

Supporting Information

Cu-Catalyzed Photoredox Chlorosulfonation of Alkenes and Alkynes

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Experimental Procedure for Photoredox Catalysis

Large Scale Reaction

A Schlenk flask (100 mL) was charged with a magnetic stirring bar, catalyst **1** (120 mg, 2.5 mol%), N-[4-cyano-3-(trifluoromethyl)phenyl]-2-methylacrylamide (1.27 g, 5 mmol) and transferred into a glovebox. Solvents (DCM:MeCN, 12.5:12.5 mL) and 4-fluorobenzenesulfonyl chloride (0.973 g, 5mmol) were added. The Schlenk flask was closed with a septum and transferred outside the glovebox. The reaction mixture was stirred for 24h at rt, irradiated by two 40 W blue LEDs from Kessil Co., Ltd (ca. 10 cm distance) and cooled by two fans.

Optimization

In order to optimize the reaction conditions, parameters were changed as described and the conversion of alkene was analyzed via the ratio of starting material to product in the ^1H NMR spectra. To verify the ^1H NMR conversions, the most promising conditions were further purified to get the isolated yields.

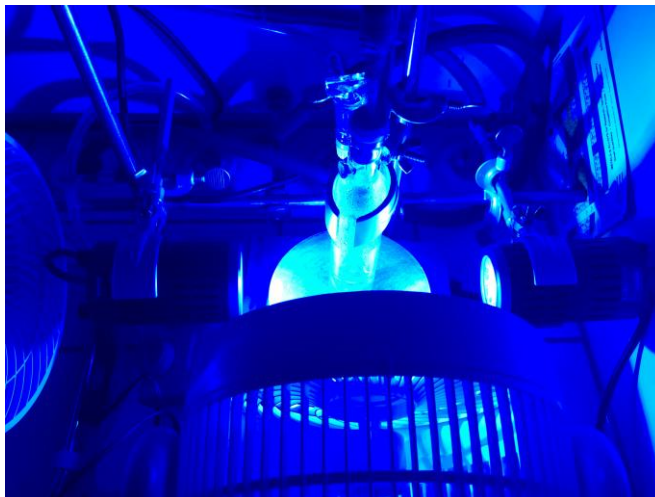


Figure S1. Large scale setup of photoredox catalyzed sulfonation of **2** using sulfonyl chloride **3**.

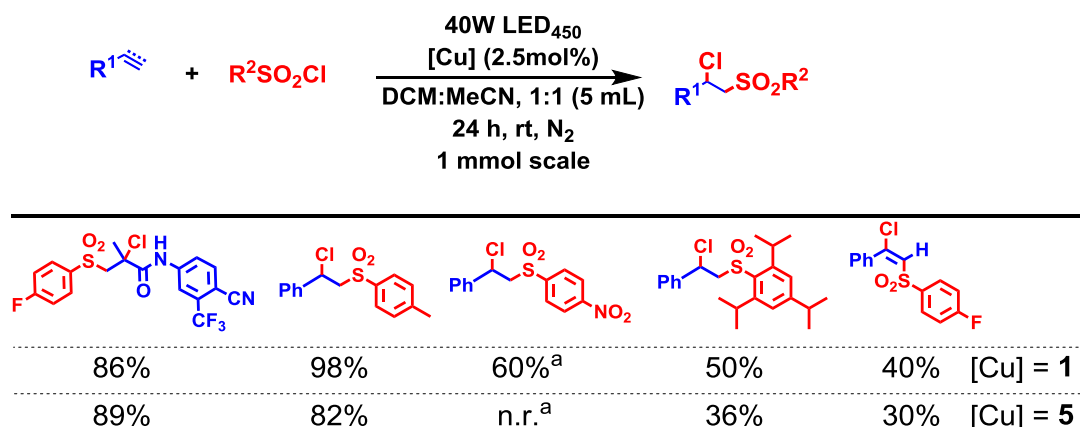
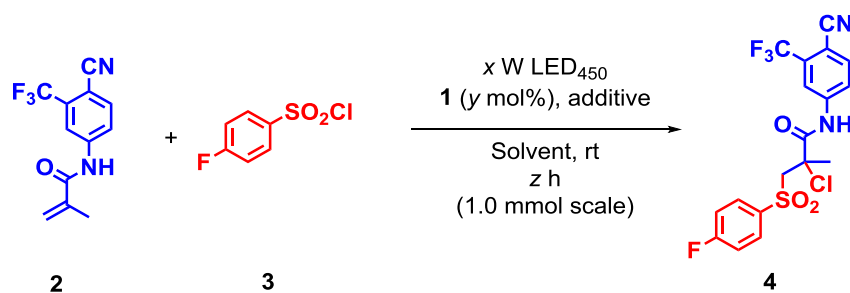


Figure S2. Comparison of catalytic activities using **PC 1** or **5**. ^a2W LED₄₅₀, 1 (1.0 mol%), DCM (3 mL), 24h, rt, N₂ atm., 1 mmol scale.

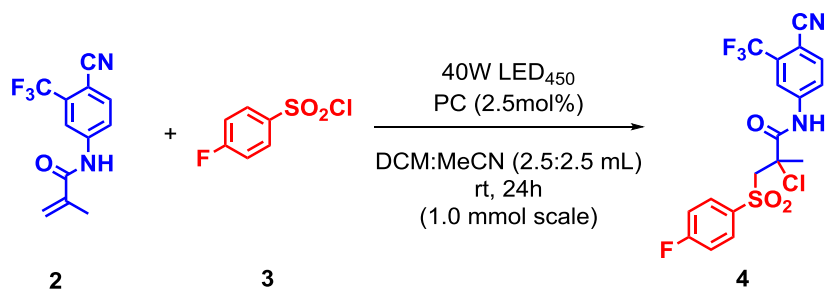
Table S1. Control reactions with standard photoredox catalysts.



Entry	2:3	x	y	Additive	Solvent	z	Conversion ^a (yield) ^b [%]
1	2:1	2	2.5	-	DCM (3 mL)	40	45 (n.d.)
2	3:1	2	2.5	-	DCM (3 mL)	40	40 (n.d.)
3	1:2	2	2.5	-	DCM (3 mL)	24	15 (n.d.)
4	1:3	2	2.5	-	DCM (3 mL)	24	50 (20)
5	1:1	40	2.5	ZnCl ₂ (20 mol%)	DCM (3 mL)	24	n.r. (n.d.)
6	1:1	40	2.5	KBr (20 mol%)	DCM (3 mL)	24	70 (n.d.)
7	1:1	40	2.5	K ₂ CO ₃ (1 equiv.)	DCM (3 mL)	24	30 (n.d.)
8	1:1	40	2.5	KBr (20 mol%)	DCM+MeCN (1.5+1.5 mL)	66	80 (72)
9	1:1	40	2.5	KBr (20 mol%)	DCM+MeCN (2.5+2.5 mL)	66	70 (n.d.)
10	1:1	2	2.5	-	DCM+MeCN (2.5+2.5 mL)	24	5 (n.d.)

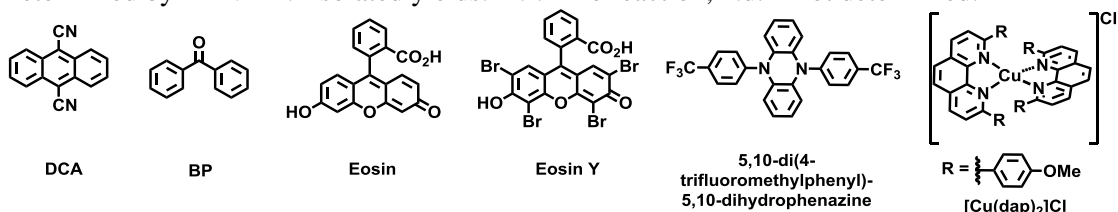
^aDetermined by ¹H NMR. ^bIsolated yields. ^cn.r. = no reaction, n.d. = not determined.

Table S2. Control experiments and reactions using other photoredox catalysts.



Entry	PC	conversion ^a (yield ^b) [%]
1	1 - dark	n.r. ^c
2	-	n.r.
3	1 – no N ₂ atm.	25 (n.d.)
4	[Cu(MeCN) ₄]PF ₆	n.r.
5	[Cu(dap) ₂]Cl (5)	100 (89)
6	Eosin	n.r.
7	Eosin Y	n.r.
8	DCA	n.r.
9	BP	n.r.
10	5,10-Di(4-trifluoromethylphenyl)-5,10-dihydrophenazine	50 (43)
11	[Ru(bpy) ₃][PF ₆] ₂ • 6H ₂ O	Trace amounts
12	[Ru(phen) ₃][PF ₆] ₂ • xH ₂ O	25 (n.d.)
13	[Ir(dtbbpy)(ppy) ₂]PF ₆	100 (42)
14	AIBN (10 mol%)	n.r.

^aDetermined by ¹H NMR. ^bIsolated yields. ^cn.r. = no reaction, n.d. = not determined.



Characterization Data

Single Crystal X-Ray Structure of **12d**

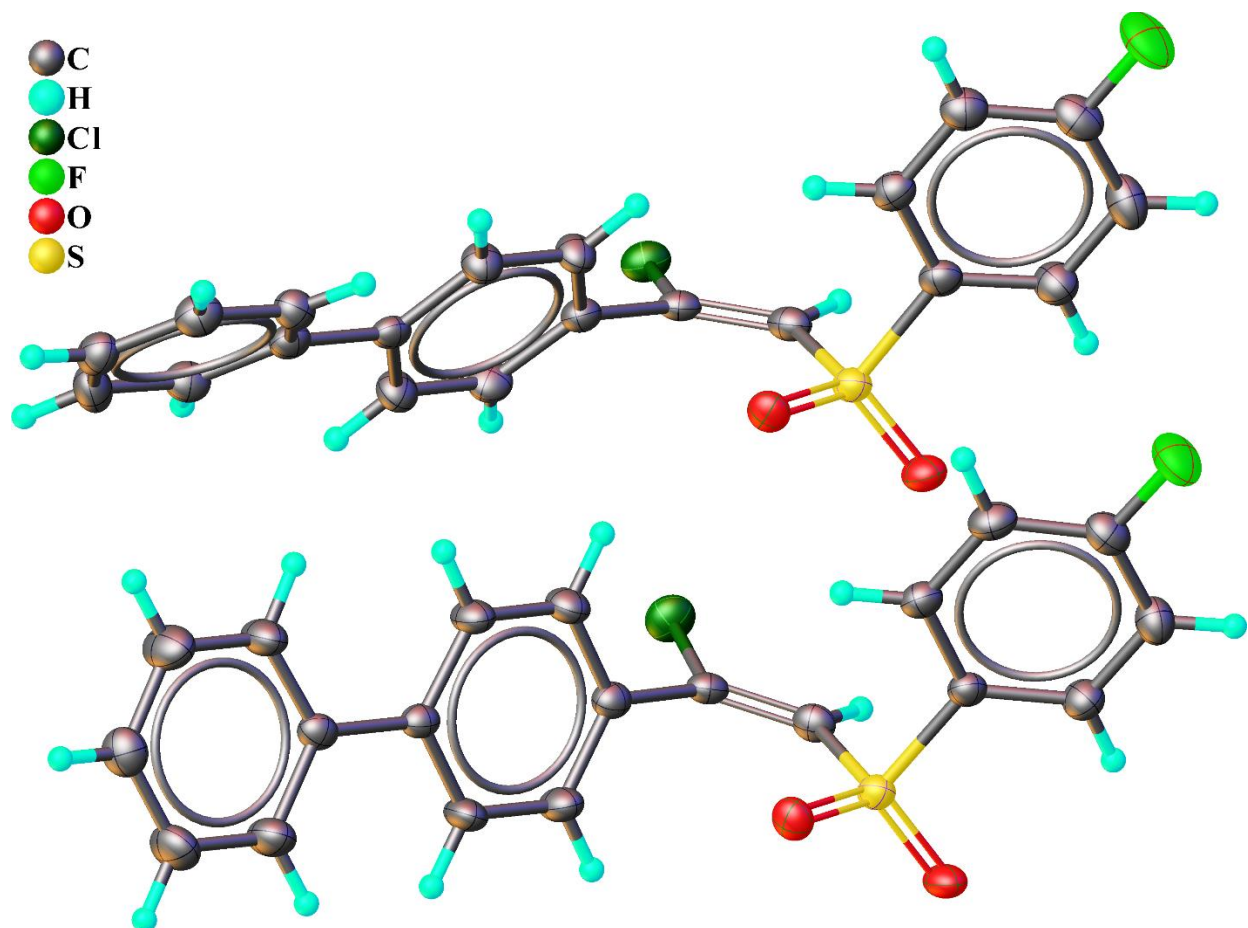


Figure S3. Single crystal X-ray structure of **12d**. The thermal ellipsoids were plotted at the 50% probability level.

Crystallographic details: Colourless needle-shaped crystals of **12d** were obtained by layering of Et₂O onto a solution of **12d** in DCM at room temperature. A suitable crystal of 0.65×0.07×0.06 mm³ was selected and mounted on a suitable support on a SuperNova, Dual, Cu at home/near, Atlas diffractometer. The crystal was kept at a $T = 140.00(10)$ K during data collection. The structure was solved with the **ShelXT** (Sheldrick, 2015) structure solution program using the dual solution method and by using **Olex2** (Dolomanov et al., 2009) as the graphical interface. The model was refined with version 2018/3 of **ShelXL** (Sheldrick, 2015) using full matrix least squares on $|F|^2$ minimisation.

Table S3. Single crystal parameters of **12d** and refinement parameters.

Compound	12d
Formula	C ₂₀ H ₁₄ ClFO ₂ S
<i>D</i> _{calc.} / g cm ⁻³	1.491
μ /mm ⁻¹	3.401
Formula Weight	372.82
Colour	colourless
Shape	needle
Size/mm ³	0.65×0.07×0.06
<i>T</i> /K	140.00(10)
Crystal System	triclinic
Space Group	<i>P</i> $\bar{1}$
<i>a</i> /Å	9.3502(6)
<i>b</i> /Å	9.7552(5)
<i>c</i> /Å	18.8817(13)
α /°	91.916(5)
β /°	92.417(5)
γ /°	104.915(5)
<i>V</i> /Å ³	1661.00(18)
<i>Z</i>	4
<i>Z</i> '	2
Wavelength/Å	1.54184
Radiation type	CuK α
Θ _{min} /°	4.693
Θ _{max} /°	73.520
Measured Refl.	6731
Independent Refl.	6731
Reflections with <i>I</i> > 2(<i>I</i>)	4978
<i>R</i> _{int}	n/a
Parameters	452
Restraints	0
Largest Peak/e Å ⁻³	0.339
Deepest Hole/e Å ⁻³	-0.532
GooF	1.022
<i>wR</i> ₂ (all data)	0.1296
<i>wR</i> ₂	0.1180
<i>R</i> ₁ (all data)	0.0650
<i>R</i> ₁	0.0450

NMR Spectra

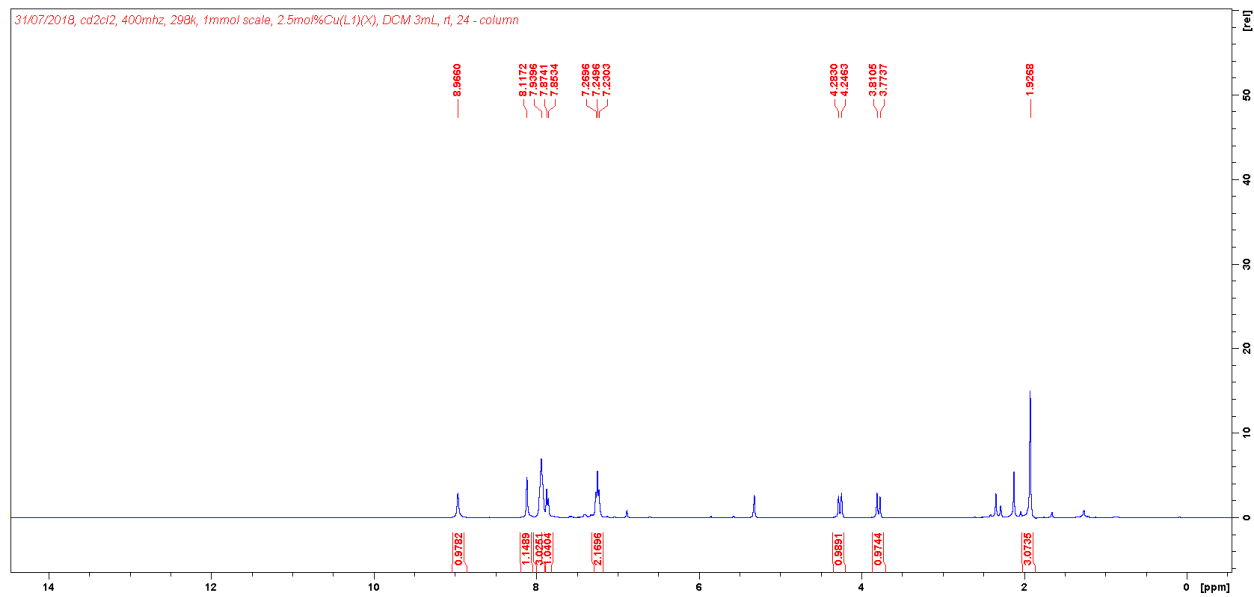
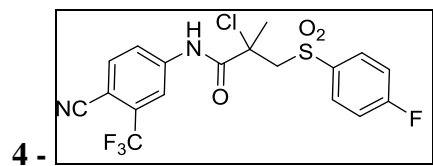


Figure S4. ¹H NMR spectrum of 4.

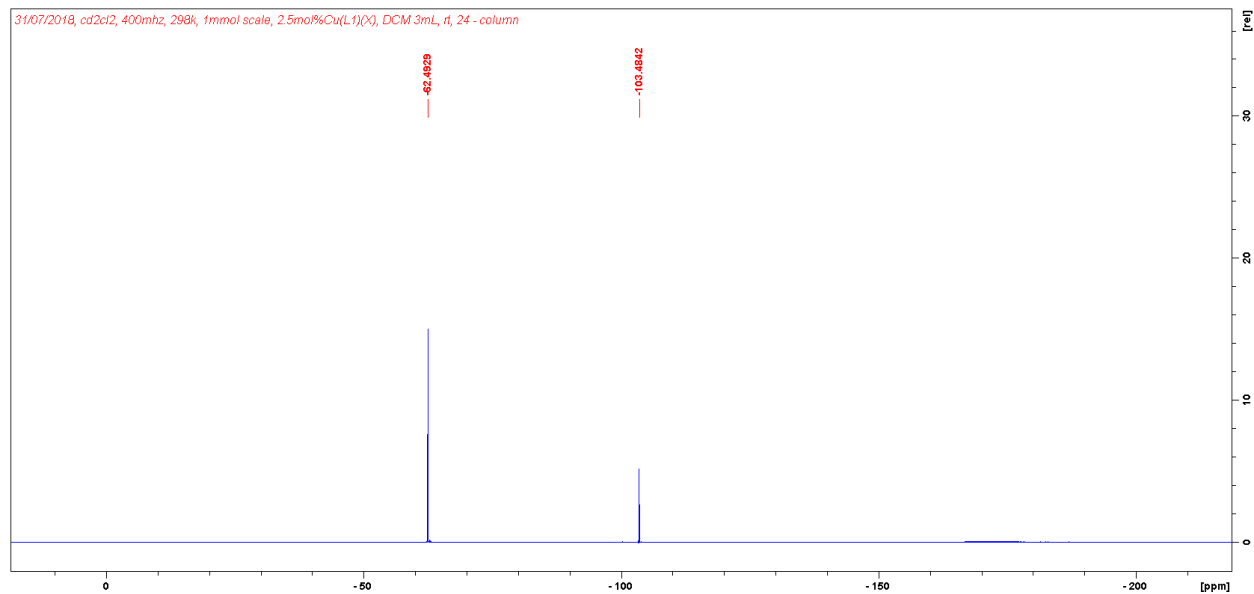


Figure S5. ¹⁹F{¹H} NMR spectrum of 4.

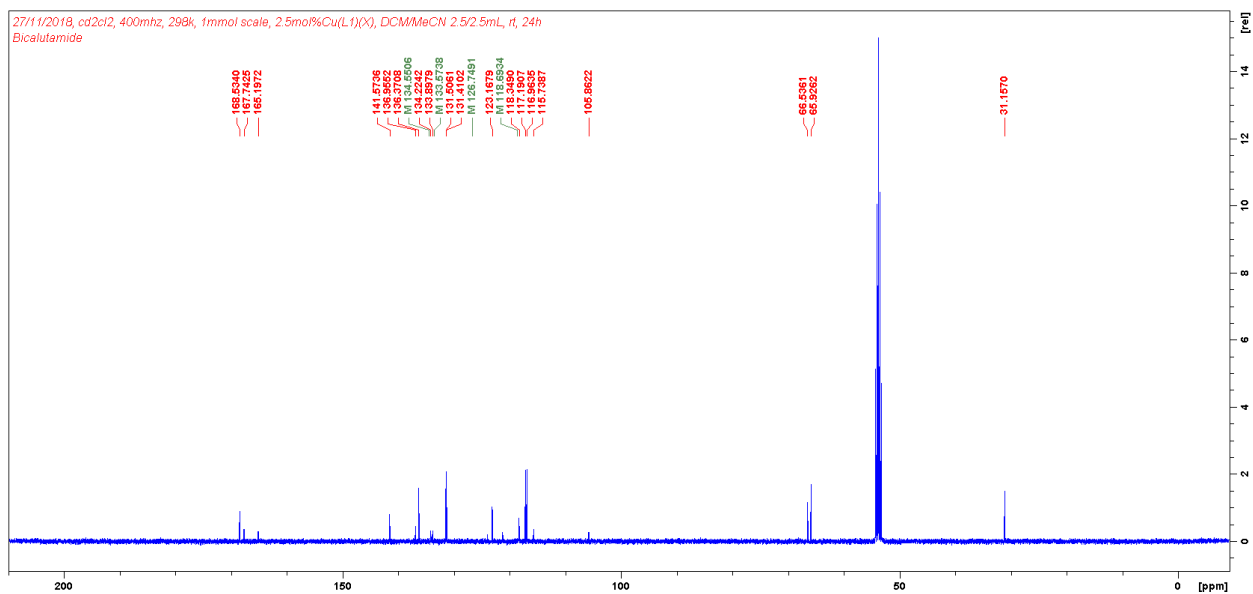


Figure S6. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **4**.

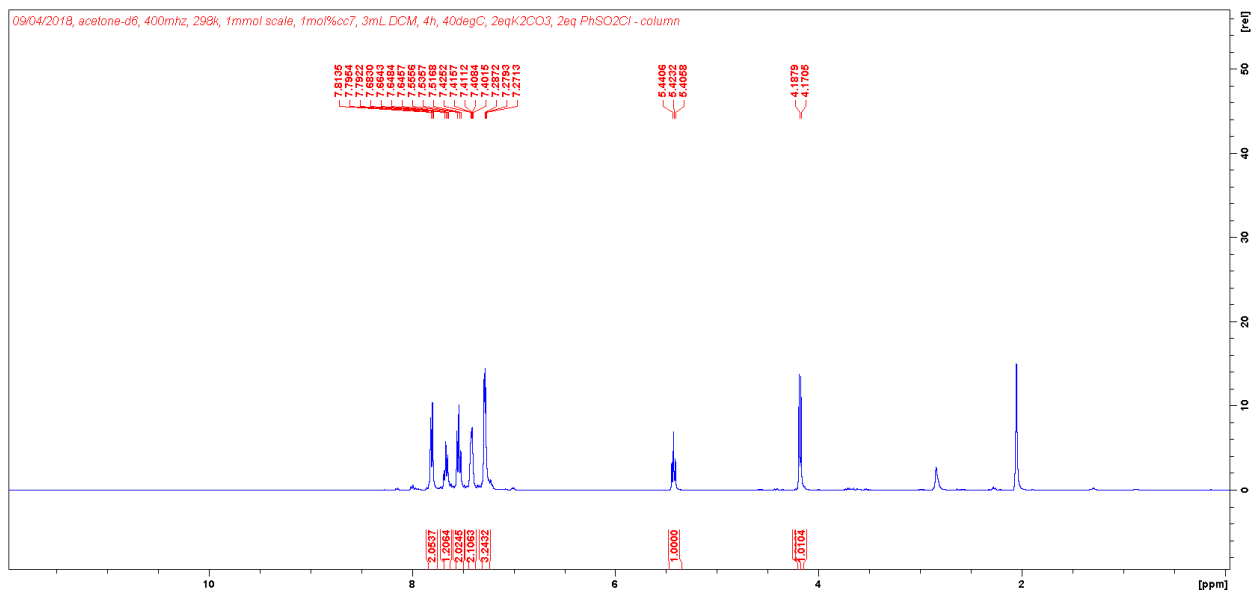
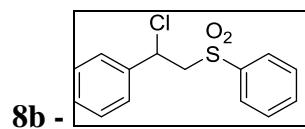


Figure S7. ^1H NMR spectrum of **8b**.

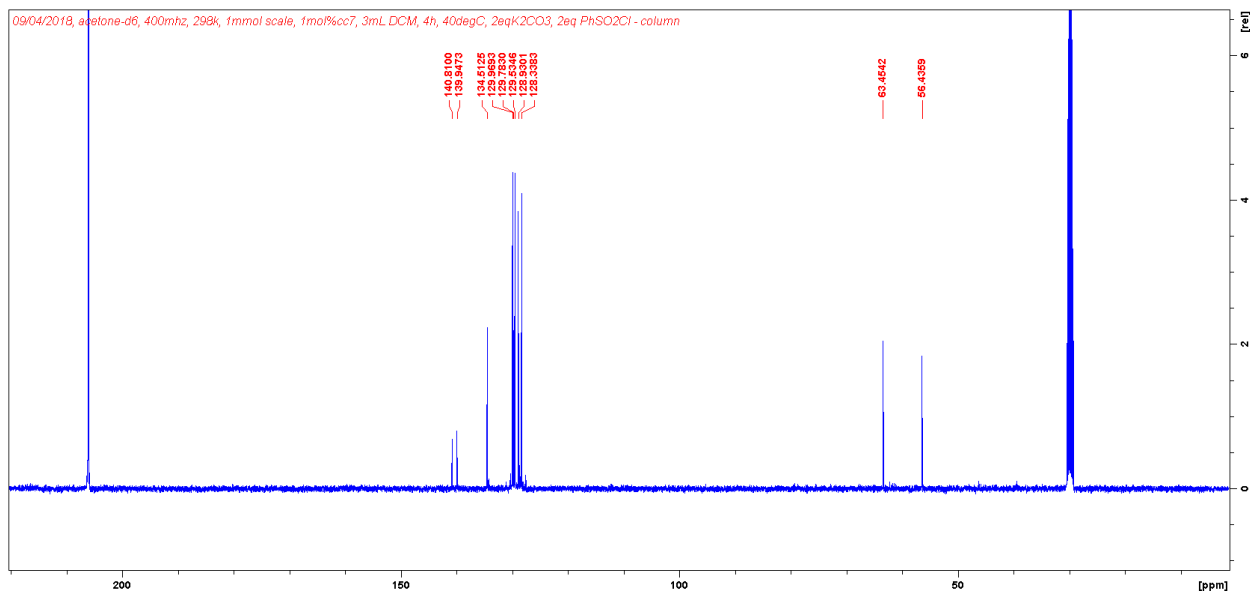


Figure S8. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8b**.

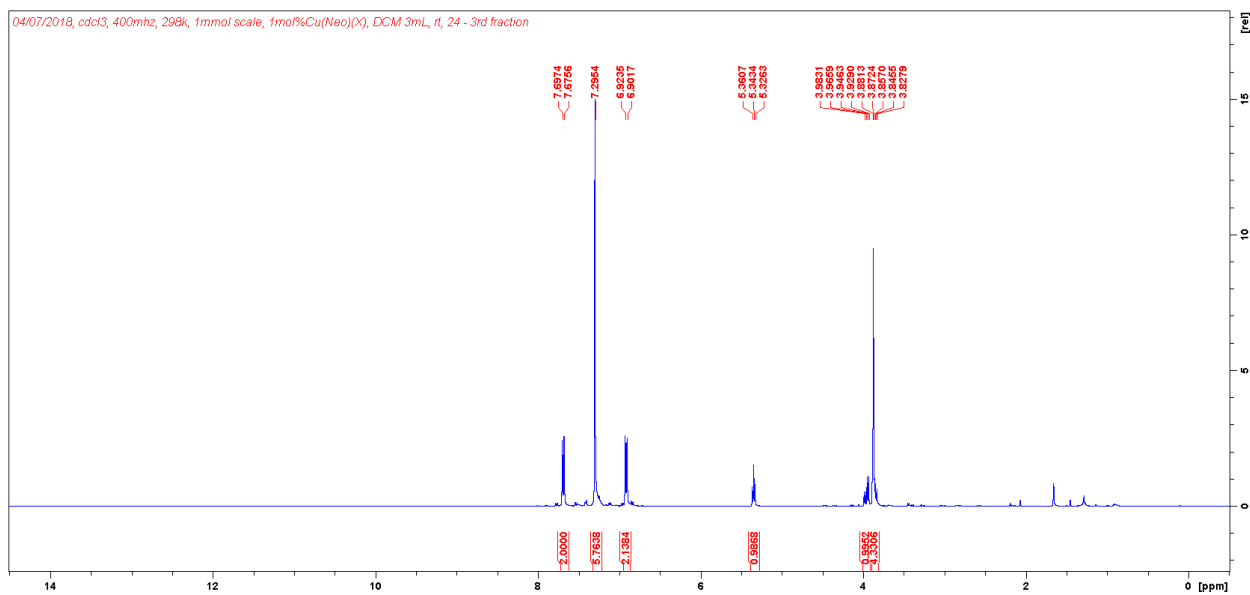
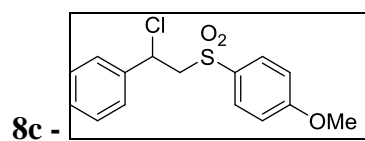


Figure S9. ^1H NMR spectrum of **8c**.

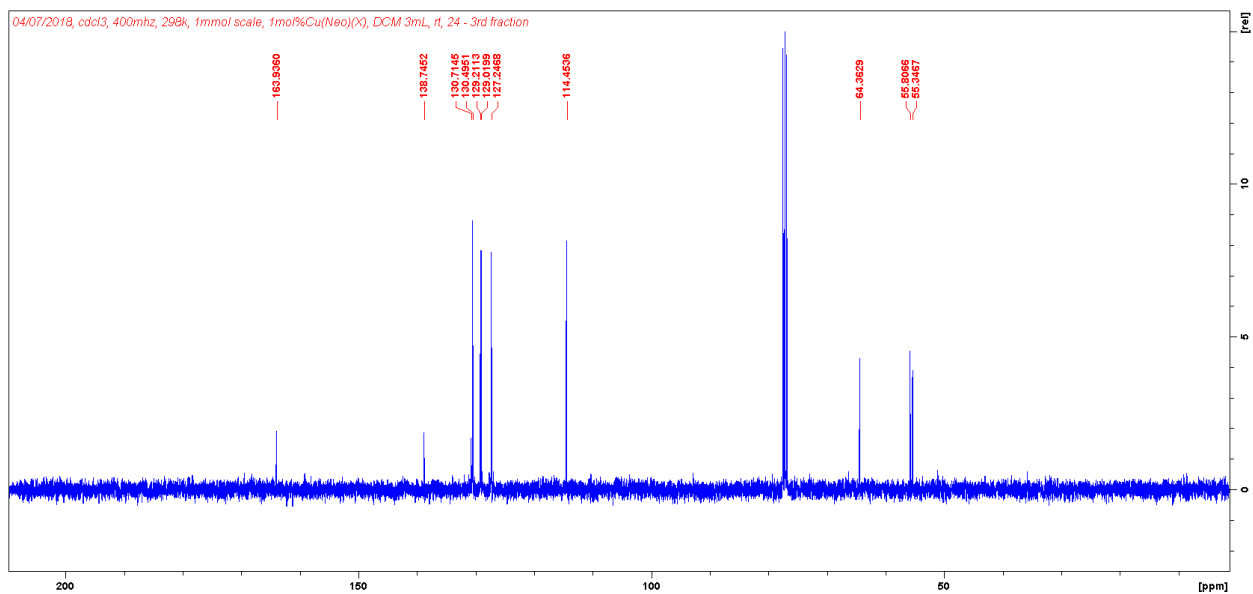


Figure S10. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8c**.

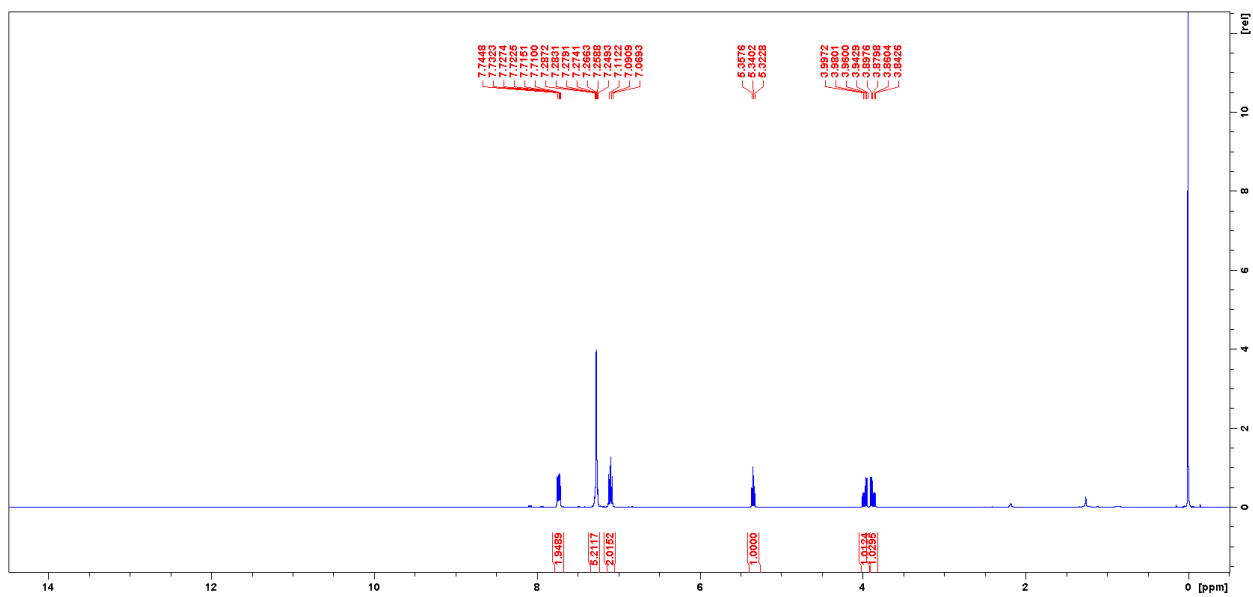
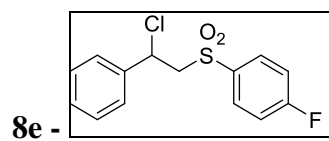


Figure S11. ^1H NMR spectrum of **8e**.

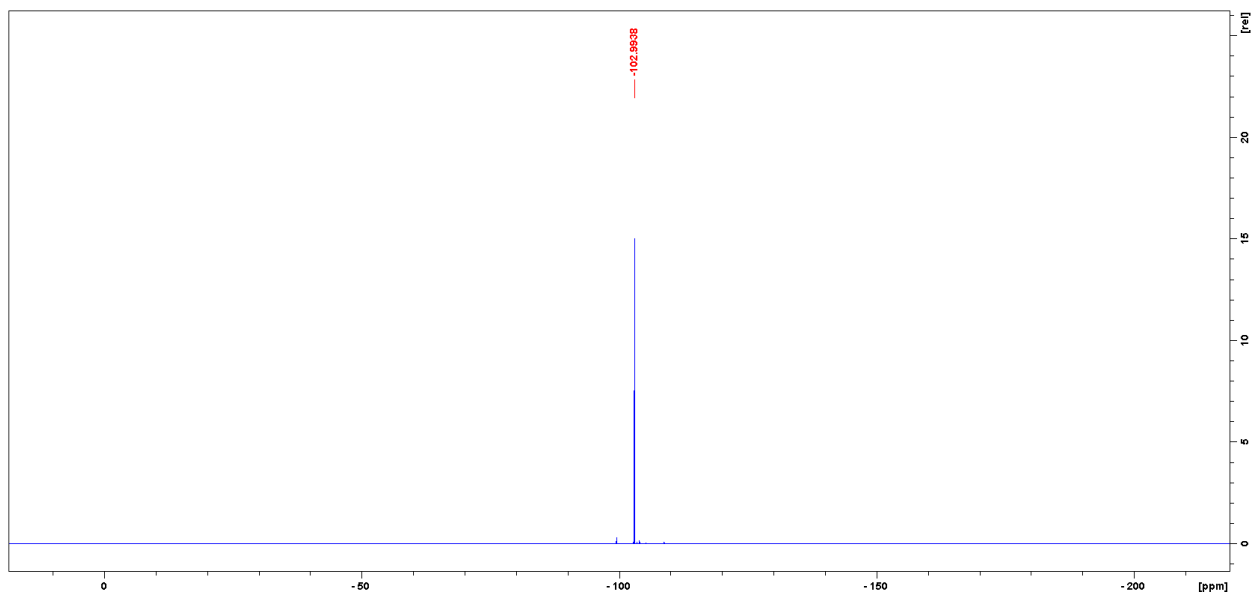


Figure S12. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **8e**.

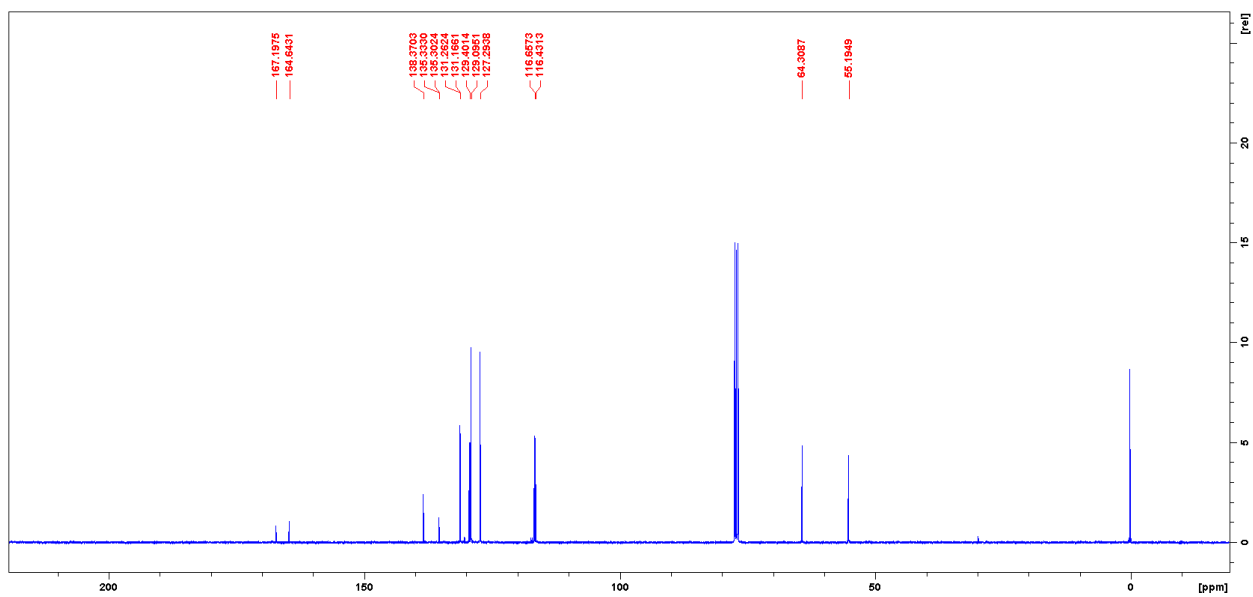
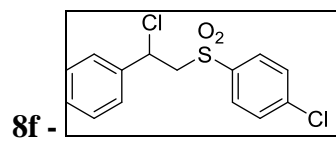


Figure S13. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8e**.



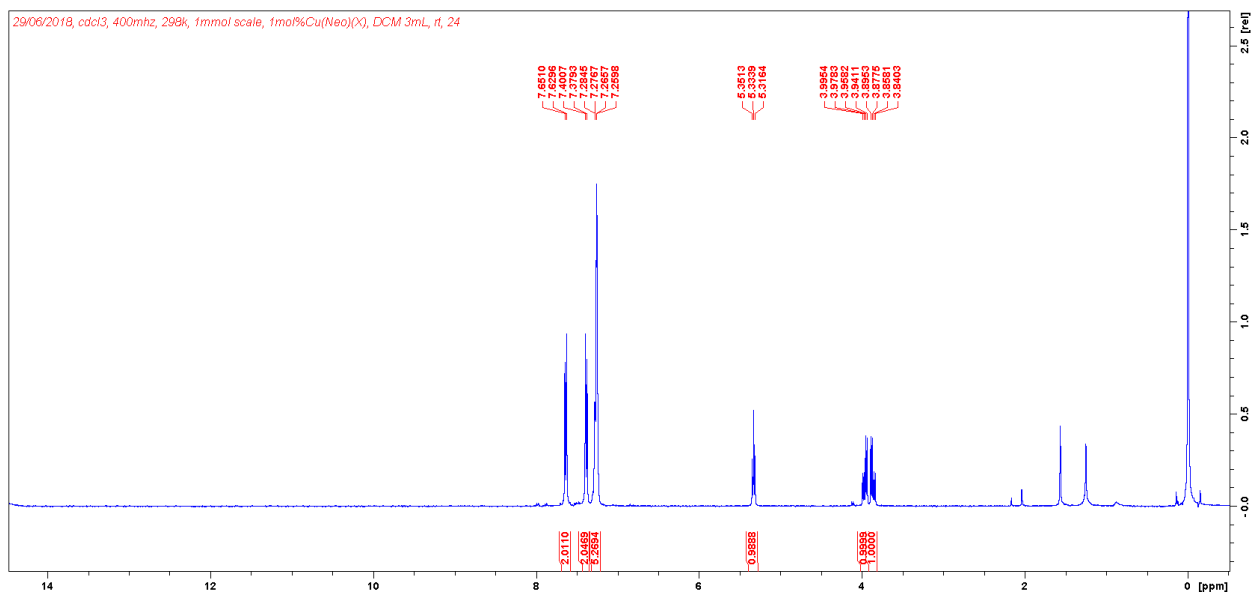


Figure S14. ^1H NMR spectrum of **8f**.

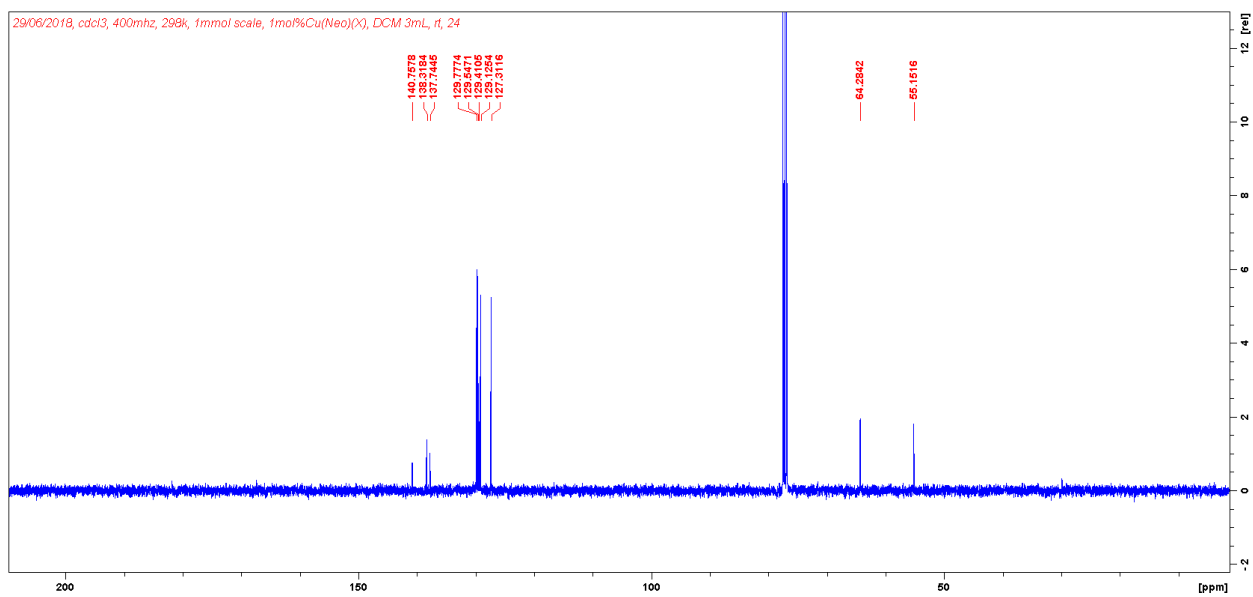
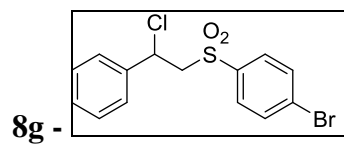


Figure S15. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8f**.



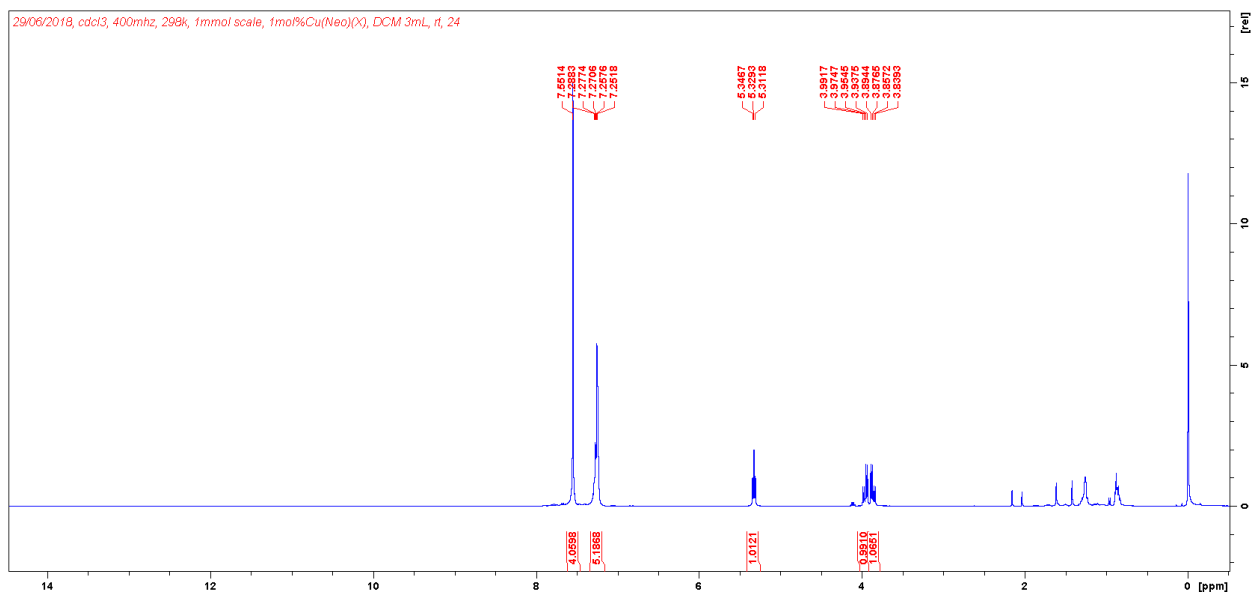


Figure S16. ^1H NMR spectrum of **8g**.

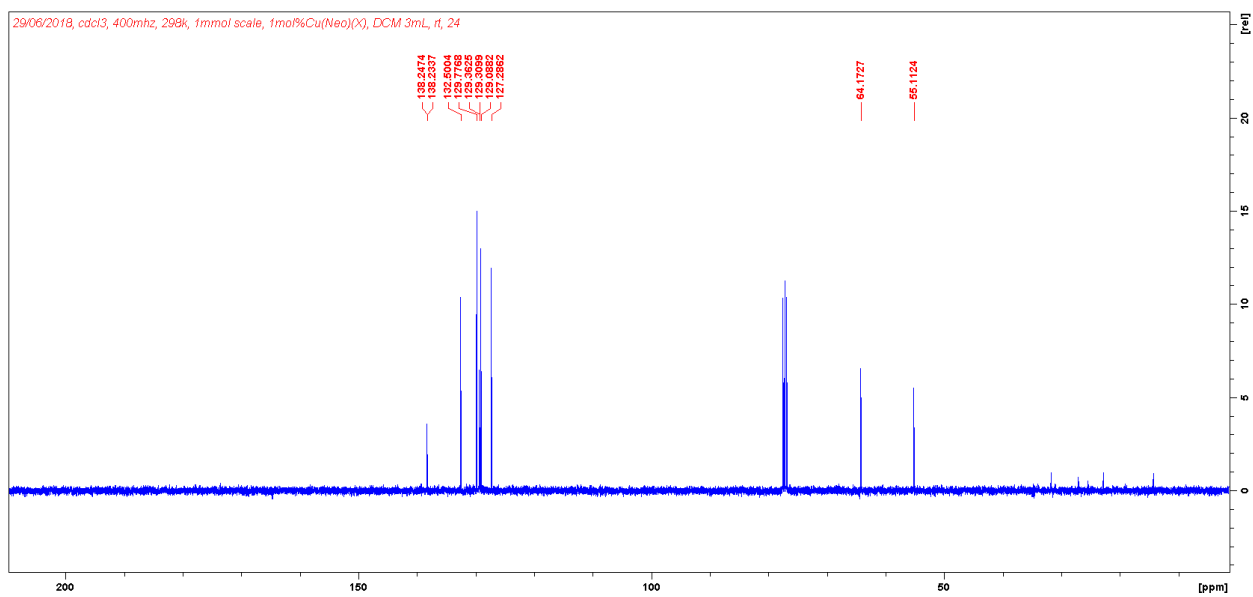
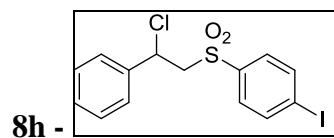


Figure S17. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8g**.



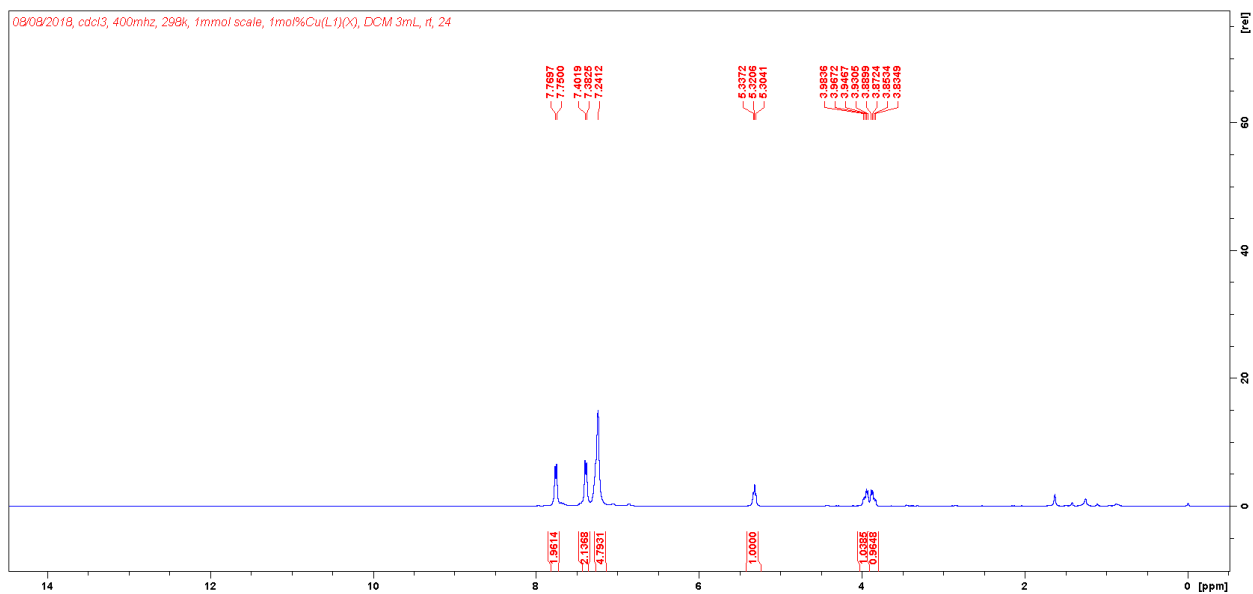


Figure S18. ^1H NMR spectrum of **8h**.

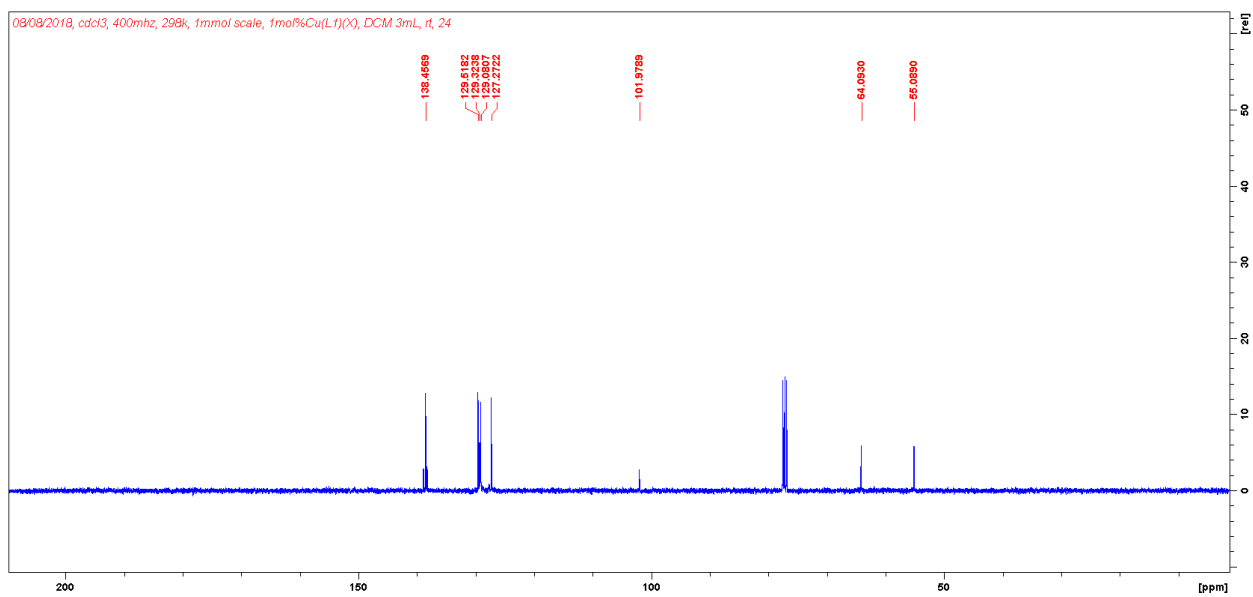
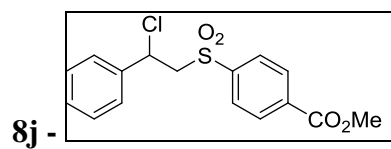


Figure S19. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8h**.



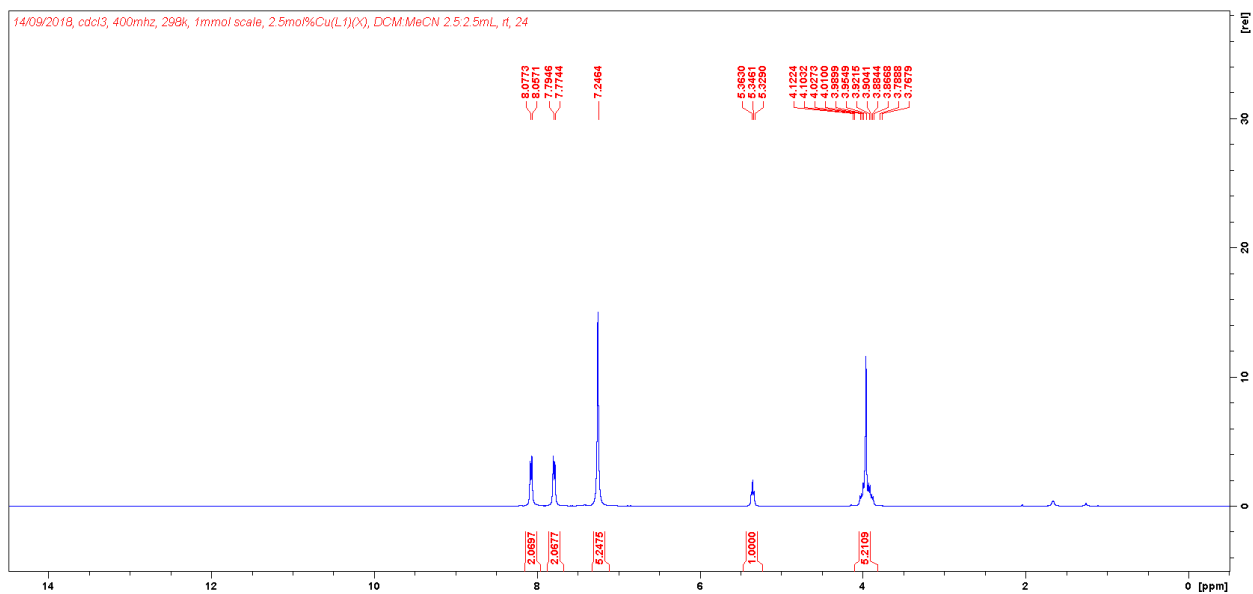


Figure S20. ^1H NMR spectrum of **8j**.

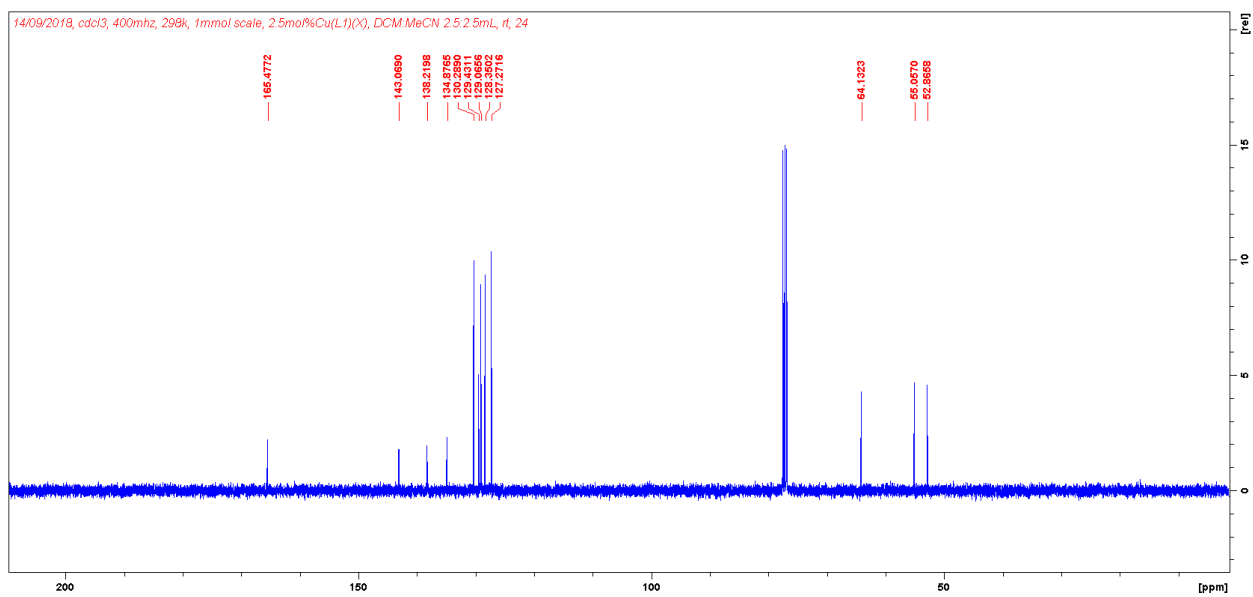
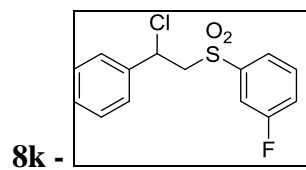


Figure S21. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8j**.



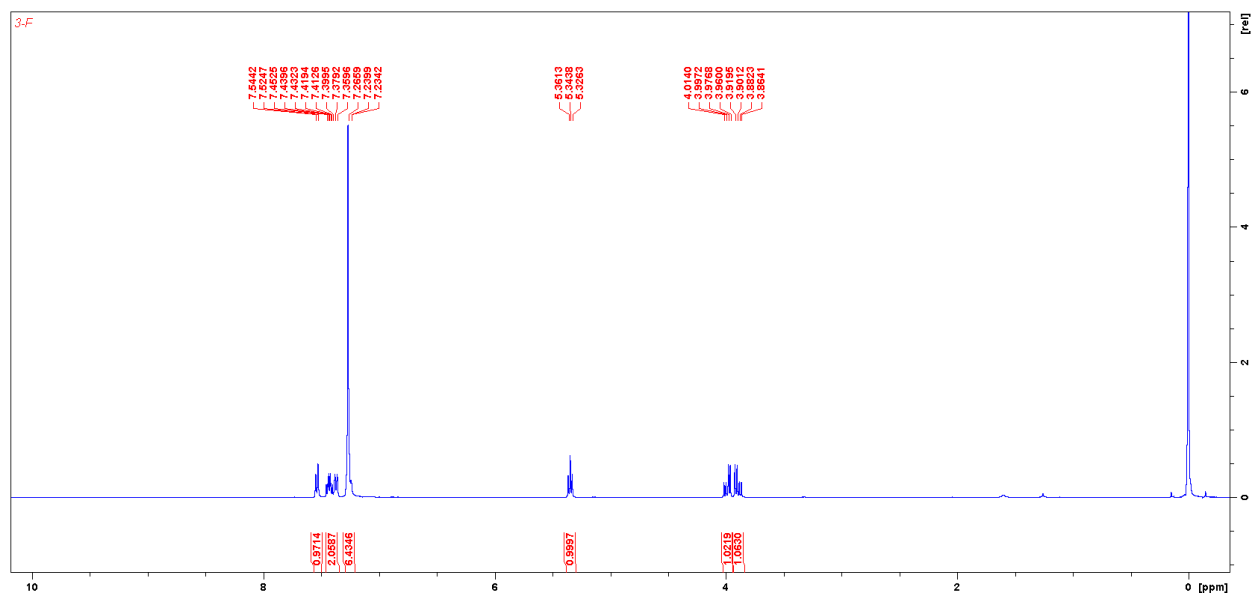


Figure S22. ^1H NMR spectrum of **8k**.

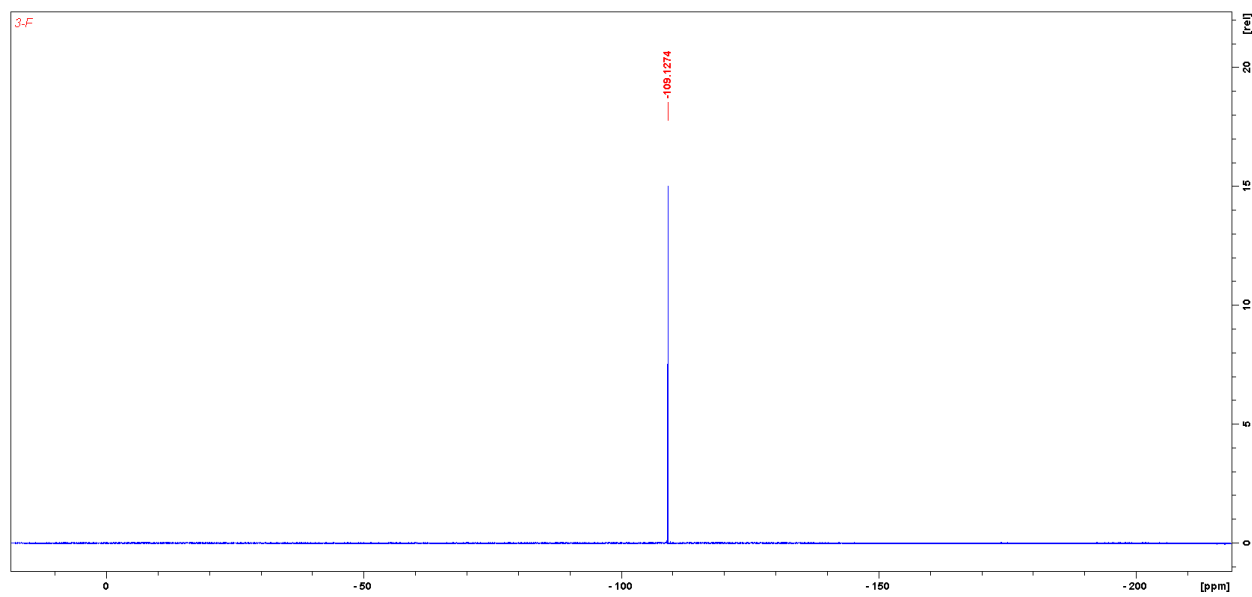


Figure S23. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **8k**.

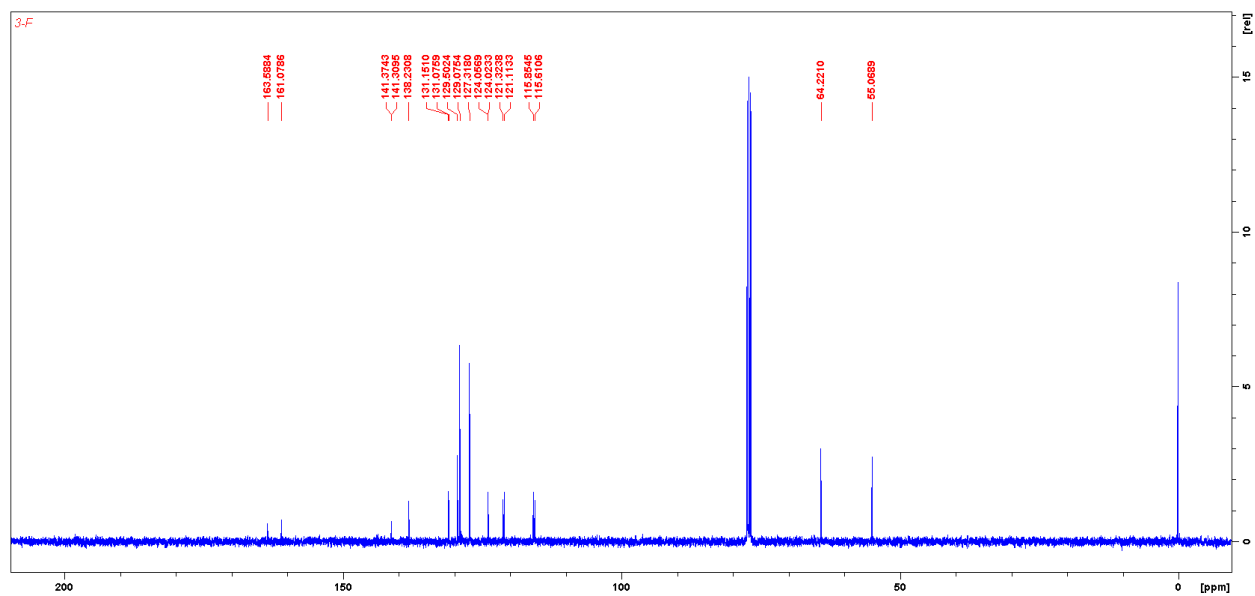


Figure S24. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8k**.

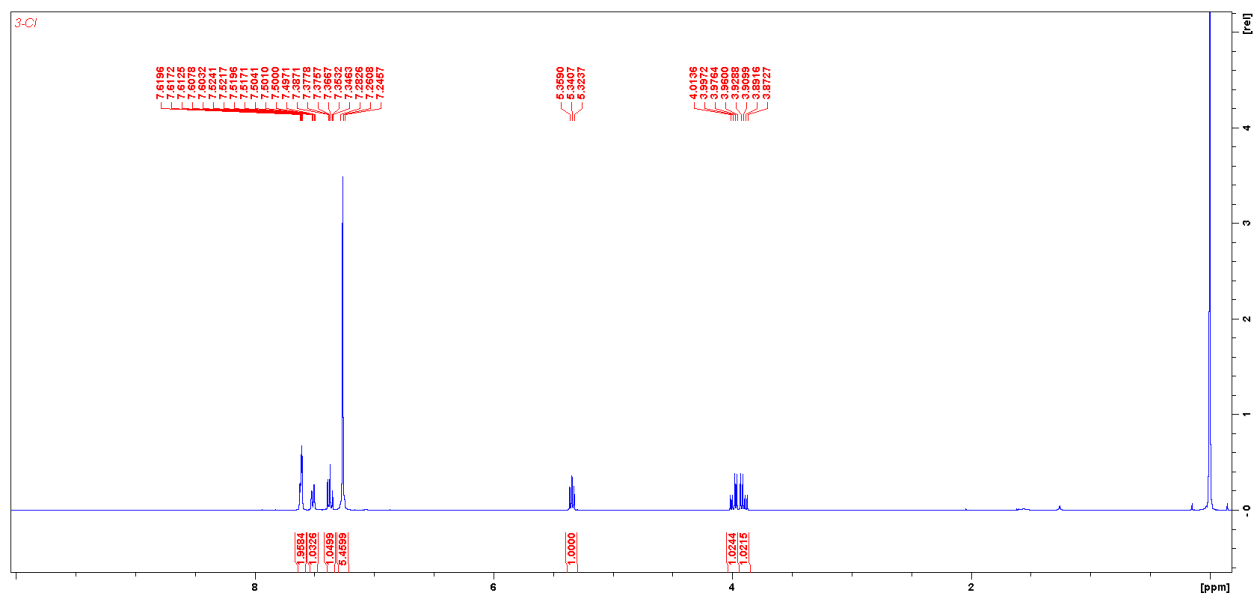
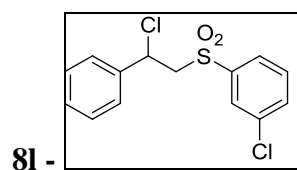


Figure S25. ^1H NMR spectrum of **8l**.

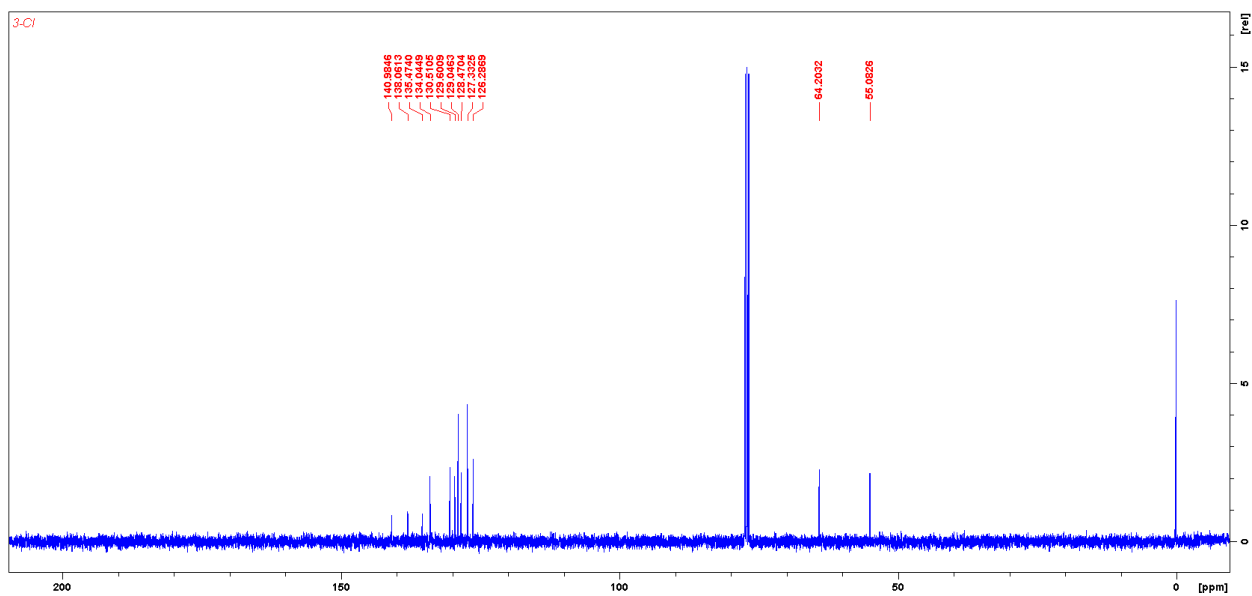


Figure S26. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8l**.

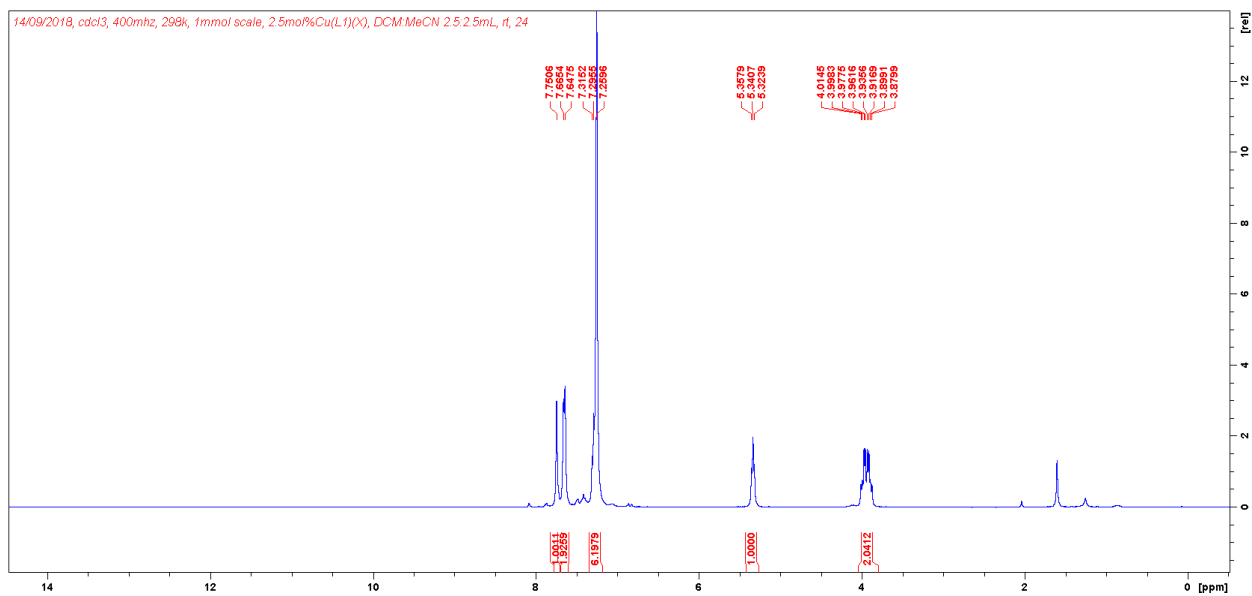
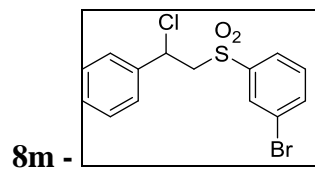


Figure S27. ^1H NMR spectrum of **8m**.

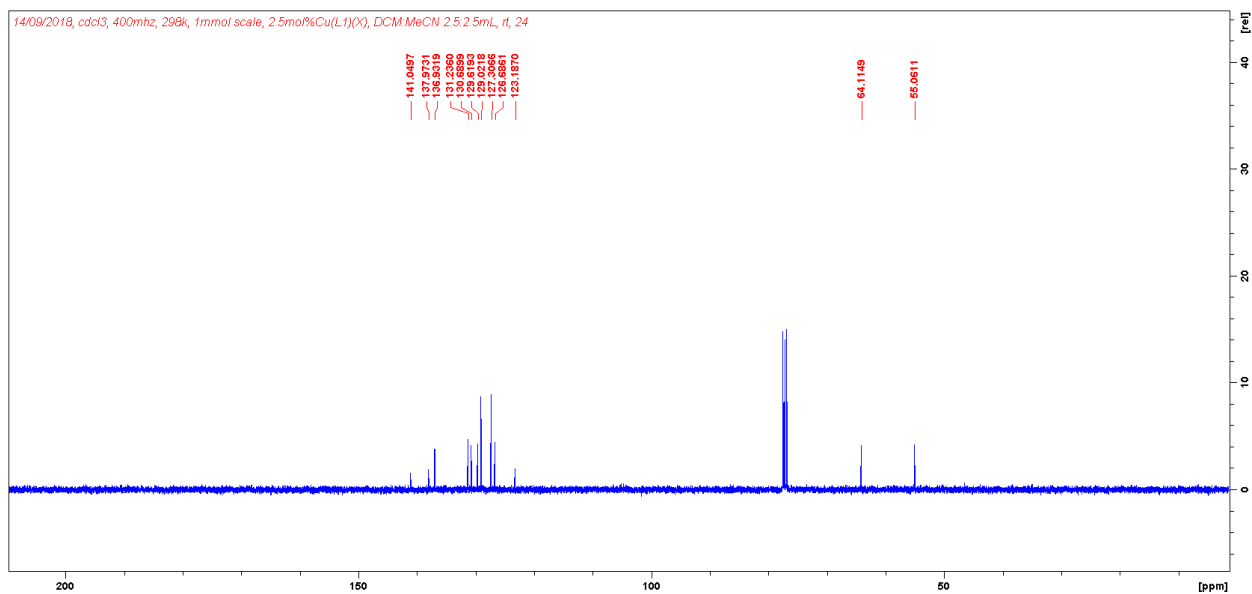


Figure S28. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8m**.

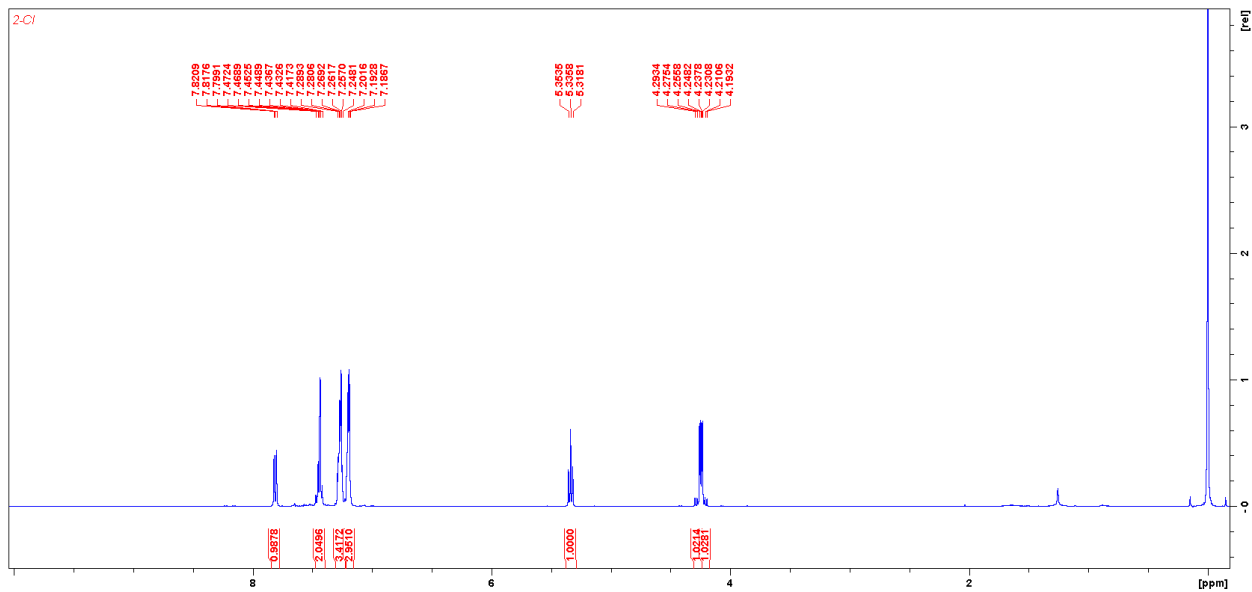
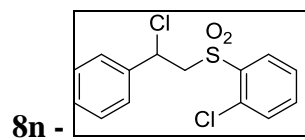


Figure S29. ^1H NMR spectrum of **8n**.

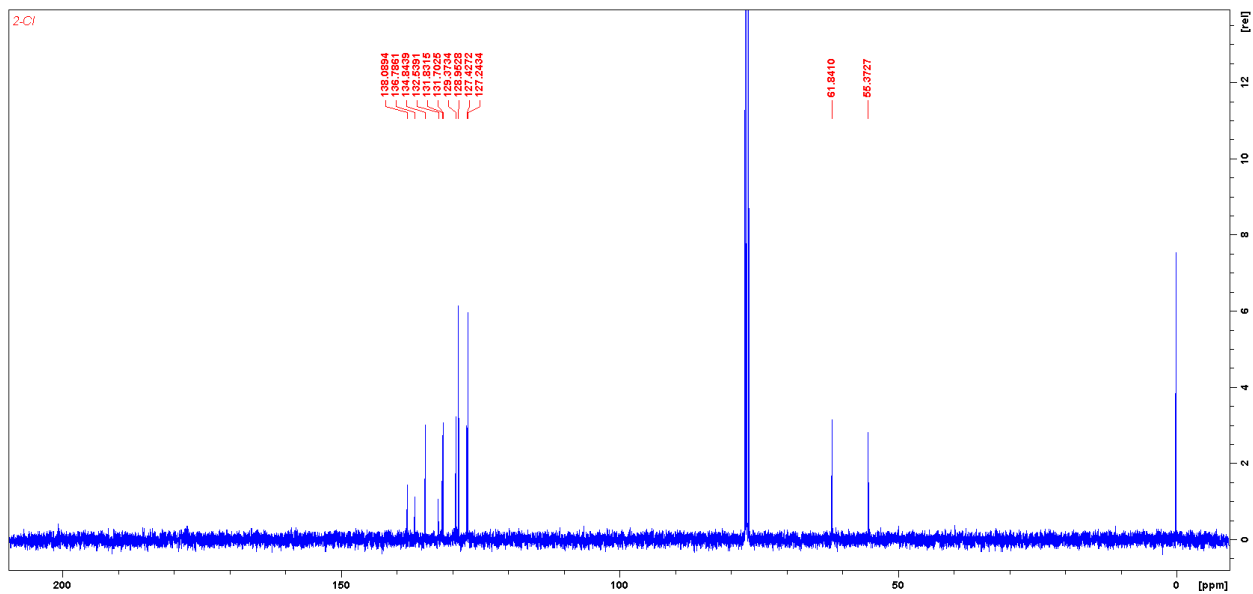


Figure S30. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8n**.

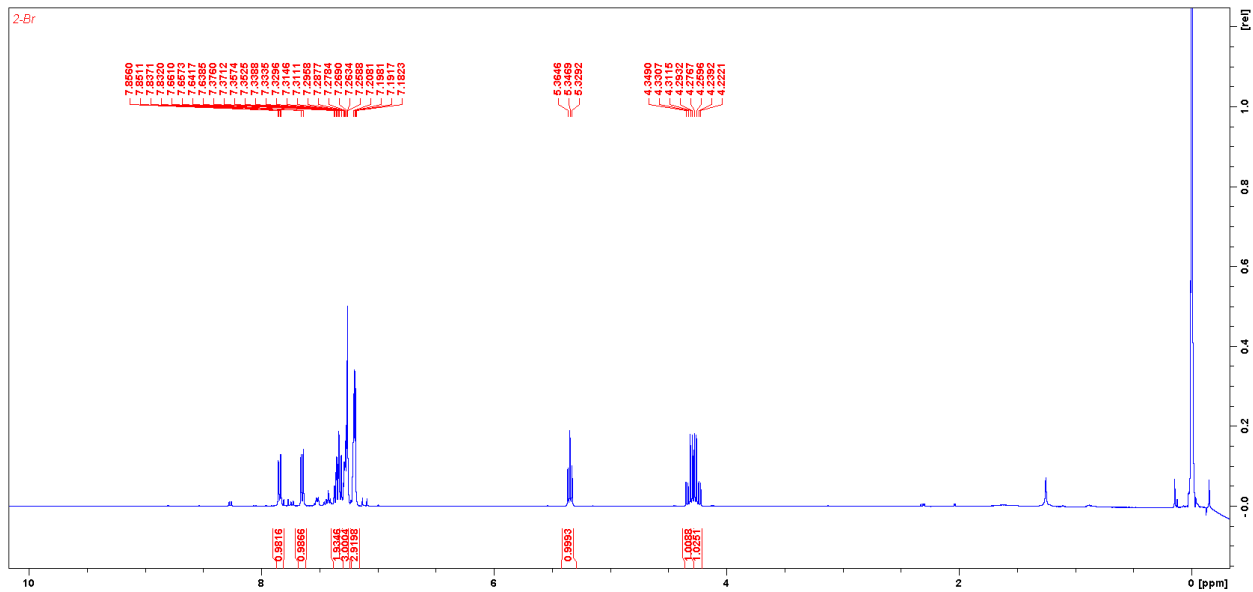
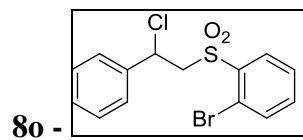


Figure S31. ^1H NMR spectrum of **8o**.

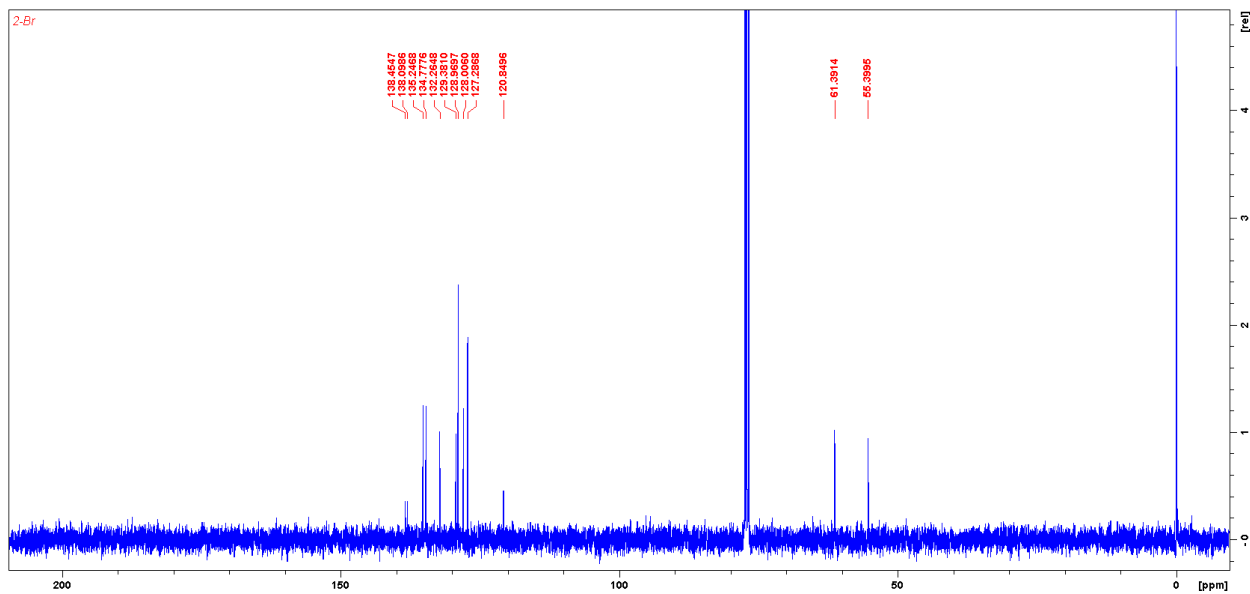


Figure S32. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8o**.

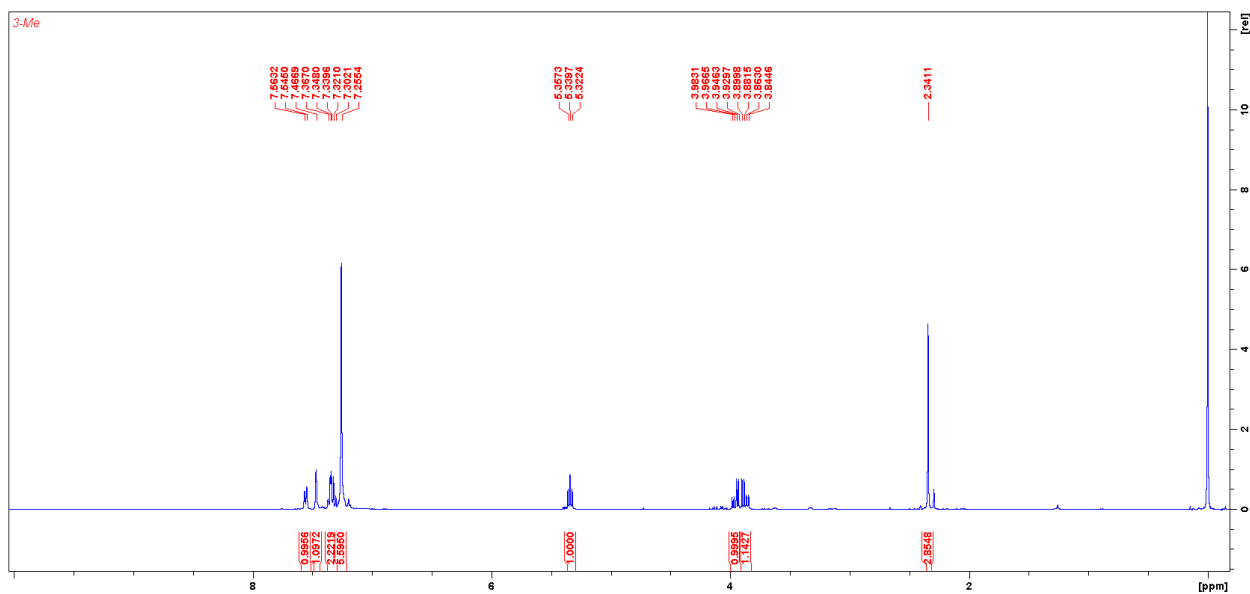
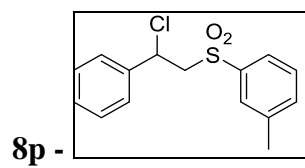


Figure S33. ^1H NMR spectrum of **8p**.

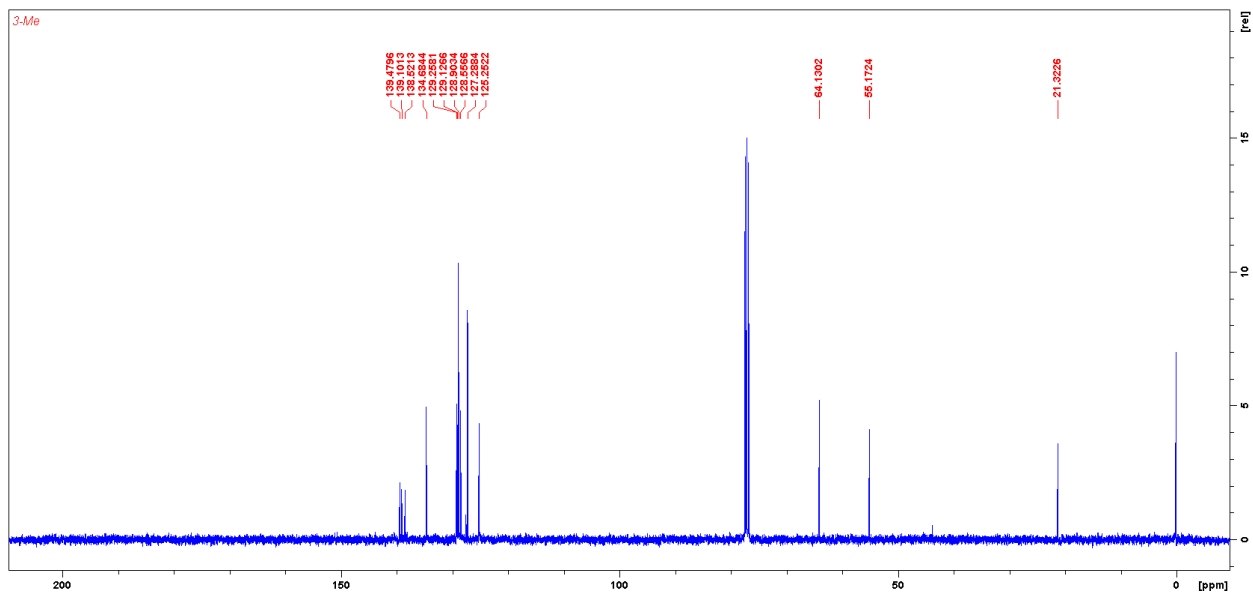


Figure S34. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8p**.

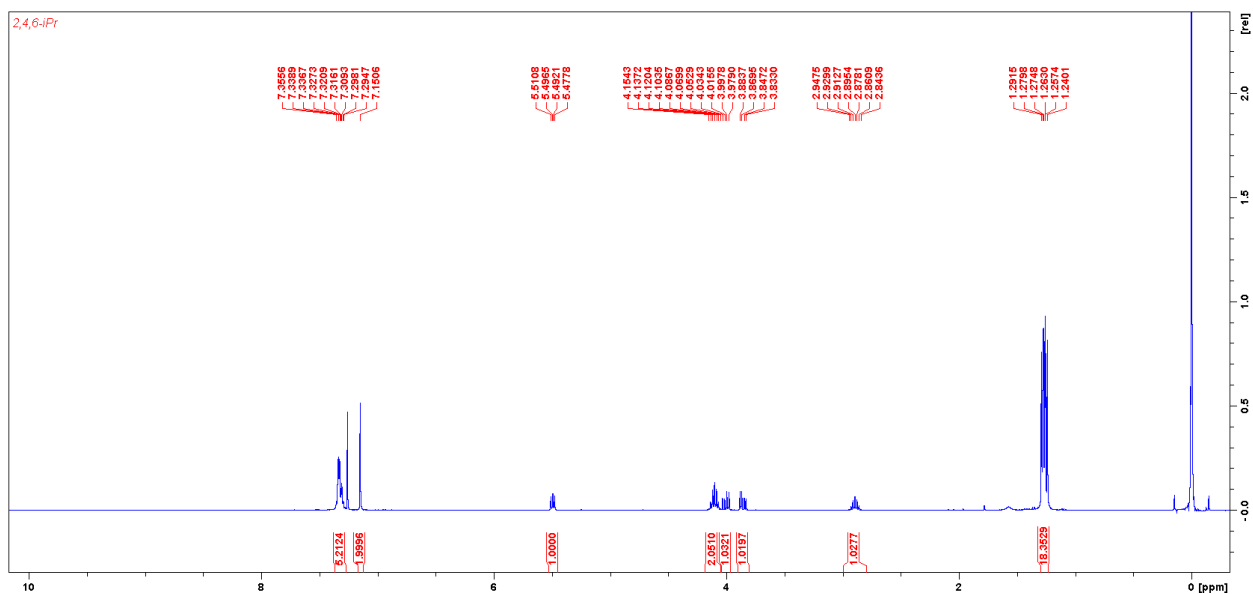
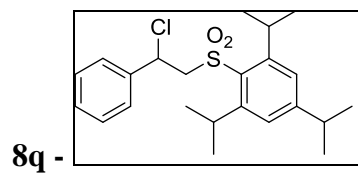


Figure S35. ^1H NMR spectrum of **8q**.

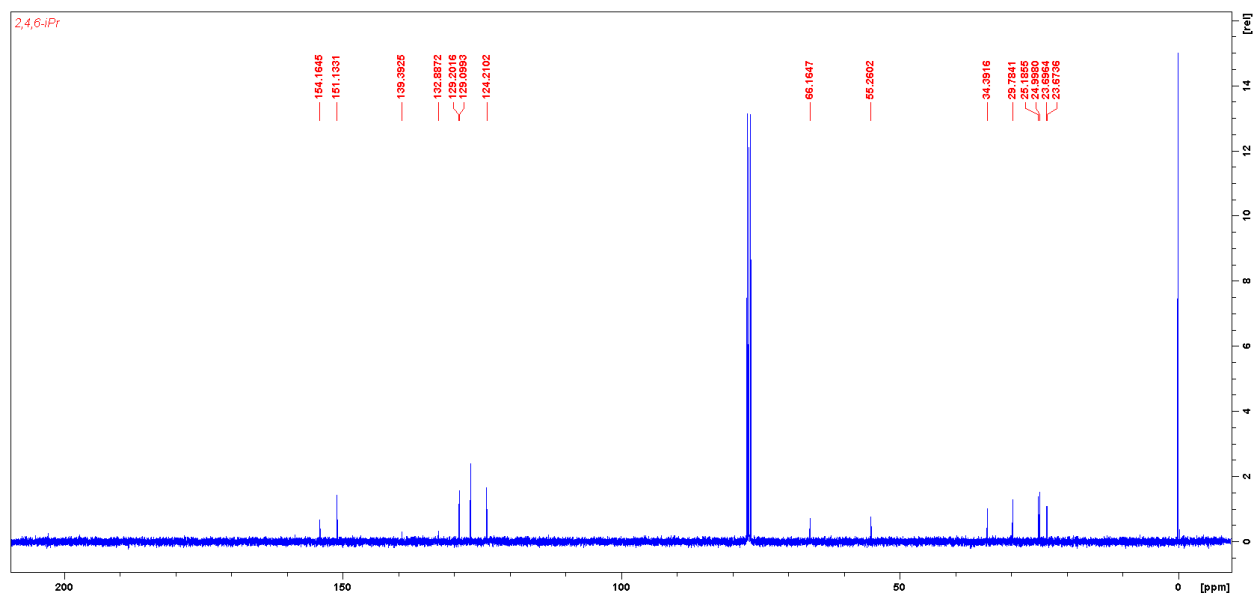


Figure S36. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8q**.

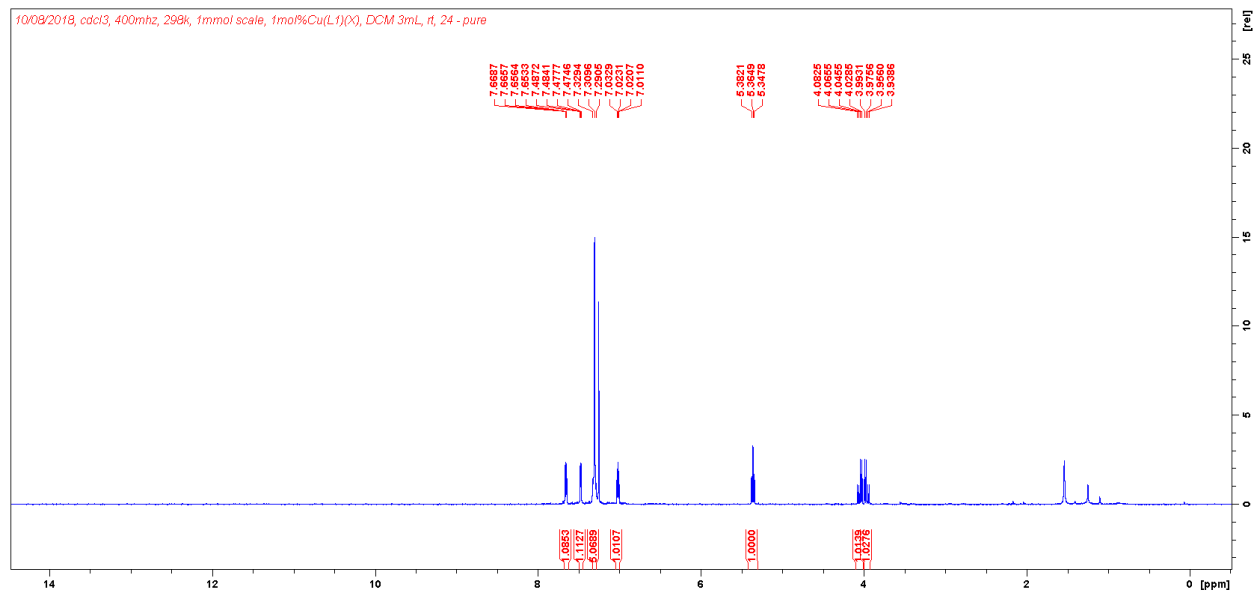
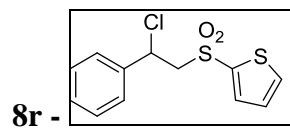


Figure S37. ^1H NMR spectrum of **8r**.

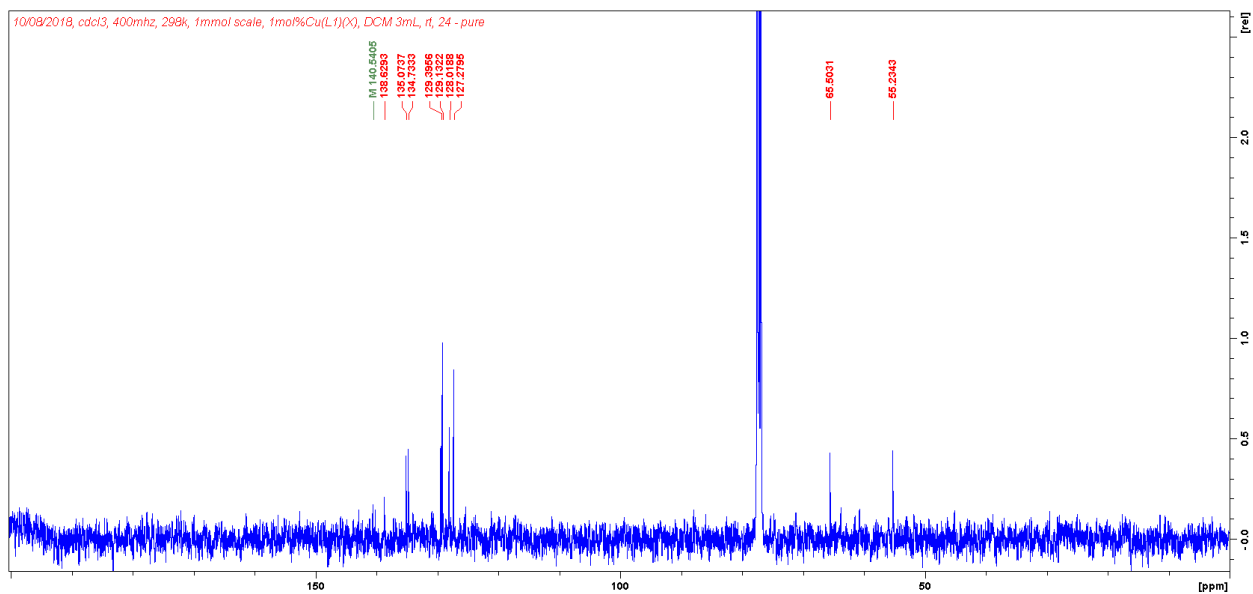


Figure S38. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8r**.

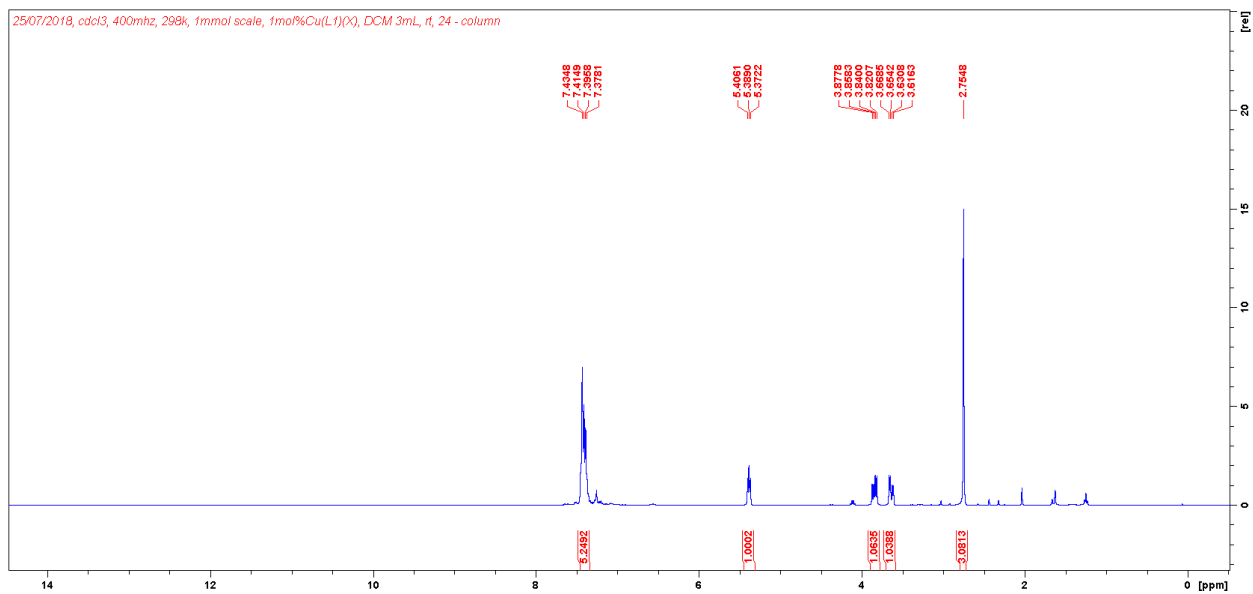
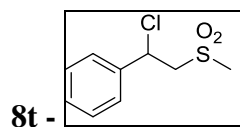


Figure S39. ^1H NMR spectrum of **8t**.

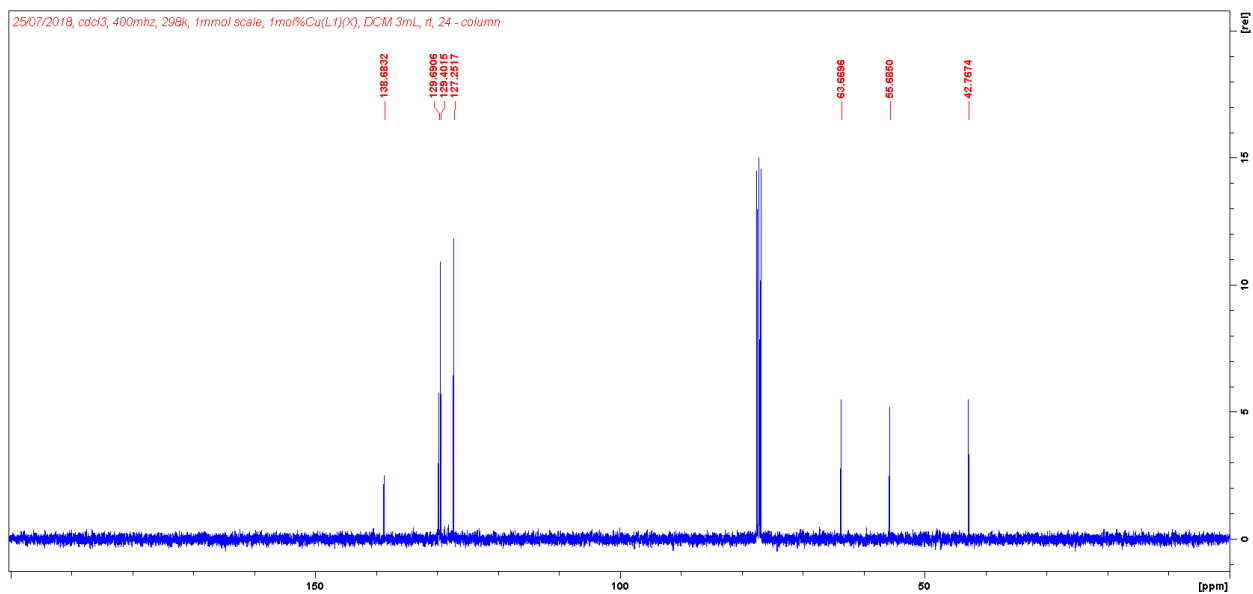


Figure S40. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8t**.

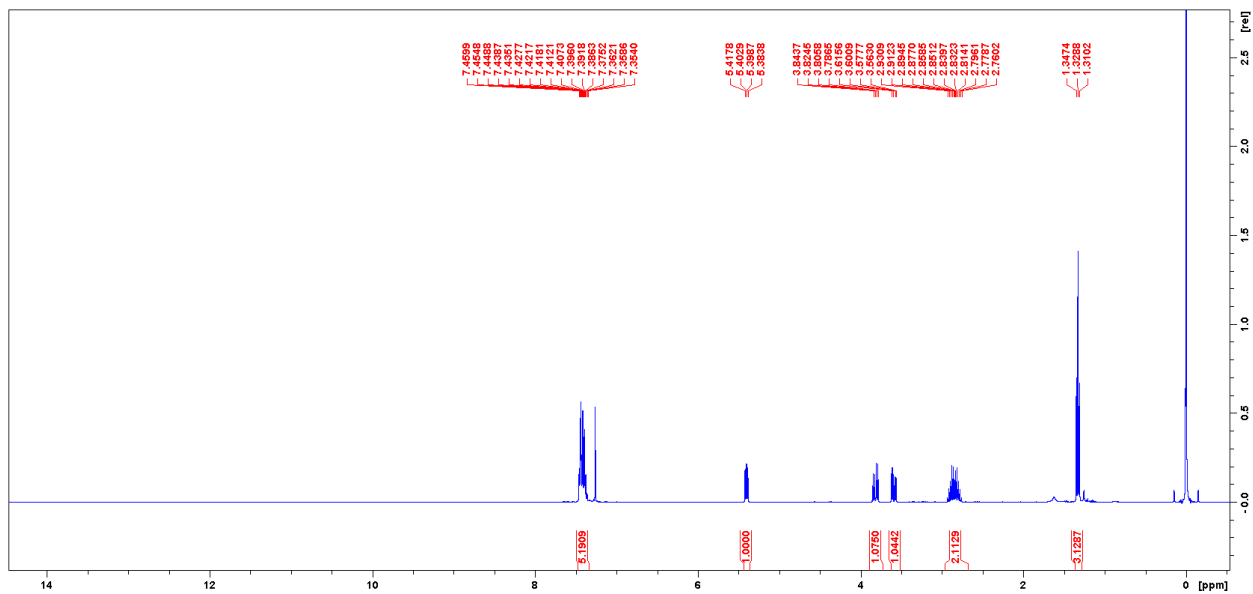
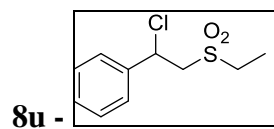


Figure S41. ^1H NMR spectrum of **8u**.

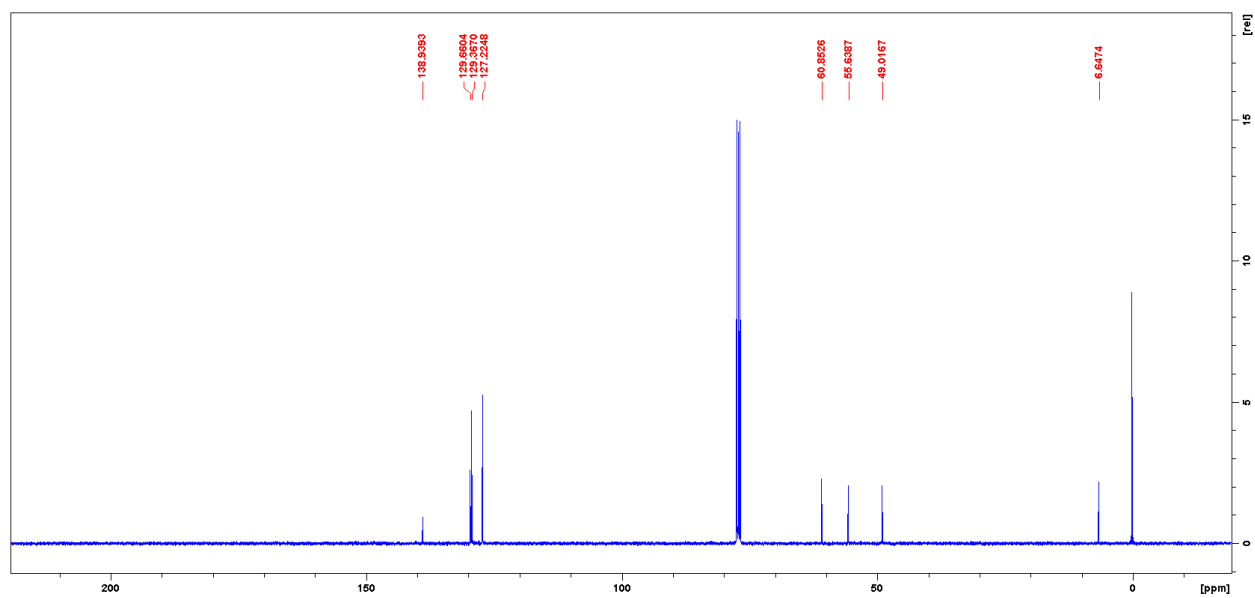


Figure S42. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8u**.

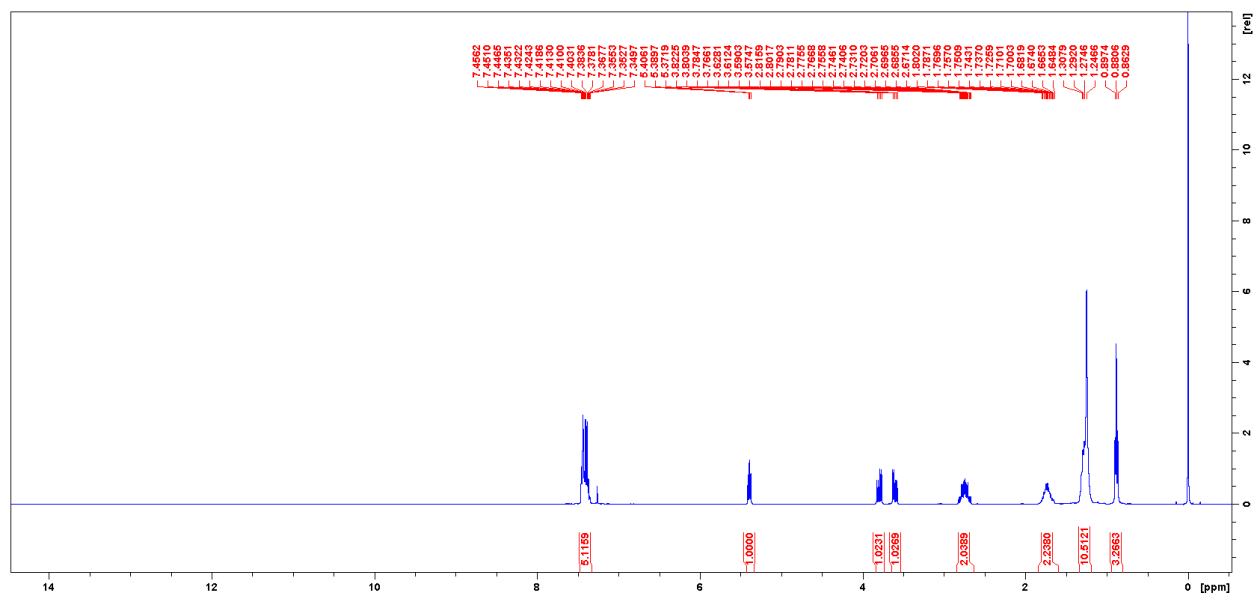
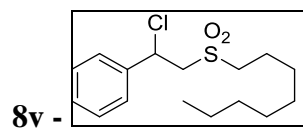


Figure S43. ^1H NMR spectrum of **8v**.

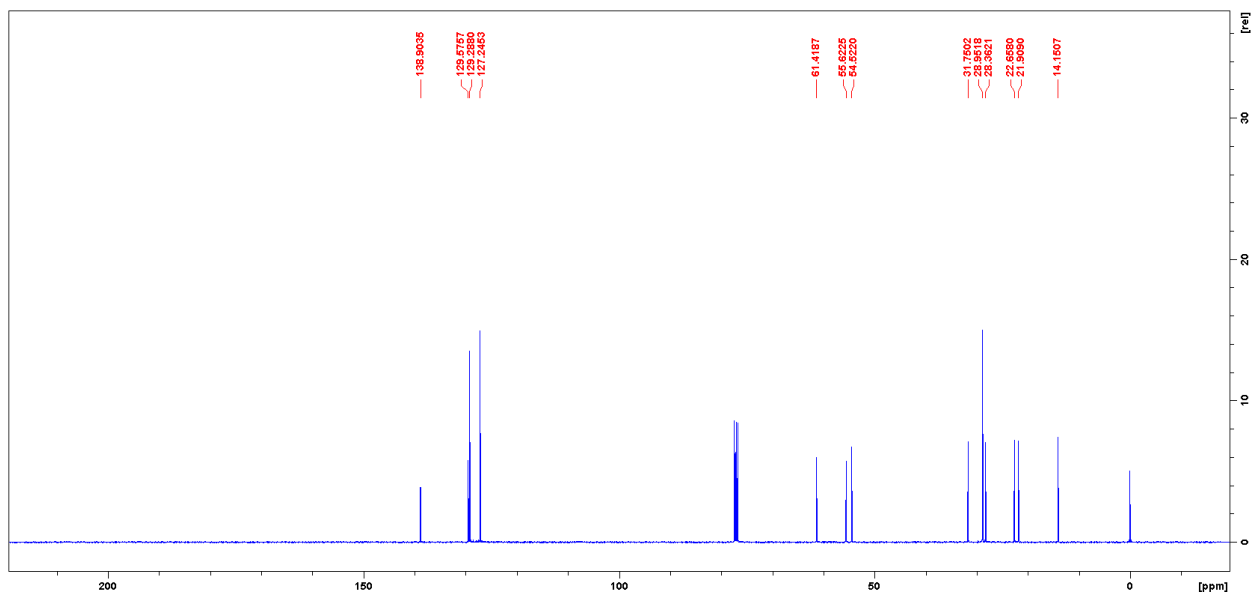


Figure S44. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8v**.

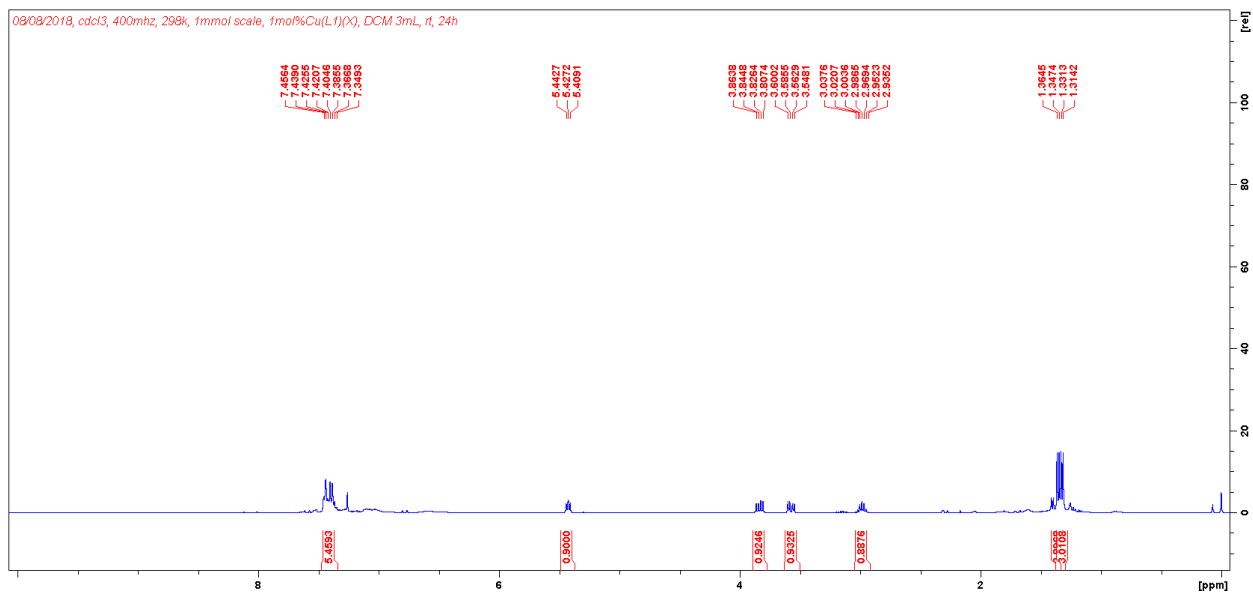
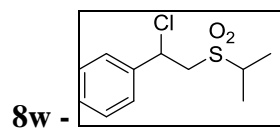


Figure S45. ^1H NMR spectrum of **8w**.

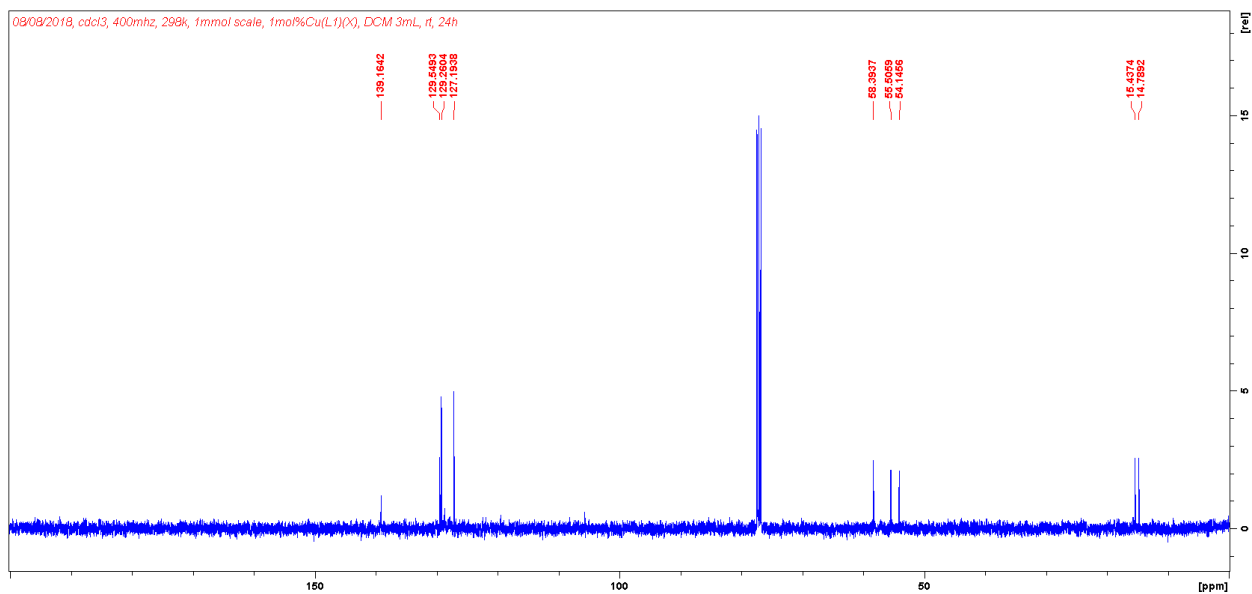


Figure S46. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8w**.

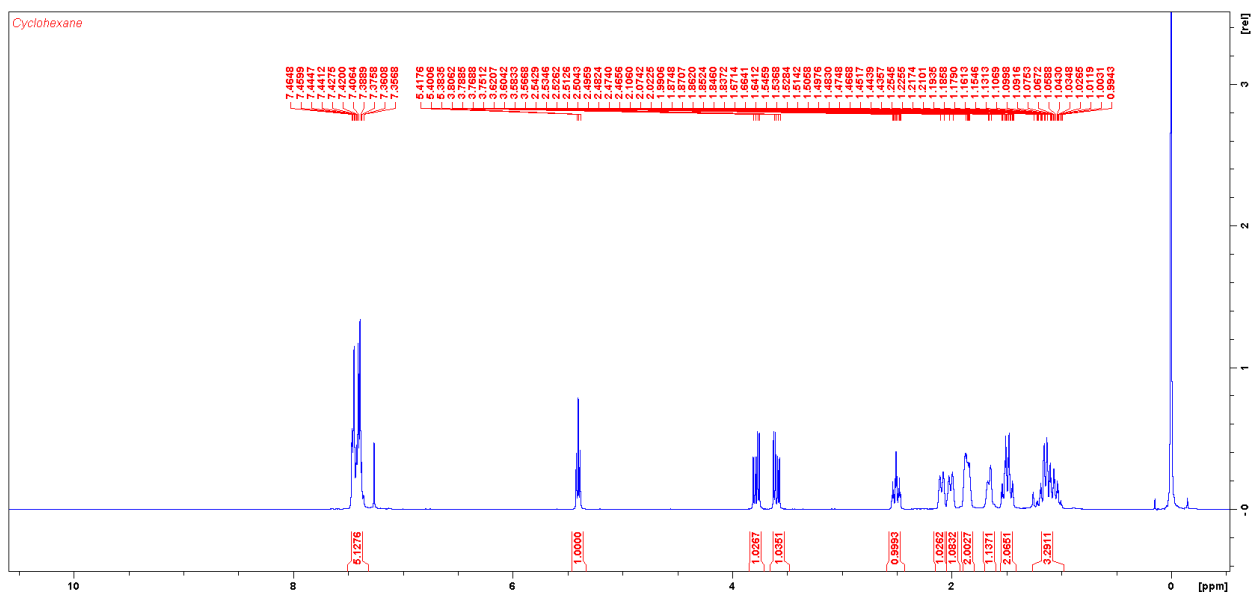
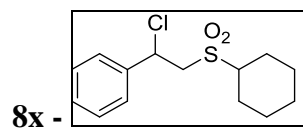


Figure S47. ^1H NMR spectrum of **8x**.

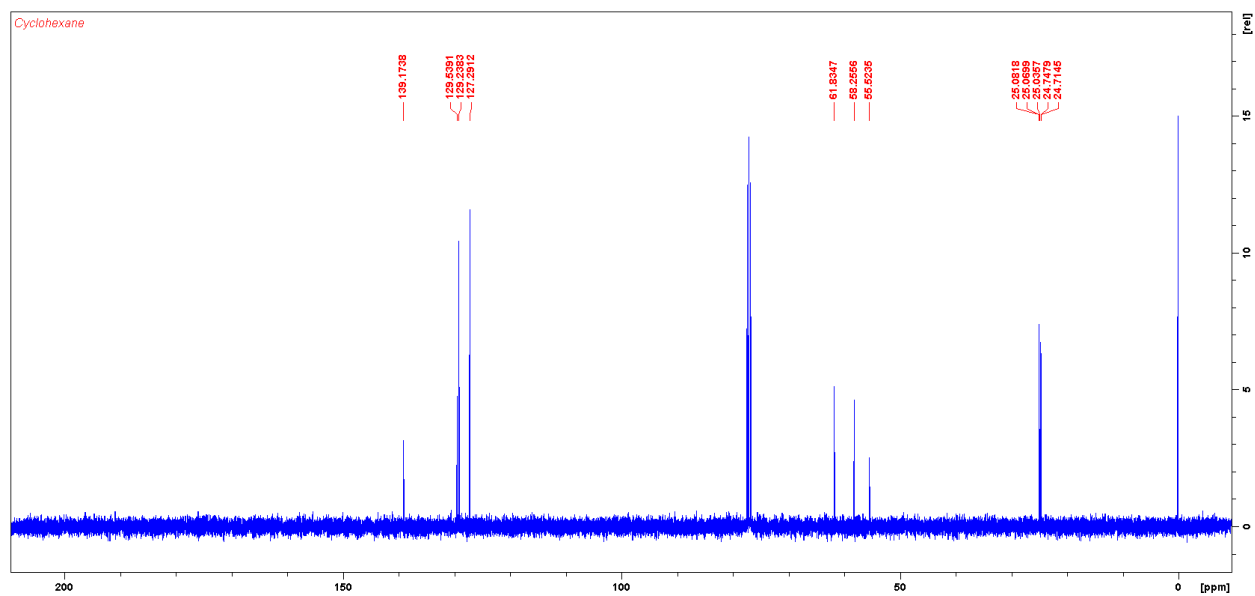


Figure S48. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8x**.

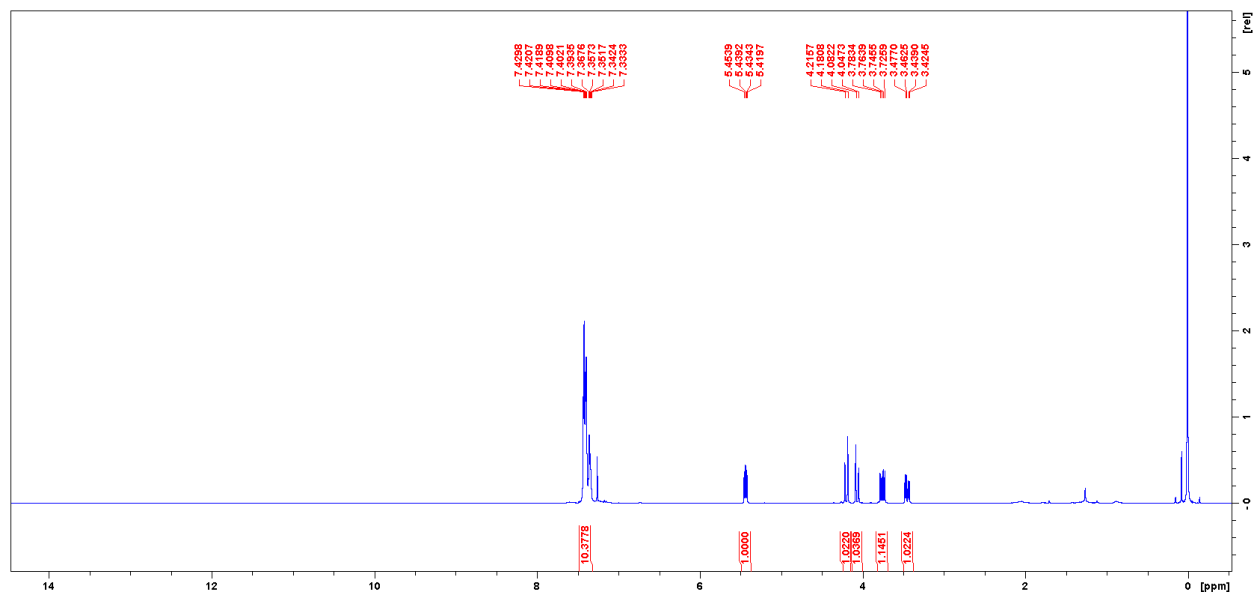
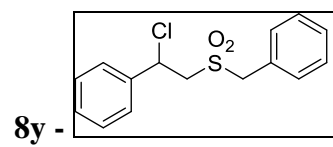


Figure S49. ^1H NMR spectrum of **8y**.

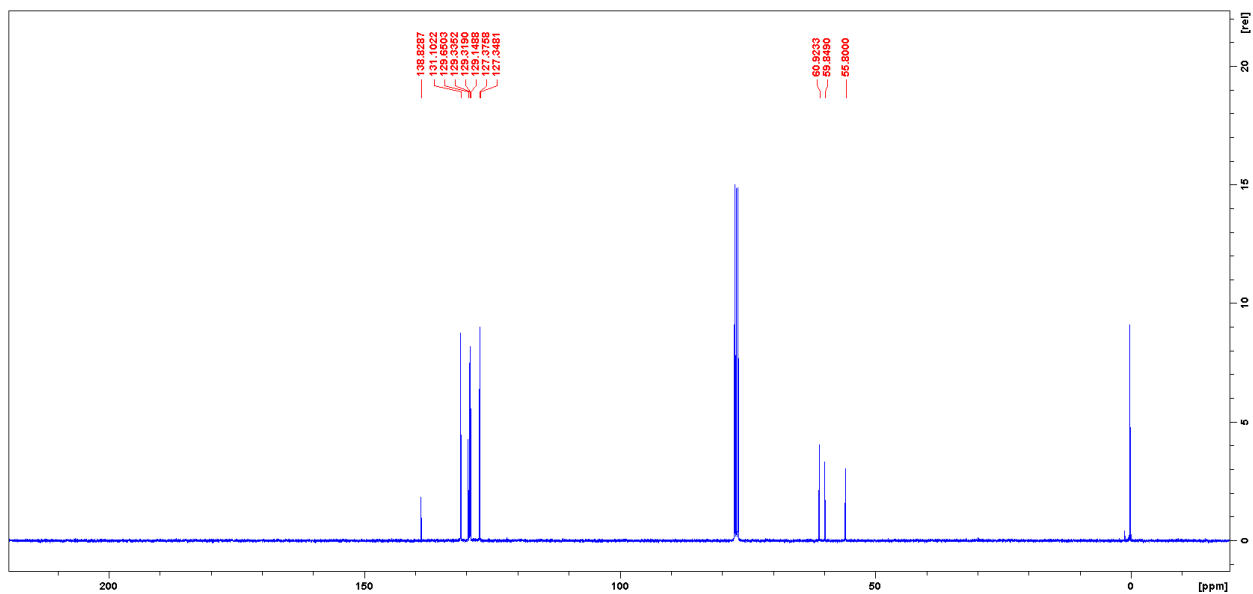


Figure S50. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8y**.

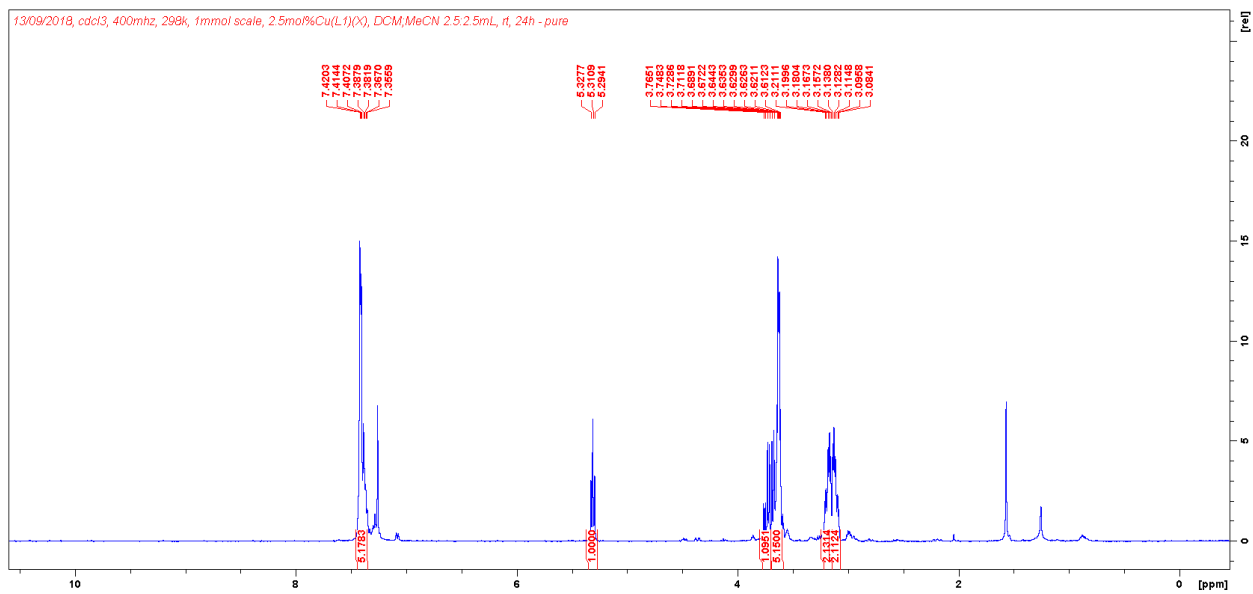
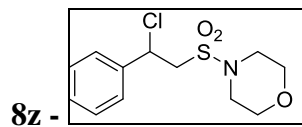


Figure S51. ^1H NMR spectrum of **8z**.

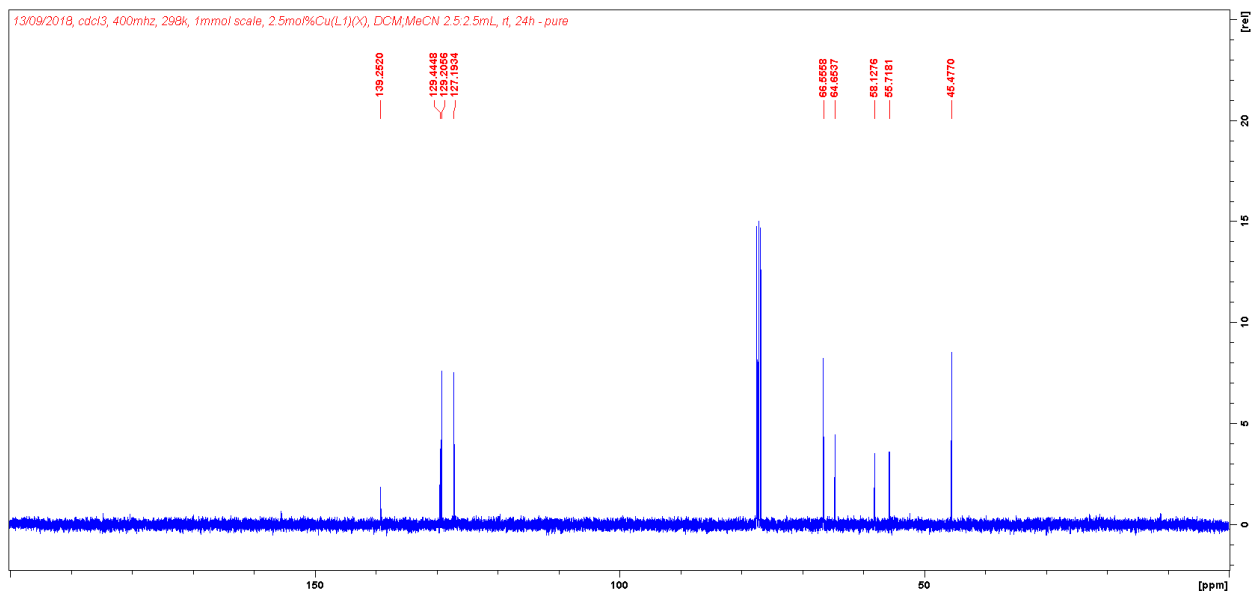


Figure S52. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8z**.

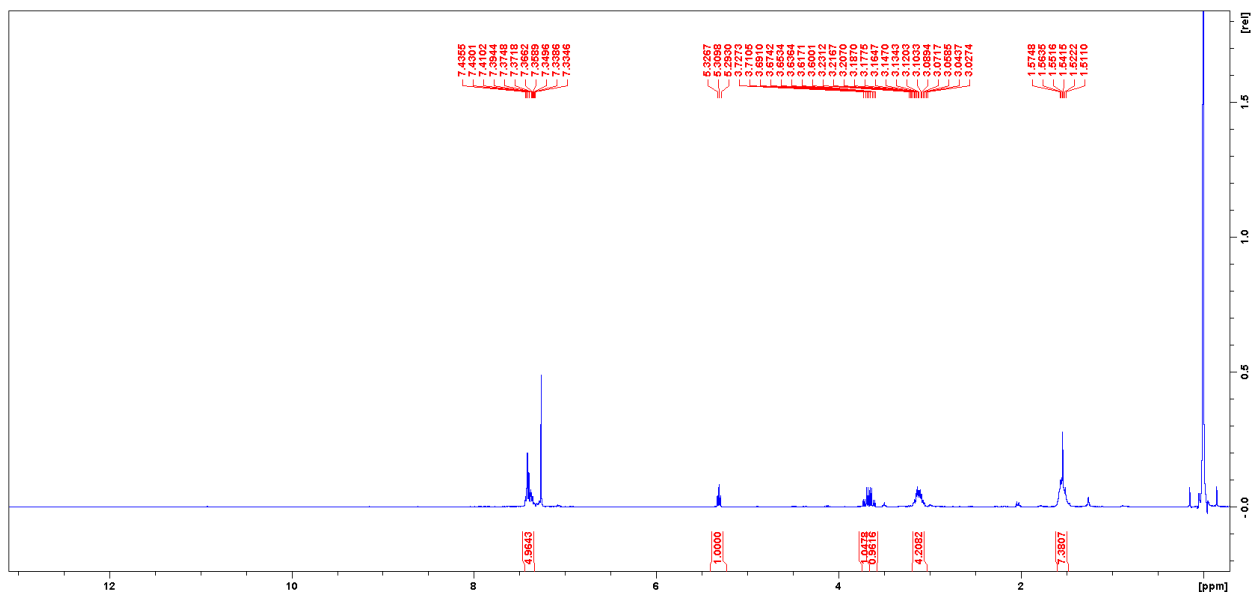
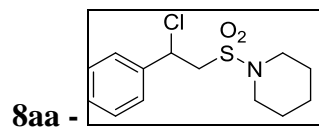


Figure S53. ^1H NMR spectrum of **8aa**.

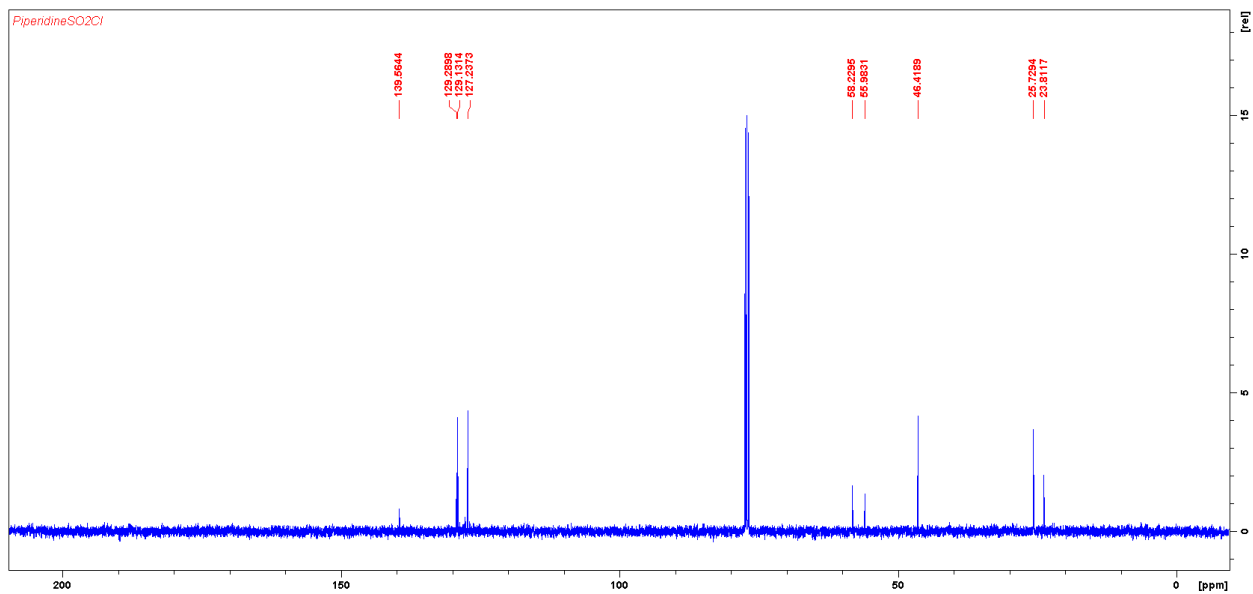


Figure S54. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8aa**.

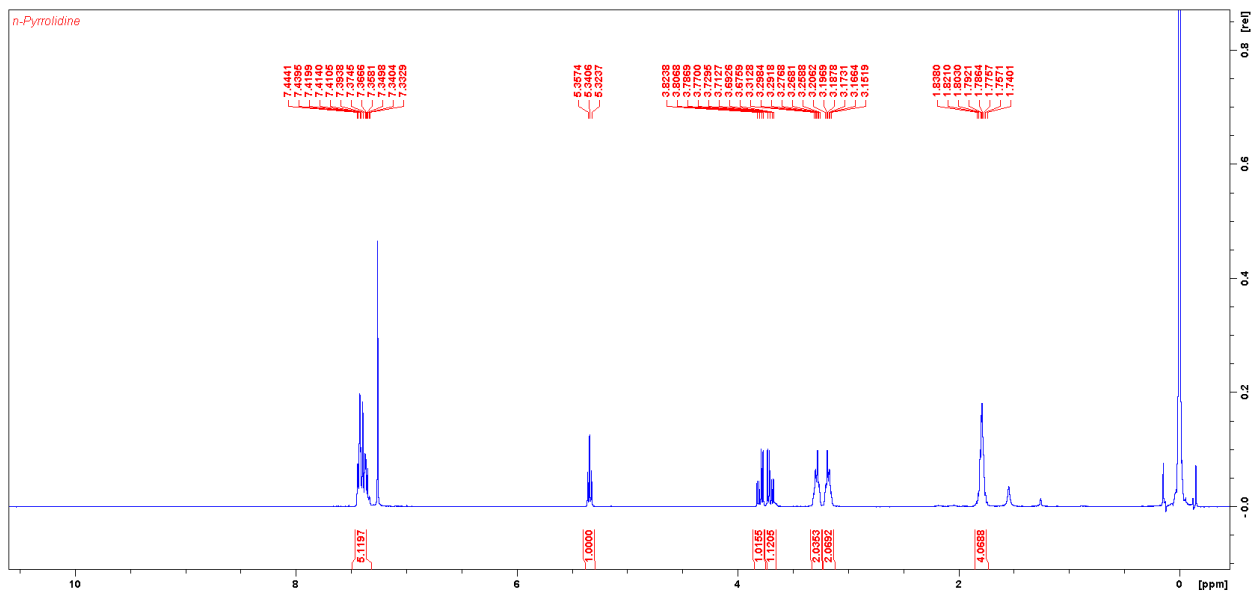
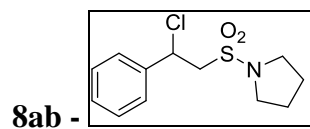


Figure S55. ^1H NMR spectrum of **8ab**.

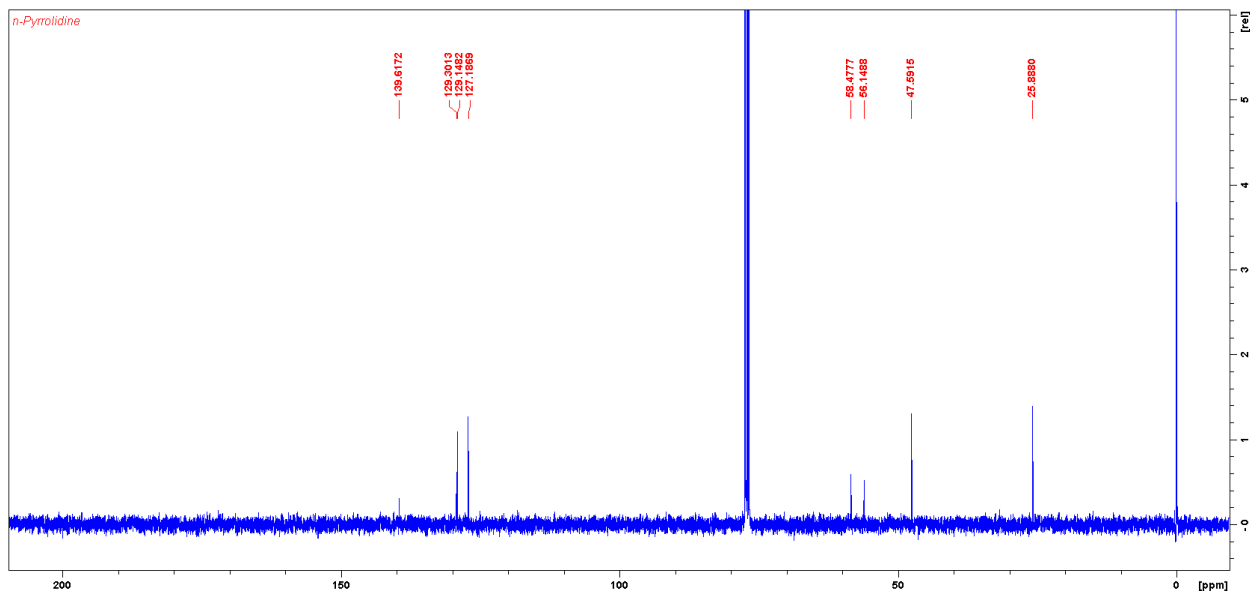


Figure S56. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **8ab**.

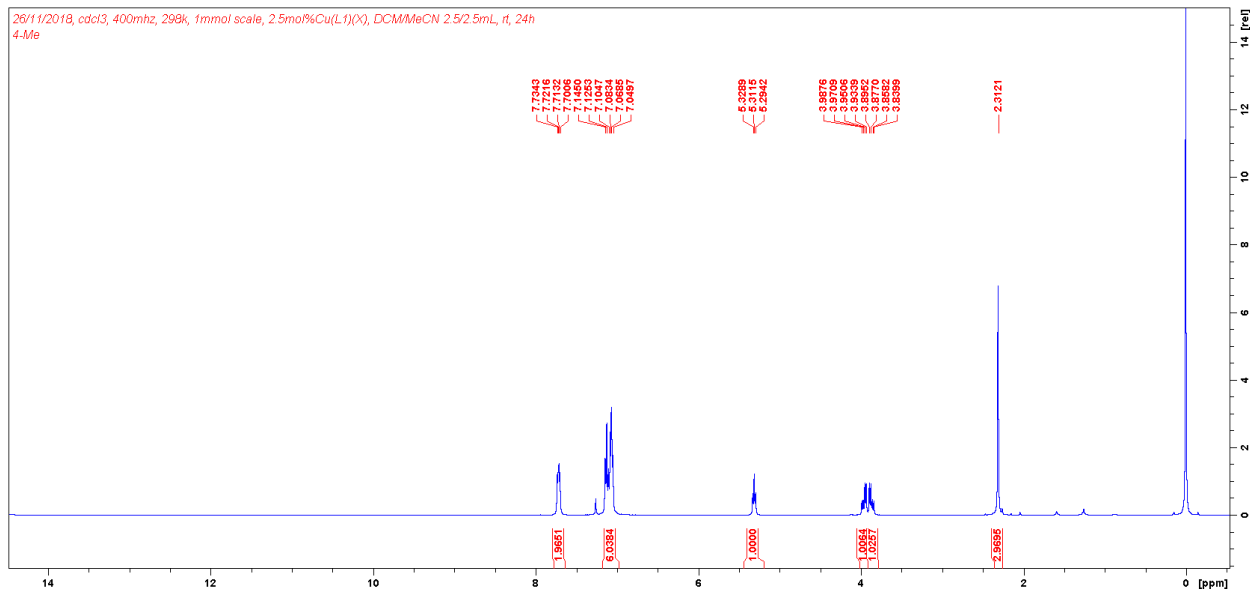
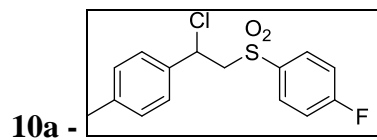


Figure S57. ^1H NMR spectrum of **10a**.

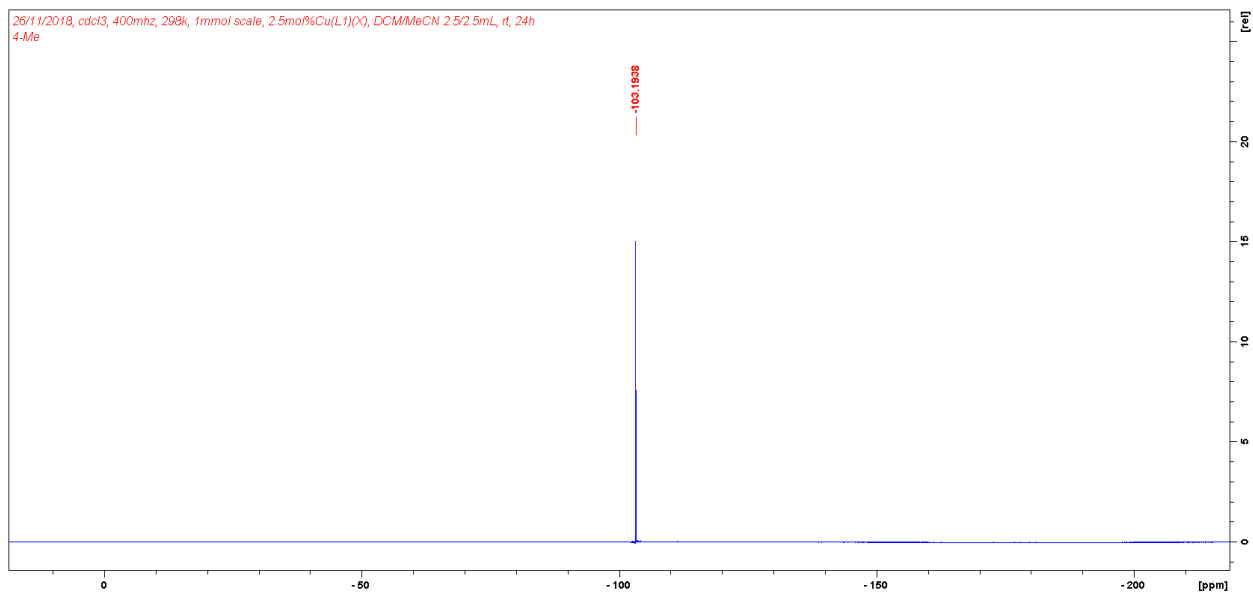


Figure S58. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10a**.

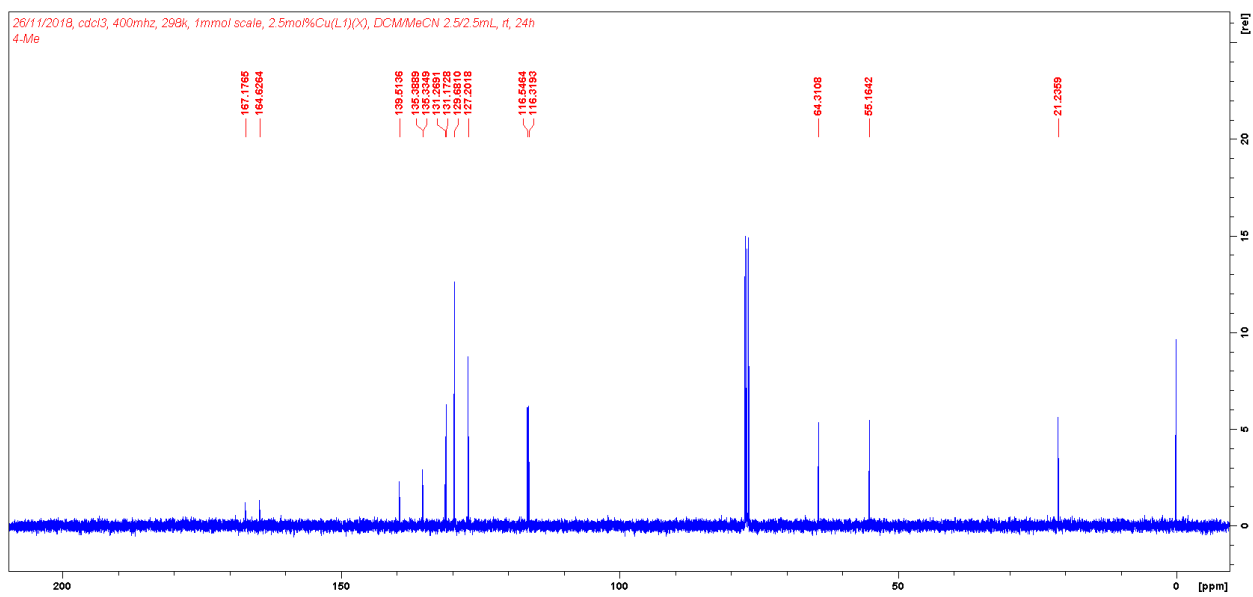
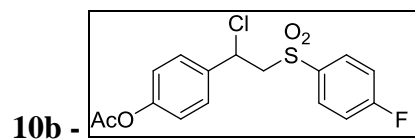


Figure S59. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10a**.



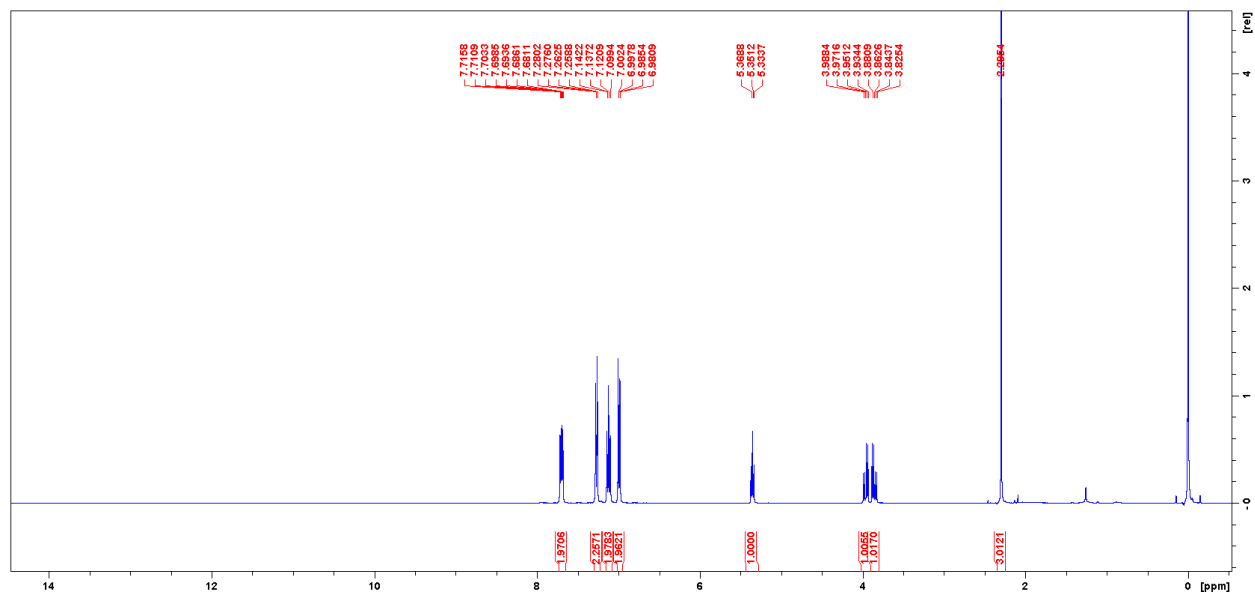


Figure S60. ^1H NMR spectrum of **10b**.

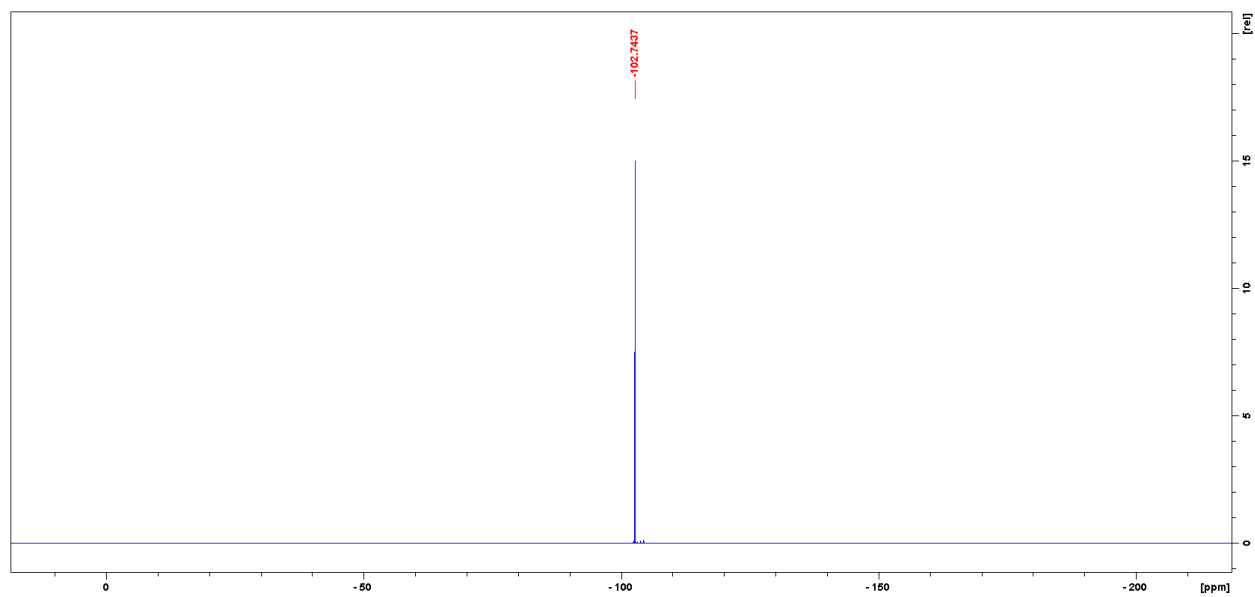


Figure S61. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10b**.

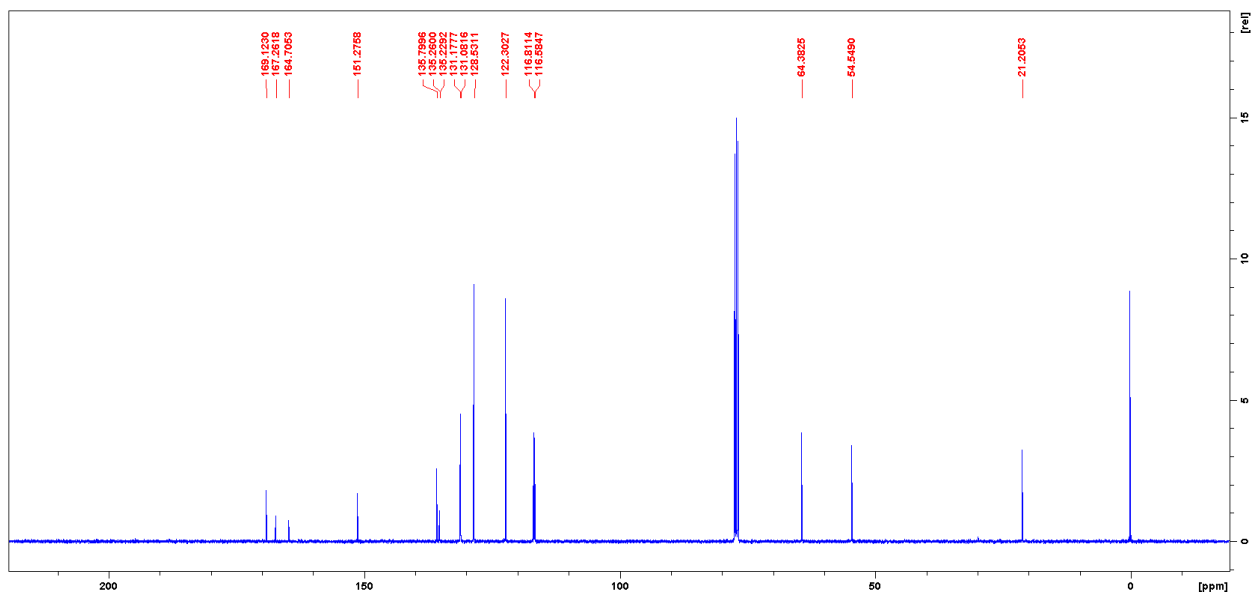


Figure S62. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10b**.

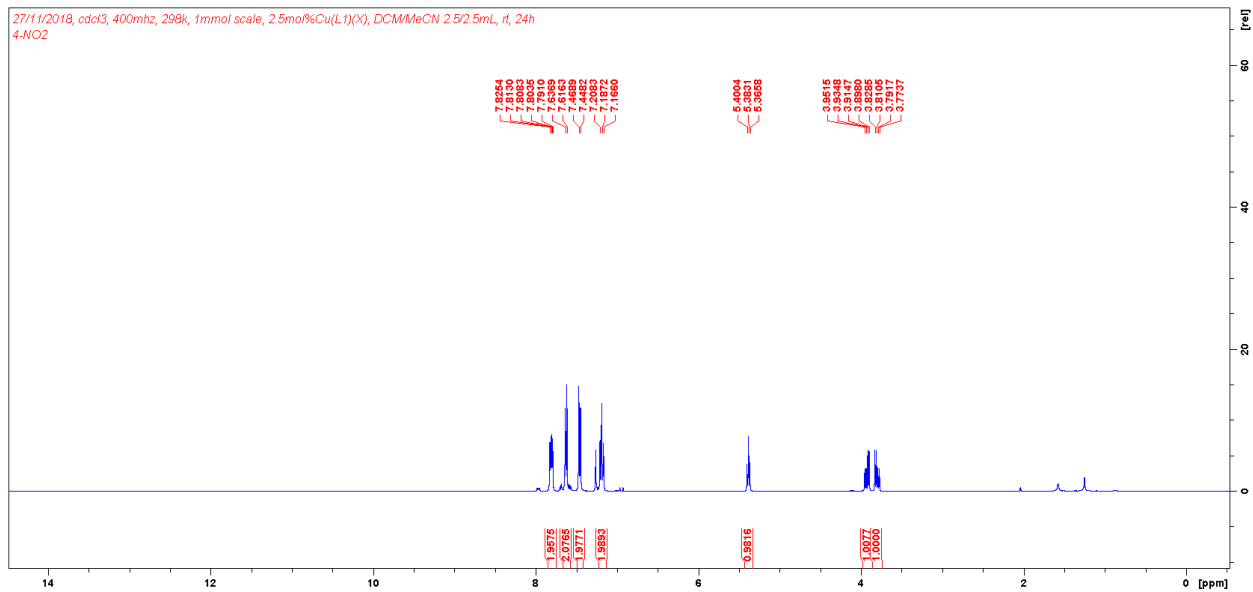
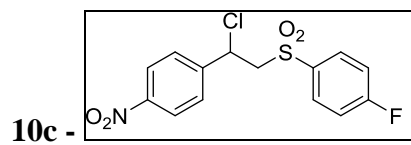


Figure S63. ^1H NMR spectrum of **10c**.

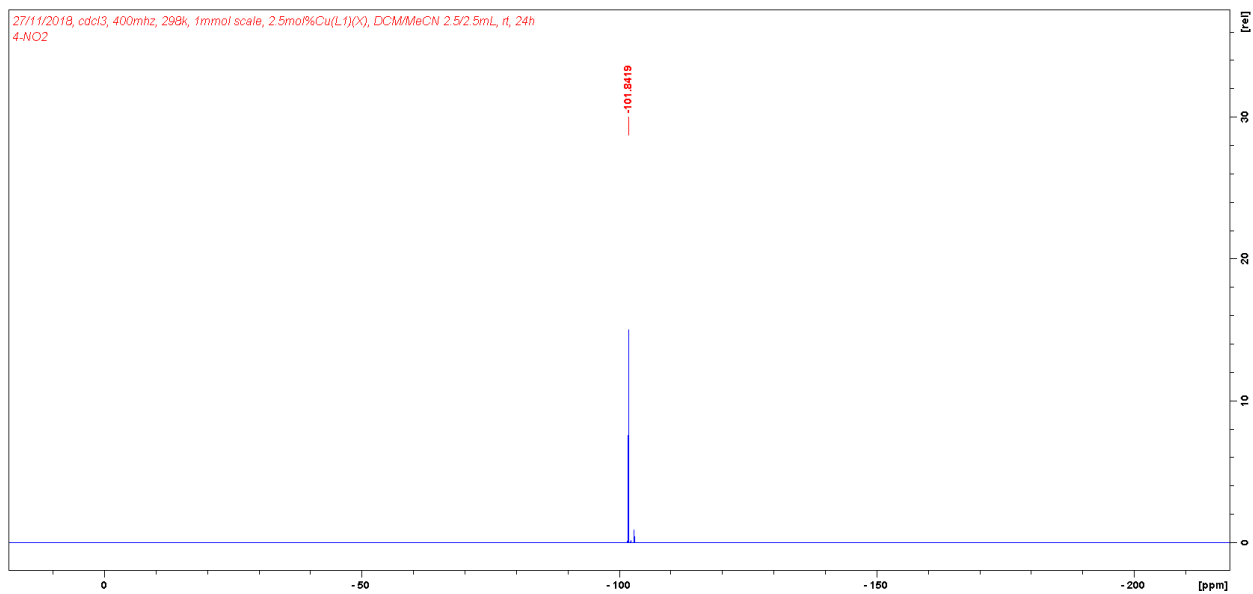


Figure S64. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10c**.

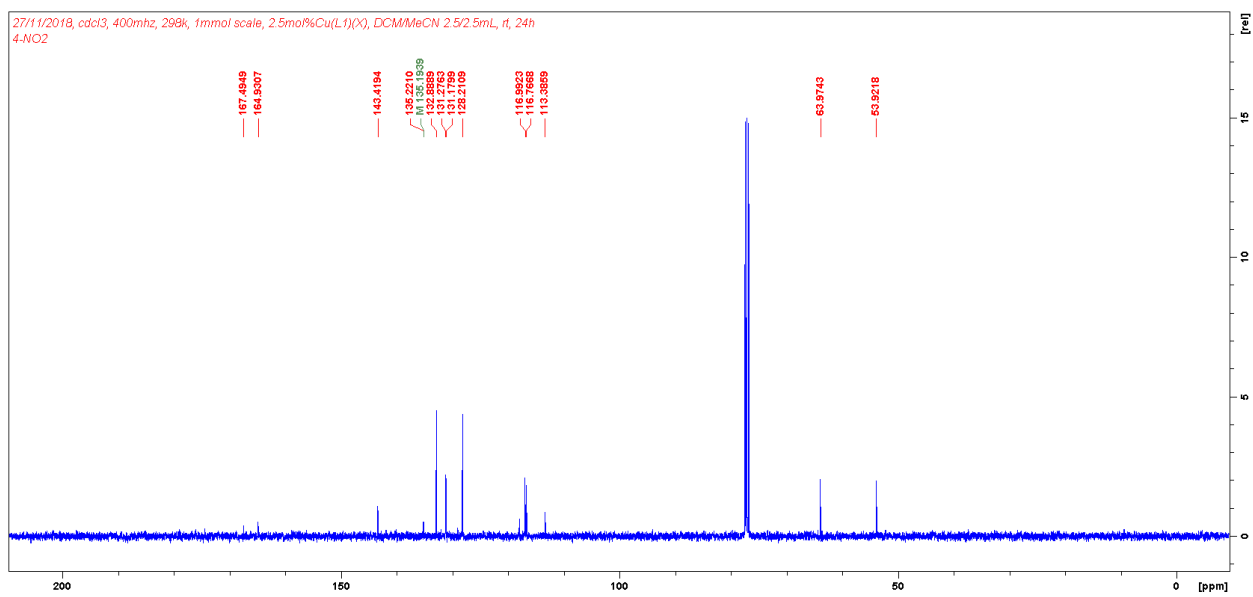
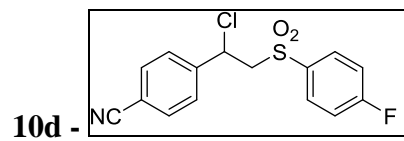


Figure S65. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10c**.



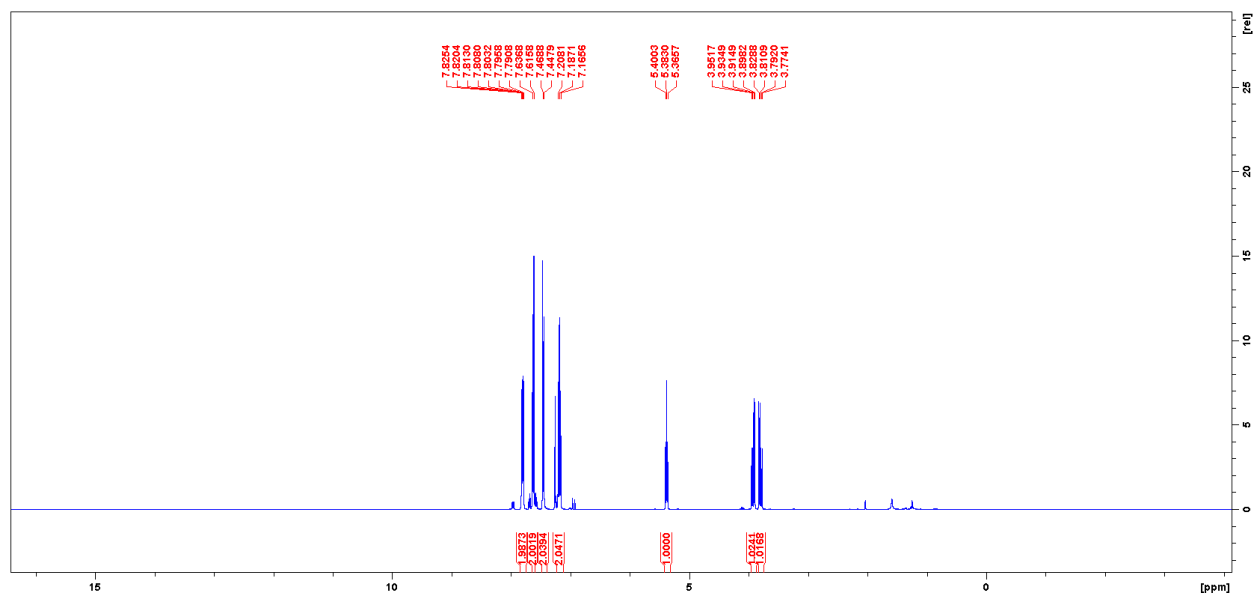


Figure S66. ^1H NMR spectrum of **10d**.

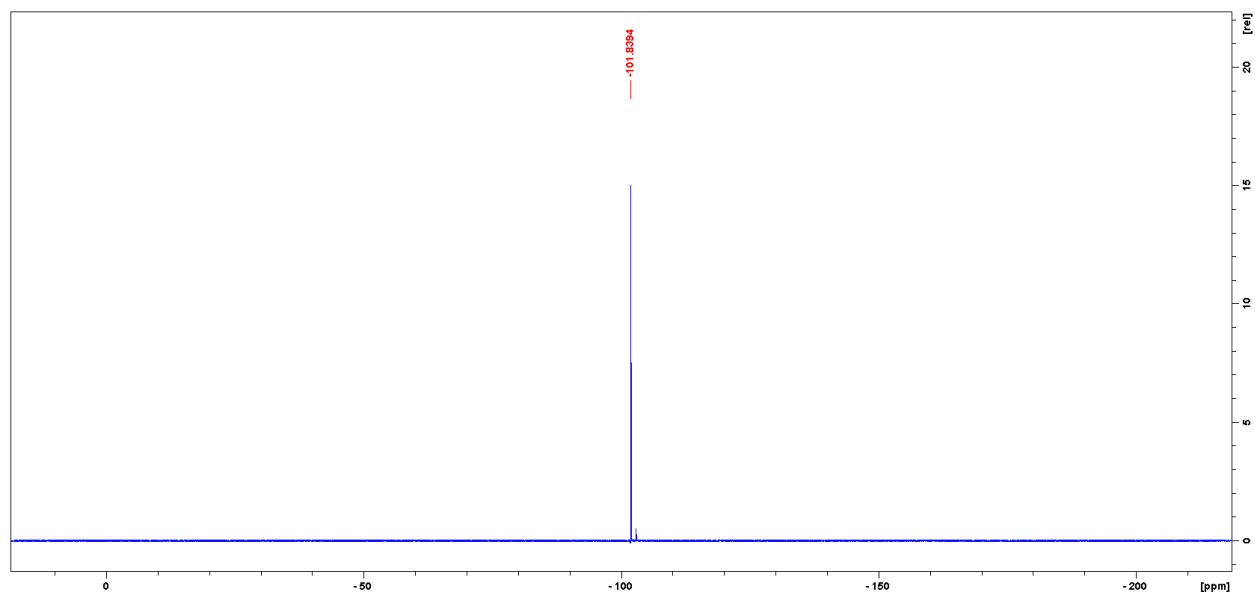


Figure S67. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10d**.

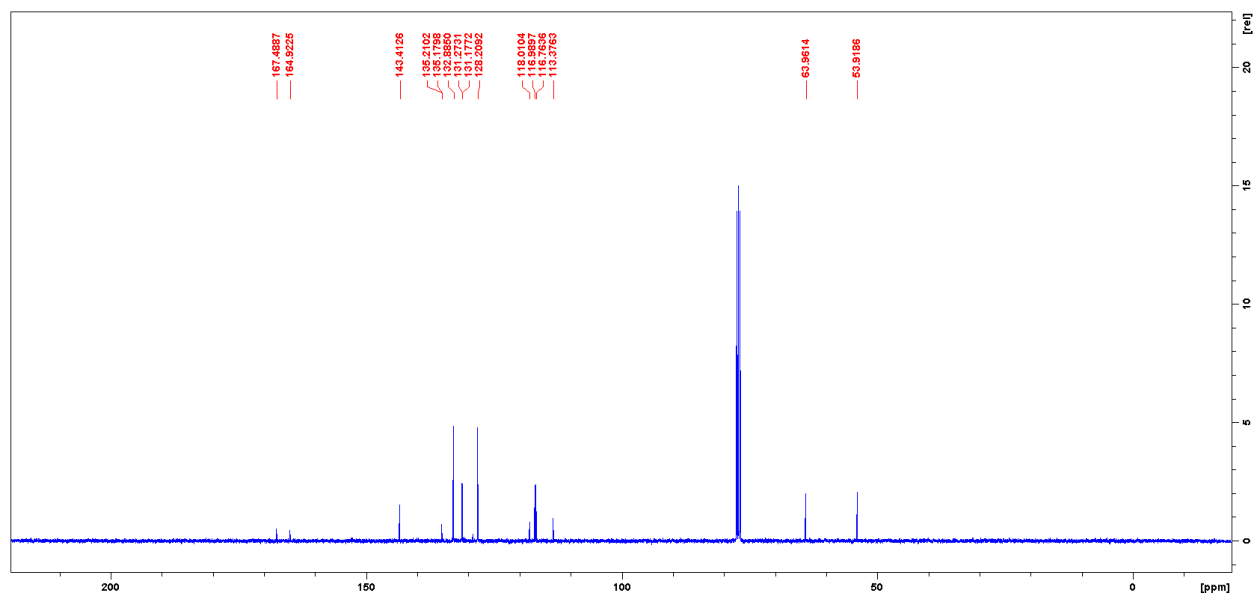


Figure S68. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10d**.

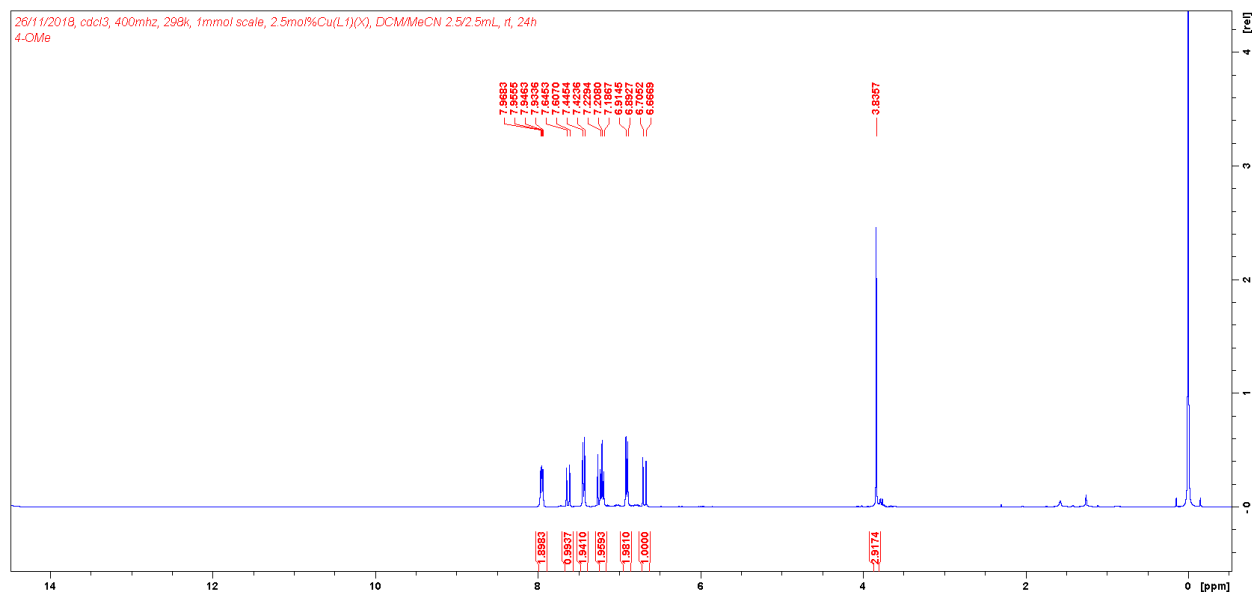
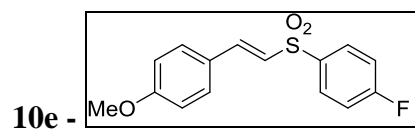


Figure S69. ^1H NMR spectrum of **10e**.

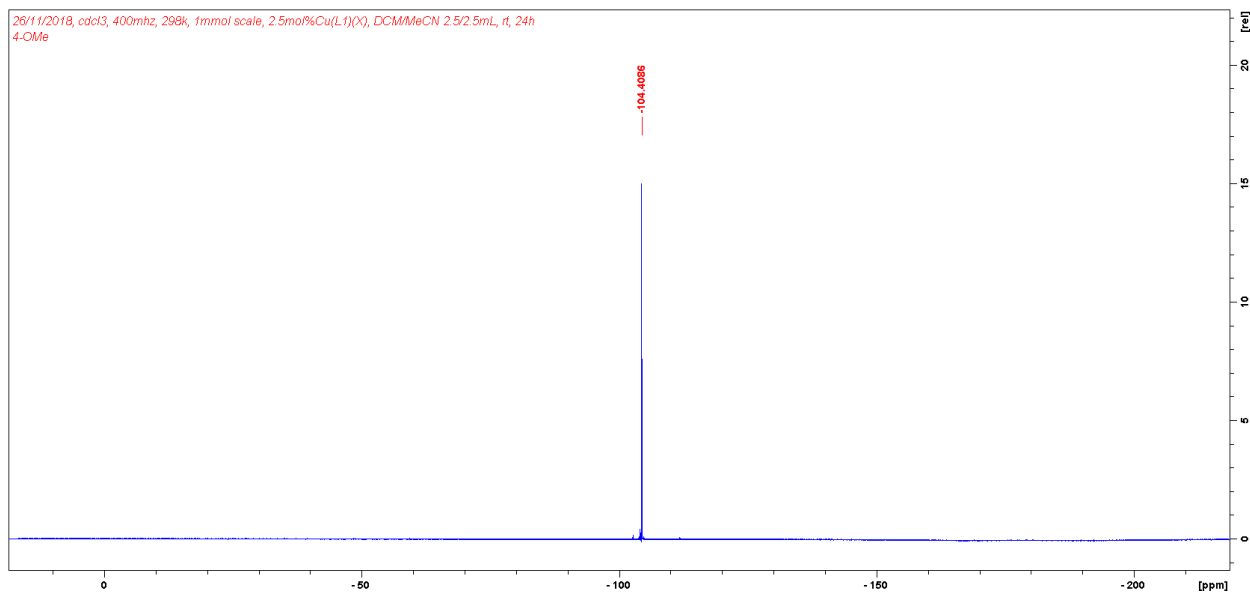


Figure S70. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10e**.

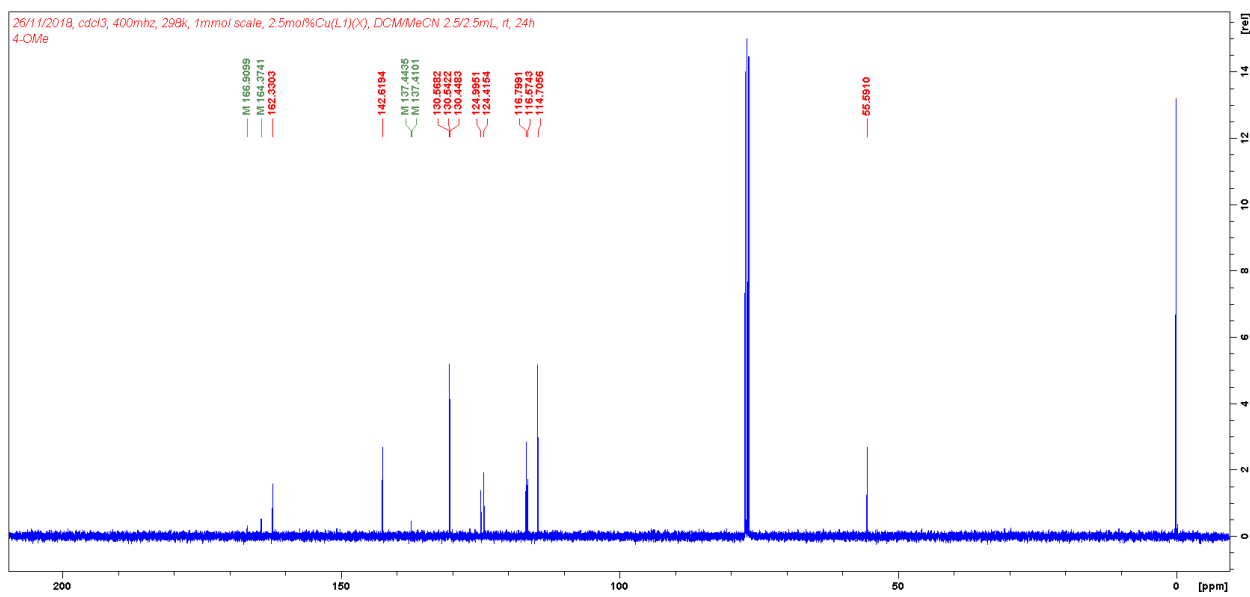
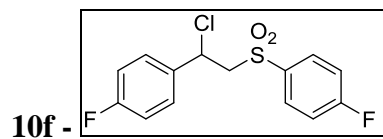


Figure S71. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10e**.



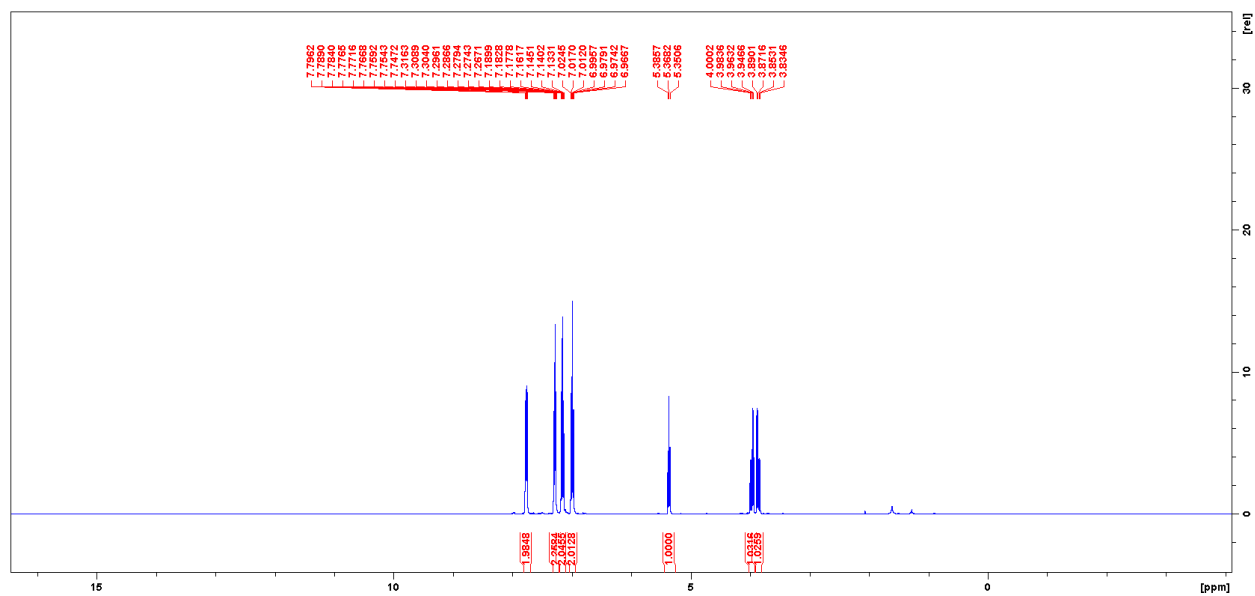


Figure S72. ^1H NMR spectrum of **10f**.

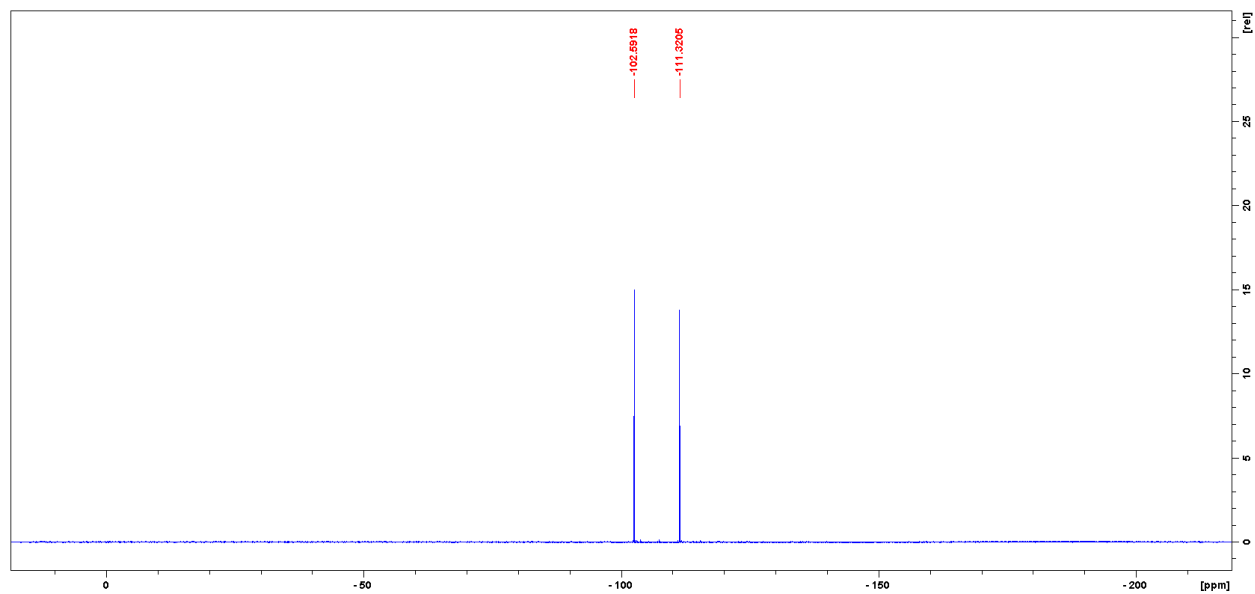


Figure S73. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10f**.

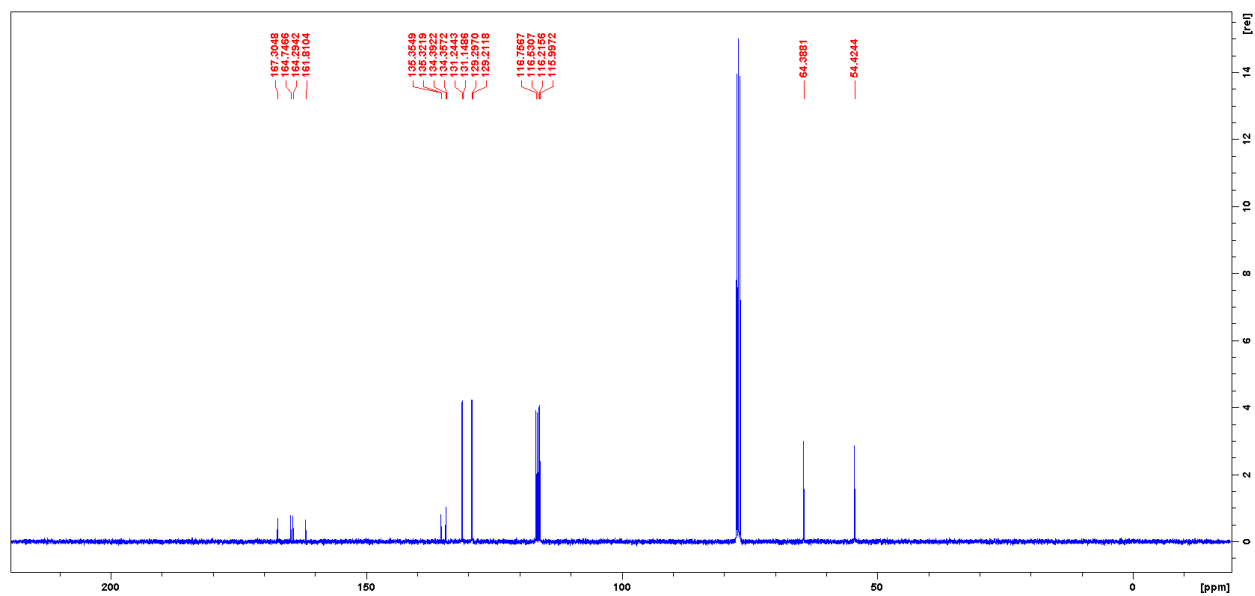


Figure S74. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10f**.

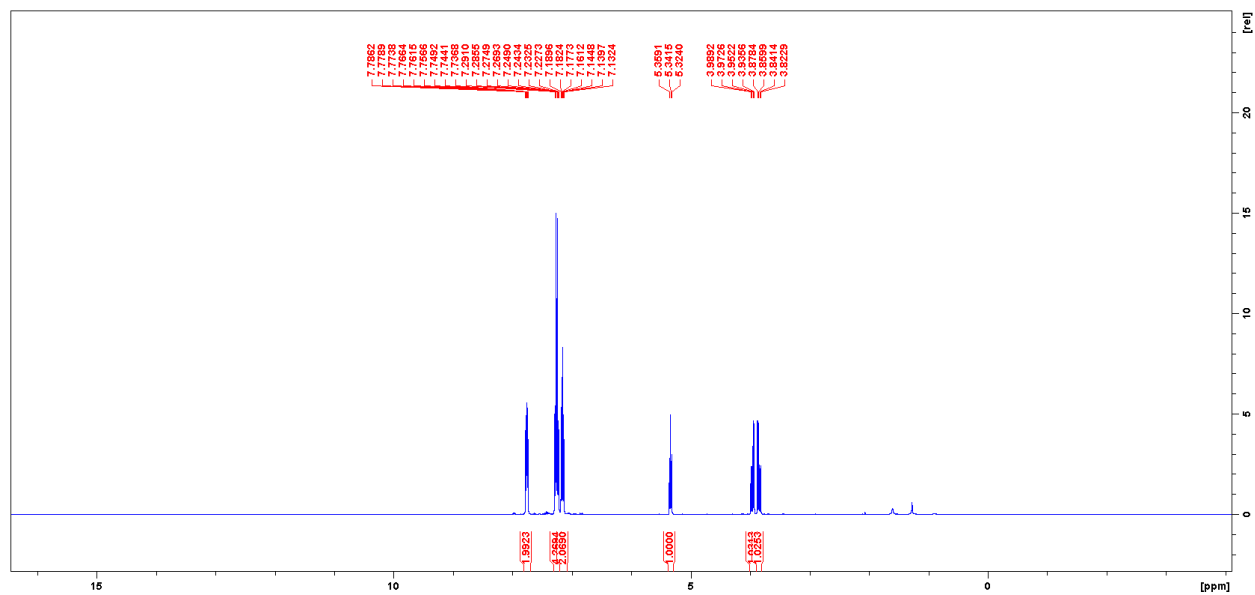
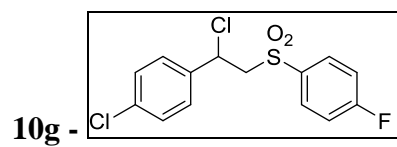


Figure S75. ^1H NMR spectrum of **10g**.

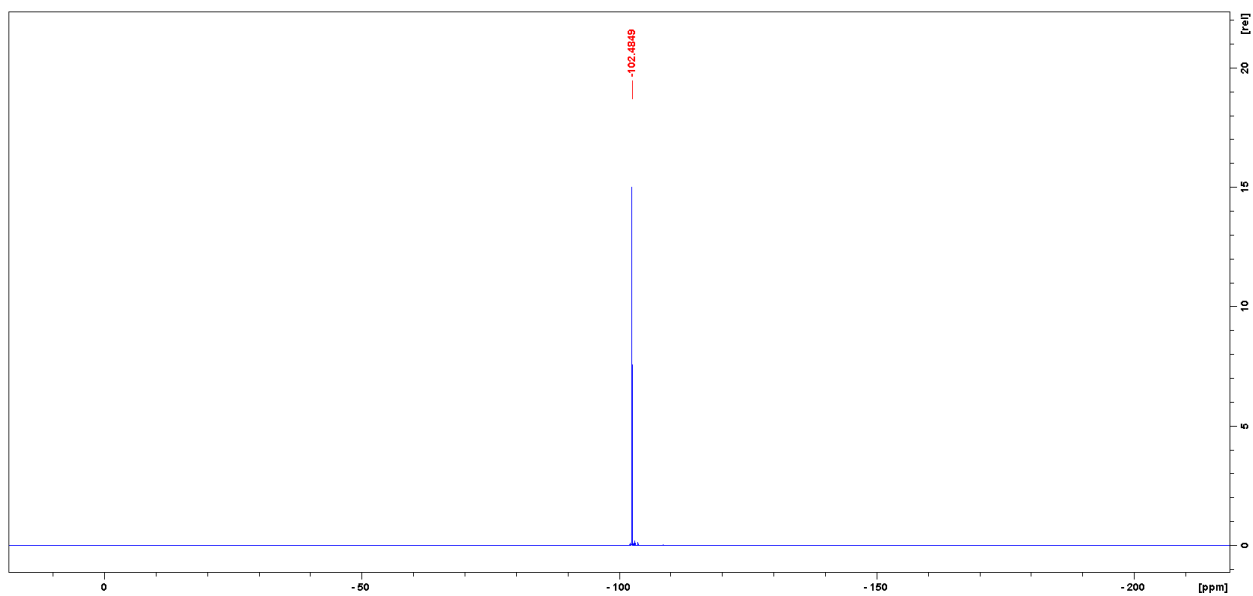


Figure S76. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10g**.

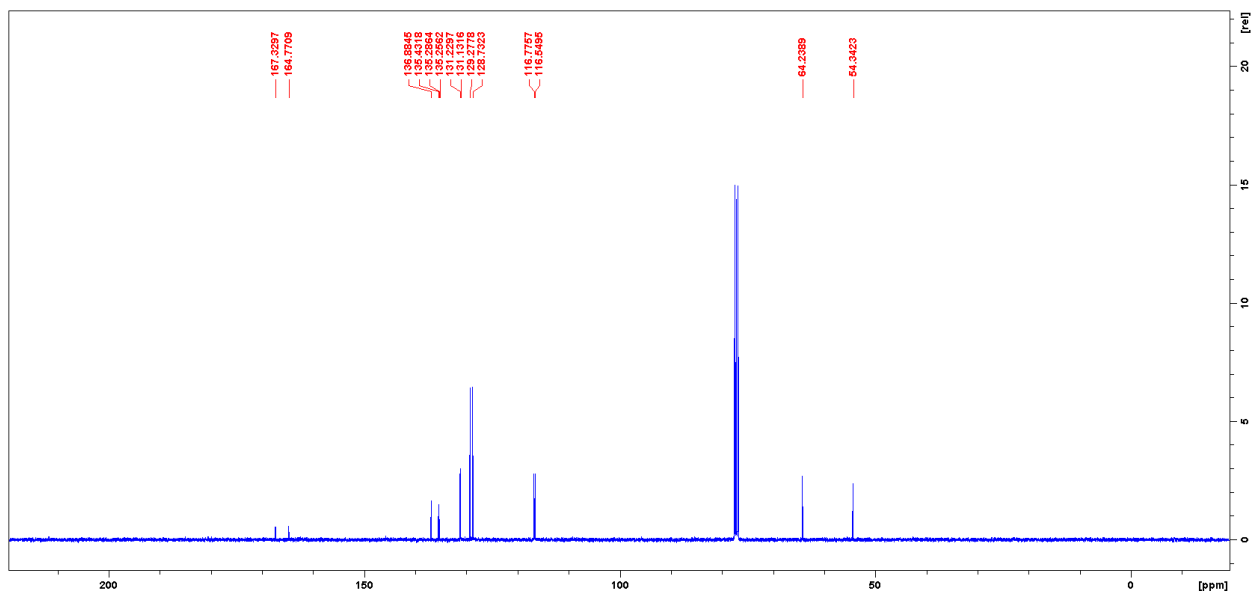
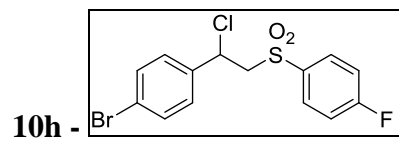


Figure S77. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10g**.



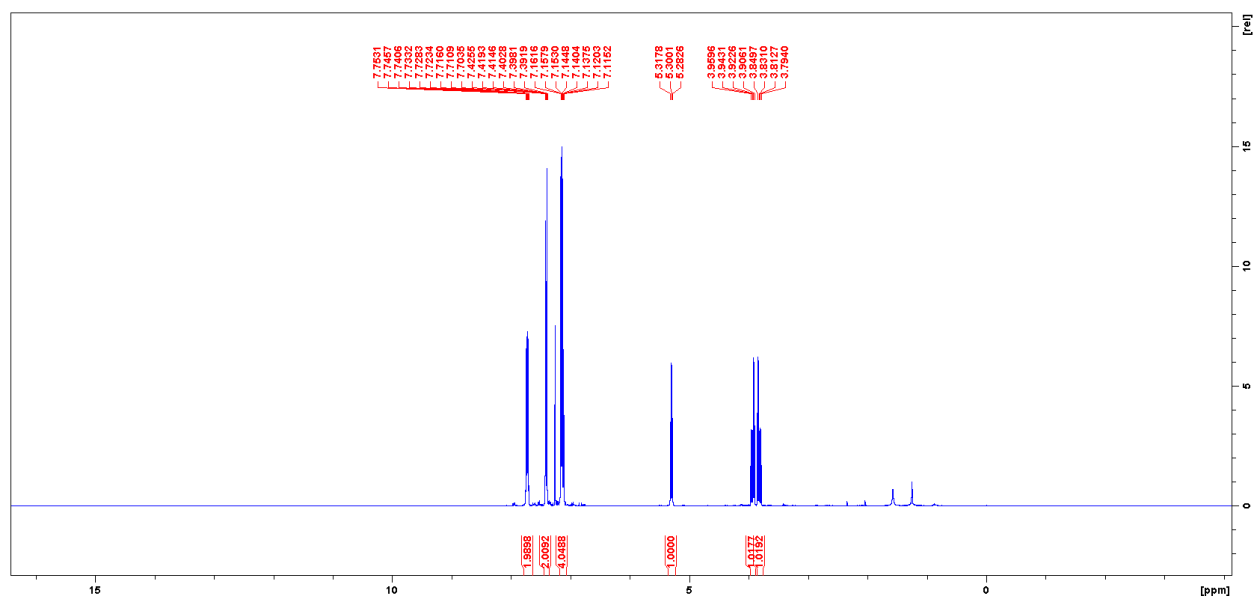


Figure S78. ^1H NMR spectrum of **10h**.

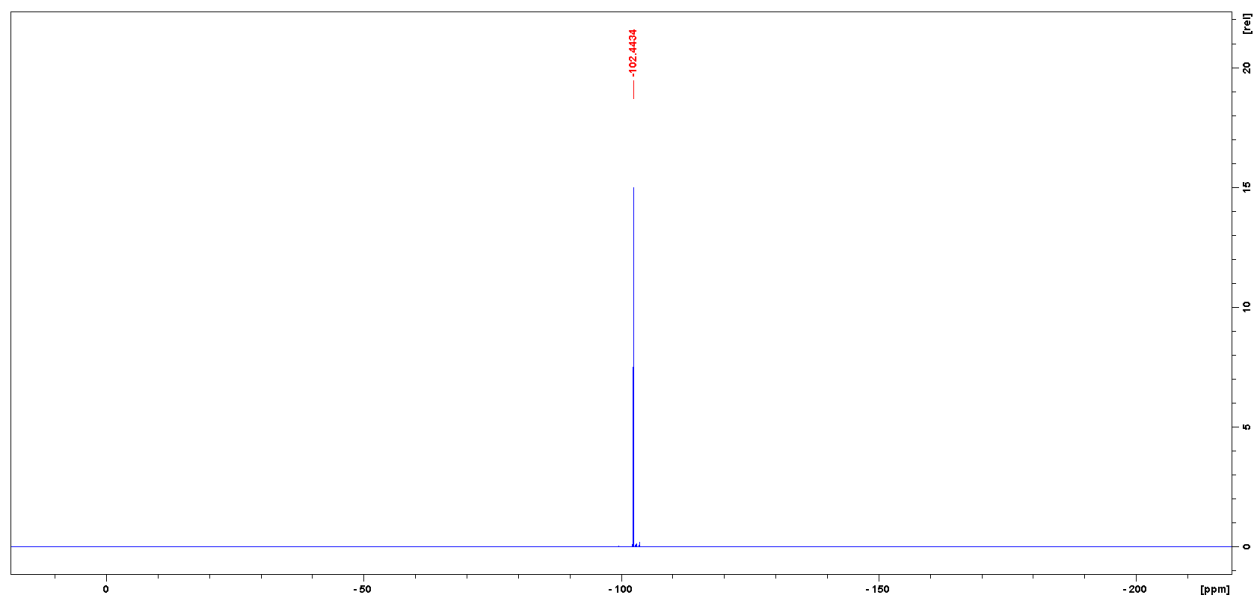


Figure S79. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10h**.

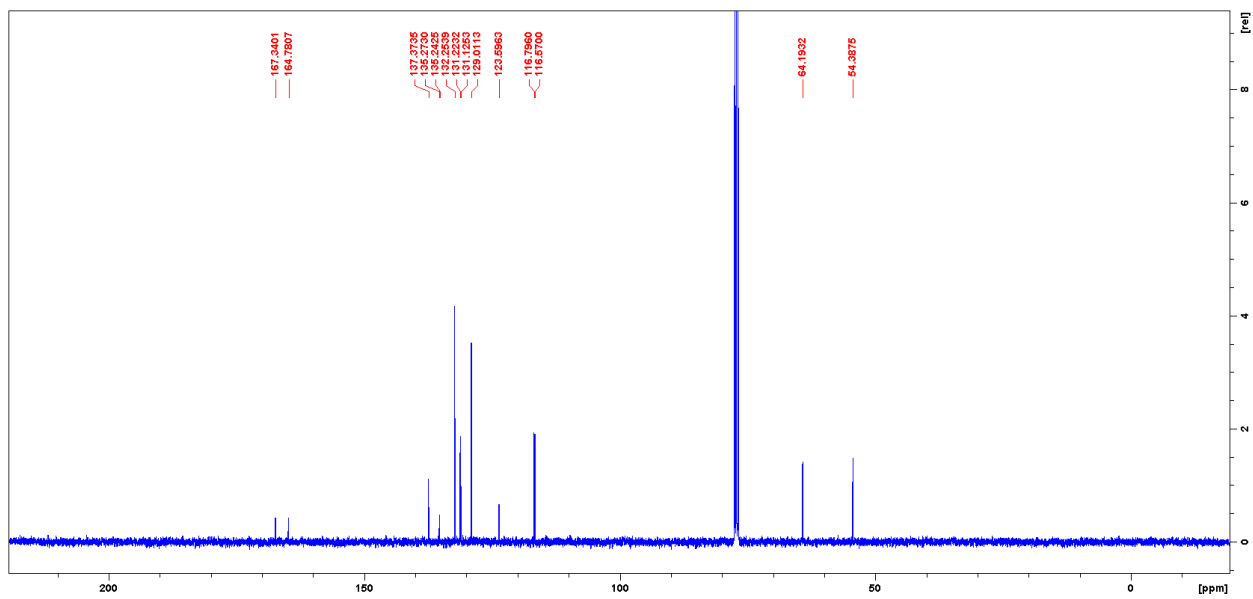


Figure S80. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10h**.

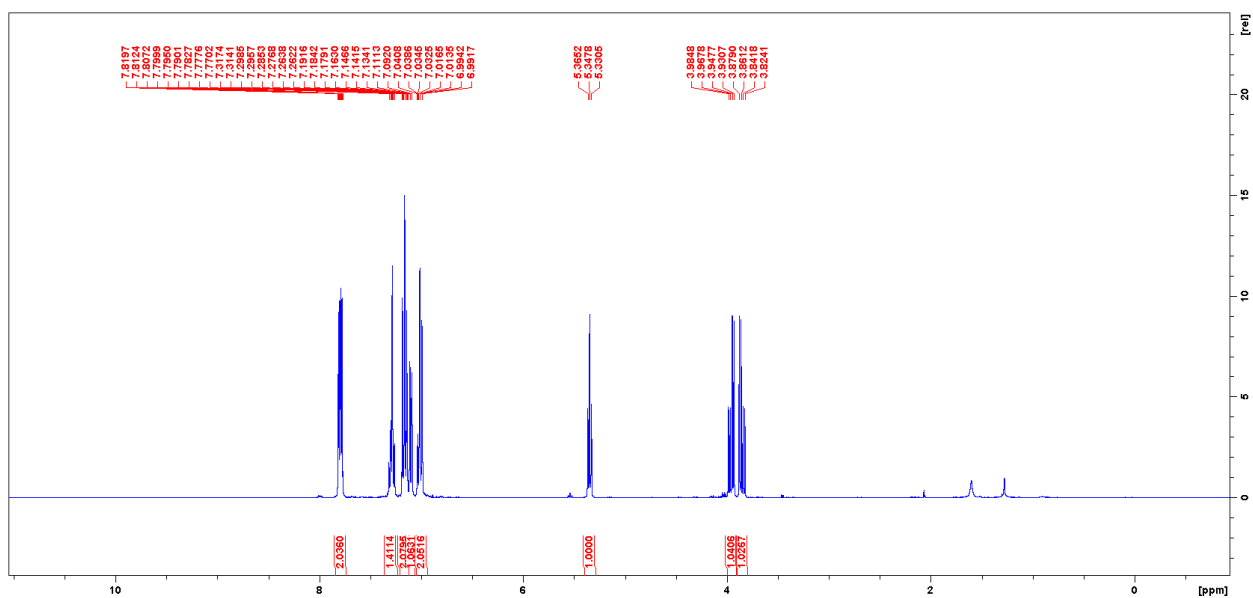
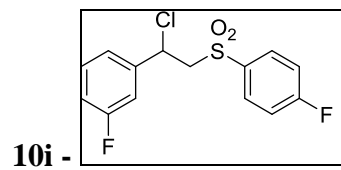


Figure S81. ^1H NMR spectrum of **10i**.

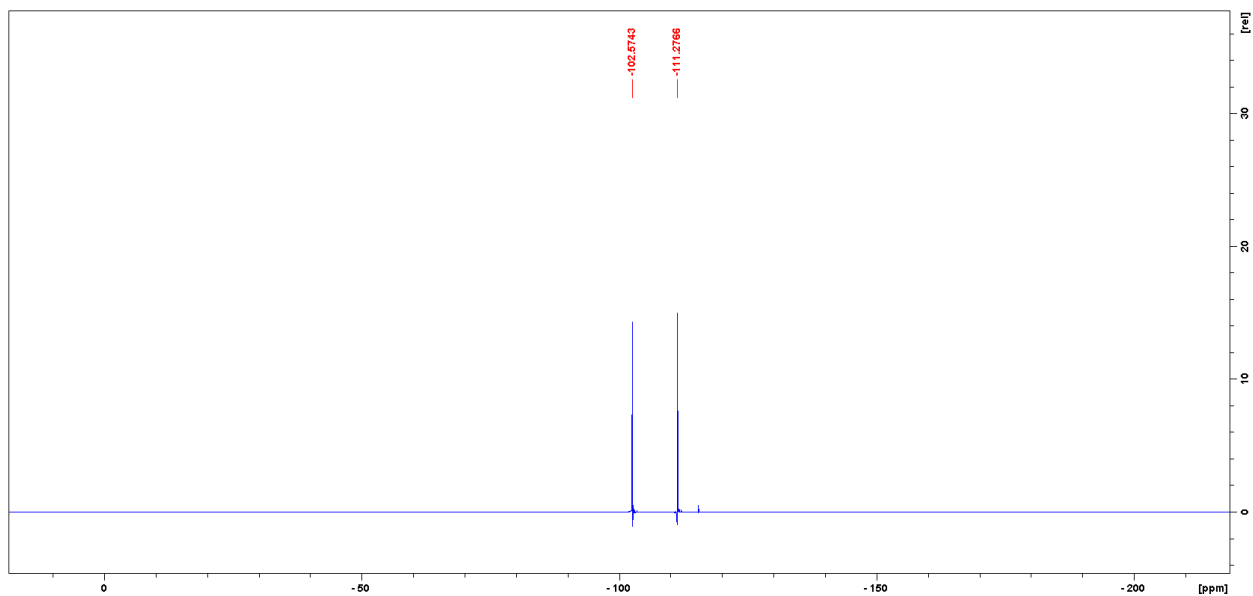


Figure S82. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10i**.

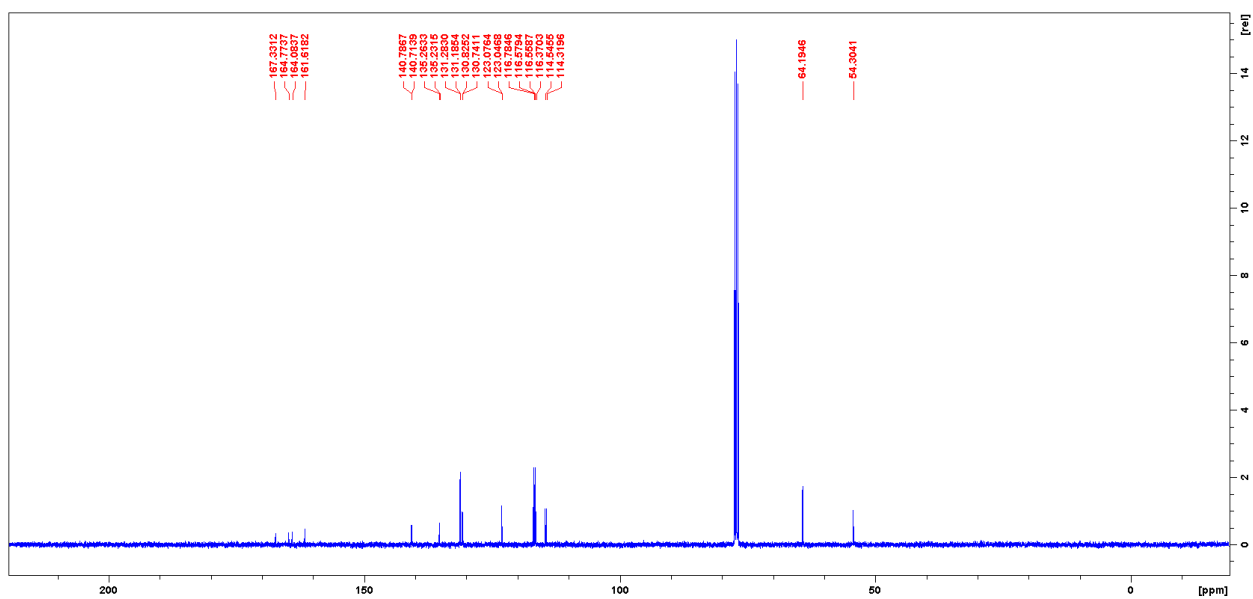
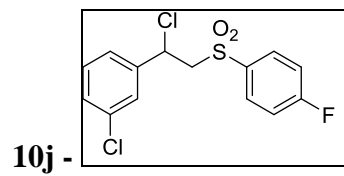


Figure S83. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10i**.



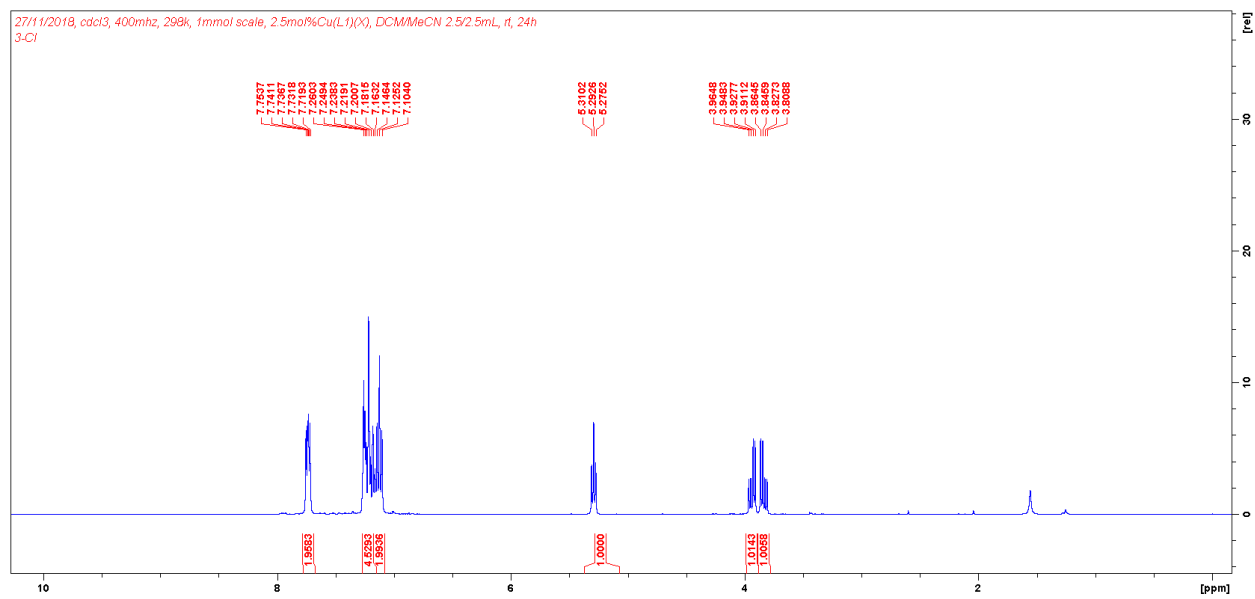


Figure S84. ^1H NMR spectrum of **10j**.

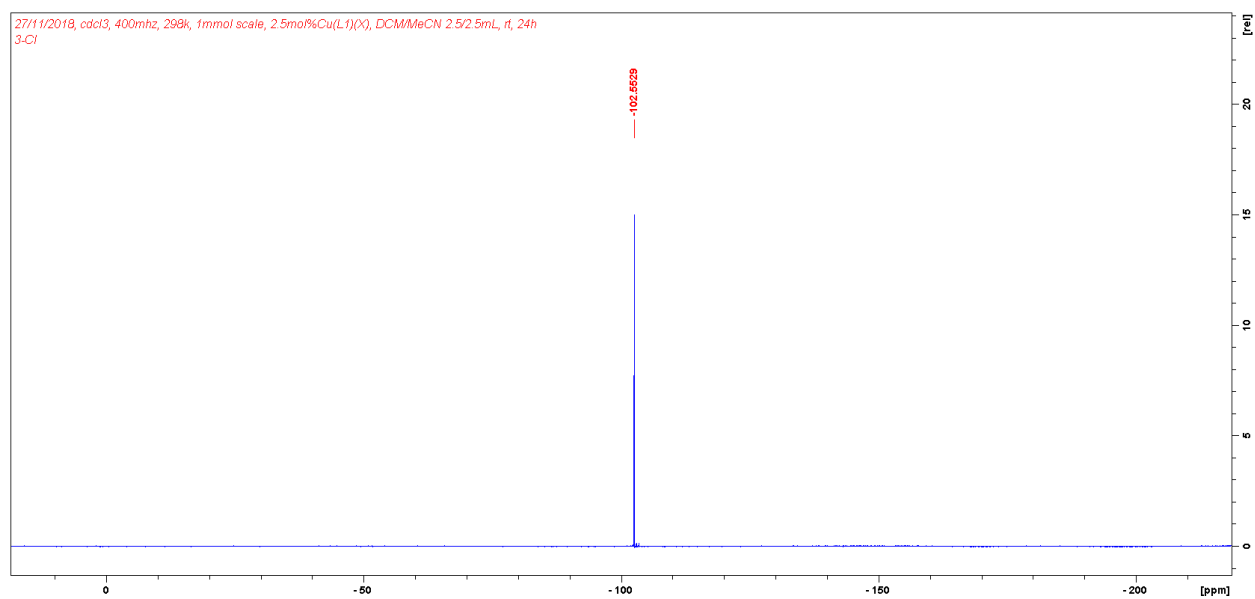


Figure S85. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10j**.

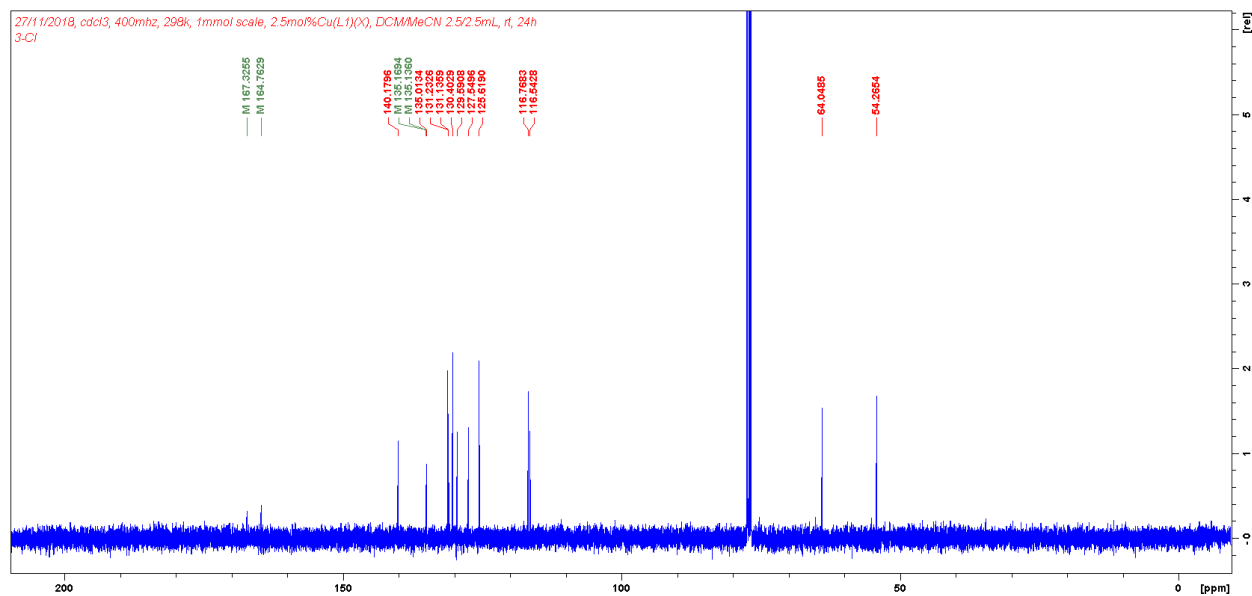


Figure S86. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10j**.

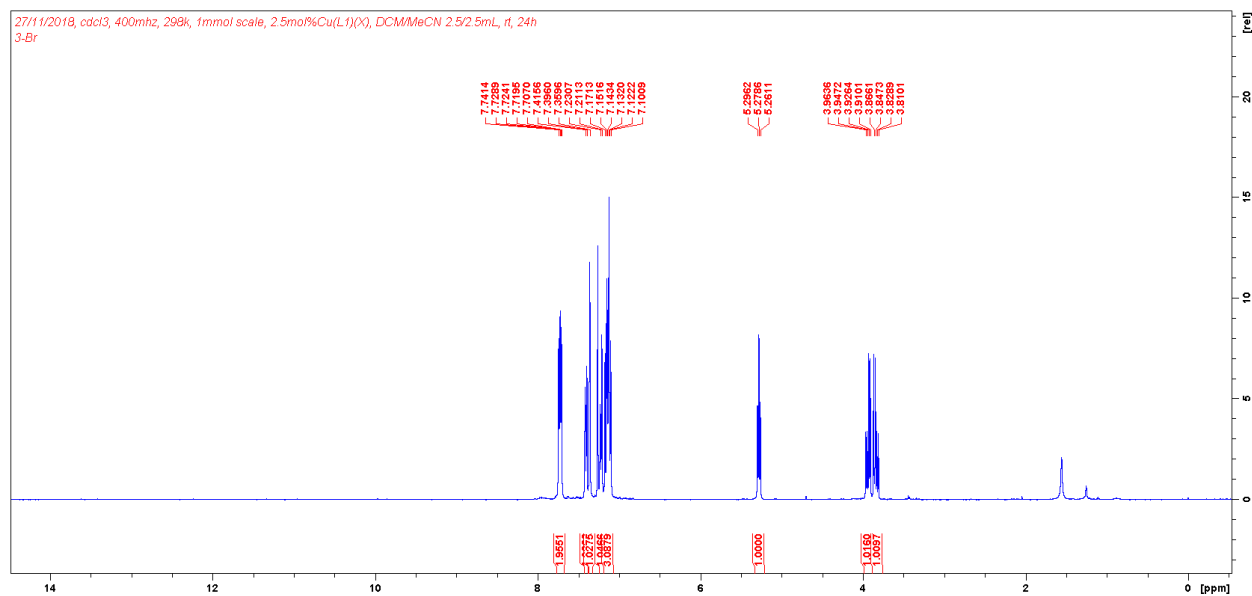
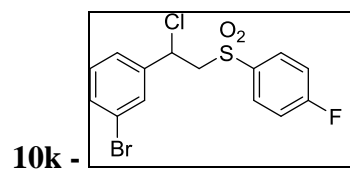


Figure S87. ^1H NMR spectrum of **10k**.

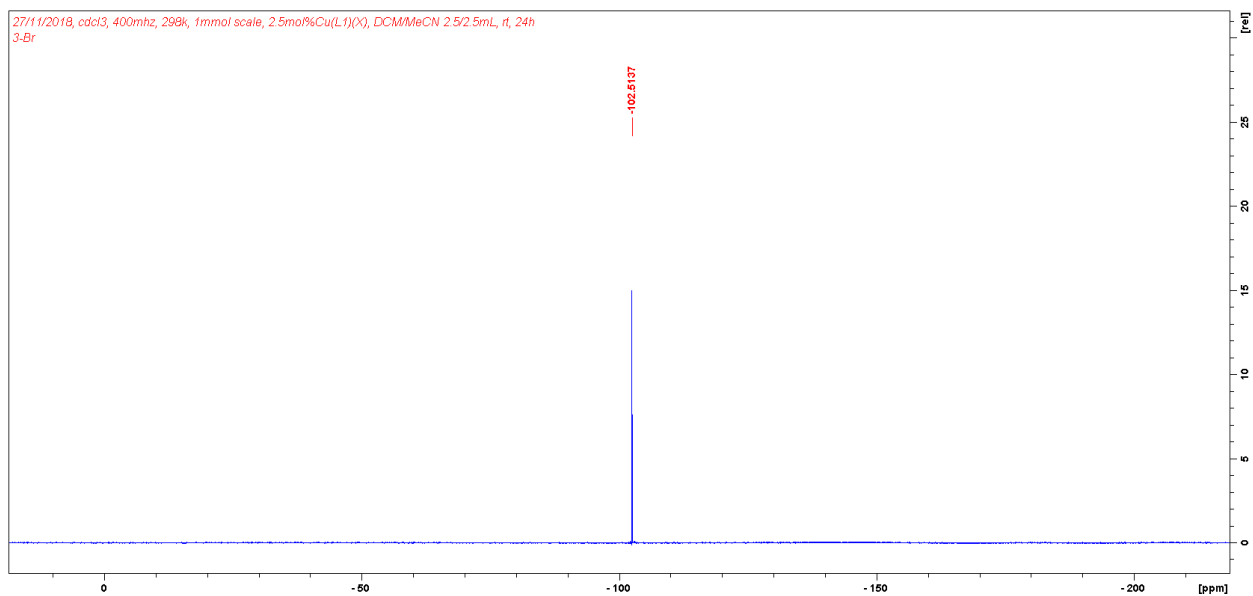


Figure S88. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10k**.

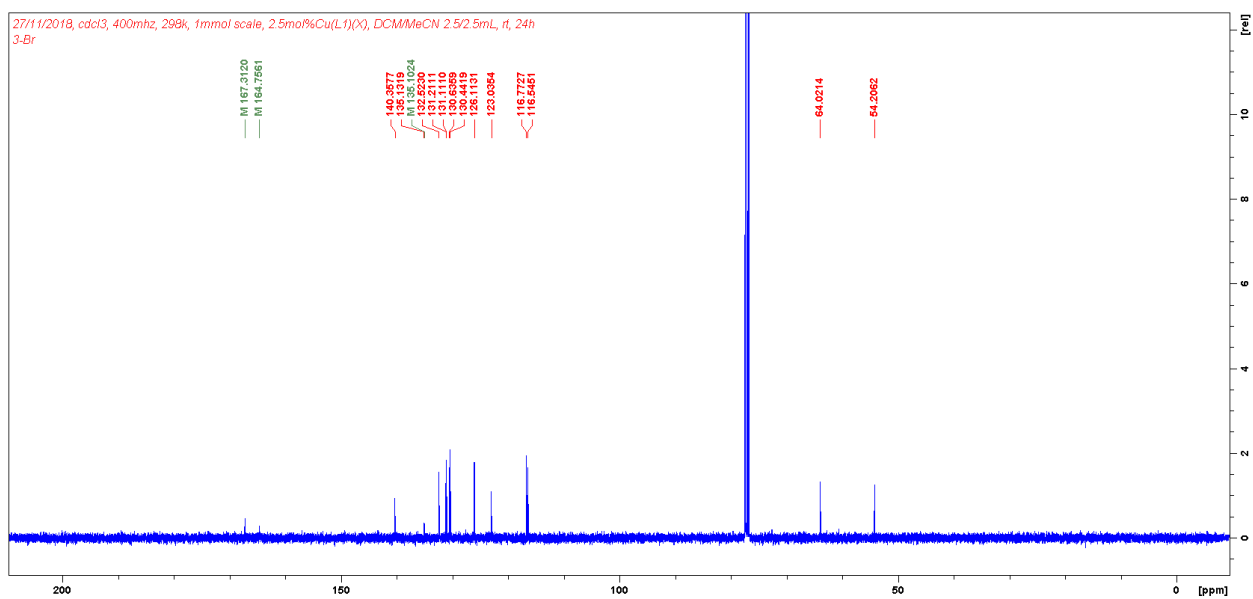
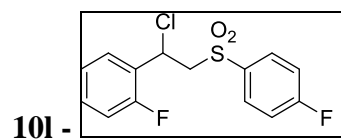


Figure S89. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10k**.



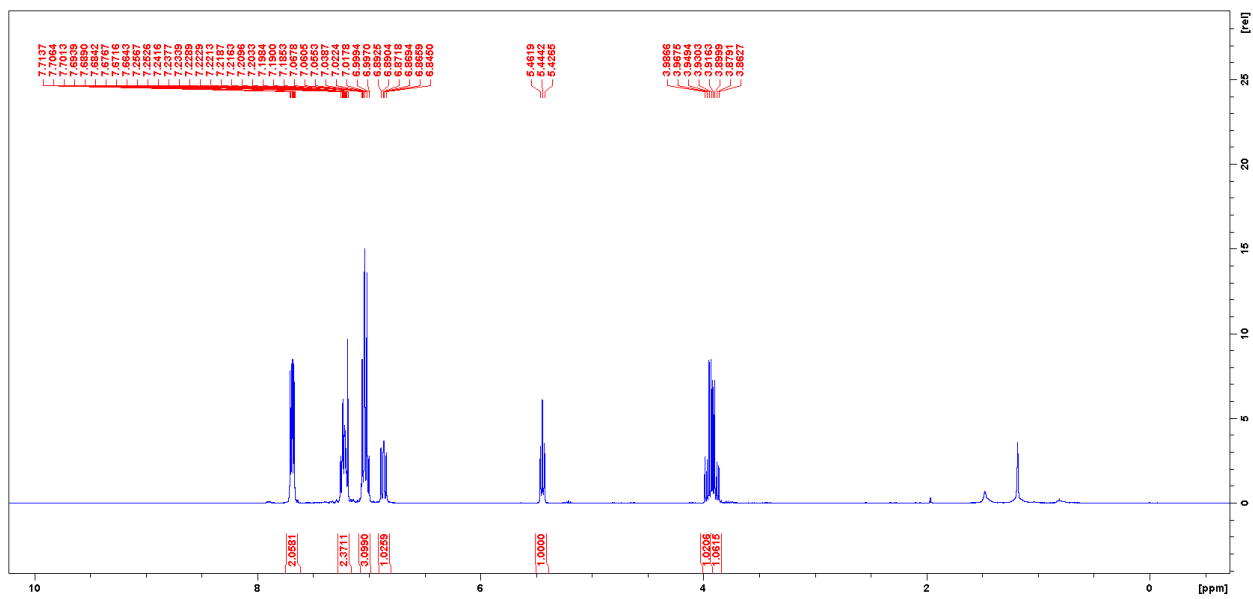


Figure S90. ^1H NMR spectrum of **101**.

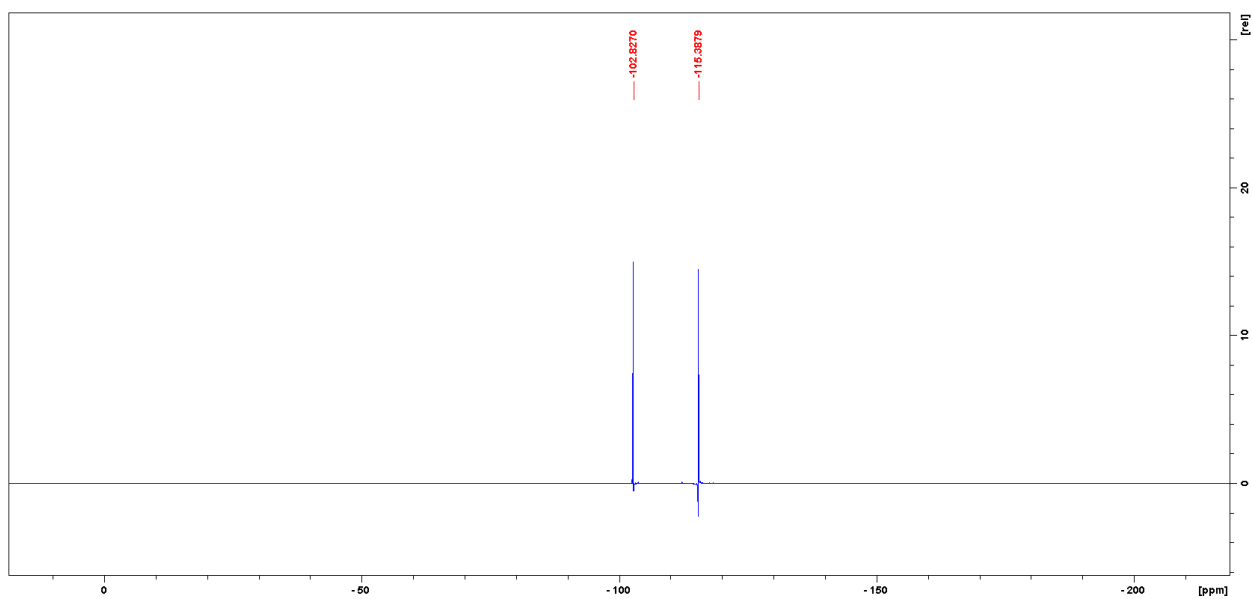


Figure S91. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **101**.

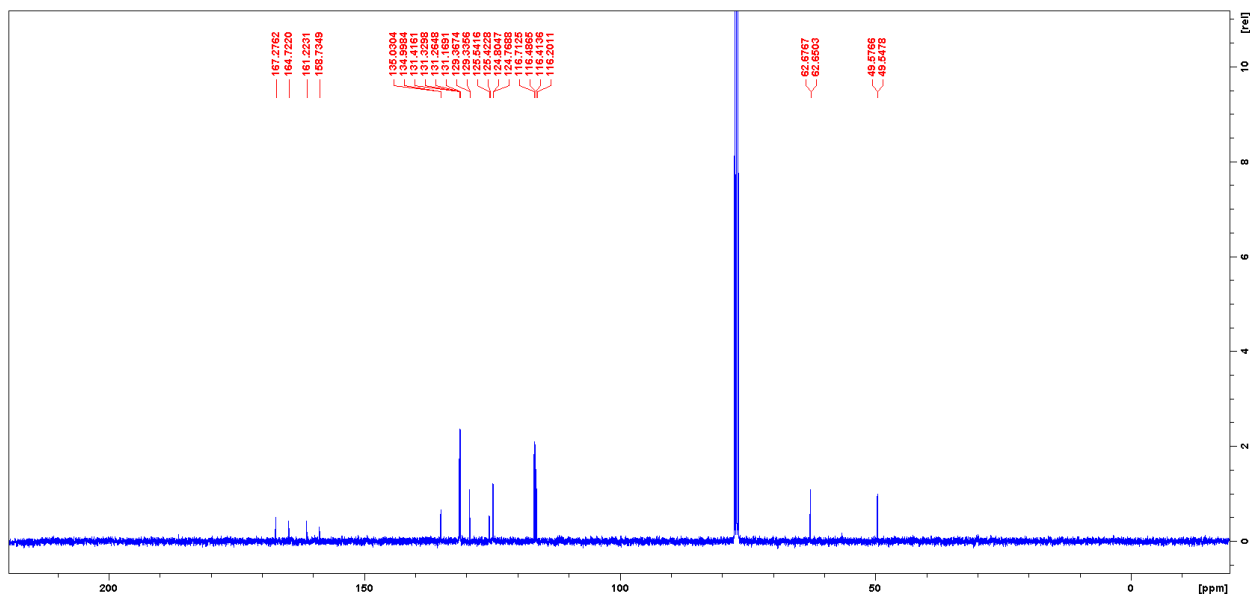


Figure S92. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10l**.

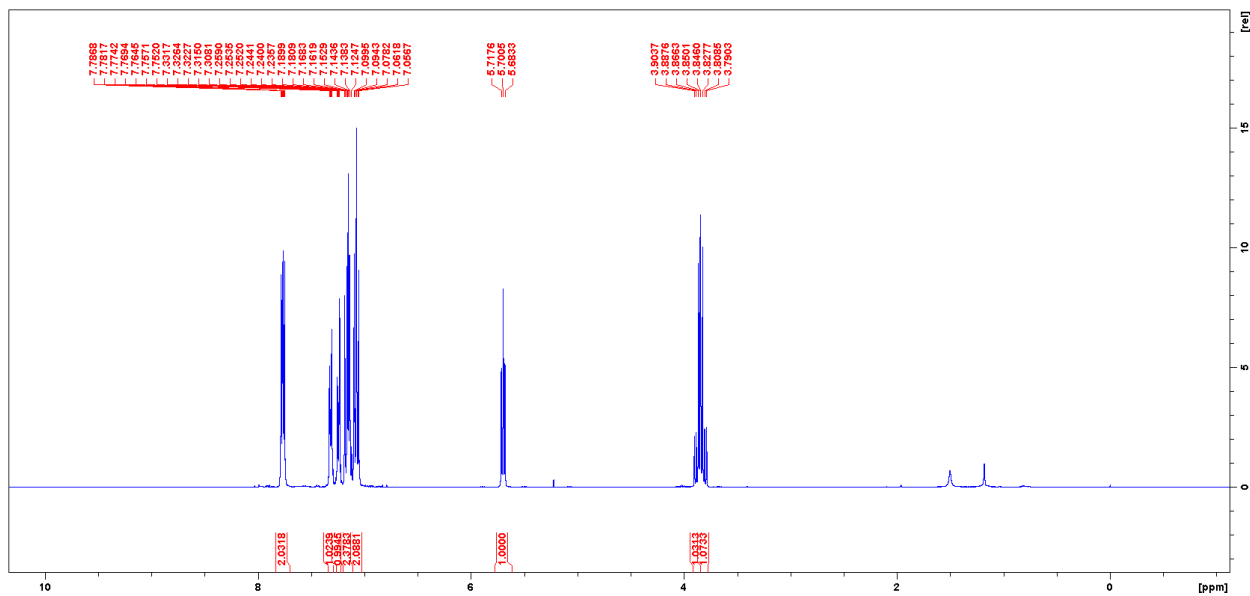
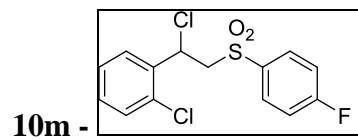


Figure S93. ^1H NMR spectrum of **10m**.

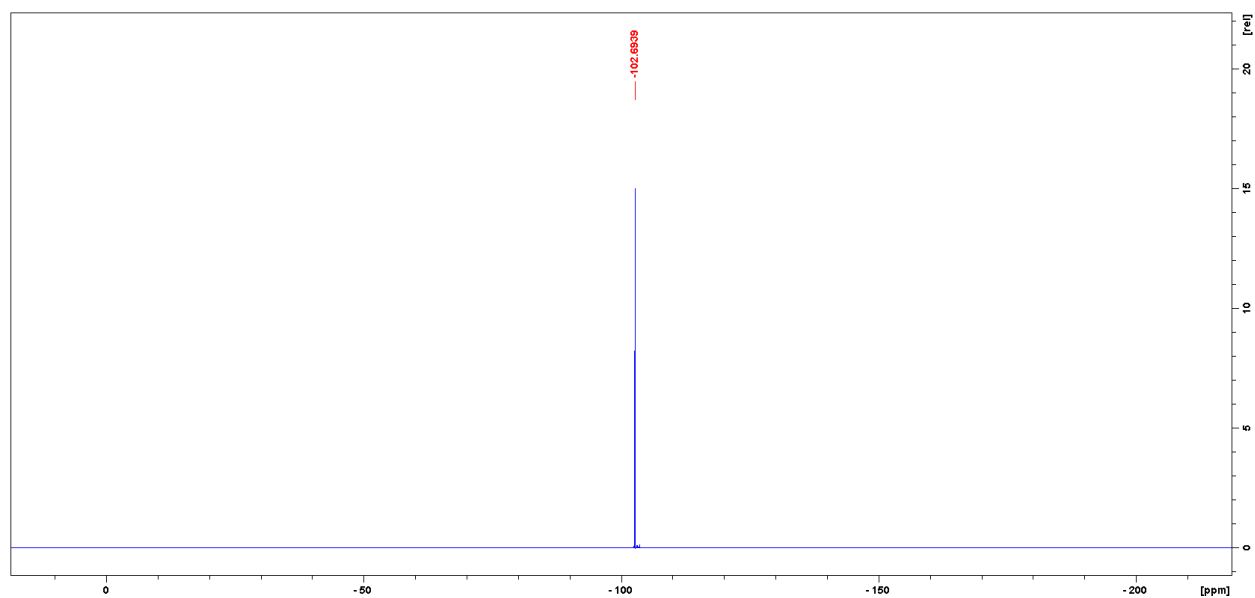


Figure S94. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10m**.

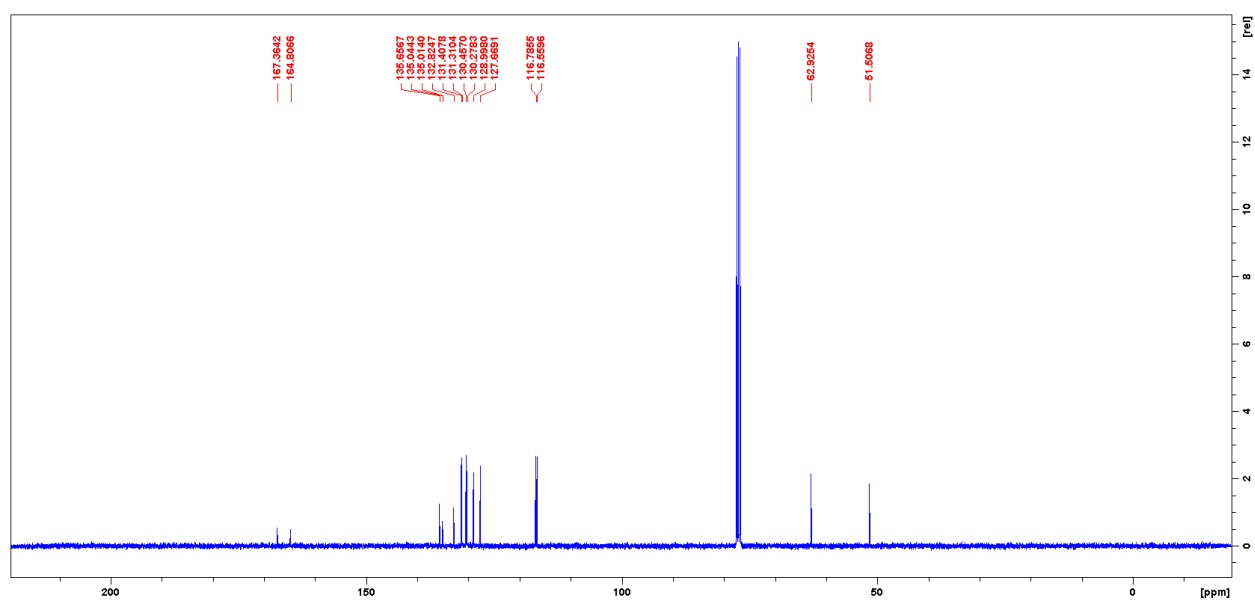
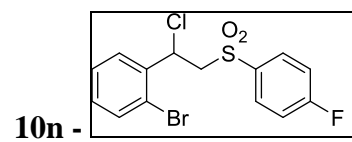


Figure S95. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10m**.



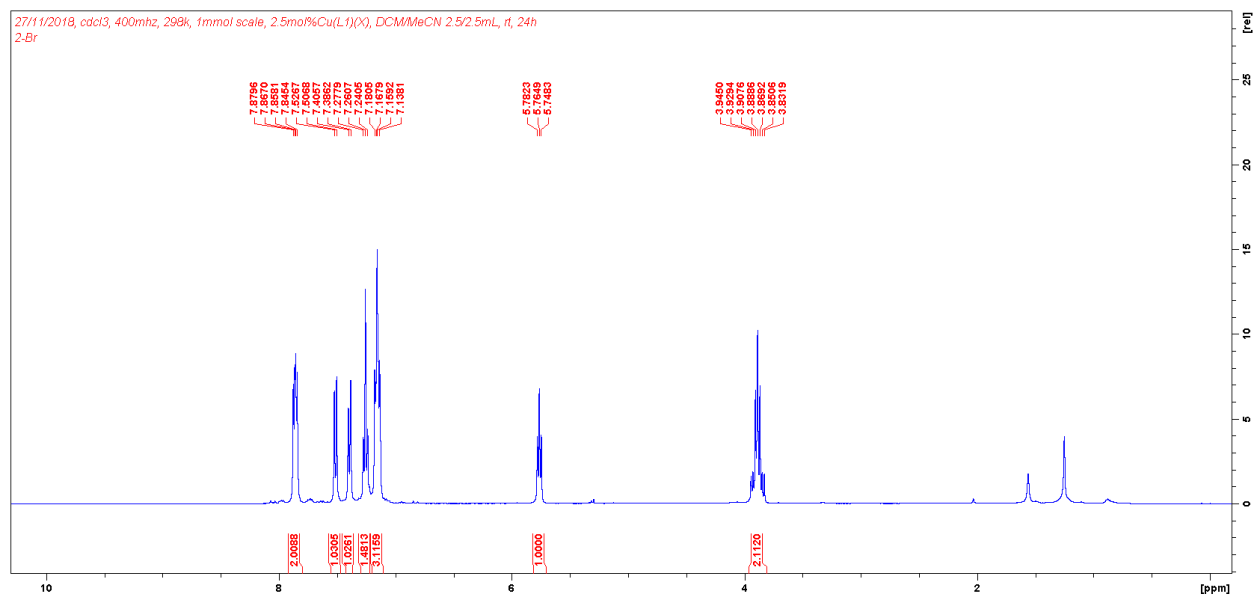


Figure S96. ^1H NMR spectrum of **10n**.

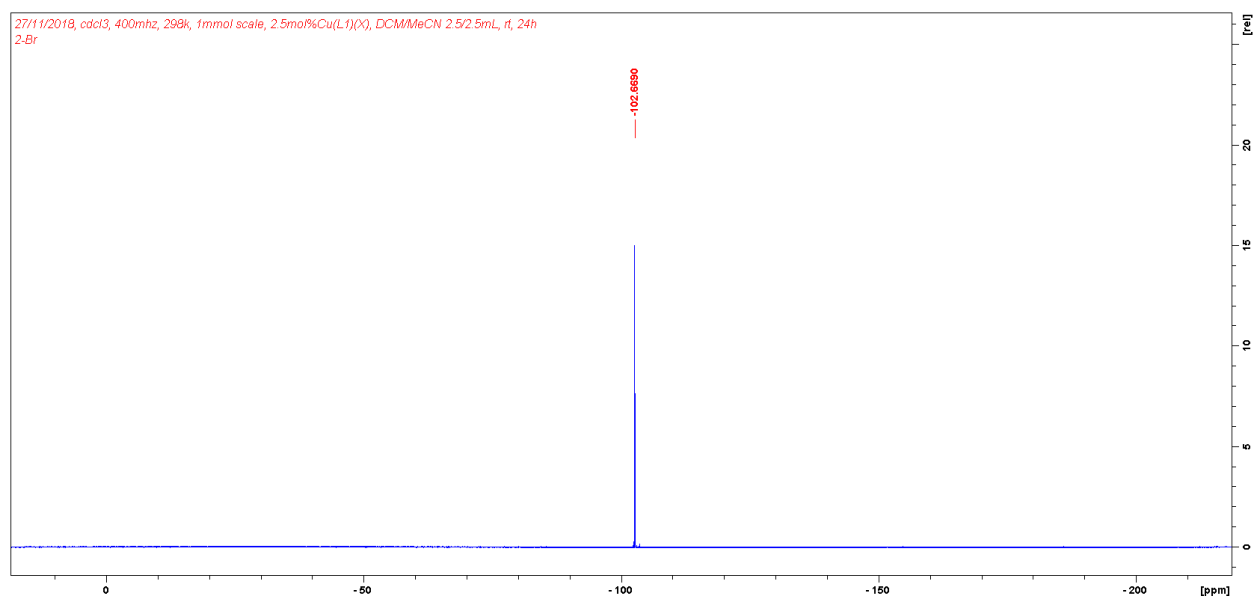


Figure S97. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10n**.

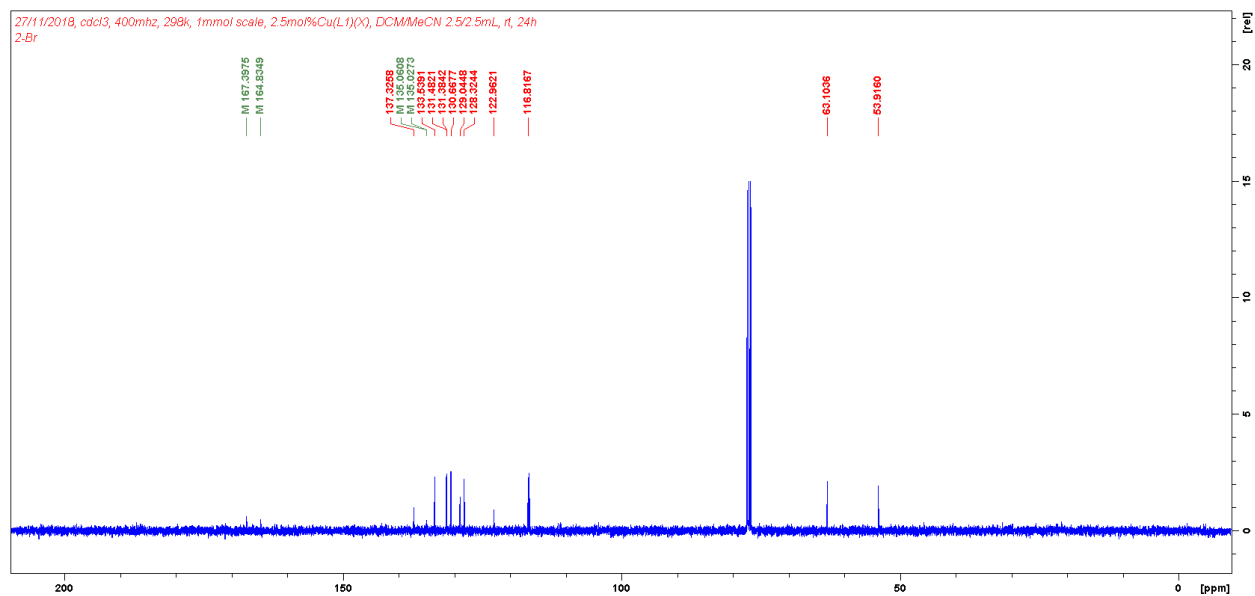


Figure S98. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10n**.

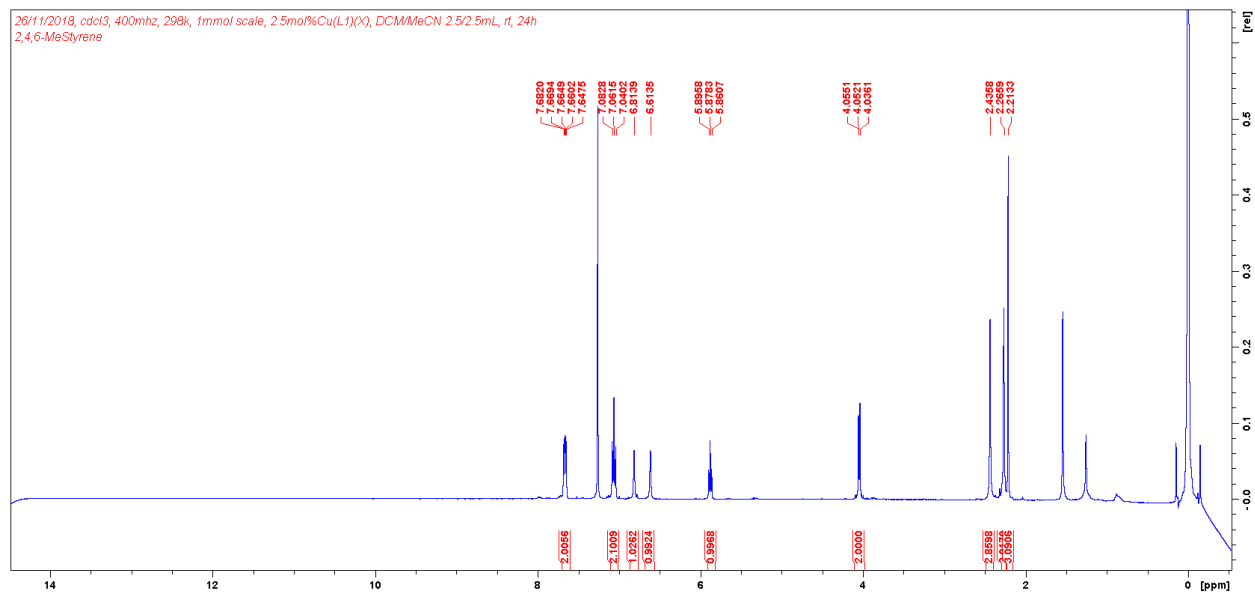
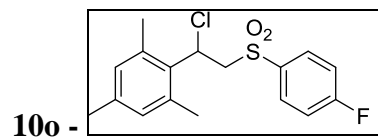


Figure S99. ^1H NMR spectrum of **10o**.

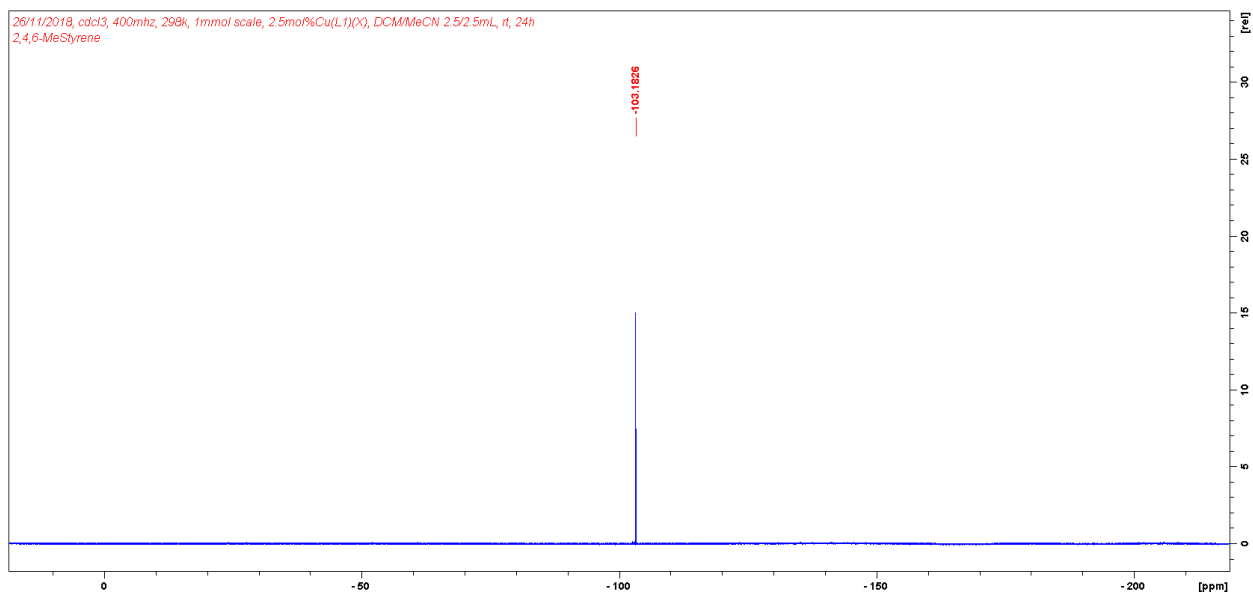


Figure S100. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10o**.

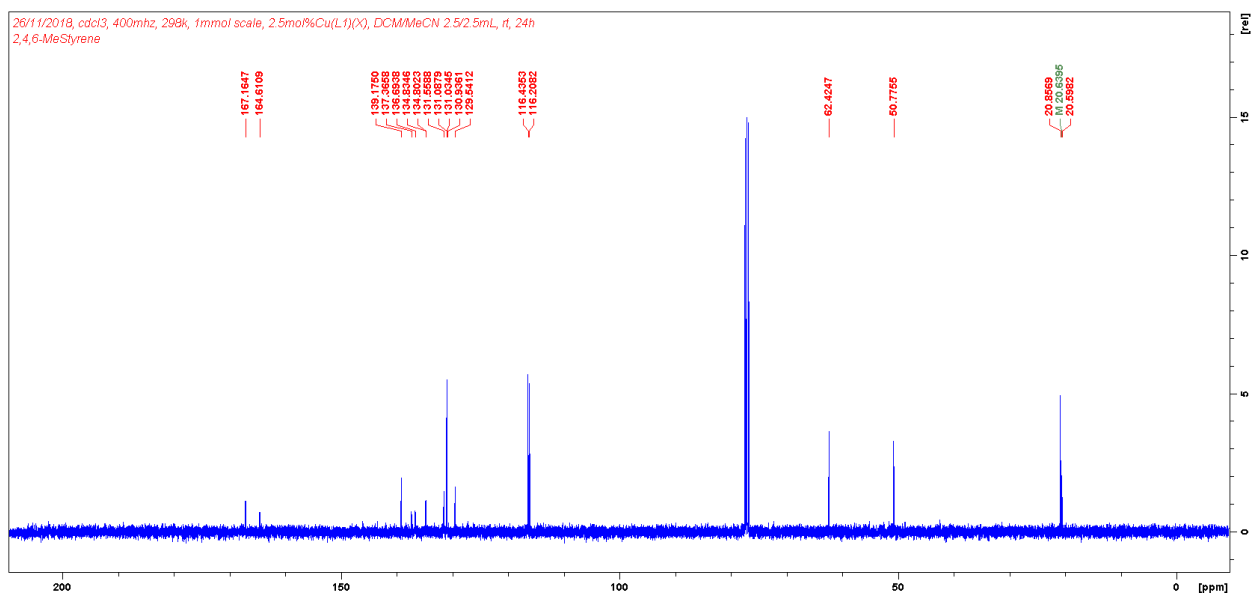
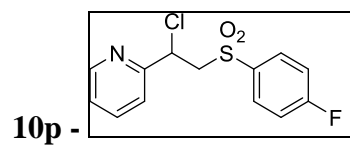


Figure S101. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10o**.



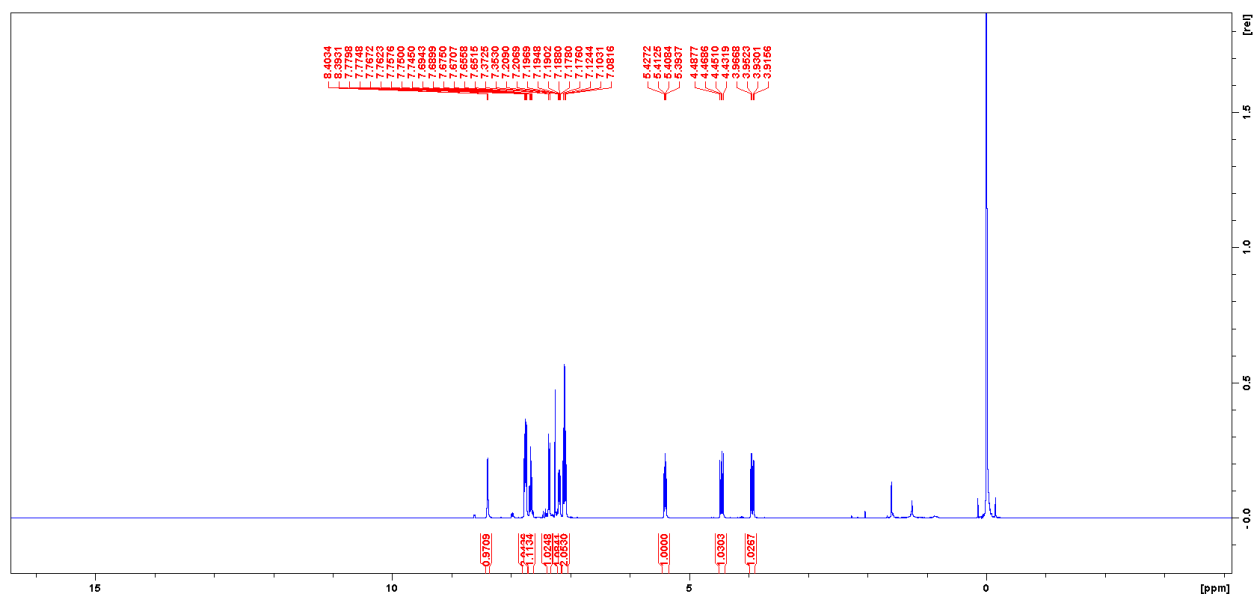


Figure S102. ^1H NMR spectrum of **10p**.

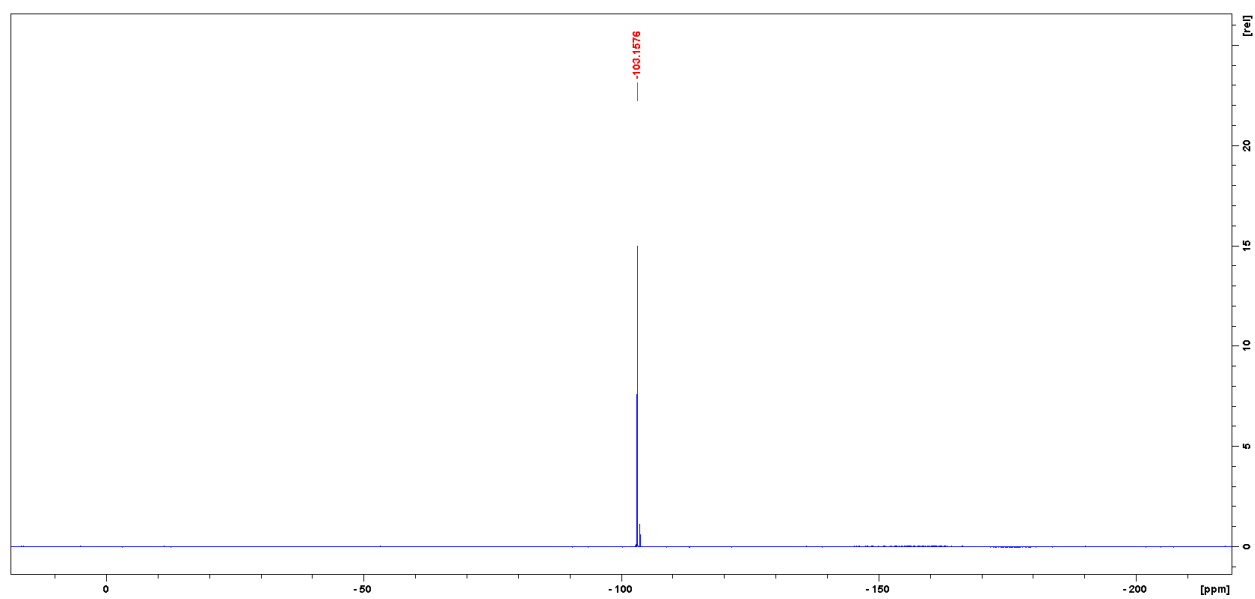


Figure S103. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10p**.

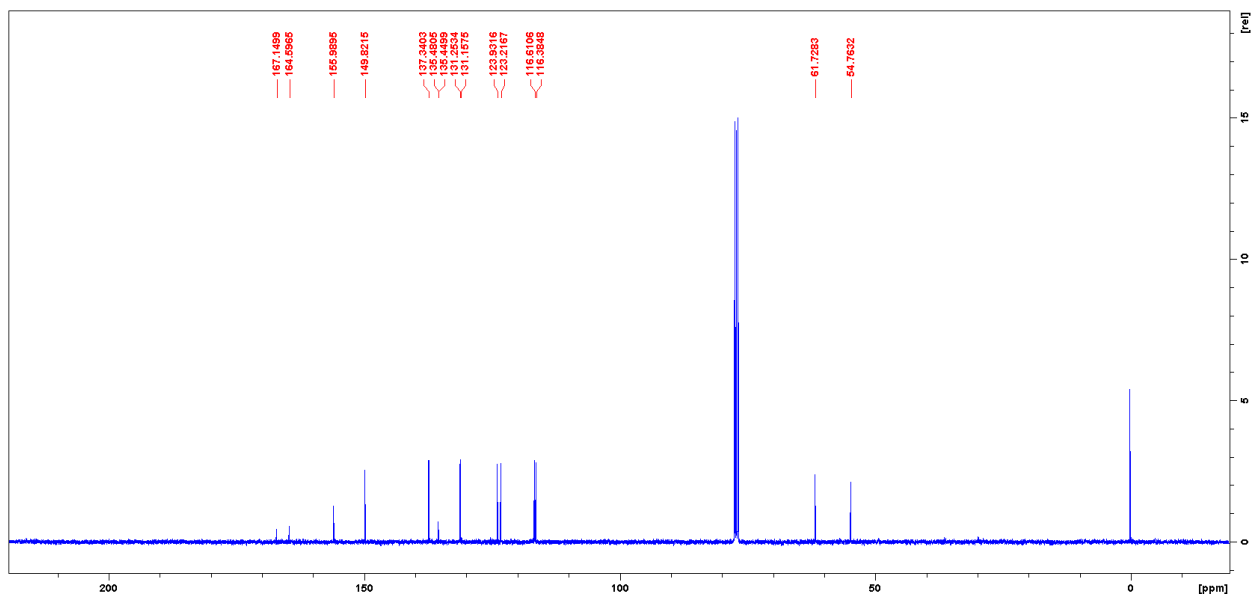


Figure S104. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10p**.

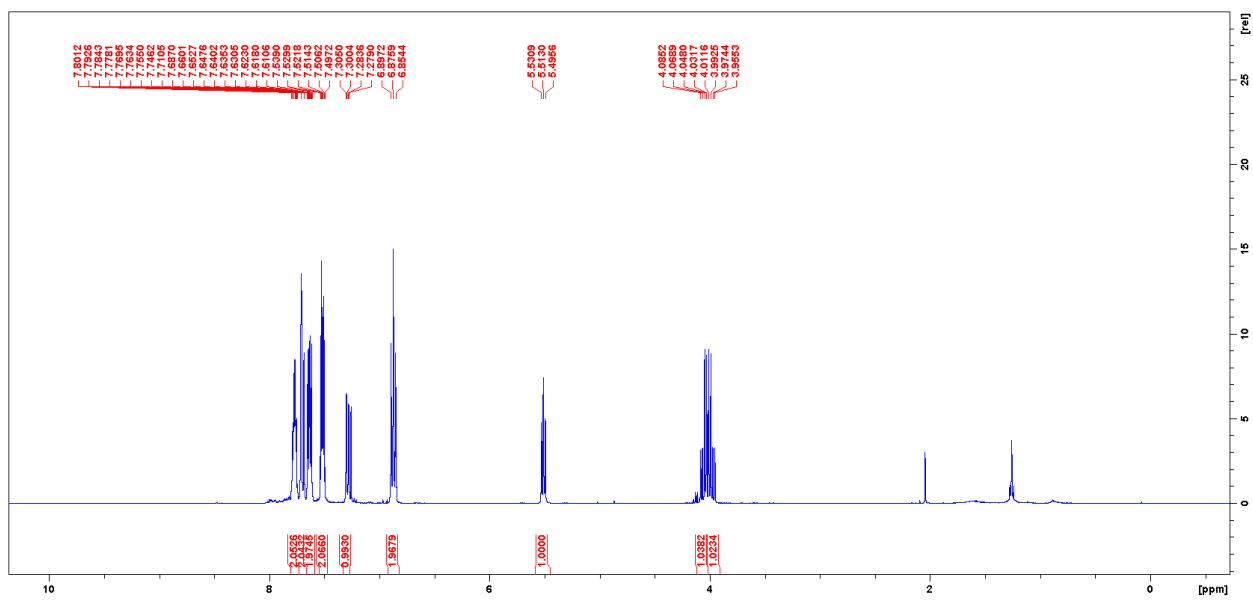
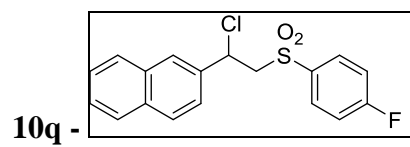


Figure S105. ^1H NMR spectrum of **10q**.

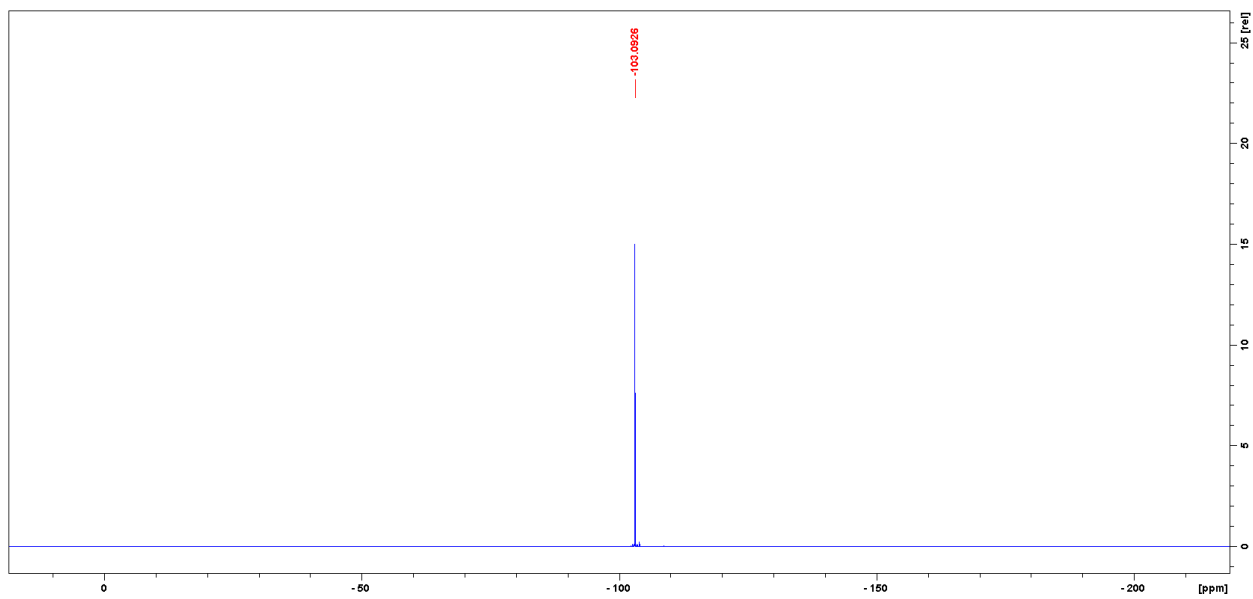


Figure S106. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10q**.

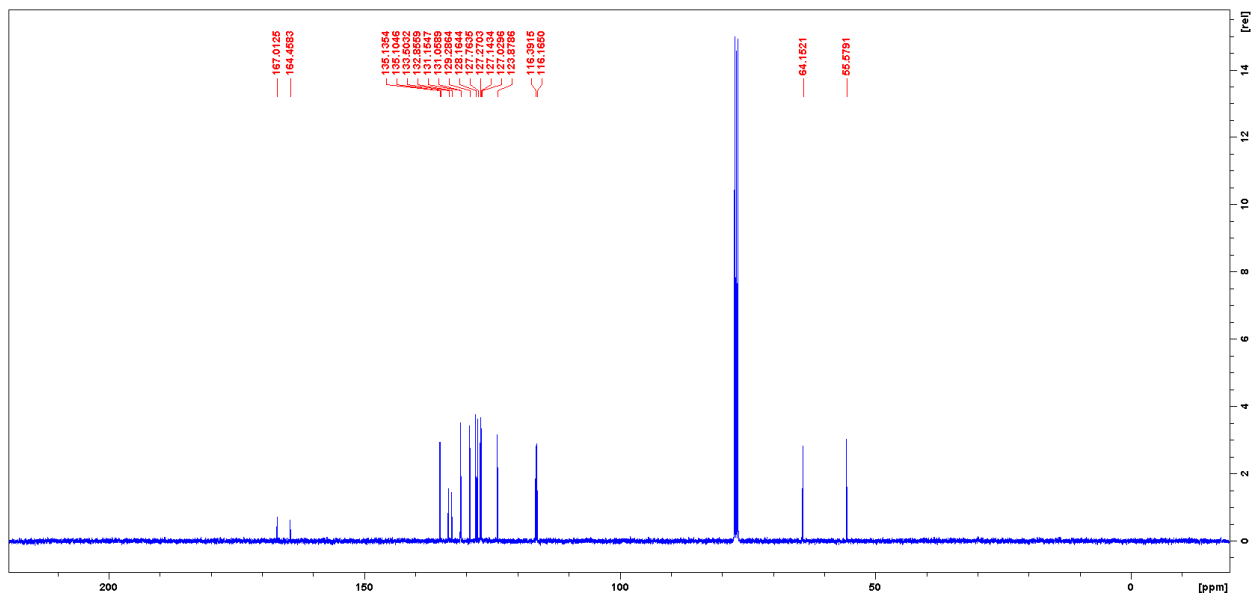
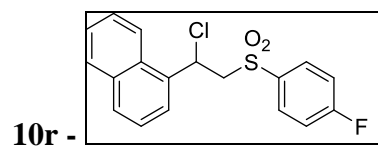


Figure S107. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10q**.



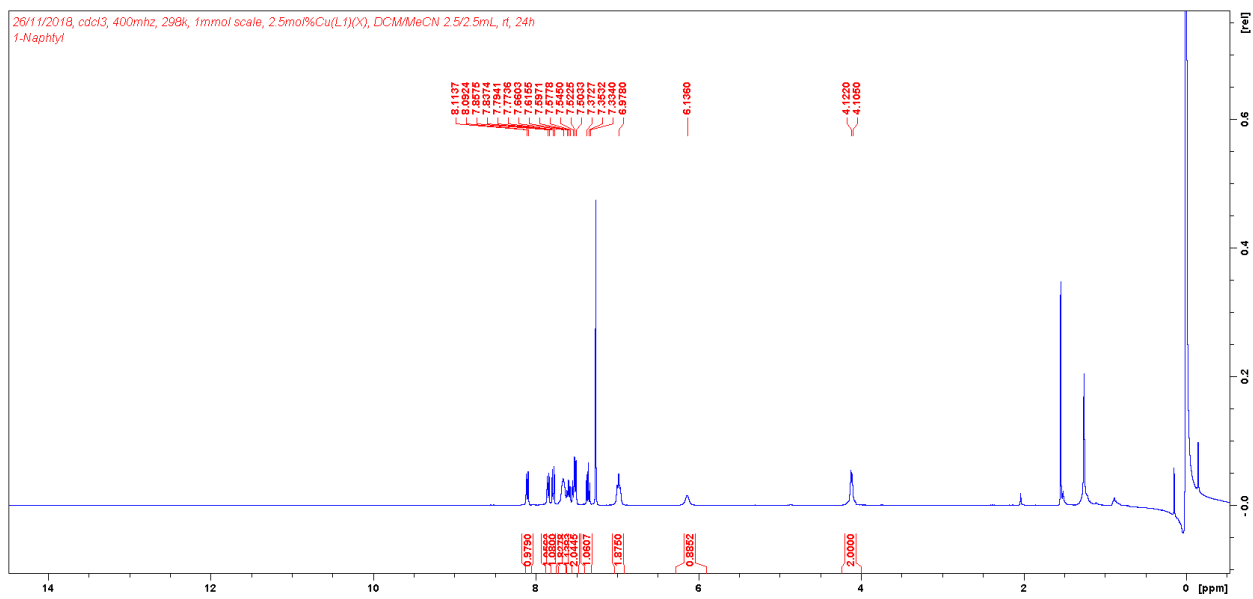


Figure S108. ^1H NMR spectrum of **10r**.

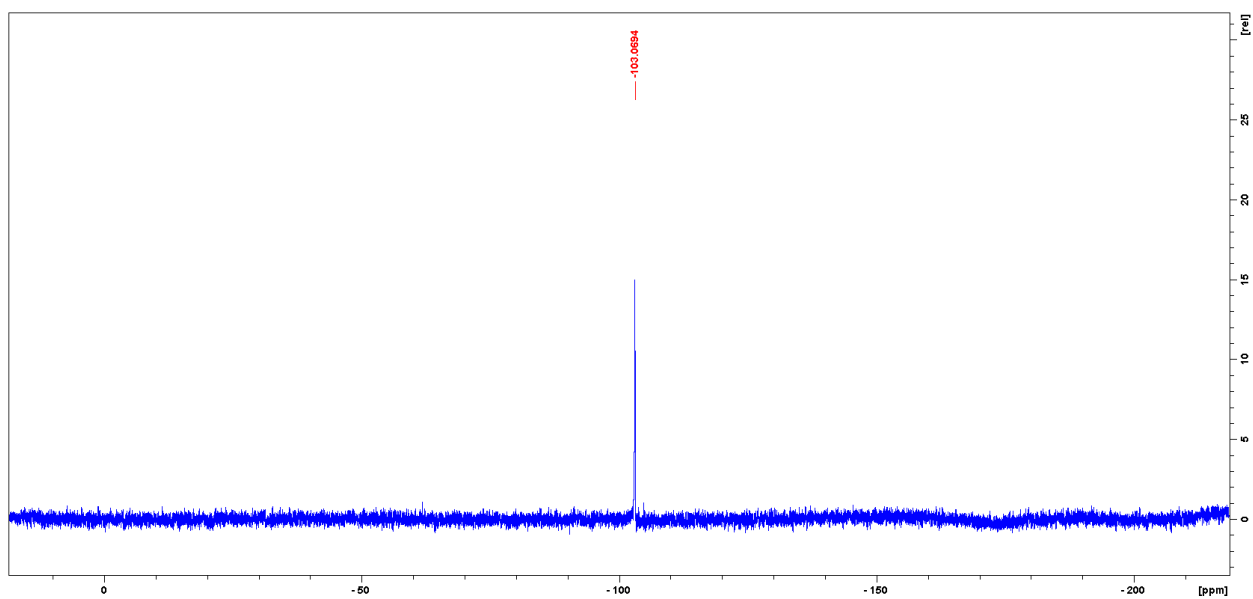


Figure S109. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10r**.

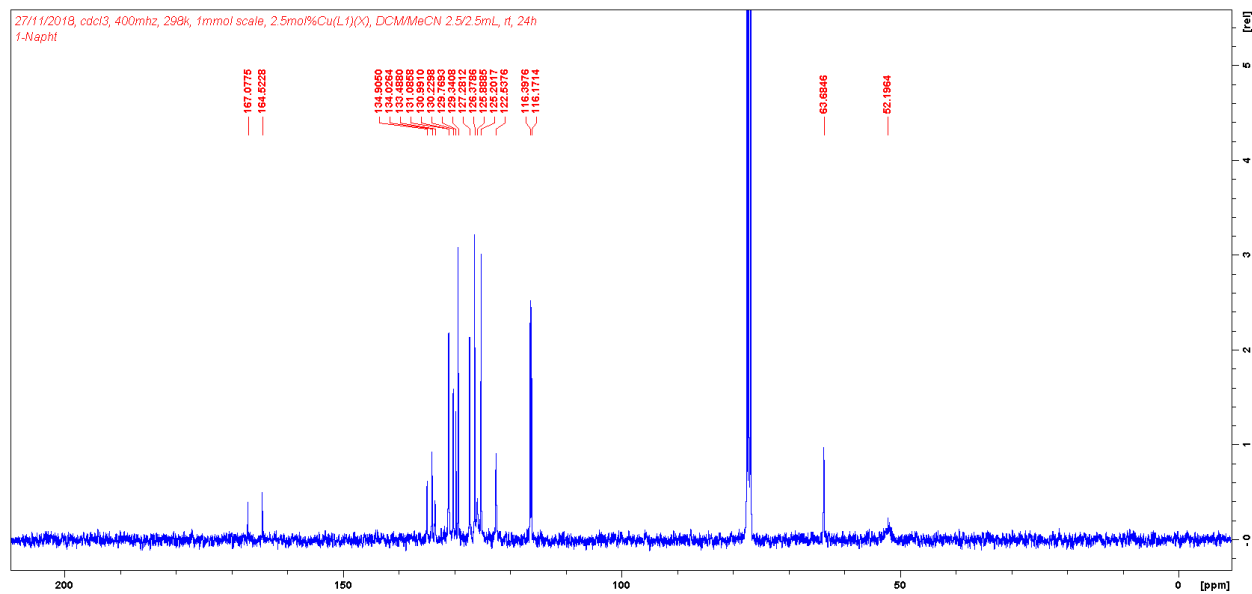


Figure S110. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10r**.

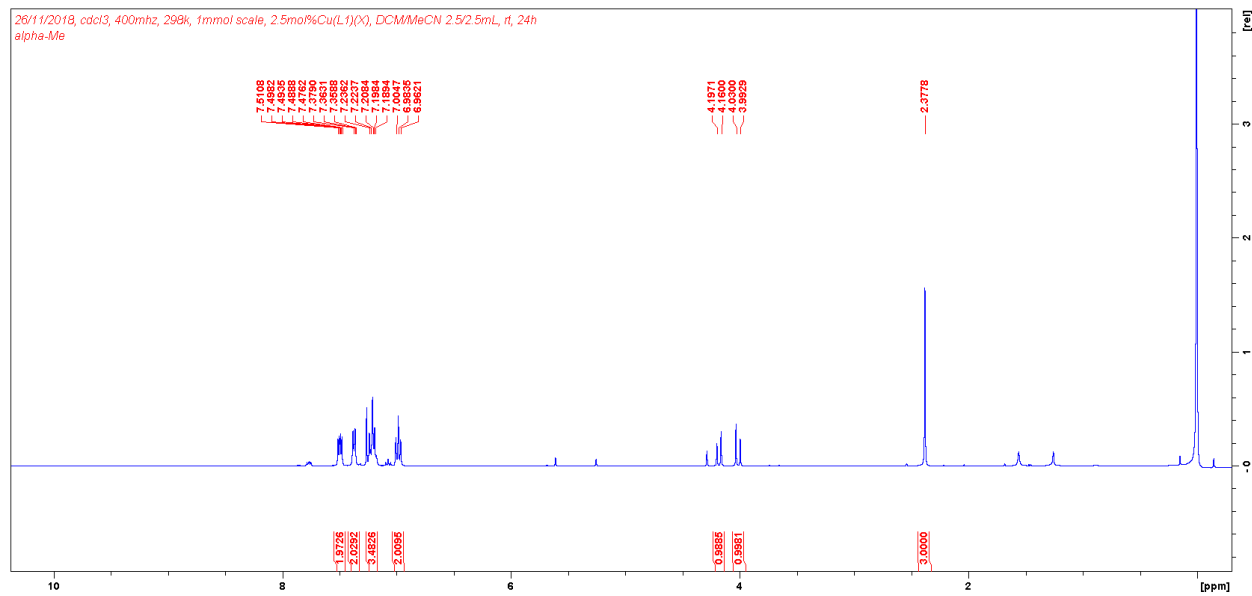
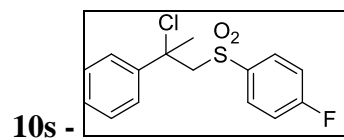


Figure S111. ^1H NMR spectrum of **10s**.

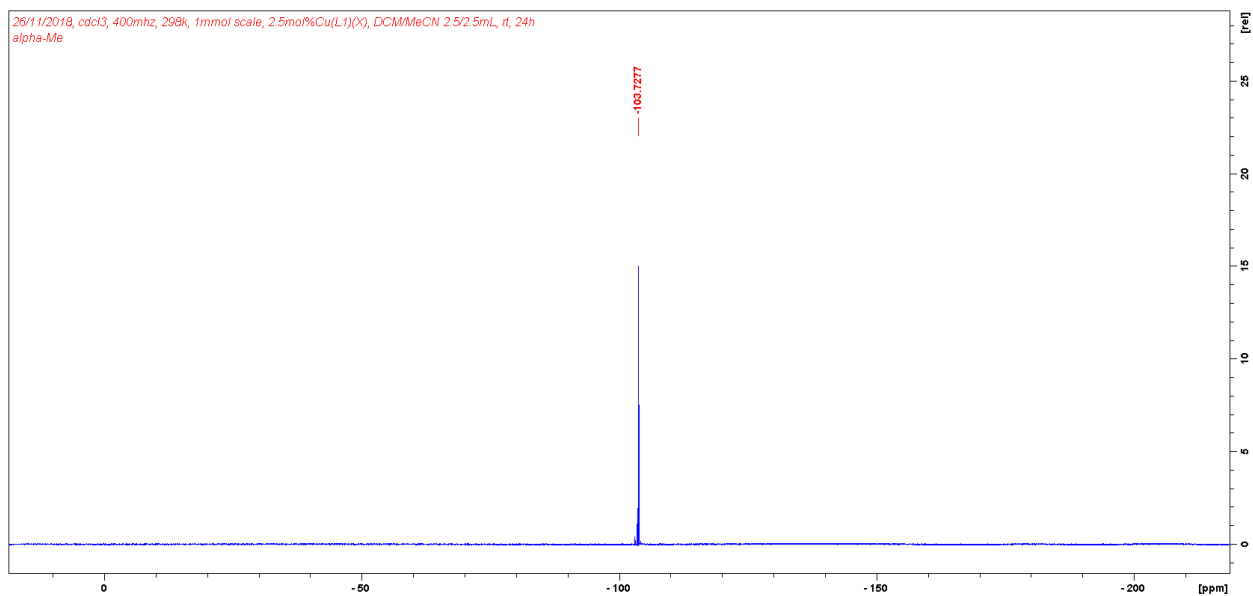


Figure S112. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10s**.

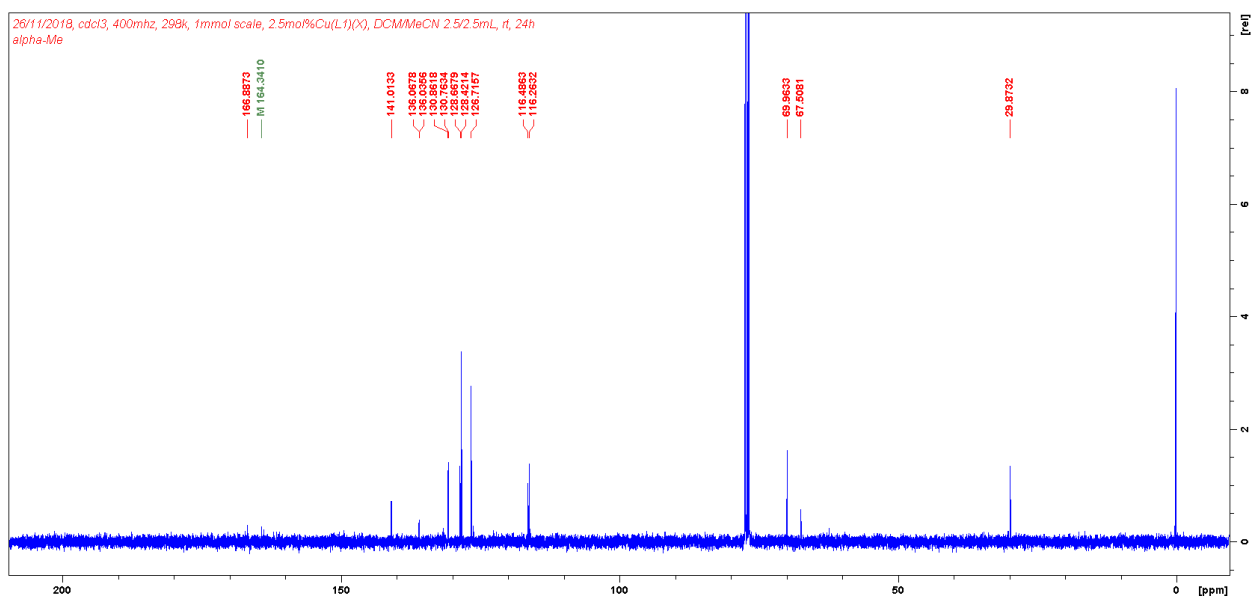
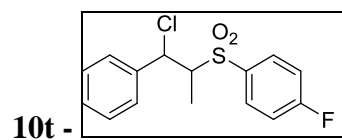


Figure S113. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10s**.



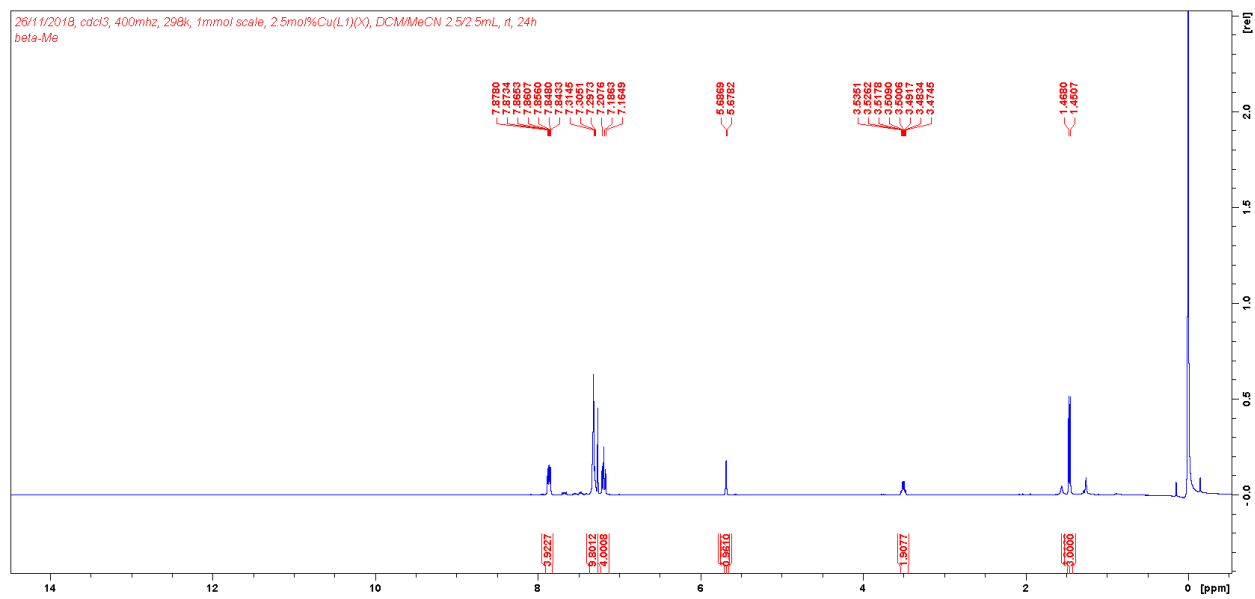


Figure S114. ^1H NMR spectrum of **10t**.

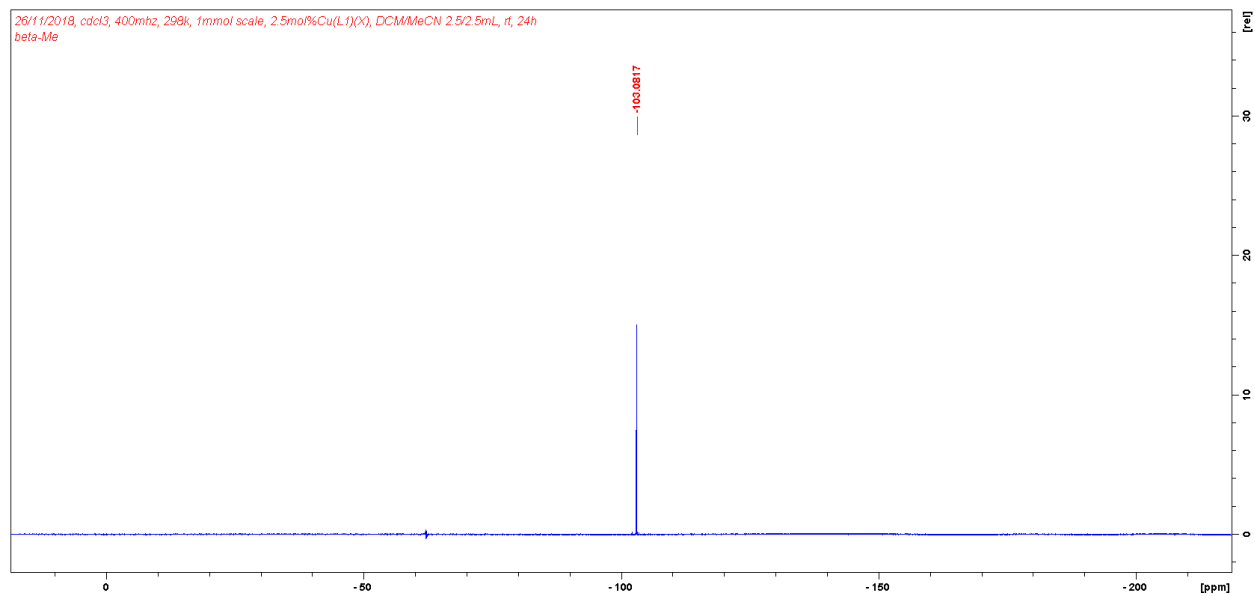


Figure S115. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10t**.

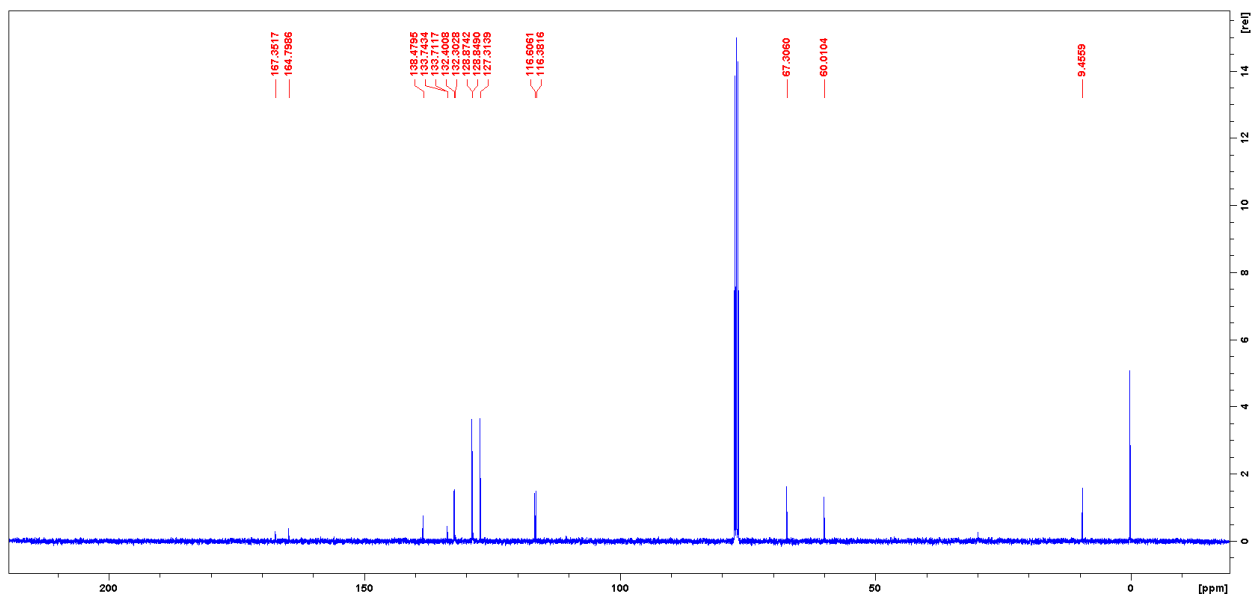


Figure S116. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10t**.

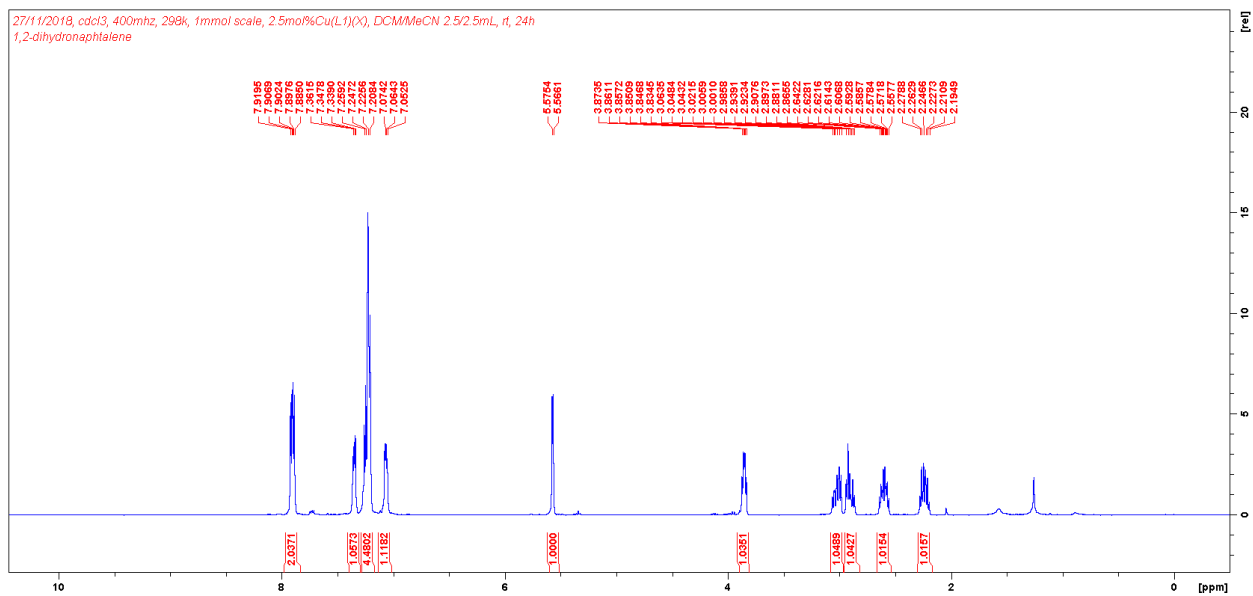
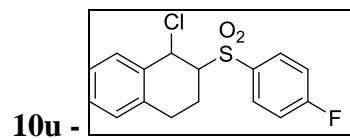


Figure S117. ^1H NMR spectrum of **10u**.

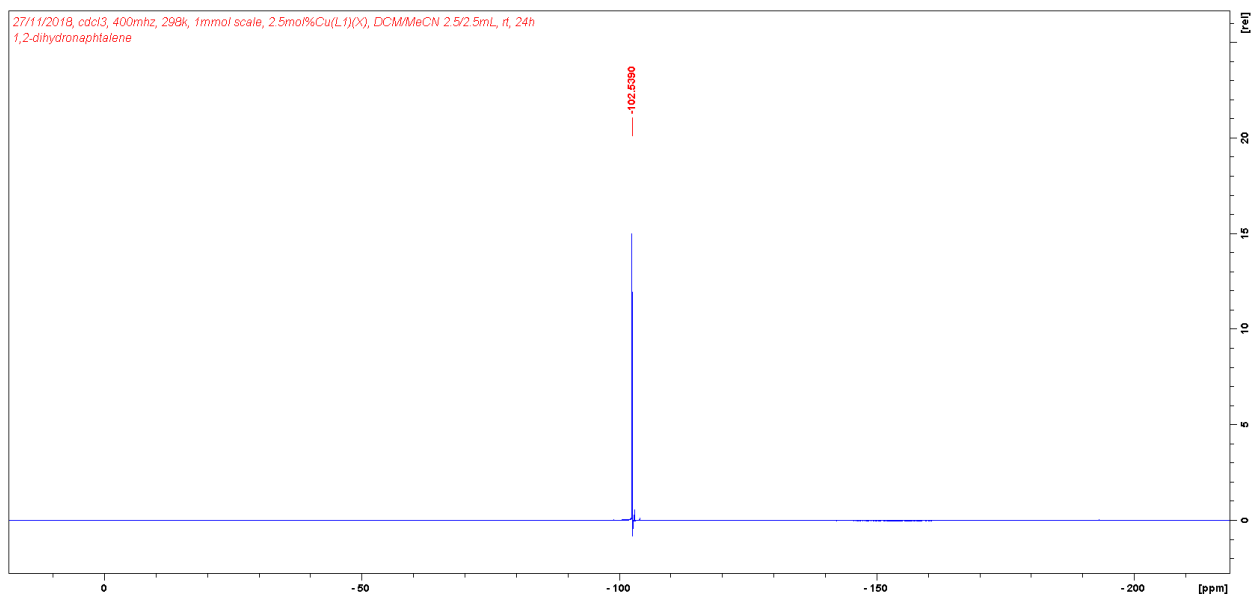


Figure S118. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10u**.

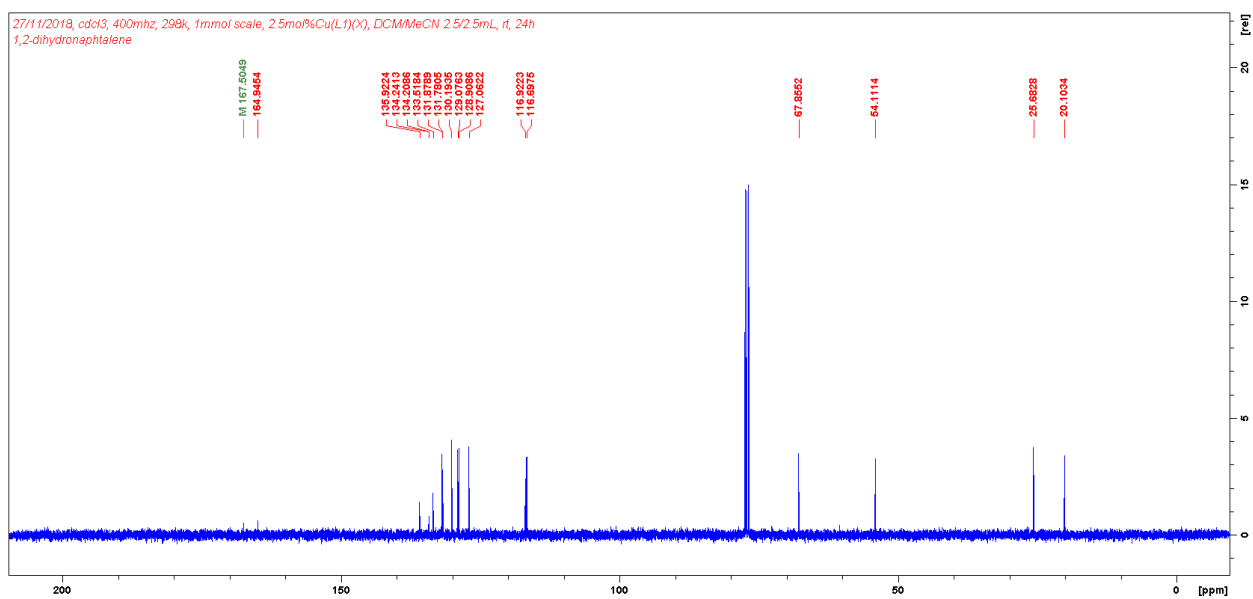
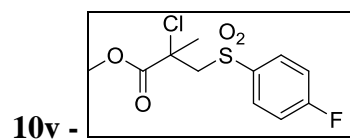


Figure S119. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10u**.



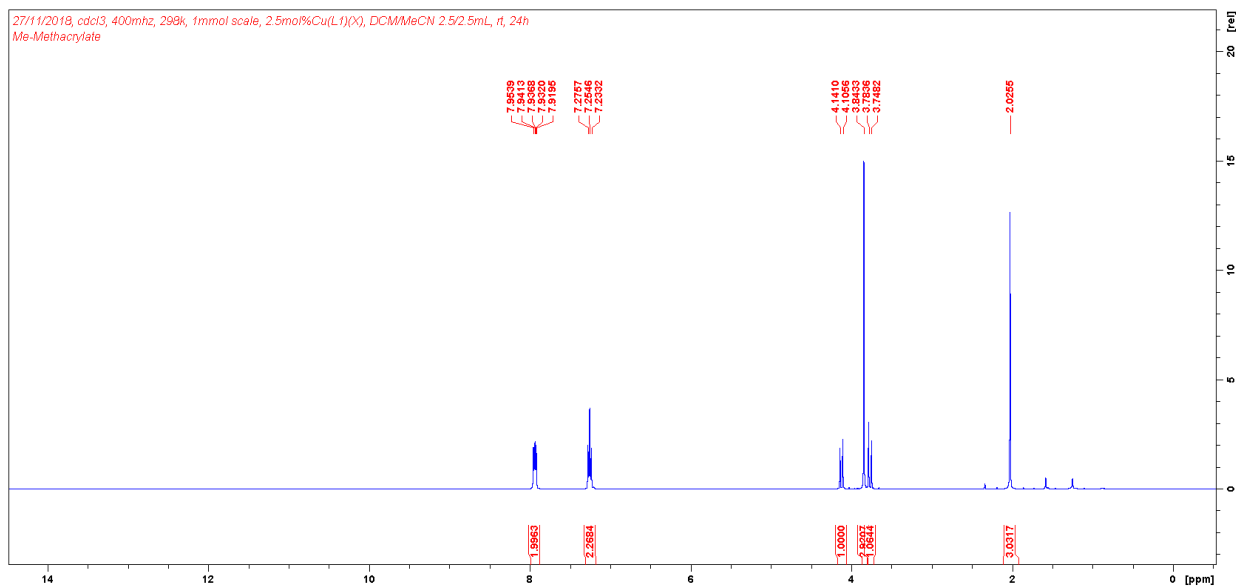


Figure S120. ^1H NMR spectrum of **10v**.

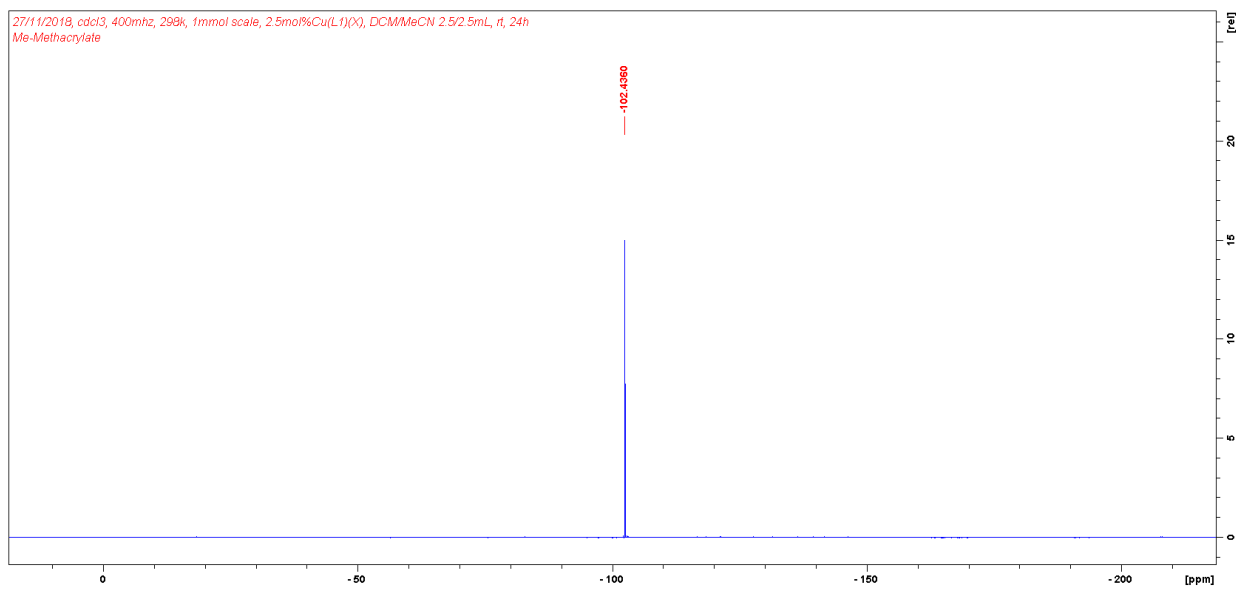


Figure S121. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10v**.

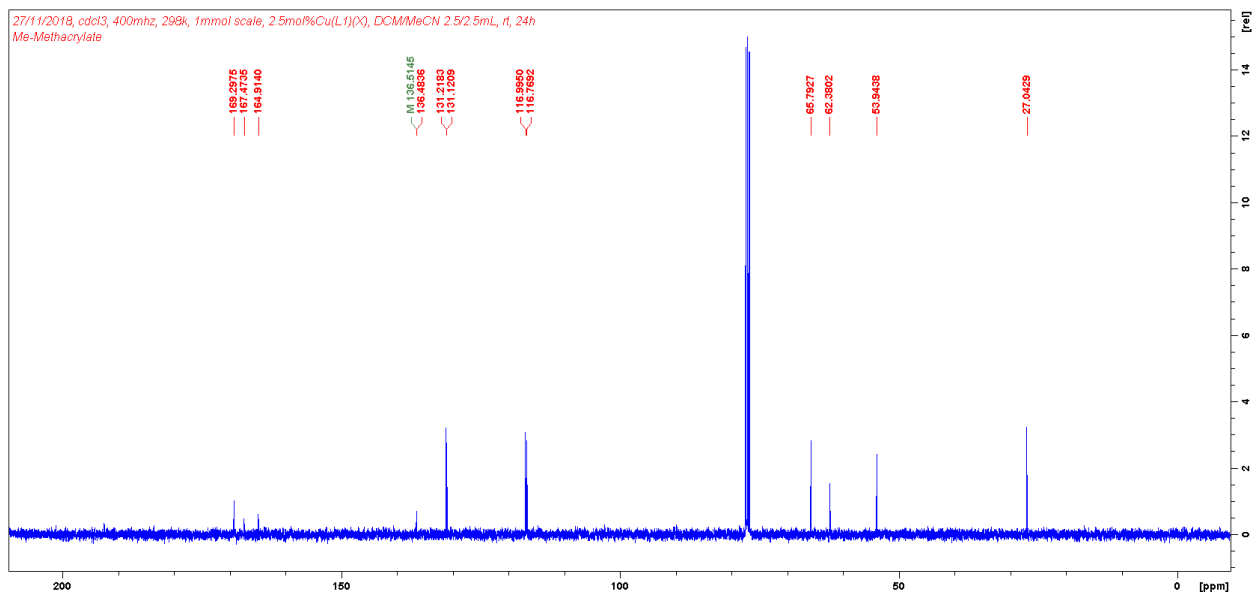


Figure S122. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10v**.

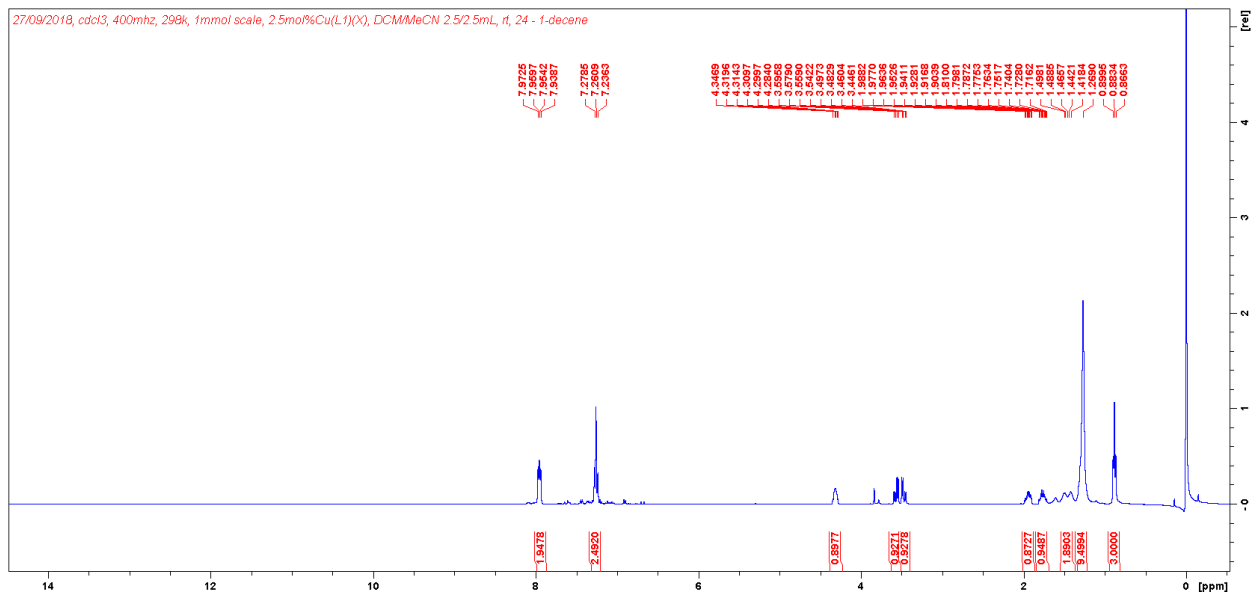
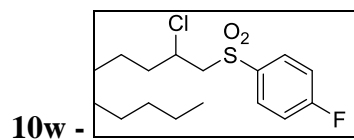


Figure S123. ^1H NMR spectrum of **10w**.

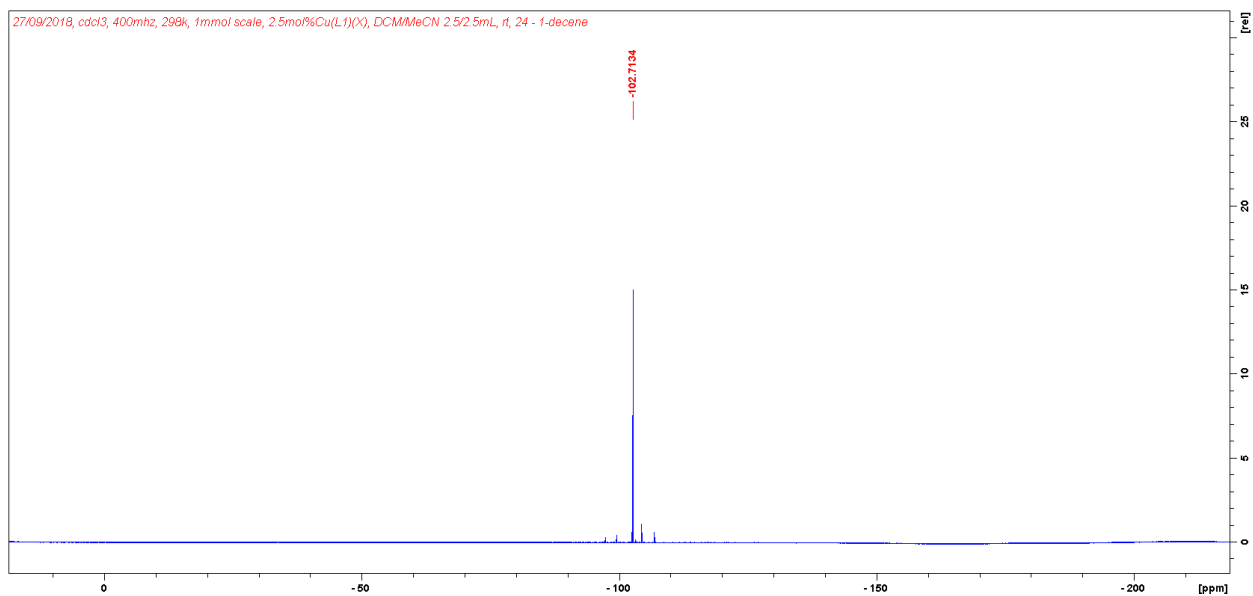


Figure S124. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10w**.

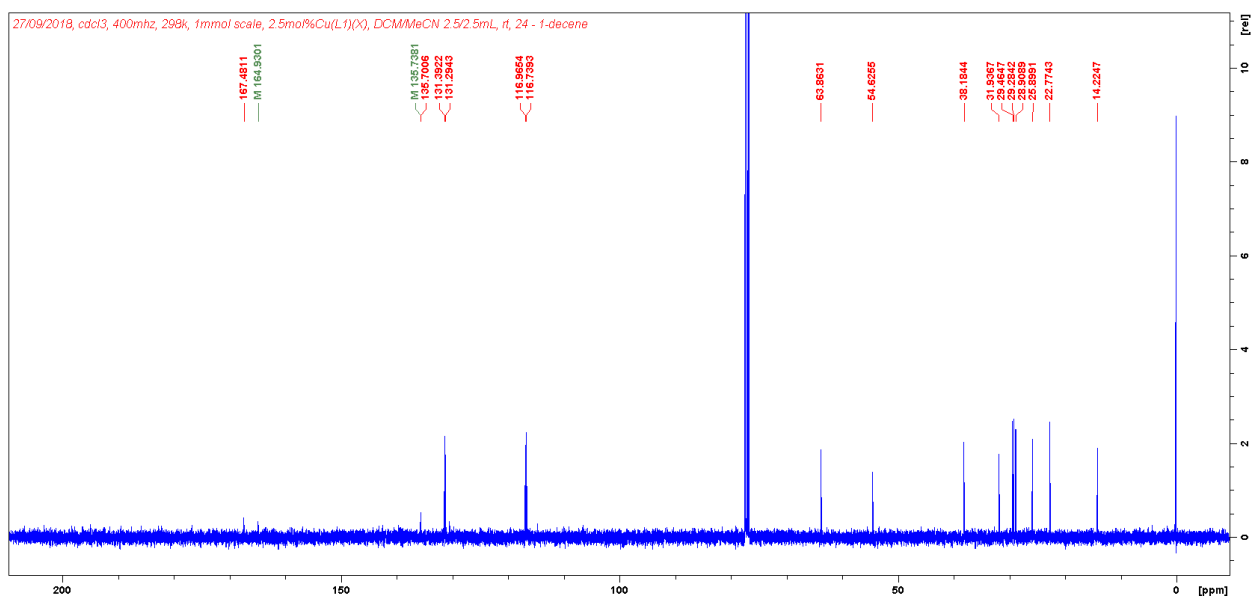
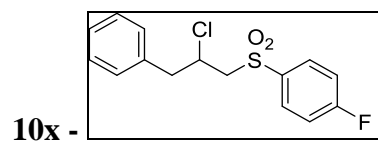


Figure S125. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10w**.



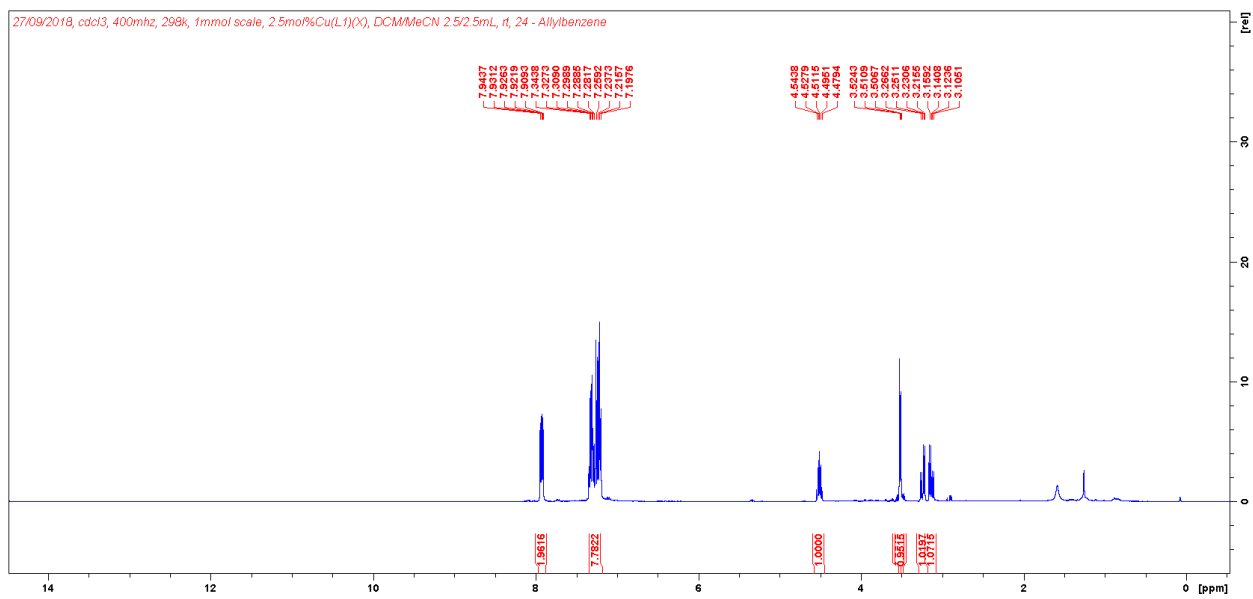


Figure S126. ^1H NMR spectrum of **10x**.

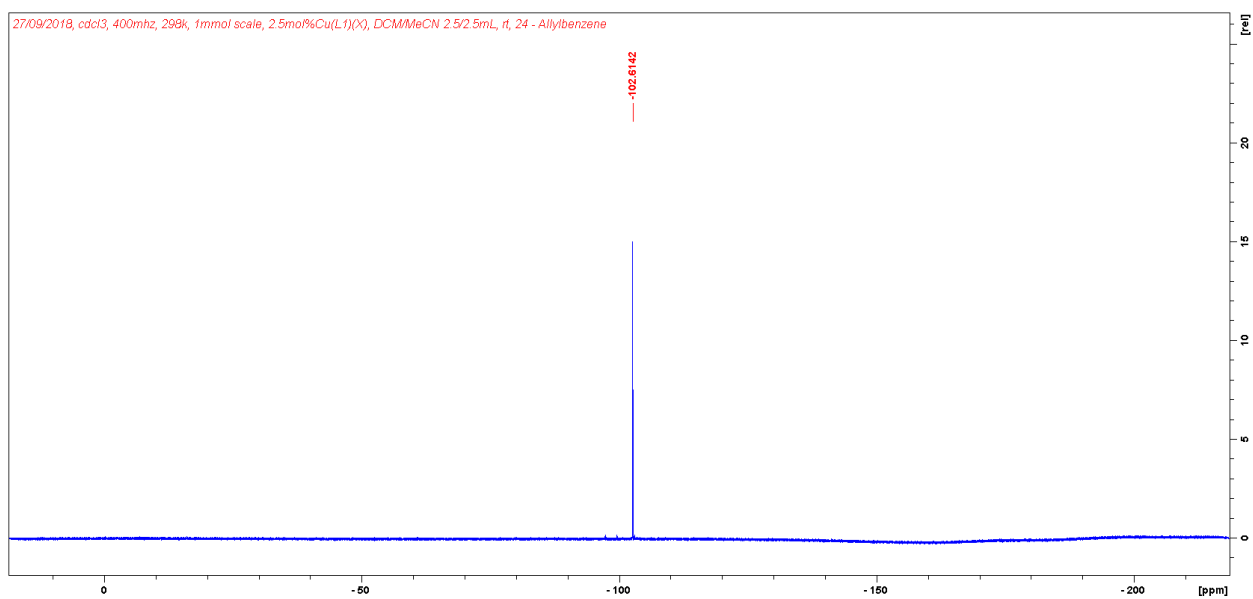


Figure S127. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10x**.

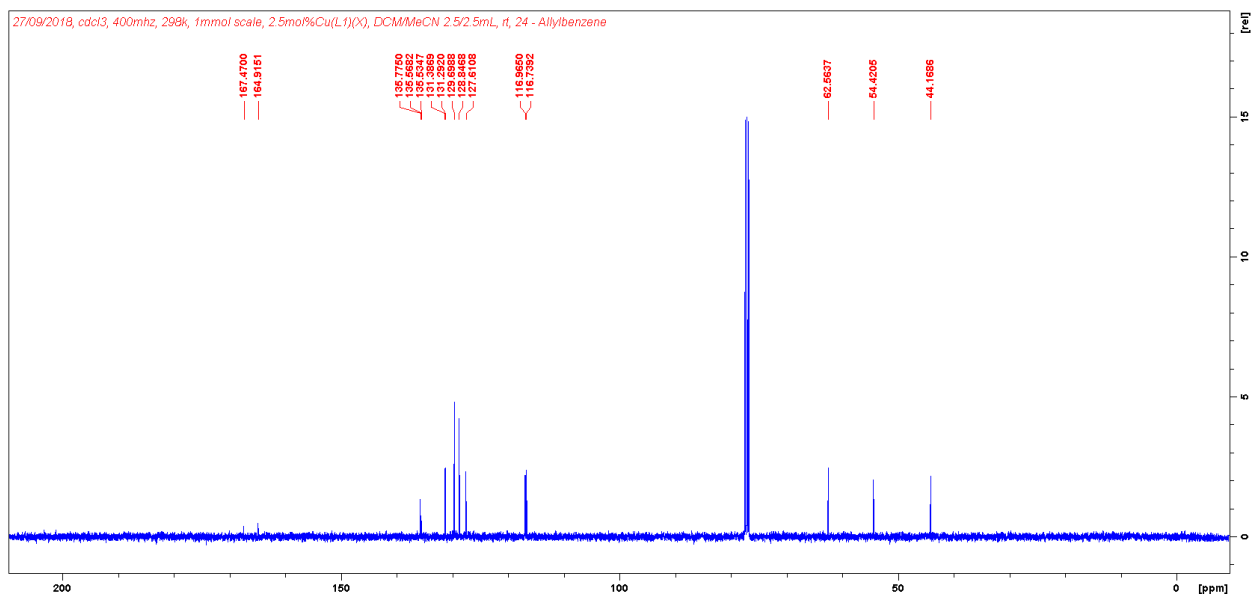


Figure S128. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10x**.

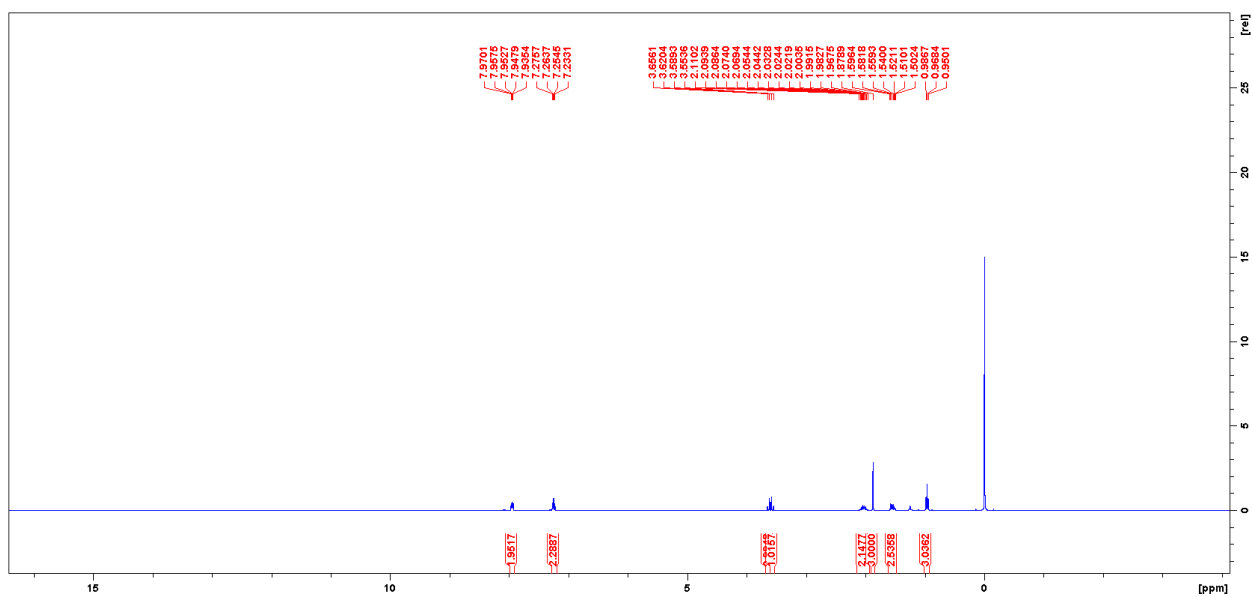
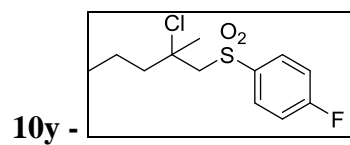


Figure S129. ^1H NMR spectrum of **10y**.

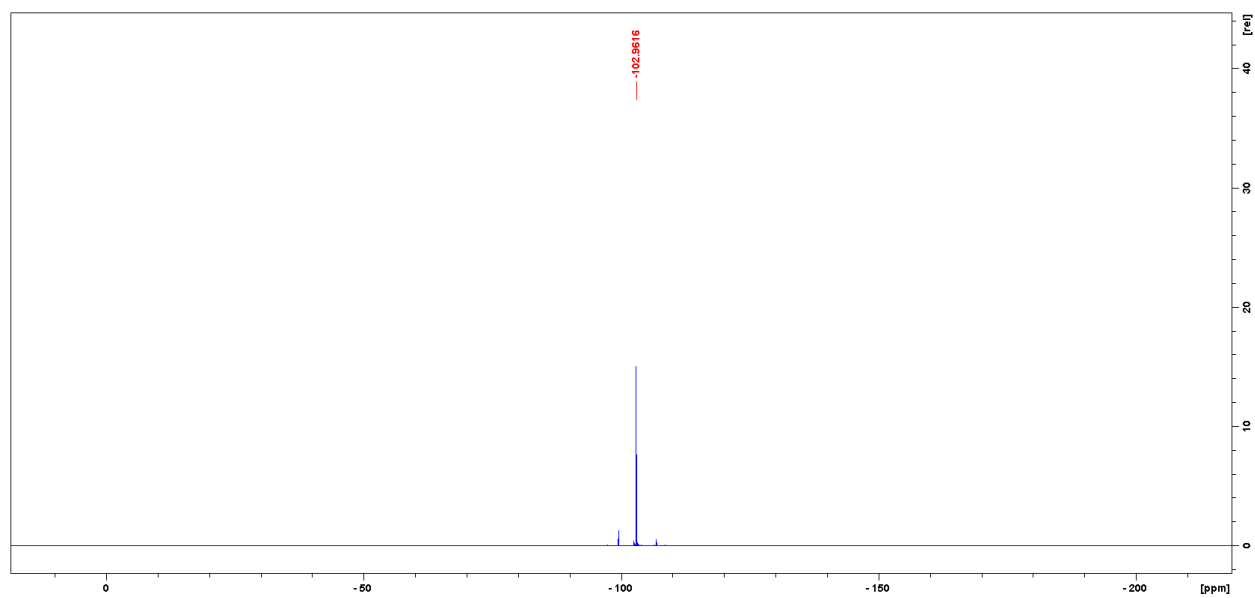


Figure S130. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10y**.

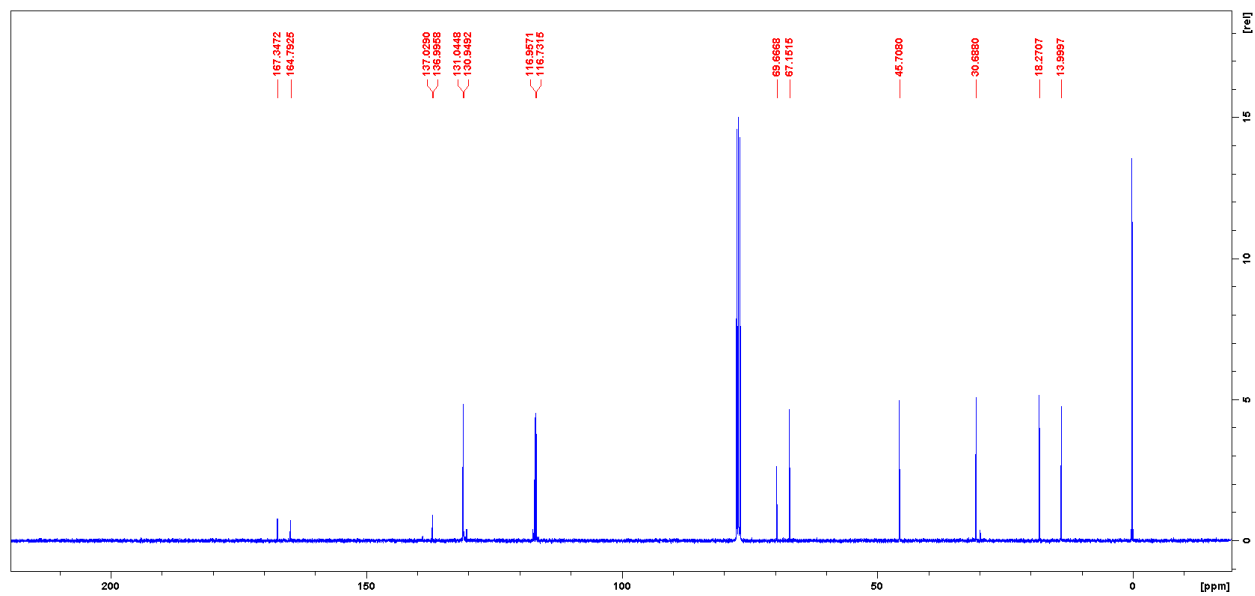
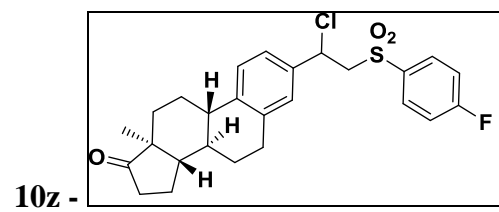


Figure S131. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10y**.



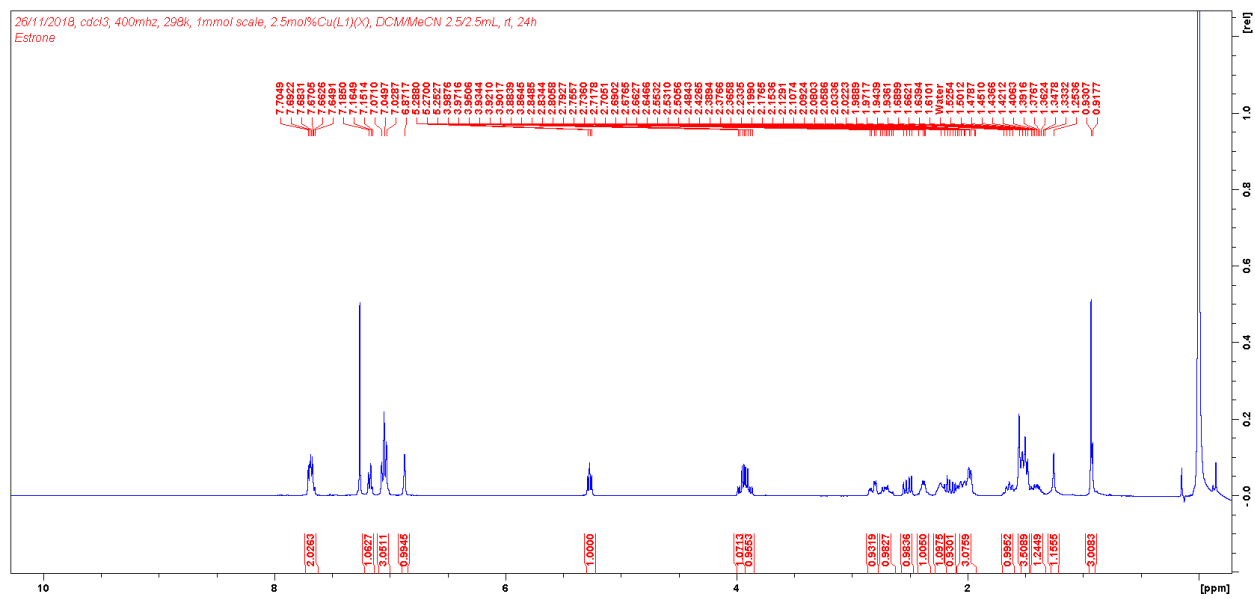


Figure S132. ^1H NMR spectrum of **10z**.

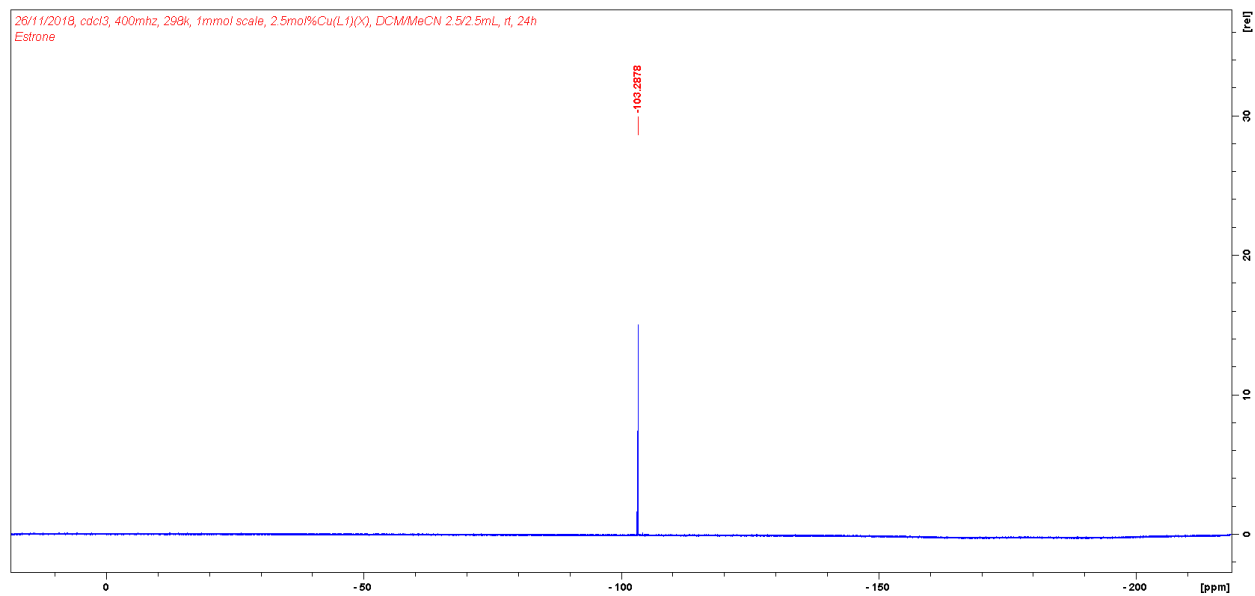


Figure S133. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **10z**.

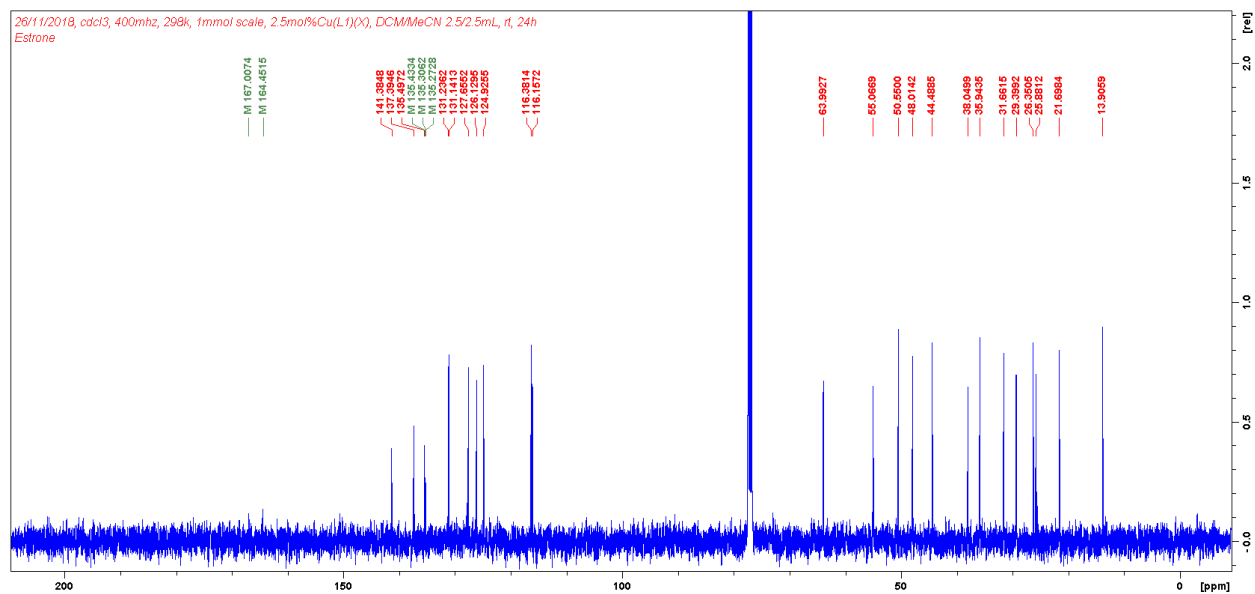


Figure S134. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **10z**.

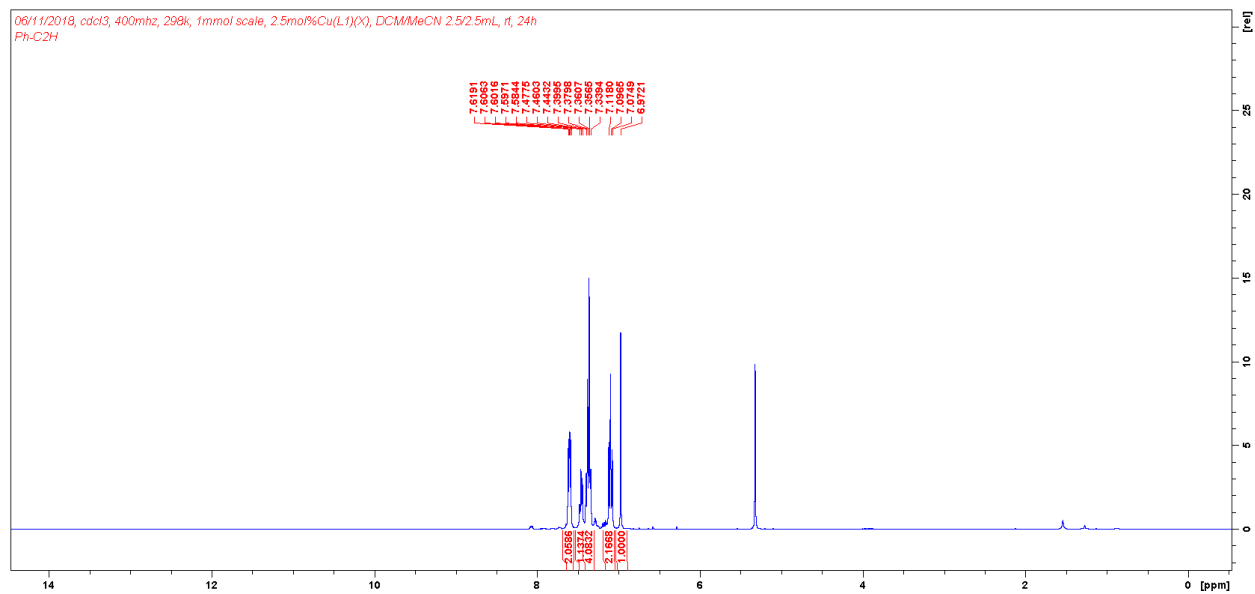
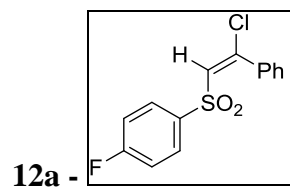


Figure S135. ^1H NMR spectrum of **12a**.

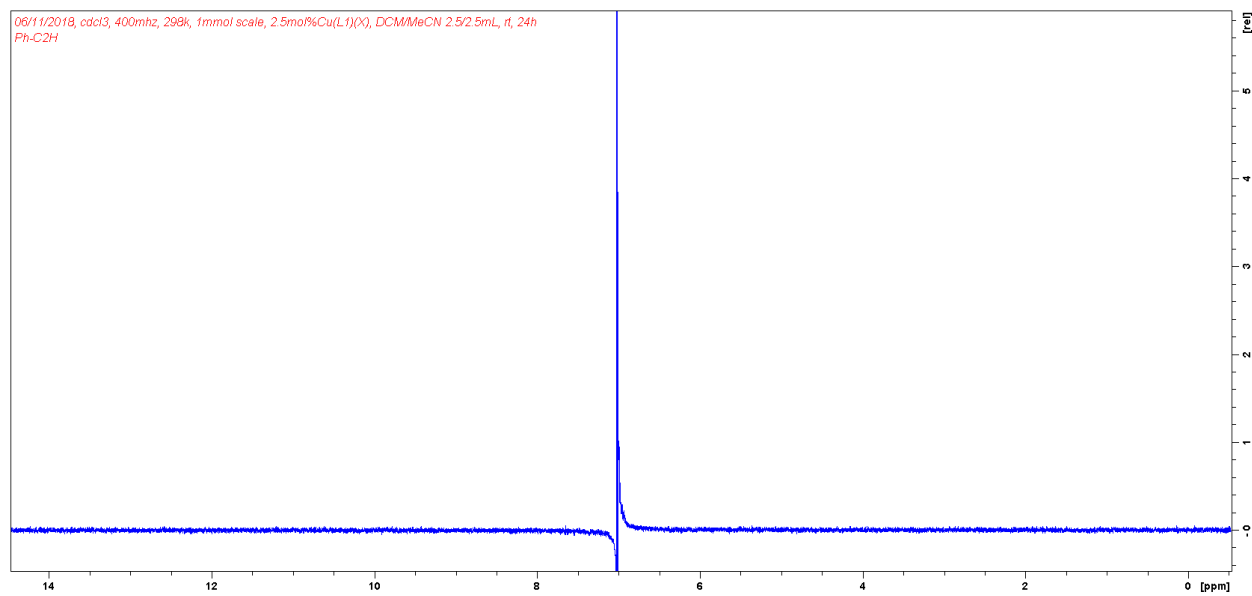


Figure S136. 1D NOESY spectrum of **12a**.

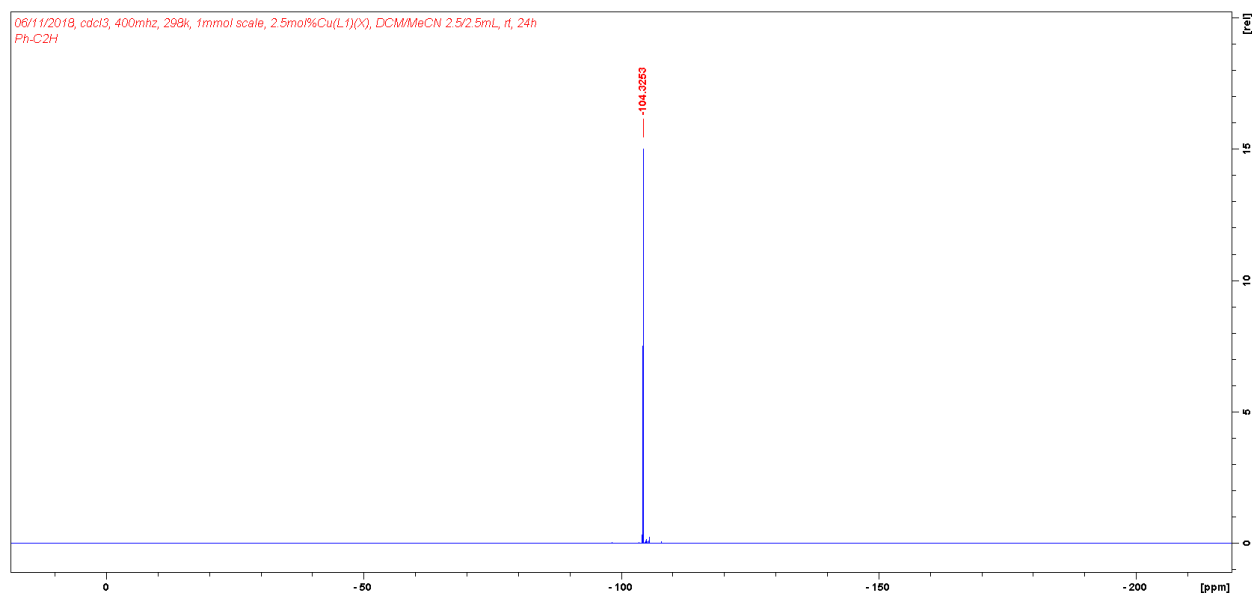


Figure S137. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **12a**.

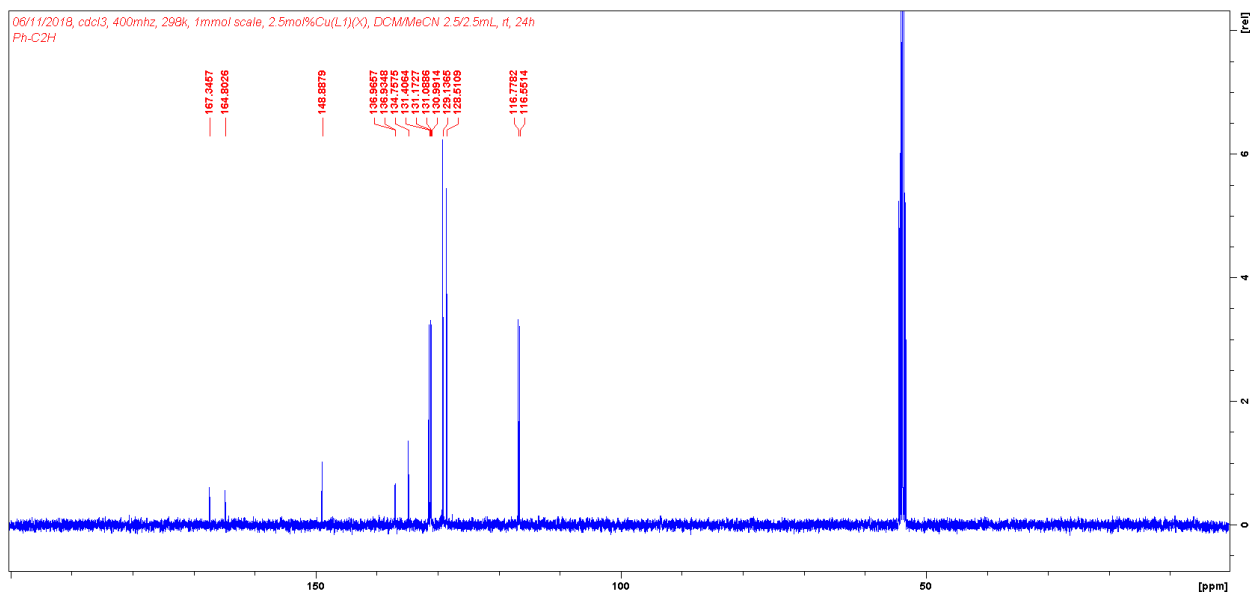


Figure S138. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **12a**.

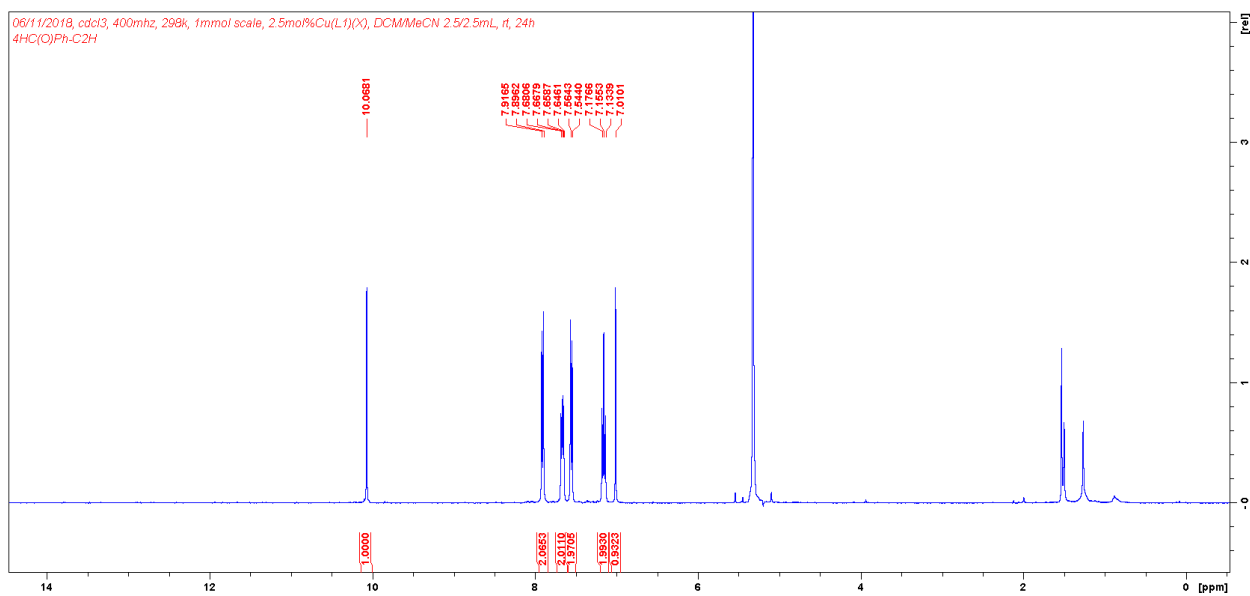
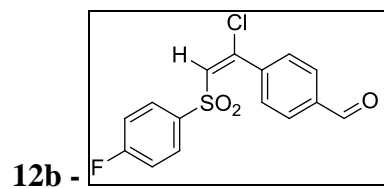


Figure S139. ^1H NMR spectrum of **12b**.

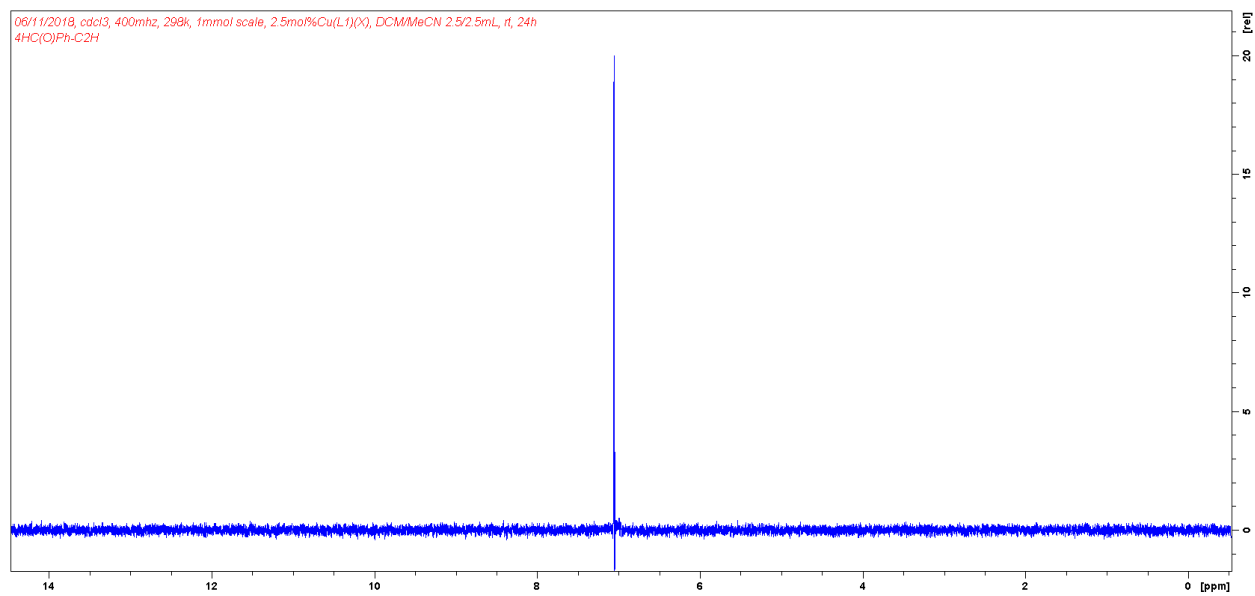


Figure S140. 1D NOESY spectrum of **12b**.

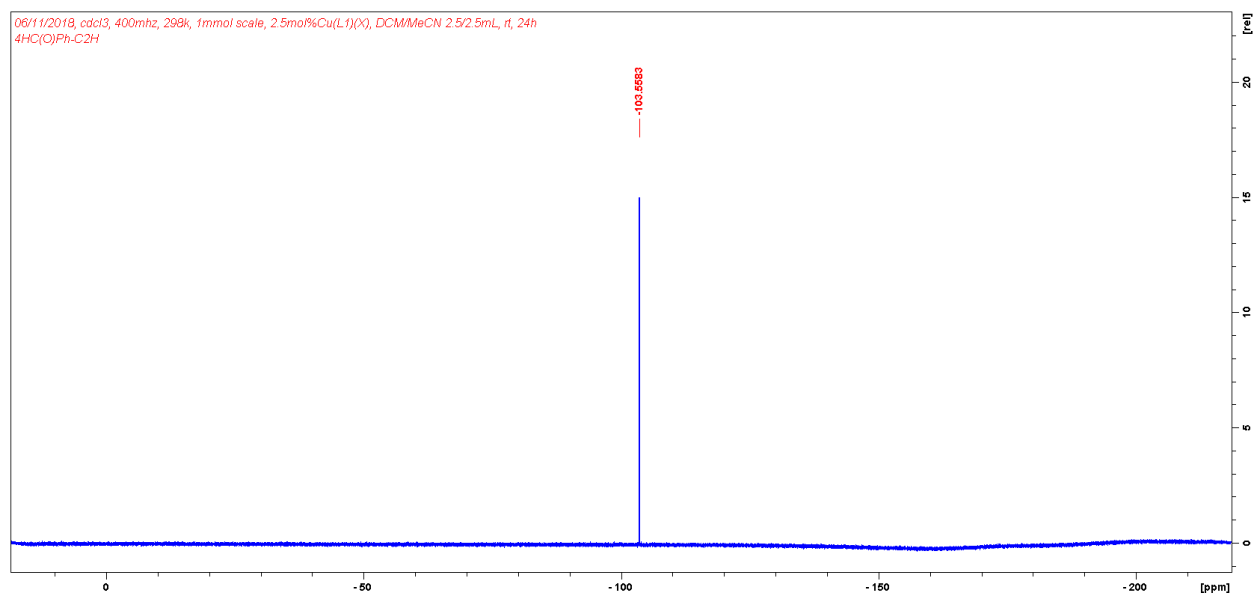


Figure S141. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **12b**.

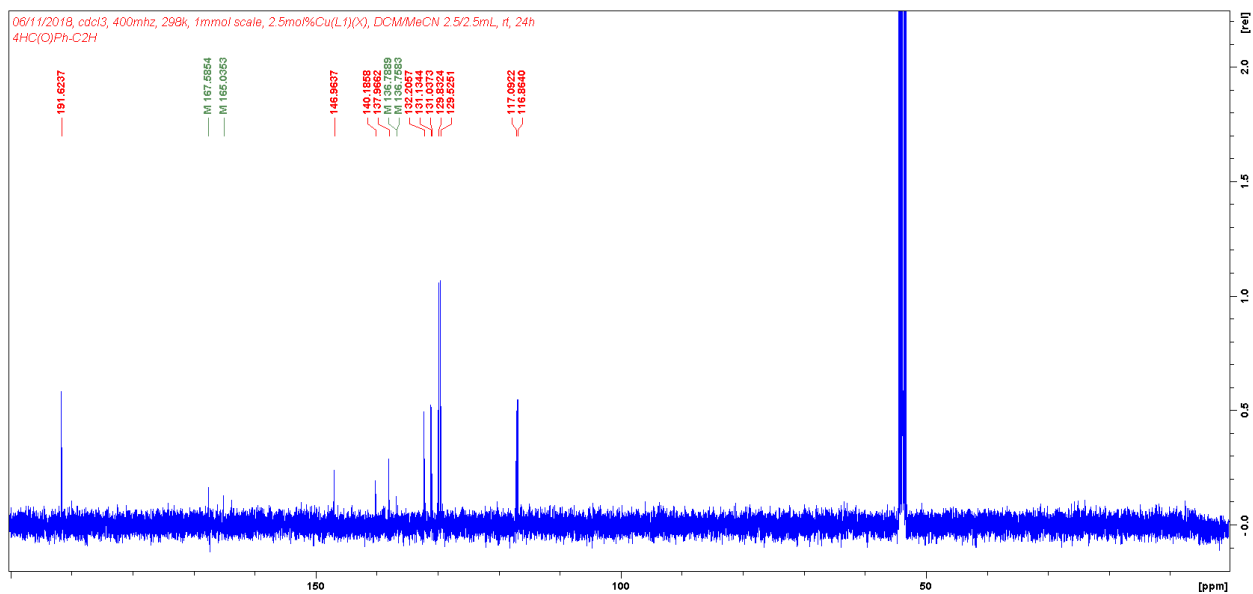


Figure S142. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **12b**.

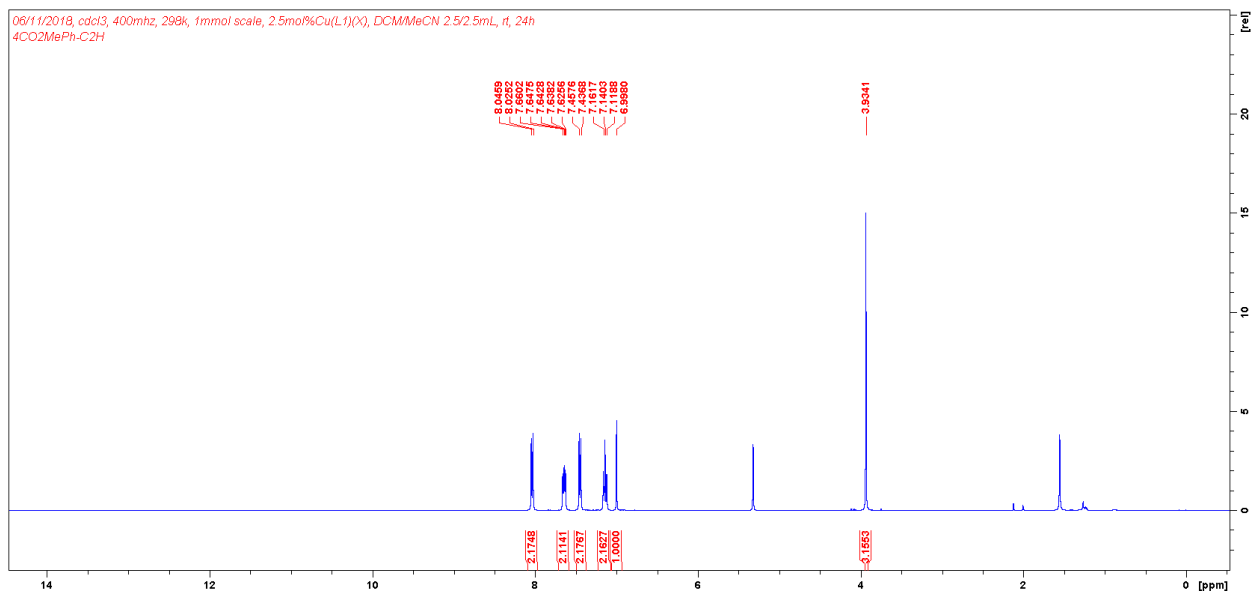
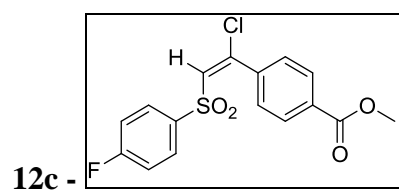


Figure S143. ^1H NMR spectrum of **12c**.

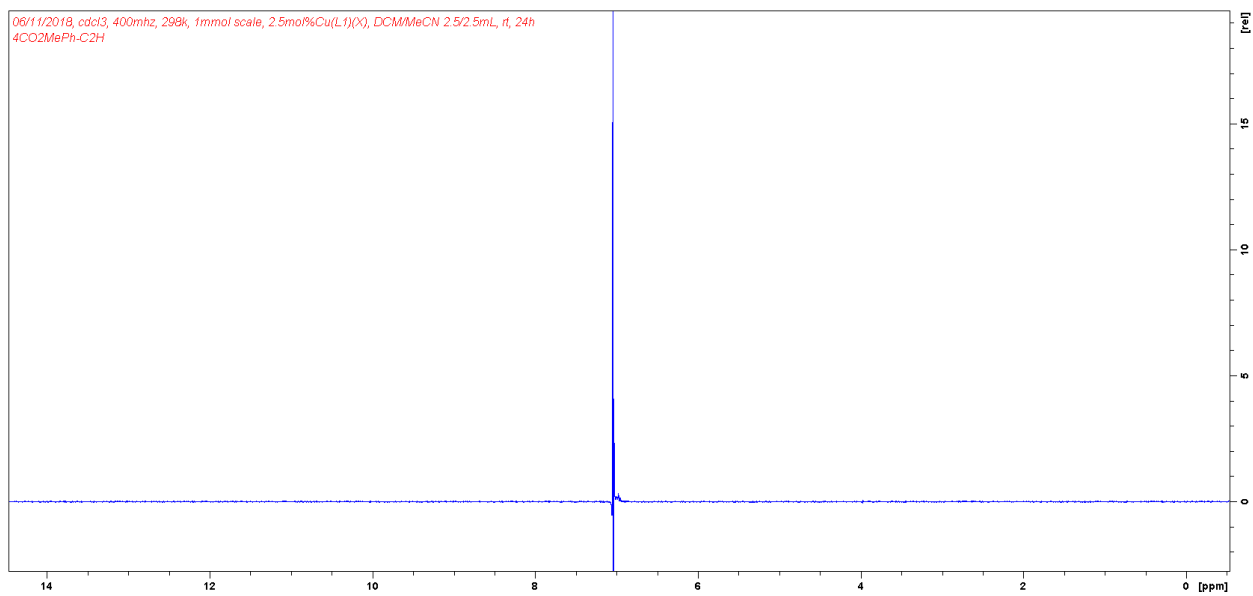


Figure S144. 1D NOESY spectrum of **12c**.

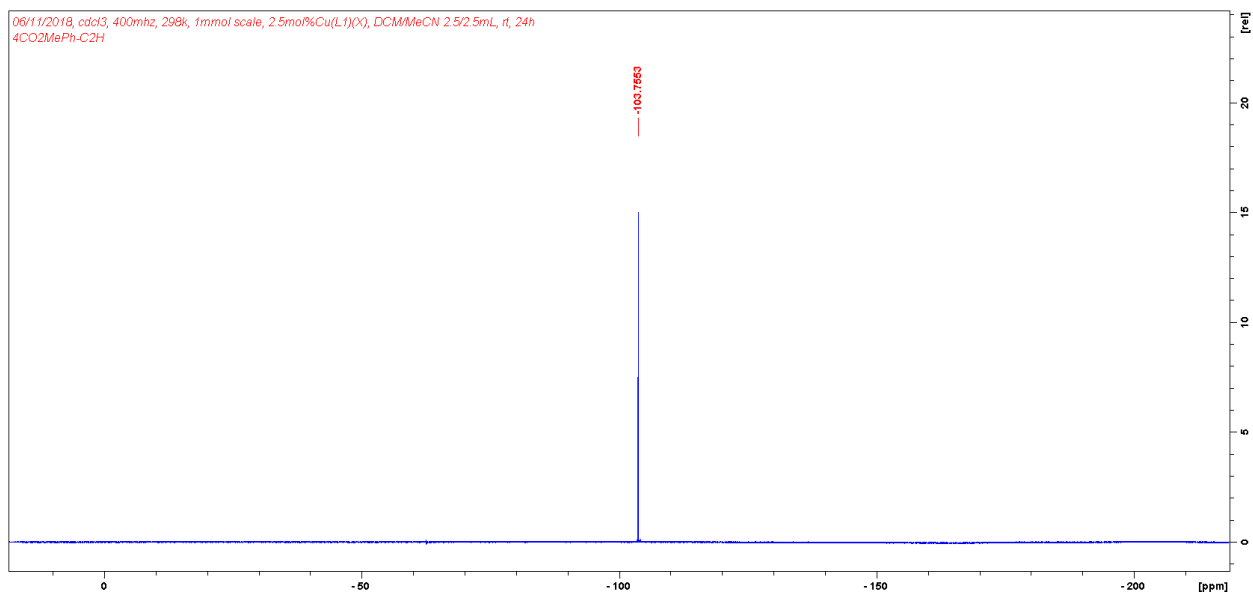


Figure S145. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **12c**.

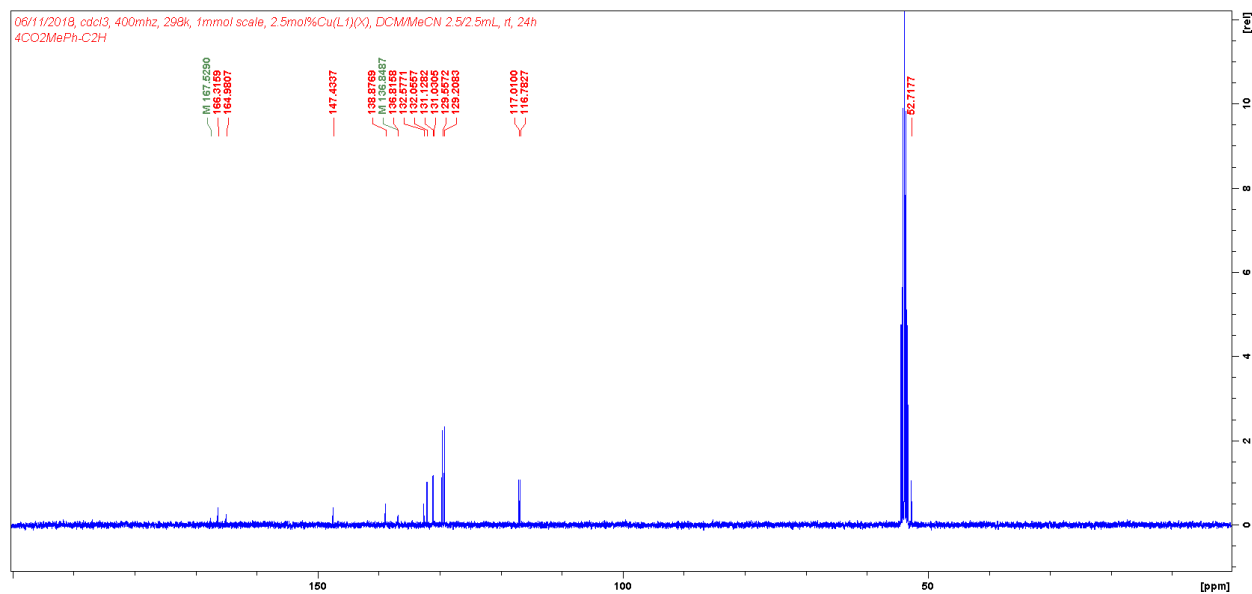


Figure S146. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **12c**.

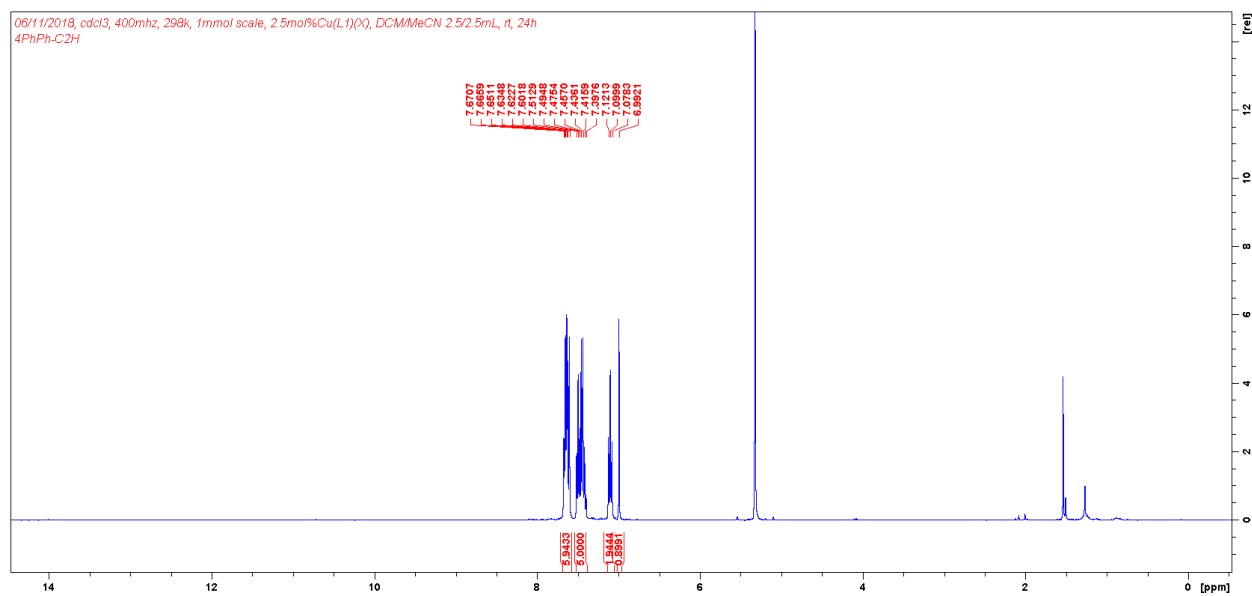
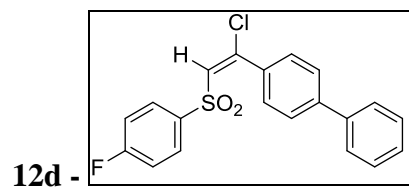


Figure S147. ^1H NMR spectrum of **12d**.

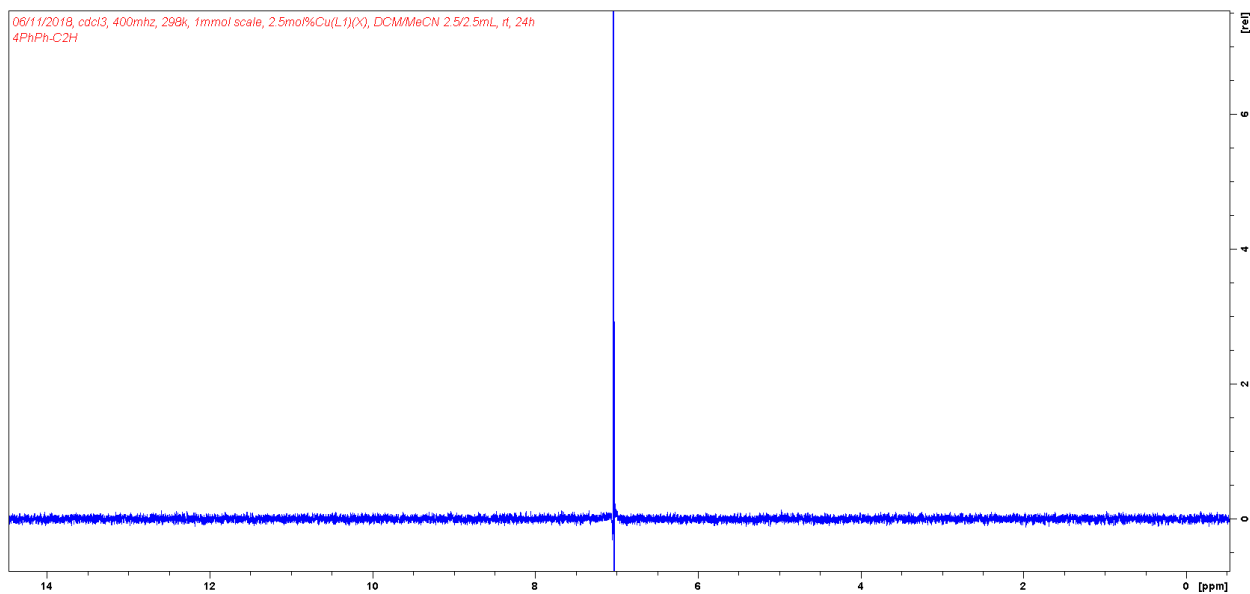


Figure S148. 1D NOESY spectrum of **12d**.

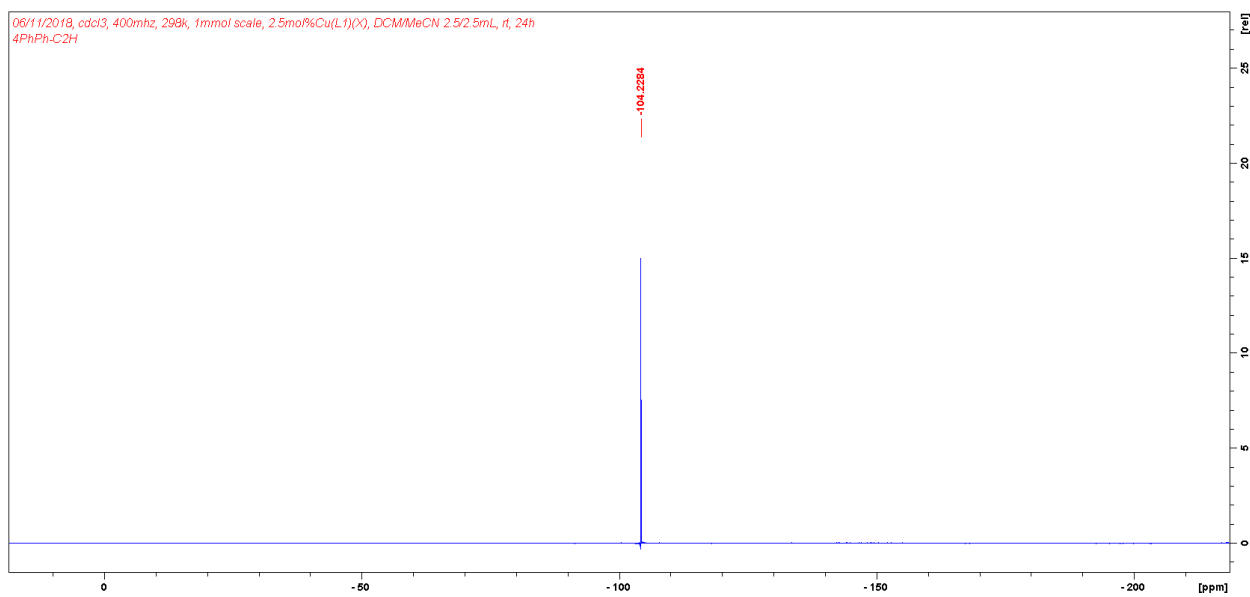


Figure S149. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **12d**.

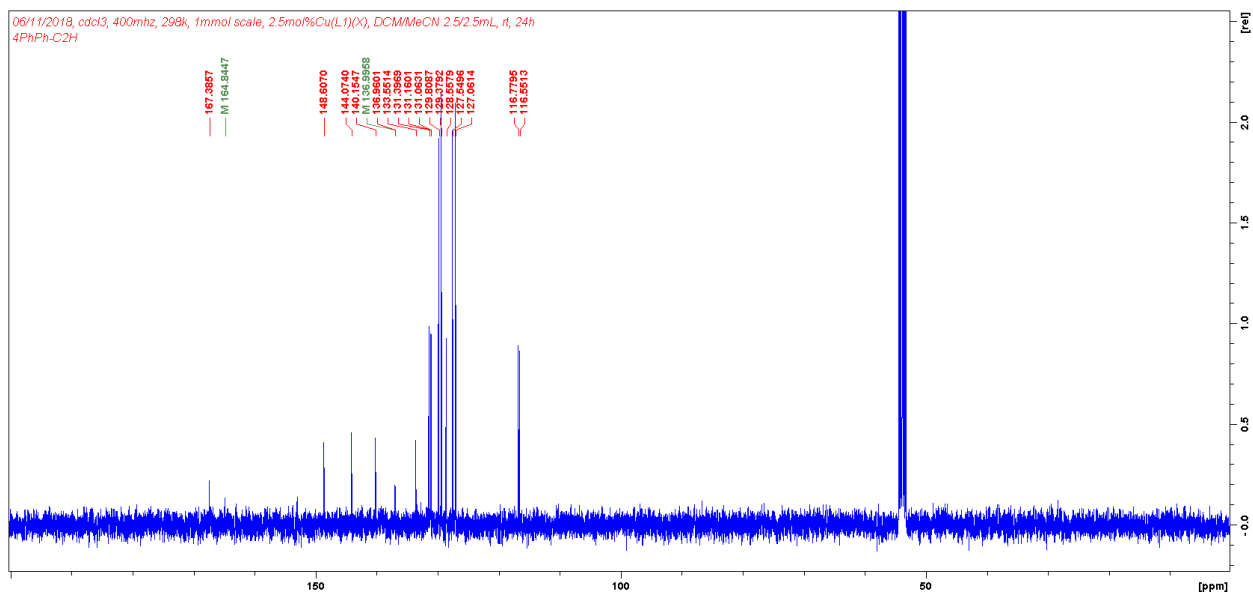


Figure S150. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **12d**.

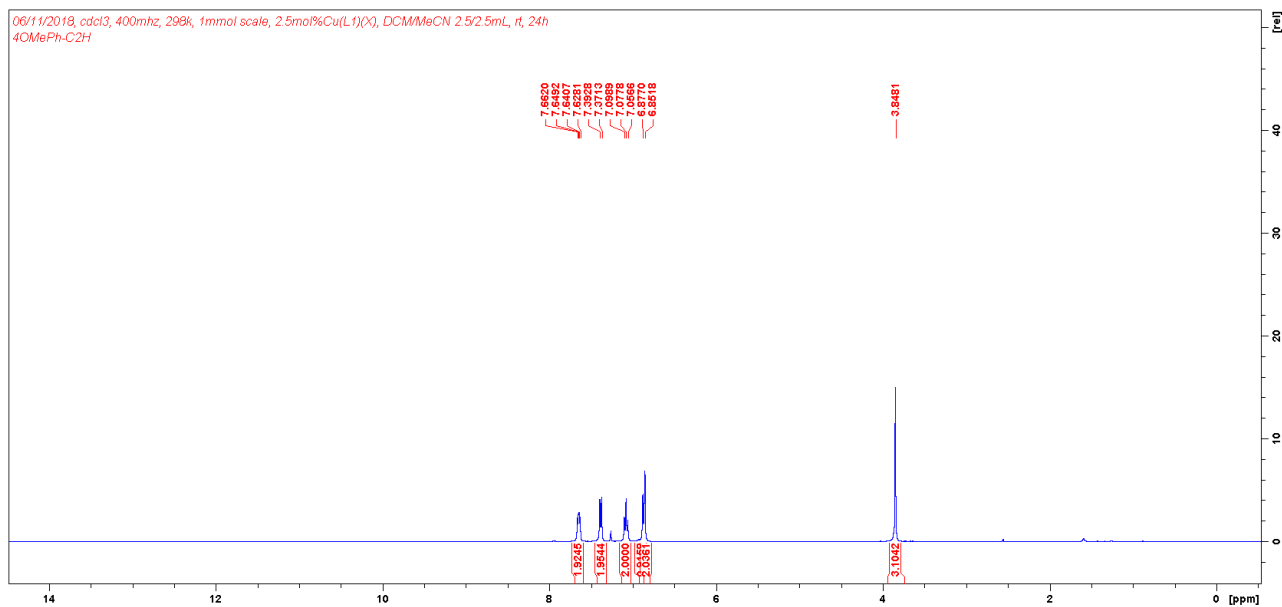
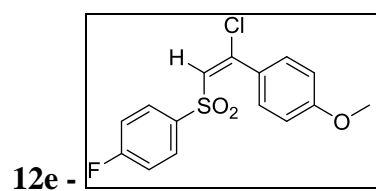


Figure S151. ^1H NMR spectrum of **12e**.

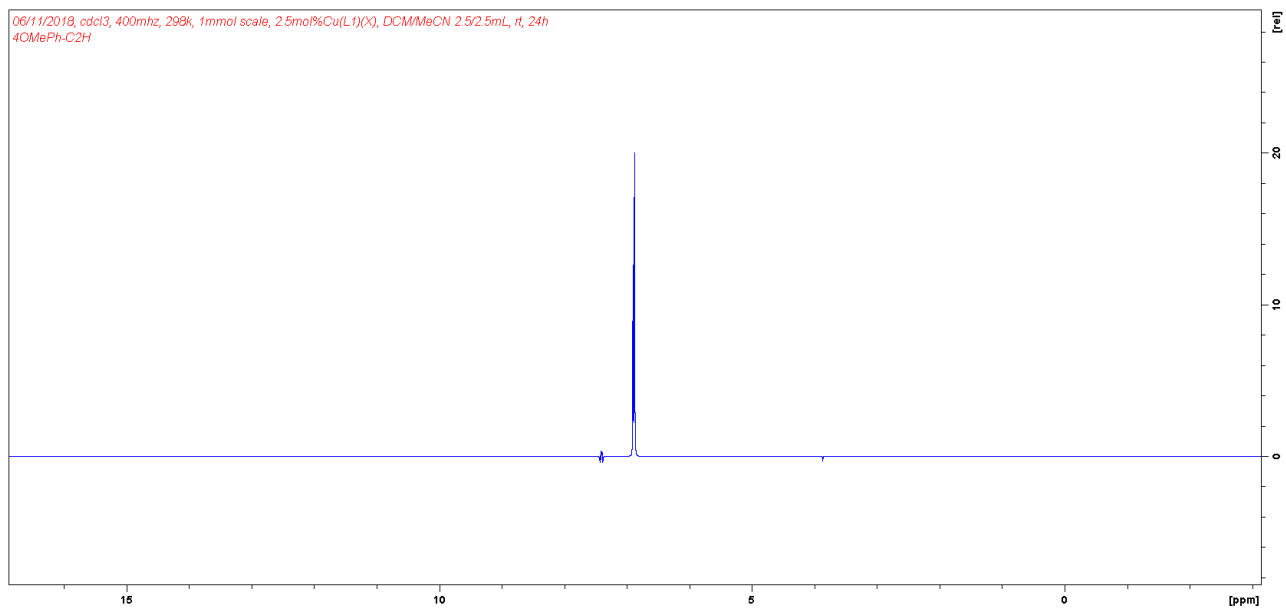


Figure S152. 1D NOESY spectrum of **12e**.

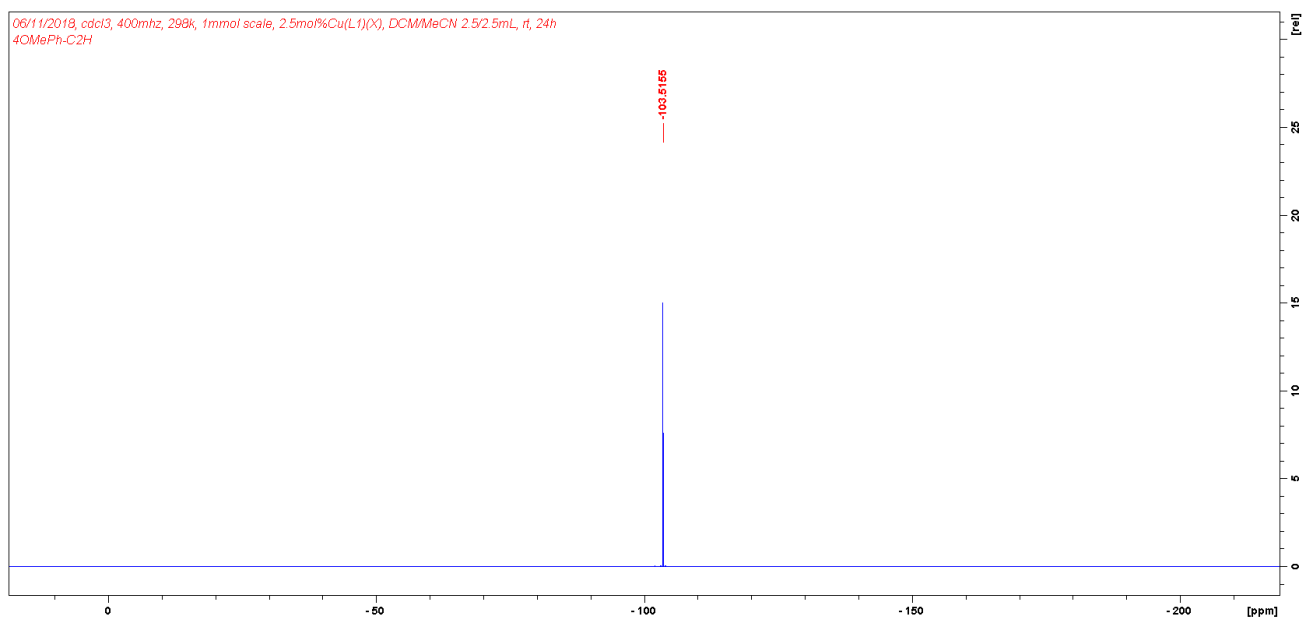


Figure S153. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **12e**.

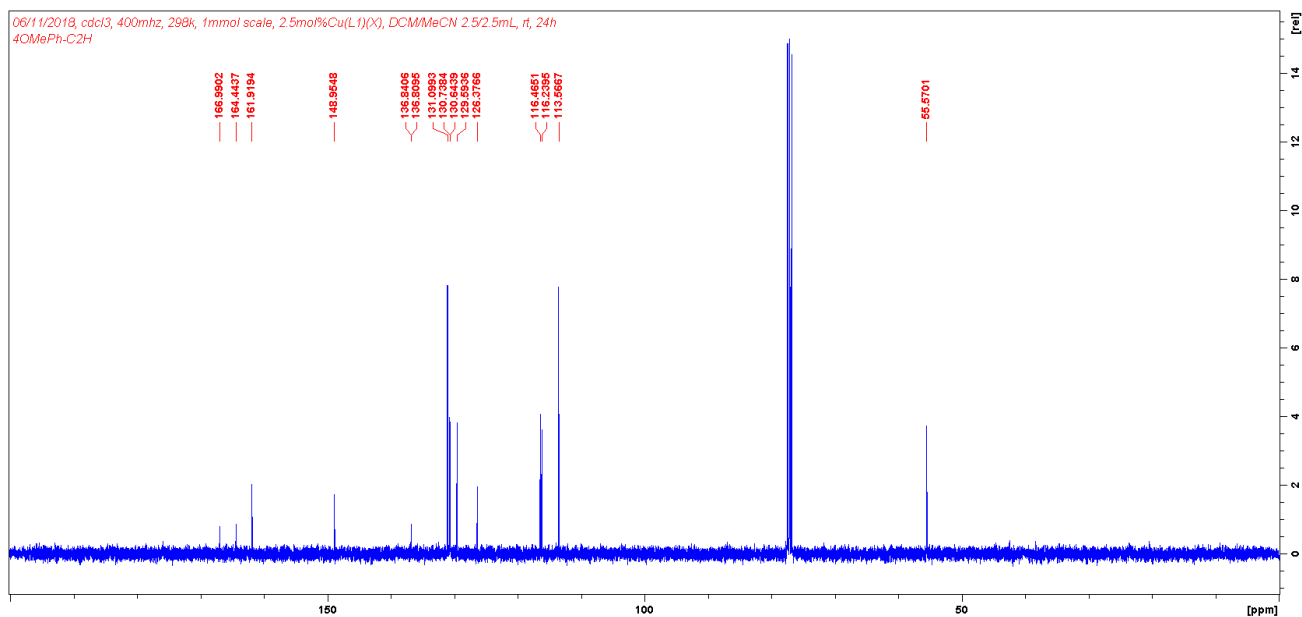


Figure S154. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **12e**.

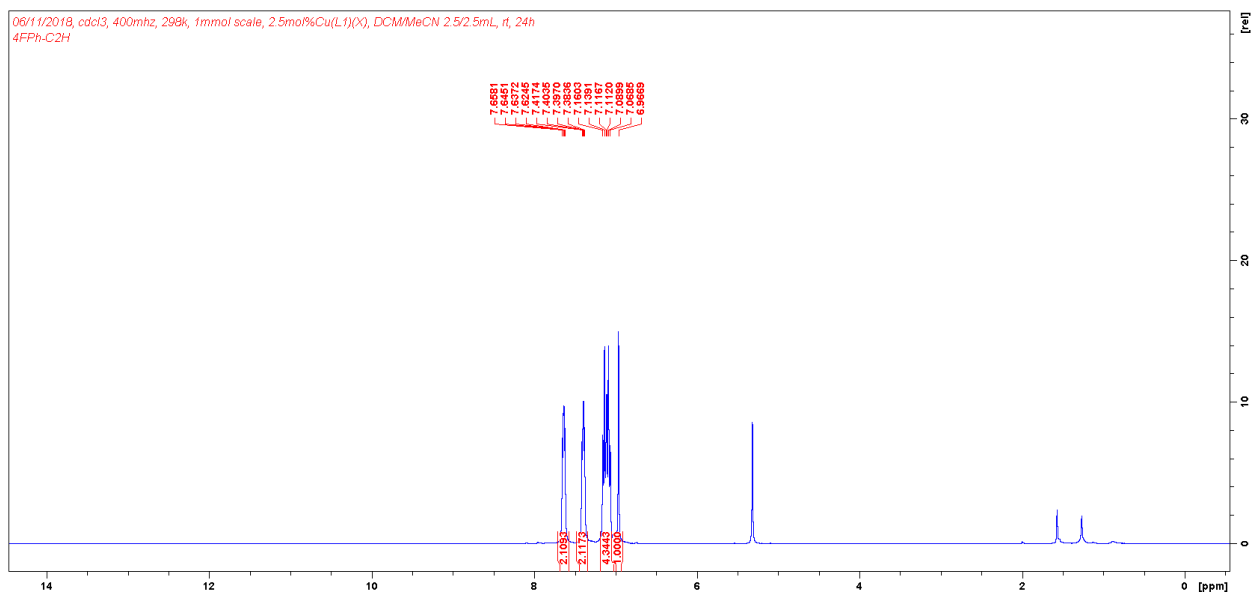
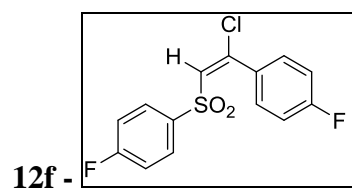


Figure S155: ^1H NMR spectrum of **12f**.

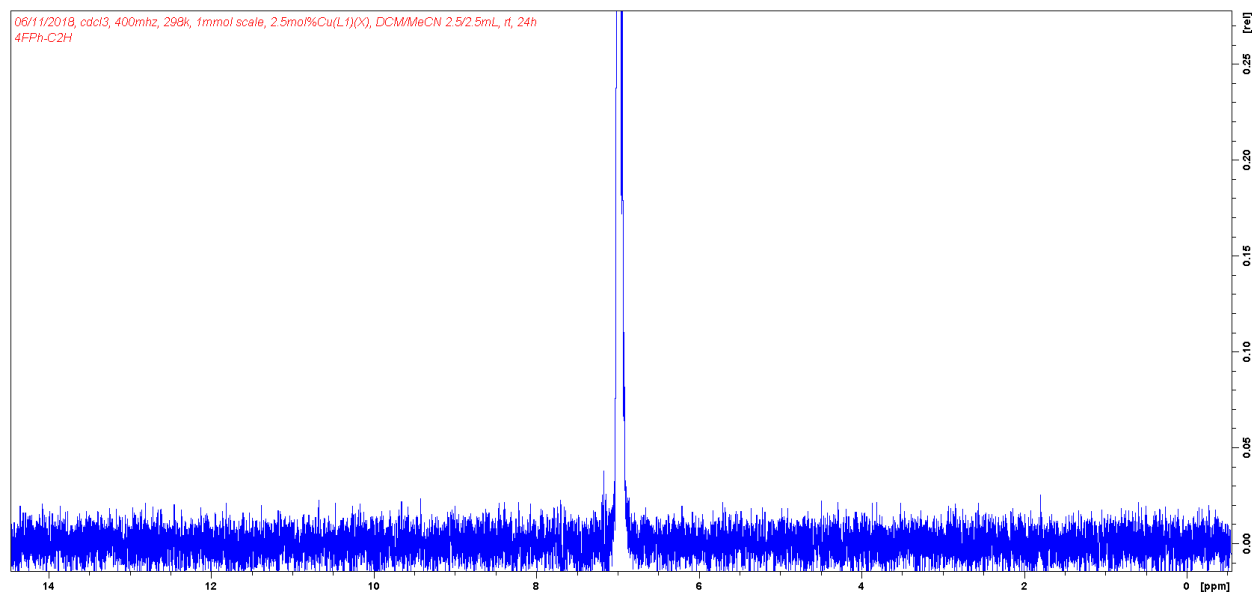


Figure S156. 1D NOESY spectrum of **12f**.

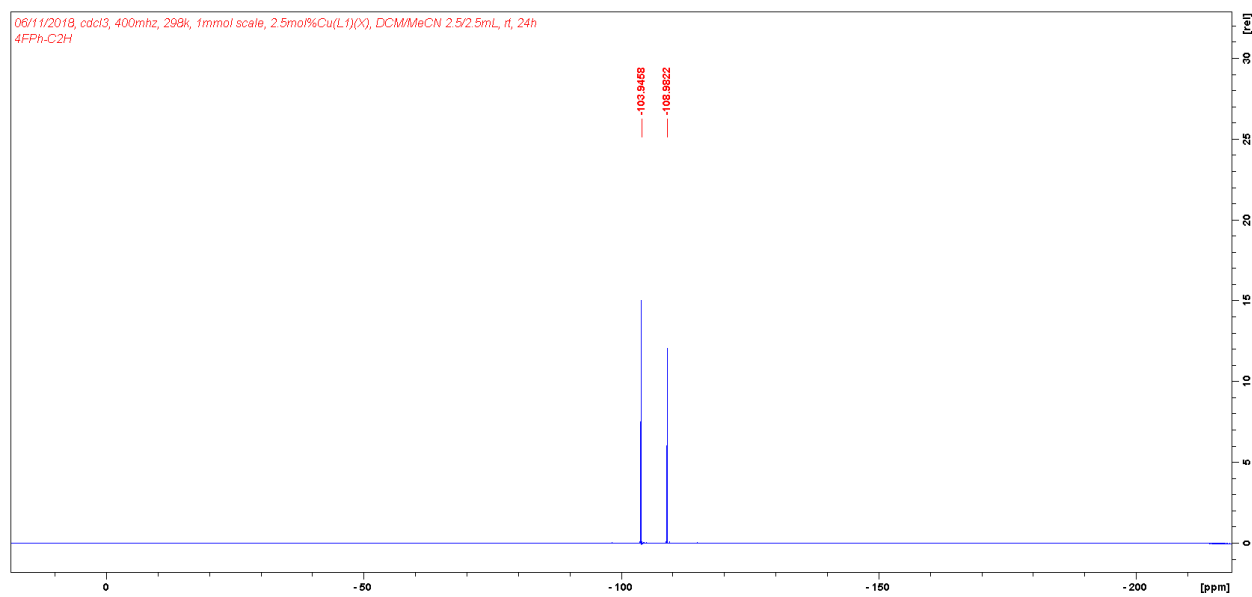


Figure S157. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **12f**.

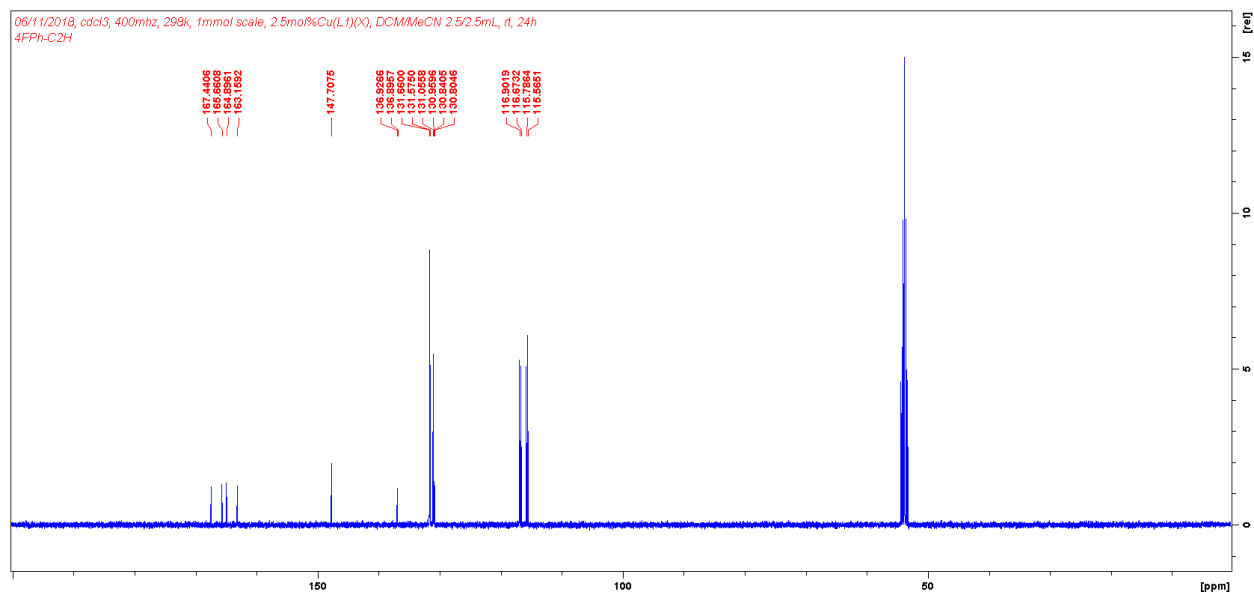


Figure S158. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **12f**.

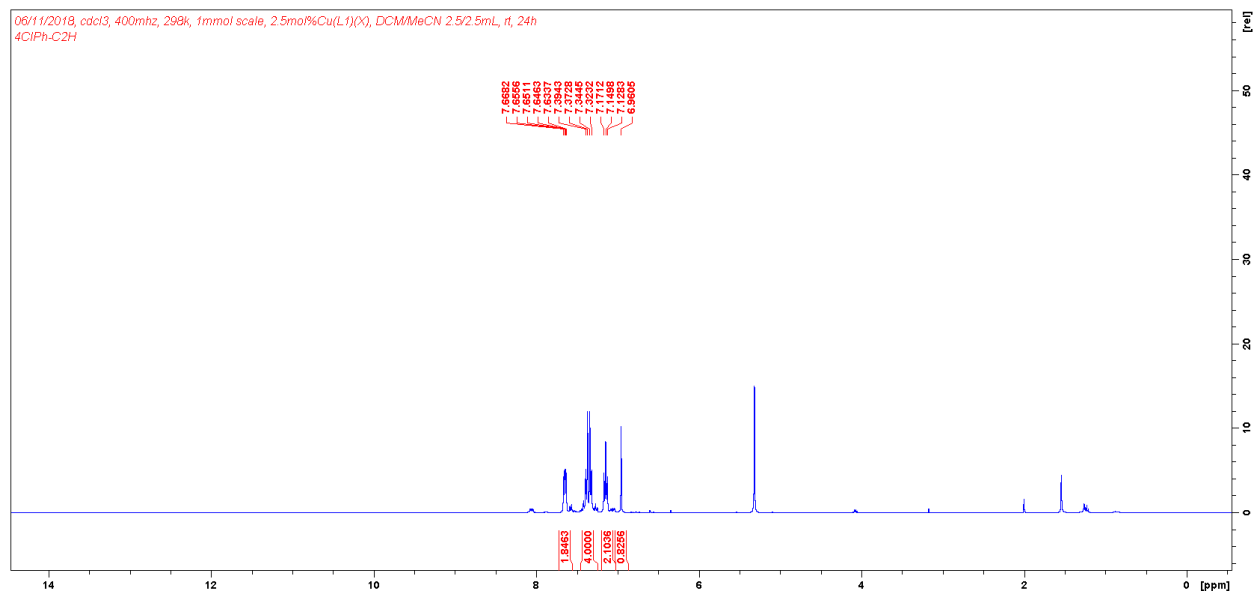
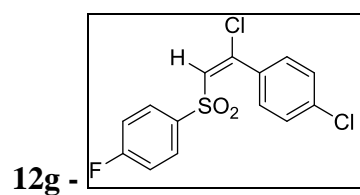


Figure S159. ^1H NMR spectrum of **12g**.

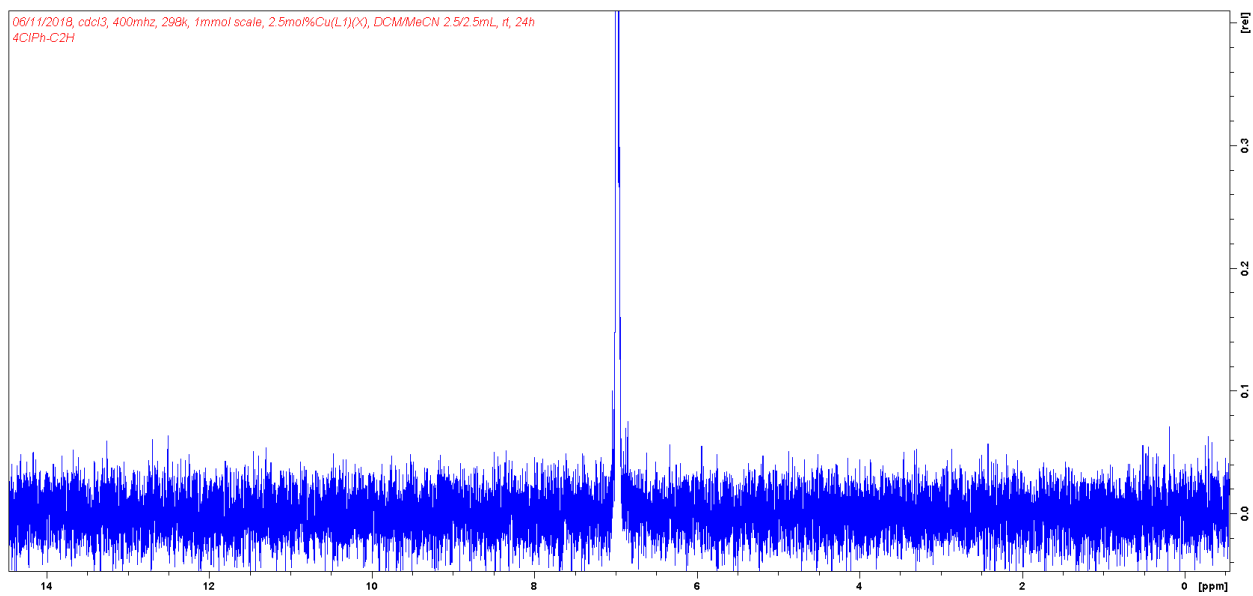


Figure S160. 1D NOESY spectrum of **12g**.

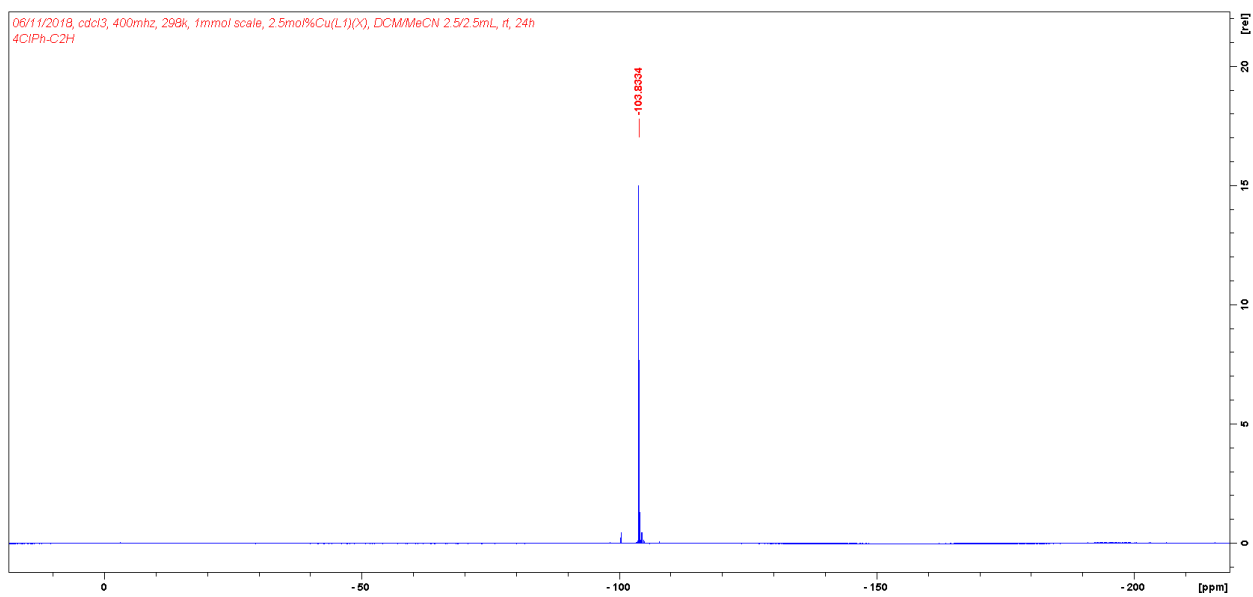


Figure S161. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **12g**.

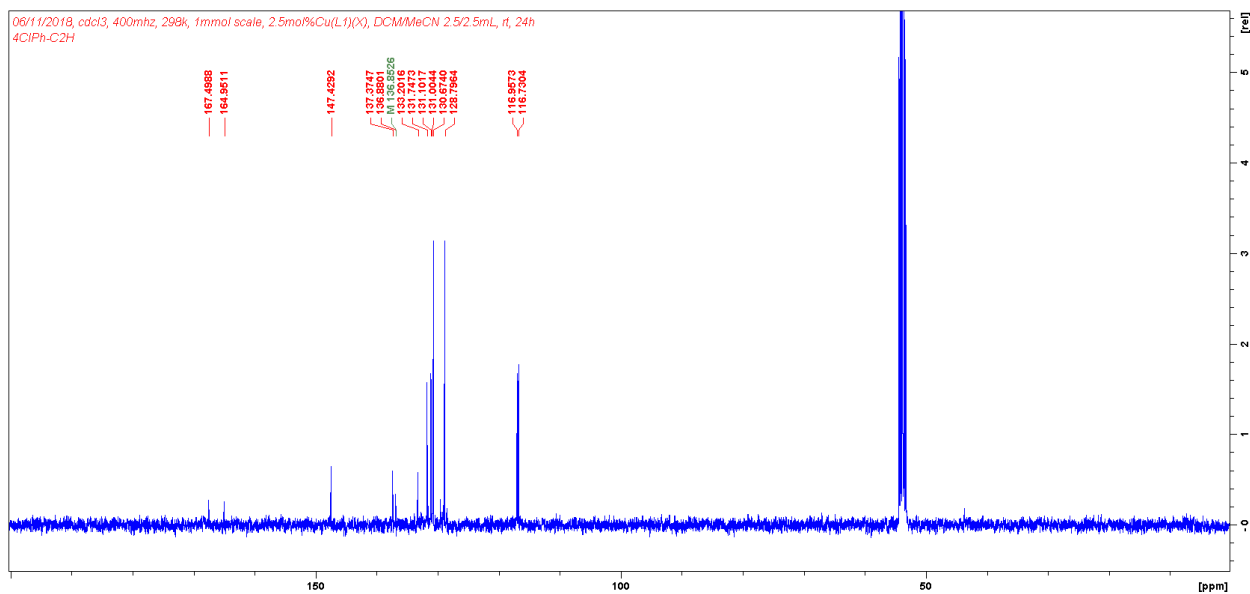


Figure S162. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **12g**.

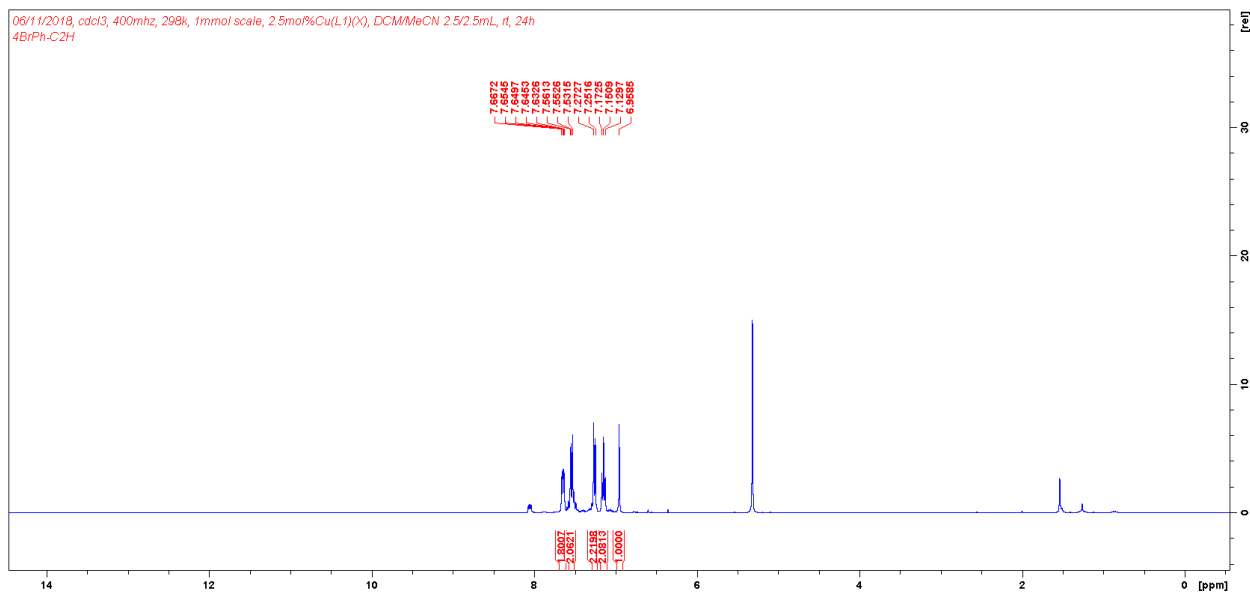
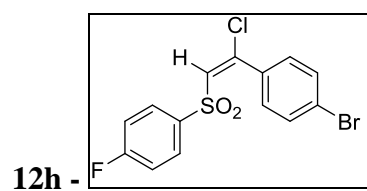


Figure S163. ^1H NMR spectrum of **12h**.

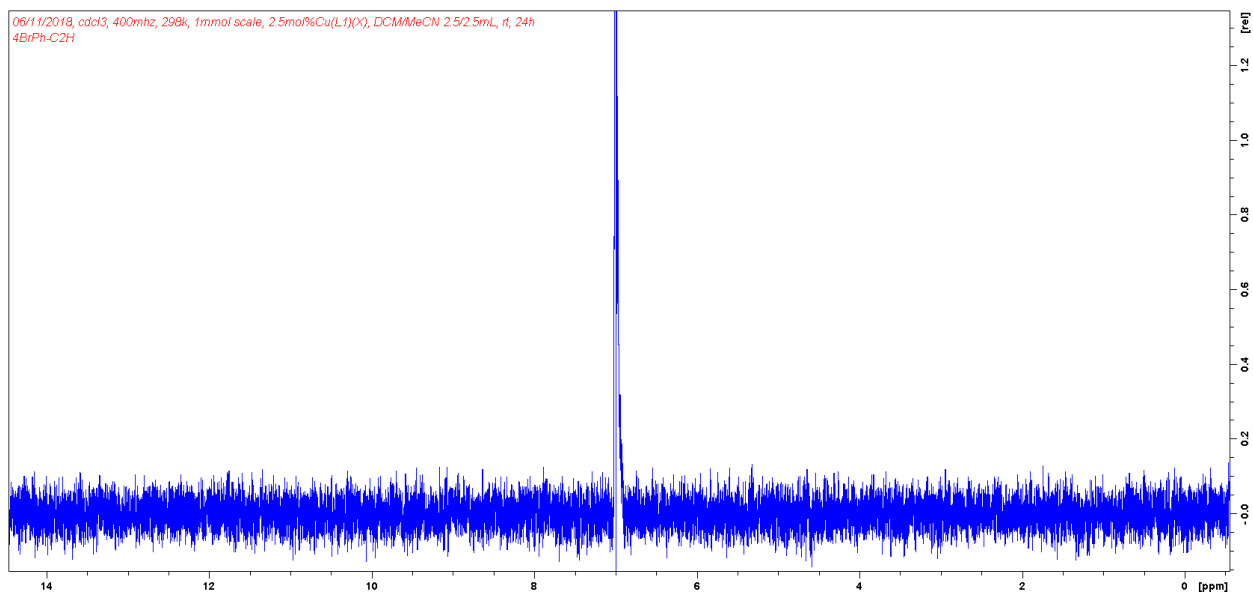


Figure S164. 1D NOESY spectrum of **12h**.

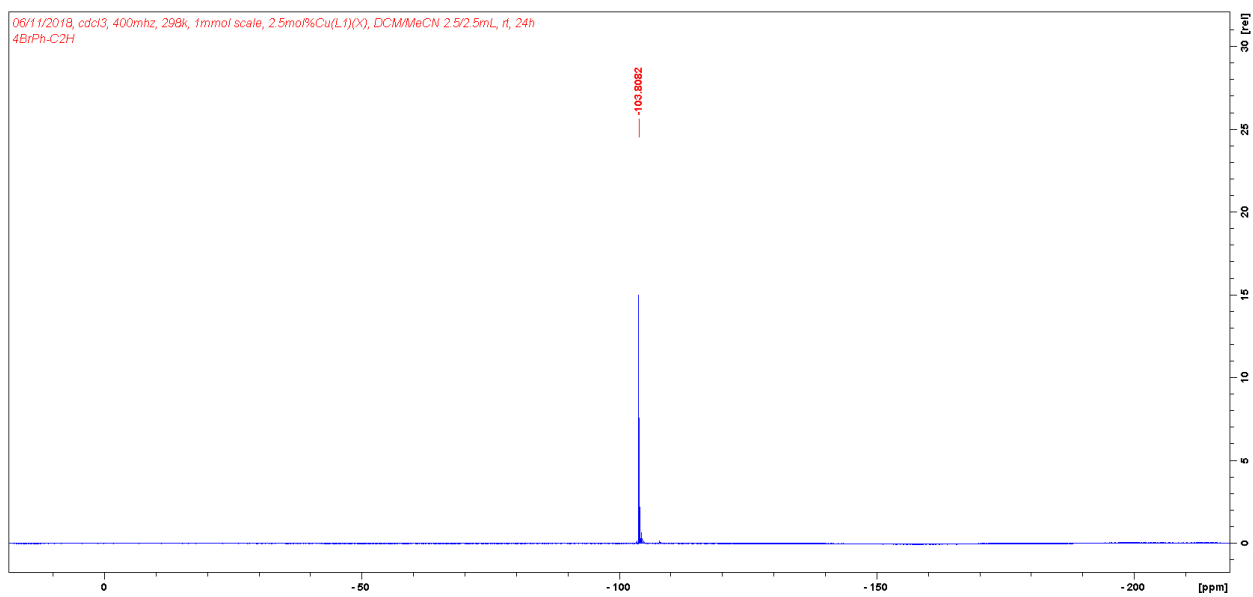


Figure S165. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **12h**.

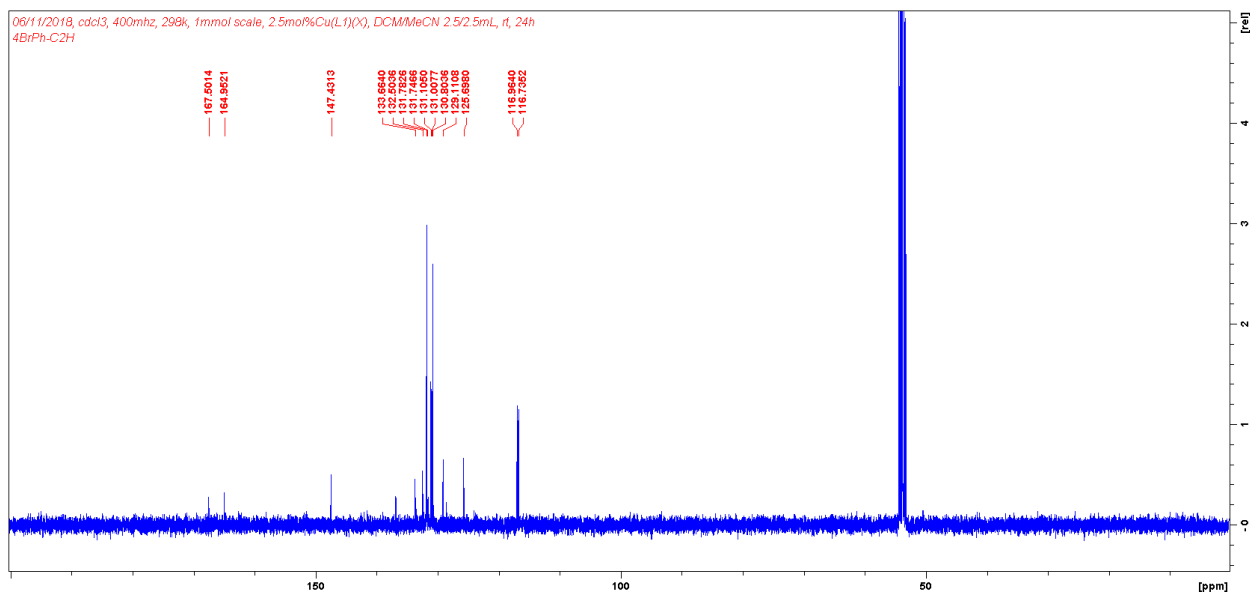


Figure S166. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **12h**.

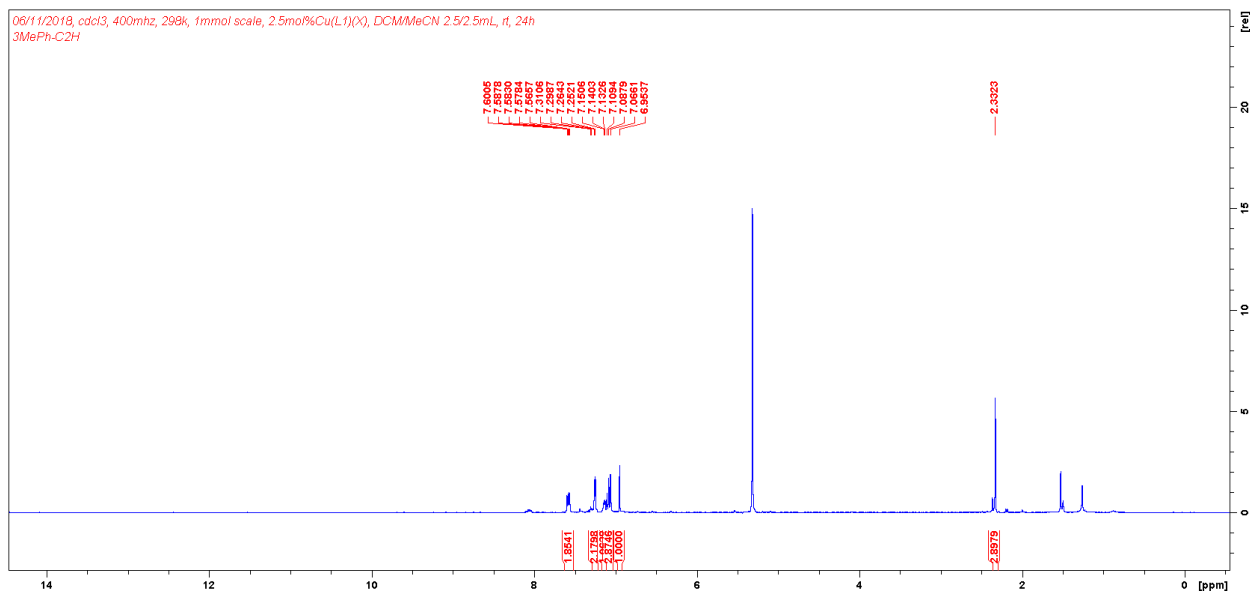
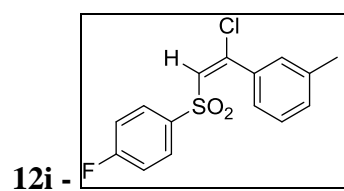


Figure S167. ^1H NMR spectrum of **12i**.

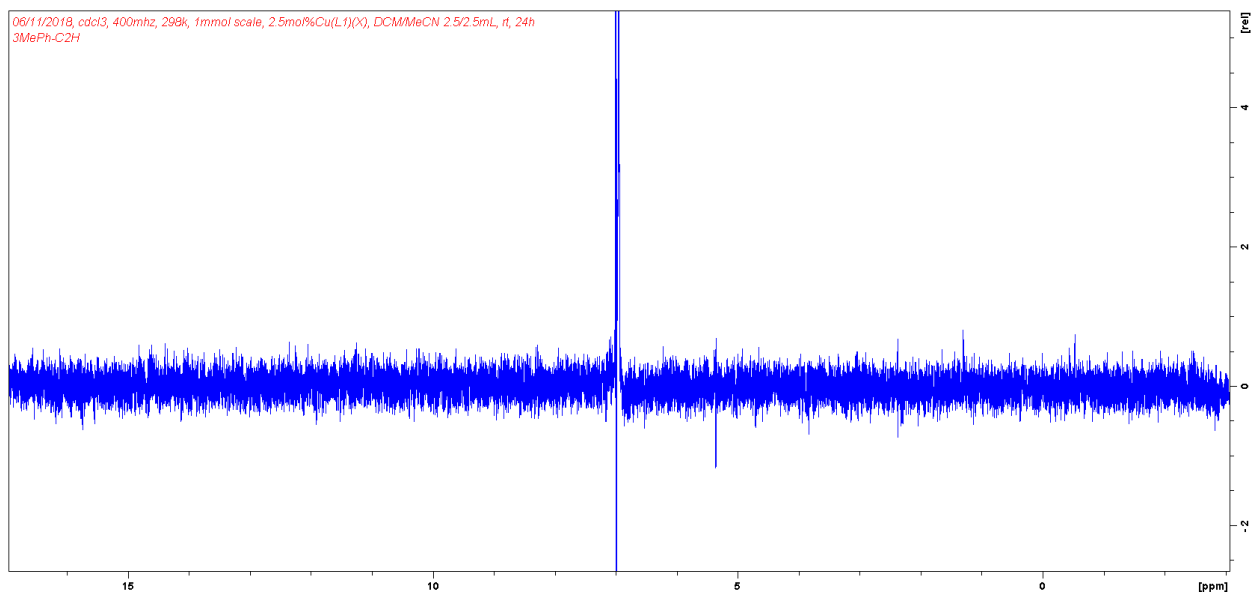


Figure S168. 1D NOESY spectrum of **12i**.

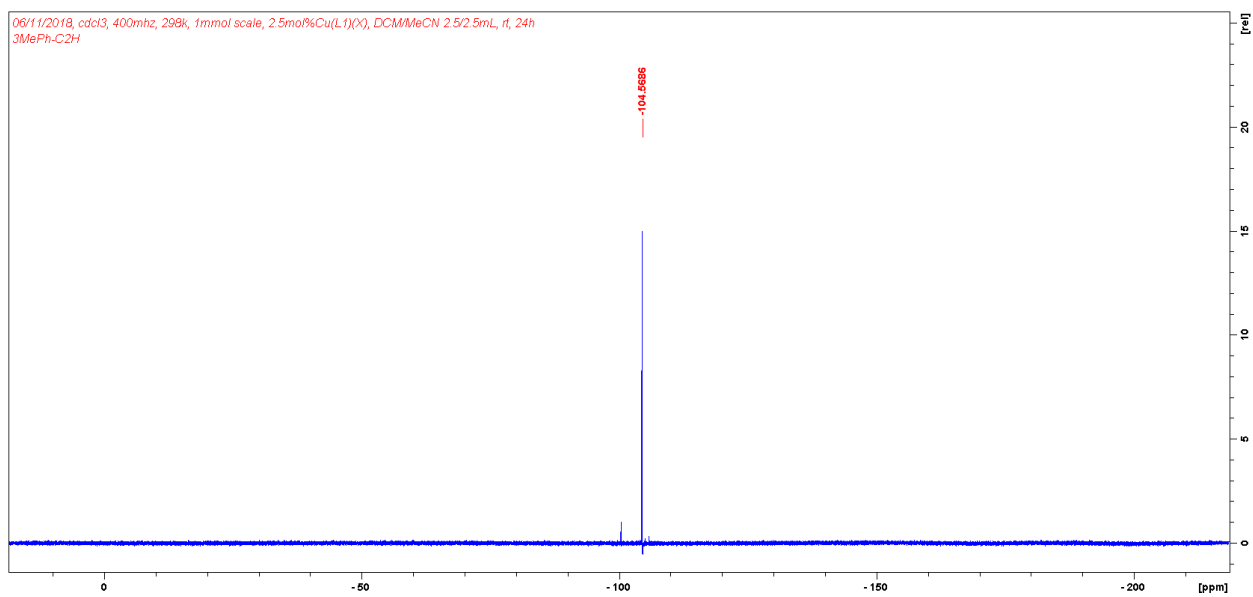


Figure S169. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **12i**.

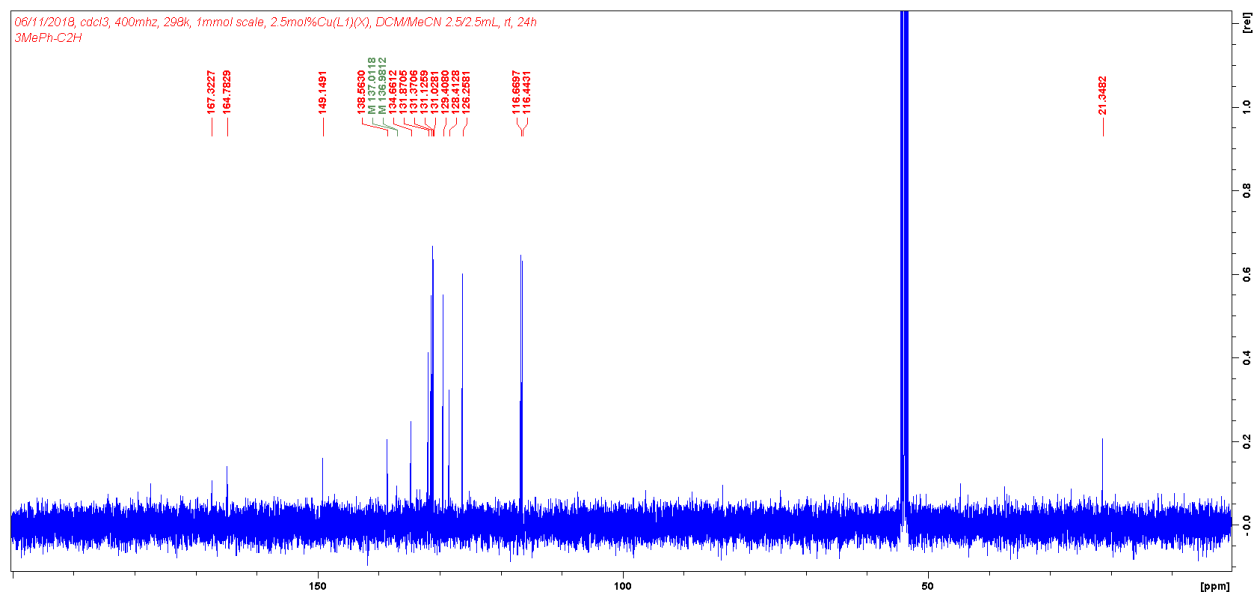


Figure S170. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **12i**.

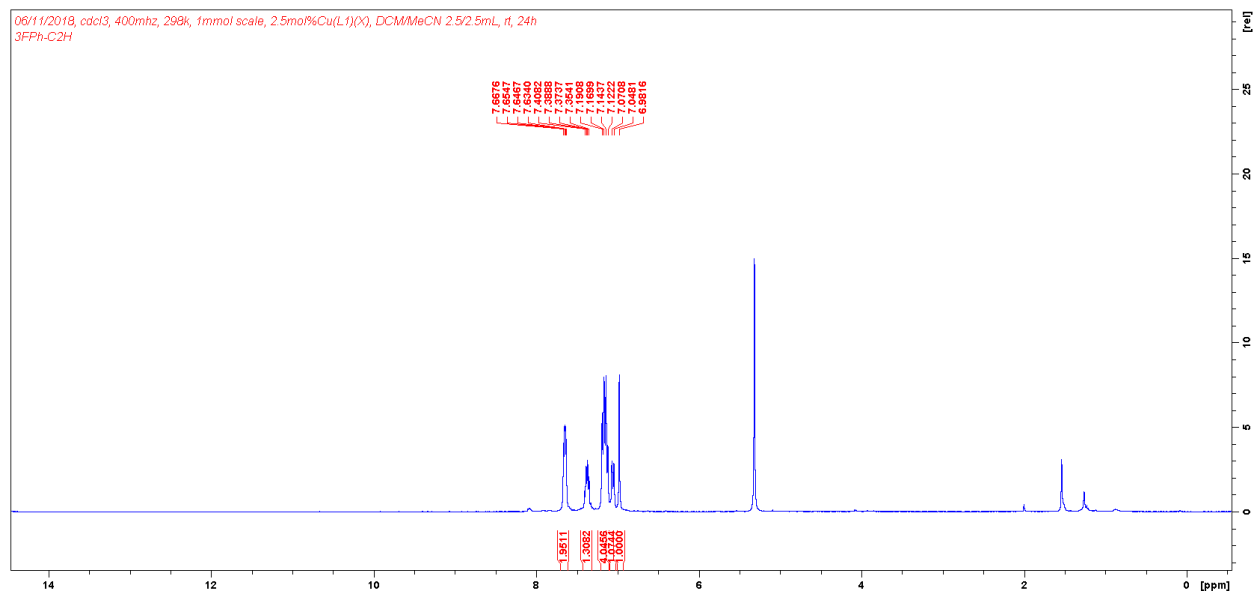
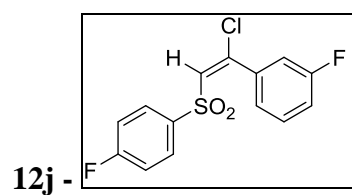


Figure S171. ^1H NMR spectrum of **12h**.

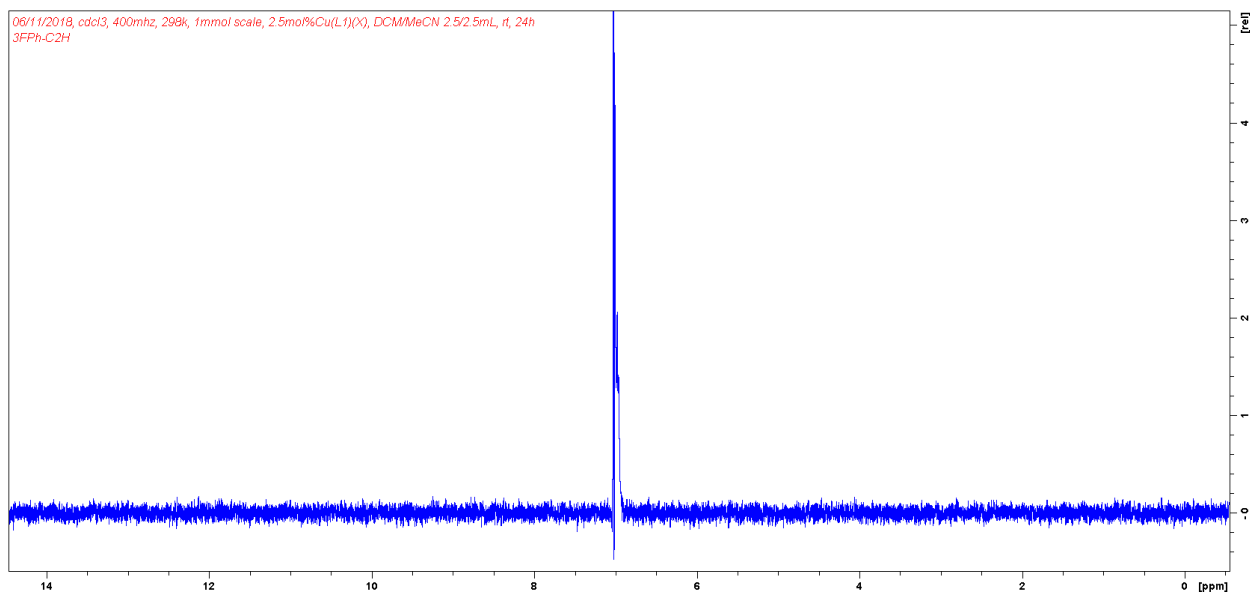


Figure S172. 1D NOESY spectrum of **12h**.

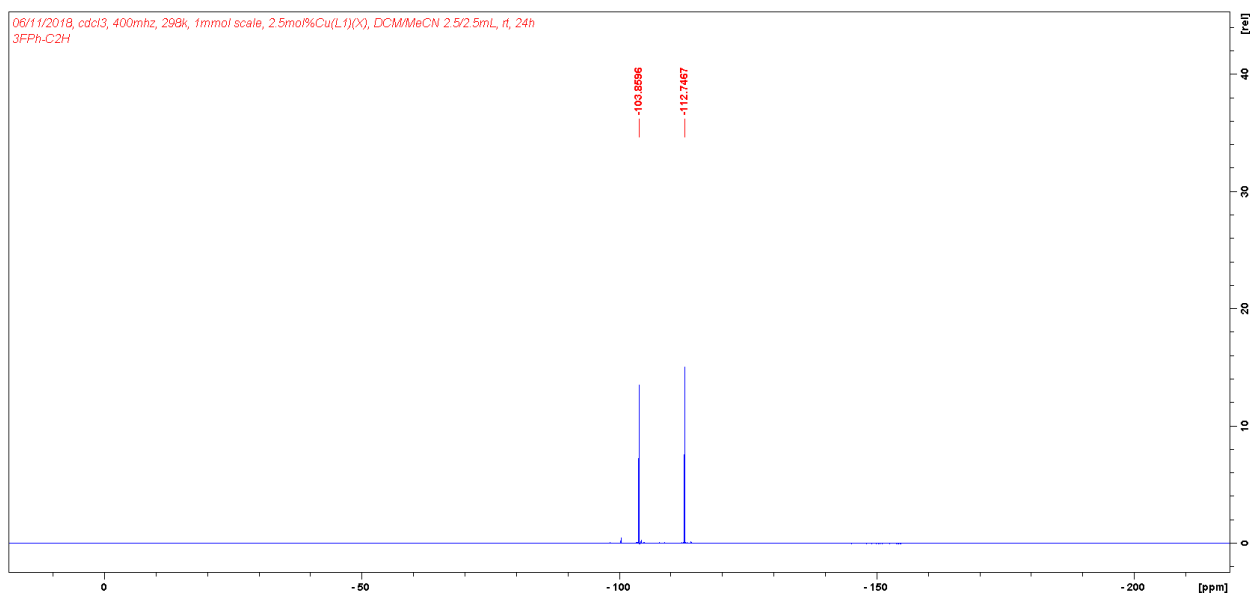


Figure S173. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **12h**.

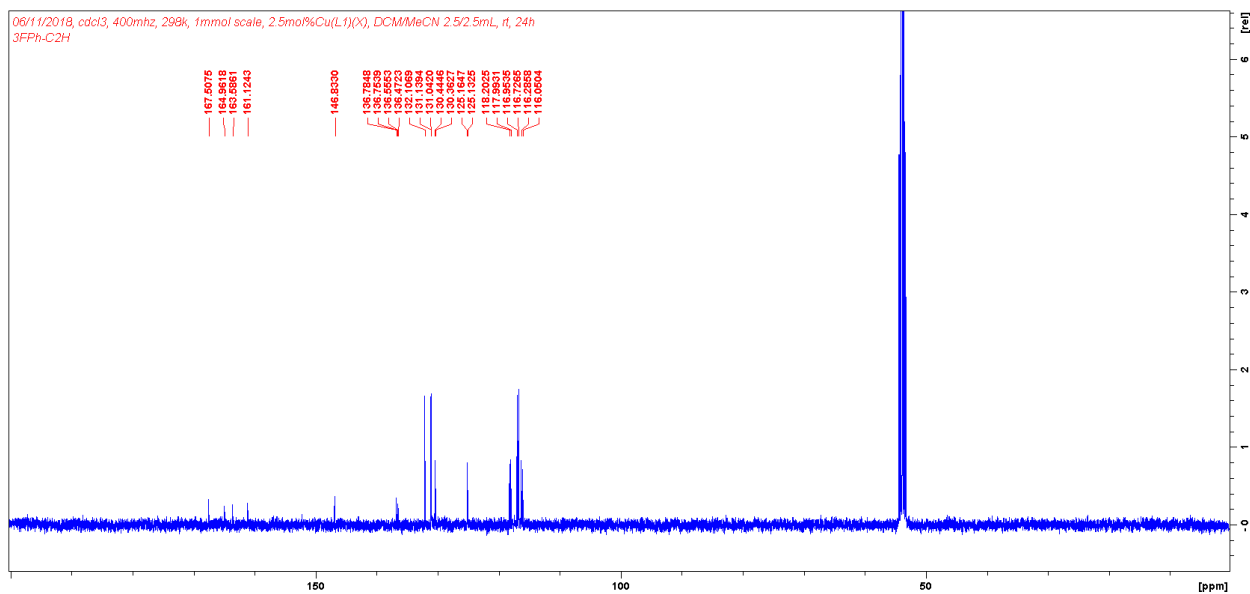


Figure S174. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **12h**.

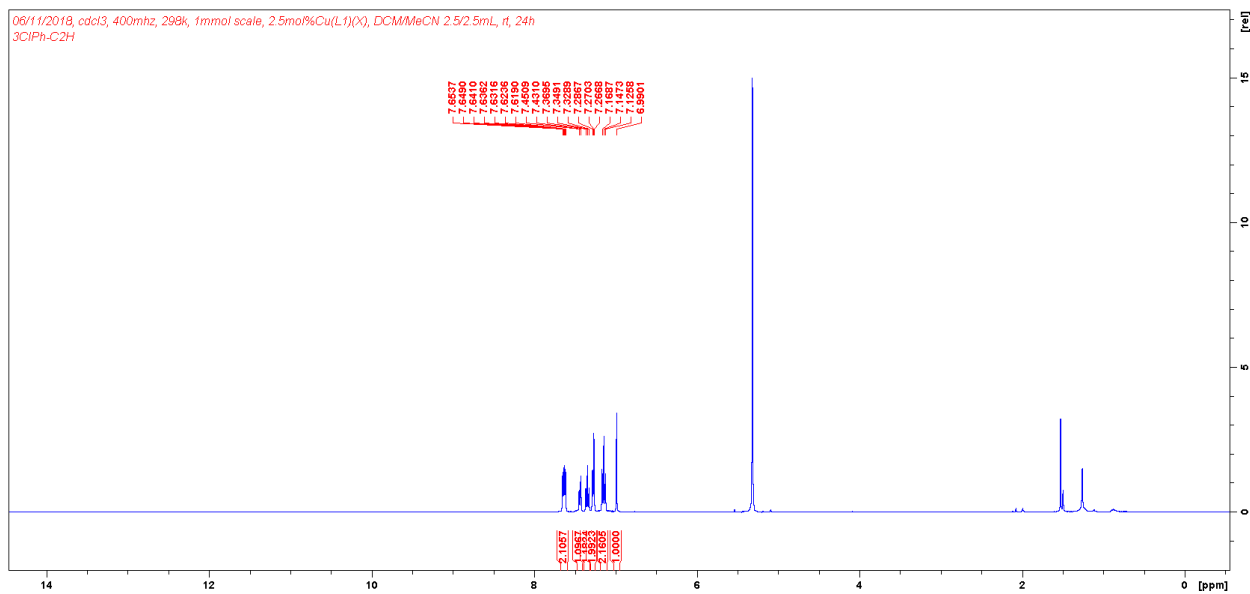
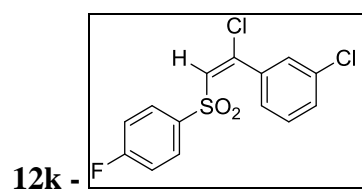


Figure S175. ^1H NMR spectrum of **12k**.

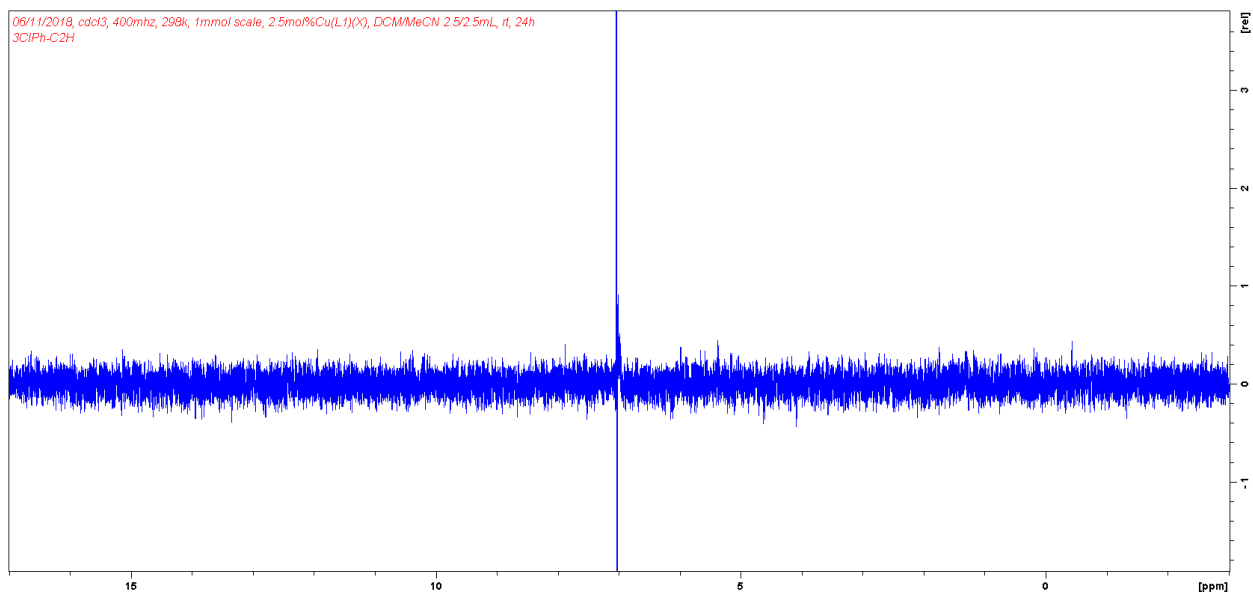


Figure S176. 1D NOESY spectrum of **12k**.

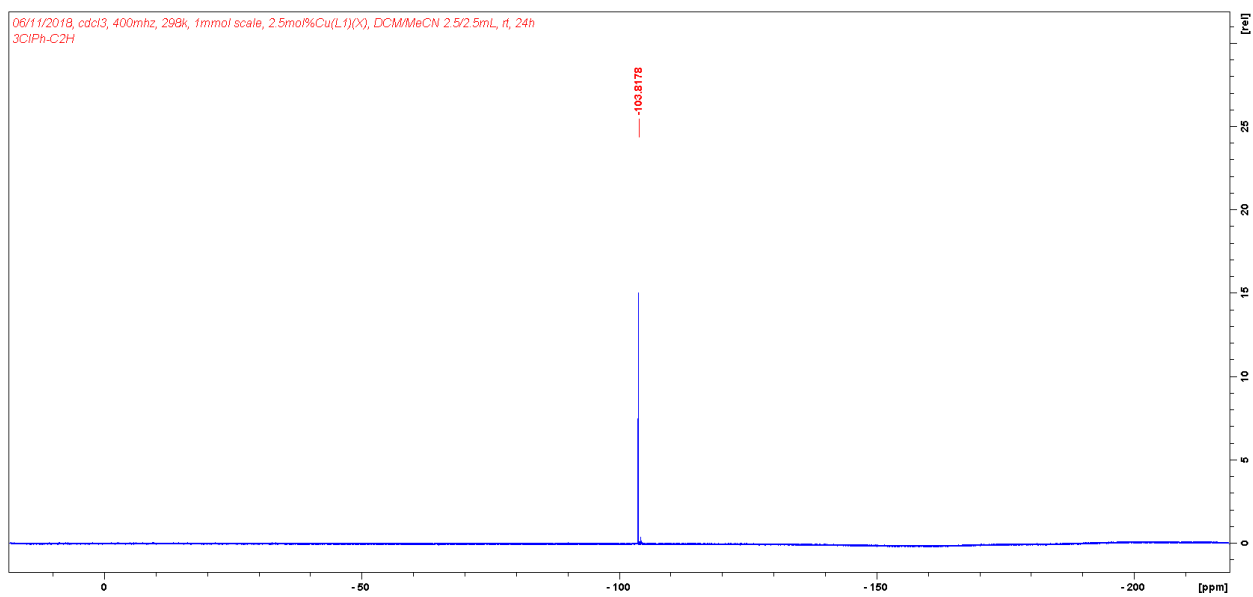


Figure S177. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **12k**.

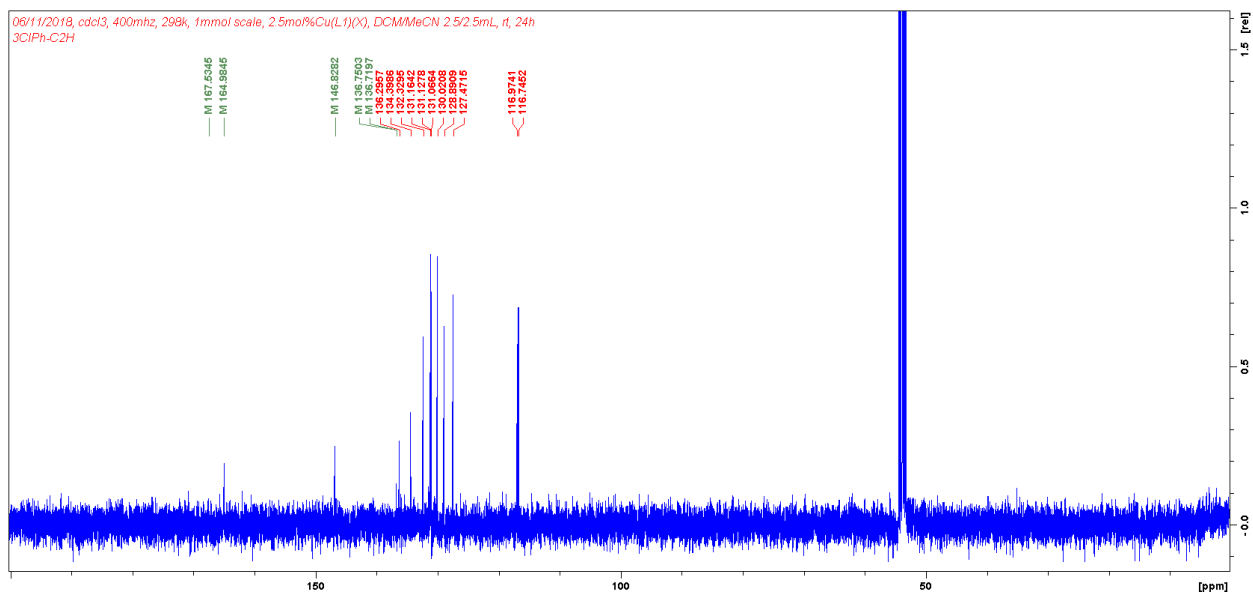


Figure S178. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **12k**.

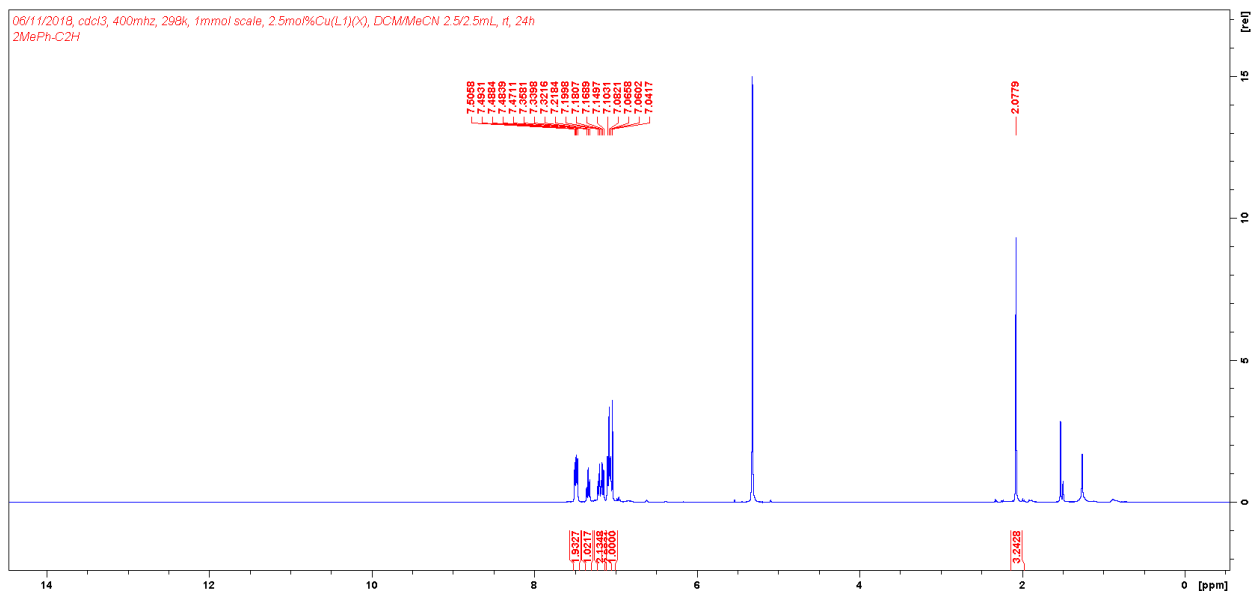
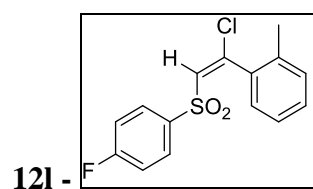


Figure S179. ^1H NMR spectrum of **12l**.

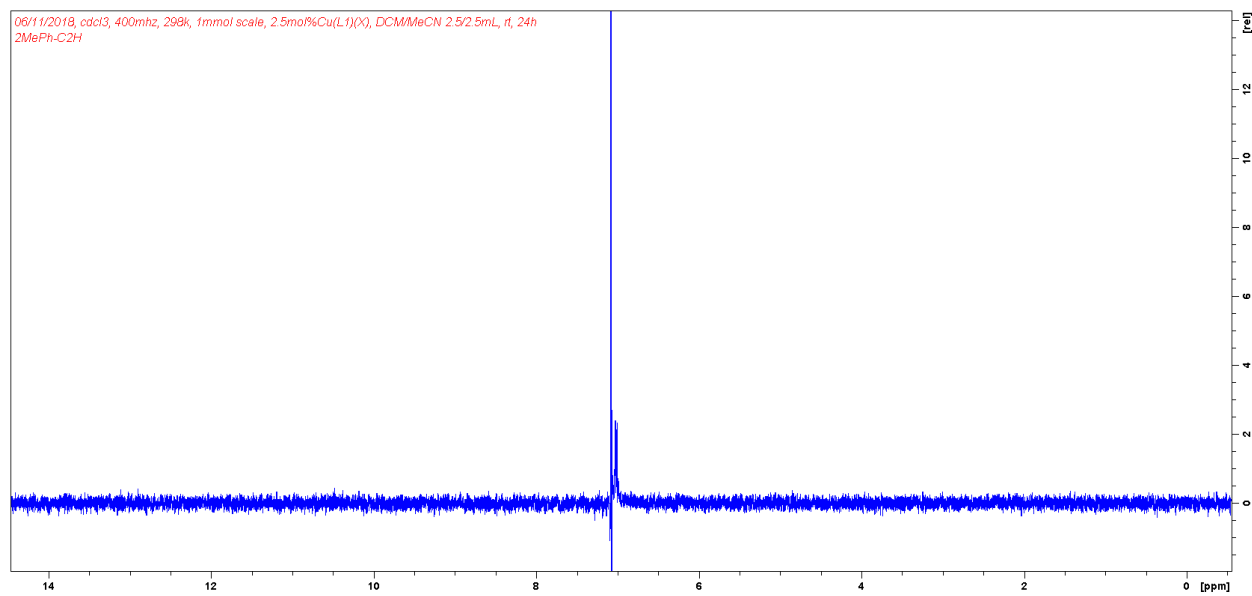


Figure S180. 1D NOESY spectrum of **12l**.

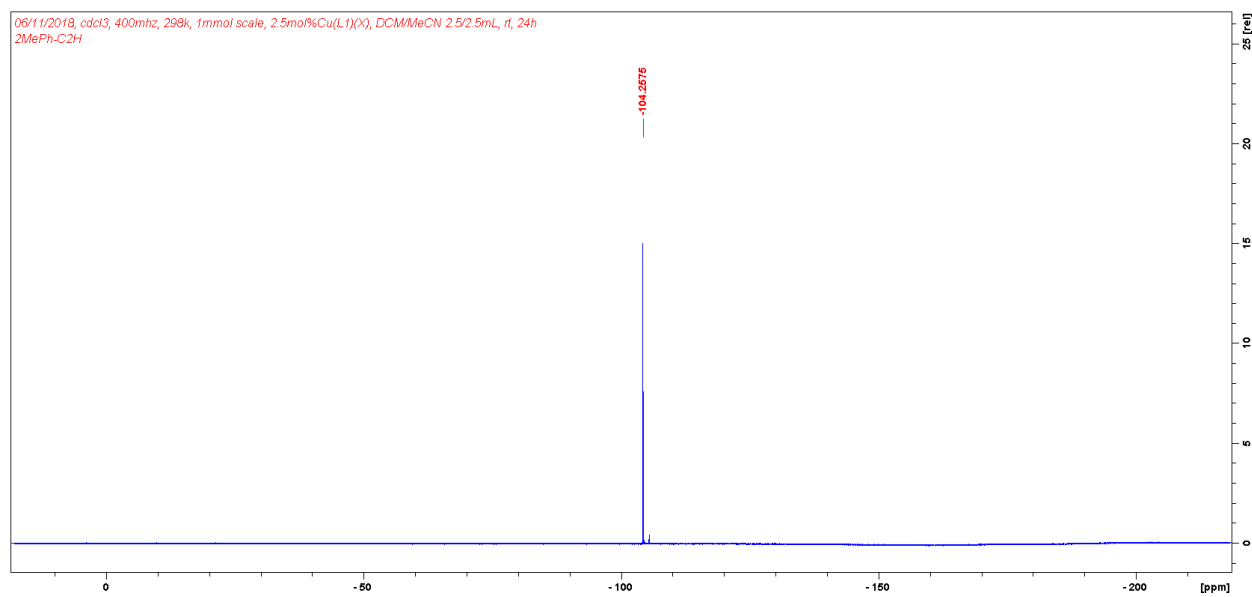


Figure S181. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **12l**.

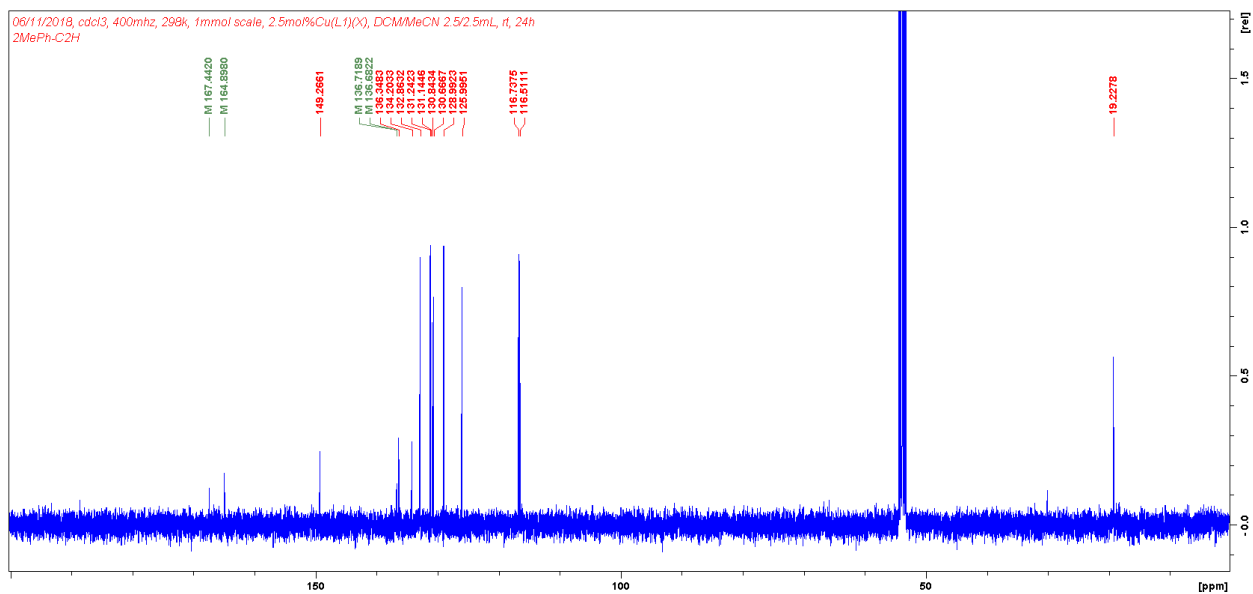


Figure S182. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **12l**.

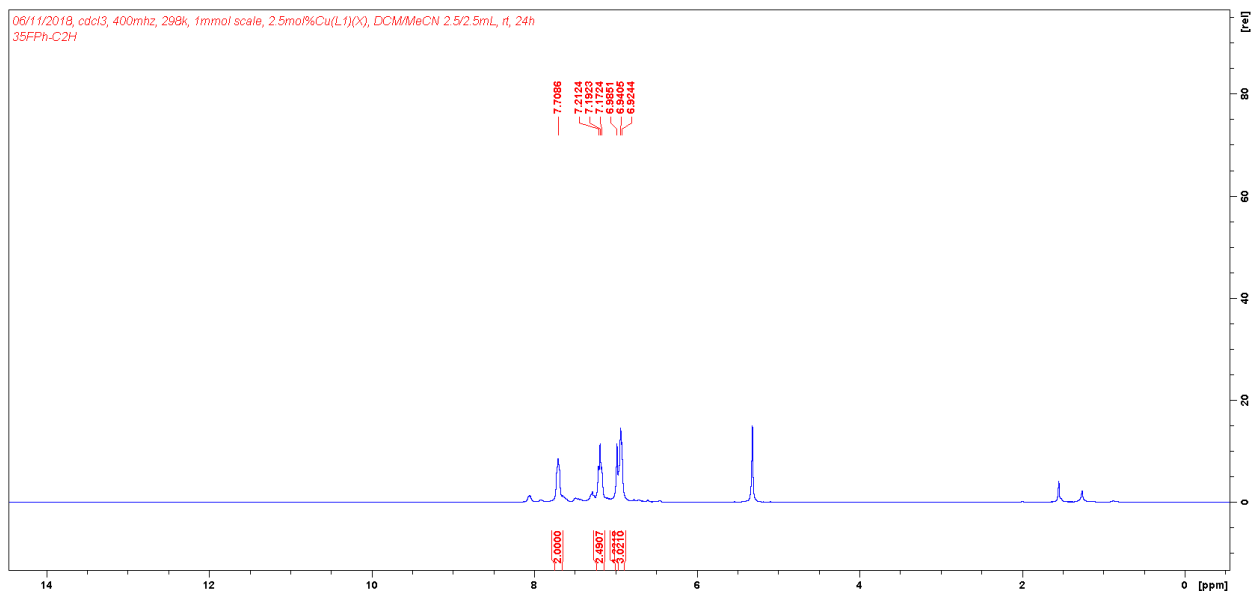
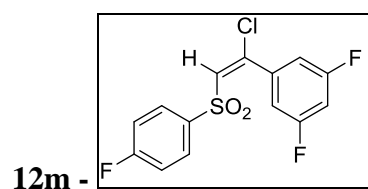


Figure S183. ^1H NMR spectrum of **12m**.

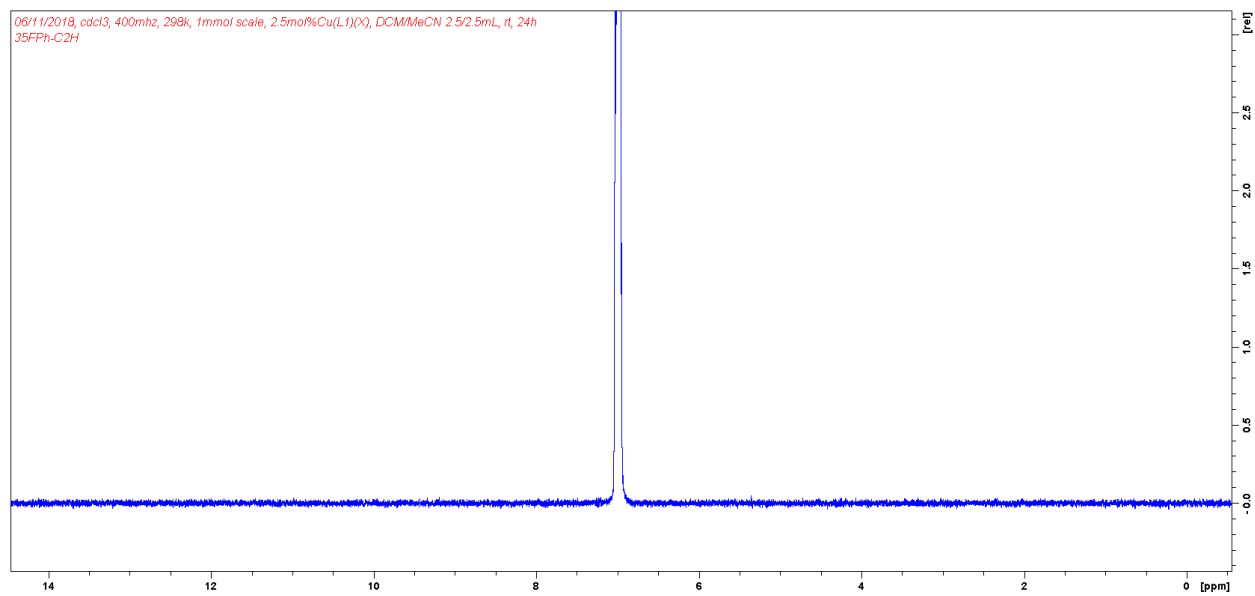


Figure S184. 1D NOESY spectrum of **12m**.

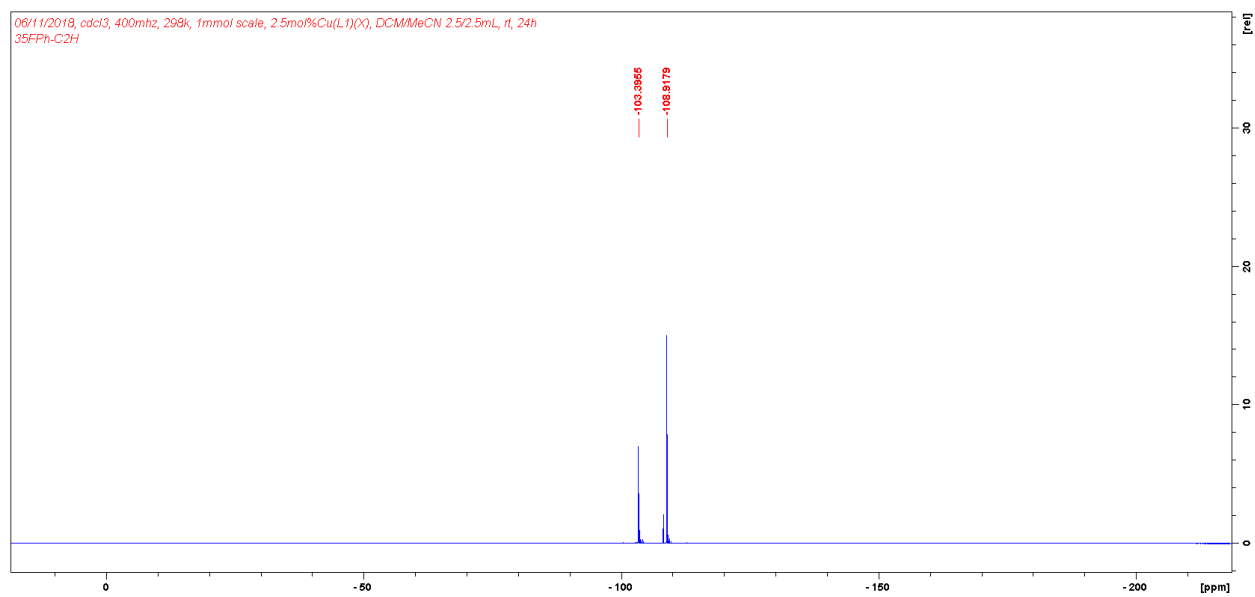


Figure S185. $^{19}\text{F}\{^1\text{H}\}$ NMR spectrum of **12m**.

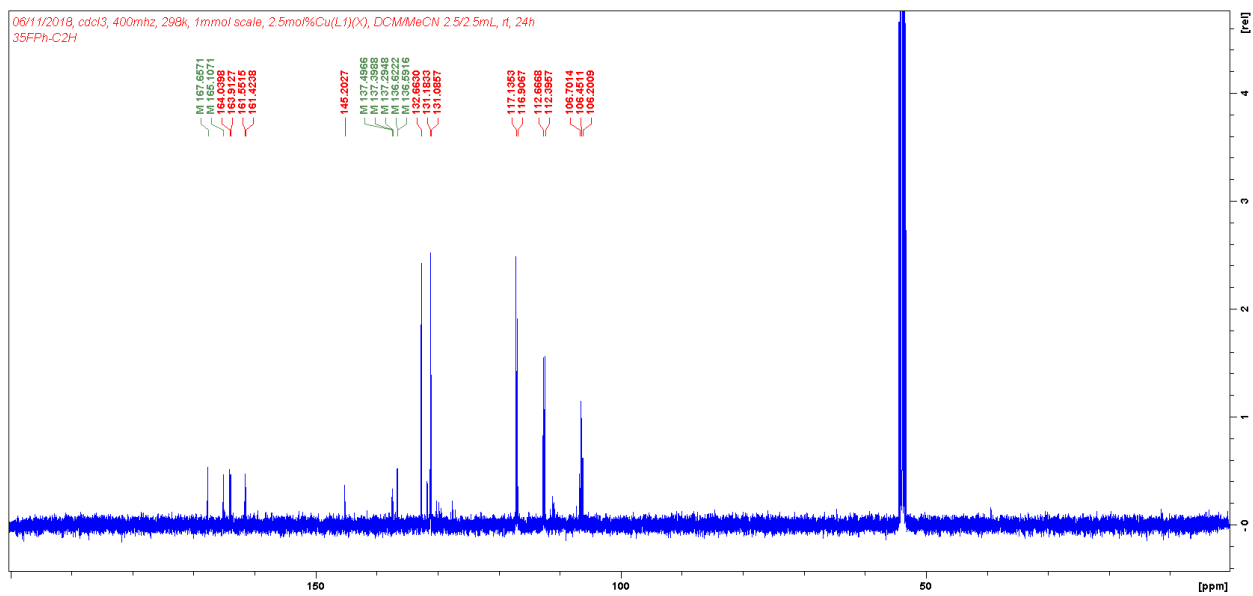


Figure S186. $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **12m**.