Supporting Information

Integrating Continuous-Flow Electrochemistry and Photochemistry for the Synthesis of Acridinium Photocatalysts via Site-Selective C–H Alkylation

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1. General Information

Flash column chromatography was performed with silica gel (230–400 mesh). NMR spectra were recorded on Bruker AV-400, Bruker AV-500 and Quantum-Iplus 400 instruments. Data were reported as chemical shifts in ppm relative to CD_3CN (1.94 ppm), $CDCl_3$ (7.27 ppm) for ^1H, and CD_3CN (118.3 ppm), $CDCl_3$ (77.2 ppm) for ^{13}C, respectively. All the acridinium salts have been reported.[1]

2. General Procedure for the Monoalkylation of Acridinium Salts

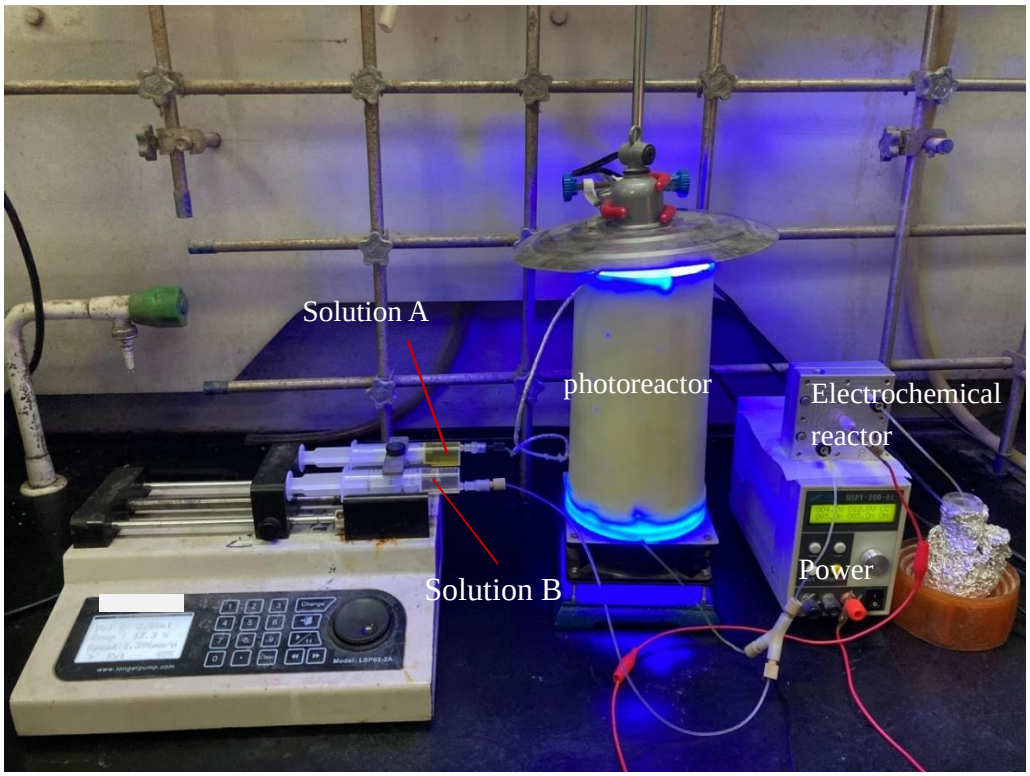


Figure S1. Setup for monoalkylation of acridinium salts.

Degassed solution (solution A) of $MesAcrClO_4$ (**1**, 0.05 M) and RBF_3K (1.5 equiv, 0.075 M) in CH_3CN-H_2O (5:1) was pumped at 0.2 mL min^{-1} into the photoreactor (PFA tubing, O.D. 1.6 mm, I.D. 0.9 mm, 3 meters, volume = 2 mL) and then mixed with solution B [TEMPO (0.01 M), in CH_3CN-pyridine-H_2O (5:6:1), 0.2 mL/min] before entering the electrochemical flow reactor (graphite anode, Pt cathode, interelectrode distance = 250 μm, volume = 250 μL, constant current = 85 mA) (Figure S1). The outlet solution was collected into a receiving flask for 30 min. The collected solution was diluted with DCM and water. The aqueous phase was extracted with DCM several times until a colorless organic phase. The combined organic solution was concentrated under reduced pressure. The residue was dissolved in DCM and washed with hydrochloric acid (1 N, 100 mL), deionized water and KPF_6 aqueous

solution (0.2 M, 3 × 100 mL). In each wash, the aqueous phase was extracted with DCM several times until the organic phase was colorless. The combined organic solution was concentrated under reduced pressure. The residue was chromatographed through silica gel eluting with DCM/acetone to give the product.

3. General Procedure for the Dialkylation of Acridinium Salts

Here, the syringe pump was replaced with a peristatic pump that was convenient for long time operation. A degassed solution (solution A, 0.4 mL/min) of $MesAcrClO_4$ (**1**, 0.025 M, 0.3 mmol) and RBF_3K (1.5 equiv, 0.038 M) in CH_3CN-H_2O (5:1) was pumped into the photoreactor and then mixed with solution B [TEMPO (0.005 M) and Et_3N (0.15 M) in CH_3CN-acetone-H_2O (5:6:1), 0.4 mL/min] before entering the electrochemical reactor (constant current = 85 mA). The collected outlet solution was diluted with DCM and washed with KPF_6 aqueous solution (0.2 M, 100 mL). The aqueous phase was extracted with DCM several times until the organic phase was colorless. The combined organic solution was washed by deionized water. The water was also extracted with DCM several times until the organic phase was colorless. The combined organic solvent was concentrated under reduced pressure. The residue was dried in vacuo and then washed with hexanes to afford the crude 3-alkyl acridinium salt. Without further purification, the crude 3-alkyl acridinium salt was subjected to a second alkylation by following the above procedure to produce the final dialkylated products, which were purified by silica gel chromatography eluting with DCM/acetone.

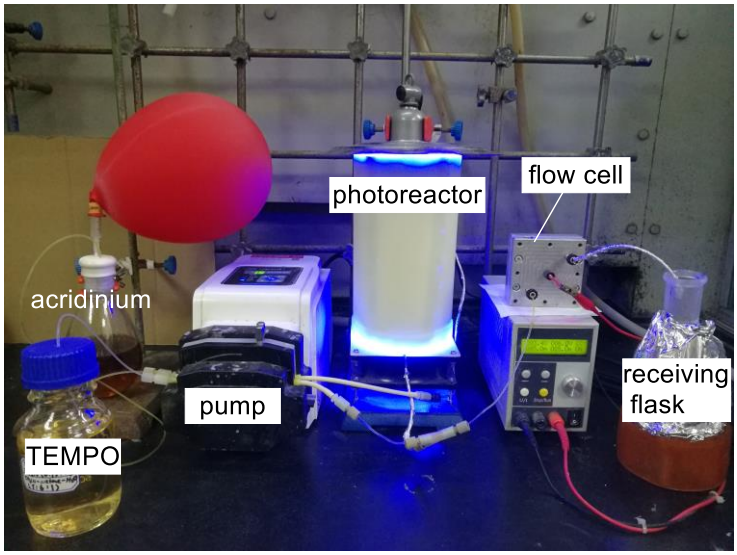
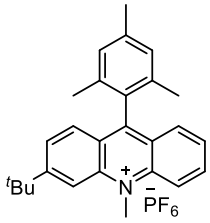


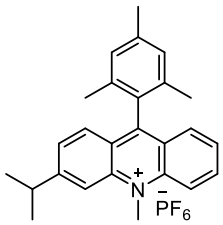
Figure S2. Reaction setup for the gram scale synthesis.

For the gram scale synthesis of **19** and **20** (Figrue S2), 2 grams of **1** was employed. For the synthesis of **19**, the crude product was dissolved in 10 mL of toluene. 30 mL of Et_2O was added to precipitate the acridinium salt. The mixture was sonicated for half an hour and filtered. The filter cake was washed with toluene-Et_2O (1:3, 3 × 5 mL) and Et_2O. **19** was obtained as dark yellow solid (1.35 g, 51% yield). For the synthesis of **20**, the crude was recrystallized in 10 mL of methanol. The filter cake was collected. The filtrate was concentrated and recrystallized. **20** was obtained as a yellow solid (1.76 g, 64% yield).

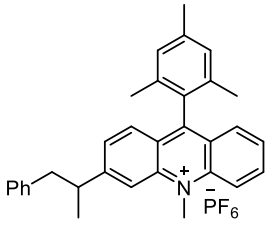
4. Characterization Data for Acridinium Salts



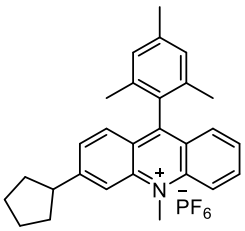
3-(*tert*-Butyl)-9-mesityl-10-methylacridin-10-ium hexafluorophosphate (**3**). ^{1}H NMR (400 MHz, CD_3CN) δ 8.61 (d, *J* = 9.2 Hz, 1H), 8.43 – 8.32 (m, 2H), 7.97 (dd, *J* = 9.1, 1.6 Hz, 1H), 7.82 (d, *J* = 4.1 Hz, 2H), 7.75 (d, *J* = 9.1 Hz, 1H), 7.24 (s, 2H), 4.84 (s, 3H), 2.47 (s, 3H), 1.71 (s, 6H), 1.53 (s, 9H); ^{13}C NMR (101 MHz, CD_3CN) δ 164.9, 162.1, 143.0, 142.6, 141.0, 139.2, 136.9, 130.6, 129.7, 129.6, 129.4, 128.9, 128.6, 126.6, 125.5, 119.8, 114.8, 39.3, 37.8, 30.7, 21.3, 19.9; ^{19}F NMR (376 MHz, CD_3CN) δ -71.9, -73.7.



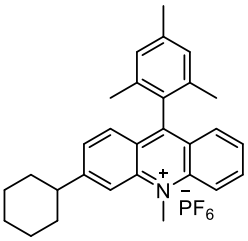
3-Isopropyl-9-mesityl-10-methylacridin-10-ium hexafluorophosphate (**6**). ^{1}H NMR (400 MHz, CD_3CN) δ 8.60 (d, *J* = 9.2 Hz, 1H), 8.40 – 8.33 (m, 2H), 7.86 – 7.72 (m, 4H), 7.23 (s, 2H), 4.82 (s, 3H), 3.49 – 3.31 (m, 1H), 2.47 (s, 3H), 1.71 (s, 6H), 1.44 (d, *J* = 6.9 Hz, 6H); ^{13}C NMR (101 MHz, CD_3CN) δ 162.9, 162.3, 143.2, 142.5, 141.0, 139.3, 136.9, 130.6, 129.8, 129.8, 129.6, 129.3, 128.9, 126.5, 125.9, 119.7, 116.2, 39.3, 36.5, 23.3, 21.3, 19.9; ^{19}F NMR (376 MHz, CD_3CN) δ -71.9, -73.7.



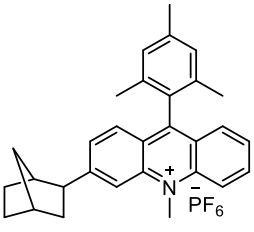
9-Mesityl-10-methyl-3-(1-phenylpropan-2-yl)acridin-10-ium hexafluorophosphate (**7**). ^{1}H NMR (400 MHz, CD_3CN) δ 8.59 (d, *J* = 9.3 Hz, 1H), 8.40 – 8.28 (m, 2H), 7.80 (d, *J* = 4.0 Hz, 2H), 7.76 – 7.68 (m, 2H), 7.28 – 7.09 (m, 7H), 4.76 (s, 3H), 3.64 – 3.48 (m, 1H), 3.08 (dd, *J* = 7.6, 1.8 Hz, 2H), 2.47 (s, 3H), 1.70 (s, 6H), 1.43 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, CD_3CN) δ 162.3, 161.0, 142.9, 142.5, 141.0, 140.6, 139.3, 136.8, 130.6, 130.1, 129.7, 129.6, 129.5, 129.2, 127.1, 126.4, 125.8, 119.7, 117.3, 44.1, 44.0, 39.3, 21.3, 21.0, 19.9; ^{19}F NMR (376 MHz, CD_3CN) δ -71.7, -73.6.



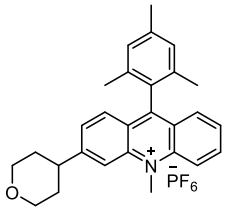
3-Cyclopentyl-9-mesityl-10-methylacridin-10-ium hexafluorophosphate (**8**). ^{1}H NMR (400 MHz, CD_3CN) δ 8.60 (d, *J* = 9.2 Hz, 1H), 8.41 – 8.30 (m, 2H), 7.85 – 7.70 (m, 4H), 7.23 (s, 2H), 4.80 (s, 3H), 3.55 – 3.43 (m, 1H), 2.46 (s, 3H), 2.30 – 2.17 (m, 4H), 1.88 – 1.77 (m, 4H), 1.70 (s, 6H); ^{13}C NMR (101 MHz, CD_3CN) δ 161.3, 160.3, 142.2, 141.6, 140.2, 138.4, 136.0, 129.8, 128.9, 128.9, 128.8, 128.8, 128.0, 125.6, 125.0, 118.9, 115.9, 47.4, 38.4, 34.5, 25.7, 20.4, 19.1; ^{19}F NMR (376 MHz, CD_3CN) δ -71.9, -73.7.



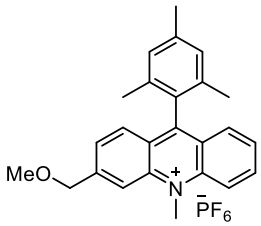
3-Cyclohexyl-9-mesityl-10-methylacridin-10-ium hexafluorophosphate (**9**). ^{1}H NMR (400 MHz, CD_3CN) δ 8.59 (d, *J* = 9.2 Hz, 1H), 8.35 (q, *J* = 5.2, 4.7 Hz, 2H), 7.84 – 7.70 (m, 4H), 7.23 (s, 2H), 4.80 (s, 3H), 3.04 (s, 1H), 2.46 (s, 3H), 2.04 – 1.97 (m, 2H), 1.96 – 1.90 (m, 2H), 1.86 – 1.78 (m, 1H), 1.70 (s, 8H), 1.52 (dt, *J* = 12.7, 3.3 Hz, 2H), 1.39 (s, 1H); ^{13}C NMR (101 MHz, CD_3CN) δ 161.4, 161.1, 142.3, 141.7, 140.1, 138.4, 136.0, 129.8, 128.9, 128.8, 128.8, 128.0, 125.6, 125.0, 118.9, 115.7, 45.9, 38.4, 33.3, 26.3, 25.6, 20.4, 19.0; ^{19}F NMR (376 MHz, CD_3CN) δ -71.9, -73.7.



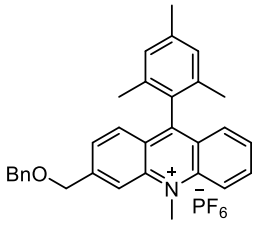
3-(Bicyclo[2.2.1]heptan-2-yl)-9-mesityl-10-methylacridin-10-ium hexafluorophosphate (**10**). ^{1}H NMR (400 MHz, CD_3CN) δ 8.59 (d, *J* = 9.2 Hz, 1H), 8.39 – 8.30 (m, 1H), 8.29 (s, 1H), 7.80 (d, *J* = 4.1 Hz, 2H), 7.72 (d, *J* = 1.8 Hz, 2H), 7.23 (s, 2H), 4.80 (s, 3H), 3.28 – 3.18 (m, 1H), 2.61 (d, *J* = 3.9 Hz, 1H), 2.50 – 2.43 (m, 4H), 2.03 – 1.95 (m, 1H), 1.87 – 1.79 (m, 1H), 1.70 (s, 9H), 1.57 – 1.47 (m, 1H), 1.43 – 1.31 (m, 2H); ^{13}C NMR (101 MHz, CD_3CN) δ 162.1, 161.5, 143.1, 142.5, 141.0, 139.2, 136.9, 130.6, 130.4, 129.7, 129.7, 129.6, 128.8, 126.4, 125.6, 119.7, 116.0, 49.7, 43.7, 39.4, 39.3, 38.0, 37.0, 31.1, 29.2, 21.3, 19.9; ^{19}F NMR (376 MHz, CD_3CN) δ -71.9, -73.7.



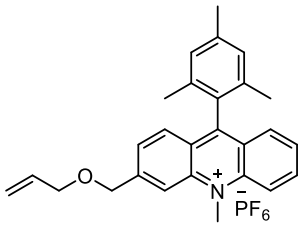
9-Mesityl-10-methyl-3-(tetrahydro-2H-pyran-4-yl)acridin-10-ium hexafluorophosphate (**11**). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.65 (d, *J* = 9.3 Hz, 1H), 8.47 (s, 1H), 8.41 – 8.31 (m, 1H), 7.88 – 7.72 (m, 3H), 7.68 (d, *J* = 8.9 Hz, 1H), 7.15 (s, 2H), 4.98 (s, 3H), 4.11 (dt, *J* = 11.6, 3.1 Hz, 2H), 3.73 – 3.57 (m, 2H), 3.47 – 3.28 (m, 1H), 2.48 (s, 3H), 1.98– 1.93 (m, 4H), 1.73 (s, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.3, 159.3, 142.1, 141.5, 140.4, 139.1, 136.0, 129.5, 129.4, 129.1, 128.2, 128.0, 125.7, 125.1, 118.9, 116.7, 67.9, 43.0, 38.6, 33.0, 21.4, 20.1; ^{19}F NMR (376 MHz, $CDCl_3$) δ -72.1, -74.0.



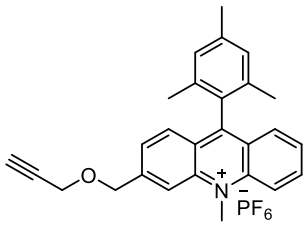
9-Mesityl-3-(methoxymethyl)-10-methylacridin-10-ium hexafluorophosphate (**12**). ^{1}H NMR (500 MHz, CD_3CN) δ 8.61 (d, *J* = 9.2 Hz, 1H), 8.53 (s, 1H), 8.41 – 8.33 (m, 1H), 7.86 – 7.73 (m, 4H), 7.24 (s, 2H), 4.87 (s, 2H), 4.82 (s, 3H), 3.54 (s, 3H), 2.47 (s, 3H), 1.71 (s, 6H); ^{13}C NMR (126 MHz, CD_3CN) δ 162.7, 152.7, 143.0, 142.6, 141.1, 139.5, 136.9, 130.6, 129.9, 129.8, 129.6, 129.1, 128.3, 126.8, 126.4, 119.8, 116.4, 74.2, 59.3, 39.5, 21.3, 19.9; ^{19}F NMR (471 MHz, CD_3CN) δ -72.1, -73.6.



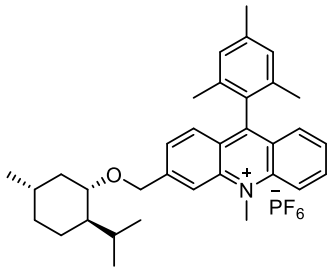
3-((Benzyloxy)methyl)-9-mesityl-10-methylacridin-10-ium hexafluorophosphate (**13**). ^{1}H NMR (400 MHz, CD_3CN) δ 8.61 (d, *J* = 9.3 Hz, 1H), 8.55 (s, 1H), 8.42 – 8.33 (m, 1H), 7.83 (d, *J* = 4.1 Hz, 2H), 7.80 (d, *J* = 0.9 Hz, 2H), 7.47 (d, *J* = 7.0 Hz, 2H), 7.43 – 7.37 (m, 2H), 7.37 – 7.30 (m, 1H), 7.24 (s, 2H), 4.98 (s, 2H), 4.80 (s, 3H), 4.75 (s, 2H), 2.47 (s, 3H), 1.70 (s, 6H); ^{13}C NMR (101 MHz, CD_3CN) δ 162.7, 152.7, 142.9, 142.6, 141.1, 139.5, 139.0, 136.9, 130.6, 129.9, 129.8, 129.6, 129.4, 129.1, 128.9, 128.8, 128.4, 126.7, 126.4, 119.8, 116.6, 73.8, 72.0, 39.4, 21.3, 19.9; ^{19}F NMR (376 MHz, CD_3CN) δ -71.9, -73.8.



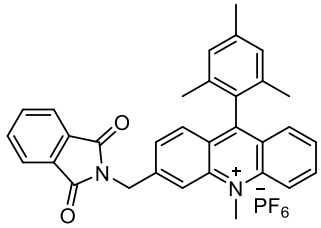
3-((Allyloxy)methyl)-9-mesityl-10-methylacridin-10-ium hexafluorophosphate (**14**). ^{1}H NMR (400 MHz, CD_3CN) δ 8.61 (d, *J* = 9.2 Hz, 1H), 8.54 (s, 1H), 8.42 – 8.33 (m, 1H), 7.89 – 7.73 (m, 4H), 7.24 (s, 2H), 6.13 – 6.00 (m, 1H), 5.46 – 5.36 (m, 1H), 5.31 – 5.22 (m, 1H), 4.94 (s, 2H), 4.81 (s, 3H), 4.22 (dt, *J* = 5.5, 1.5 Hz, 2H), 2.47 (s, 3H), 1.71 (s, 6H); ^{13}C NMR (101 MHz, CD_3CN) δ 162.7, 152.8, 143.0, 142.6, 141.1, 139.5, 136.9, 135.6, 130.6, 129.9, 129.8, 129.6, 129.1, 128.4, 126.7, 126.4, 119.8, 117.7, 116.4, 72.7, 71.9, 39.4, 21.3, 19.9; ^{19}F NMR (376 MHz, CD_3CN) δ -71.9, -73.8.



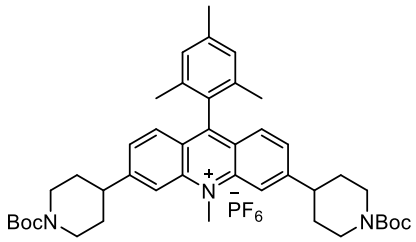
9-Mesityl-10-methyl-3-((prop-2-yn-1-yloxy)methyl)acridin-10-ium hexafluorophosphate (**15**). ^{1}H NMR (500 MHz, CD_3CN) δ 8.61 (d, *J* = 9.2 Hz, 1H), 8.55 (s, 1H), 8.41 – 8.34 (m, 1H), 7.89 – 7.75 (m, 4H), 7.24 (s, 2H), 5.01 (s, 2H), 4.81 (s, 3H), 4.40 (d, *J* = 2.4 Hz, 2H), 2.84 (t, *J* = 2.4 Hz, 1H), 2.47 (s, 3H), 1.70 (s, 6H); ^{13}C NMR (126 MHz, CD_3CN) δ 162.8, 151.8, 142.9, 142.7, 141.1, 139.6, 136.9, 130.6, 130.0, 129.9, 129.7, 129.2, 128.4, 126.8, 126.5, 119.8, 116.9, 80.2, 76.7, 71.6, 59.1, 39.5, 21.3, 19.9; ^{19}F NMR (471 MHz, CD_3CN) δ -72.2, -73.7.



3-((((1*S*,2*R*,5*S*)-2-Isopropyl-5-methylcyclohexyl)oxy)methyl)-9-mesityl-10-methyl acridin-10-ium hexafluorophosphate (**16**). ^{1}H NMR (400 MHz, CD_3CN) δ 8.59 (d, *J* = 9.2 Hz, 1H), 8.53 (s, 1H), 8.41 – 8.32 (m, 1H), 7.83 (d, *J* = 3.9 Hz, 2H), 7.78 (s, 2H), 7.23 (s, 2H), 5.07 (d, *J* = 14.7 Hz, 1H), 4.82 (d, *J* = 15.3 Hz, 1H), 4.79 (s, 3H), 3.44 – 3.32 (m, 1H), 2.47 (s, 3H), 2.37 – 2.27 (m, 1H), 1.76 – 1.62 (m, 8H), 1.51 – 1.30 (m, 3H), 1.13 – 0.86 (m, 9H), 0.77 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, CD_3CN) δ 162.6, 153.7, 142.9, 142.5, 141.1, 139.5, 136.9, 130.6, 129.8, 129.7, 129.6, 129.1, 128.7, 126.7, 126.3, 119.7, 116.4, 80.7, 70.1, 49.2, 41.0, 39.3, 35.2, 32.2, 26.7, 24.1, 22.5, 21.2, 21.2, 19.9, 16.6; ^{19}F NMR (376 MHz, CD_3CN) δ -71.9, -73.8.

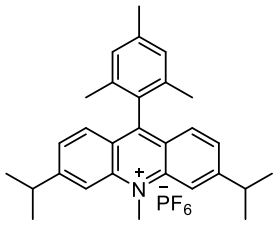


3-((1,3-Dioxoisoindolin-2-yl)methyl)-9-mesityl-10-methylacridin-10-ium hexafluorophosphate (**17**). ^{1}H NMR (500 MHz, CD_3CN) δ 8.58 (d, *J* = 9.3 Hz, 1H), 8.52 (s, 1H), 8.41 – 8.33 (m, 1H), 7.94 – 7.88 (m, 2H), 7.88 – 7.74 (m, 6H), 7.21 (s, 2H), 5.22 (s, 2H), 4.78 (s, 3H), 2.45 (s, 3H), 1.68 (s, 6H); ^{13}C NMR (126 MHz, CD_3CN) δ 169.1, 163.0, 149.9, 142.9, 142.8, 141.2, 139.8, 136.9, 135.6, 133.1, 130.5, 129.9, 129.7, 129.3, 128.8, 126.9, 126.3, 124.3, 119.8, 117.7, 42.7, 39.6, 21.3, 19.9; ^{19}F NMR (471 MHz, CD_3CN) δ -72.2, -73.7.

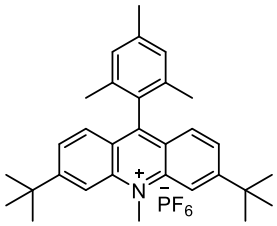


3,6-Bis(1-(*tert*-butoxycarbonyl)piperidin-4-yl)-9-mesityl-10-methylacridin-10-ium hexafluorophosphate (**18**). ^{1}H NMR (400 MHz, CD_3CN) δ 8.32 (s, 2H), 7.72 (s, 4H), 7.22 (s, 2H), 4.77 (s, 3H), 4.27 (d, *J* = 13.2 Hz, 4H), 3.26 – 3.13 (m, 2H), 2.91 (s, 4H), 2.46 (s, 3H), 1.99 – 1.92 (m, 4H), 1.87 – 1.70 (m, 4H), 1.69 (s, 6H), 1.46 (s, 18H); ^{13}C NMR (101 MHz, CD_3CN) δ 161.7, 159.3, 155.4, 143.0, 141.0, 136.8, 130.6,

129.9, 129.6, 129.3, 125.6, 116.8, 79.9, 44.6, 39.2, 33.1, 28.6, 21.3, 19.9; ^{19}F NMR (376 MHz, CD_3CN) δ -71.9, -73.8.



3,6-Diisopropyl-9-mesityl-10-methylacridin-10-ium hexafluorophosphate (**19**). ^{1}H NMR (400 MHz, CD_3CN) δ 8.34 (s, 2H), 7.78 – 7.68 (m, 4H), 7.22 (s, 2H), 4.78 (s, 3H), 3.47 – 3.28 (m, 2H), 2.46 (s, 3H), 1.70 (s, 6H), 1.43 (d, *J* = 6.9 Hz, 12H); ^{13}C NMR (101 MHz, CD_3CN) δ 162.3, 161.5, 143.0, 140.9, 136.8, 130.7, 129.8, 129.6, 128.8, 125.4, 116.2, 39.1, 36.5, 23.4, 21.3, 19.9; ^{19}F NMR (376 MHz, CD_3CN) δ -71.9, -73.8.

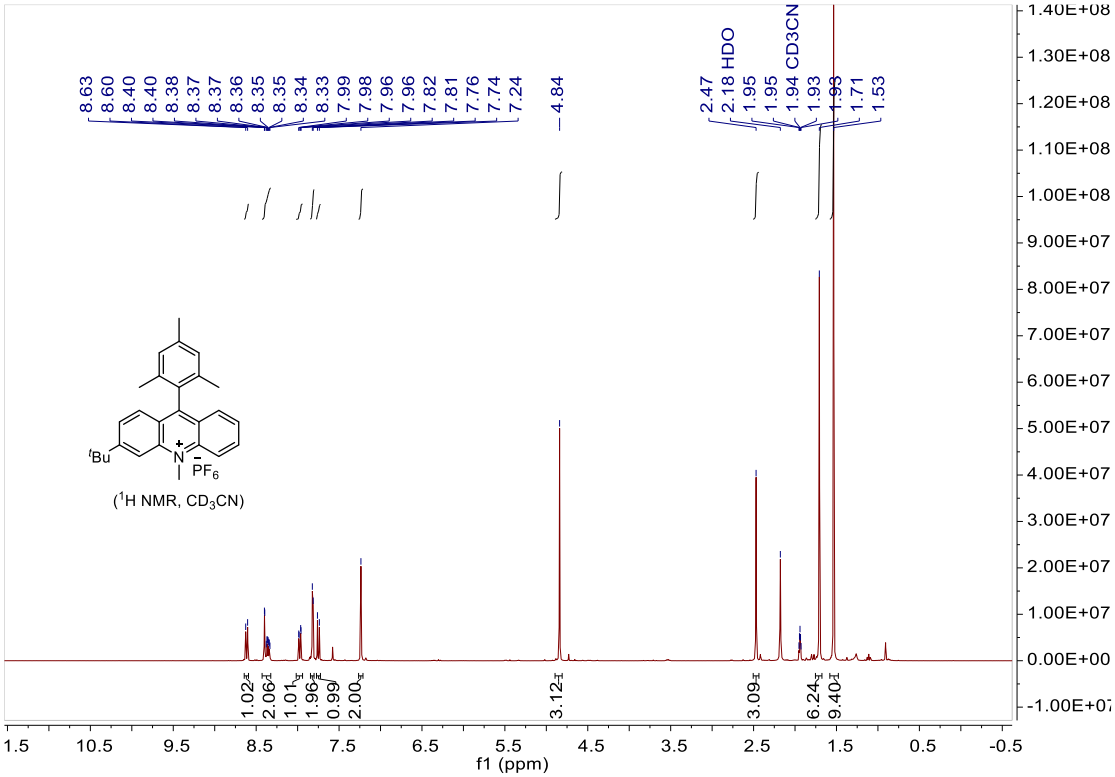


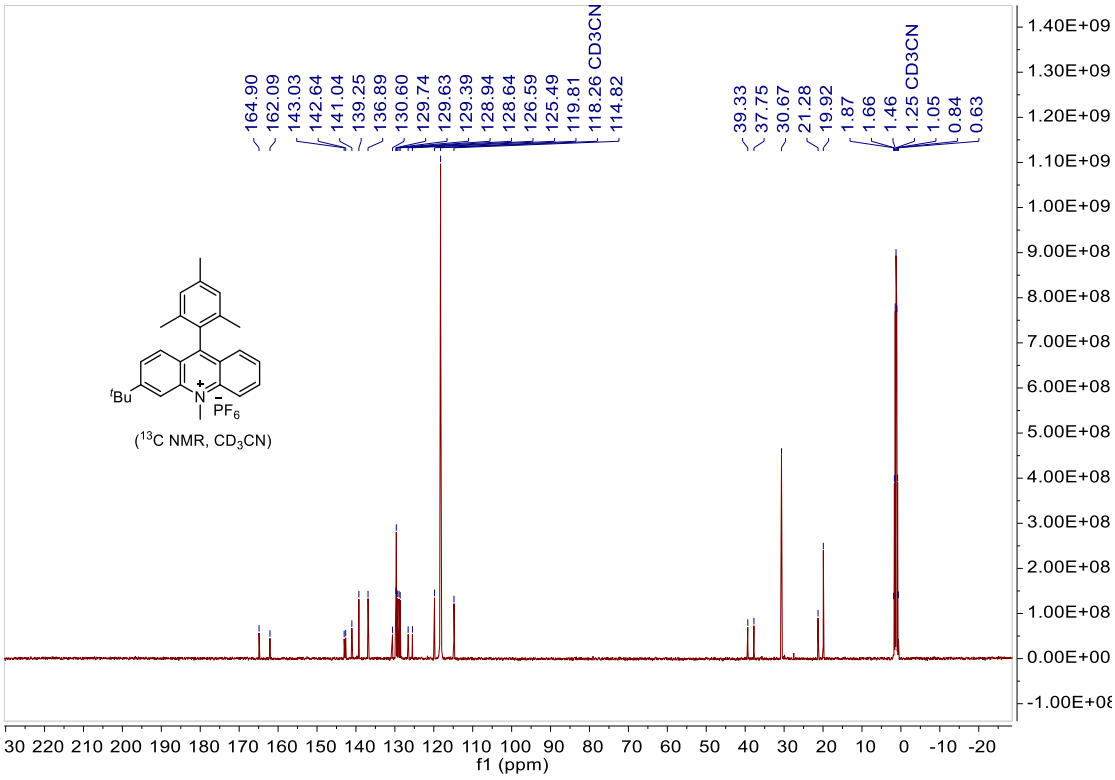
3,6-Di-*tert*-butyl-9-mesityl-10-methylacridin-10-ium hexafluorophosphate (**20**). ^{1}H NMR (400 MHz, CD_3CN) δ 8.39 (s, 2H), 7.94 (dd, *J* = 9.1, 1.6 Hz, 2H), 7.72 (d, *J* = 9.0 Hz, 2H), 7.23 (s, 2H), 4.84 (s, 3H), 2.47 (s, 3H), 1.71 (s, 6H), 1.53 (s, 18H); ^{13}C NMR (101 MHz, CD_3CN) δ 164.3, 161.0, 143.0, 141.0, 136.9, 130.6, 129.6, 129.3, 128.3, 125.1, 114.9, 39.2, 37.7, 30.7, 21.3, 19.9; ^{19}F NMR (376 MHz, CD_3CN) δ -71.9, -73.7.

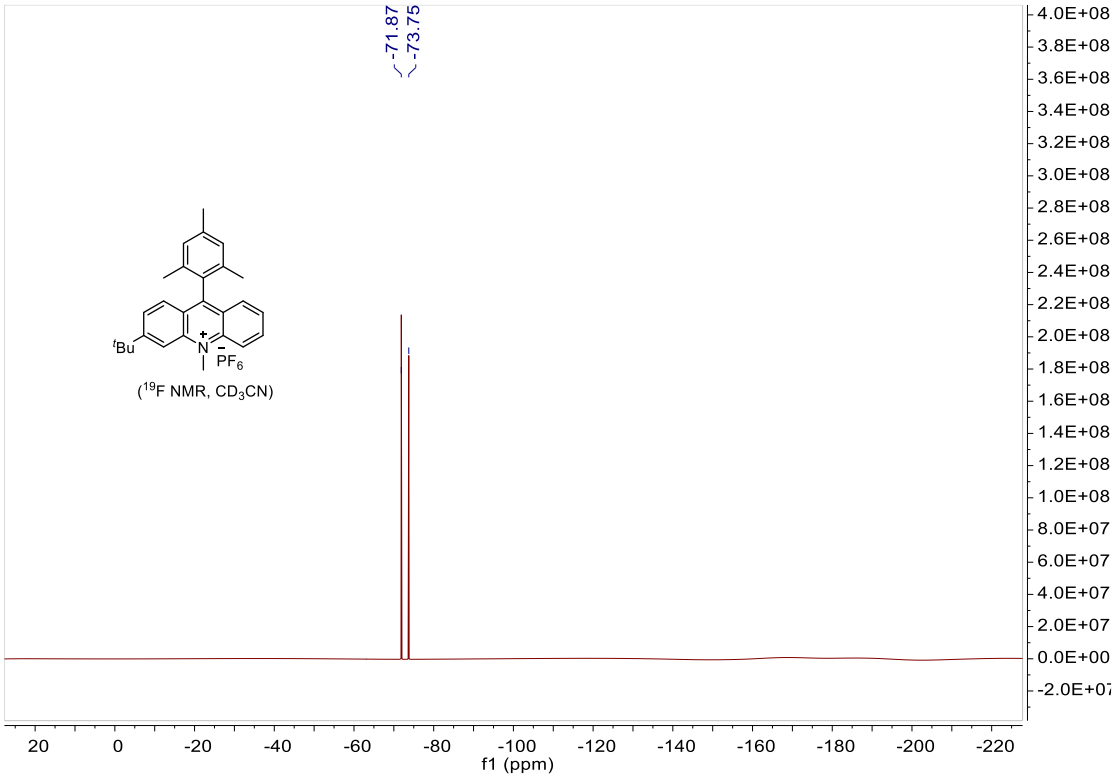
5. Reference

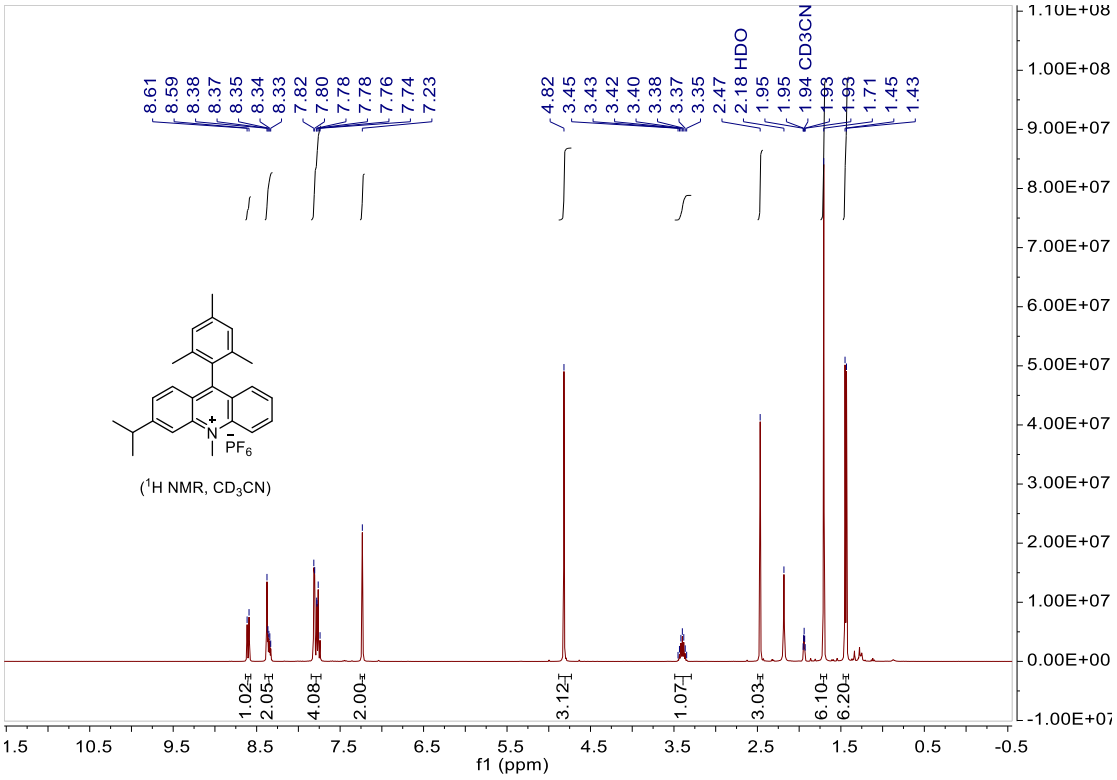
1. Yan, H.; Song, J.; Zhu, S. and Xu, H.-C. Synthesis of Acridinium Photocatalysts via Site-Selective C−H Alkylation. *CCS Chem.* **2021**, DOI: 10.31635/ccschem.021.202000743.

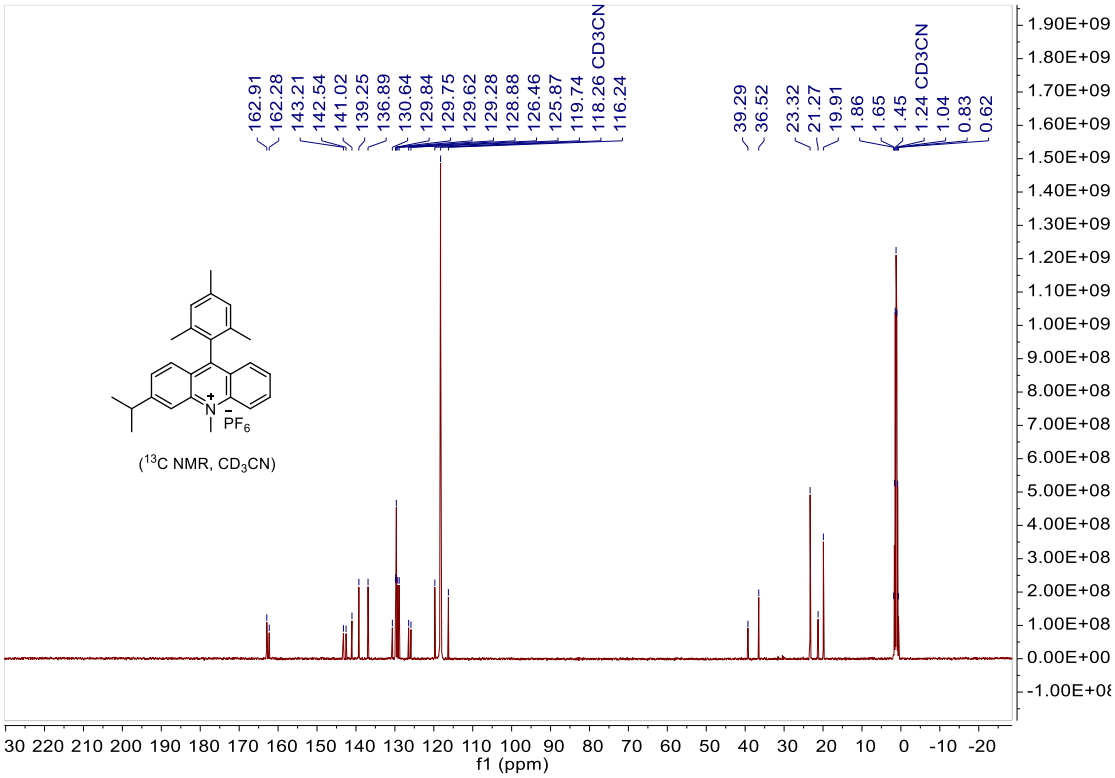
6. NMR Spectra






-71.87
-73.75
tBu
PF6
(19F NMR, CD3CN)
f1 (ppm)


8.61
8.59
8.38
8.37
8.35
8.34
8.33
7.82
7.80
7.78
7.78
7.76
7.74
7.23
4.82
3.45
3.43
3.42
3.40
3.38
3.37
3.35
2.47
2.18 HDO
1.95
1.95
1.94 CD3CN
1.93
1.93
1.71
1.45
1.43
PF6
(1H NMR, CD3CN)
1.02
2.05
4.08
2.00
3.12
1.07
3.03
6.10
6.20
f1 (ppm)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162.28
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139.25
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130.64
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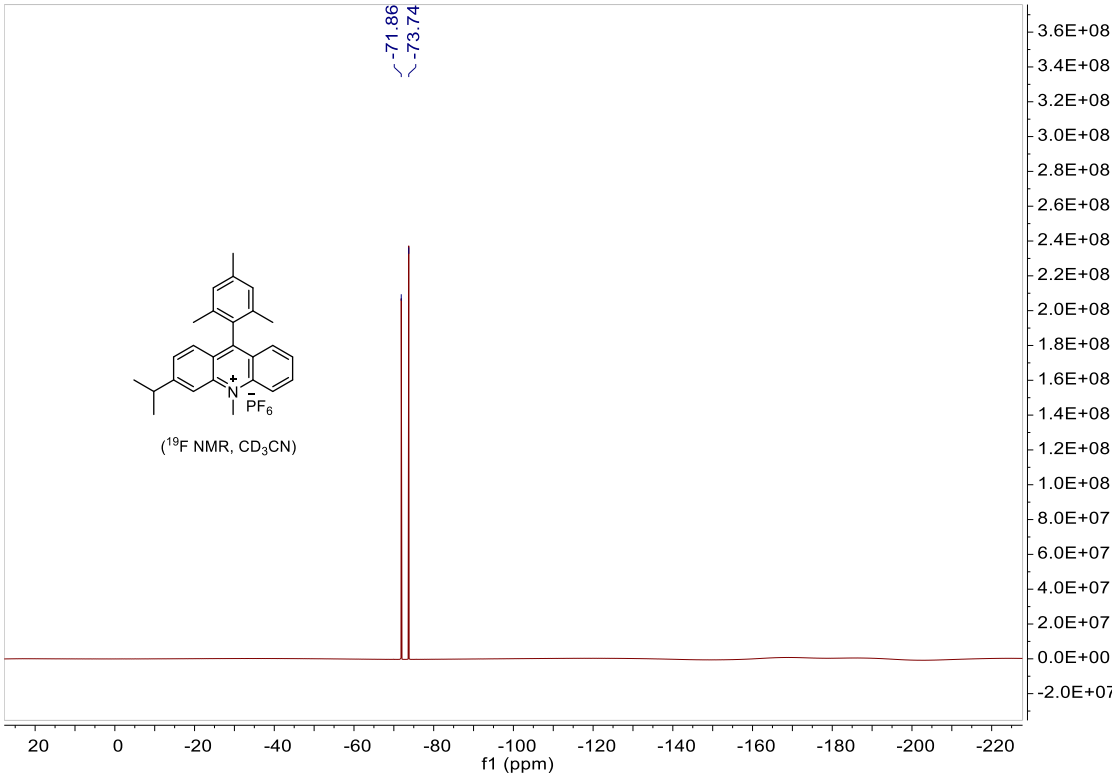
129.75
129.62
129.28
128.88
126.46
125.87
119.74
118.26 CD3CN
116.24
39.29
36.52
23.32
21.27
19.91
1.86
1.65
1.45
1.24 CD3CN
1.04
0.83
0.62
PF6
(13C NMR, CD3CN)
f1 (ppm)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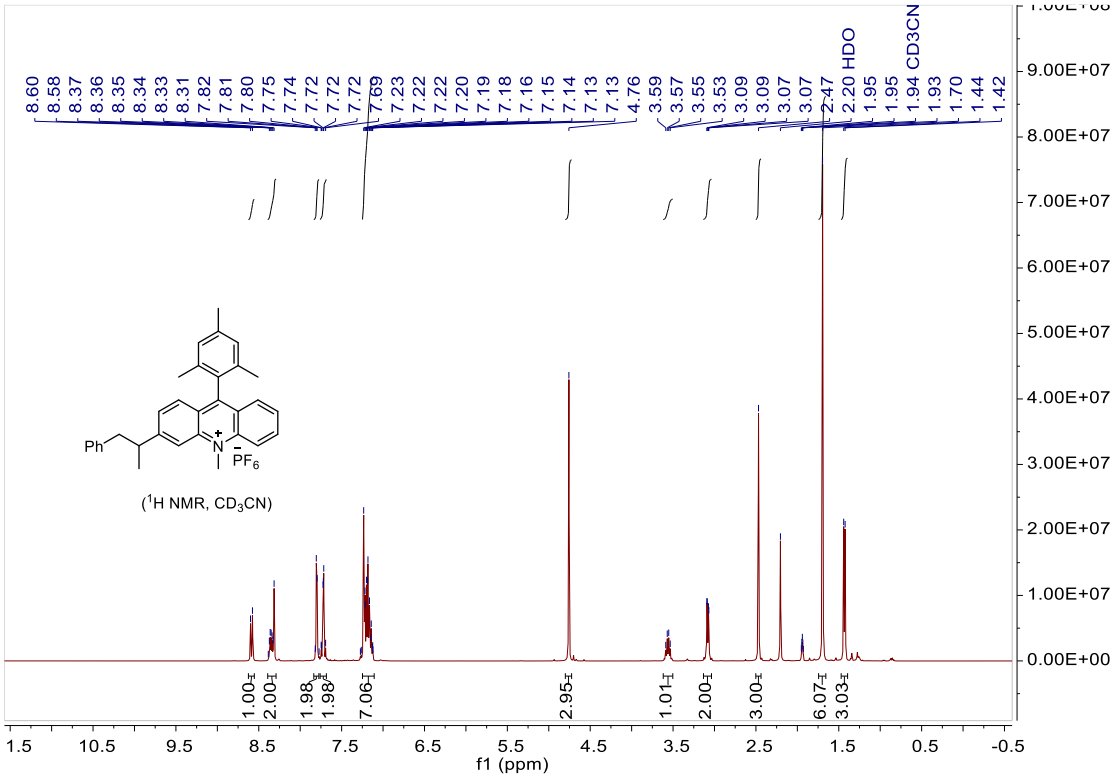
-73.74
PF6
(19F NMR, CD3CN)
f1 (ppm)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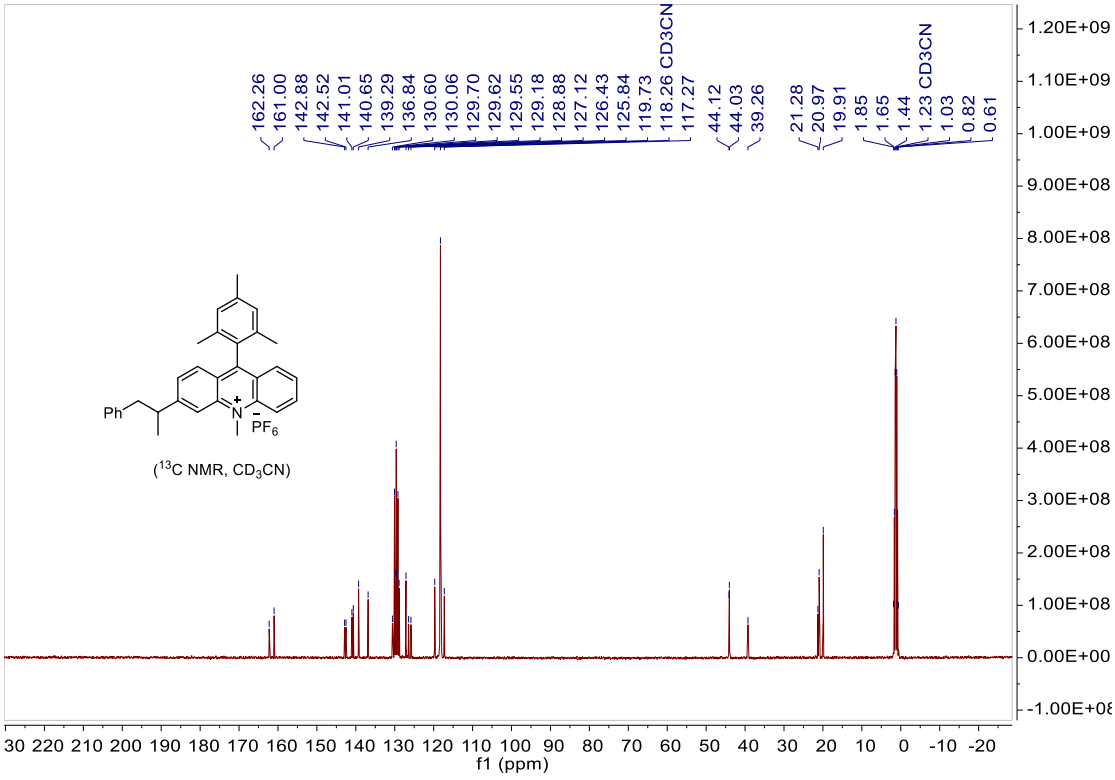
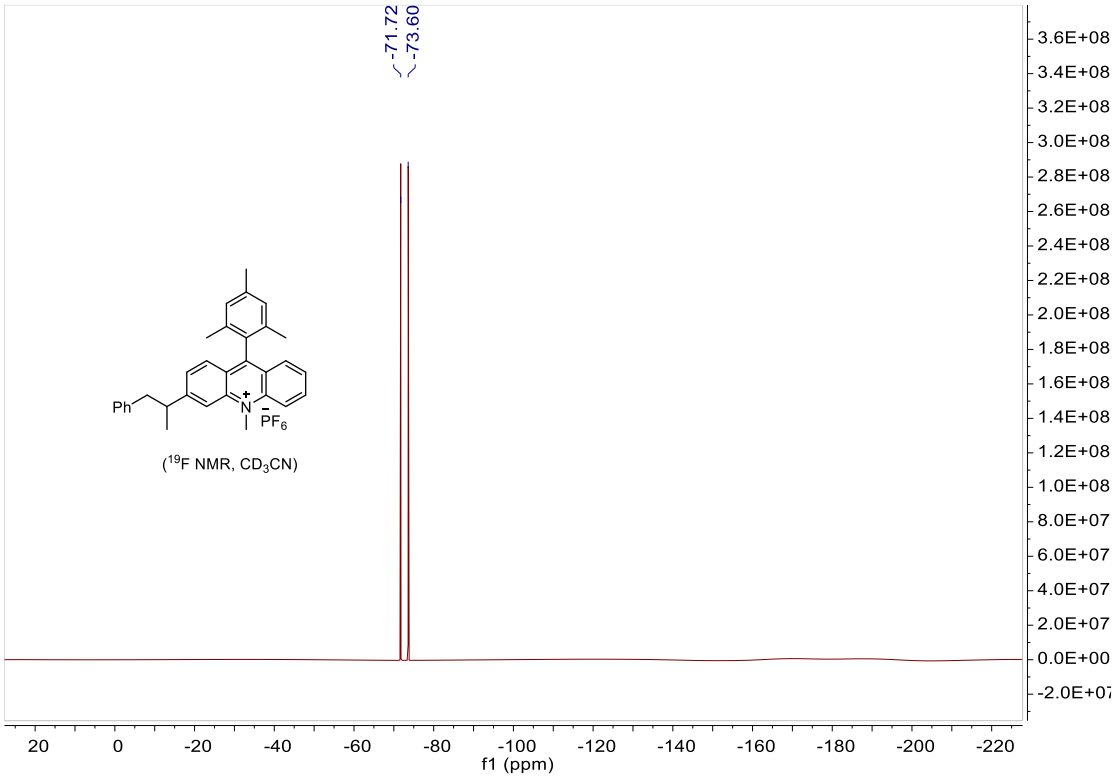
Ph
N
PF6
(1H NMR, CD3CN)
1.00 2.00 1.98 1.98 7.06 2.95 1.01 2.00 3.00 6.07 3.03
f1 (ppm)


162.26 161.00 142.88 142.52 141.01 140.65 139.29 136.84 130.60 130.06 129.70 129.62 129.55 129.18 128.88 127.12 126.43 125.84 119.73 118.26 CD3CN 117.27 44.12 44.03 39.26 21.28 20.97 19.91 1.85 1.65 1.44 1.23 CD3CN 1.03 0.82 0.61
Ph
N
PF6
(13C NMR, CD3CN)
f1 (ppm)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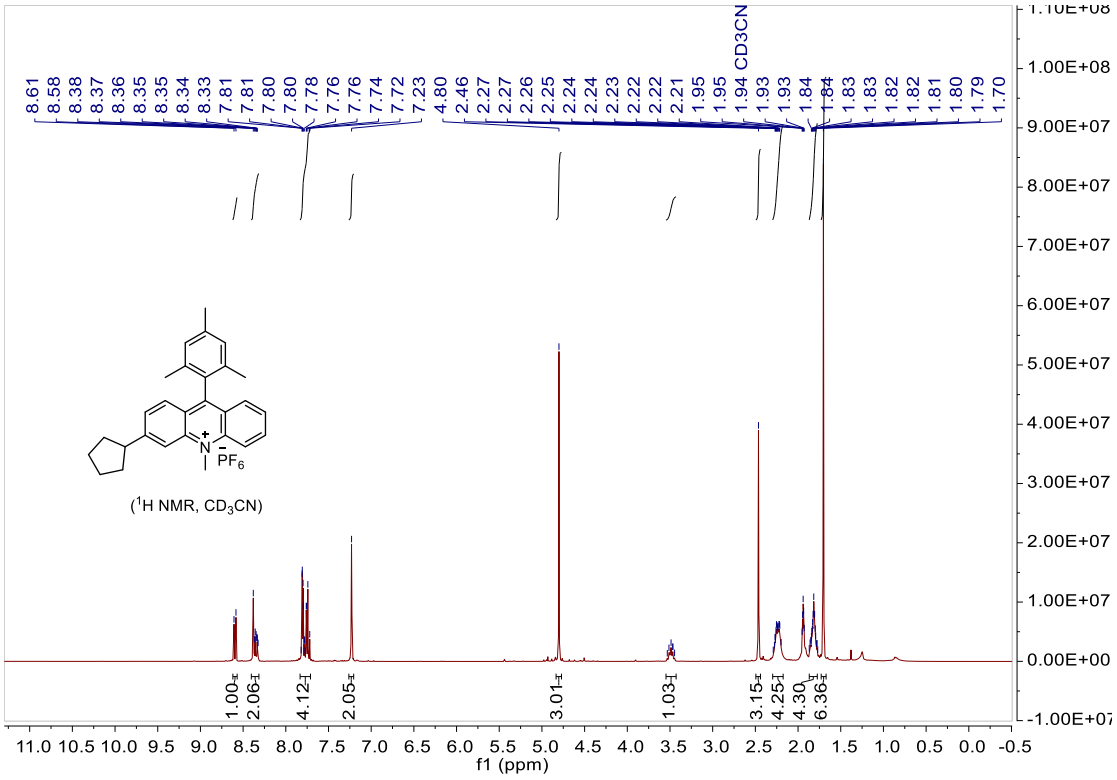
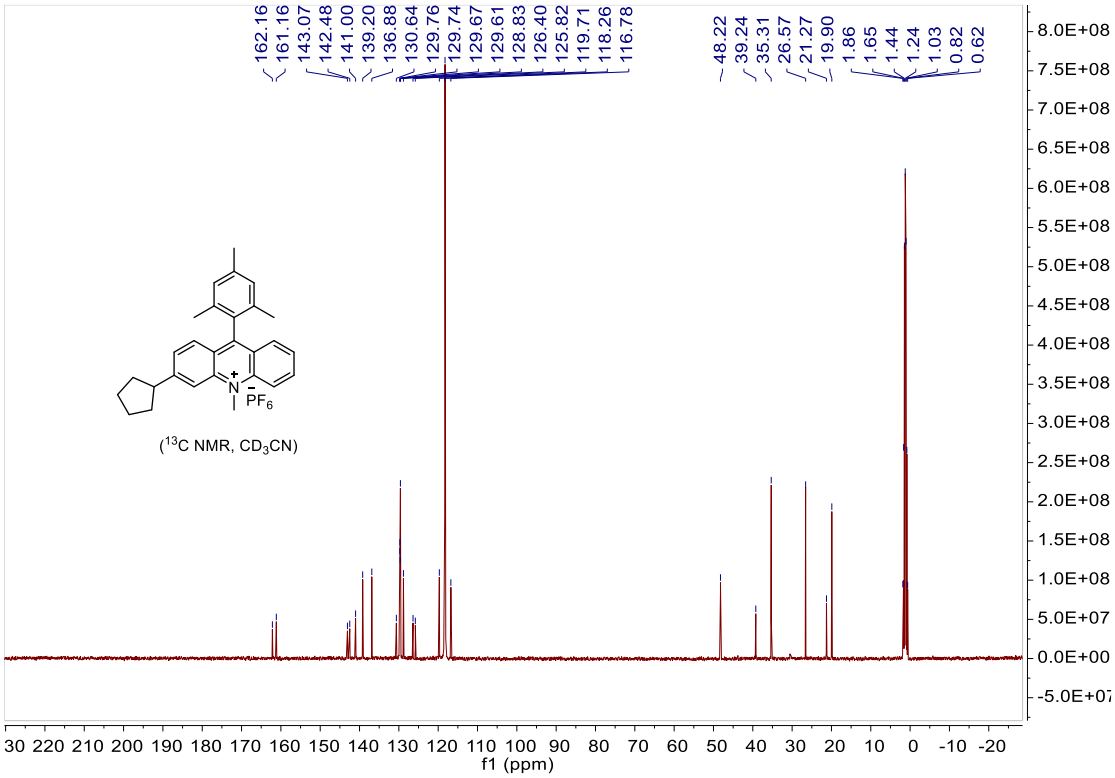
Ph
N
PF6
(19F NMR, CD3CN)
f1 (ppm)


CD3CN
N
PF6
(1H NMR, CD3CN)
f1 (ppm)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142.48
141.00
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129.74
129.67
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128.83
126.40
125.82
119.71
118.26
116.78
48.22
39.24
35.31
26.57
21.27
19.90
1.86
1.65
1.44
1.24
1.03
0.82
0.62
8.0E+08
7.5E+08
7.0E+08
6.5E+08
6.0E+08
5.5E+08
5.0E+08
4.5E+08
4.0E+08
3.5E+08
3.0E+08
2.5E+08
2.0E+08
1.5E+08
1.0E+08
5.0E+07
0.0E+00
-5.0E+07
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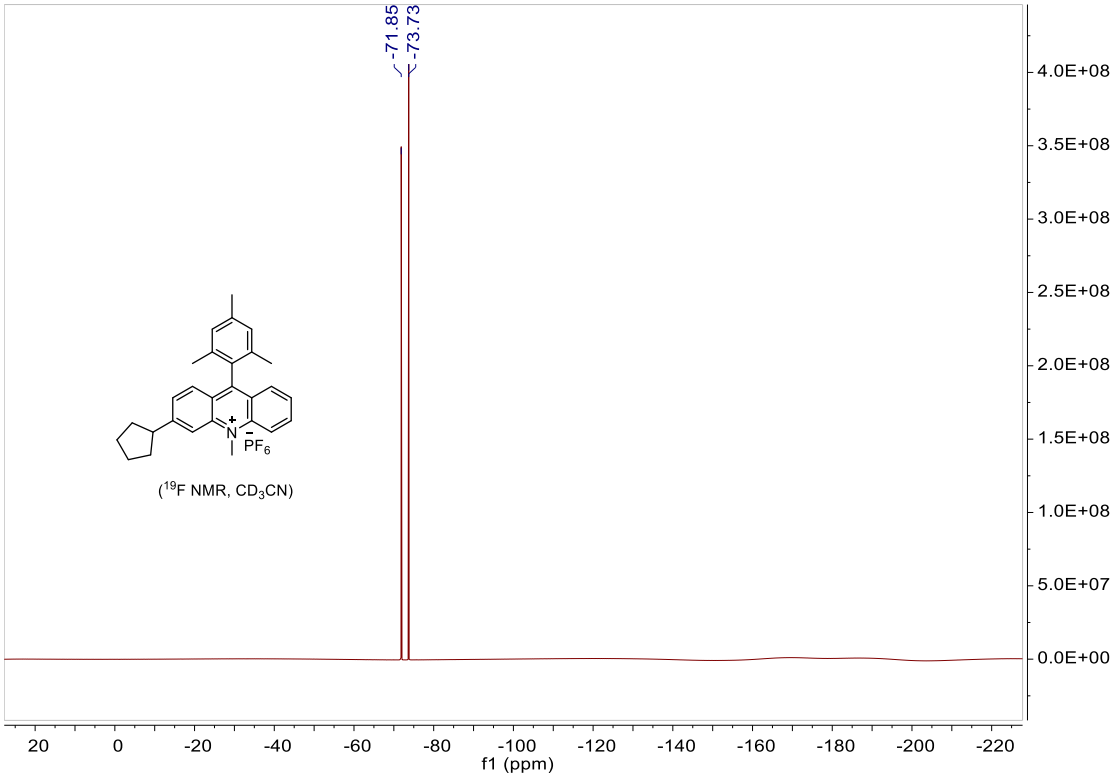
f1 (ppm)
N
PF6
(13C NMR, CD3CN)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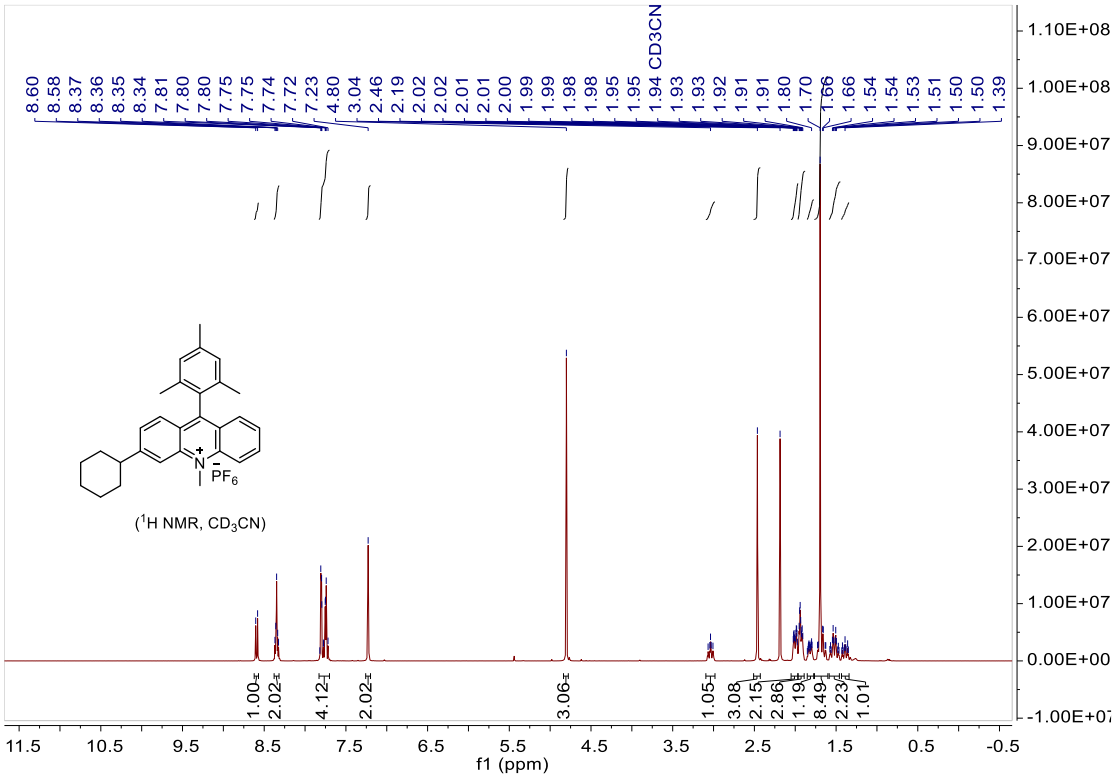
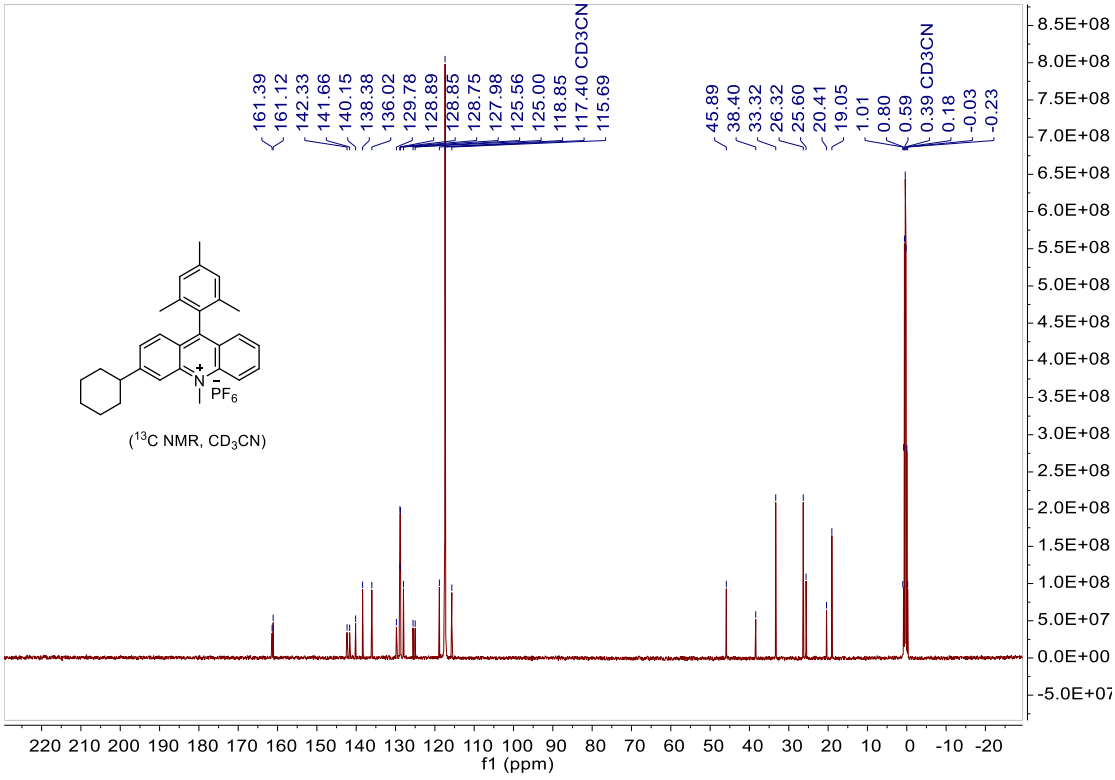
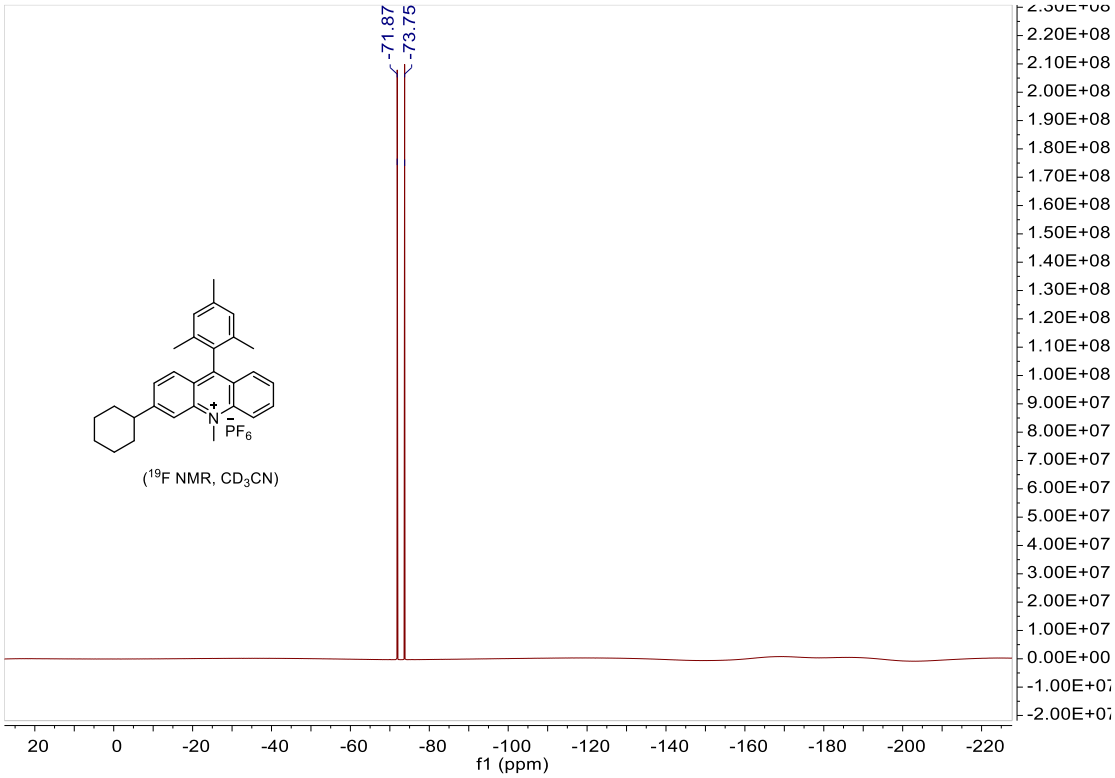
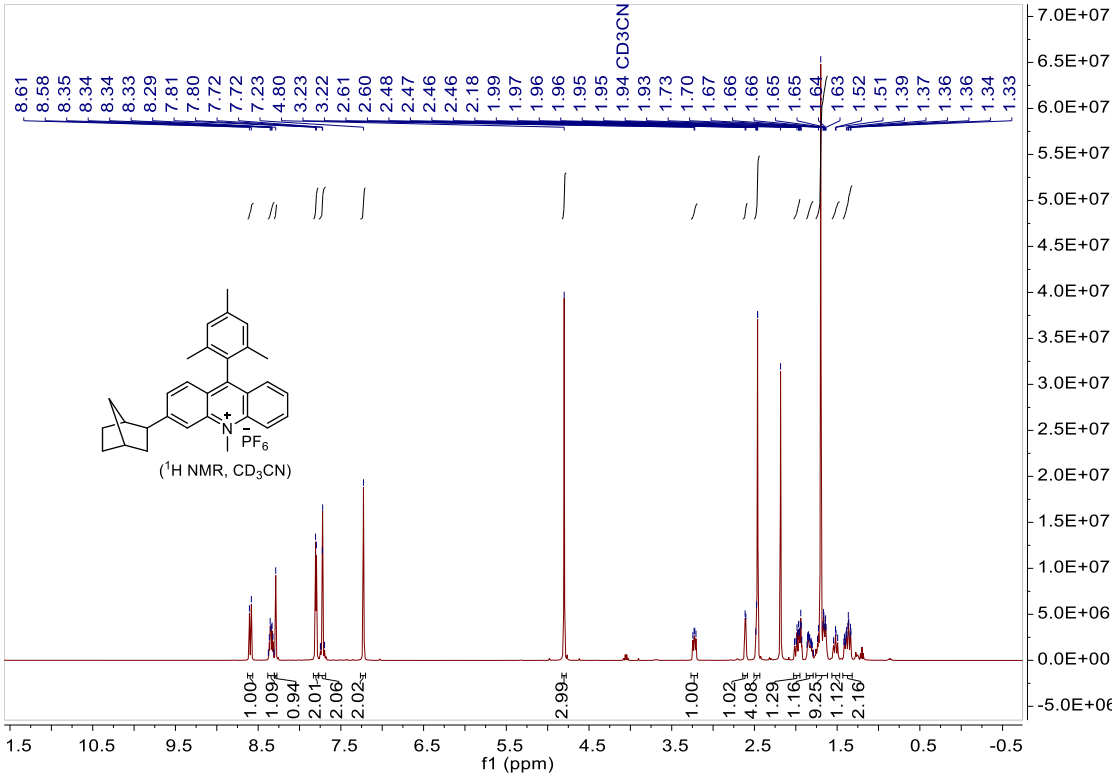
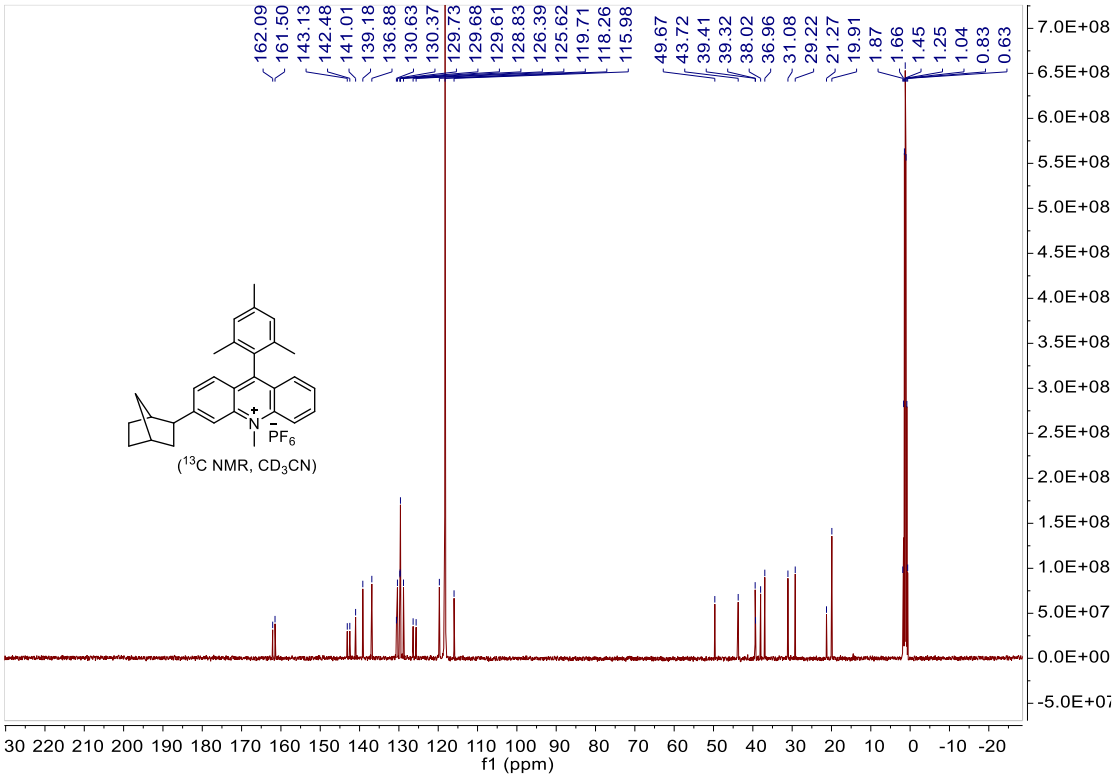
-73.73
4.0E+08
3.5E+08
3.0E+08
2.5E+08
2.0E+08
1.5E+08
1.0E+08
5.0E+07
0.0E+00
20 0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 -220
f1 (ppm)
N
PF6
(19F NMR, CD3CN)


8.60 8.58 8.37 8.36 8.35 8.34 7.81 7.80 7.80 7.75 7.75 7.74 7.72 7.23 4.80 3.04 2.46 2.19 2.02 2.02 2.01 2.01 2.00 1.99 1.99 1.98 1.98 1.95 1.95 1.94 CD3CN 1.93 1.93 1.92 1.91 1.91 1.80 1.70 1.66 1.66 1.54 1.54 1.53 1.51 1.50 1.50 1.39
1.00 2.02 4.12 2.02 3.06 1.05 3.08 2.15 2.86 1.19 8.49 2.23 1.01
f1 (ppm)
(^{1}H NMR, CD_3CN)


161.39 161.12 142.33 141.66 140.15 138.38 136.02 129.78 128.89 128.85 128.75 127.98 125.56 125.00 118.85 117.40 CD3CN 115.69
45.89 38.40 33.32 26.32 25.60 20.41 19.05 1.01 0.80 0.59 0.39 CD3CN 0.18 -0.03 -0.23
f1 (ppm)
(^{13}C NMR, CD_3CN)


-71.87
-73.75
PF6
(19F NMR, CD3CN)
f1 (ppm)


CD3CN
PF6
(1H NMR, CD3CN)
f1 (ppm)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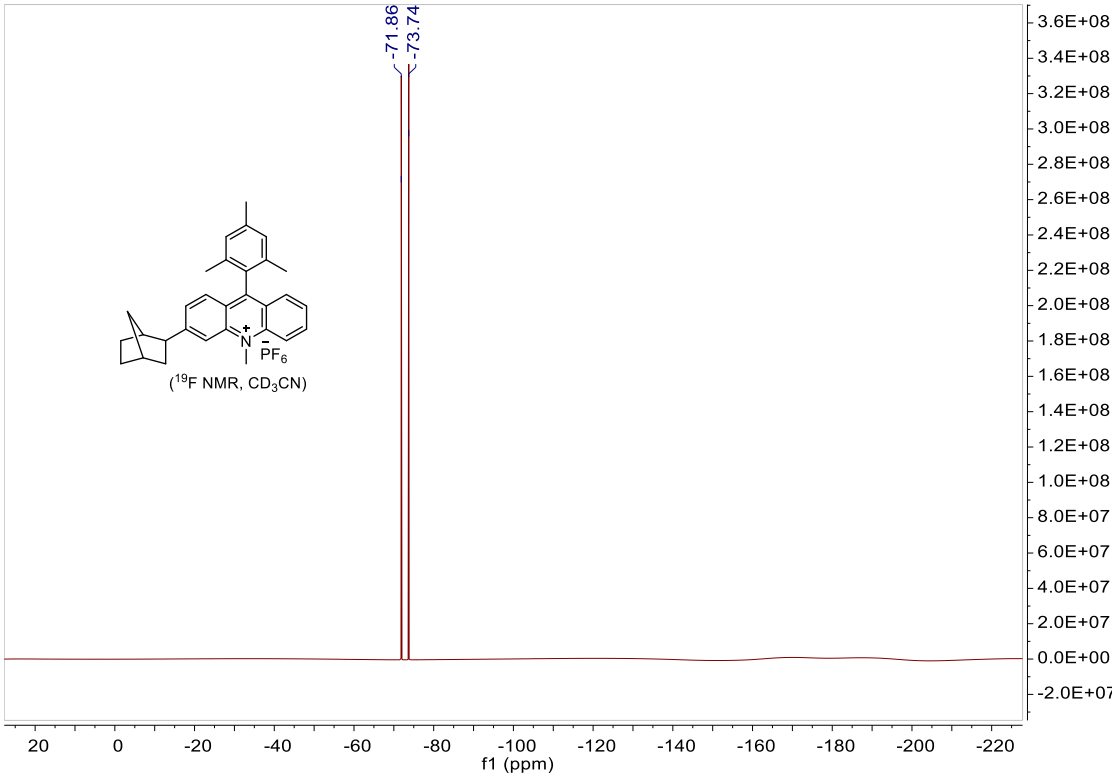
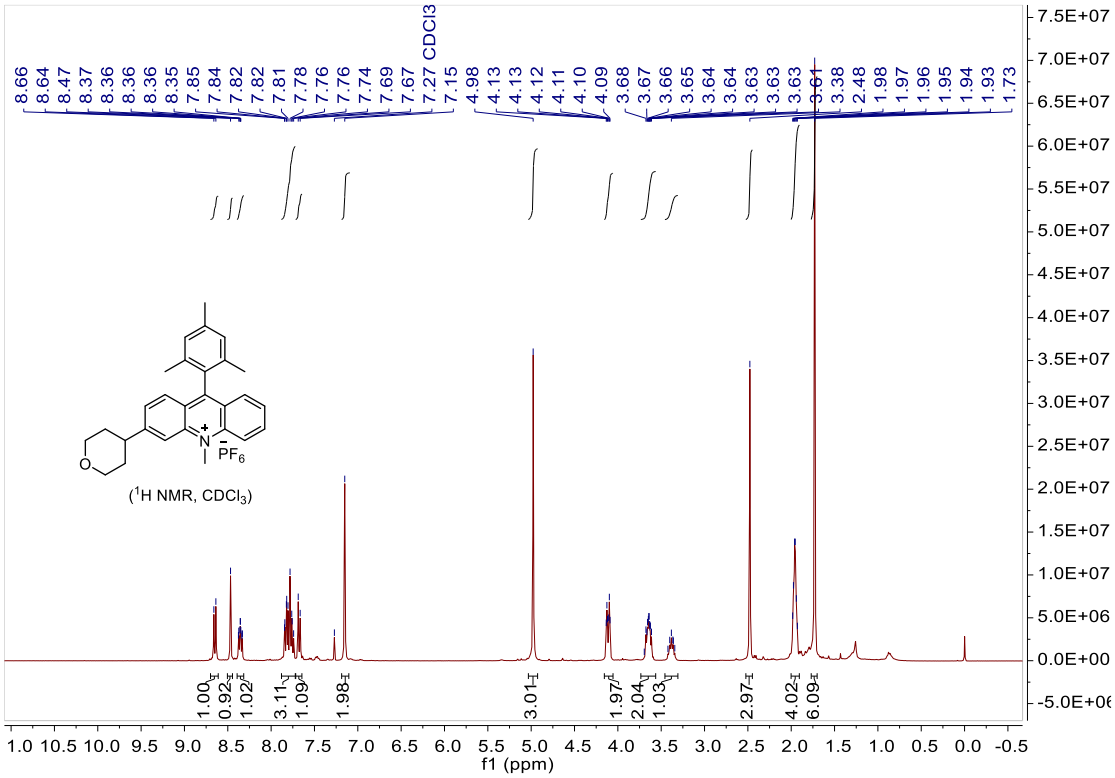
126.39
125.62
119.71
118.26
115.98
49.67
43.72
39.41
39.32
38.02
36.96
31.08
29.22
21.27
19.91
1.87
1.66
1.45
1.25
1.04
0.83
0.63
PF6
(13C NMR, CD3CN)
f1 (ppm)


-71.86
-73.74
PF6
(19F NMR, CD3CN)
f1 (ppm)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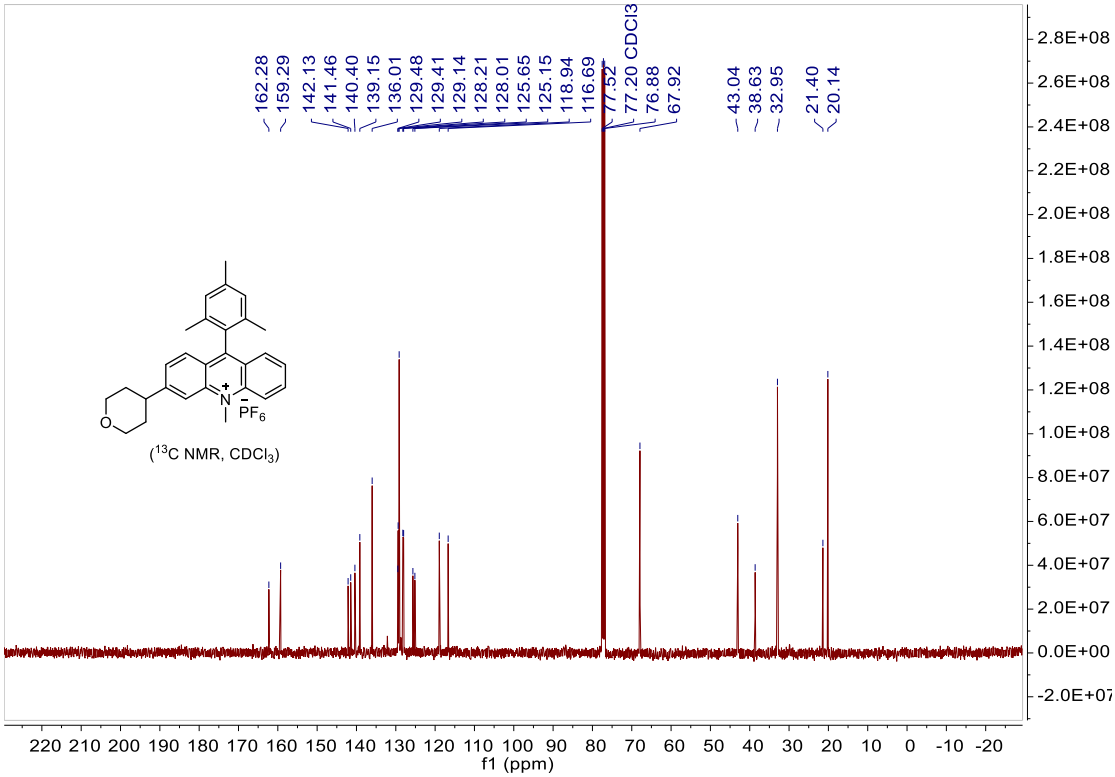
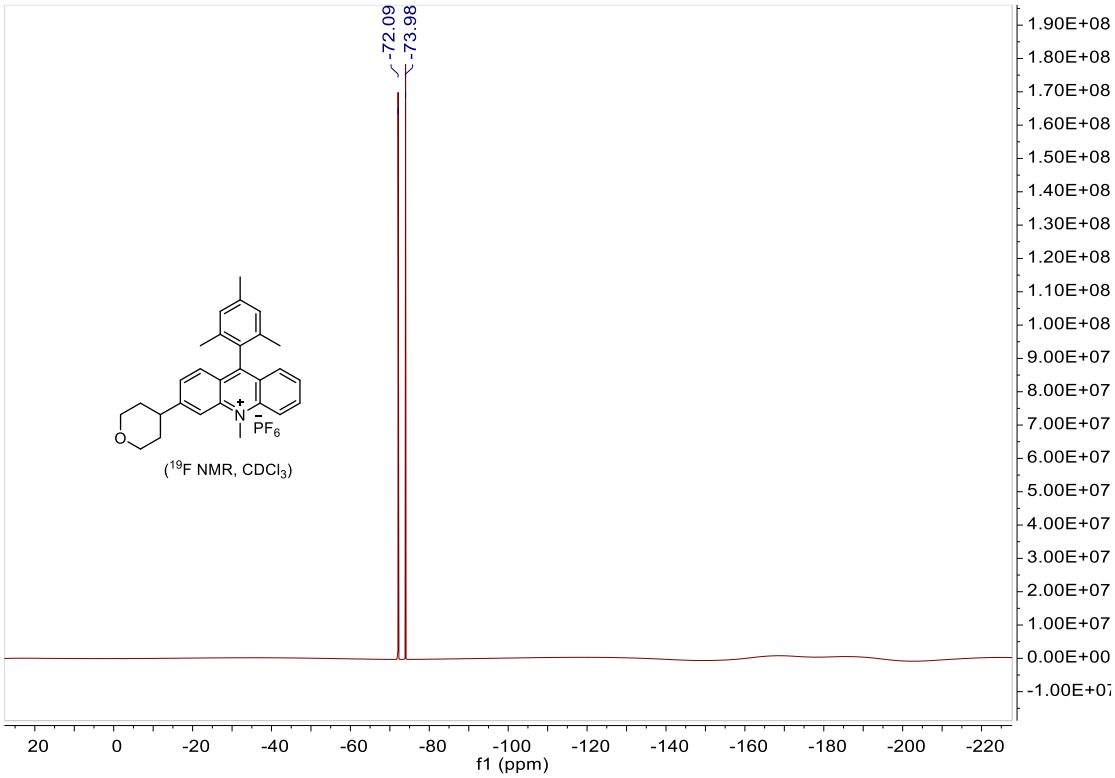
1.00 0.92 1.02 3.11 1.09 1.98 3.01 1.97 2.04 1.03 2.97 4.02 6.09
f1 (ppm)
(^{1}H NMR, $CDCl_3$)


162.28 159.29 142.13 141.46 140.40 139.15 136.01 129.48 129.41 129.14 128.21 128.01 125.65 125.15 118.94 116.69 77.52 77.20 CDCl3 76.88 67.92 43.04 38.63 32.95 21.40 20.14
f1 (ppm)
(^{13}C NMR, $CDCl_3$)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O
N
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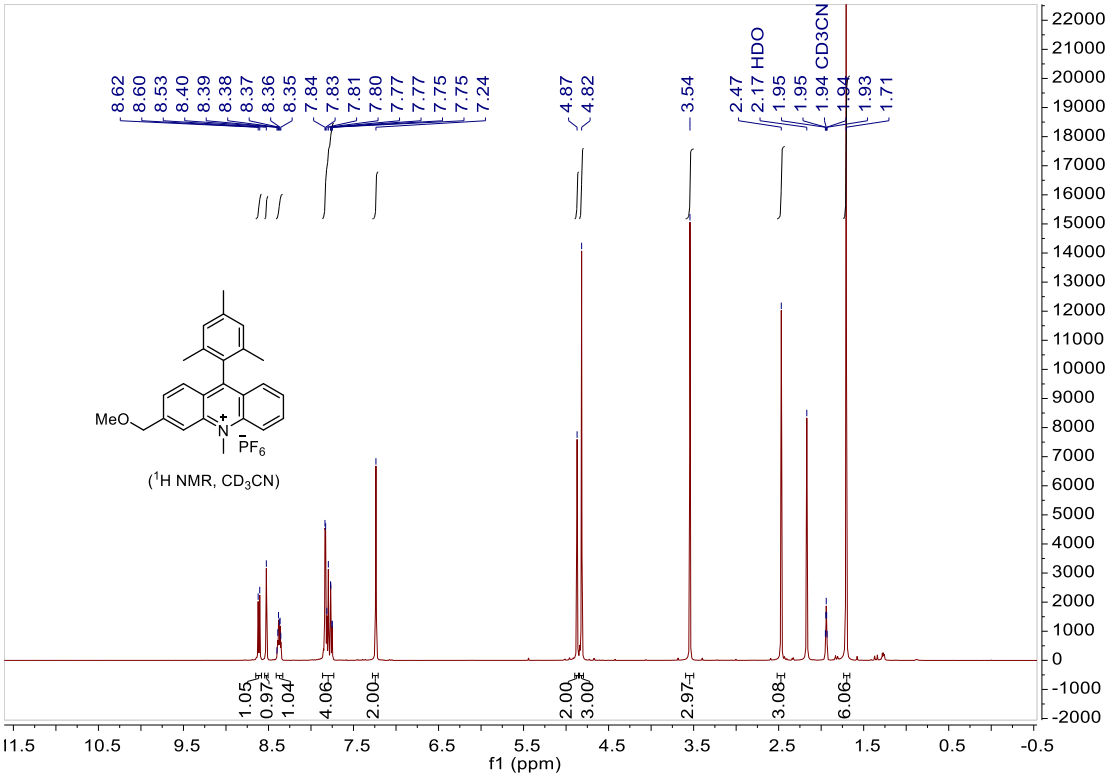
(19F NMR, CDCl3)
f1 (ppm)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2.17 HDO
1.95
1.95
1.94 CD3CN
1.94
1.93
1.71
MeO
N
PF6
(1H NMR, CD3CN)
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4.06
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2.00
3.00
2.97
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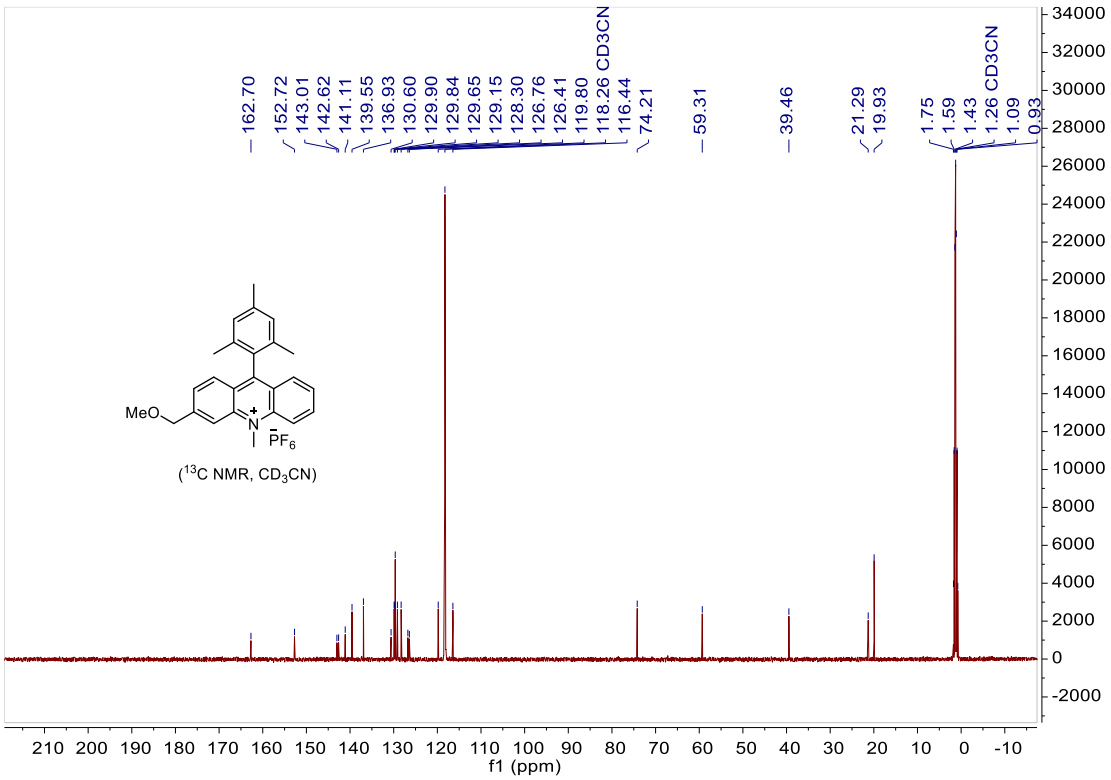
6.06
f1 (ppm)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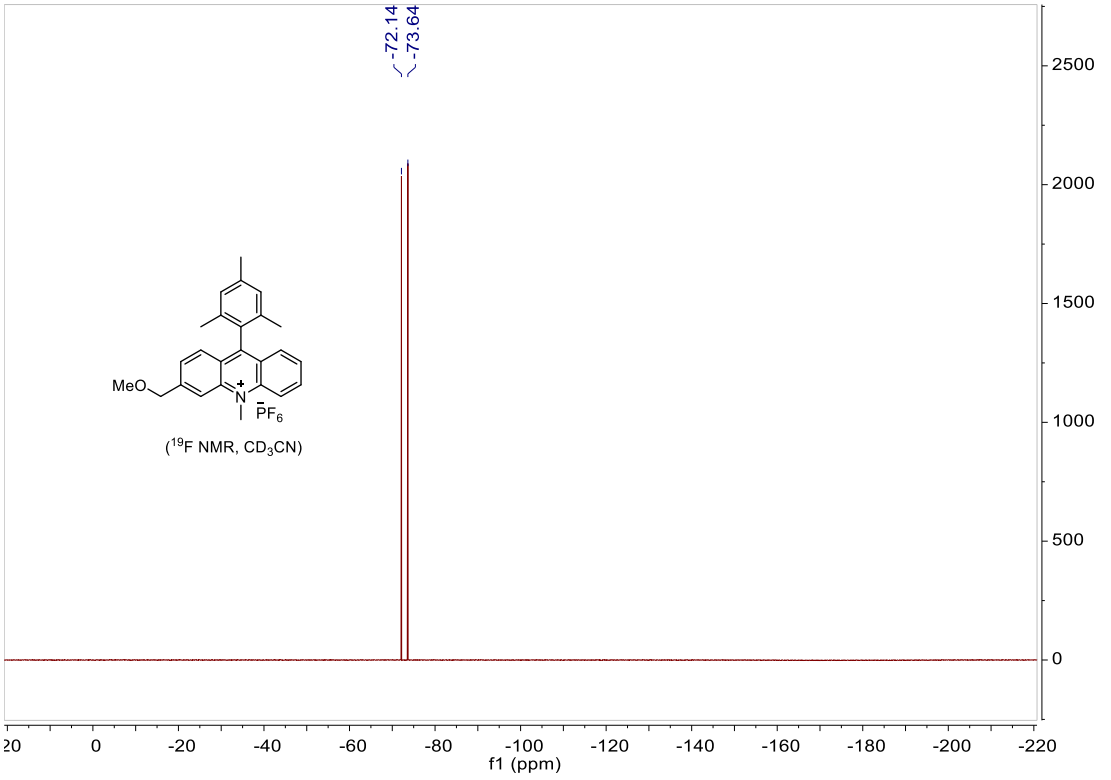
126.76
126.41
119.80
118.26 CD3CN
116.44
74.21
59.31
39.46
21.29
19.93
1.75
1.59
1.43
1.26 CD3CN
1.09
0.93
MeO
PF6
(13C NMR, CD3CN)
f1 (ppm)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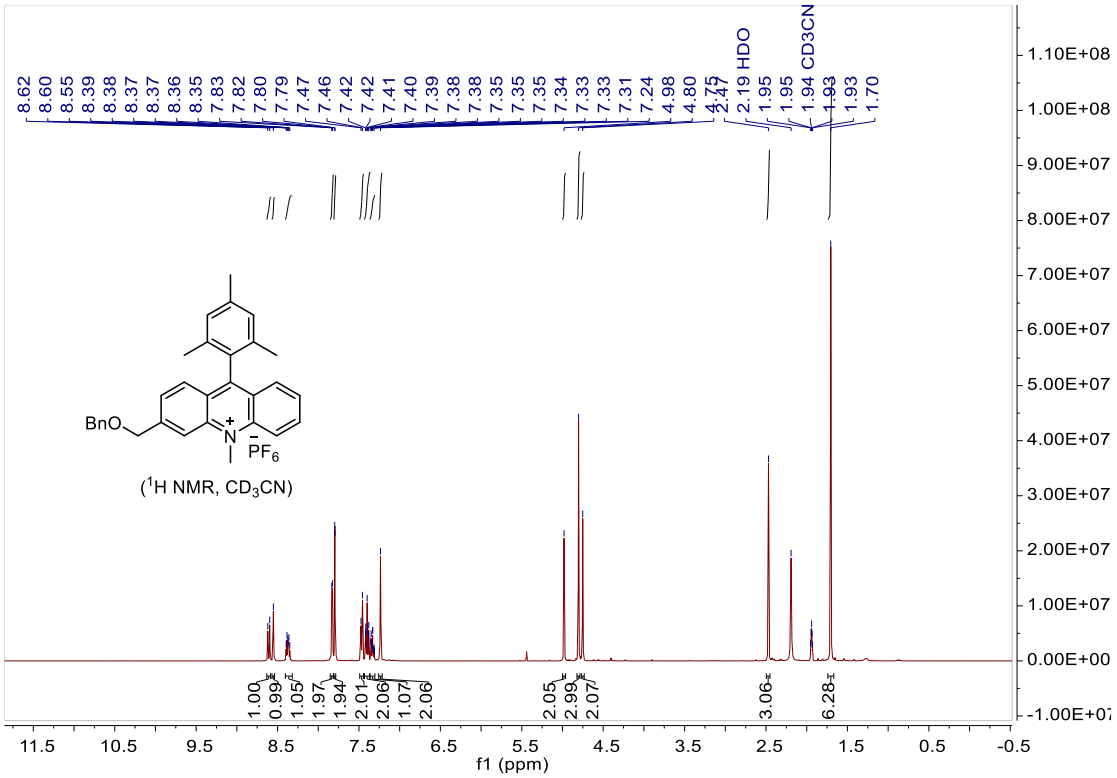
(19F NMR, CD3CN)
f1 (ppm)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7.33
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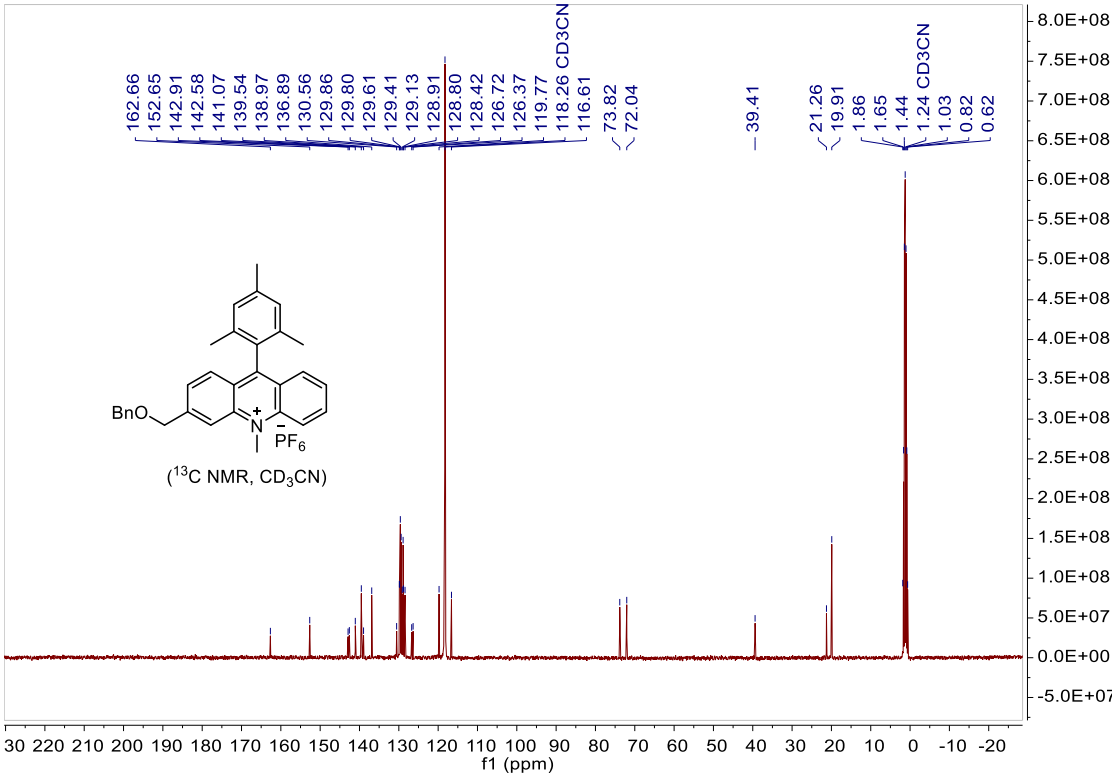
7.24
4.98
4.80
4.75
4.47
2.19 HDO
1.95
1.95
1.94 CD3CN
1.93
1.93
1.70
BnO
N
PF6
(1H NMR, CD3CN)
1.00
0.99
1.05
1.97
1.94
2.01
2.06
1.07
2.06
2.05
2.99
2.07
3.06
6.28
f1 (ppm)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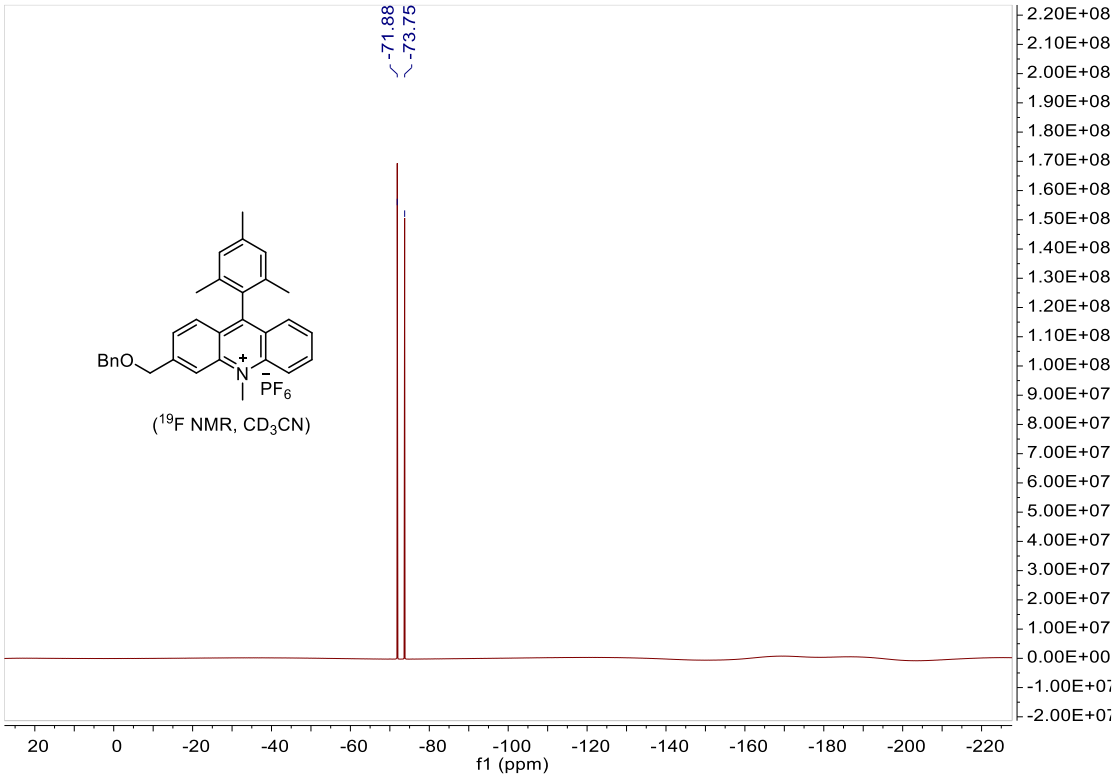
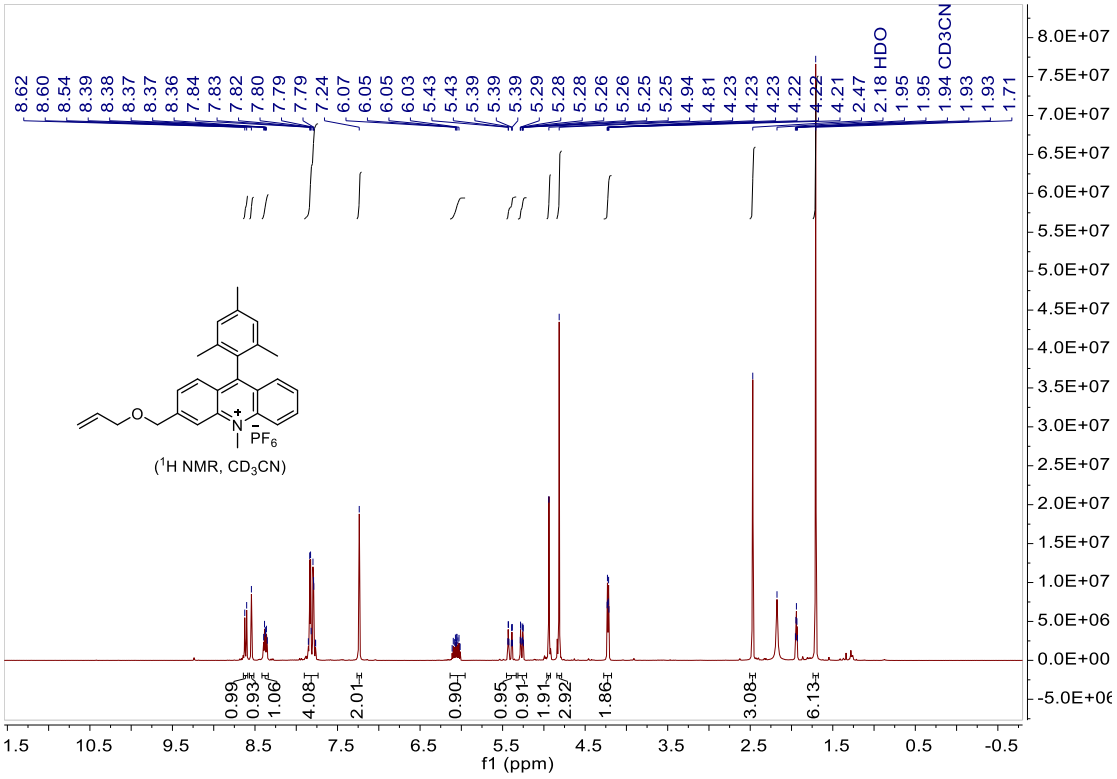
128.80
128.42
126.72
126.37
119.77
118.26 CD3CN
116.61
73.82
72.04
39.41
21.26
19.91
1.86
1.65
1.44
1.24 CD3CN
1.03
0.82
0.62
BnO
N
PF6
(13C NMR, CD3CN)
f1 (ppm)


-71.88
-73.75
BnO
N
PF6
(19F NMR, CD3CN)
f1 (ppm)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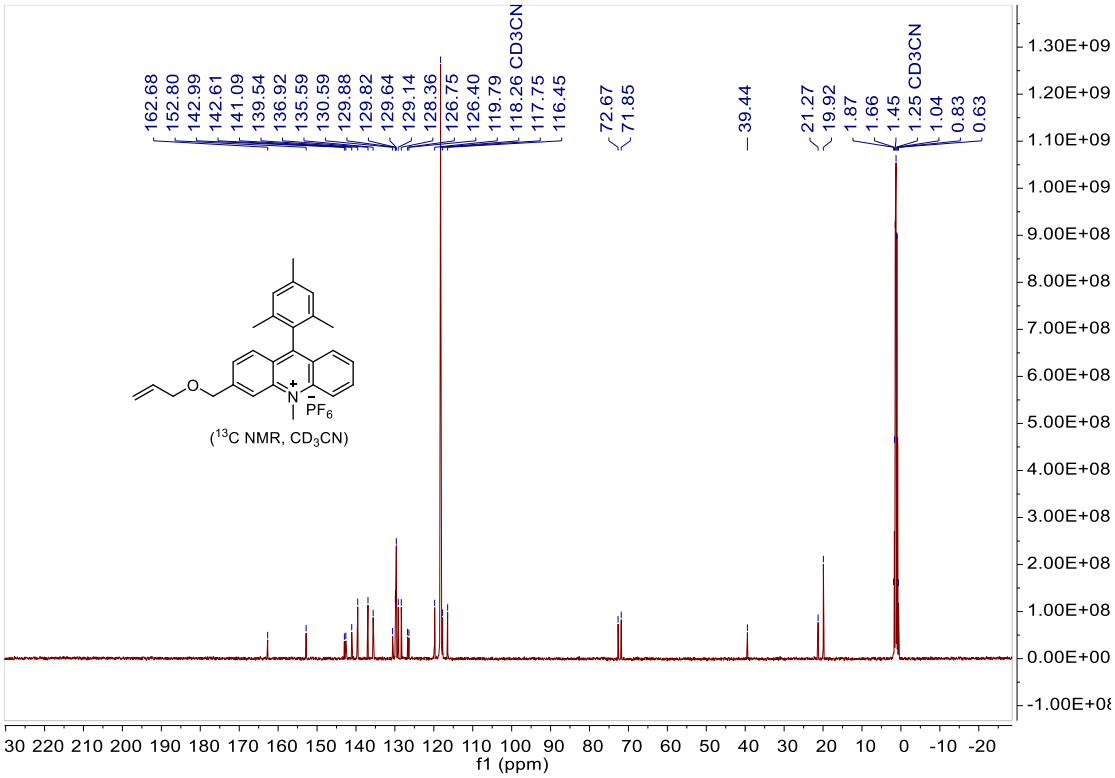
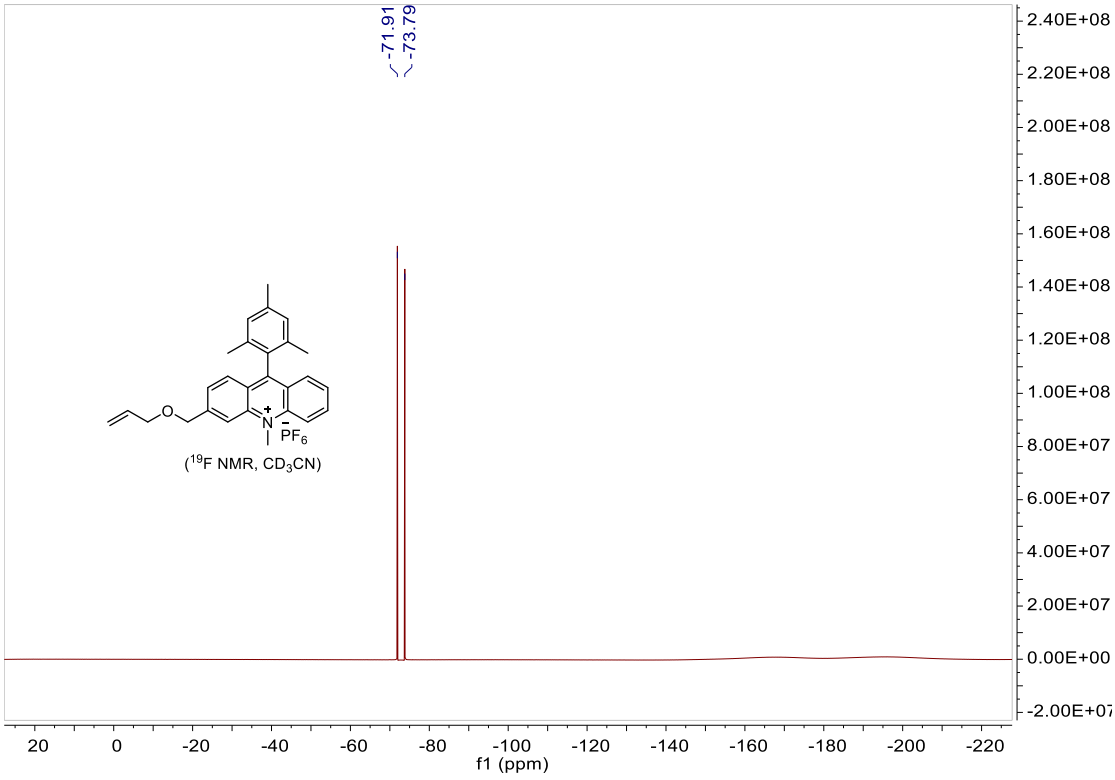
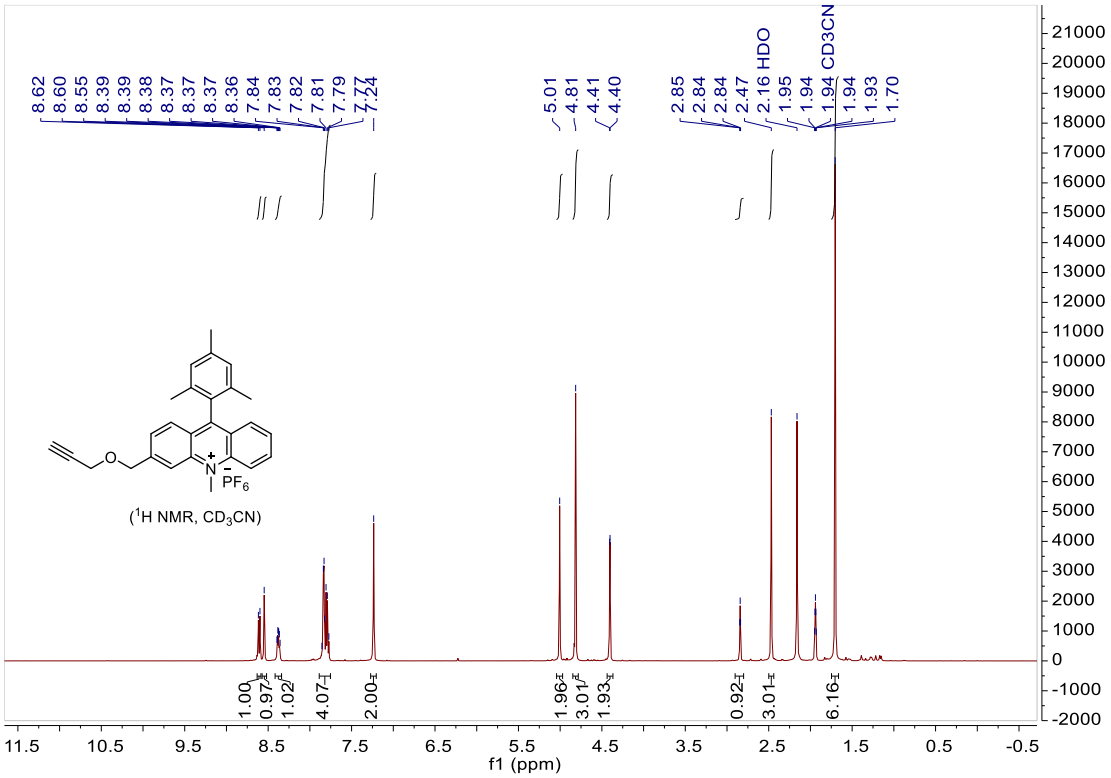
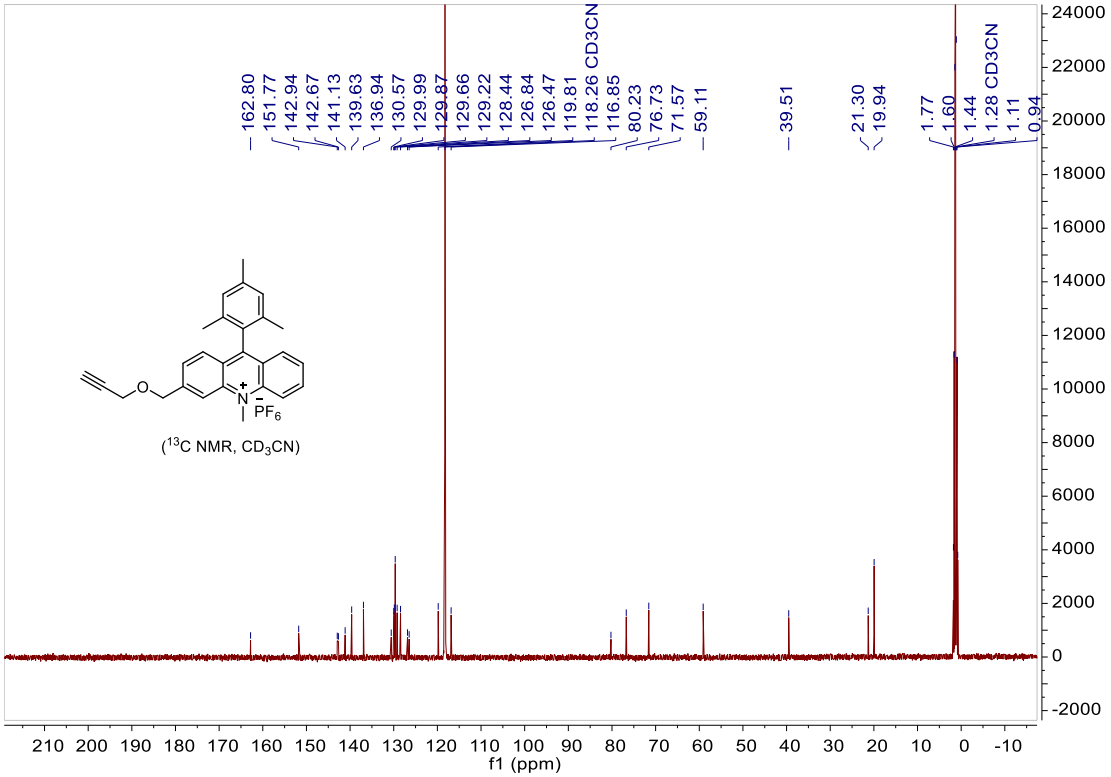
8.60
8.54
8.39
8.38
8.37
8.37
8.36
7.84
7.83
7.82
7.80
7.79
7.79
7.24
6.07
6.05
6.05
6.03
5.43
5.43
5.39
5.39
5.39
5.29
5.28
5.28
5.26
5.26
5.25
5.25
4.94
4.81
4.23
4.23
4.22
4.22
4.21
2.47
2.18 HDO
1.95
1.95
1.94 CD3CN
1.93
1.93
1.71
O
N
PF6
(1H NMR, CD3CN)
0.99
0.93
1.06
4.08
2.01
0.90
0.95
0.91
1.91
2.92
1.86
3.08
6.13
f1 (ppm)


162.68
152.80
142.99
142.61
141.09
139.54
136.92
135.59
130.59
129.88
129.82
129.64
129.14
128.36
126.75
126.40
119.79
118.26 CD3CN
117.75
116.45
72.67
71.85
39.44
21.27
19.92
1.87
1.66
1.45
1.25 CD3CN
1.04
0.83
0.63
PF6
(13C NMR, CD3CN)
f1 (ppm)


-71.91
-73.79
PF6
(19F NMR, CD3CN)
f1 (ppm)


8.62
8.60
8.55
8.39
8.39
8.38
8.37
8.37
8.37
8.36
7.84
7.83
7.82
7.81
7.79
7.77
7.24
5.01
4.81
4.41
4.40
2.85
2.84
2.84
2.47
2.16 HDO
1.95
1.94
1.94 CD3CN
1.94
1.93
1.70
1.00
0.97
1.02
4.07
2.00
1.96
3.01
1.93
0.92
3.01
6.16
f1 (ppm)
PF6
(1H NMR, CD3CN)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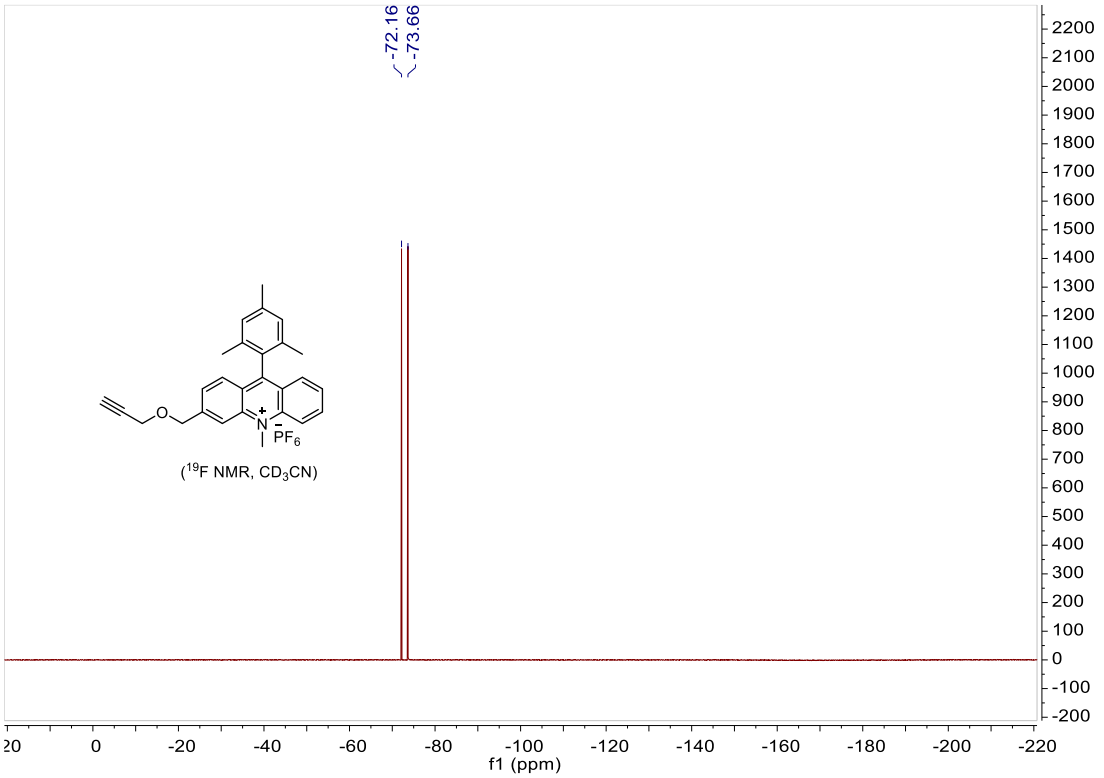
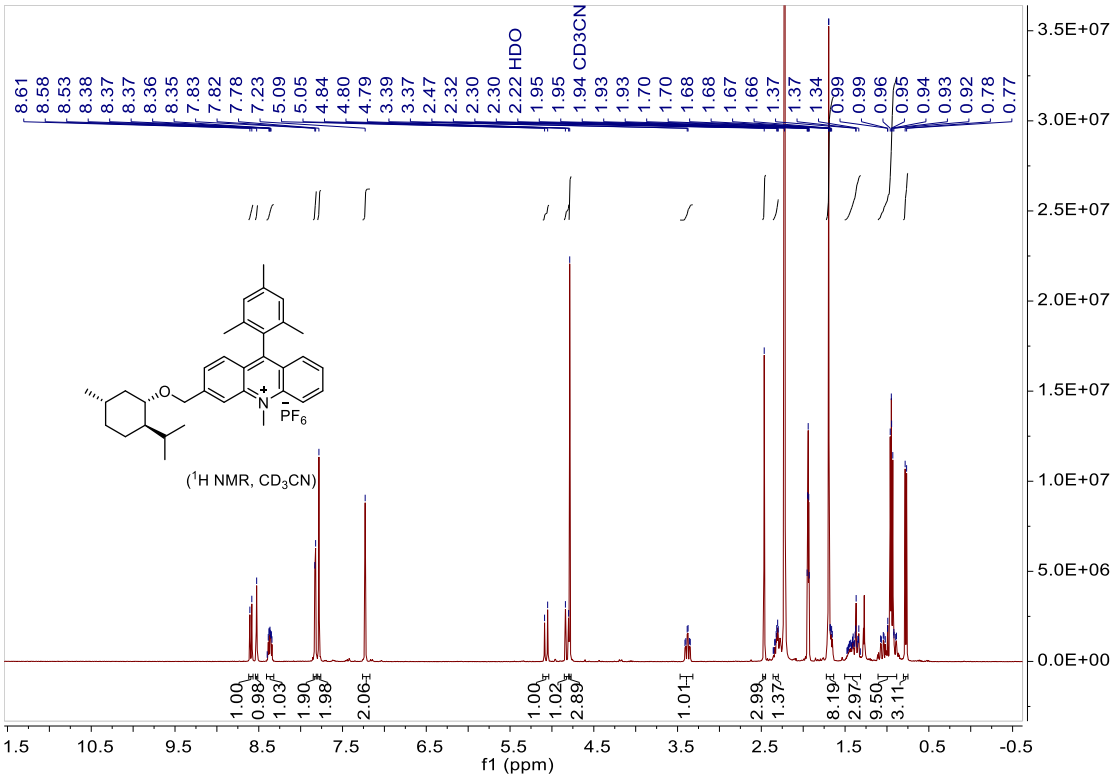
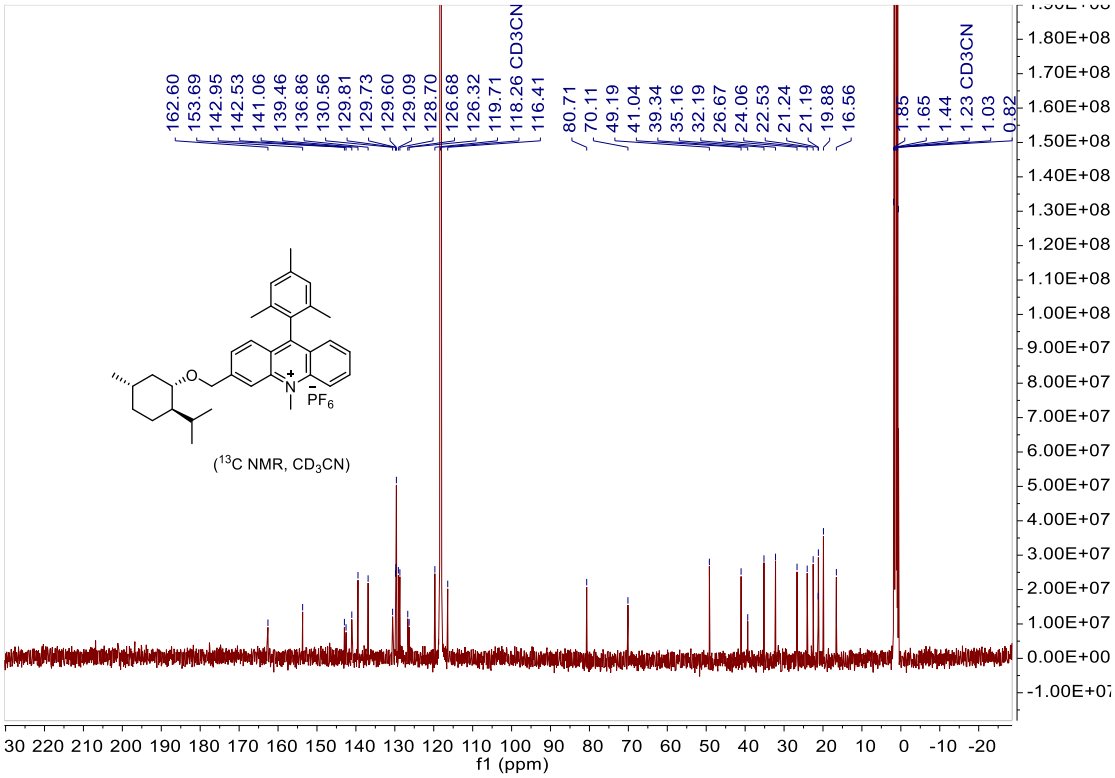
151.77
142.94
142.67
141.13
139.63
136.94
130.57
129.99
129.87
129.66
129.22
128.44
126.84
126.47
119.81
118.26 CD3CN
116.85
80.23
76.73
71.57
59.11
39.51
21.30
19.94
1.77
1.60
1.44
1.28 CD3CN
1.11
0.94
f1 (ppm)
PF6
(13C NMR, CD3CN)


-72.16
-73.66
PF6
(^{19}F NMR, CD_3CN)
f1 (ppm)


HDO
CD3CN
PF6
(^{1}H NMR, CD_3CN)
f1 (ppm)


162.60
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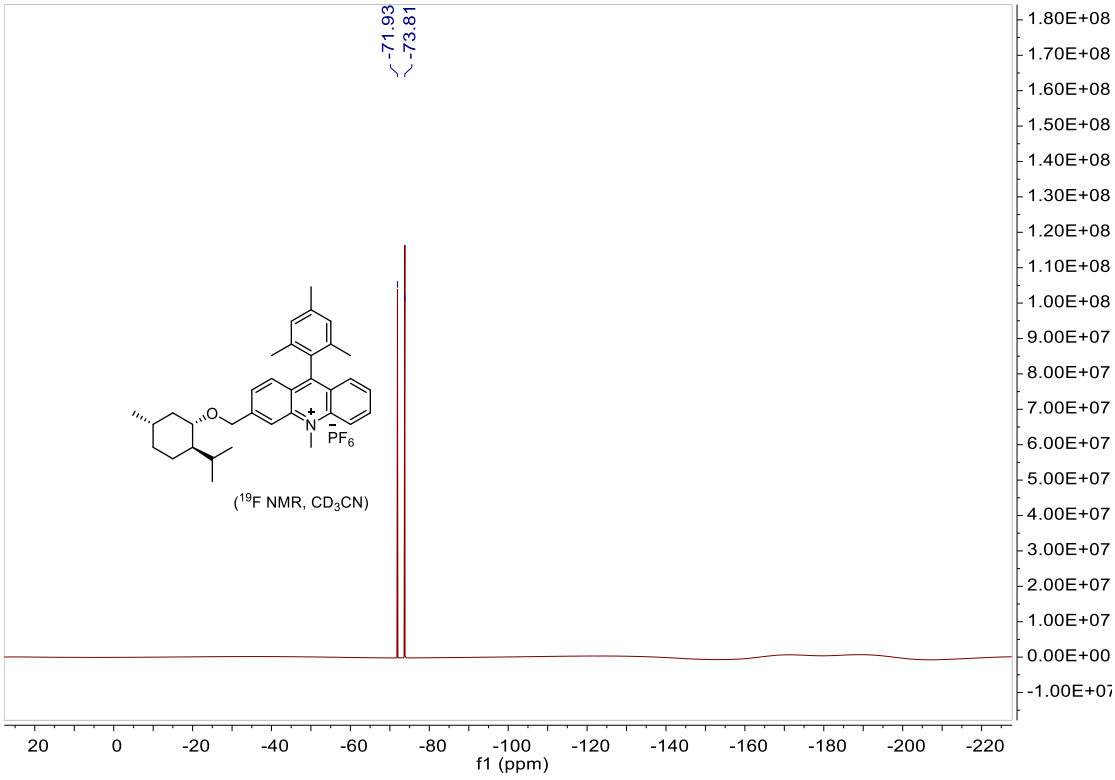
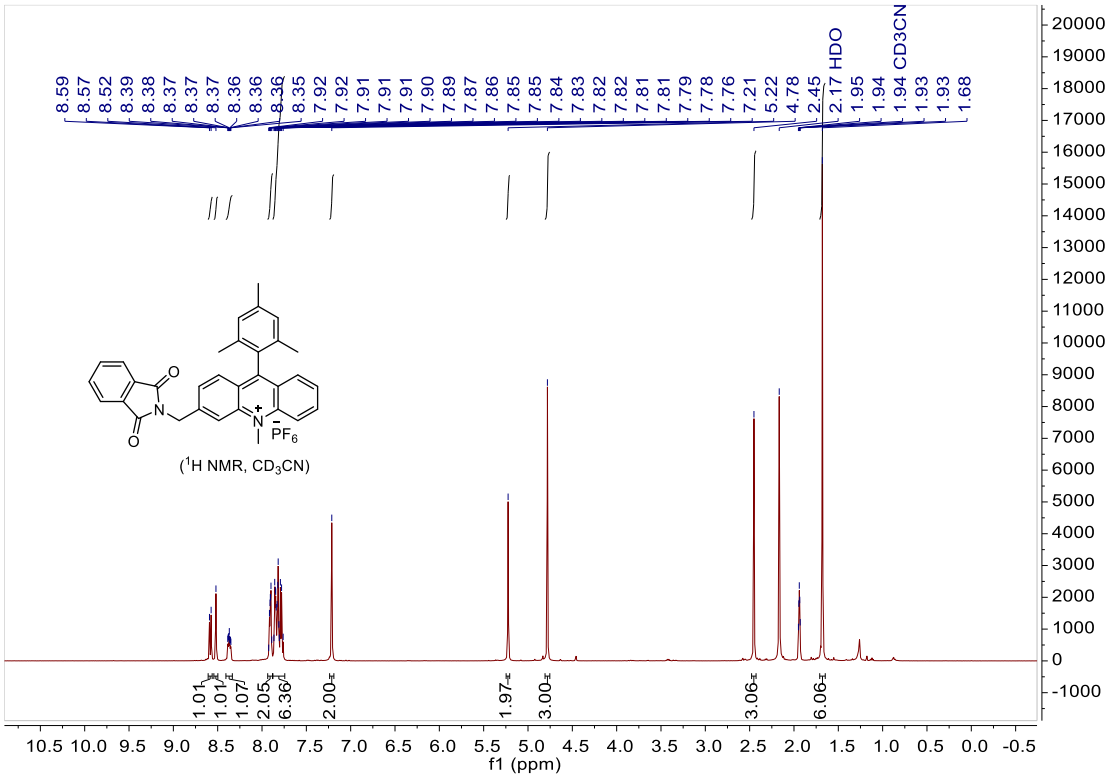
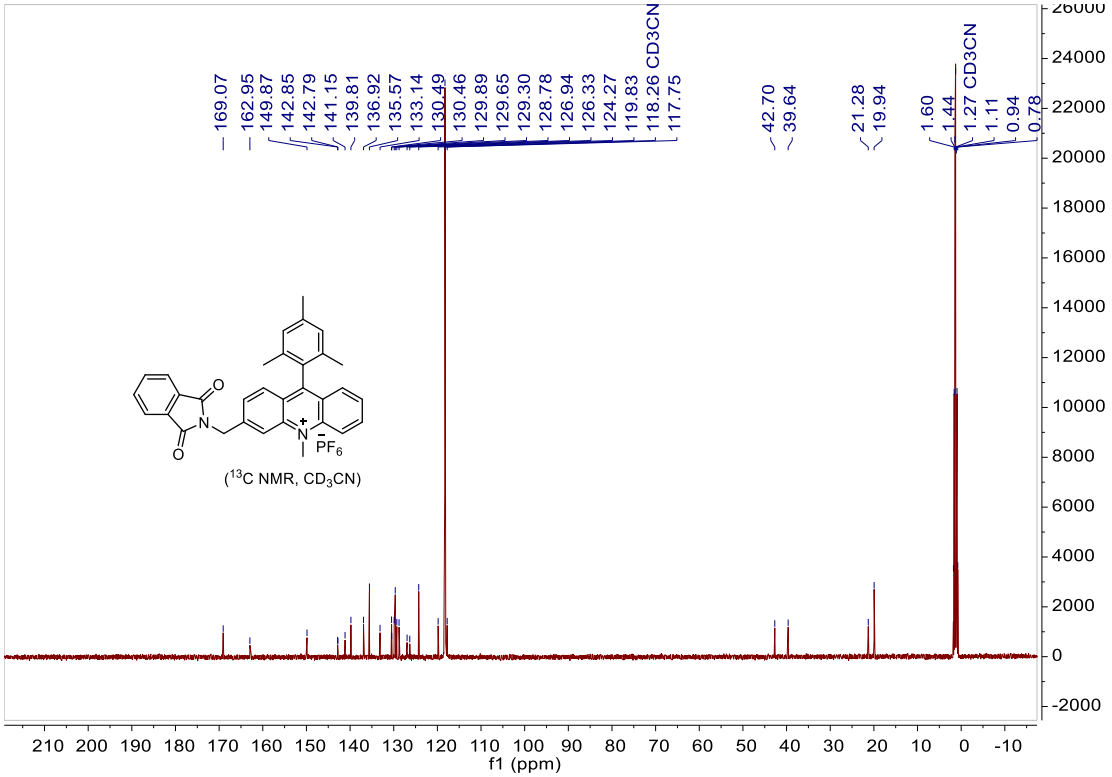
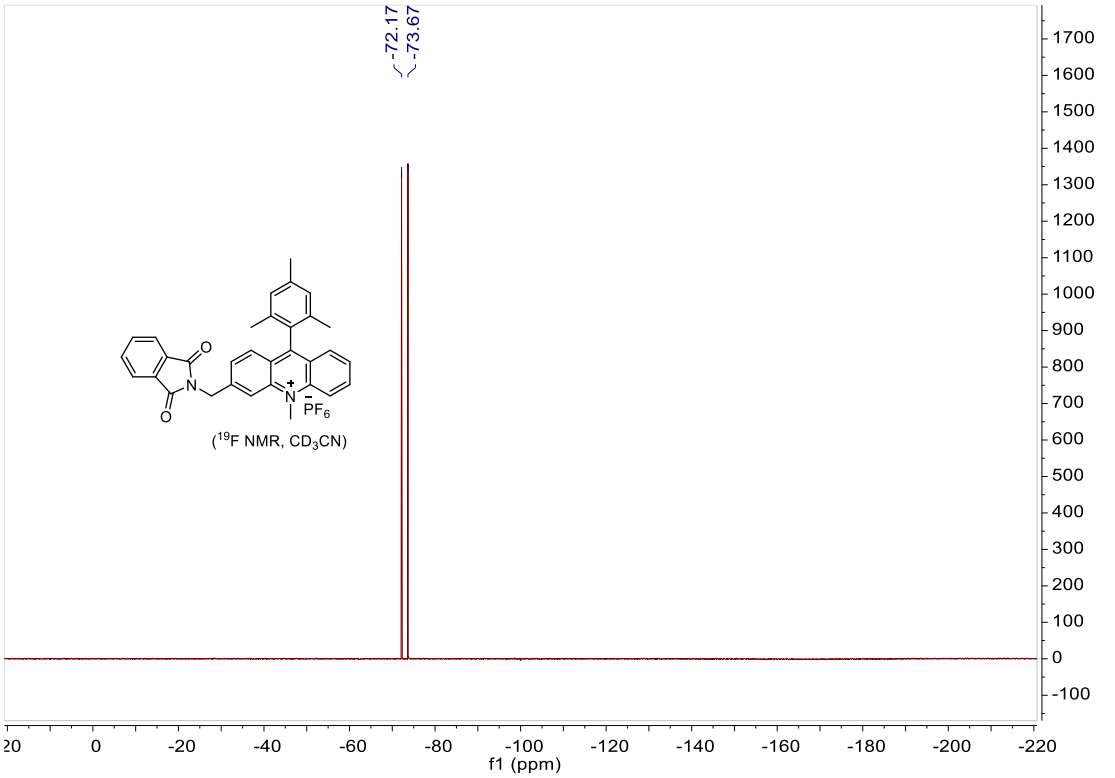
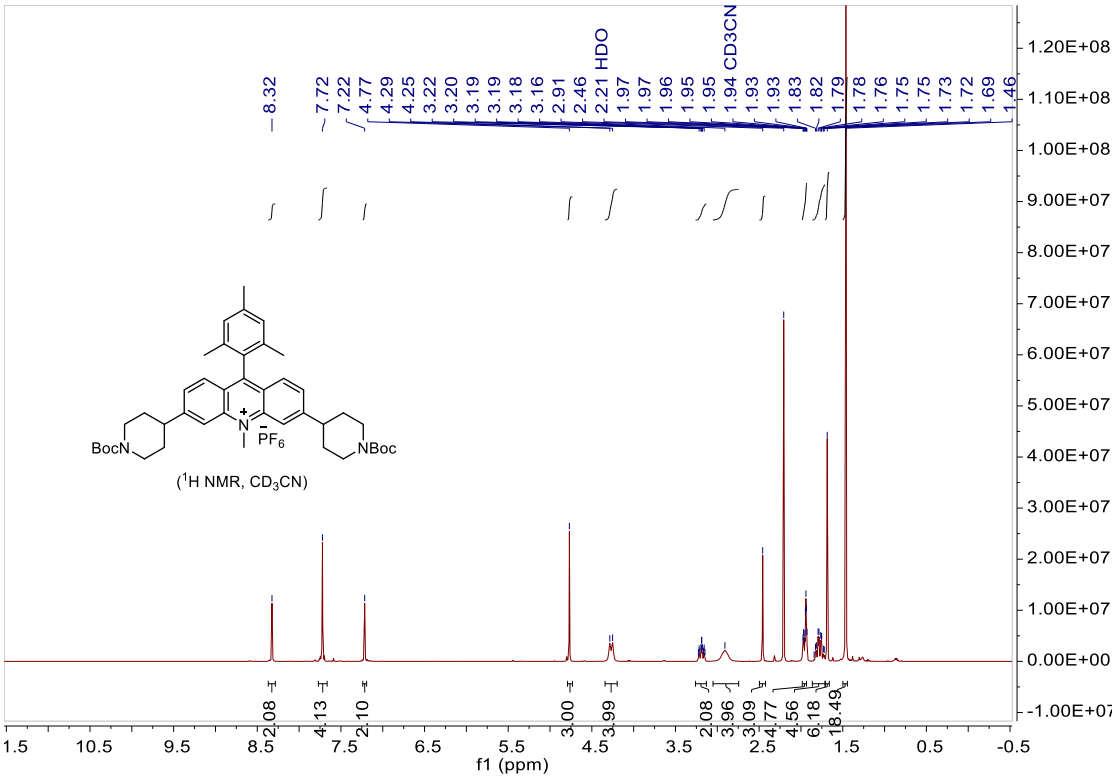
142.95
142.53
141.06
139.46
136.86
130.56
129.81
129.73
129.60
129.09
128.70
126.68
126.32
119.71
118.26 CD3CN
116.41
80.71
70.11
49.19
41.04
39.34
35.16
32.19
26.67
24.06
22.53
21.24
21.19
19.88
16.56
1.85
1.65
1.44
1.23 CD3CN
1.03
0.82
PF6
(^{13}C NMR, CD_3CN)
f1 (ppm)

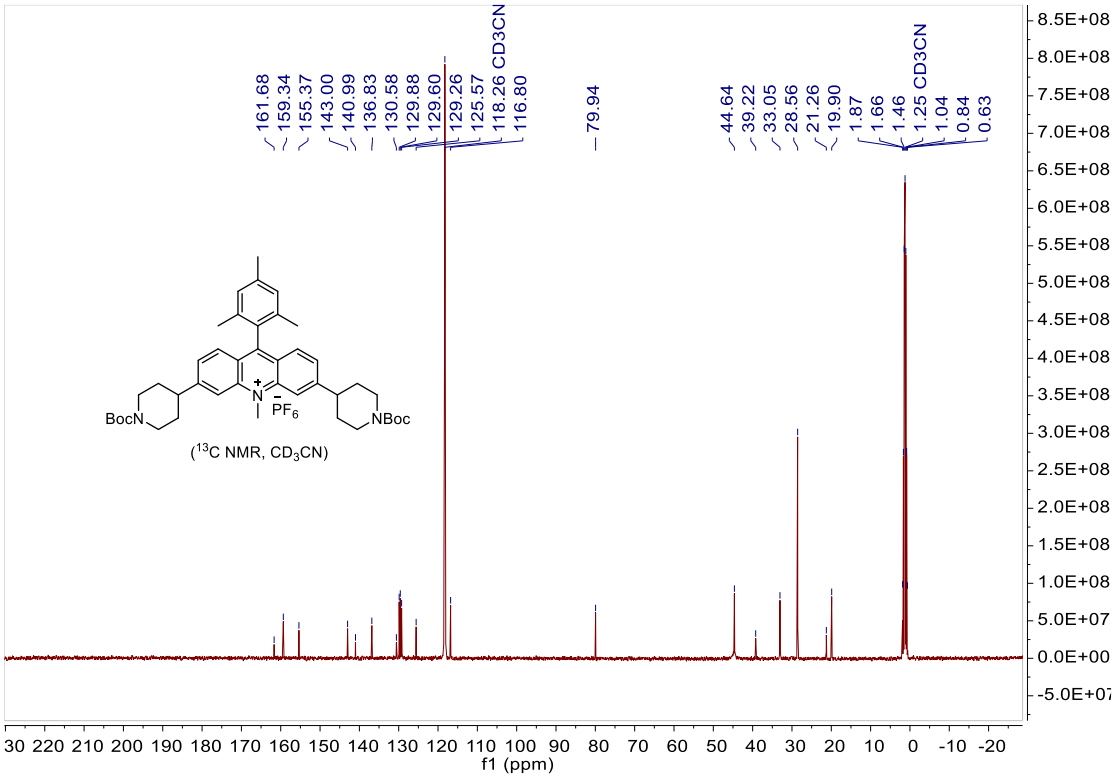

-71.93
-73.81
PF6
(^{19}F NMR, CD_3CN)
f1 (ppm)


8.59 8.57 8.52 8.39 8.38 8.37 8.37 8.37 8.36 8.36 8.36 8.35 7.92 7.92 7.91 7.91 7.91 7.90 7.89 7.87 7.86 7.85 7.85 7.84 7.83 7.82 7.82 7.81 7.81 7.79 7.78 7.76 7.21 5.22 4.78 2.45 2.17 HDO 1.95 1.94 1.94 CD3CN 1.93 1.93 1.68
1.01 1.01 1.07 2.05 6.36 2.00 1.97 3.00 3.06 6.06
f1 (ppm)
(^{1}H NMR, CD_3CN)


169.07 162.95 149.87 142.85 142.79 141.15 139.81 136.92 135.57 133.14 130.49 130.46 129.89 129.65 129.30 128.78 126.94 126.33 124.27 119.83 118.26 CD3CN 117.75
42.70 39.64
21.28 19.94
1.60 1.44 1.27 CD3CN 1.11 0.94 0.78
f1 (ppm)
(^{13}C NMR, CD_3CN)


-72.17
-73.67
O
O
N
N
PF6
(19F NMR, CD3CN)
f1 (ppm)


8.32
7.72
7.22
4.77
4.29
4.25
3.22
3.20
3.19
3.19
3.18
3.16
2.91
2.46
2.21 HDO
1.97
1.97
1.96
1.95
1.95
1.94 CD3CN
1.93
1.93
1.83
1.82
1.79
1.78
1.76
1.75
1.75
1.73
1.72
1.69
1.46
BocN
NBoc
PF6
(1H NMR, CD3CN)
2.08
4.13
2.10
3.00
3.99
2.08
3.96
3.09
4.77
4.56
6.18
18.49
f1 (ppm)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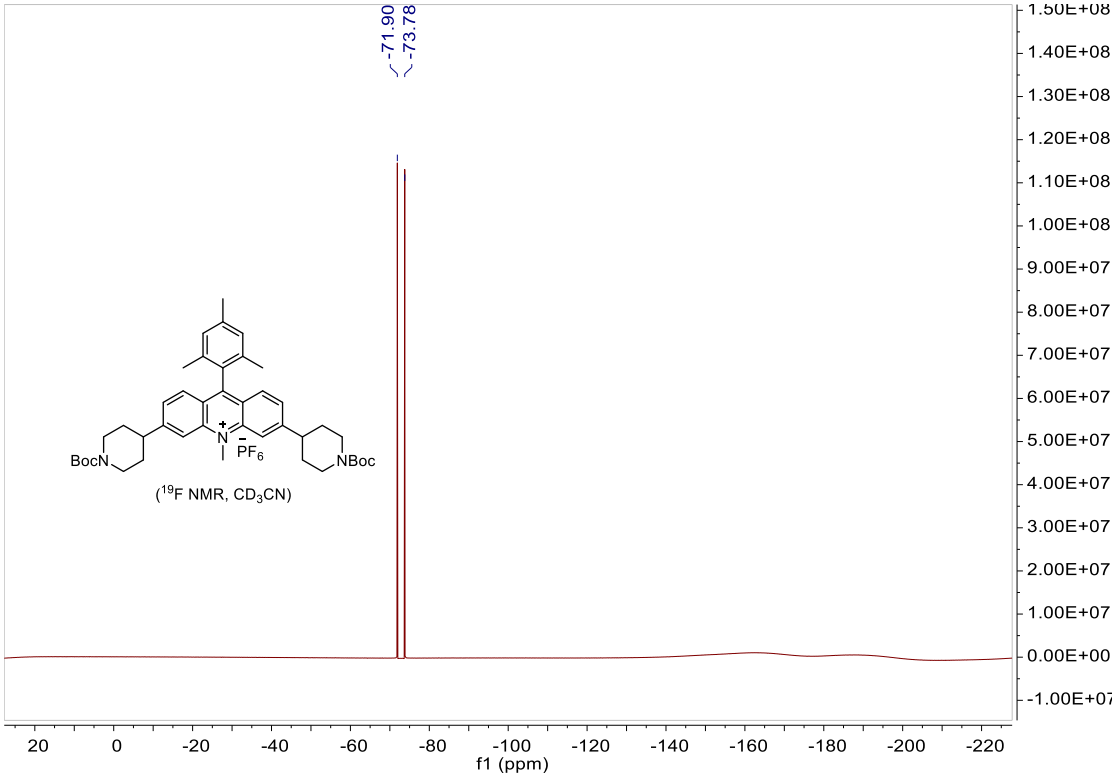
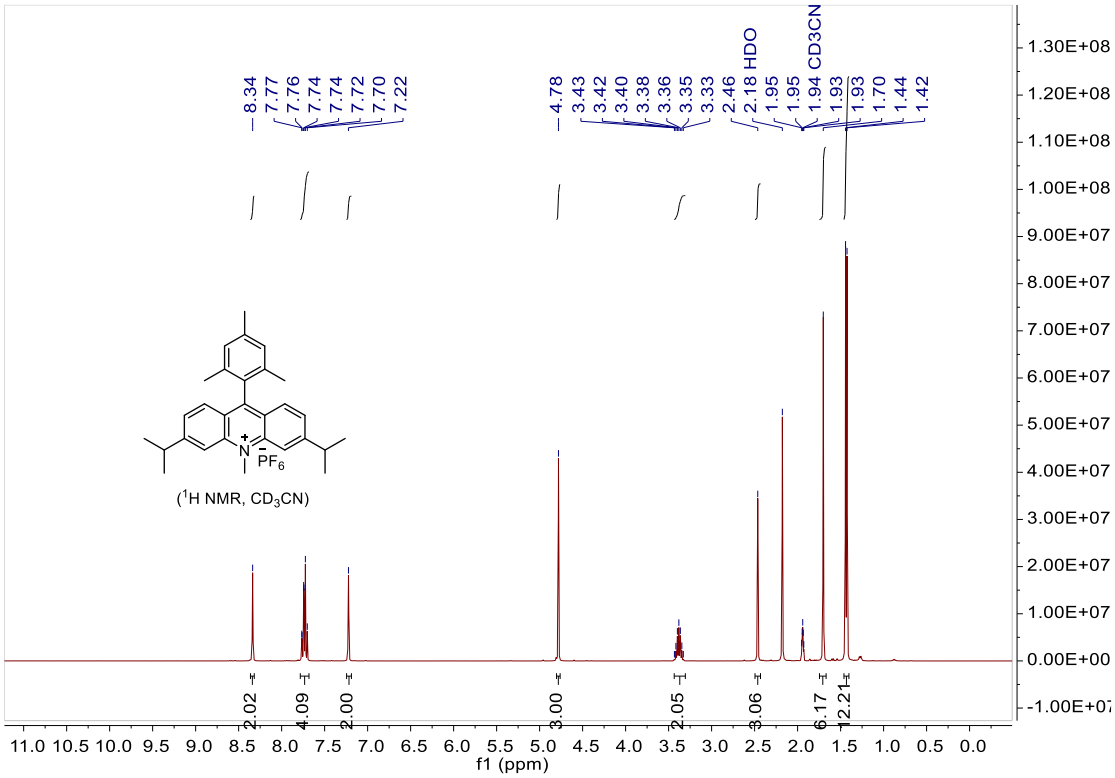
136.83
130.58
129.88
129.60
129.26
125.57
118.26 CD3CN
116.80
79.94
44.64
39.22
33.05
28.56
21.26
19.90
1.87
1.66
1.46
1.25 CD3CN
1.04
0.84
0.63
BocN
NBoc
PF6
(13C NMR, CD3CN)
f1 (ppm)


-71.90
-73.78
BocN
NBoc
PF6
(19F NMR, CD3CN)
f1 (ppm)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7.76
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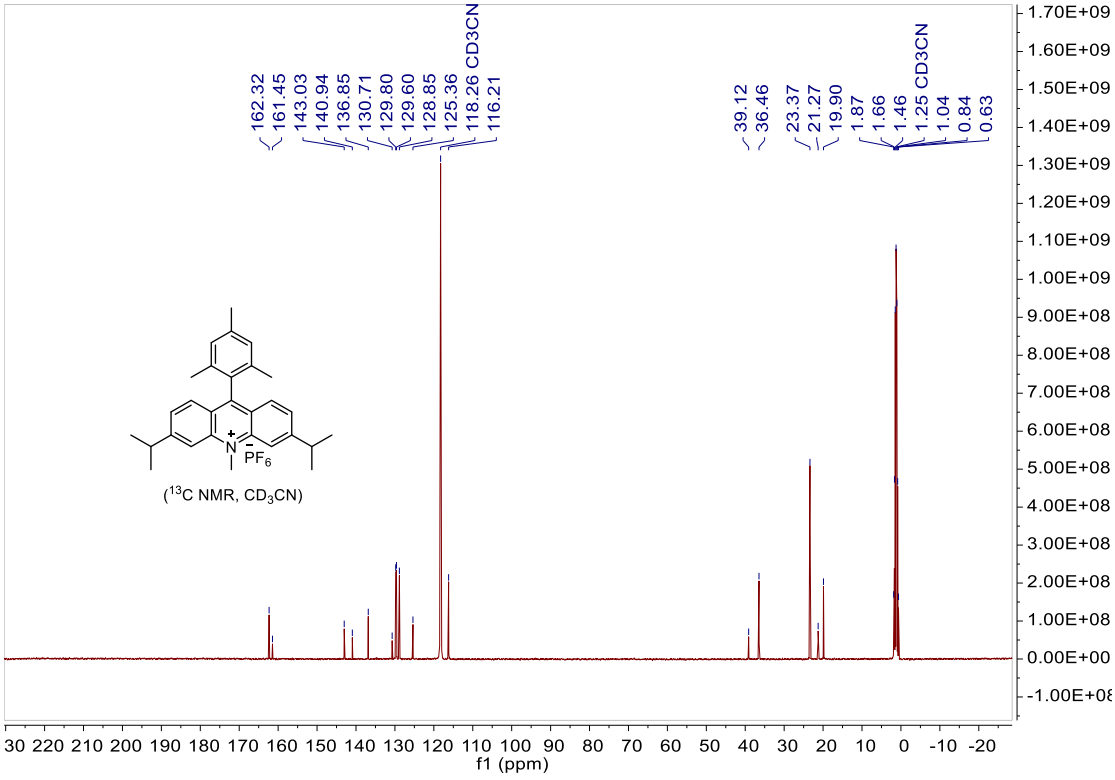
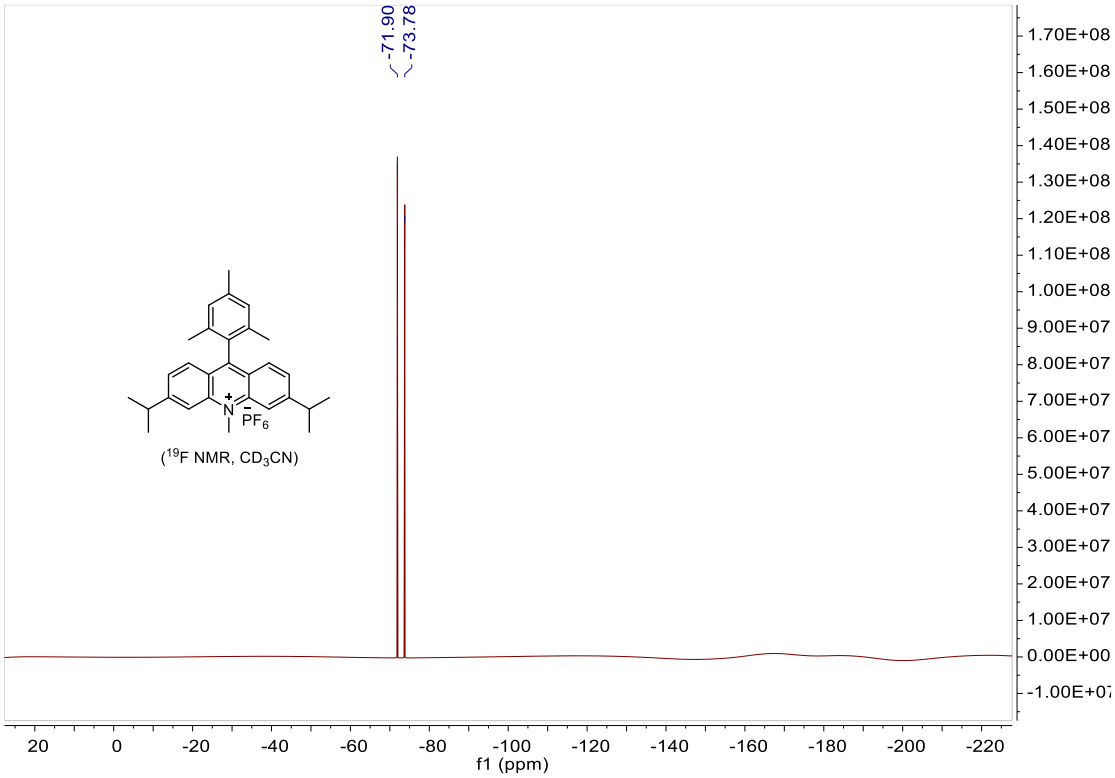
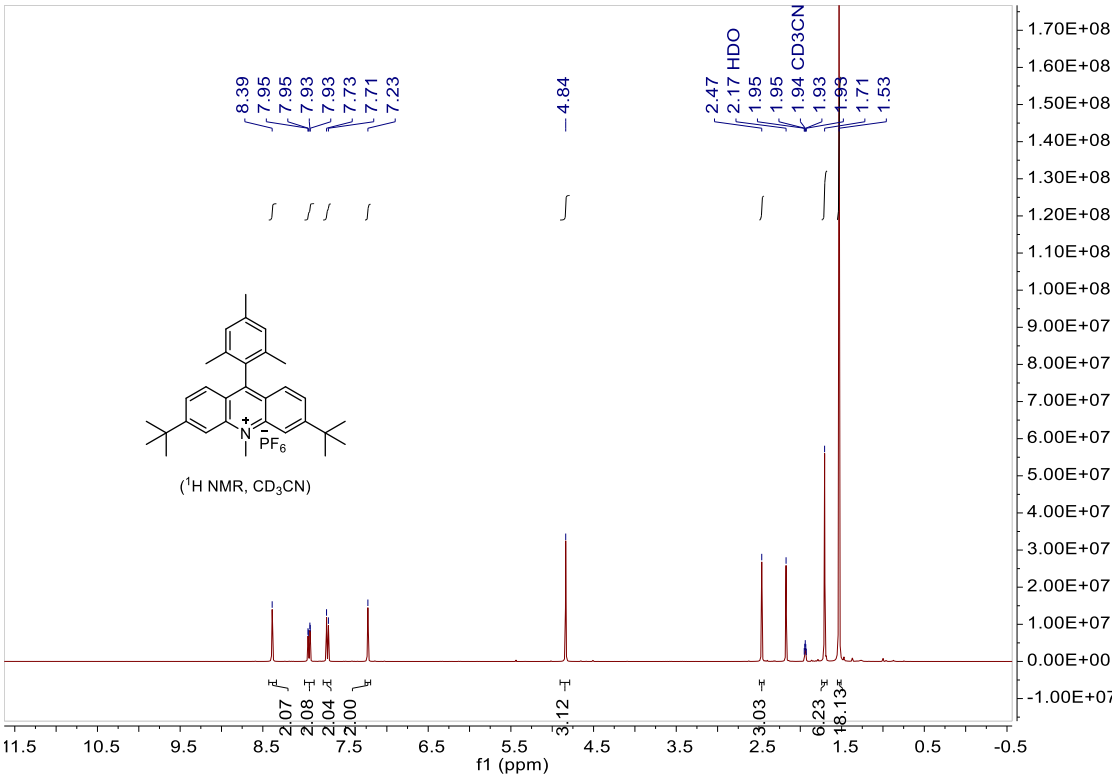
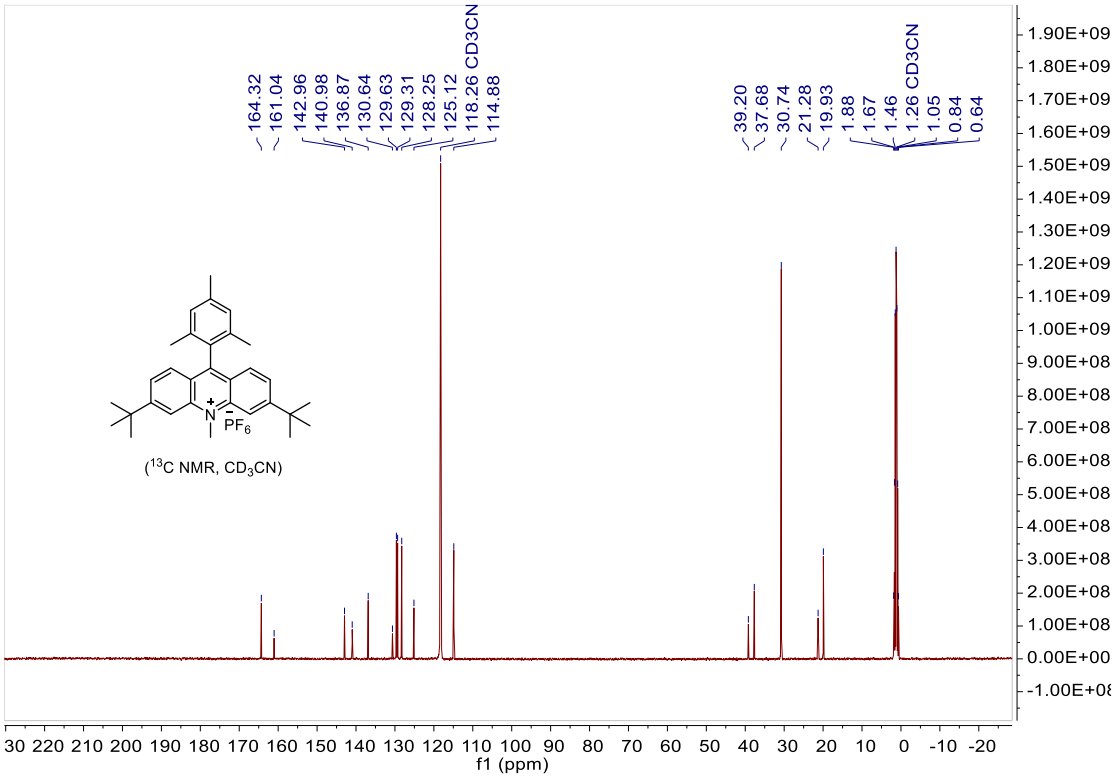
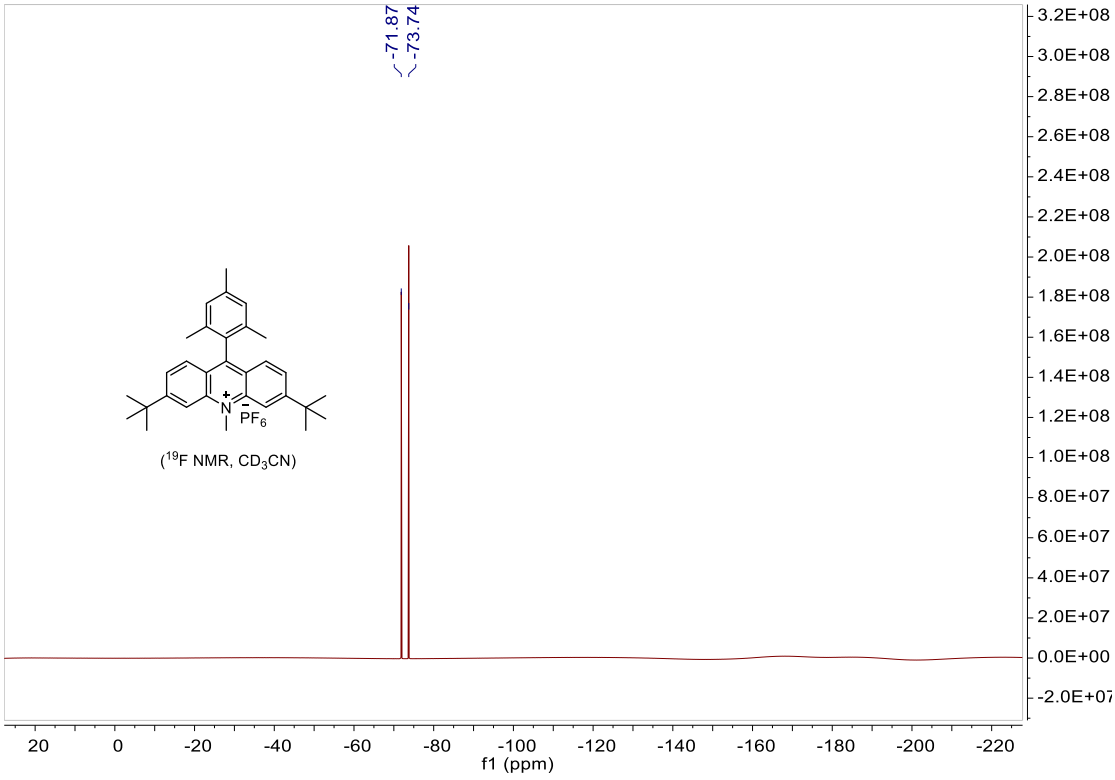
7.74
7.72
7.70
7.22
4.78
3.43
3.42
3.40
3.38
3.36
3.35
3.33
2.46
2.18 HDO
1.95
1.95
1.94 CD3CN
1.93
1.93
1.70
1.44
1.42
2.02
4.09
2.00
3.00
2.05
3.06
6.17
12.21
PF6
(1H NMR, CD3CN)
f1 (ppm)


162.32
161.45
143.03
140.94
136.85
130.71
129.80
129.60
128.85
125.36
118.26 CD3CN
116.21
39.12
36.46
23.37
21.27
19.90
1.87
1.66
1.46
1.25 CD3CN
1.04
0.84
0.63
PF6
(13C NMR, CD3CN)
f1 (ppm)


-71.90
-73.78
PF6
(19F NMR, CD3CN)
f1 (ppm)


8.39
7.95
7.95
7.93
7.93
7.73
7.71
7.23
4.84
2.47
2.17 HDO
1.95
1.95
1.94 CD3CN
1.93
1.93
1.71
1.53
2.07
2.08
2.04
2.00
3.12
3.03
6.23
18.13
PF6
(1H NMR, CD3CN)
f1 (ppm)


164.32
161.04
142.96
140.98
136.87
130.64
129.63
129.31
128.25
125.12
118.26 CD3CN
114.88
39.20
37.68
30.74
21.28
19.93
1.88
1.67
1.46
1.26 CD3CN
1.05
0.84
0.64
PF6
(13C NMR, CD3CN)
f1 (ppm)


-71.87
-73.74
PF6
(19F NMR, CD3CN)
f1 (ppm)