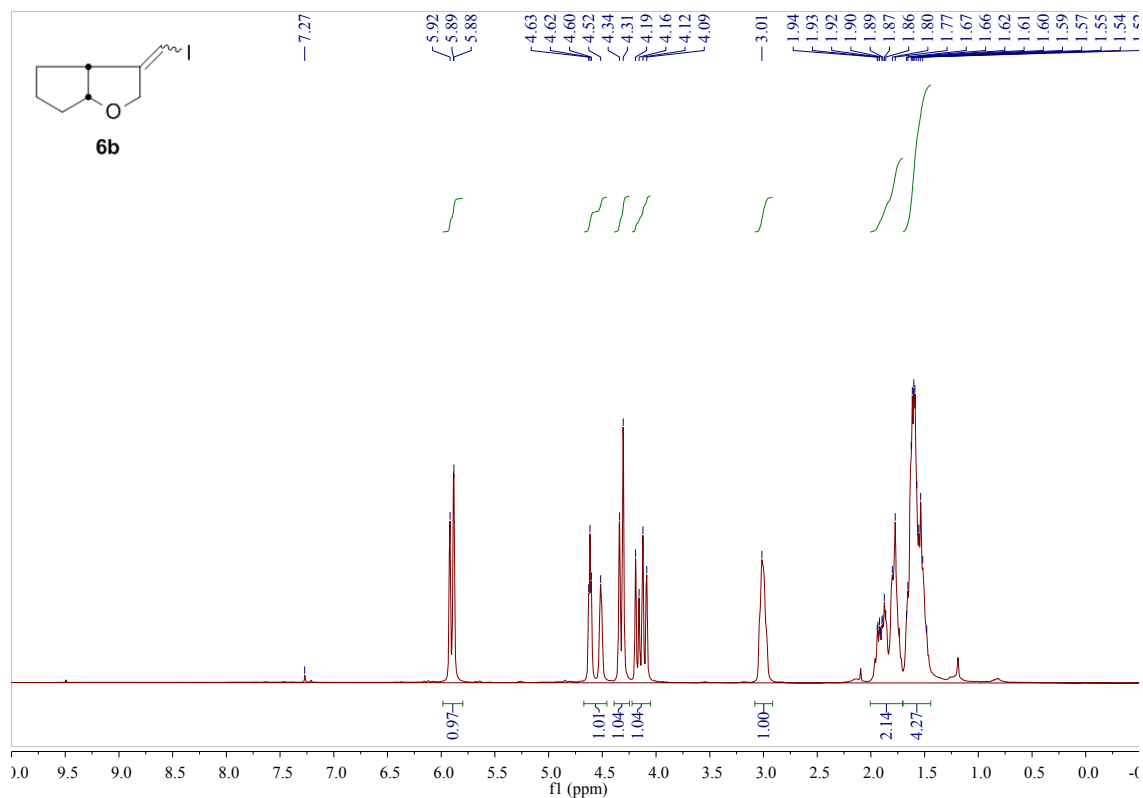


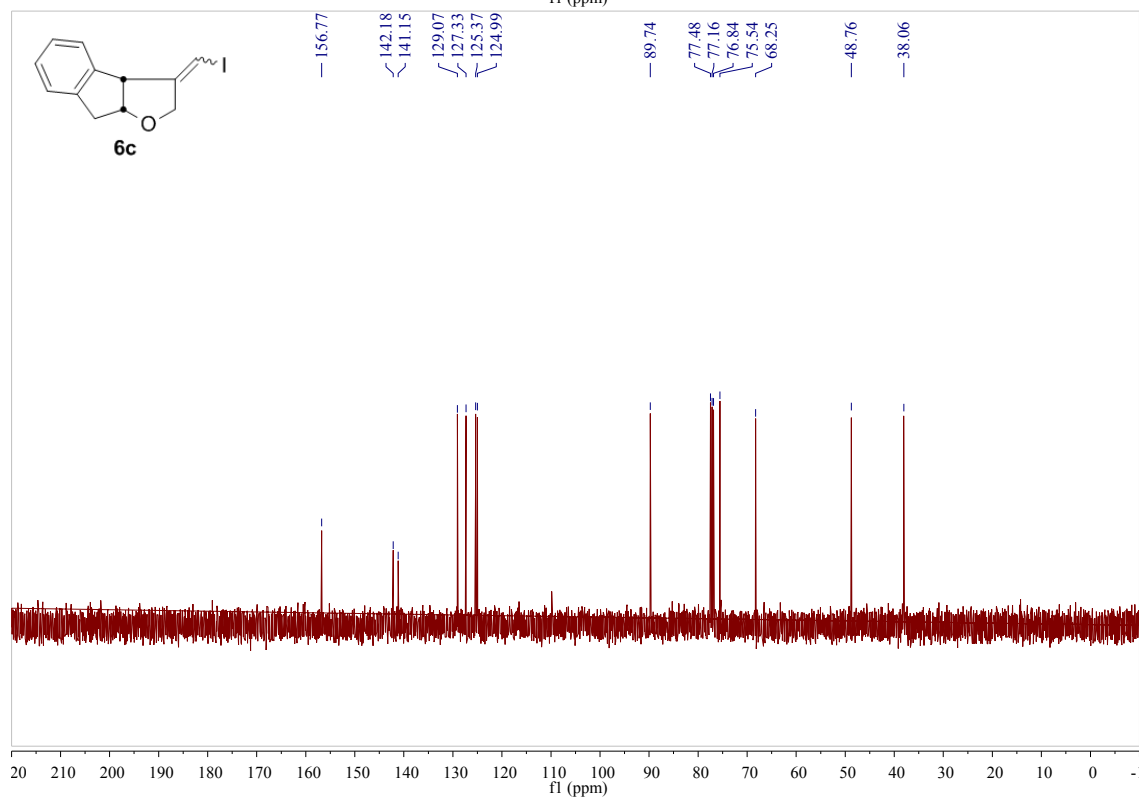
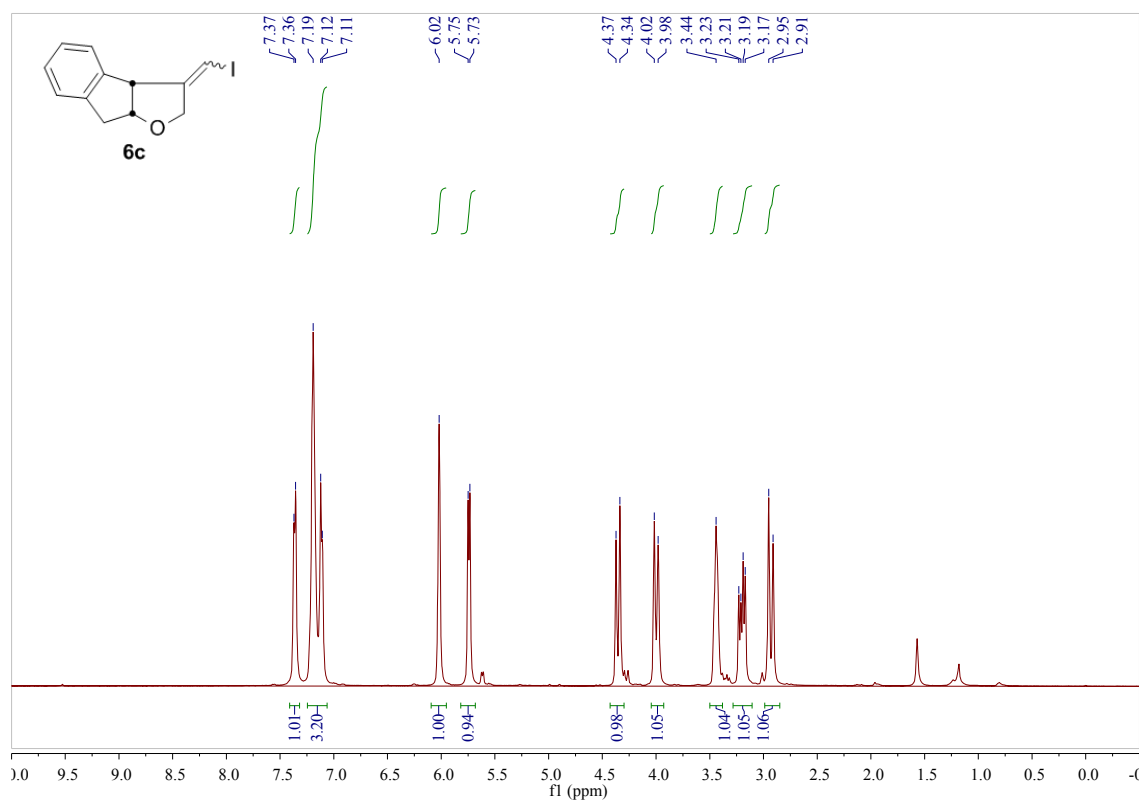


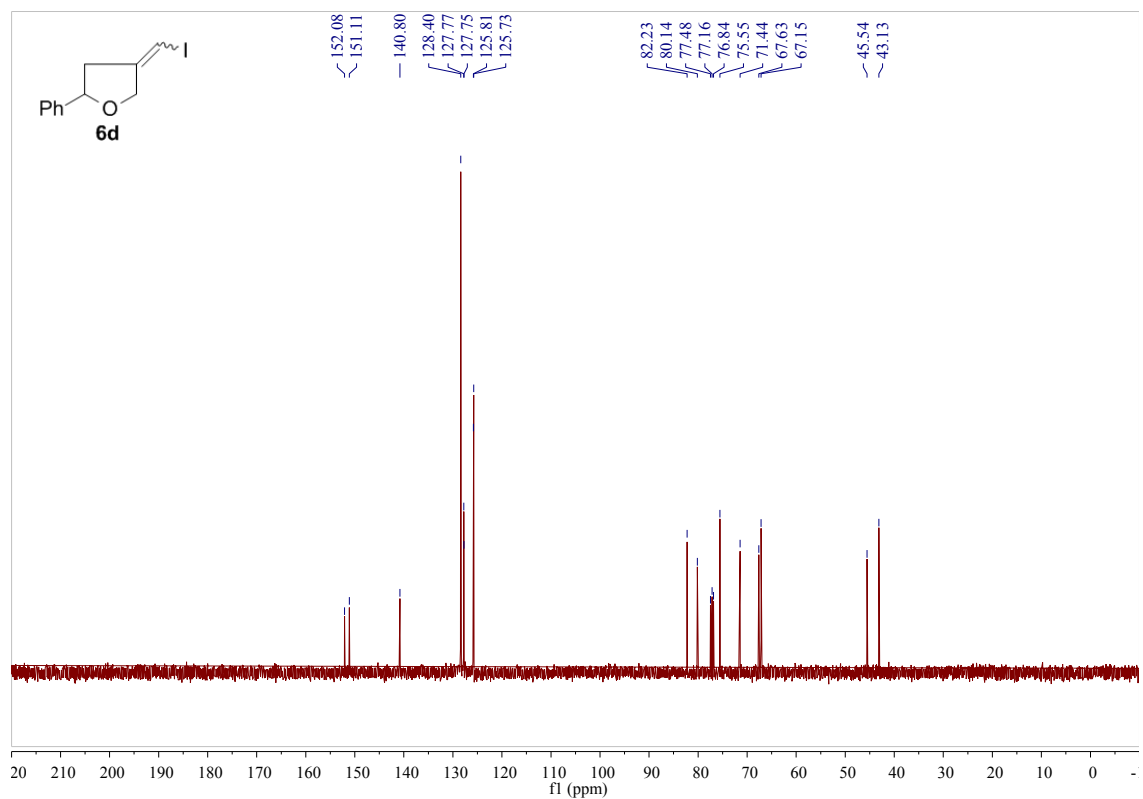
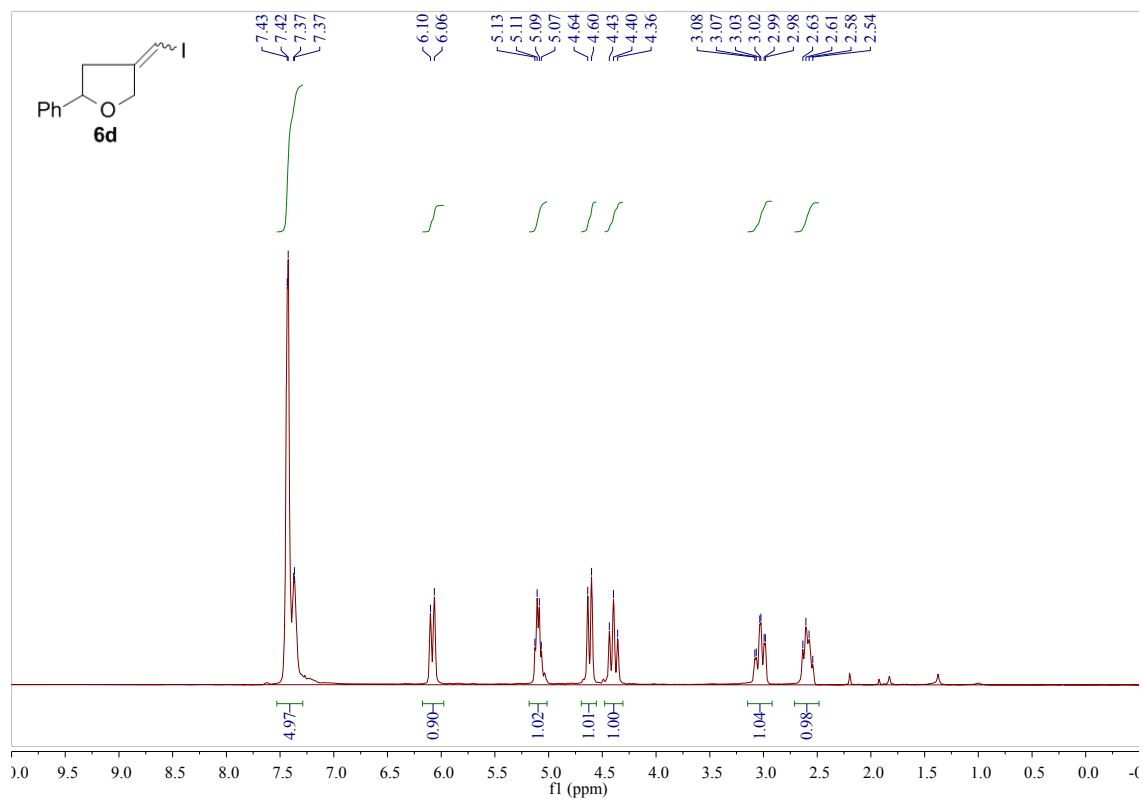












Data for X-ray Crystal Structure of Compound **6c**

Suitable single crystal of compound **6c** was sealed in a thin-walled glass capillary for determination the single-crystal structures. Intensity data were collected with a Rigaku Mercury CCD area detector in ω scan mode using Mo-K α radiation ($\lambda = 0.71070 \text{ \AA}$). The diffracted intensities were corrected for Lorentz/polarization effects and empirical absorption corrections. The structures were solved by direct methods and refined by full-matrix least-squares procedures based on $|F|^2$. The hydrogen atoms in these complexes were generated geometrically, assigned appropriate isotropic thermal parameters, and allowed to ride on their parent carbon atoms. All of the hydrogen atoms were held stationary and included in the structure factor calculation in the final stage of full-matrix least-squares refinement. The structures were solved and refined using SHELEXL-97 programs.

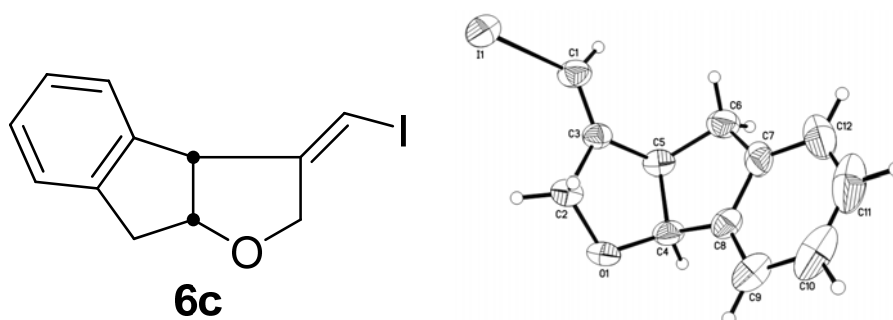


Table 1. Crystal data and structure refinement for shelxl.

Identification code	shelxl
Empirical formula	C12 H11 I O
Formula weight	298.11
Temperature	293(2) K
Wavelength	0.71070 A
Crystal system, space group	Triclinic, P -1
Unit cell dimensions	a = 6.6246(16) A alpha = 107.355(7) deg. b = 8.867(3) A beta = 91.179(5) deg. c = 9.748(3) A gamma = 95.669(7) deg.
Volume	543.1(3) A ³
Z, Calculated density	2, 1.823 Mg/m ³
Absorption coefficient	2.912 mm ⁻¹
F(000)	288
Crystal size	0.55 x 0.20 x 0.15 mm
Theta range for data collection	3.09 to 25.34 deg.

Limiting indices	-7<=h<=7, -10<=k<=9, -10<=l<=11
Reflections collected / unique	5185 / 1964 [R(int) = 0.0283]
Completeness to theta = 25.34	98.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.328 and 0.163
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	1964 / 0 / 128
Goodness-of-fit on F ²	1.009
Final R indices [I>2sigma(I)]	R1 = 0.0321, wR2 = 0.0765
R indices (all data)	R1 = 0.0406, wR2 = 0.0820
Largest diff. peak and hole	0.581 and -0.827 e.A ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for shelxl.

U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
I(1)	4317(1)	692(1)	2817(1)	65(1)
O(1)	10735(4)	2776(4)	5413(3)	56(1)
C(1)	5401(6)	2675(5)	4524(4)	51(1)
C(2)	8934(6)	1882(5)	4655(4)	51(1)
C(3)	7274(5)	2936(4)	5085(4)	39(1)
C(4)	10166(5)	3896(5)	6686(4)	45(1)
C(5)	8070(6)	4354(5)	6351(4)	46(1)
C(6)	6881(6)	4525(5)	7714(5)	57(1)
C(7)	7971(7)	3603(5)	8506(4)	50(1)
C(8)	9816(6)	3239(4)	7921(4)	44(1)
C(9)	11098(7)	2394(5)	8478(5)	61(1)
C(10)	10469(11)	1913(6)	9645(6)	83(2)
C(11)	8653(13)	2283(7)	10225(6)	91(2)
C(12)	7372(9)	3117(6)	9684(5)	73(2)

Table 3. Bond lengths [Å] and angles [deg] for shelxl.

I(1)-C(1)	2.082(4)
O(1)-C(4)	1.423(5)
O(1)-C(2)	1.423(5)
C(1)-C(3)	1.316(5)
C(1)-H(1)	0.9300
C(2)-C(3)	1.499(5)
C(2)-H(2A)	0.9700
C(2)-H(2B)	0.9700
C(3)-C(5)	1.516(5)
C(4)-C(8)	1.498(5)
C(4)-C(5)	1.540(5)
C(4)-H(4)	0.9800
C(5)-C(6)	1.534(5)
C(5)-H(5)	0.9800
C(6)-C(7)	1.502(6)
C(6)-H(6A)	0.9700
C(6)-H(6B)	0.9700
C(7)-C(8)	1.386(6)
C(7)-C(12)	1.394(6)
C(8)-C(9)	1.388(6)
C(9)-C(10)	1.387(7)
C(9)-H(9)	0.9300
C(10)-C(11)	1.367(9)
C(10)-H(10)	0.9300
C(11)-C(12)	1.372(9)
C(11)-H(11)	0.9300
C(12)-H(12)	0.9300

C(4)-O(1)-C(2)	108.1(3)
C(3)-C(1)-I(1)	123.8(3)
C(3)-C(1)-H(1)	118.1
I(1)-C(1)-H(1)	118.1
O(1)-C(2)-C(3)	105.9(3)
O(1)-C(2)-H(2A)	110.6
C(3)-C(2)-H(2A)	110.6
O(1)-C(2)-H(2B)	110.6
C(3)-C(2)-H(2B)	110.6
H(2A)-C(2)-H(2B)	108.7
C(1)-C(3)-C(2)	127.0(4)
C(1)-C(3)-C(5)	124.4(4)
C(2)-C(3)-C(5)	108.5(3)
O(1)-C(4)-C(8)	114.3(3)
O(1)-C(4)-C(5)	107.3(3)
C(8)-C(4)-C(5)	103.7(3)
O(1)-C(4)-H(4)	110.4
C(8)-C(4)-H(4)	110.4
C(5)-C(4)-H(4)	110.4
C(3)-C(5)-C(6)	114.3(3)
C(3)-C(5)-C(4)	101.4(3)
C(6)-C(5)-C(4)	106.3(3)
C(3)-C(5)-H(5)	111.5
C(6)-C(5)-H(5)	111.5
C(4)-C(5)-H(5)	111.5
C(7)-C(6)-C(5)	103.6(3)
C(7)-C(6)-H(6A)	111.0
C(5)-C(6)-H(6A)	111.0
C(7)-C(6)-H(6B)	111.0
C(5)-C(6)-H(6B)	111.0

H(6A)-C(6)-H(6B)	109.0
C(8)-C(7)-C(12)	119.7(5)
C(8)-C(7)-C(6)	111.2(3)
C(12)-C(7)-C(6)	129.1(4)
C(7)-C(8)-C(9)	121.6(4)
C(7)-C(8)-C(4)	110.7(3)
C(9)-C(8)-C(4)	127.6(4)
C(10)-C(9)-C(8)	117.8(5)
C(10)-C(9)-H(9)	121.1
C(8)-C(9)-H(9)	121.1
C(11)-C(10)-C(9)	120.5(5)
C(11)-C(10)-H(10)	119.8
C(9)-C(10)-H(10)	119.8
C(10)-C(11)-C(12)	122.4(5)
C(10)-C(11)-H(11)	118.8
C(12)-C(11)-H(11)	118.8
C(11)-C(12)-C(7)	118.1(5)
C(11)-C(12)-H(12)	120.9
C(7)-C(12)-H(12)	120.9

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{Å}^2 \times 10^3$) for shelxl.

The anisotropic displacement factor exponent takes the form: $-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h$
 $k a^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
I(1)	50(1)	70(1)	63(1)	5(1)	-6(1)	-1(1)
O(1)	28(1)	82(2)	57(2)	19(2)	7(1)	6(1)
C(1)	38(2)	49(2)	62(2)	12(2)	-1(2)	9(2)
C(2)	35(2)	64(3)	51(2)	12(2)	4(2)	9(2)
C(3)	34(2)	42(2)	44(2)	19(2)	2(2)	2(2)
C(4)	31(2)	50(2)	52(2)	17(2)	-2(2)	-4(2)
C(5)	39(2)	43(2)	56(2)	18(2)	0(2)	3(2)
C(6)	42(2)	57(3)	61(3)	2(2)	4(2)	7(2)
C(7)	58(3)	41(2)	39(2)	-3(2)	3(2)	-8(2)
C(8)	43(2)	40(2)	45(2)	10(2)	-8(2)	-6(2)
C(9)	64(3)	52(3)	64(3)	20(2)	-21(2)	-3(2)
C(10)	122(5)	53(3)	71(4)	25(3)	-45(3)	-13(3)
C(11)	144(7)	74(4)	48(3)	23(3)	-11(3)	-35(4)
C(12)	92(4)	66(3)	50(3)	6(2)	18(3)	-19(3)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{Å}^2 \times 10^3$) for shelxl.

	x	y	z	U(eq)
H(1)	4510	3420	4913	61
H(2A)	8637	913	4916	61
H(2B)	9084	1602	3624	61
H(4)	11168	4838	6973	54
H(5)	8194	5340	6086	55
H(6A)	6919	5632	8282	68
H(6B)	5476	4078	7474	68
H(9)	12336	2158	8082	73
H(10)	11287	1335	10036	100
H(11)	8273	1958	11015	109
H(12)	6140	3350	10092	88

Table 6. Torsion angles [deg] for shelxl.

C(4)-O(1)-C(2)-C(3)	26.3(4)
I(1)-C(1)-C(3)-C(2)	-1.7(6)
I(1)-C(1)-C(3)-C(5)	-178.4(3)
O(1)-C(2)-C(3)-C(1)	171.5(4)
O(1)-C(2)-C(3)-C(5)	-11.4(4)
C(2)-O(1)-C(4)-C(8)	83.3(4)
C(2)-O(1)-C(4)-C(5)	-31.2(4)
C(1)-C(3)-C(5)-C(6)	57.0(5)
C(2)-C(3)-C(5)-C(6)	-120.2(4)
C(1)-C(3)-C(5)-C(4)	170.9(4)
C(2)-C(3)-C(5)-C(4)	-6.4(4)
O(1)-C(4)-C(5)-C(3)	22.3(4)
C(8)-C(4)-C(5)-C(3)	-99.1(3)
O(1)-C(4)-C(5)-C(6)	142.0(3)
C(8)-C(4)-C(5)-C(6)	20.7(4)
C(3)-C(5)-C(6)-C(7)	90.7(4)
C(4)-C(5)-C(6)-C(7)	-20.2(4)
C(5)-C(6)-C(7)-C(8)	12.5(4)
C(5)-C(6)-C(7)-C(12)	-167.8(4)
C(12)-C(7)-C(8)-C(9)	0.1(6)
C(6)-C(7)-C(8)-C(9)	179.8(4)
C(12)-C(7)-C(8)-C(4)	-179.1(4)
C(6)-C(7)-C(8)-C(4)	0.7(4)
O(1)-C(4)-C(8)-C(7)	-130.0(3)
C(5)-C(4)-C(8)-C(7)	-13.5(4)
O(1)-C(4)-C(8)-C(9)	50.9(5)
C(5)-C(4)-C(8)-C(9)	167.4(4)
C(7)-C(8)-C(9)-C(10)	0.3(6)

C(4)-C(8)-C(9)-C(10)	179.3(4)
C(8)-C(9)-C(10)-C(11)	-0.7(7)
C(9)-C(10)-C(11)-C(12)	0.8(8)
C(10)-C(11)-C(12)-C(7)	-0.4(8)
C(8)-C(7)-C(12)-C(11)	0.0(6)
C(6)-C(7)-C(12)-C(11)	-179.7(5)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for shelxl [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
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