Supporting Information

for

High-Throughput Evaluation of *In Situ*-generated Cobalt (III) Catalysts for Acyl Fluoride Synthesis

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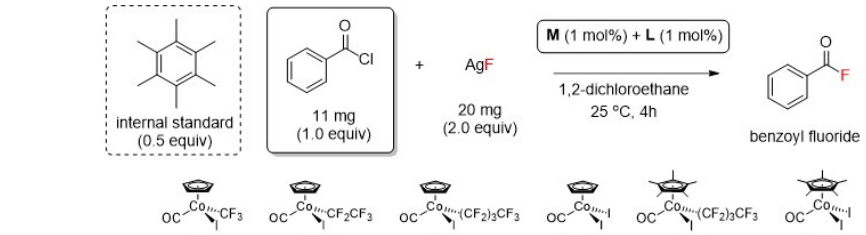
Table of Contents

I. Supplementary High-Throughput Experiment Data



		M1	M2	M3	M4	M5	M6	
PPh_3	L1	62	53	46	81	43	69	59
PPh_2Me	L2	99	99	99	99	99	88	97
$PPhMe_2$	L3	85	69	56	99	99	99	85
$P(n\text{-}Pr)_3$	L4	81	56	59	89	79	99	77
$P(n\text{-}Bu)_3$	L5	86	55	58	99	96	99	82
$P(i\text{-}Bu)_3$	L6	77	77	69	99	68	76	78
$PCyp_3$	L7	84	86	84	83	99	99	89
PCy_3	L8	79	87	65	74	78	99	80
$P(t\text{-}Bu)_2H$	L9	80	77	78	81	54	85	76
$P(t\text{-}Bu)H_2$	L10	75	74	74	75	99	82	80
$P(C_6F_5)_3$	L11	63	53	55	69	99	49	65
$P(OMe)_3$	L12	67	70	73	53	82	99	74
$P(OEt)_3$	L13	70	71	68	52	76	66	67
$P(O\text{-}i\text{-}Pr)_3$	L14	65	42	58	74	89	48	63
$P(OPh)_3$	L15	86	74	75	74	51	82	74
none	L16	70	70	65	81	74	60	70
		77	70	68	80	80	81	*Average*

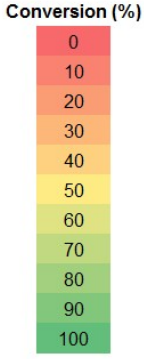
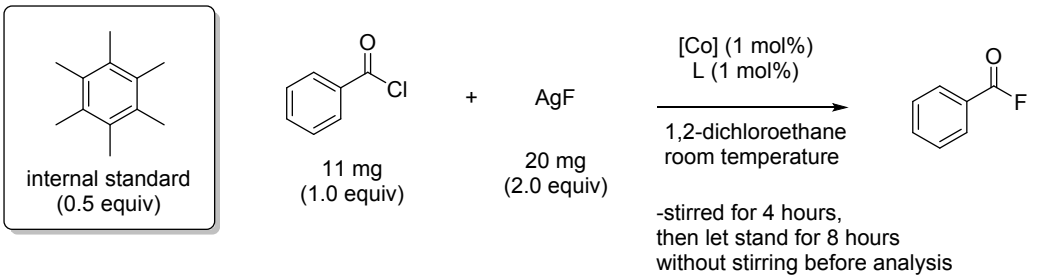


Figure S1. Conversion of **1a** during high-throughput experiment.



	N=1		N=2	
	(% conversion)	**(% Yield)**	**(% conversion)**	**(% Yield)**
CpCo(CF$_3$)(I)(PPh$_2$Me) ***(no ligand)***	**72.7**	**66.4**	**71.9**	**62.1**
CpCo(CF$_3$)(I)(CO) + PPh$_2$Me	**84.2**	**74.5**	**83.9**	**72.7**
CpCo(CF$_3$)(I)(CO) + PPh$_3$	**63.2**	**50.1**	**65.8**	**57.3**
Control (no catalyst or ligand)	**33.2**	**24.4**	**38.9**	**22.1**

Table S1. Results of test screen for validation of high-throughput experiment procedure.

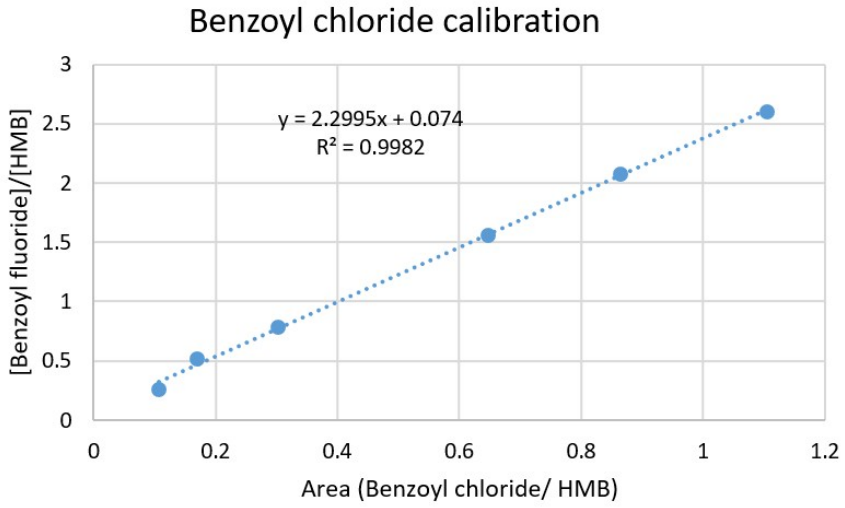


Figure S2. GC-FID calibration curve for **1a** and HMB.

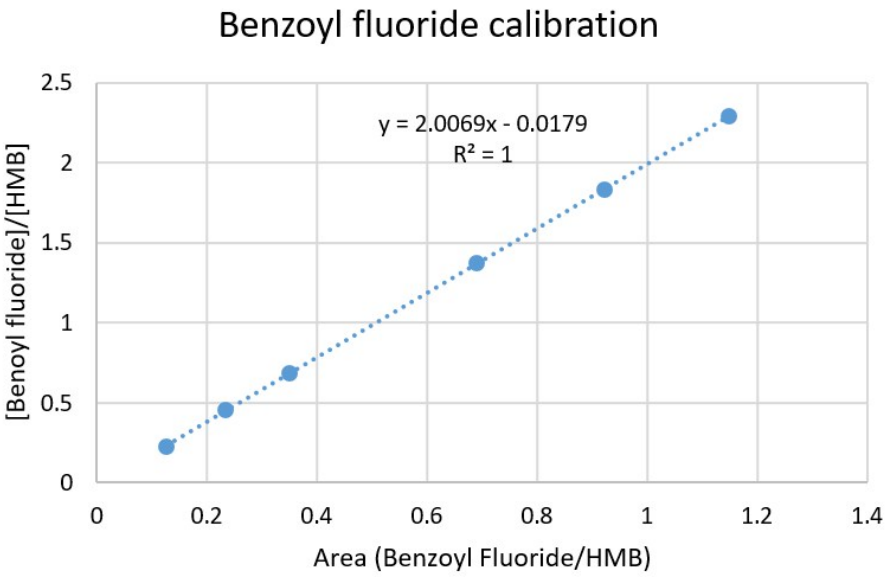
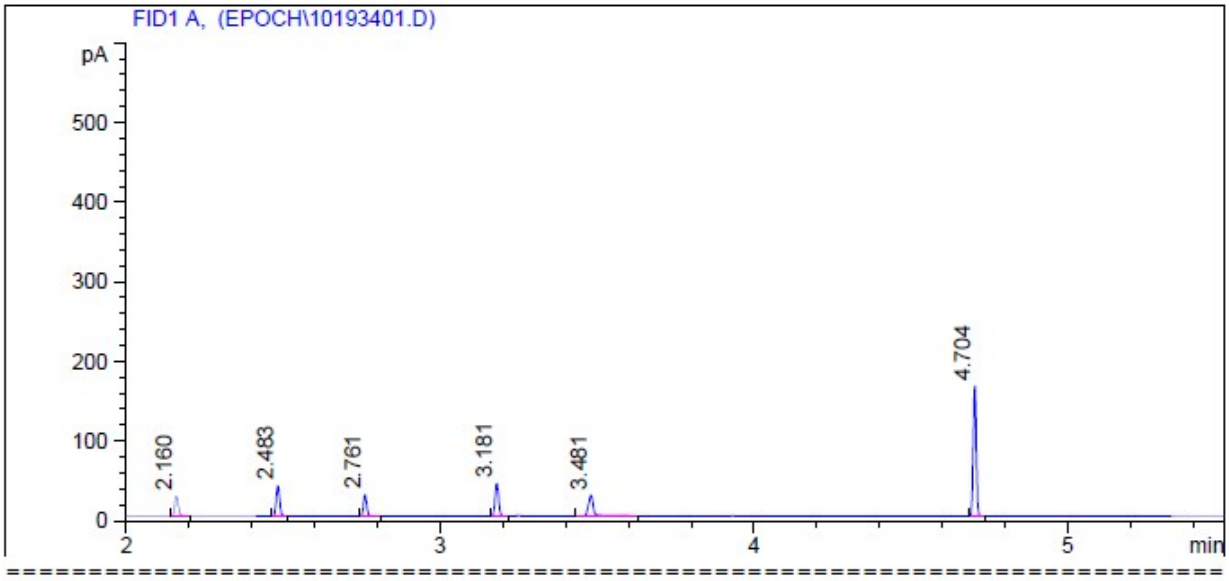


Figure S3. GC-FID calibration curve for **2a** and HMB.



Signal 1: FID1 A,

Peak #	RT [min]	Name	Area	Area %	Amount
1	2.160		20.516	15.358	0.00000
2	2.483	Benzoyl fluoride	29.667	22.208	0.00000
3	2.761		19.730	14.770	0.00000
4	3.181	Benzoyl chloride	32.971	24.681	0.00228
5	3.481		30.704	22.984	0.00000
6	0.000	Hexamethylbenzene_BF	0.000	0.000	0.00000
7	4.704	Hexamethylbenzene_BC	111.573	83.520	0.00315

Figure S4. Example of GC-FID chromatogram for high-throughput experiment.

II. NMR/IR Spectra of Isolated Compounds

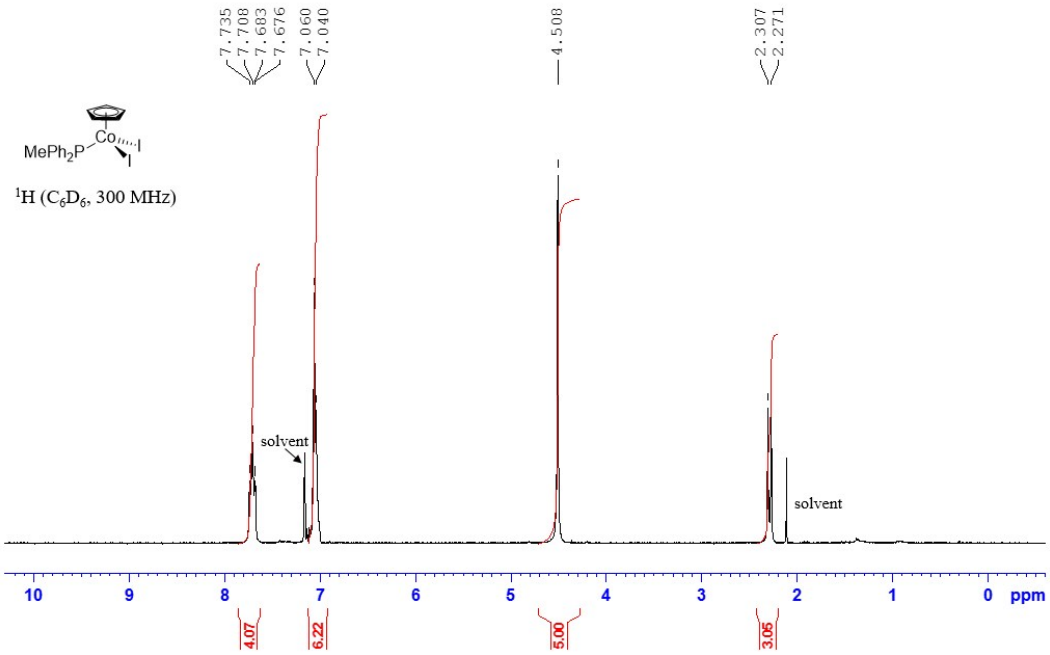


Figure S5. ^{1}H NMR spectrum of $CpCo(I)_2(PPh_2Me)$.

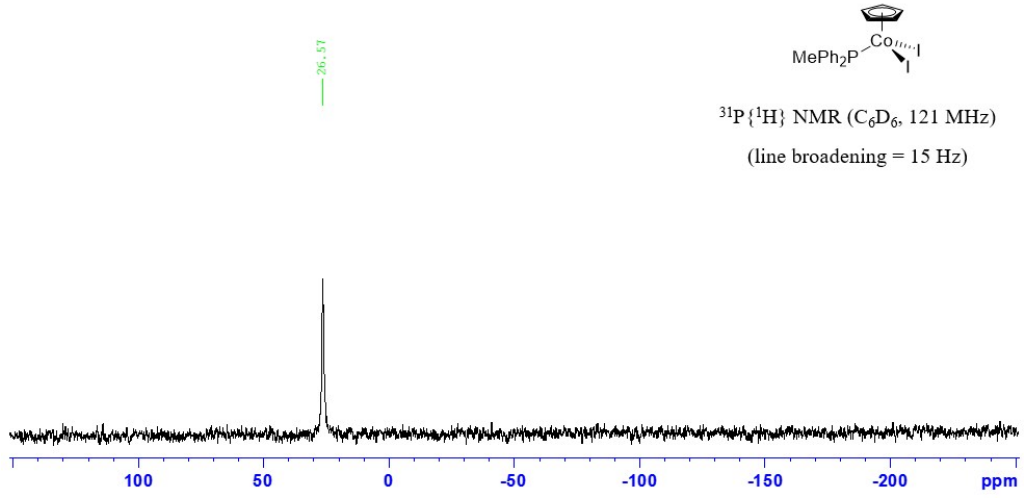


Figure S6. $^{31}P\{^{1}H\}$ NMR spectrum of $CpCo(I)_2(PPh_2Me)$.

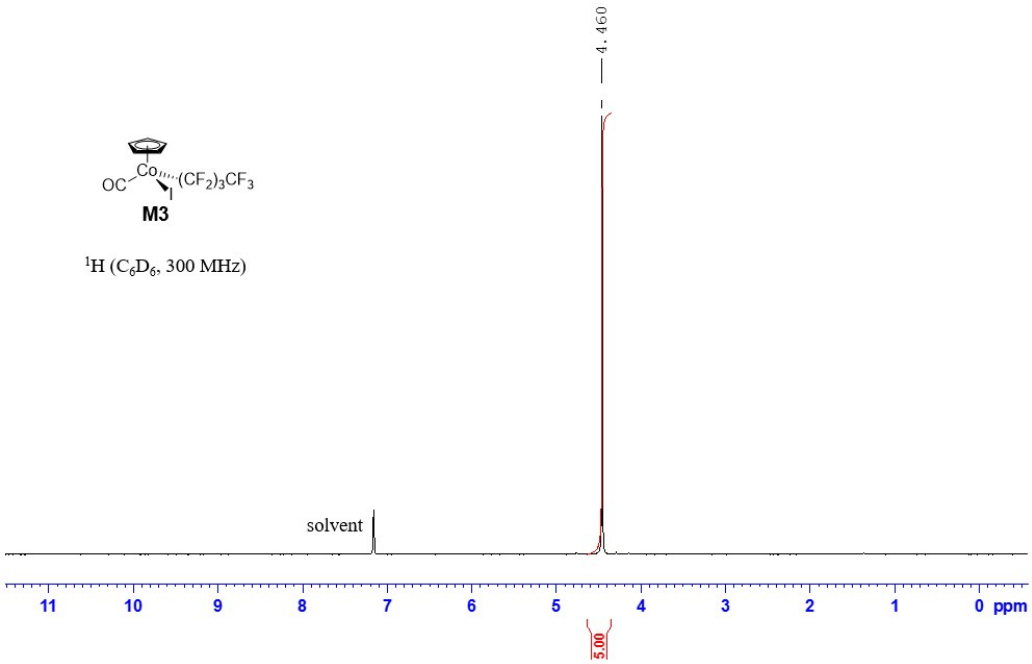


Figure S7. ^{1}H NMR spectrum of CpCo(I)(CO)($CF_2CF_2CF_2CF_3$) (**M3**).

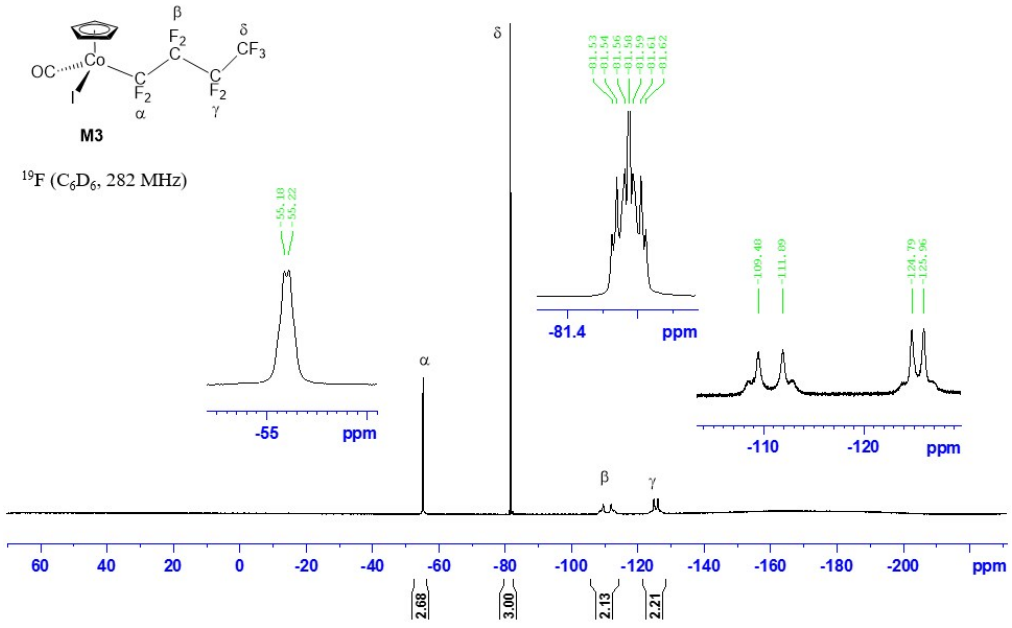


Figure S8. ^{19}F NMR spectrum of CpCo(I)(CO)($CF_2CF_2CF_2CF_3$) (**M3**).

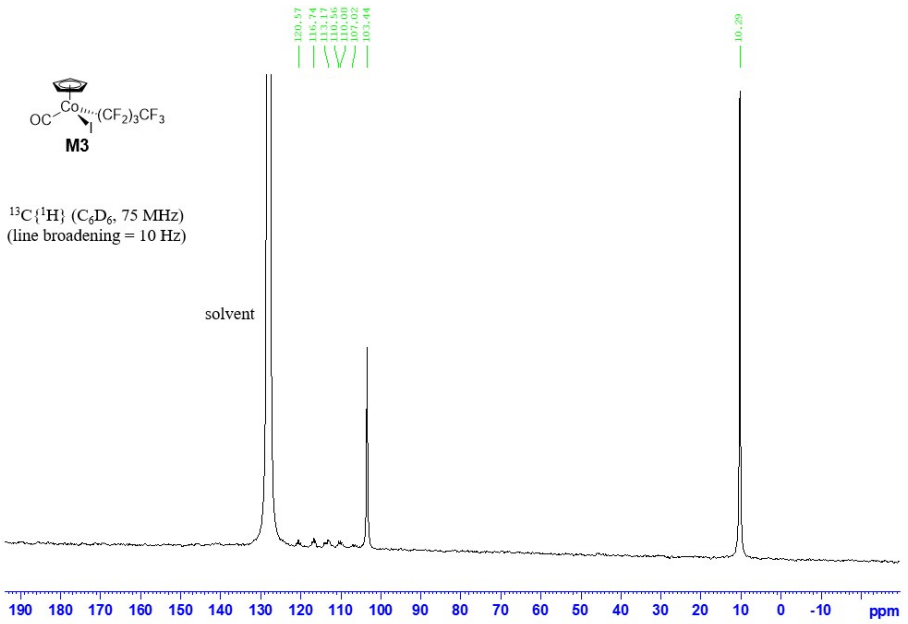


Figure S9. $^{13}C\{^{1}H\}$ NMR spectrum of CpCo(I)(CO)($CF_2CF_2CF_2CF_3$) (**M3**).

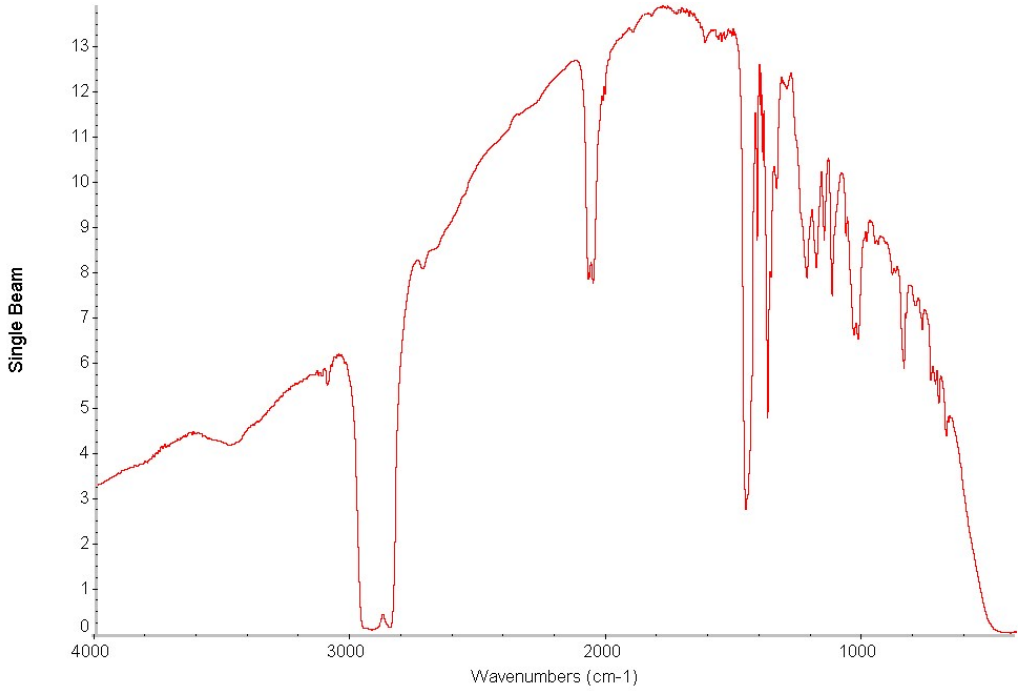


Fig S10. IR spectrum of CpCo(I)(CO)($CF_2CF_2CF_2CF_3$) (**M3**).

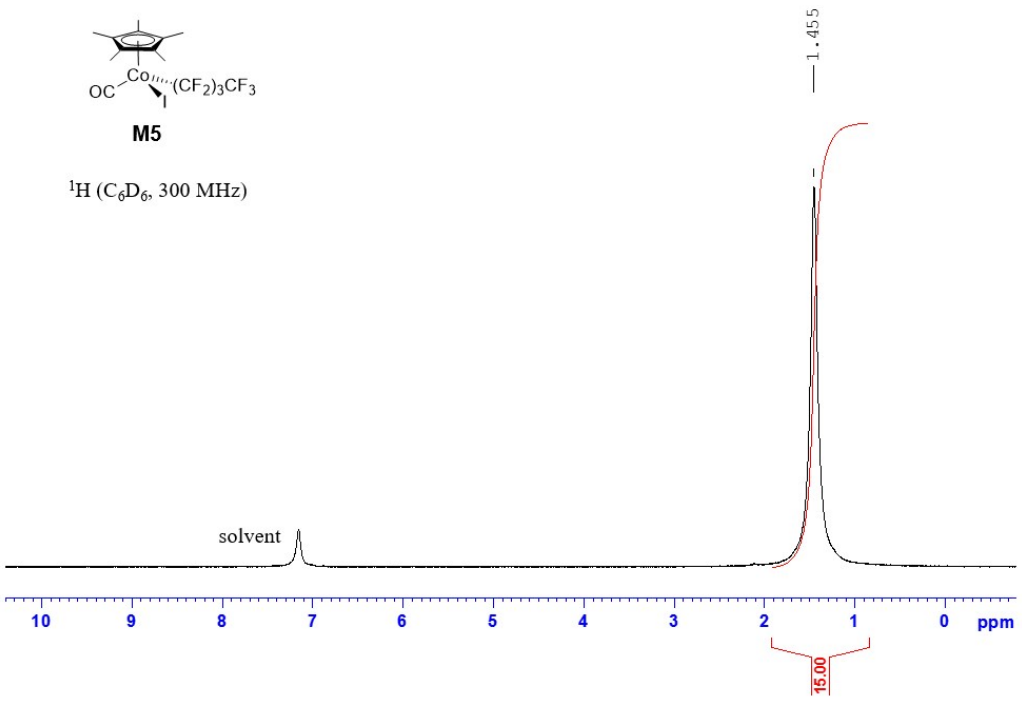


Figure S11. ^{1}H NMR spectrum of Cp*Co(I)(CO)($CF_2CF_2CF_2CF_3$) (**M5**).

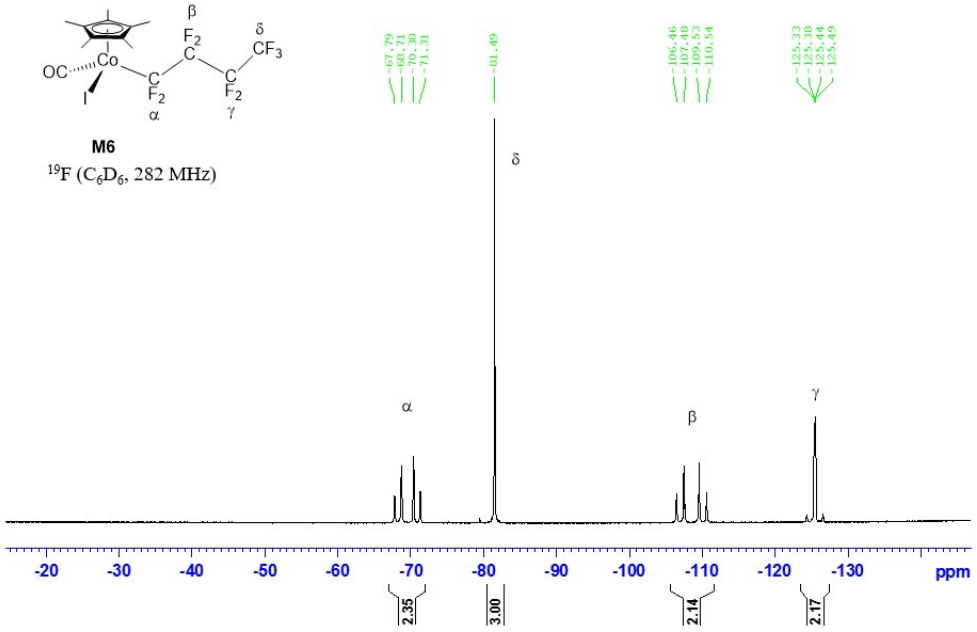


Figure S12. ^{19}F NMR spectrum of Cp*Co(I)(CO)($CF_2CF_2CF_2CF_3$) (**M5**).

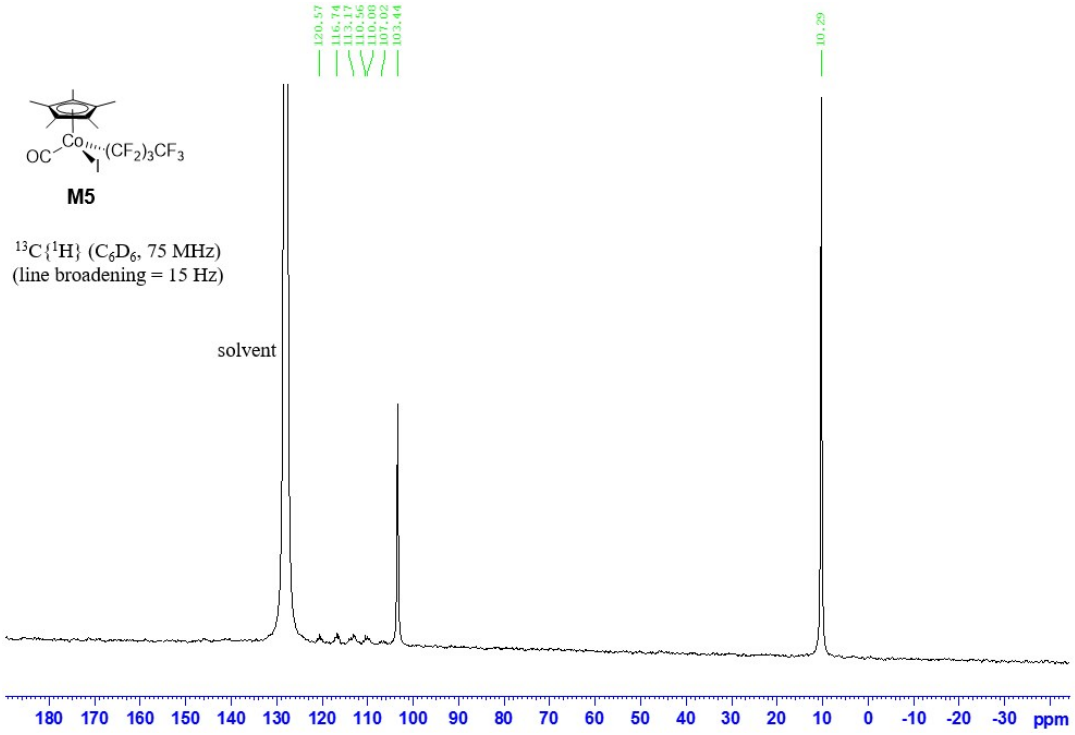


Figure S13. $^{13}C\{^{1}H\}$ NMR spectrum of Cp*Co(I)(CO)($CF_2CF_2CF_2CF_3$) (**M5**).

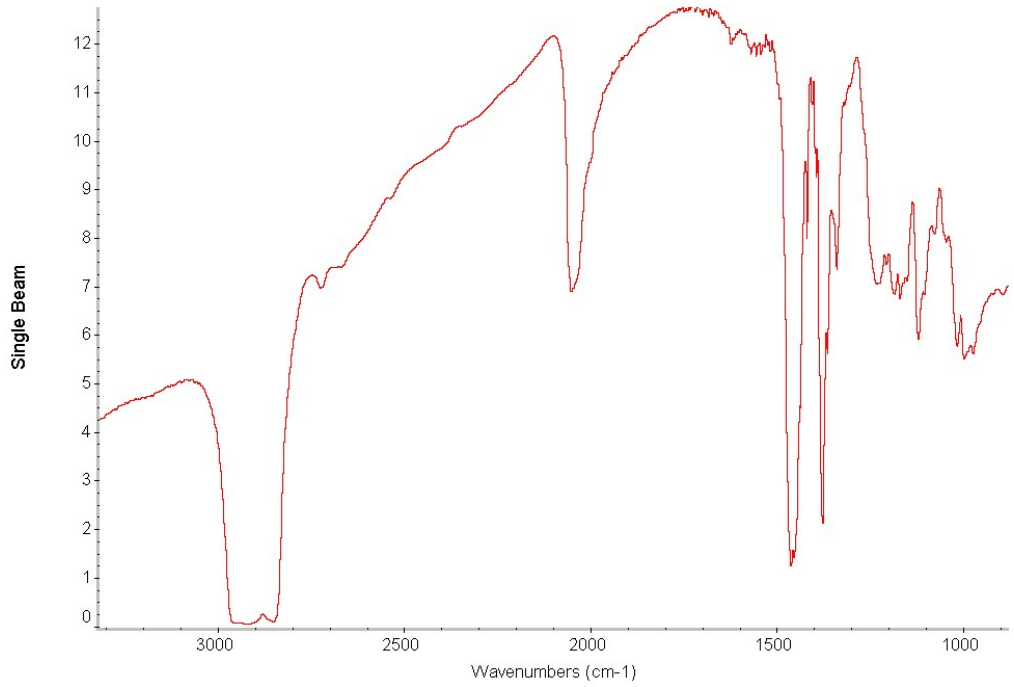


Fig S14. IR spectrum of Cp*Co(I)(CO)($CF_2CF_2CF_2CF_3$) (**M5**).

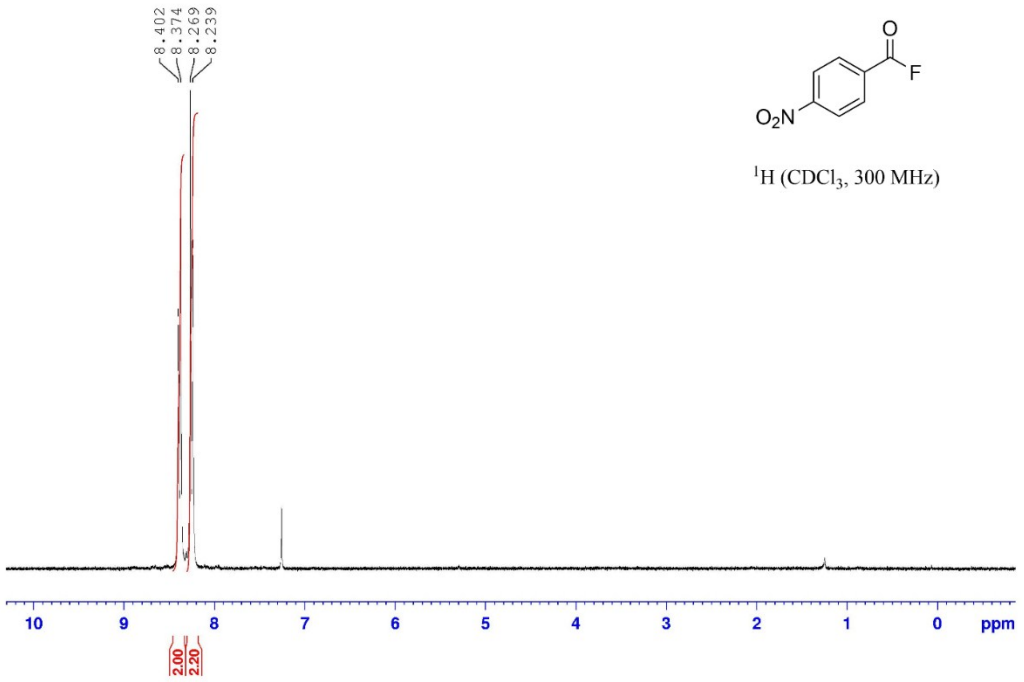


Figure S15. ^{1}H NMR spectrum of 4-nitrobenzoyl fluoride (**2b**).

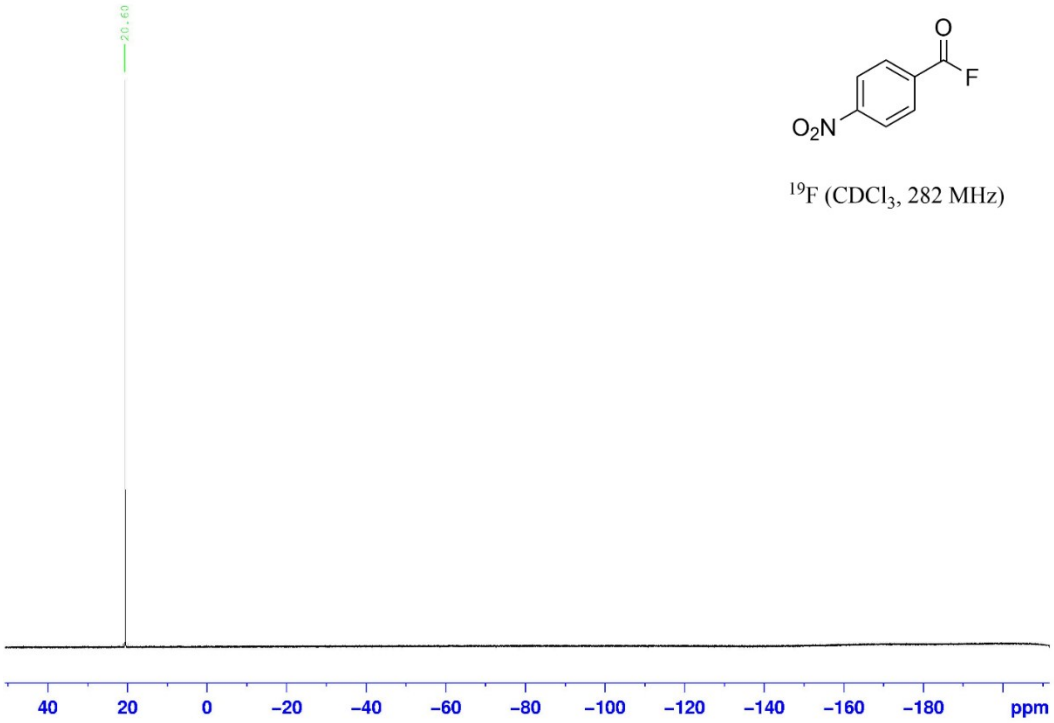


Figure S16. ^{19}F NMR spectrum of 4-nitrobenzoyl fluoride (**2b**).

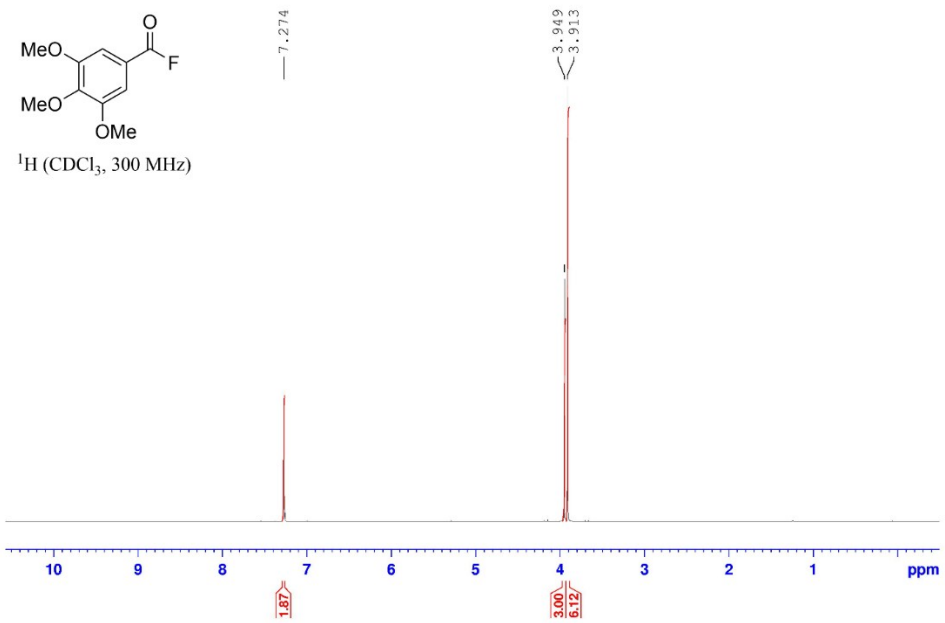


Figure S17. ^{1}H NMR spectrum of 3,4,5-tris(methoxy)benzoyl fluoride (**2k**).

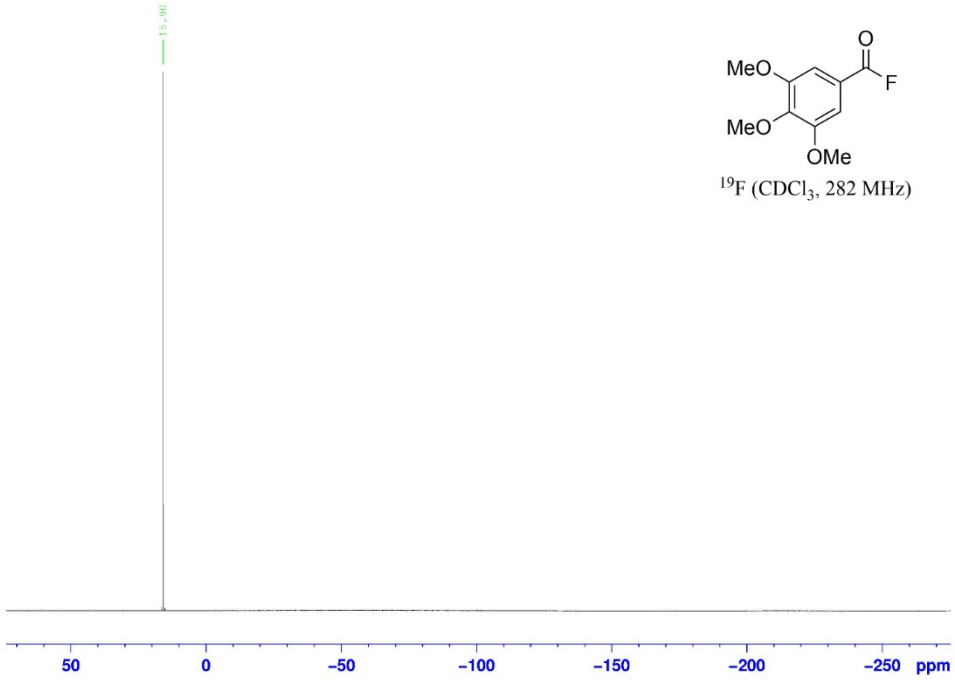


Figure S18. ^{19}F NMR spectrum of 3,4,5-tris(methoxy)benzoyl fluoride (**2k**).

III. Crude ^{19}F NMR Spectra of Acyl Fluorides

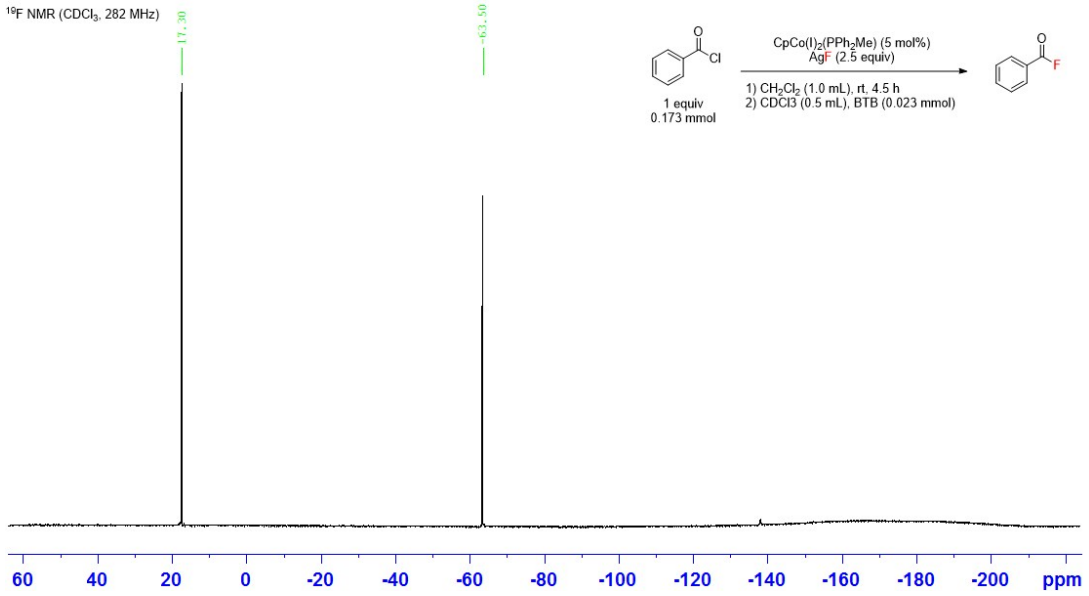


Figure S19. Crude ^{19}F NMR spectrum of **2a**.

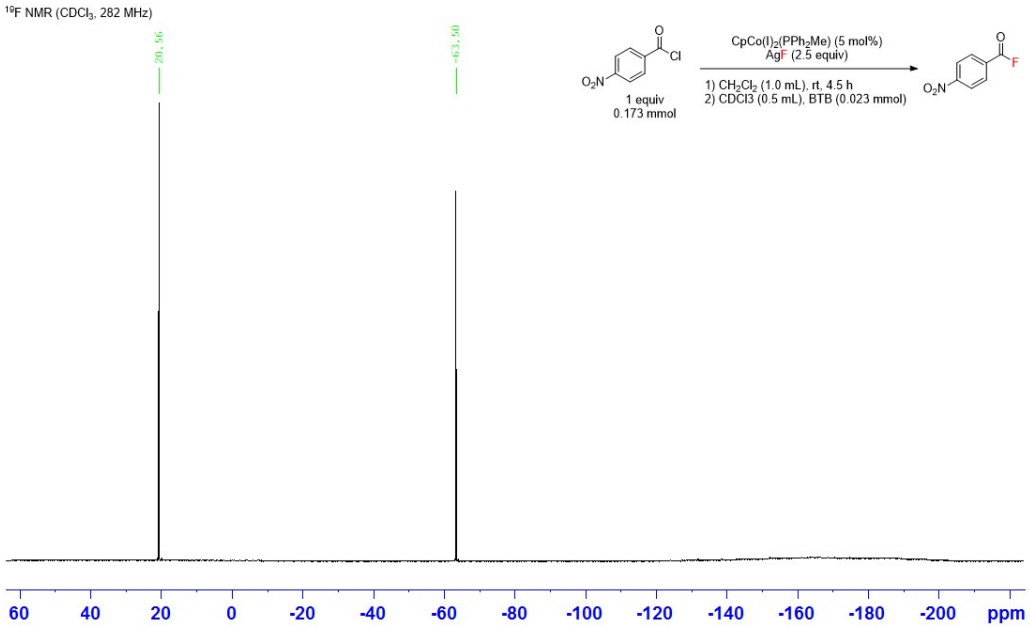


Figure S20. Crude ^{19}F NMR spectrum of **2b**.

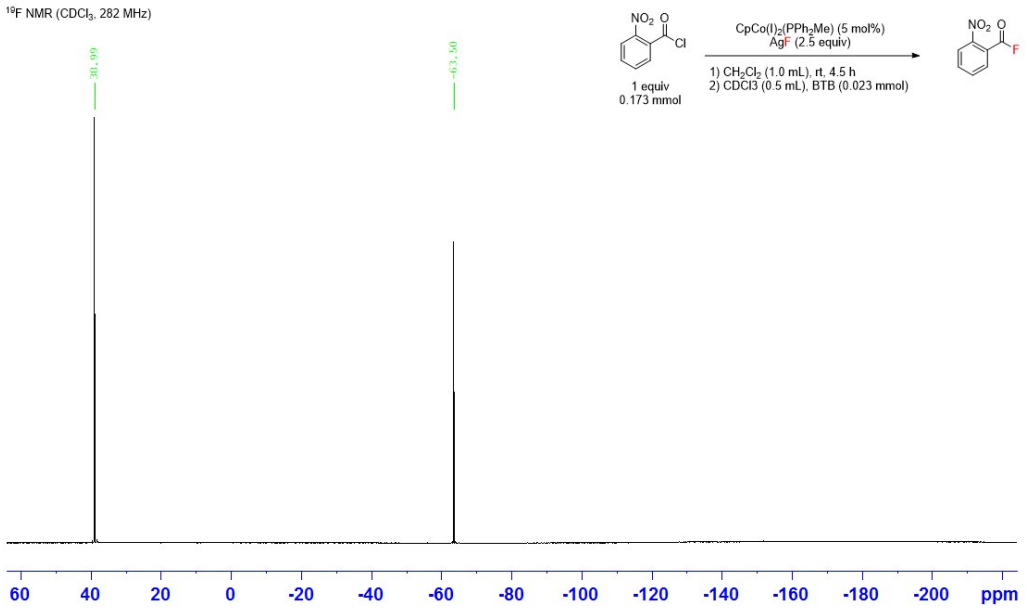


Figure S21. Crude ^{19}F NMR spectrum of **2c**.

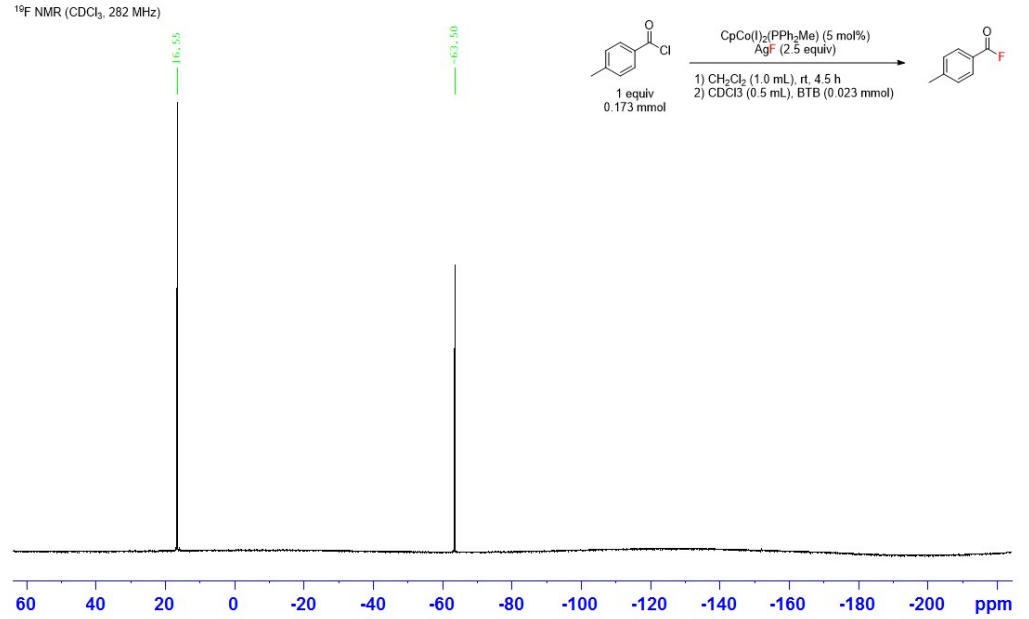


Figure S22. Crude ^{19}F NMR spectrum of **2d**.

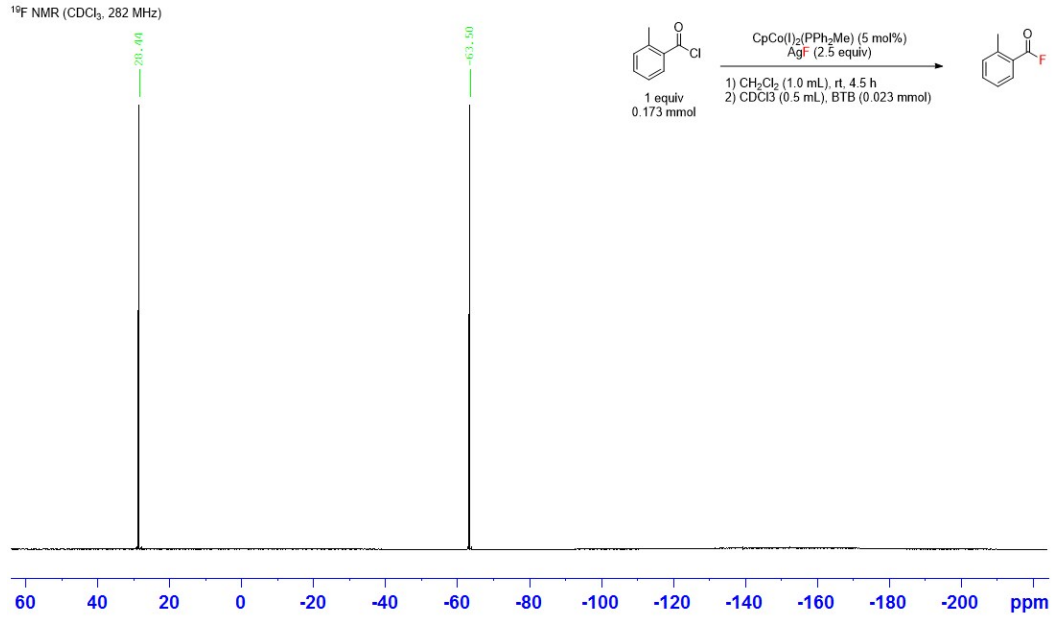


Figure S23. Crude ^{19}F NMR spectrum of **2e**.

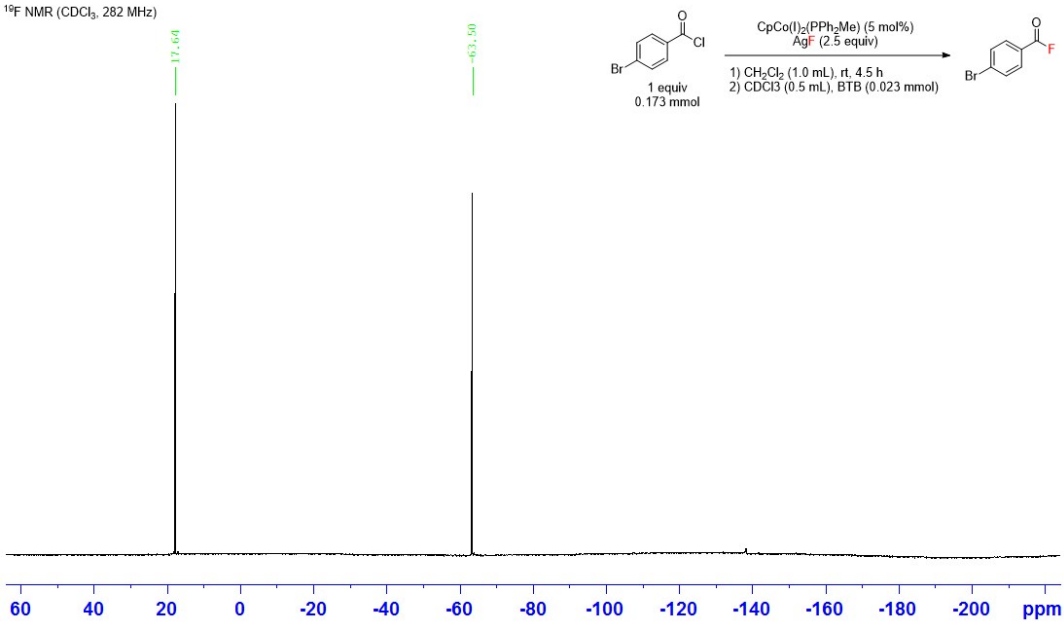


Figure S24. Crude ^{19}F NMR spectrum of **2f**.

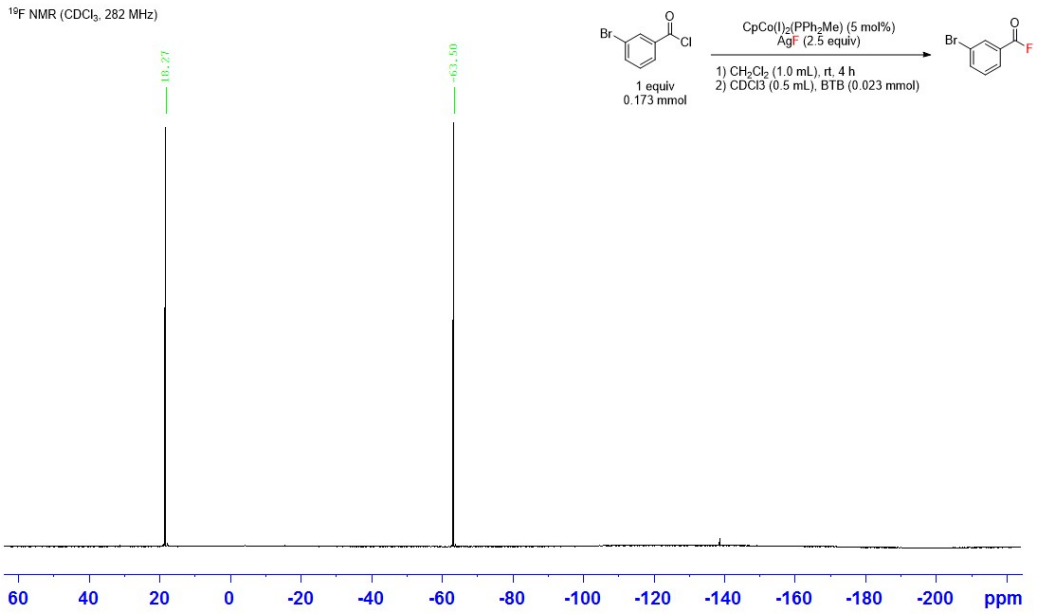


Figure S25. Crude ^{19}F NMR spectrum of **2g**.

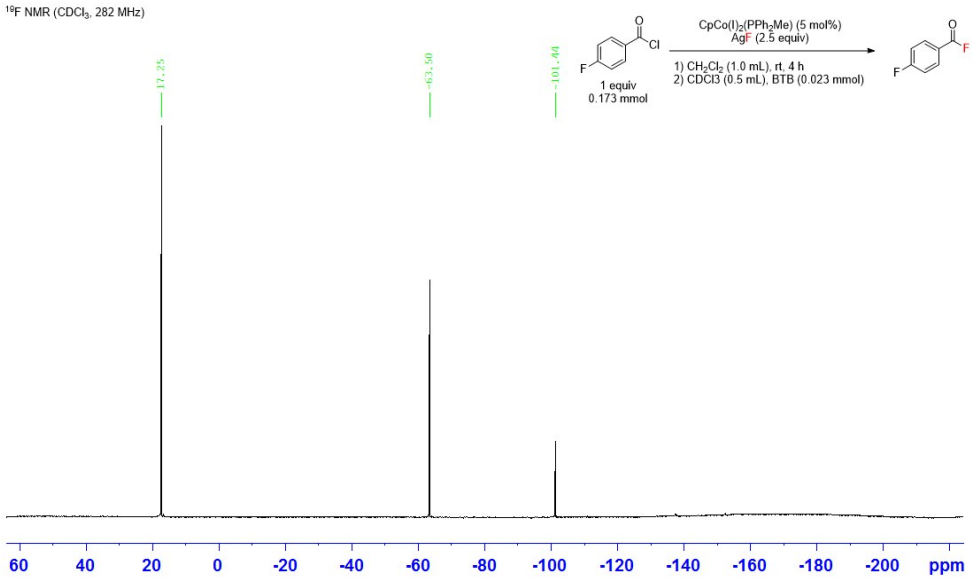


Figure S29. Crude ^{19}F NMR spectrum of **2h**.

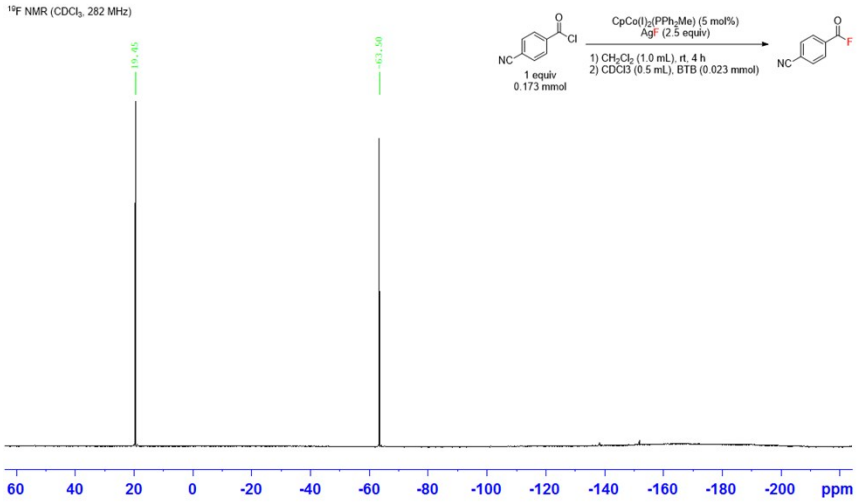


Figure S30. Crude ^{19}F NMR spectrum of **2i**.

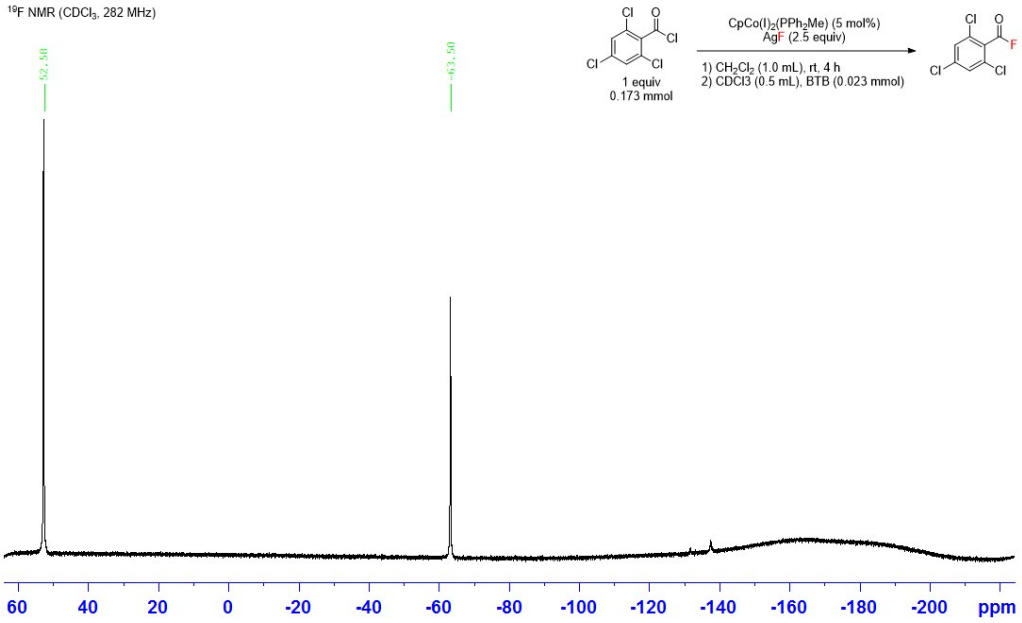


Figure S26. Crude ^{19}F NMR spectrum of **2j**.

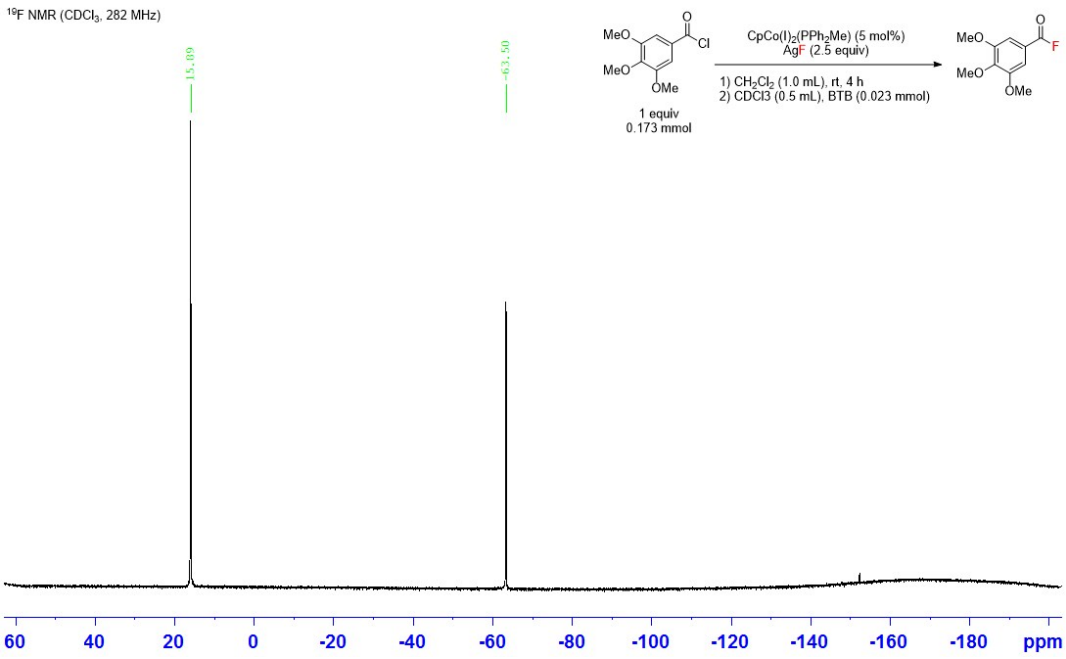


Figure S31. Crude ^{19}F NMR spectrum of **2k**.

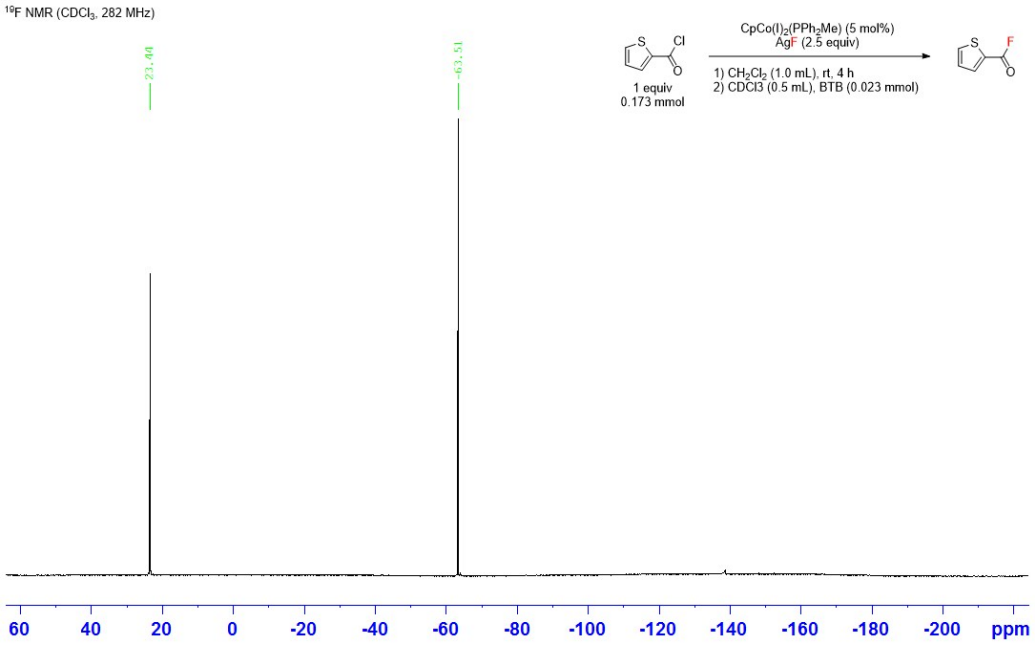


Figure S27. Crude ^{19}F NMR spectrum of **2l**.

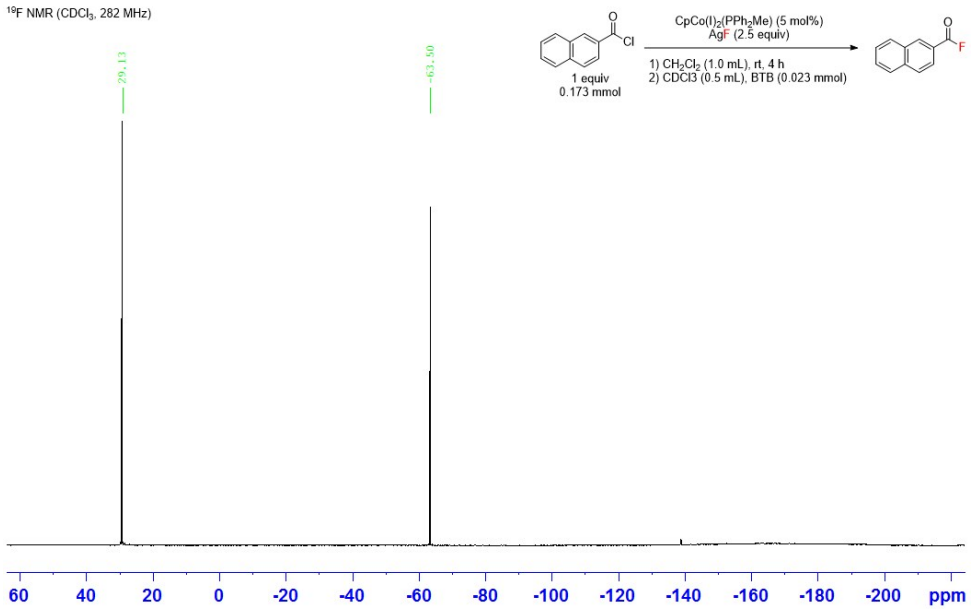


Figure S28. Crude ^{19}F NMR spectrum of **2m**.

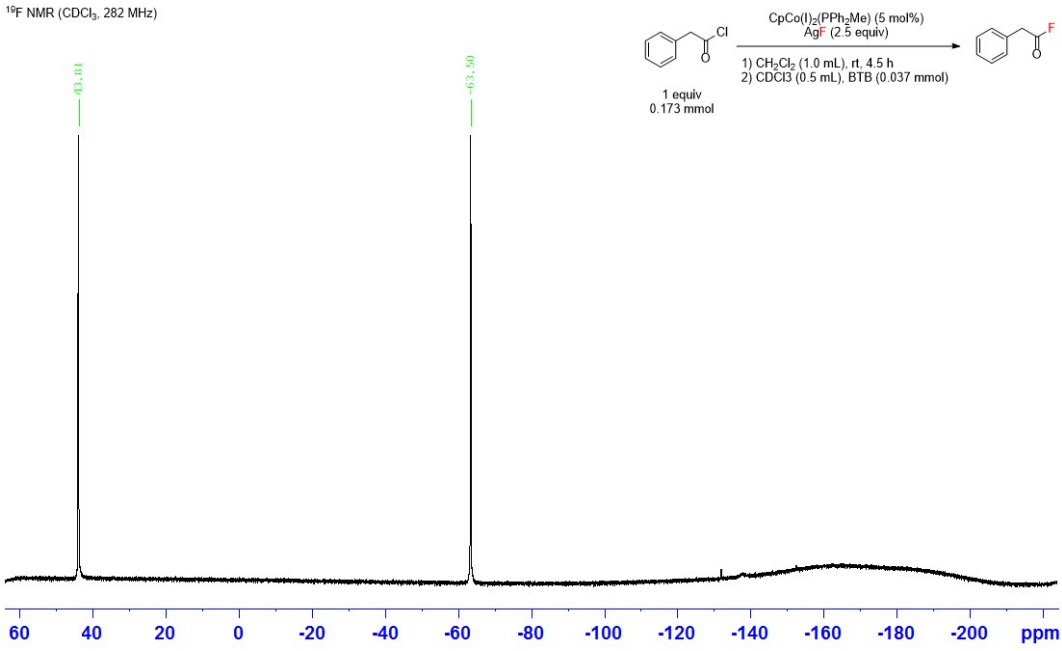


Figure S32. Crude ^{19}F NMR spectrum of **2n**.