

Supporting Information for:

Covalent Functionalization of Carbon Nanomaterials with Iodonium Salts

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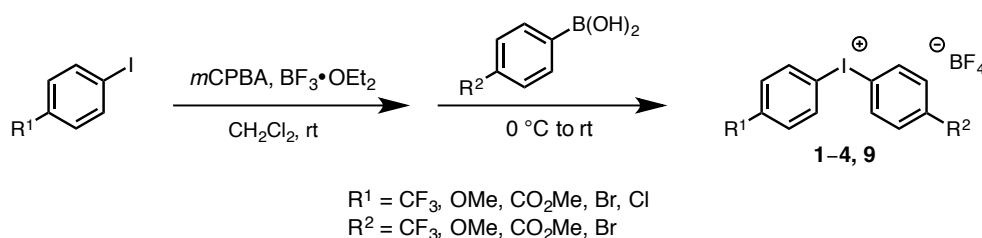
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1. General method

All reactions were carried out in dry glassware. Reagent grade CH_2Cl_2 was used for reactions. Anhydrous, inhibitor-free THF was dried over 4Å molecular sieve. Unless otherwise noted, *m*-Chlorobenzoic acid (*m*-CPBA) was purified by washing the commercial 77% material with phosphate buffer pH 7.5 and dried under reduced pressure. SWCNT used in this work was purified SWCNTs from Nano-C (diameter: 0.9–1.3 nm), DWCNTs was from Aldrich (outer diameter: 5 nm, inner diameter: 1.3–2.0 nm, length: 50 μm), MWCNTs was from Aldrich (outer diameter: 6–13 nm, length: 2.5–20 μm) and graphite powder was from Alfa Aesar (crystalline, 325 mesh). All other commercially reagents were used as received. Reactions were monitored by thin-layer chromatography (JT Baker silica gel IB-F plates), visualized by fluorescence quenching under UV light. Column chromatography was performed using silica gel (60 Å pore size, 230–400 mesh, Aldrich). ^1H , ^{11}B , ^{13}C and ^{19}F NMR spectra were recorded on Bruker-ADVANCE 400 spectrometer at 400 MHz, 101 MHz, 128 MHz and 376 MHz respectively. ^1H and ^{13}C NMR chemical shifts are reported as δ in ppm relative to the residual solvent peaks. ^1H NMR and ^{13}C NMR coupling constants (J) are reported in Hertz (Hz), and multiplicities are indicated as follow: s (singlet), d (doublet), t (triplet), q (quartet), sept (septet), dd (doublet of doublets), ddd (doublet of doublet of doublets), m (multiplet). High resolution mass spectra were determined with a Bruker Daltonics APEXIV 4.7 Tesla FT-ICR-MS using ESI ionization.

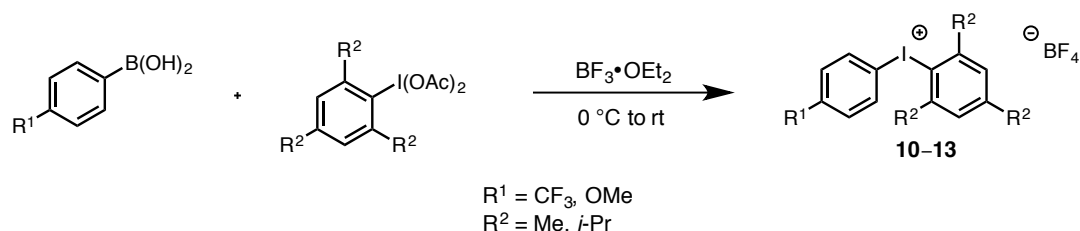
2. Synthesis and characterization of iodonium salts

General procedure A:



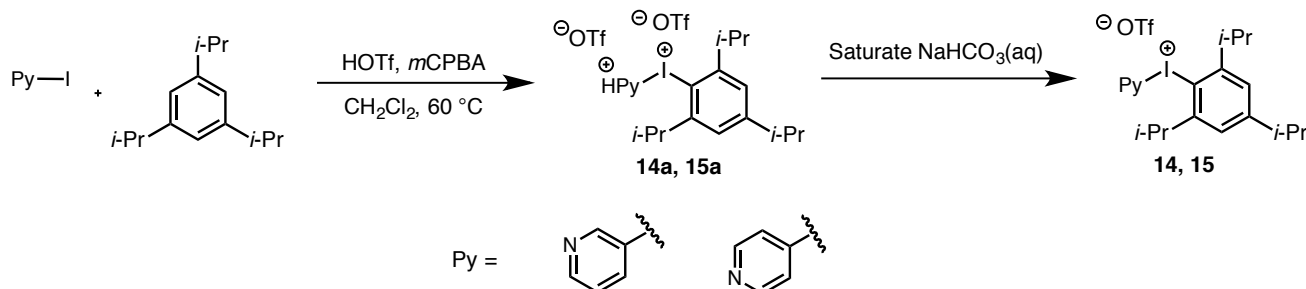
General procedure A was adapted from reference 1.¹ To a solution of iodoarene (1.00 equiv) in CH_2Cl_2 (0.30 M), *m*-CPBA (1.10 equiv) and $\text{BF}_3 \cdot \text{OEt}_2$ (2.50 equiv) was added sequentially and the yellow mixture was stirred at rt for 30 min. The reaction was cooled to 0 °C and the boronic acid (1.10 equiv) was added. After the addition of boronic acid, the cooling bath was removed and the reaction was stirred at rt for 15 min before directly loading over a silica gel column. Purification by column chromatography (100% CH_2Cl_2 to remove unreacted starting materials followed by CH_2Cl_2 :MeOH 20:1 to elute the iodonium salt) provided **1–4, 9** as a white or off-white solid in 24–91 % yield.

General procedure B:



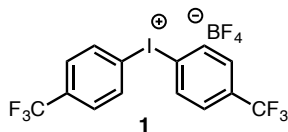
General procedure B was adapted from reference 2.² To a solution of the boronic acid (1.00 equiv) in CH_2Cl_2 (0.17 M), $\text{BF}_3 \cdot \text{OEt}_2$ (1.50 equiv) was added at 0 °C and stirred for 10 min. A solution of diacetoxiodoarene (1.30–1.50 equiv) in CH_2Cl_2 (0.79 M) was added at 0 °C and stirred for 1.5 h. Saturated aqueous NaBF_4 (3 mL/mmol boronic acid) solution was added and stirred at rt for 30 min. The reaction mixture was extracted with CH_2Cl_2 (2x) and the organic layer was dried over MgSO_4 and concentrated under reduced pressure. Precipitation by dissolving the crude in minimum amount of CH_2Cl_2 followed by addition of cold Et_2O afforded pure iodonium salt **10–13** as a white solid in 55–87% yield after filtration and drying under vacuum.

General procedure C:

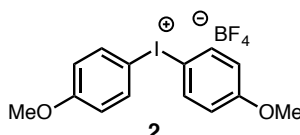


General procedure C was adapted from reference 3³ with modification. Triflic acid (4 equiv) was added slowly to a solution of iodopyridine (1.0 equiv) in CH_2Cl_2 (0.24 M) at 0 °C over 5 min in a seal tube. After 15 min of stirring, *m*-CPBA (1.50 equiv) and 1,3,5-triisopropylbenzene (1.10 equiv) was added sequentially at rt. The reaction mixture was heated to 60 °C for 1h 45 min. After cooling to rt, the reaction mixture was directly loaded over a column packed with silica gel in 100% CH_2Cl_2 . Purification by column chromatography (100% CH_2Cl_2 to elute unreacted starting materials and nonpolar impurities then CH_2Cl_2 :MeOH 20:1 to elute the product) afforded a brown solid. The brown solid was dissolved in minimum CH_2Cl_2 and Et_2O was added until a gel was formed. The gel was collected by filtration and dry under vacuum to obtain a white solid in 43–84% yield as the iodonium bistriflate products **14a** and **15a**. The conversions of **14a** and **15a** to **14** and **15** were obtained by extraction with CH_2Cl_2 :saturated aqueous NaHCO_3 3:1 (100 mL/ g of salt **14a** or **15a**). The organic layer was dried

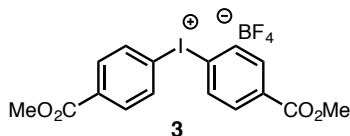
over MgSO_4 and concentrated under reduced pressure to afford product **14** and **15** a white solid in 80–98% yield.



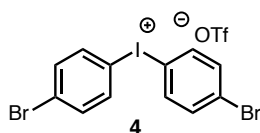
Iodonium salt 1. General procedure A was followed using 4-iodobenzotrifluoride (2.00 g, 7.35 mmol) and 4-(trifluoromethyl)phenyl boronic acid (1.54 g, 8.09 mmol). The product was obtained as a fluffy white solid in 63% yield. The ^1H , ^{19}F and ^{13}C NMR spectra were identical to those reported previously in the literature.⁴



Iodonium salt 2. General procedure A was followed using 4-iodoanisole (500 mg, 2.14 mmol) and 4-methoxybenzene boronic acid (357 mg, 2.35 mmol). The product was obtained as a beige solid in 24% yield. The ^1H , ^{19}F and ^{13}C NMR spectra were identical to those reported previously in the literature.¹

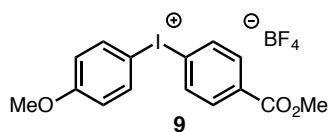


Iodonium salt 3. General procedure A was followed using methyl-4-iodobenzoate (833 mg, 3.18 mmol) and 4-(methoxycarbonyl)benzeneboronic acid (630 mg, 3.50 mmol). The product was obtained as a white solid in 60% yield. The ^1H , ^{19}F and ^{13}C NMR spectra were identical to those reported previously in the literature.⁴



Iodonium salt 4. Synthetic procedure was adapted from reference 5.⁵ A solution of bromobenzene (55 mg, 0.35 mmol) in CH_2Cl_2 (0.5 mL) was added to a stirred solution of 1-bromo-4-iodobenzene (100 mg, 0.353 mmol) and *m*-CPBA (77% active oxidant, 0.389 mmol) in CH_2Cl_2 (1.5 mL) at rt. The reaction mixture was cooled to 0 °C and triflic acid (94 μL , 1.1 mmol) was added. After stirring for 1 h, the reaction mixture was concentrated under reduced pressure. The crude was dissolved in minimum

amount of CH₂Cl₂ and precipitated by addition of cold Et₂O. Pure iodonium salt **4** was obtained as a white solid (115 mg, 55%) after filtration and drying under vacuum. The ¹H, ¹⁹F and ¹³C NMR spectra were identical to those reported previously in the literature.⁶



Iodonium salt 9. General method A was followed using methyl-4-iodobenzoate (500 mg, 1.91 mmol) and 4-methoxybenzeneboronic acid (319 mg, 2.10 mmol). The product was obtained as a beige solid in 91% yield.

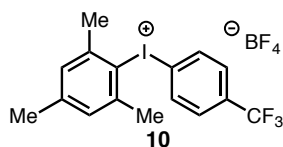
¹H NMR (400 MHz, Acetonitrile-*d*₃) δ 8.17 – 8.01 (m, 6H), 7.09 (d, *J* = 9.2 Hz, 2H), 3.89 (s, 3H), 3.85 (s, 3H).

¹⁹F NMR (376 MHz, Acetonitrile-*d*₃) δ –150.47.

¹¹B NMR (128 MHz, Acetonitrile-*d*₃) δ –1.52.

¹³C NMR (101 MHz, Acetonitrile-*d*₃) δ 166.6, 164.9, 139.5, 136.3, 135.4, 133.9, 119.7, 119.5, 102.4, 57.2, 53.8.

ESI-HRMS: calculated for C₁₅H₁₄IO₃⁺ [*M*]⁺: 368.9982, found: 368.9973



Iodonium salt 10. General method B was followed using 4-(trifluoromethyl)phenylboronic acid (100 mg, 0.526 mmol) and 2-(diacetoxyiodo)mesitylene (287 mg, 0.789 mmol). The product was obtained as a white solid in 55% yield.

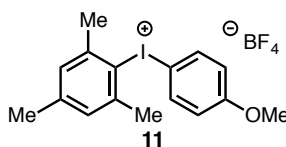
¹H NMR (400 MHz, Acetonitrile-*d*₃) δ 8.01 (d, *J* = 8.7 Hz, 2H), 7.80 (d, *J* = 8.5 Hz, 2H), 7.28 (s, 2H), 2.62 (s, 6H), 2.38 (s, 3H).

¹⁹F NMR (376 MHz, Acetonitrile-*d*₃) δ –63.83, –150.59.

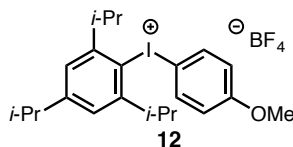
¹¹B NMR (128 MHz, Acetonitrile-*d*₃) δ –1.54.

¹³C NMR (101 MHz, Acetonitrile-*d*₃) δ 146.96, 144.40, 136.17, 134.86 (q, *J* = 33.1 Hz), 132.06, 130.48 (q, *J* = 3.8 Hz), 124.79 (q, *J* = 272.3 Hz), 121.56, 116.64 (q, *J* = 1.6 Hz), 27.76, 21.60.

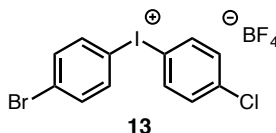
ESI-HRMS: calculated for C₁₆H₁₅F₃I⁺ [*M*]⁺: 391.0165, found: 391.0179



Iodonium salt 11. General method B was followed using (4-methoxyphenyl)boronic acid (500 mg, 3.29 mmol) and 2-(diacetoxyiodo)mesitylene (1.58 g, 4.28 mmol). The product was obtained as a white solid in 87% yield. The ^1H , ^{19}F and ^{13}C NMR spectra were identical to those reported previously in the literature.⁷



Iodonium salt 12. General method B was followed using (4-methoxyphenyl)boronic acid (100 mg, 0.658 mmol) and 2-(diacetoxyiodo)-1,3,5-triisopropylbenzene (384 mg, 0.855 mmol). The product was obtained as a white solid in 51 % yield. The ^1H , ^{19}F and ^{13}C NMR spectra were identical to those reported previously in the literature.⁸



Iodonium salt 13. General procedure A was followed using 1-bromo-4-iodobenzene (100 mg, 0.353 mmol) and 4-chlorophenylboronic acid (61 mg, 0.39 mmol). The product was isolated as a white solid in 43% yield.

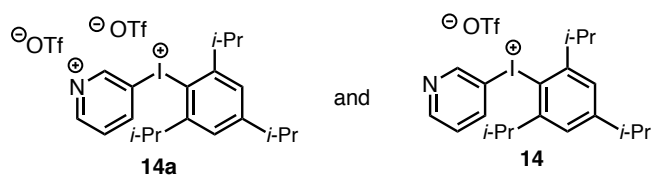
^1H NMR (400 MHz, Acetonitrile- d_3) δ 8.07 (d, J = 8.9 Hz, 2H), 7.99 (d, J = 8.8 Hz, 2H), 7.72 (d, J = 8.8 Hz, 2H), 7.57 (d, J = 8.8 Hz, 2H).

^{19}F NMR (376 MHz, Acetonitrile- d_3) δ -150.69.

^{11}B NMR (128 MHz, Acetonitrile- d_3) δ -1.54.

^{13}C NMR (101 MHz, Acetonitrile- d_3) δ 140.9, 138.5 (2 peaks, not resolved), 136.9, 134.0, 129.3, 112.9, 112.0.

ESI-HRMS: calculated for $\text{C}_{12}\text{H}_8\text{BrClI}^+$ $[M]^+$: 394.8521, found: 394.8510



Iodonium salt 14a and 14. General procedure C was followed using 3-iodopyridine (1.00 g, 4.88 mmol) and 1,3,5-triisopropylbenzene (1.10 g, 5.37 mmol). Iodonium bistriflate salt **14a** and iodonium triflate salt **14** were obtained as white solids in 84% yield (2.92 g) and 98% yield (772 mg) respectively. The ^1H , ^{19}F and ^{13}C NMR spectra of **14a** were identical to those reported previously in the literature.³ Using 2,2,2-trifluoroethanol as an internal standard, the ratio of triflate counter ion to the parent cation was calculated to be 1.9:1.0 for **14a** and 1.0:1.0 for **14**.

Iodonium salt 14a

^1H NMR (400 MHz, Methanol- d_4) δ 9.00 (d, J = 1.8 Hz, 1H), 8.82 (dd, J = 4.9, 1.3 Hz, 1H), 8.34 (ddd, J = 8.4, 2.2, 1.4 Hz, 1H), 7.68 – 7.59 (m, 1H), 7.37 (s, 2H), 3.41 (sept, J = 6.8 Hz, 2H), 3.01 (sept, J = 6.9 Hz, 1H), 1.29 (d, J = 6.7 Hz, 12H), 1.25 (d, J = 7.0 Hz, 6H).

^{19}F NMR (376 MHz, Methanol- d_4) δ –80.11.

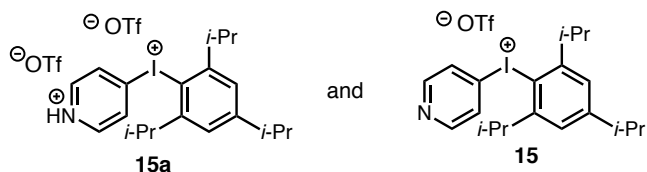
Iodonium salt 14

^1H NMR (400 MHz, Methanol- d_4) δ 8.95 (d, J = 2.3 Hz, 1H), 8.78 (dd, J = 4.8, 1.3 Hz, 1H), 8.31 (ddd, J = 8.3, 2.4, 1.3 Hz, 1H), 7.58 (ddd, J = 8.3, 4.7, 0.8 Hz, 1H), 7.36 (s, 2H), 3.44 (sept, J = 6.7 Hz, 2H), 3.02 (sept, J = 6.9 Hz, 1H), 1.31 (d, J = 6.7 Hz, 12H), 1.27 (d, J = 6.9 Hz, 6H).

^{19}F NMR (376 MHz, Methanol- d_4) δ –79.96.

^{13}C NMR (101 MHz, Methanol- d_4) δ 157.21, 153.38, 153.21, 142.83, 128.56, 126.57, 123.30, 121.77 (d, J = 318.5 Hz), 114.38, 49.43, 40.72, 35.39, 24.48, 24.00.

ESI-HRMS: calculated for $\text{C}_{20}\text{H}_{27}\text{IN}^+ [\text{M}]^+$: 408.1183, found: 408.1176.



Iodonium salt 15a and 15. General procedure C was followed using 4-iodopyridine (712 mg, 3.47 mmol) and 1,3,5-triisopropylbenzene (780 mg, 3.82 mmol). Iodonium bistriflate salt **15a** and iodonium triflate salt **15** were obtained as white solids in 43% yield (1.06 g) and 80% yield (673 mg) respectively. Using 2,2,2-trifluoroethanol as an internal standard, the ratio of triflate counter ion to the parent cation was calculated to be 1.9:1.0 for **15a** and 0.98:1.0 for **15**.

Iodonium salt 15a

^1H NMR (300 MHz, Methanol- d_4) δ 8.74 (d, J = 6.5 Hz, 2H), 8.01 – 7.94 (m, 2H), 7.45 (s, 2H), 3.29 – 3.21 (m, 2H), 3.07 (d, J = 7.0 Hz, 1H), 1.36 – 1.26 (m, 18H).

^{19}F NMR (282 MHz, Methanol- d_4) δ –80.54.

Iodonium salt 15

^1H NMR (400 MHz, Acetonitrile- d_3) δ 8.66 – 8.60 (m, 2H), 7.67 – 7.61 (m, 2H), 7.41 (s, 2H), 3.22 (sept, J = 6.7 Hz, 2H), 3.07 (sept, J = 6.9 Hz, 1H), 1.30 (d, J = 6.9 Hz, 7H), 1.26 (d, J = 6.7 Hz, 12H).

^{19}F NMR (376 MHz, Acetonitrile- d_3) δ –79.28.

^{13}C NMR (101 MHz, Acetonitrile- d_3) δ 158.07, 153.99, 153.95, 128.45, 127.42, 124.85, 121.83, 41.11, 35.47, 24.67, 24.28.

ESI-HRMS: calculated for $\text{C}_{20}\text{H}_{27}\text{IN}^+ [\text{M}]^+$: 408.1183, found: 408.1177.

3. Raman spectra of SWCNT-1 after TGA measurement

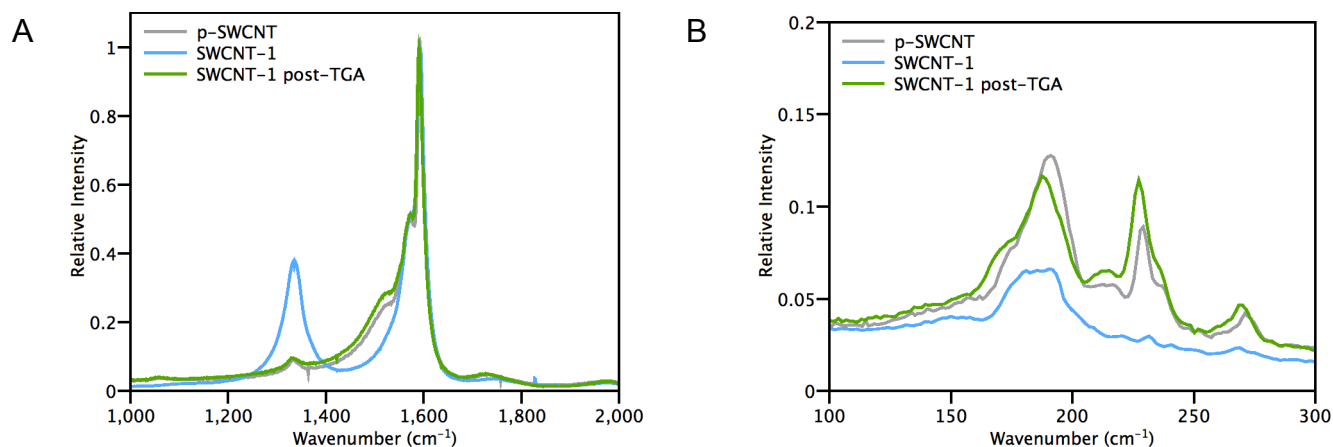
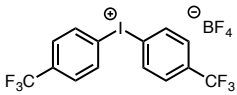
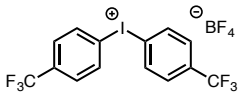
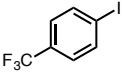
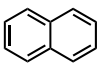
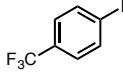
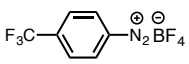


Figure S1. A) Raman spectra and B) Raman RBM region of **p-SWCNT**, **SWCNT-1** and **SWCNT-1** after TGA measurement.

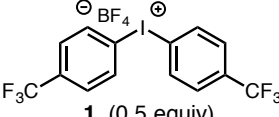
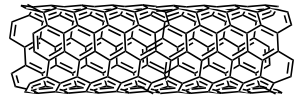
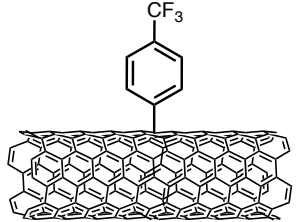
4. Control experiments

Table S1. Control experiments

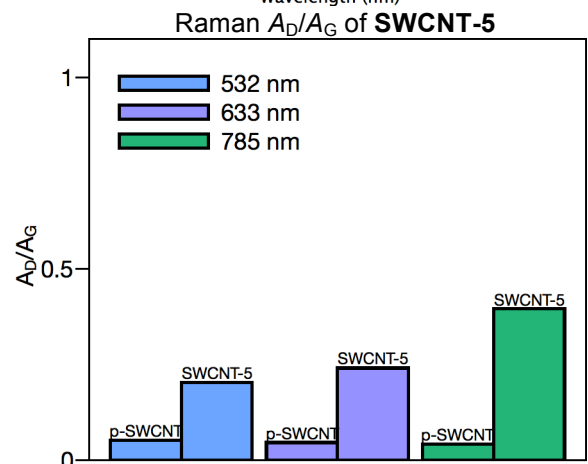
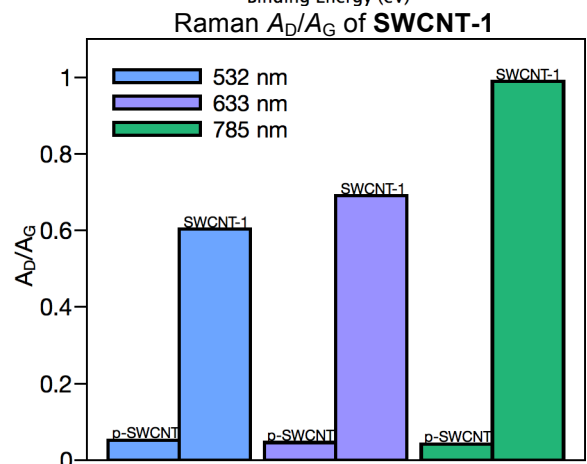
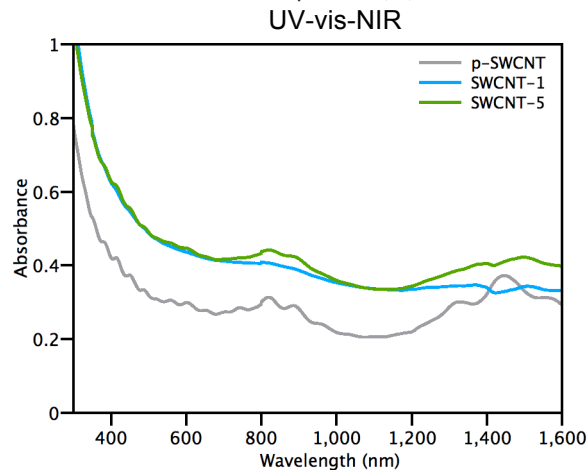
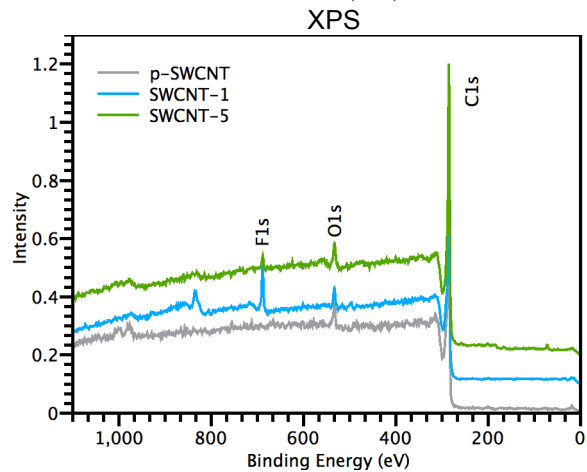
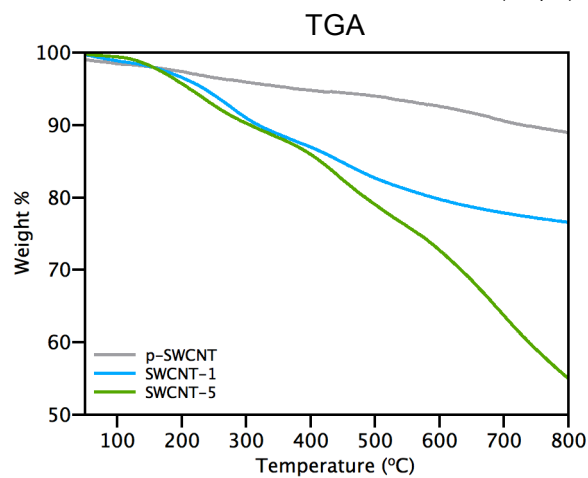
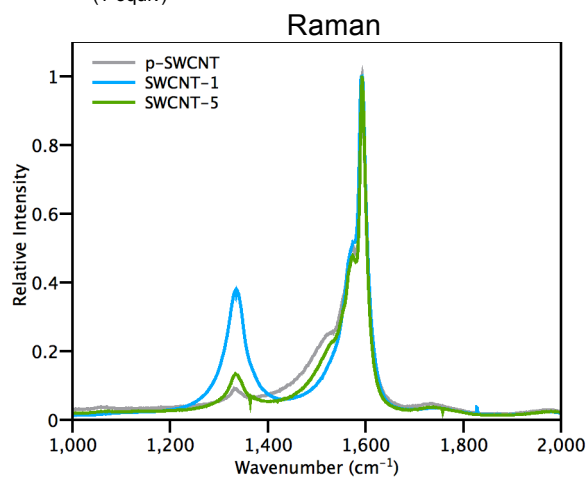
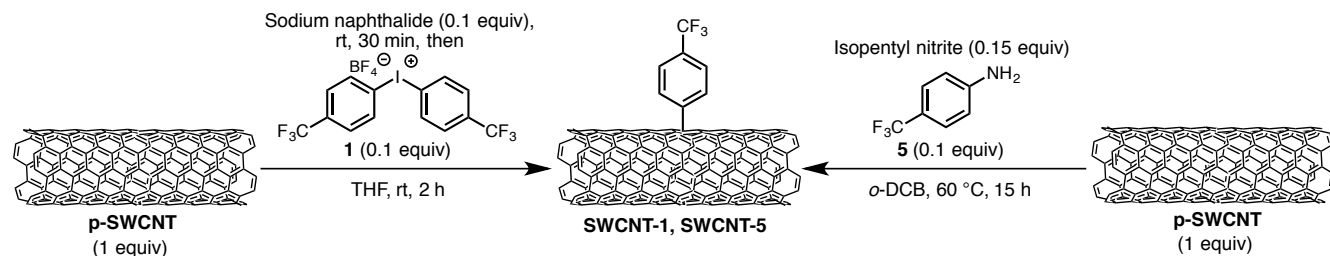
Entry	Reagents	SWCNT:NaC ₁₀ H ₈ :Reagent	Filtrate (analyzed by ¹ H NMR)	Raman A _D /A _G
1	None	1:0:0	N/A	p-SWCNT: 0.10
2	None	1:0.4:0		SWCNT-1a: 0.053
3		1:0:1		SWCNT-1b: 0.037
4		1:0.4:1	 + 	SWCNT-1c: 0.062
5		1:0.4:1	 + unidentified products	SWCNT-1d: 0.087

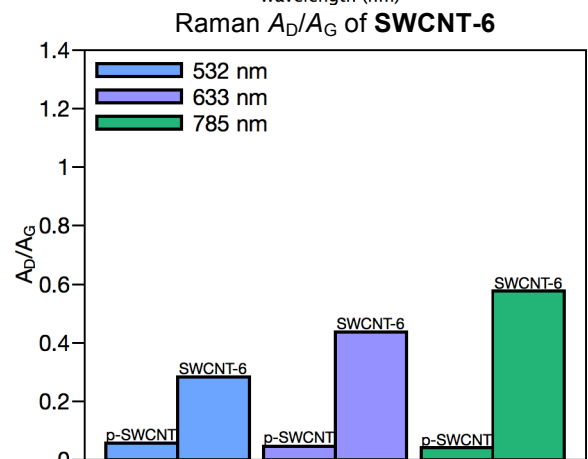
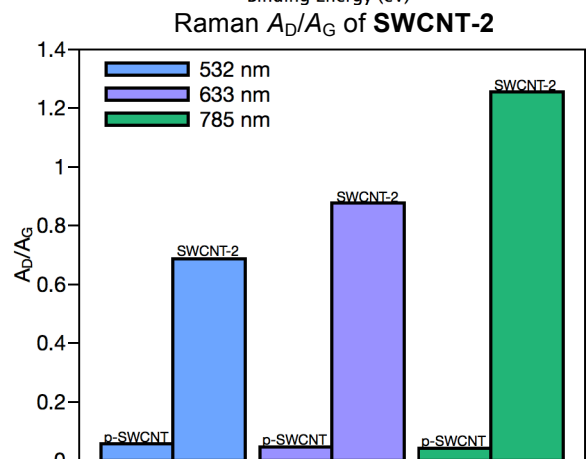
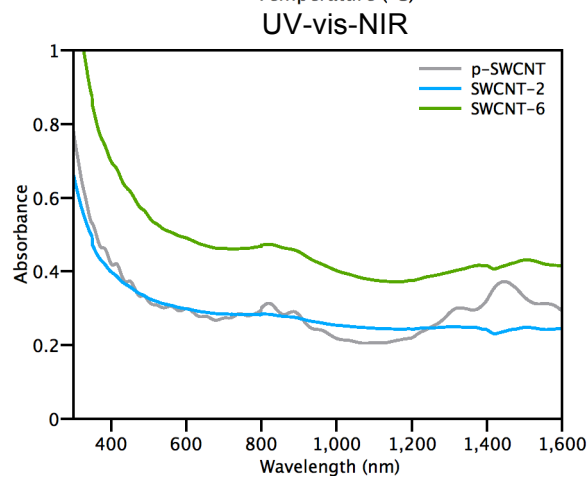
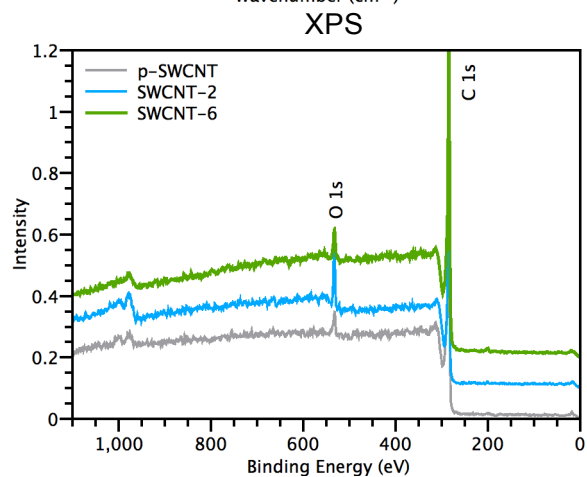
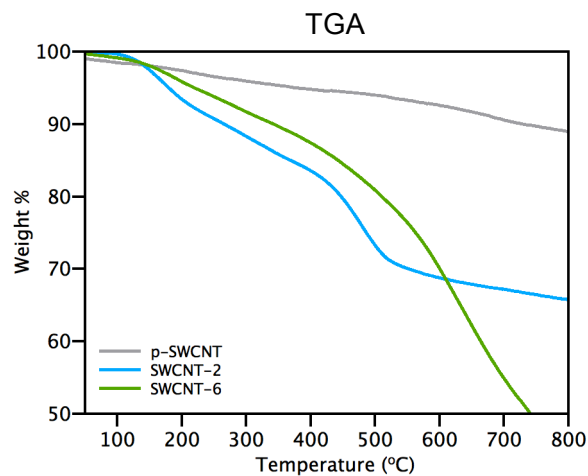
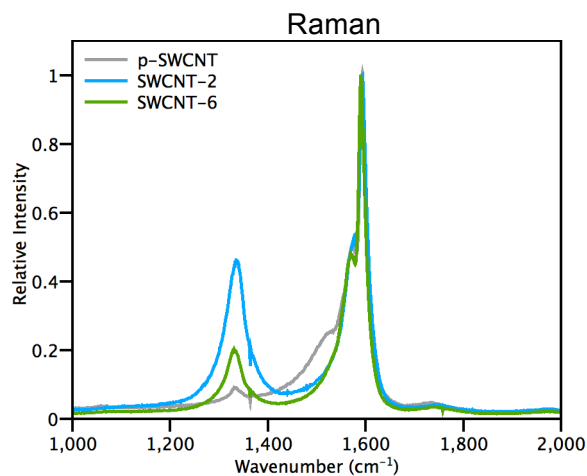
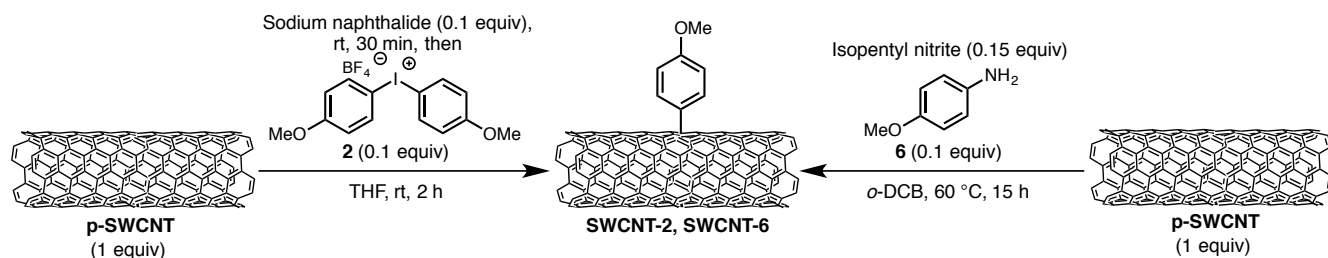
5. Reactions with different equivalents of sodium naphthalide

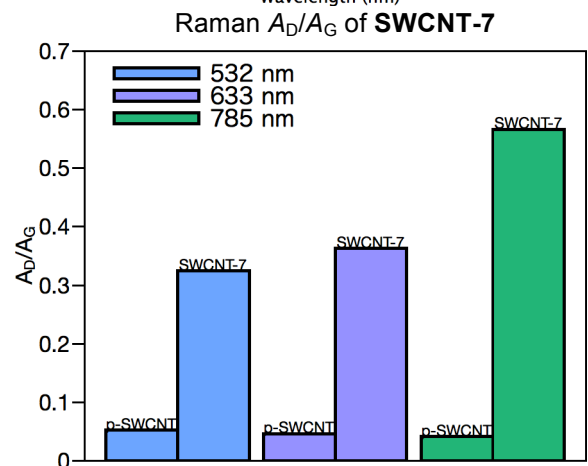
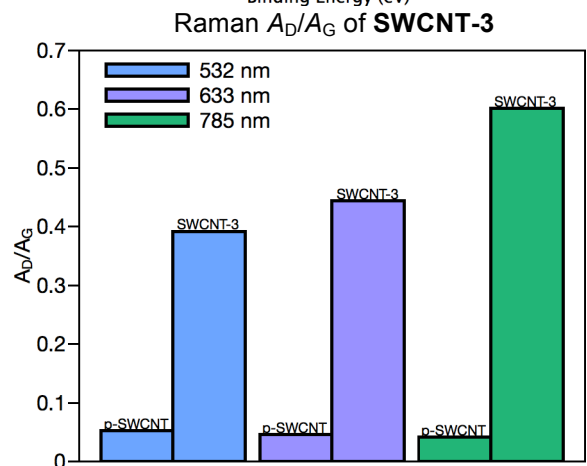
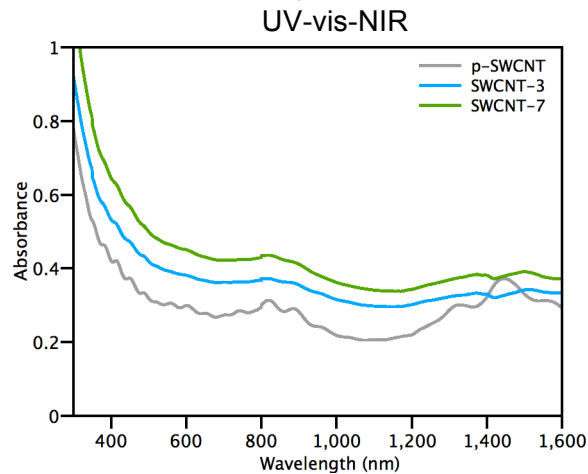
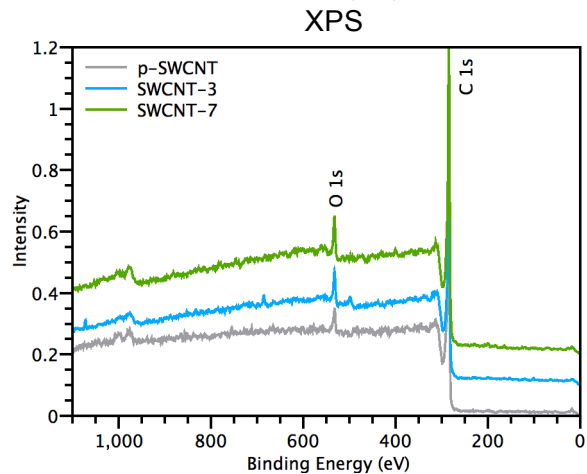
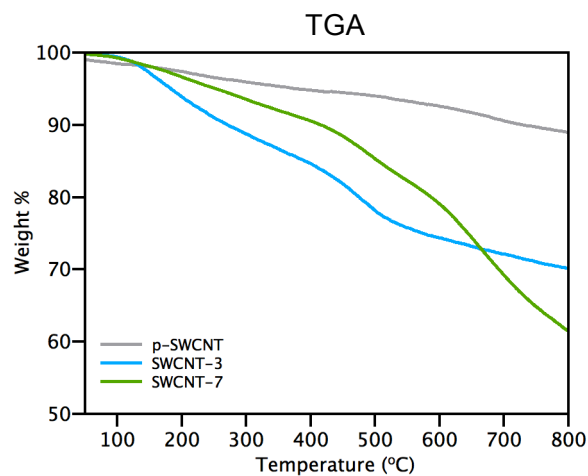
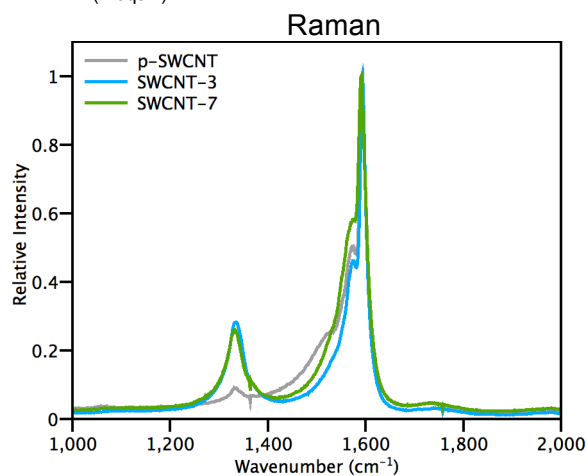
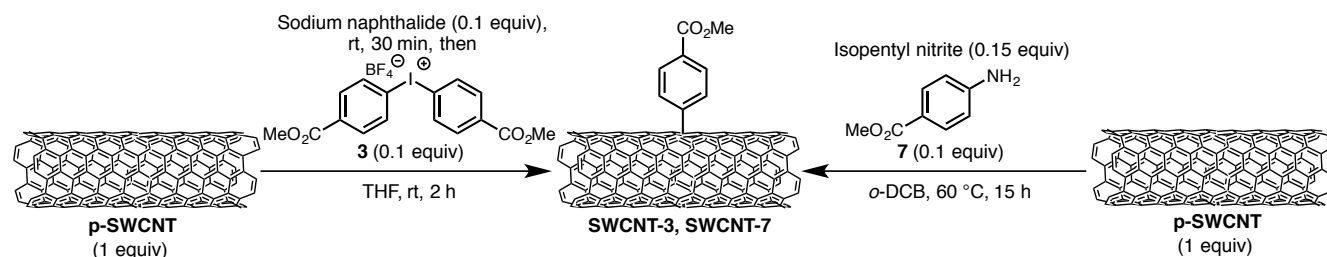
Table S2. Reactions of different equivalents of sodium naphthalide with p-SWCNT

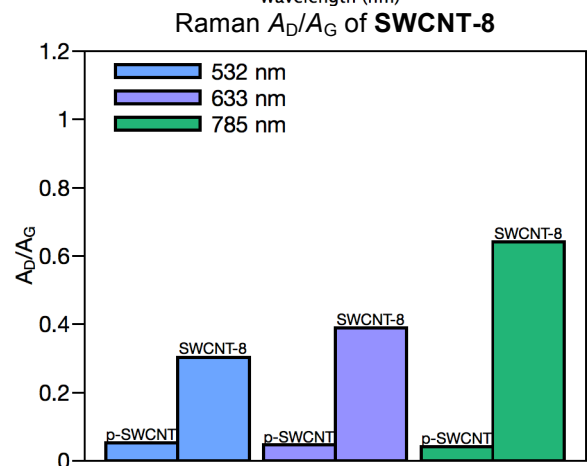
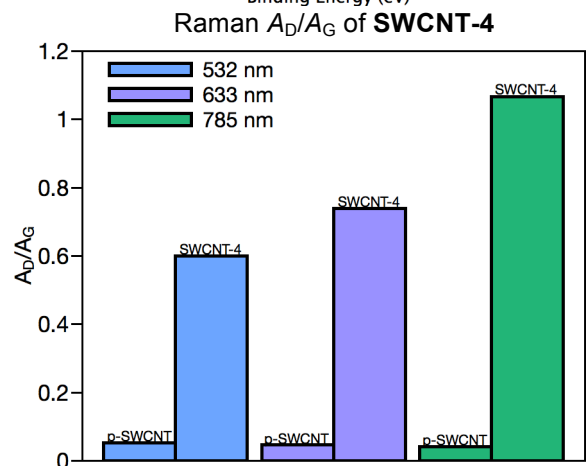
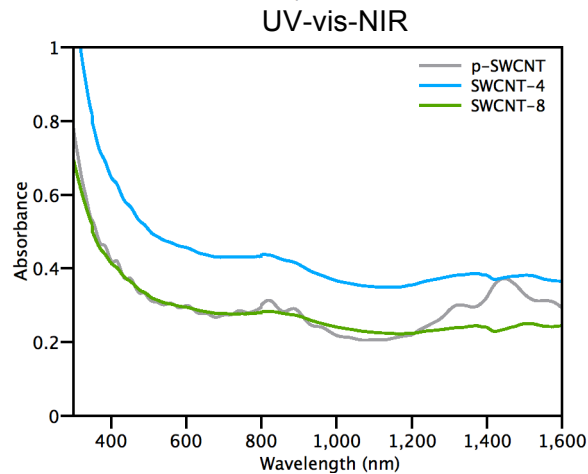
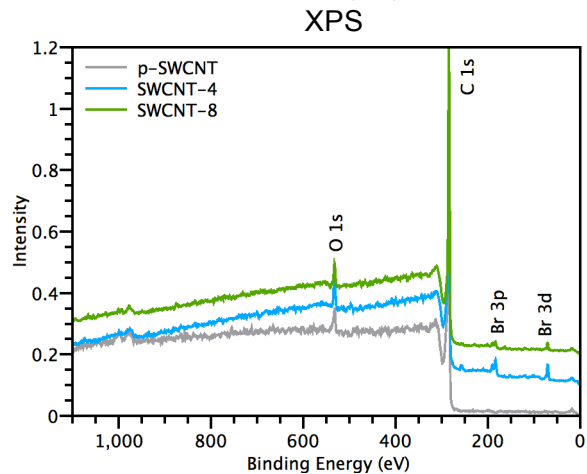
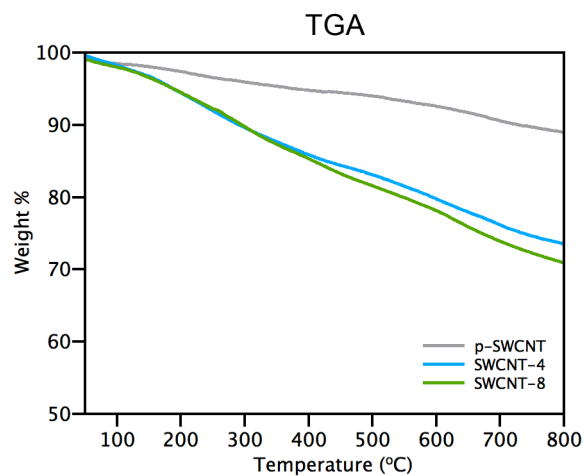
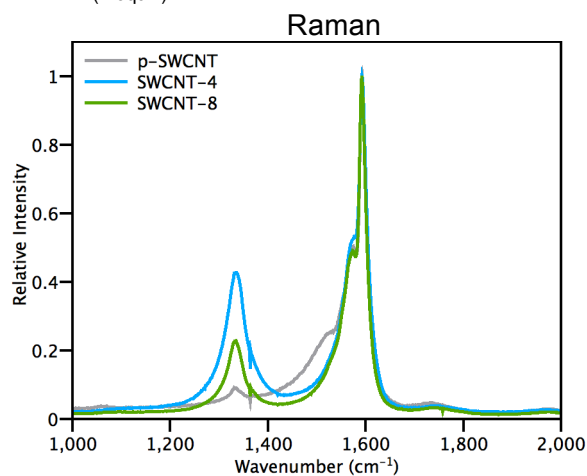
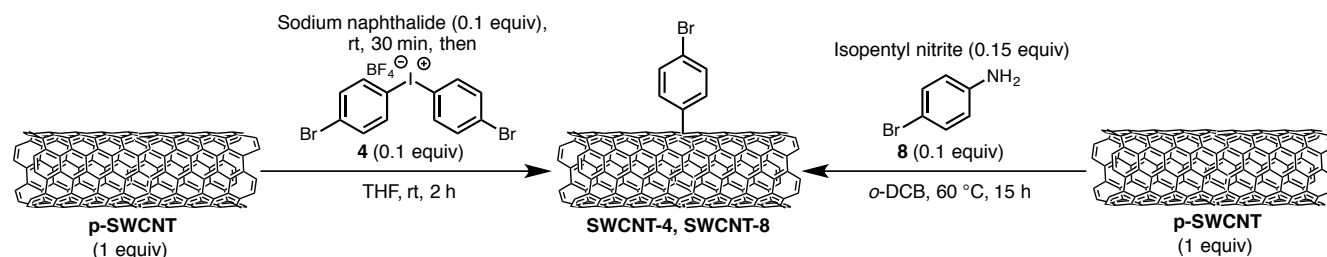
Sodium naphthalide (0.1–0.4 equiv), rt, 30 min, then  1, (0.5 equiv) THF, rt, 2 h			 p-SWCNT (1 equiv)	 SWCNT-1
<u>Equivalents of iodonium salt 1</u>	<u>Equivalents of sodium naphthalide</u>	<u>SWCNT-1 Raman A_D/A_G</u>		
0.5	0.1	0.48		
0.5	0.2	0.64		
0.5	0.3	0.63		
0.5	0.4	0.43		

6. Comparison between iodonium and diazonium methods

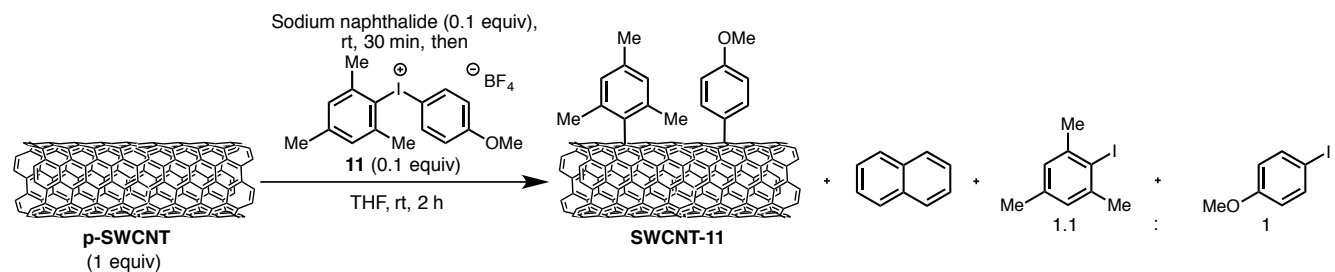
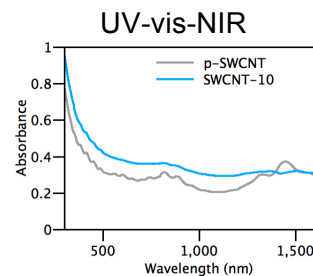
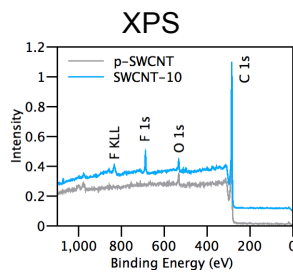
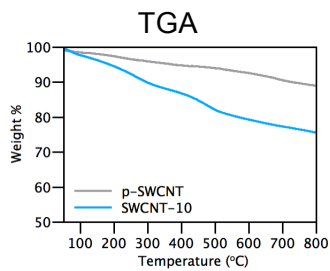
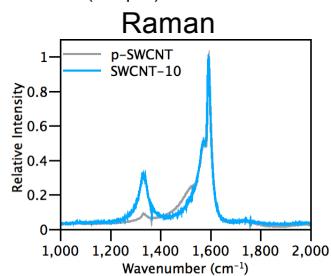
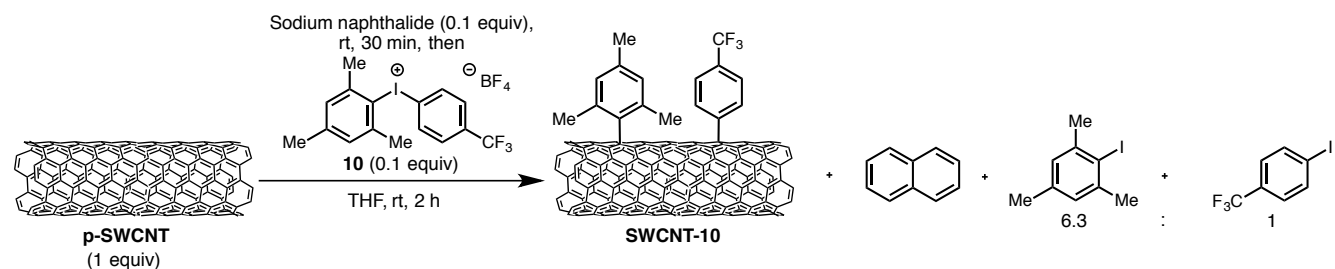
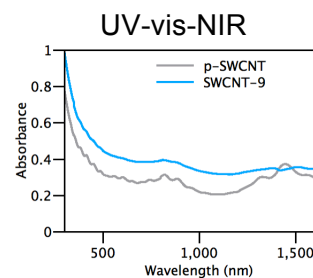
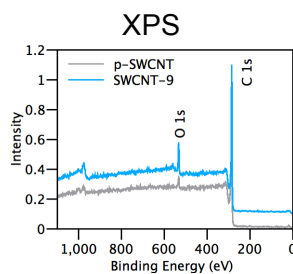
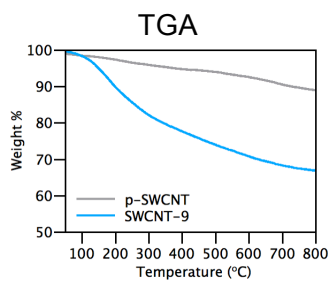
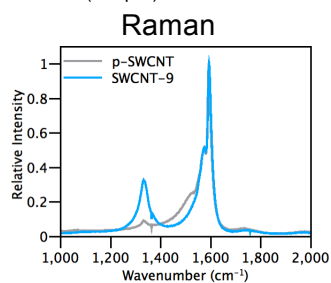
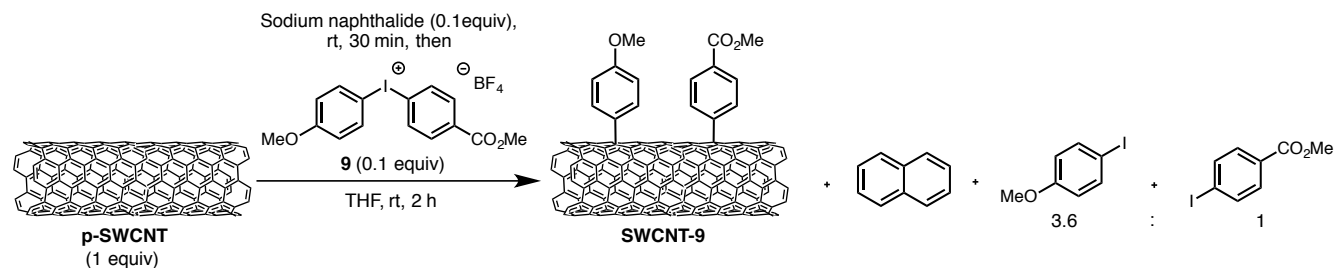


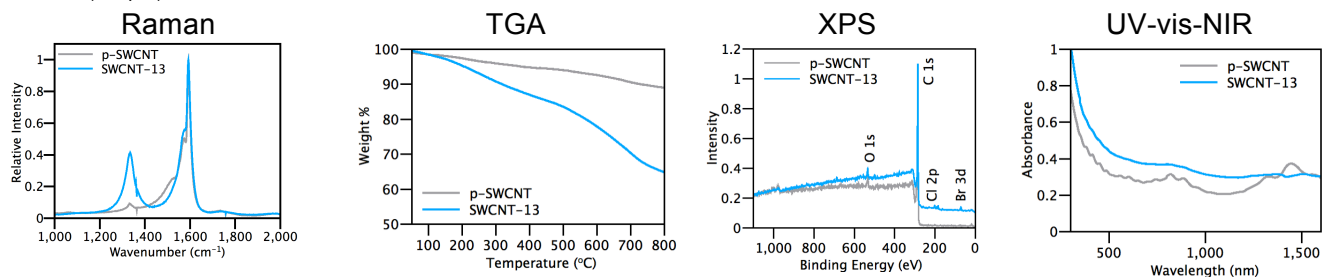
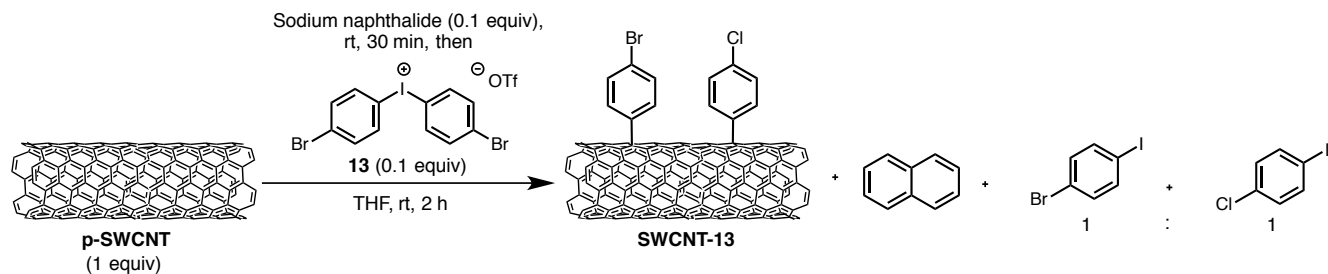
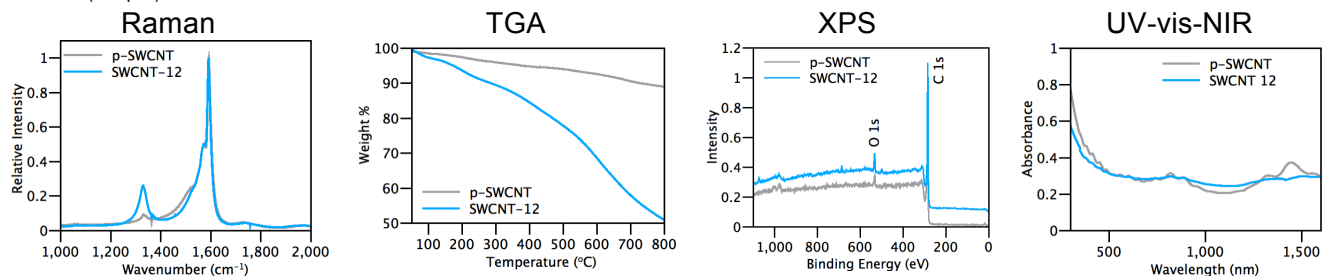
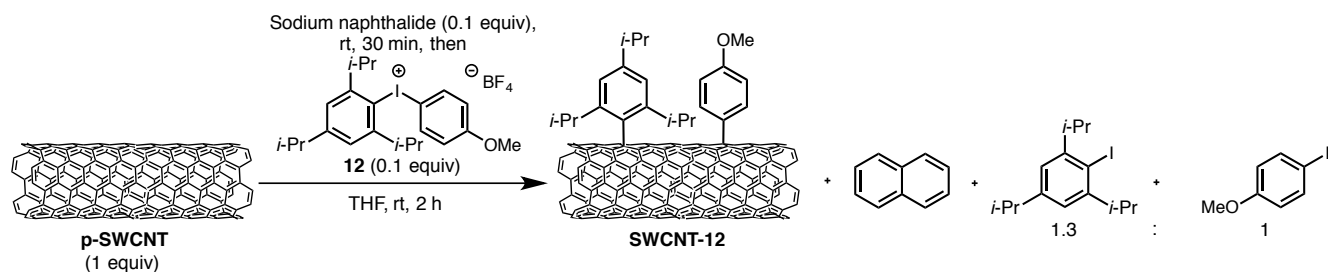
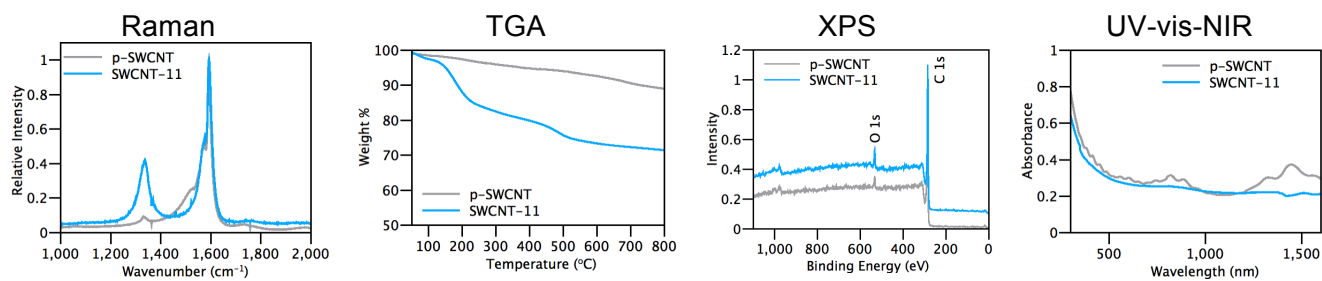


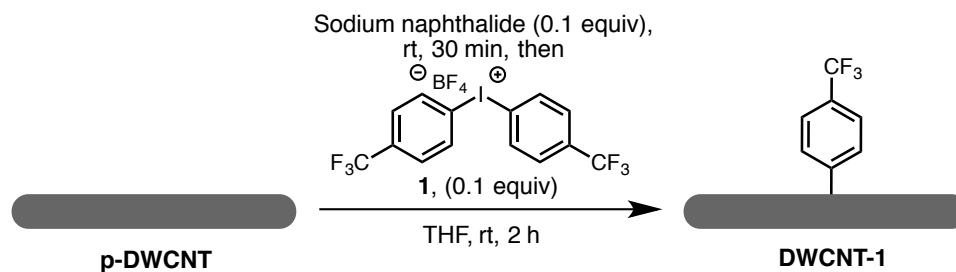
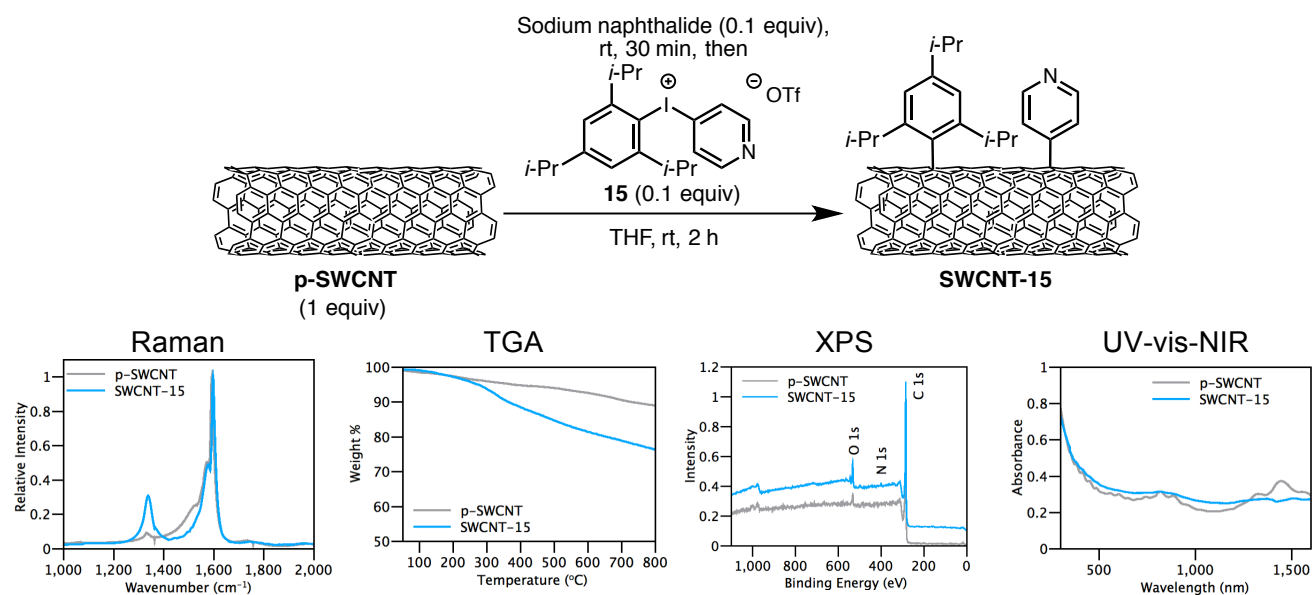
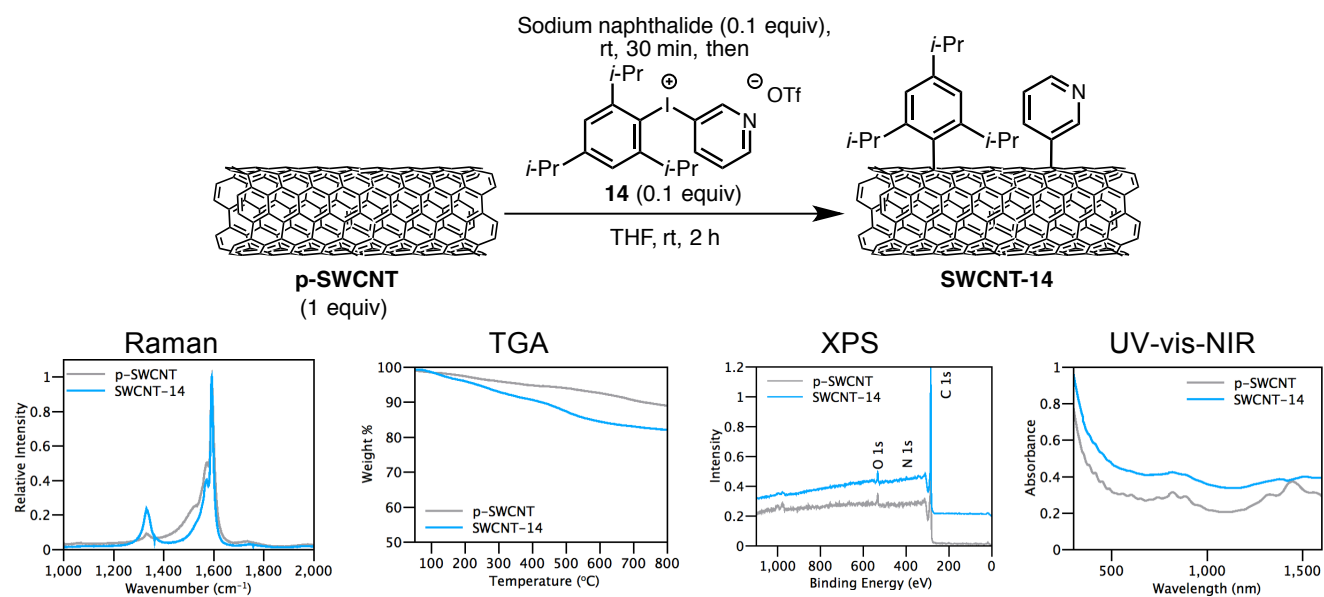


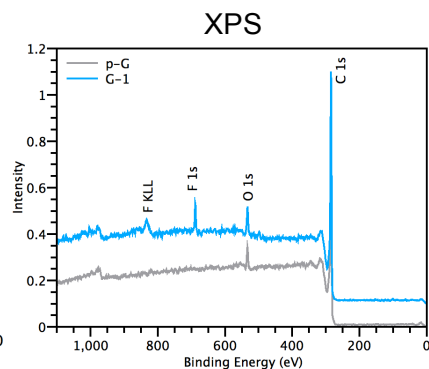
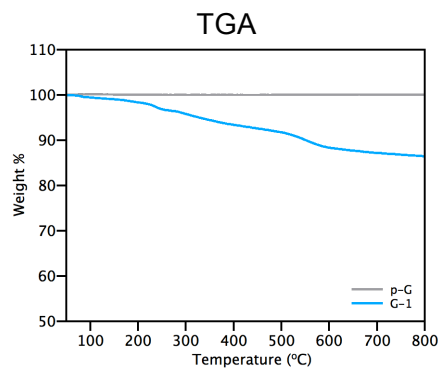
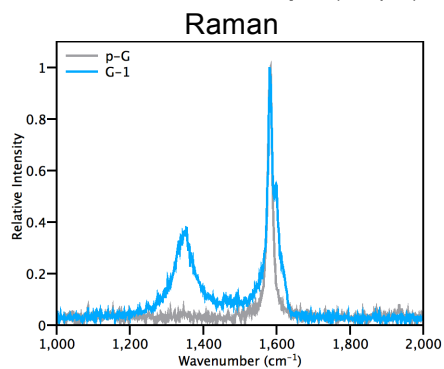
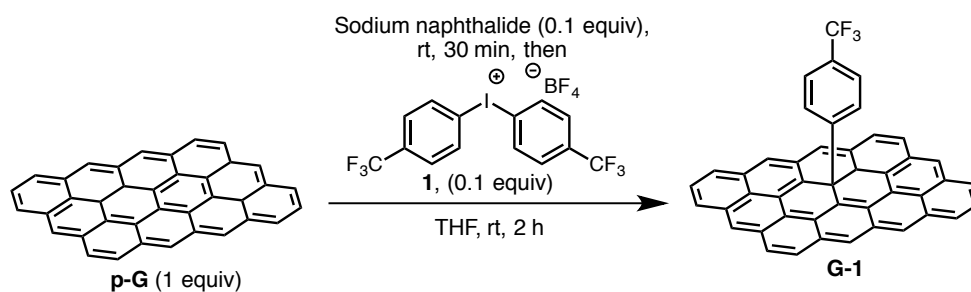
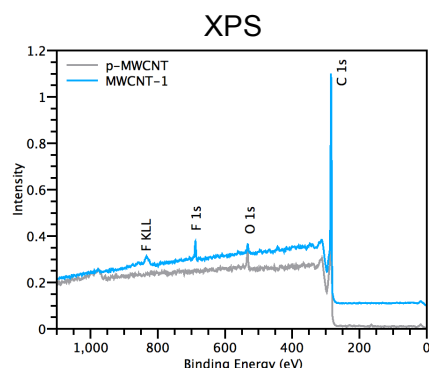
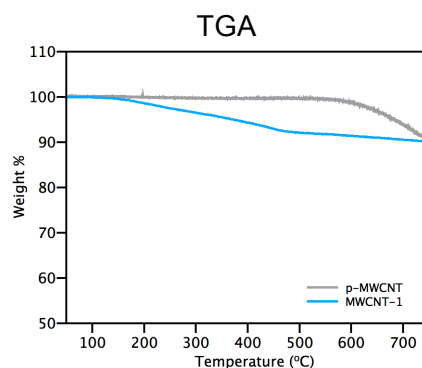
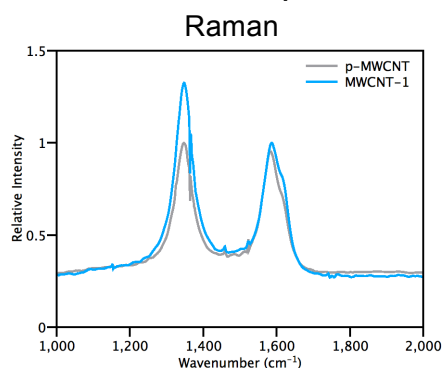
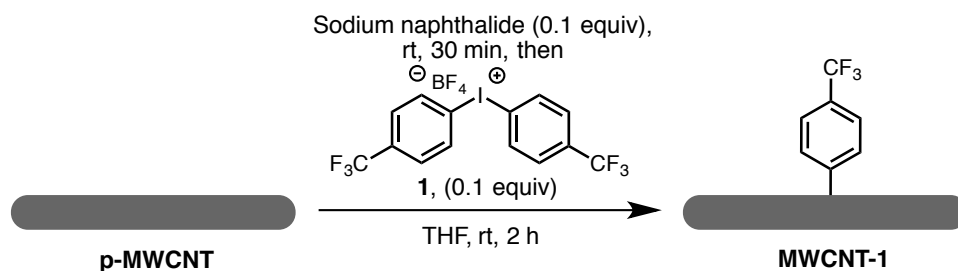
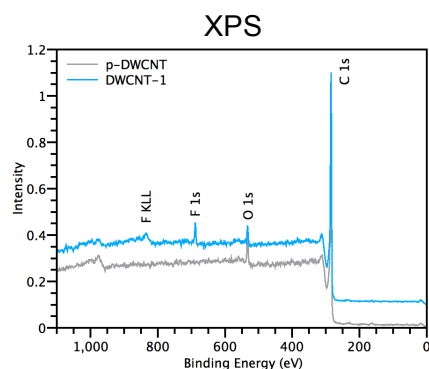
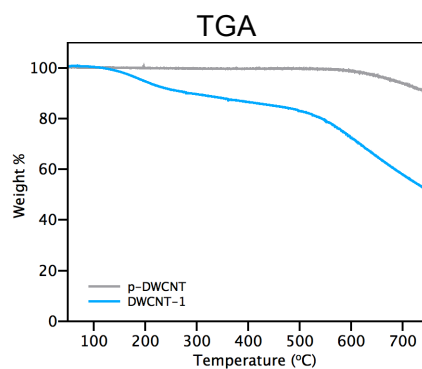
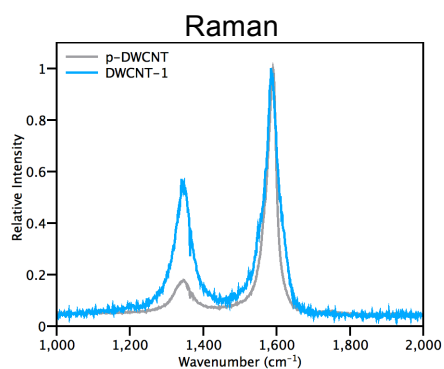


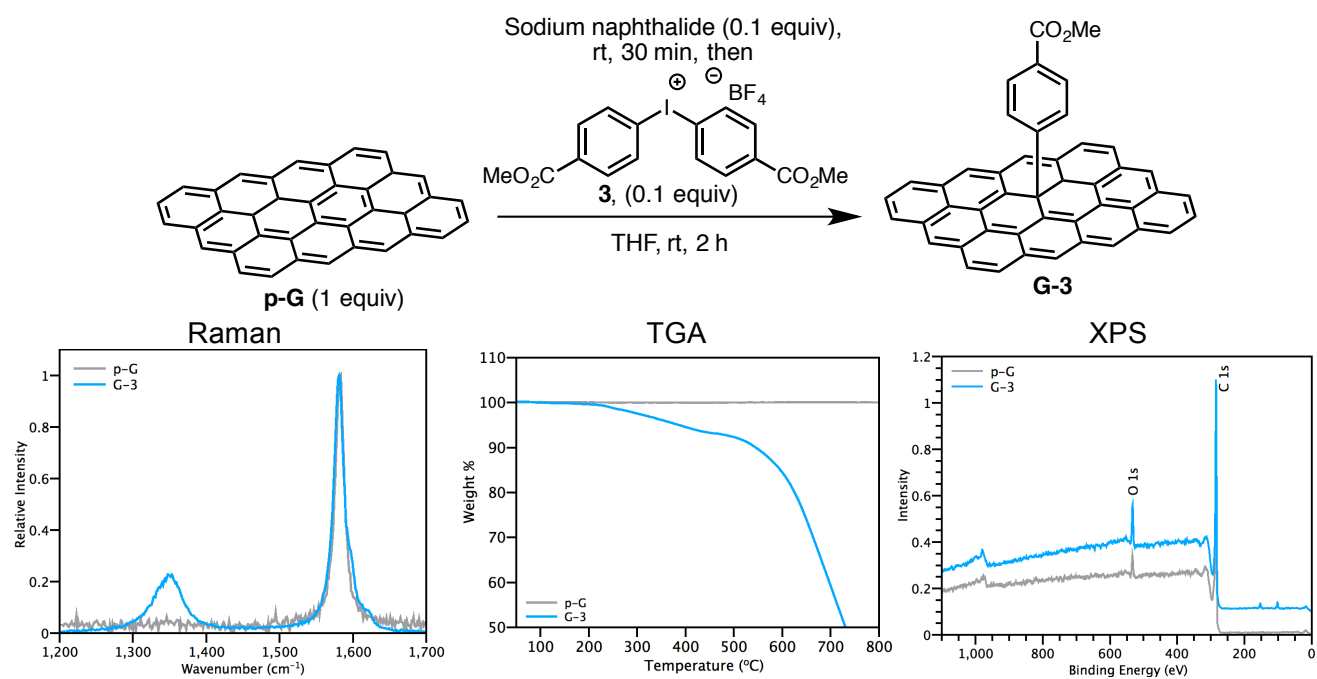
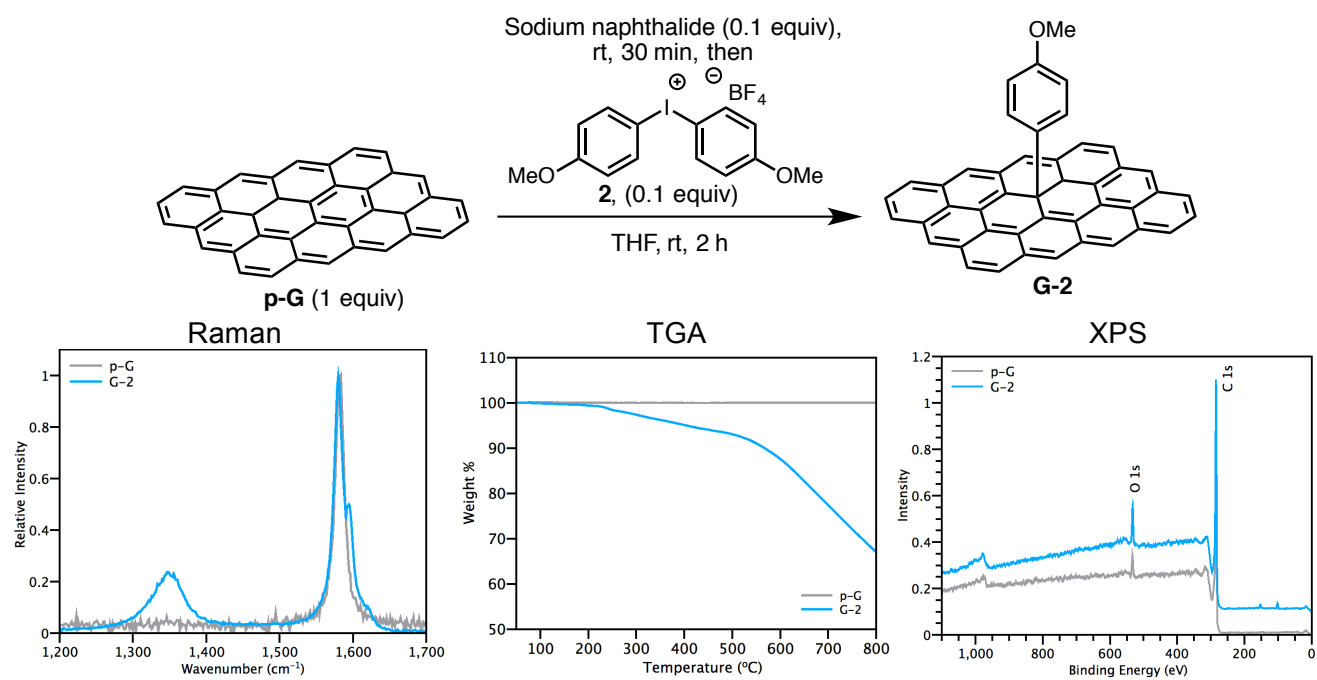
7. Raman, TGA, XPS and UV-vis-NIR of CNTs and graphites

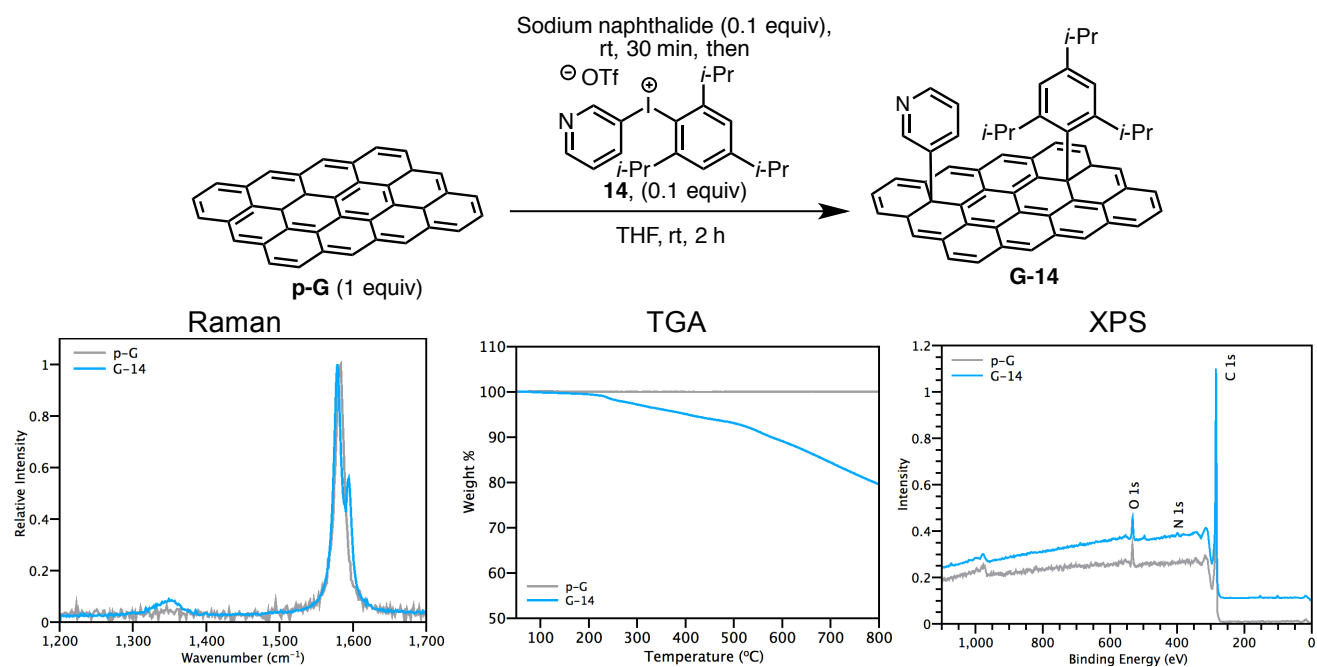












8. Control reactions with 3-iodopyridine and 4-iodopyridine

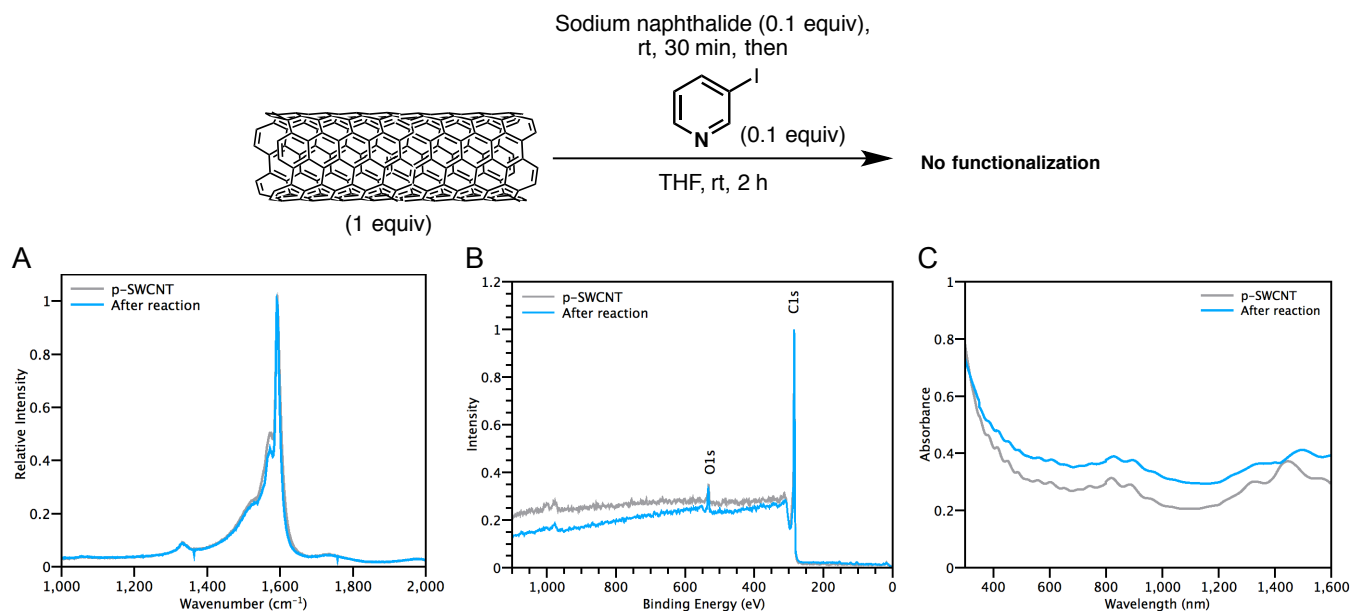


Figure S2. A) Raman, B) XPS and C) UV-vis-NIR of **p-SWCNT** after reaction with 3-iodopyridine.

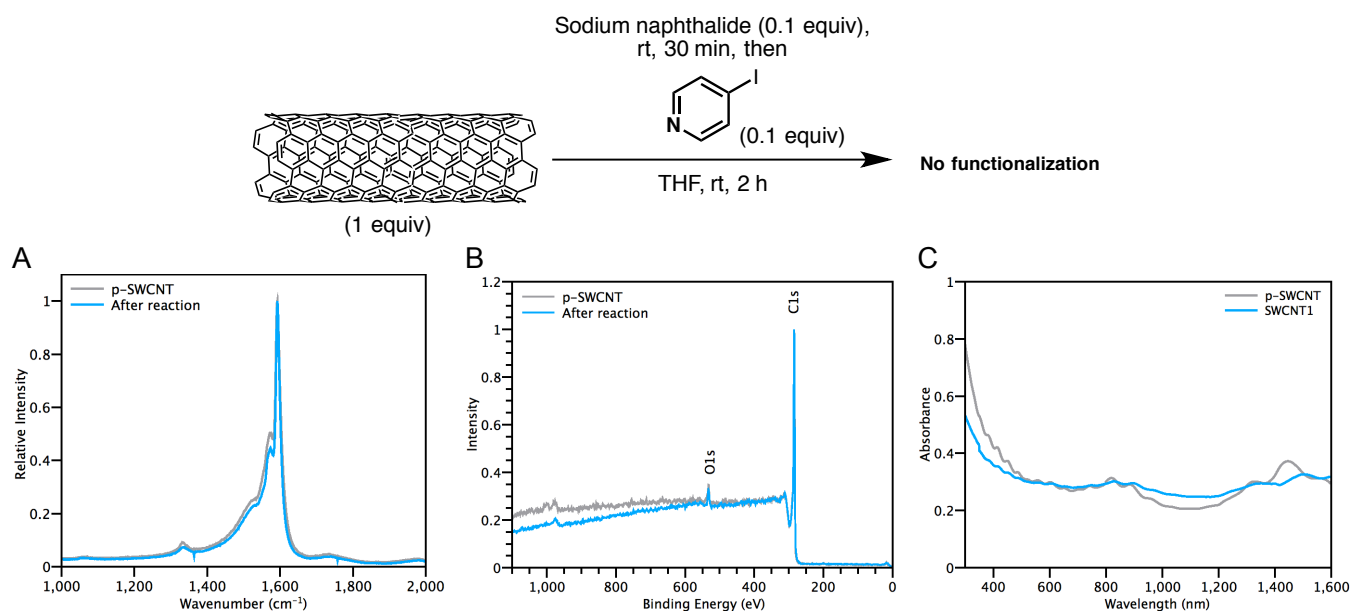


Figure S3. A) Raman, B) XPS and C) UV-vis-NIR of **p-SWCNT** after reaction with 4-iodopyridine.

9. Raman statistical analysis

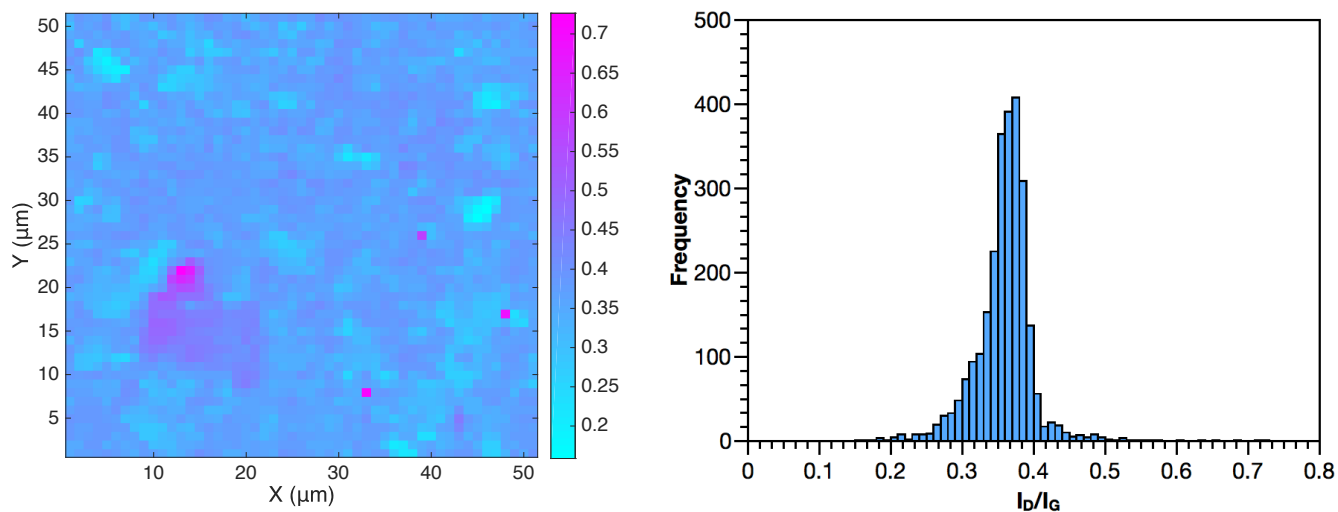


Figure S4. Spatial (left) and histogram (right) representation of the I_D/I_G ratio of **SWCNT-1**. The area of the mapped region are $100 \mu\text{m} \times 100 \mu\text{m}$.

10. Degree of functionalization

$$DF = \frac{(A_{wtloss} - N_{wtloss}) / A_{MW}}{R_{wt} / C_{MW}} \times 100$$

DF = degree of functionalization, defined as the number of addend per 100 carbon atoms on CNT or graphite

A_{wtloss} [g] = TGA weight loss of functionalized nanomaterial at 500 °C

N_{wtloss} [g] = TGA weight loss of pristine nanomaterial at 500 °C

A_{MW} [g/mol] = molecular weight of addend

R_{wt} [g] = residual weight of sample

C_{MW} [g/mol] = molecular weight of carbon (12 g/mol)

Nanomaterials	A_{wtloss} [g]	N_{wtloss} [g]	A_{MW} [g/mol]	R_{wt} [g]	DF
SWCNT-1	17	7	145	83	1.0
SWCNT-2	27	7	107	73	3.1
SWCNT-3	22	7	135	78	1.7
SWCNT-4	18	7	156	82	1.0
SWCNT-5	21	7	145	79	1.5
SWCNT-6	19	7	107	81	1.7
SWCNT-7	15	7	135	85	0.8
SWCNT-8	18	7	156	82	1.0
G-1	9	0	9	91	0.8
G-2	8	0	8	92	1.0
G-3	7	0	7	93	0.7

11. TGA-MS data

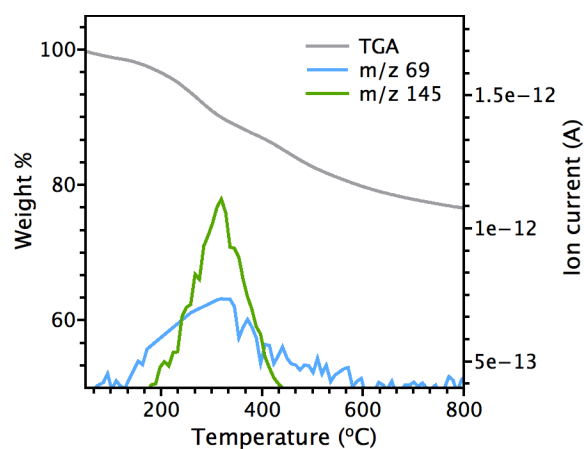


Figure S5. TGA-MS of **SWCNT-1**. Depicted MS traces for m/z 145 and m/z 69 can be attributed to the trifluoromethylphenyl fragments (M) and (M – CF₃) respectively.

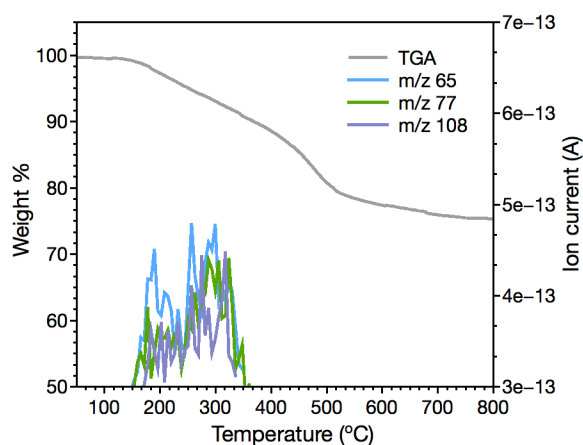


Figure S6. TGA-MS of **SWCNT-2**. Depicted MS traces for m/z 108, m/z 77 and m/z 65 can be attributed to the methoxyphenyl fragments (M), (M – OCH₃) and (M – CH₃ – CO) respectively.

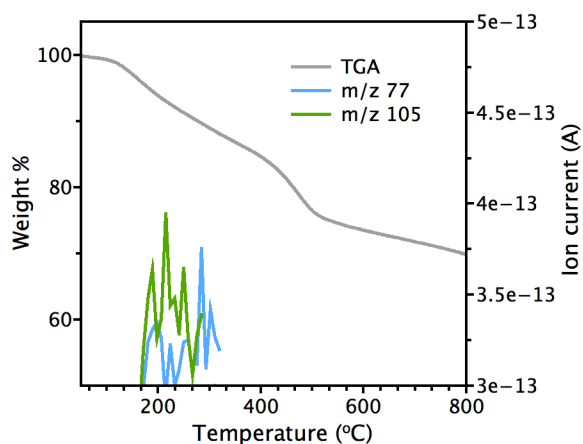


Figure S7. TGA-MS of **SWCNT-3**. Depicted MS trace for m/z 105 and m/z 77 can be attributed to the methyl benzoate fragments (M – OCH₃) and (M – OCH₃ – CO) respectively.

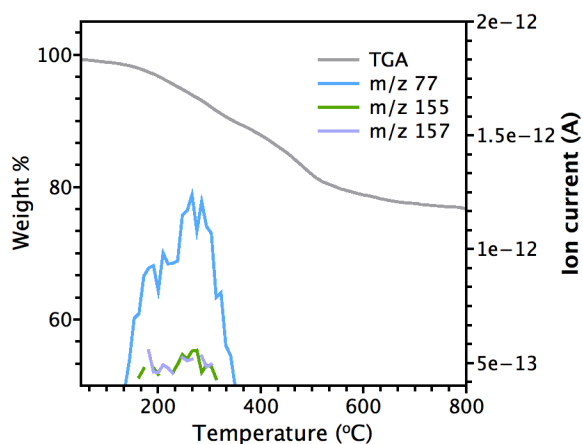


Figure S8. TGA-MS of **SWCNT-4**. Depicted MS trace for m/z 155 and m/z 155 can be attributed to the bromophenyl fragment (M). The m/z 77 trace can be attributed to the fragment (M – Br).

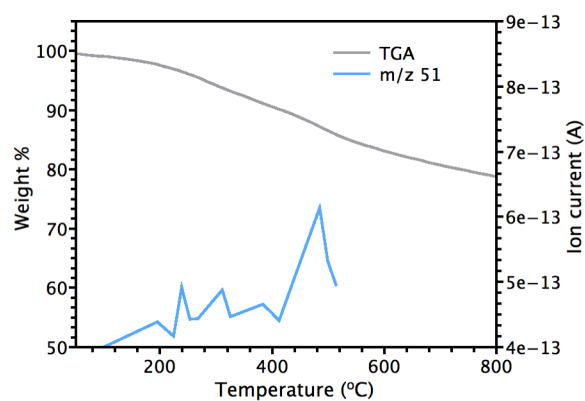


Figure S9. TGA-MS for **SWCNT-15**. Depicted MS trace for m/z can be attributed to the pyridine fragment ($M - \text{HCN}$).

12. ATR-FTIR data

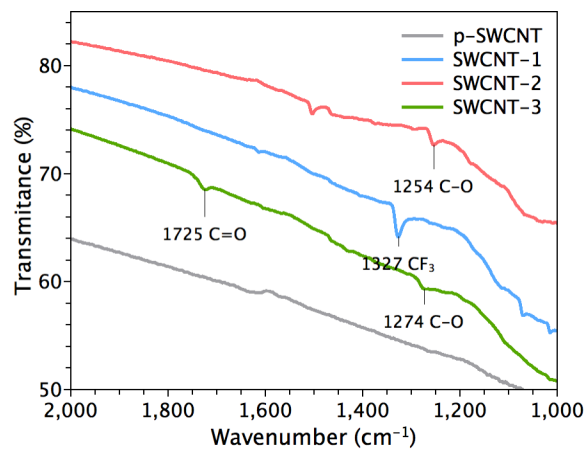


Figure S10. ATR-FTIR of **p-SWCNT**, **SWCNT-1**, **SWCNT-2** and **SWCNT-3**.

13. NMR spectra of the reaction filtrates

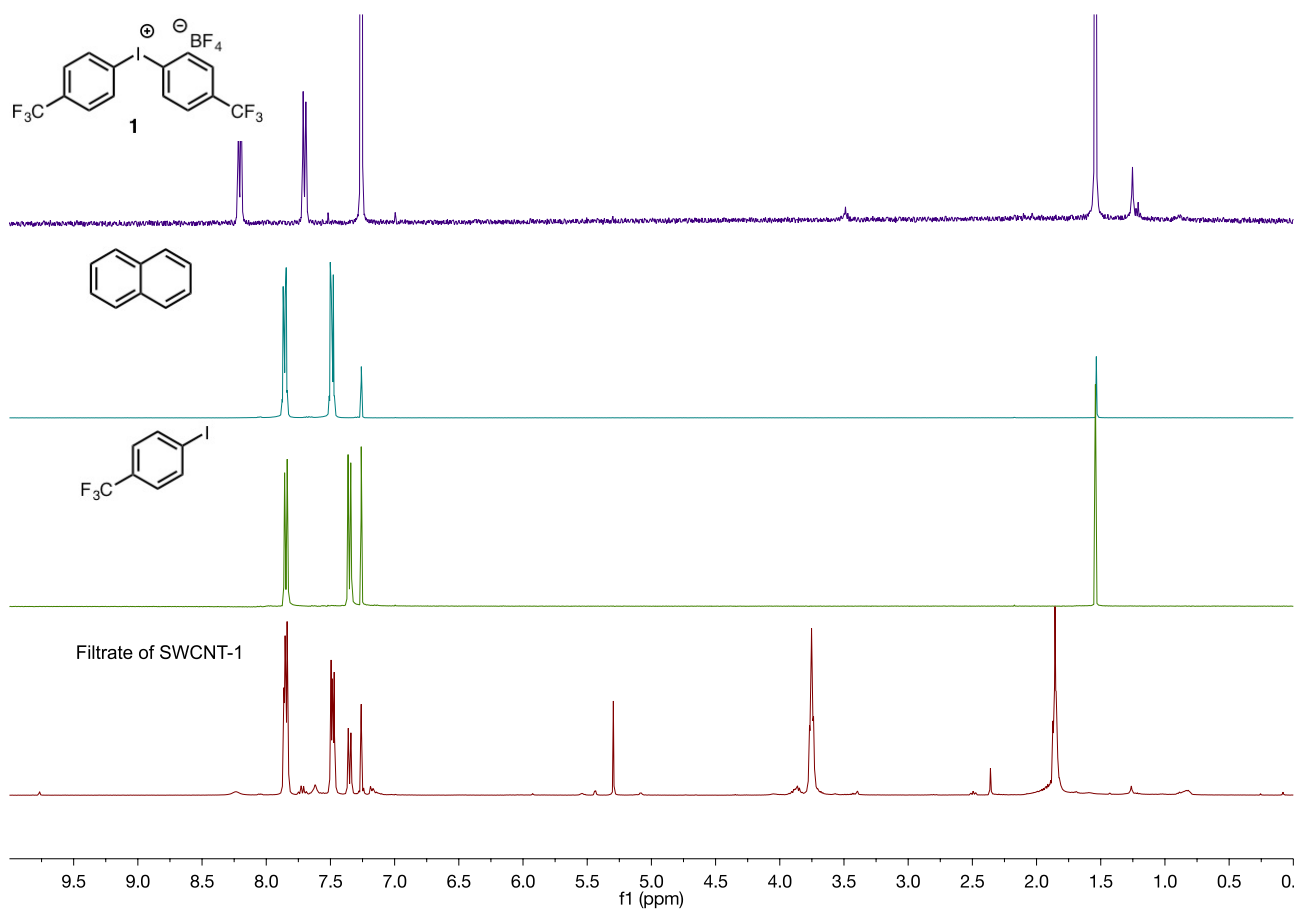


Figure S11. NMR spectra of the filtrate of **SWCNT-1** in CDCl_3 .

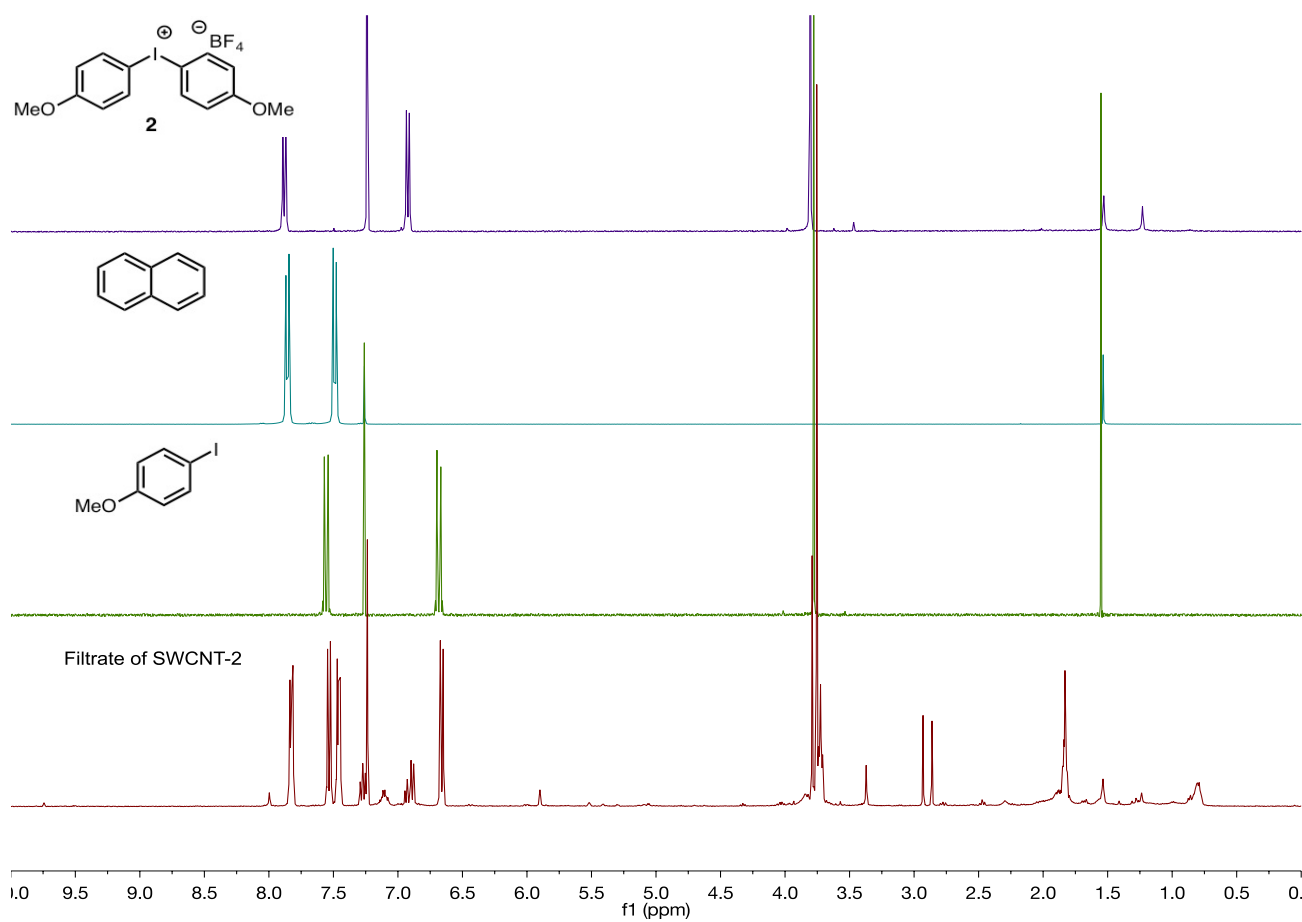


Figure S12. NMR spectra of the filtrate of **SWCNT-2** in CDCl_3 .

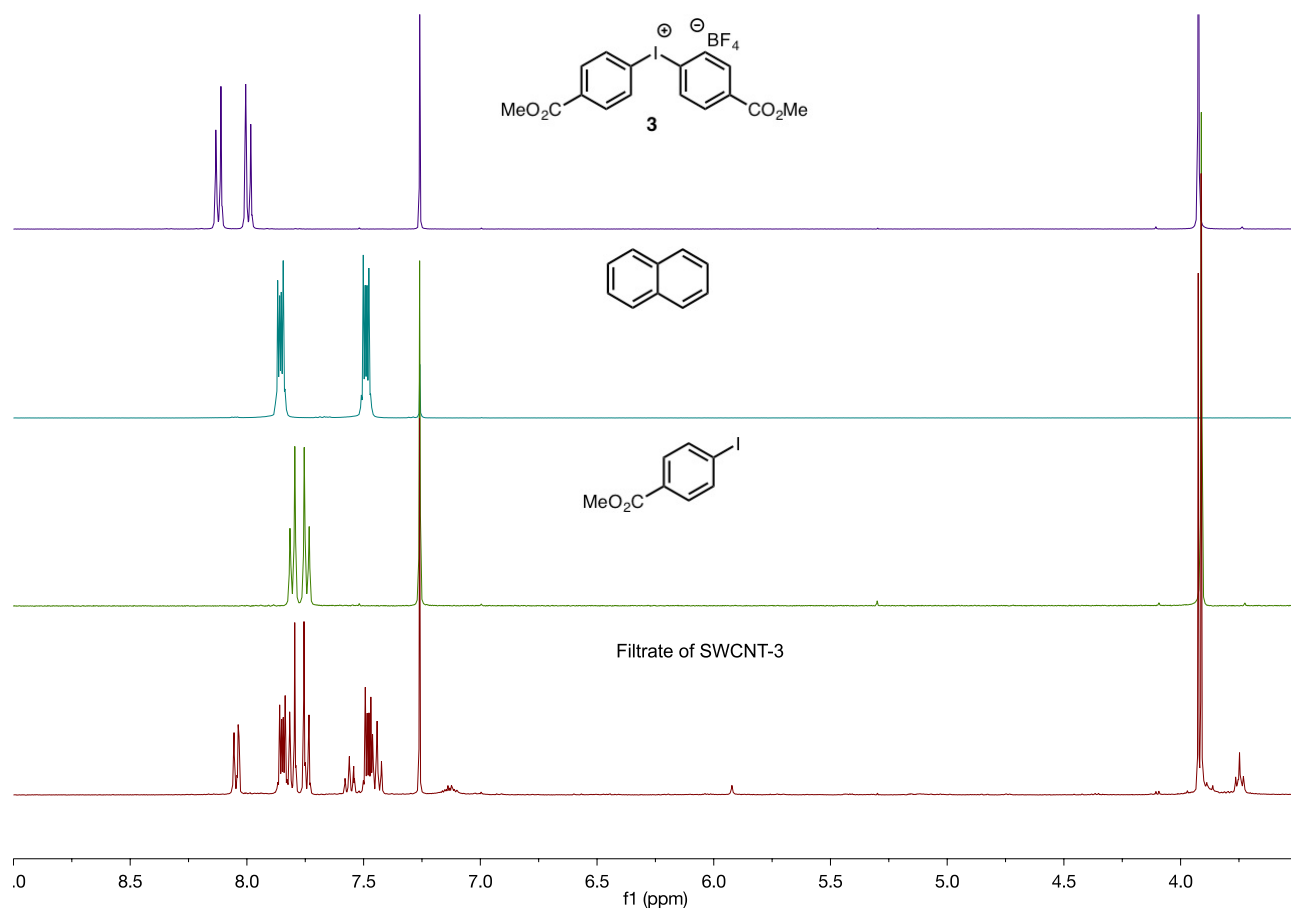
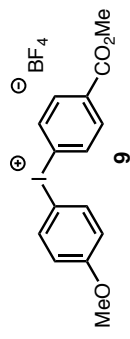


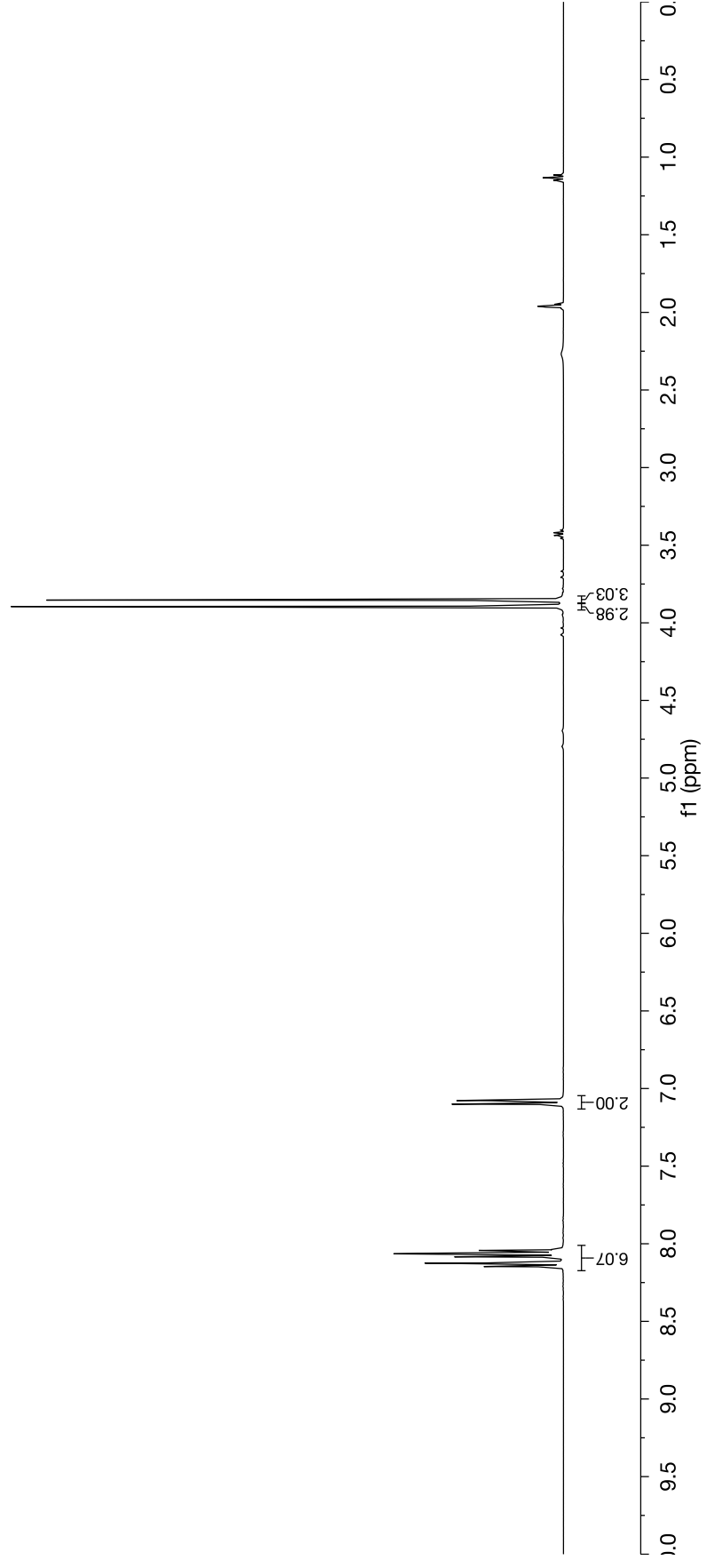
Figure S13. NMR spectra of the filtrate of **SWCNT-3** in CDCl_3 .

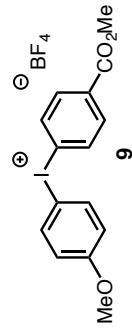
14. Reference

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- (3) Bielawski, M.; Malmgren, J.; Pardo, L. M.; Wikmark, Y.; Olofsson, B. *ChemistryOpen* **2014**, *3*, 19–22.
- (4) Monastyrskiy, A.; Namelikonda, N. K.; Manetsch, R. *J. Org. Chem.* **2015**, *80*, 2513–2520.
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- (6) Zhou, X.; Wu, Q. H.; Yu, Y.; Yu, C. J.; Hao, E. H.; Wei, Y.; Mu, X. L.; Jiao, L. *J. Org. Lett.* **2016**, *18*, 736–739.
- (7) Bigot, A.; Williamson, A. E.; Gaunt, M. J. *J. Am. Chem. Soc.* **2011**, *133*, 13778–13781.
- (8) Phipps, R. J.; Grimster, N. P.; Gaunt, M. J. *J. Am. Chem. Soc.* **2008**, *130*, 8172–8174.

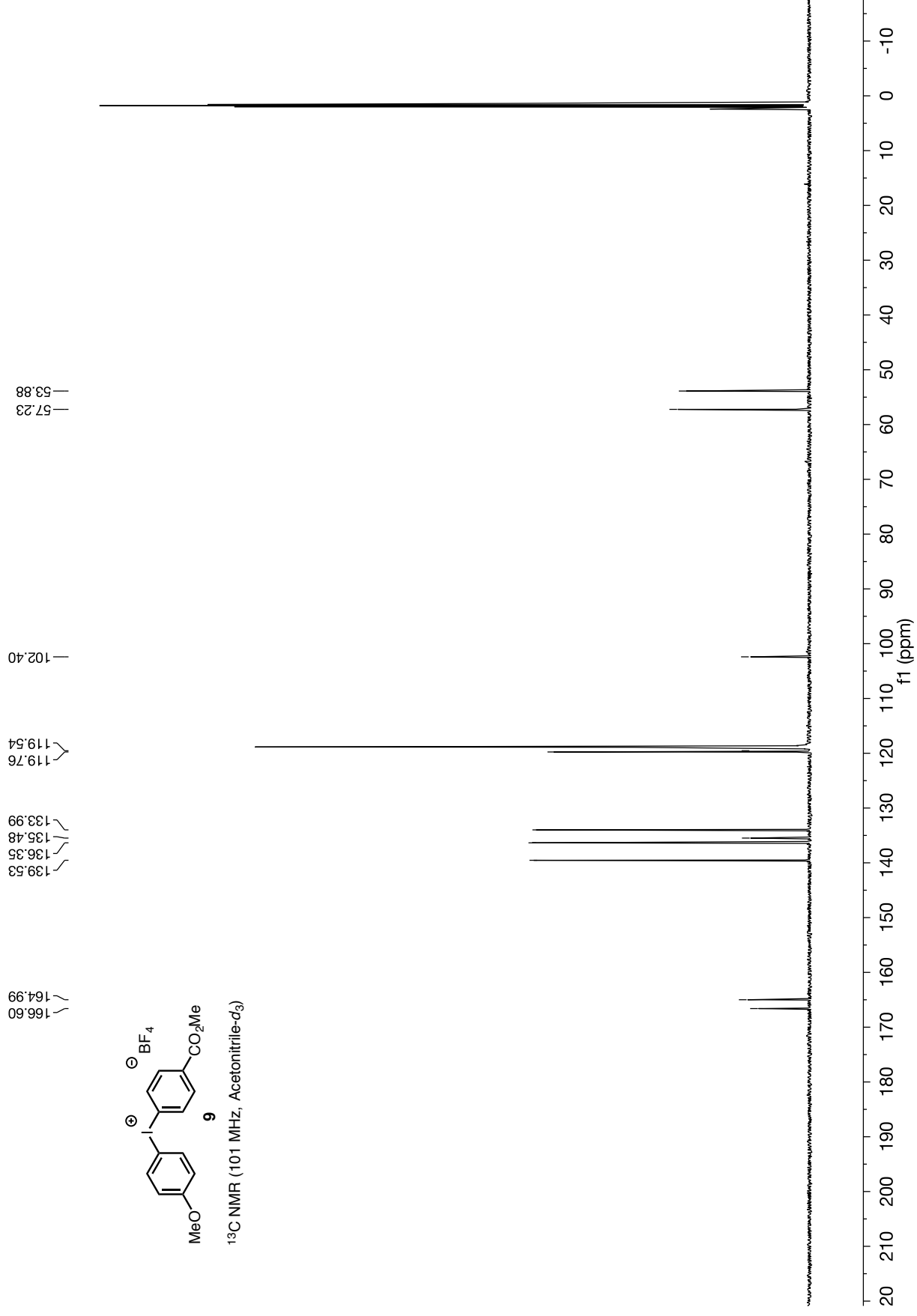


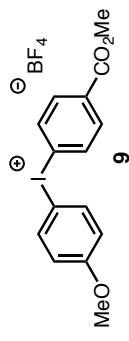
^1H NMR (400 MHz, Acetonitrile- d_3)



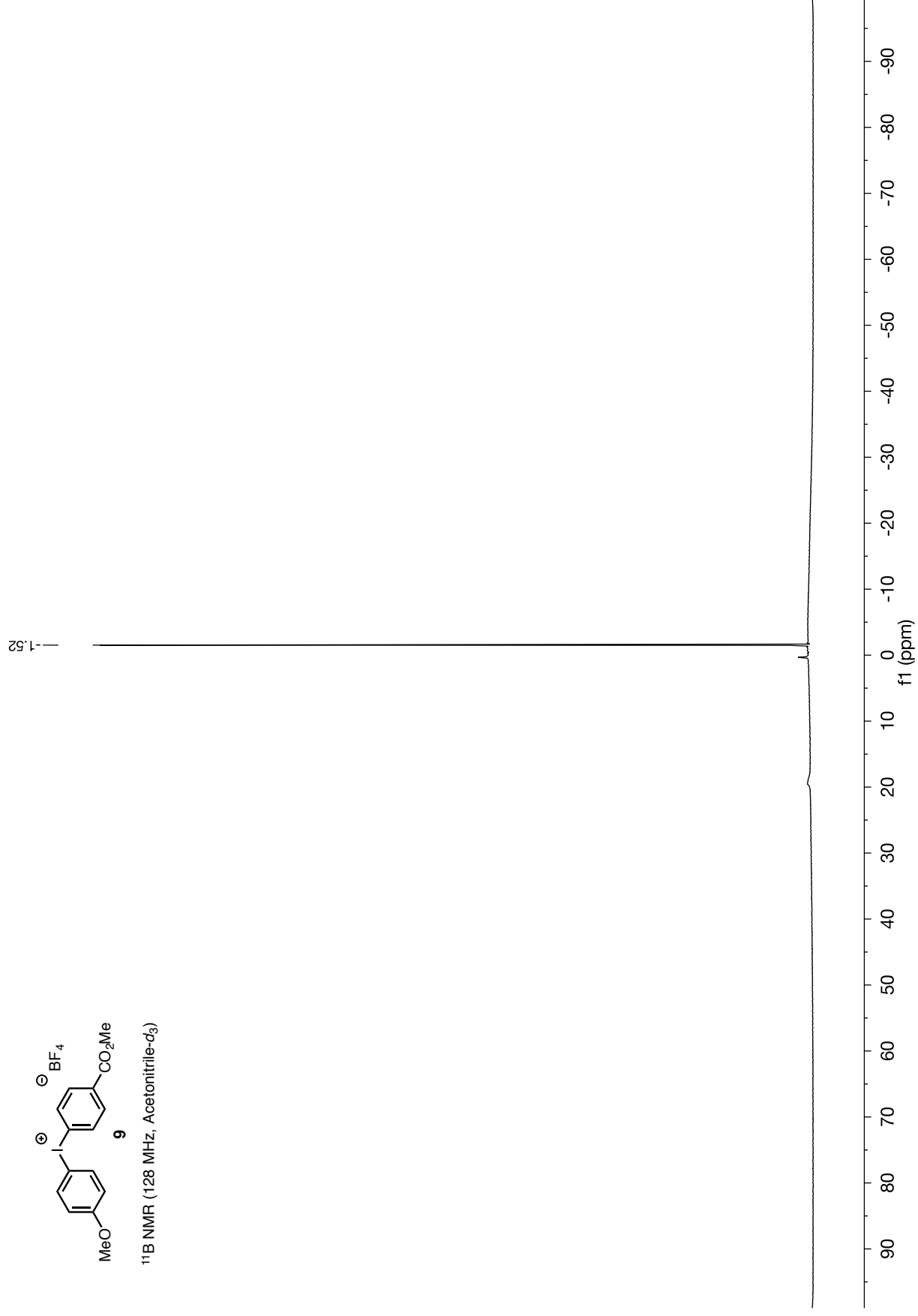


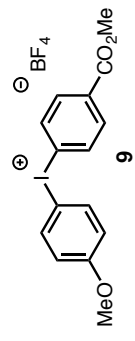
¹³C NMR (101 MHz, Acetonitrile-*d*₃)





¹¹B NMR (128 MHz, Acetonitrile-*d*₃)



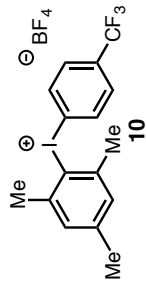


^{19}F NMR (376 MHz, Acetonitrile- d_3)

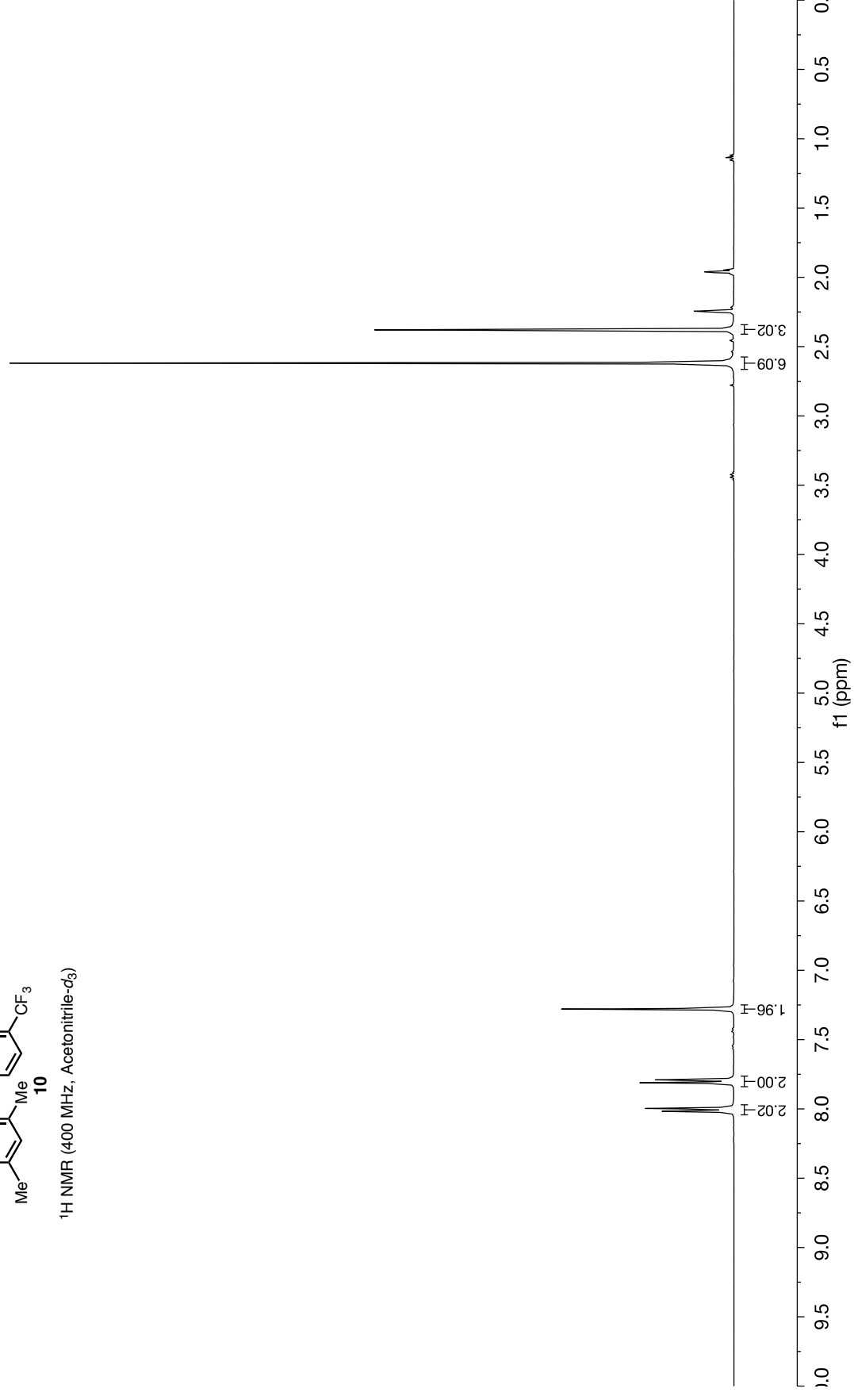
— -150.47

10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210

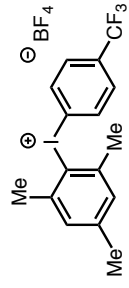
f1 (ppm)



¹H NMR (400 MHz, Acetonitrile-d₃)

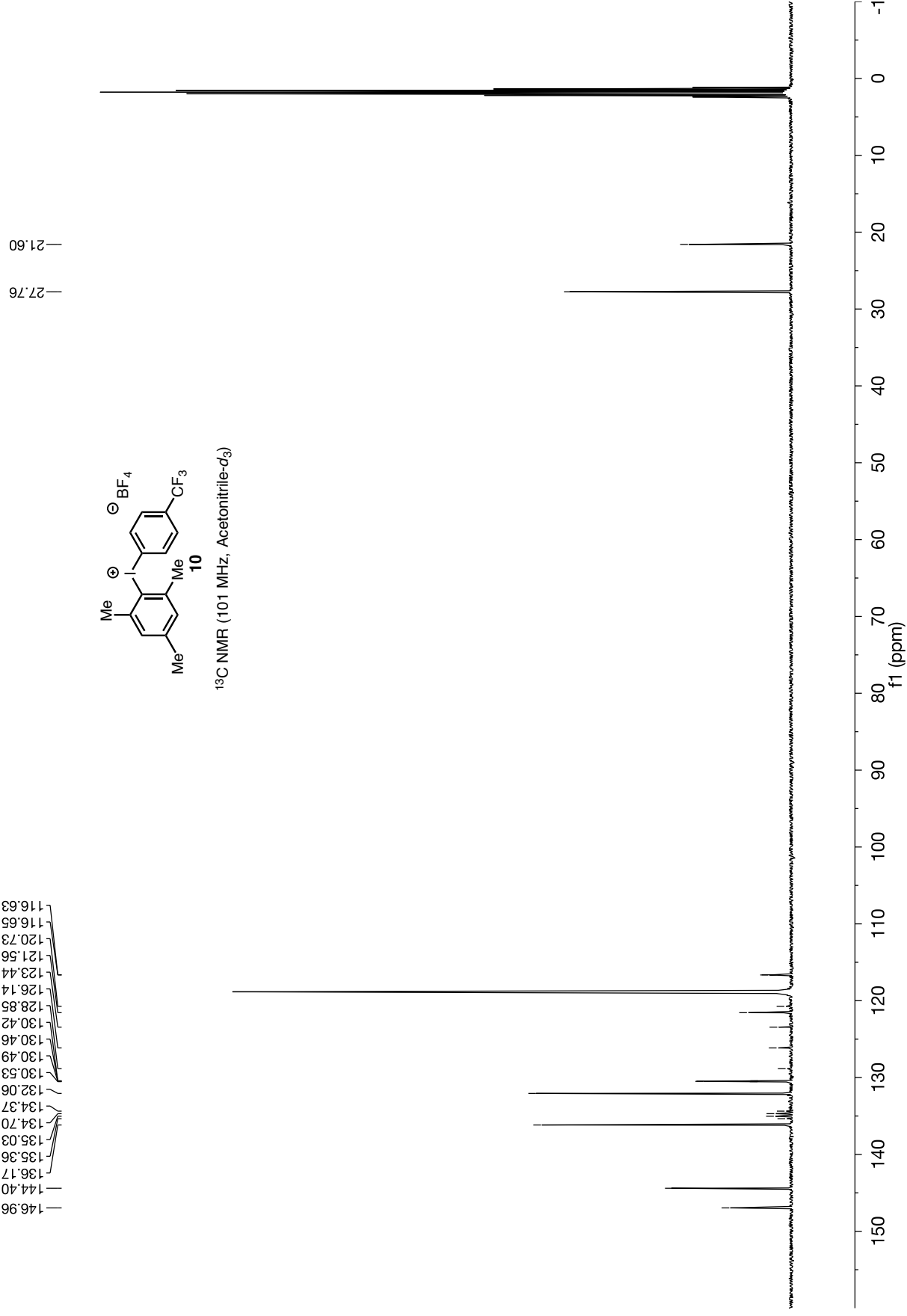


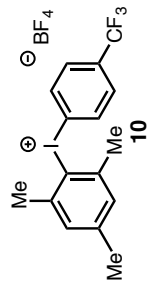
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 144.40
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 135.36
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 134.70
 134.37
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 116.63



10

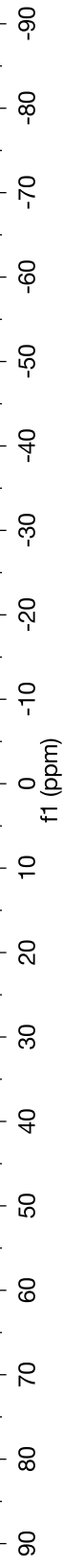
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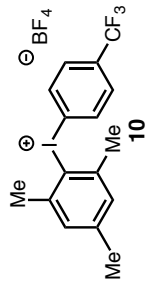




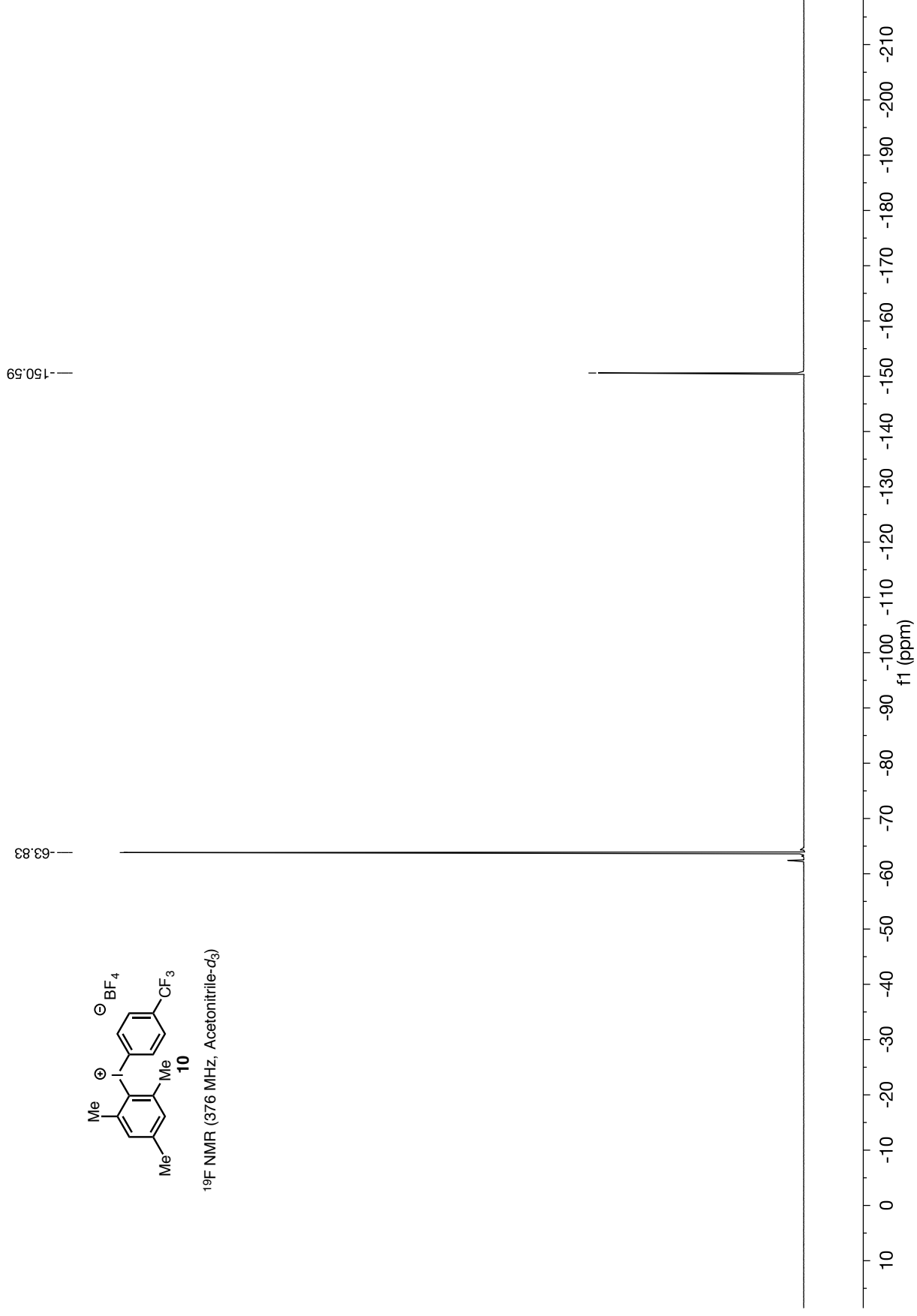
^{11}B NMR (128 MHz, Acetonitrile- d_3)

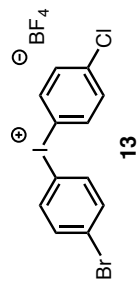
— -1.54



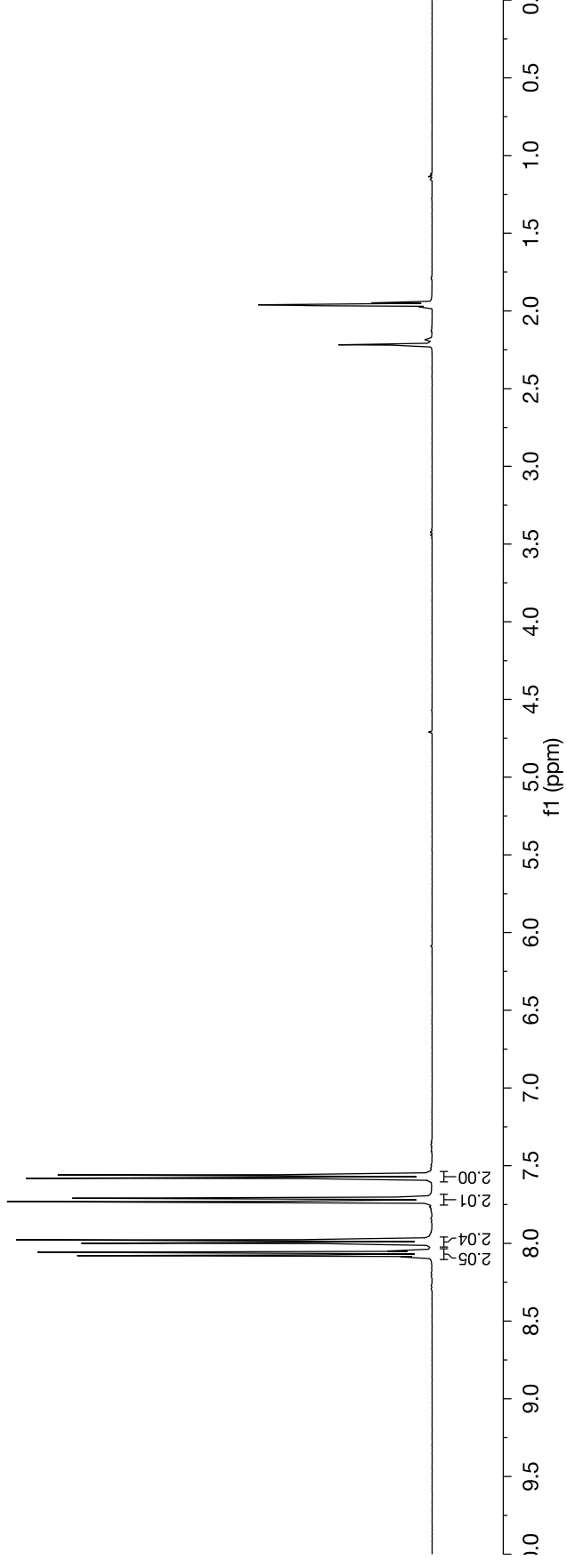


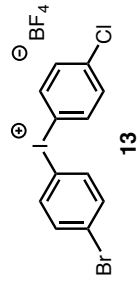
^{19}F NMR (376 MHz, Acetonitrile- d_3)



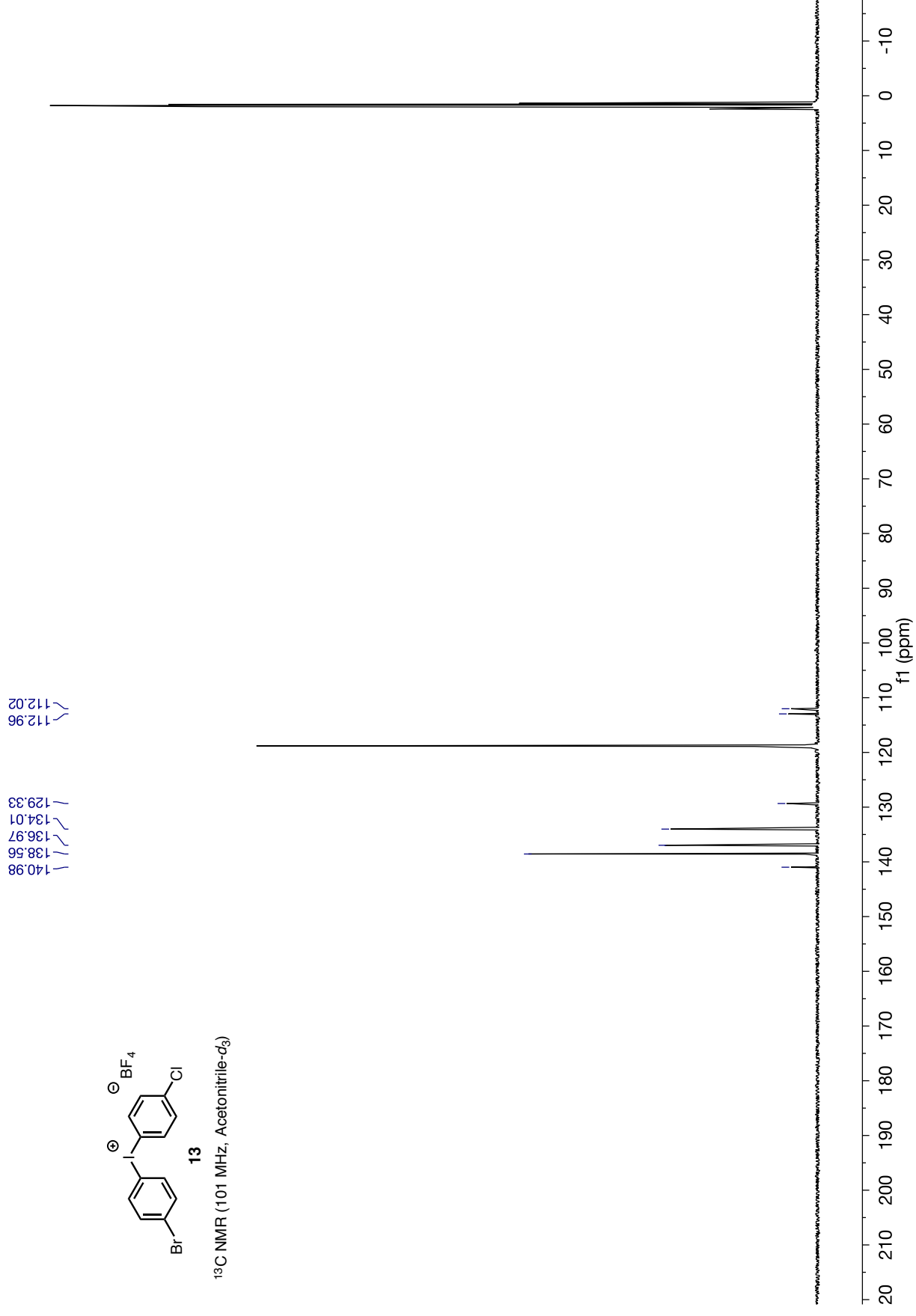


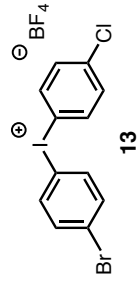
¹H NMR (400 MHz, Acetonitrile-*d*₃)





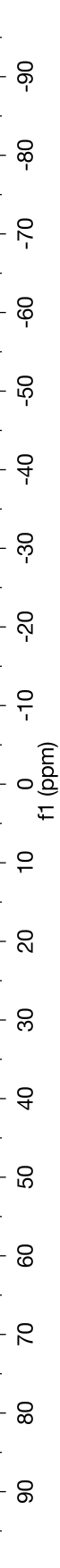
¹³C NMR (101 MHz, Acetonitrile-d₃)

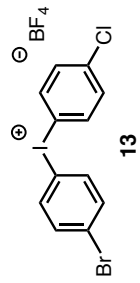




¹¹B NMR (128 MHz, Acetonitrile-*d*₃)

— -1.54

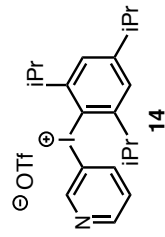




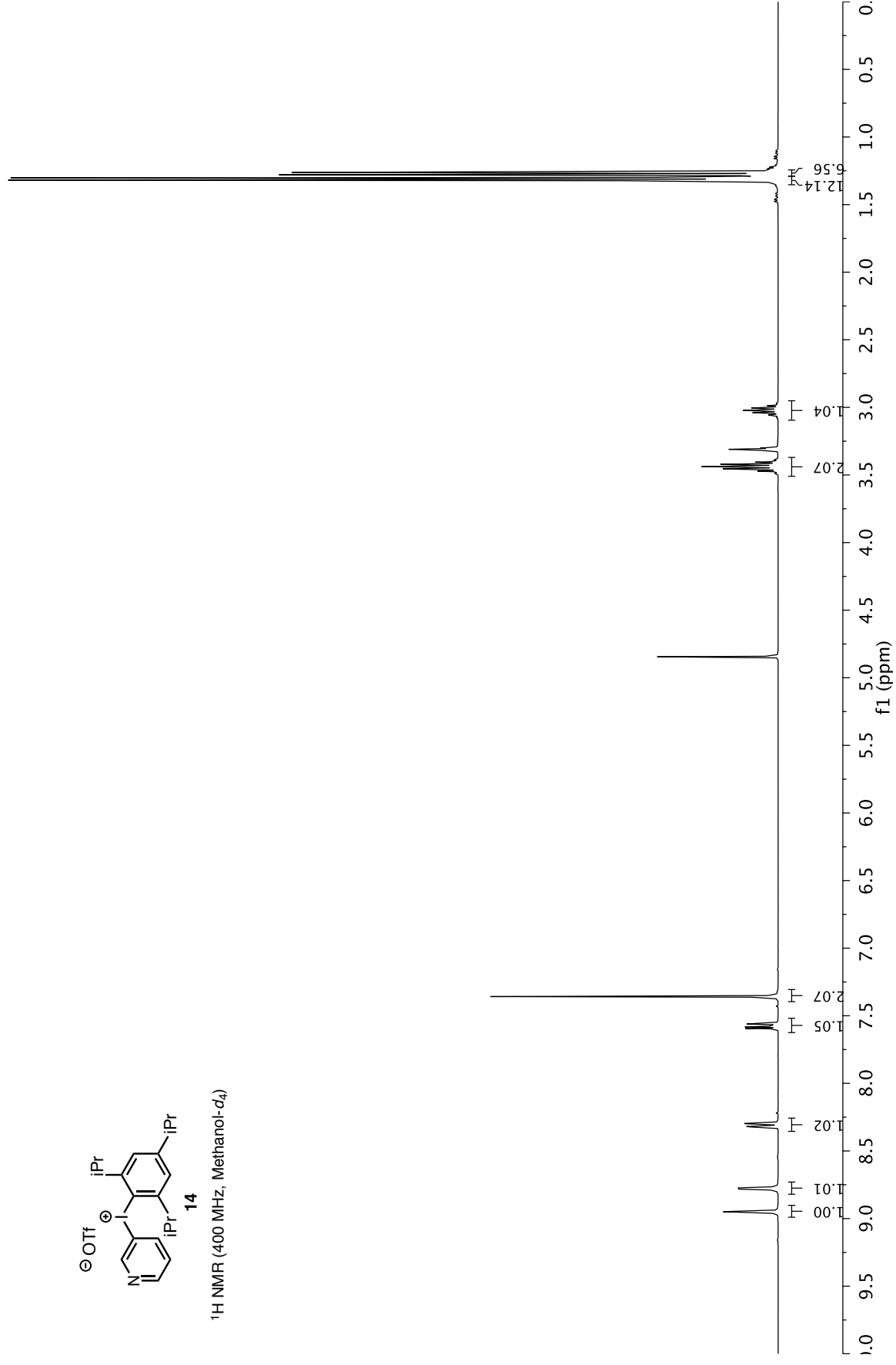
^{19}F NMR (376 MHz, Acetonitrile- d_3)

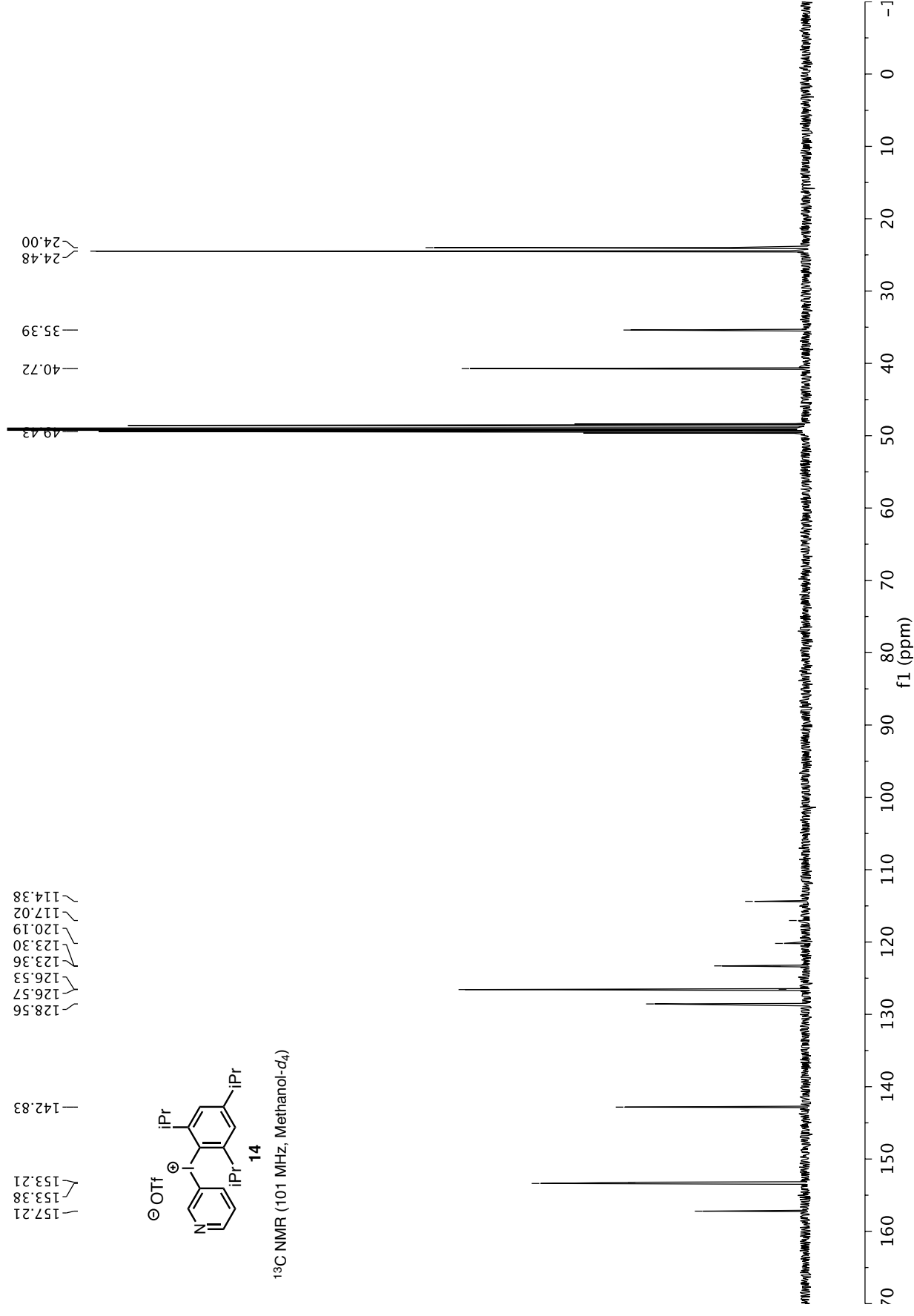
— -150.69

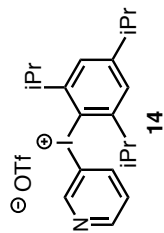
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)



^1H NMR (400 MHz, Methanol- d_4)



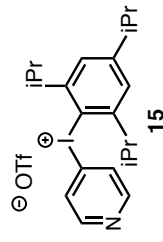




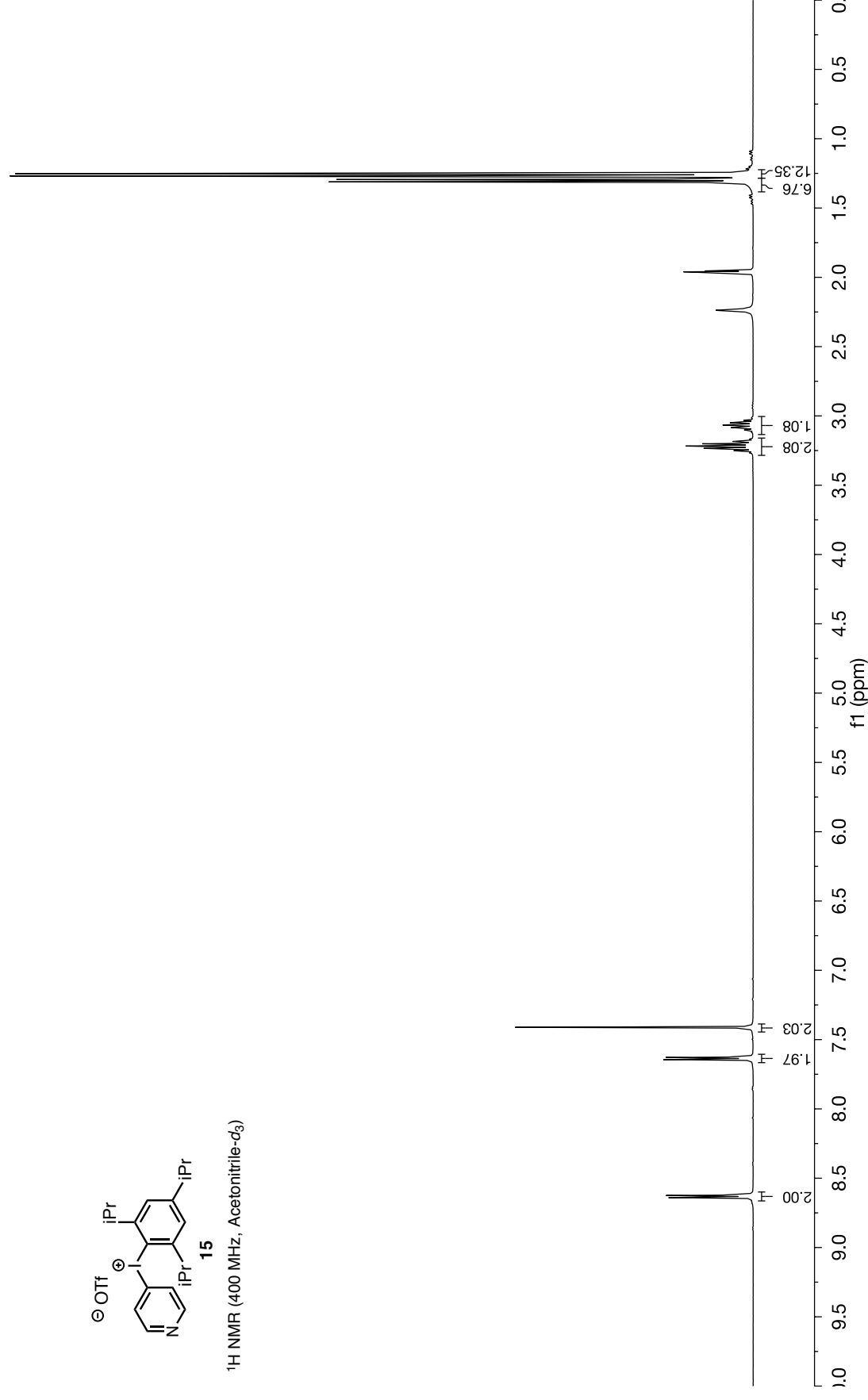
¹⁹F NMR (376 MHz, Methanol-*d*₄)

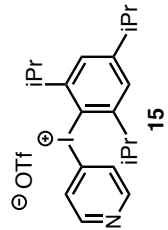
96.79, 96.79

10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

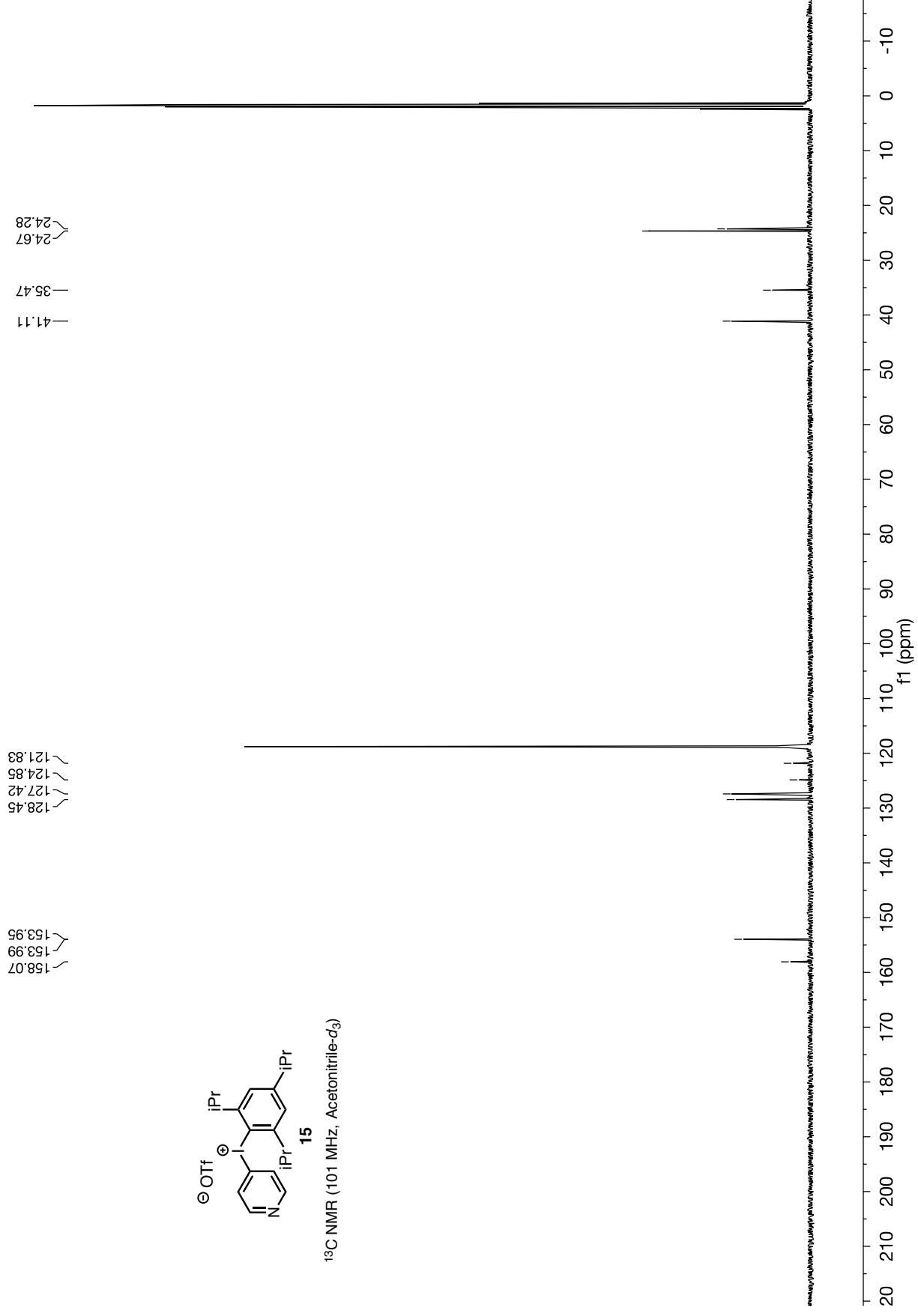


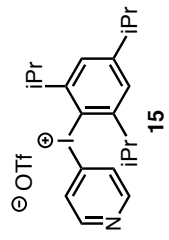
¹H NMR (400 MHz, Acetonitrile-*d*₃)





^{13}C NMR (101 MHz, Acetonitrile- d_3)





¹⁹F NMR (376 MHz, Acetonitrile-*d*₃)

— -79.28

10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)