

Supplementary Material

Synthesis and electrochemical properties of tetramethyl ammonium salts of $[(\text{PhO})\text{Ni}(\text{CF}_3)_3]^{2-}$ and $[(7\text{-azaindole})\text{Ni}(\text{CF}_3)_3]^{2-}$

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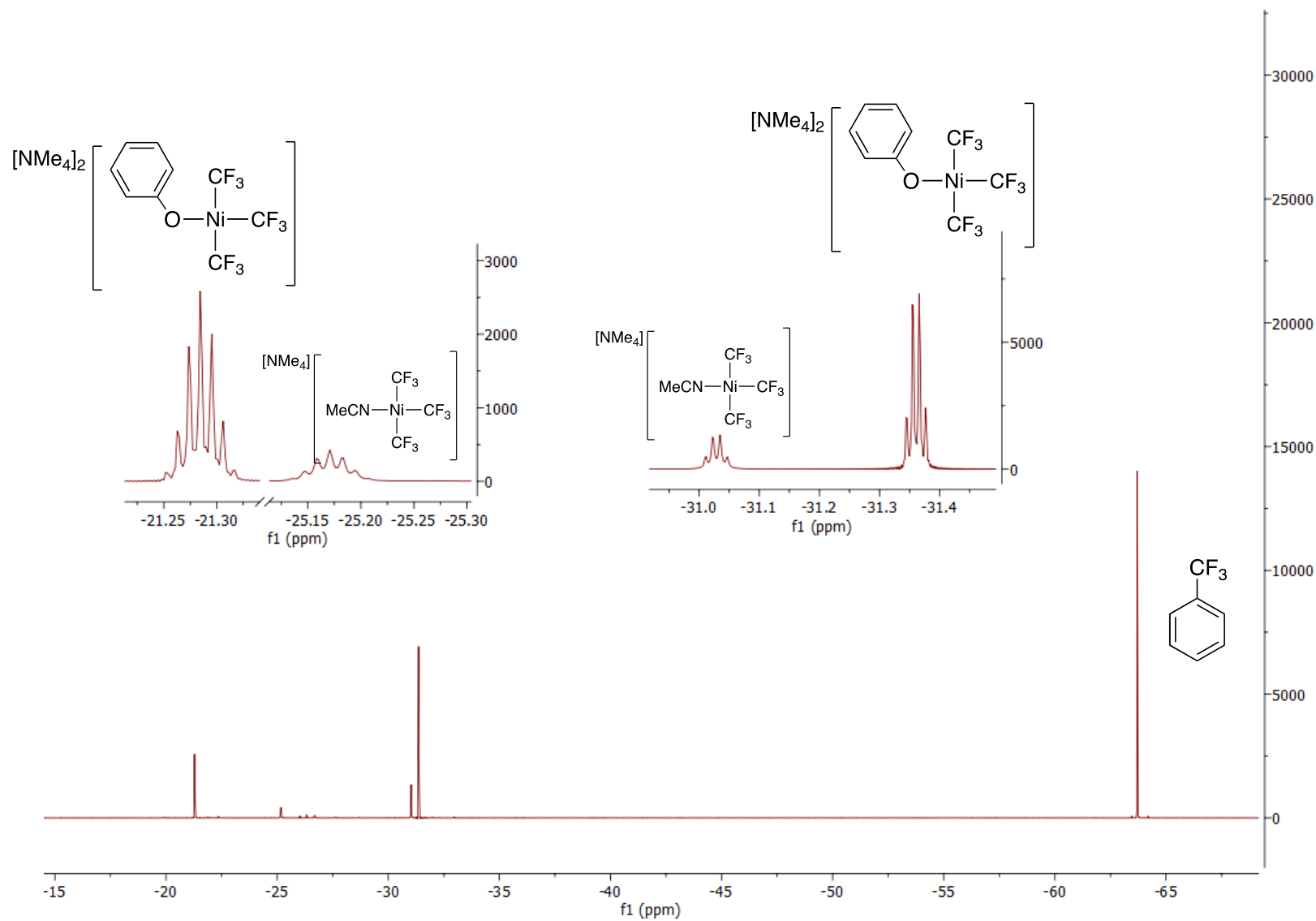
Figure S1. ^{19}F NMR of $[\text{NMe}_4]_2[(\text{PhO})\text{Ni}(\text{CF}_3)_3]$ (**2**) at room temperature in CD_3CN 

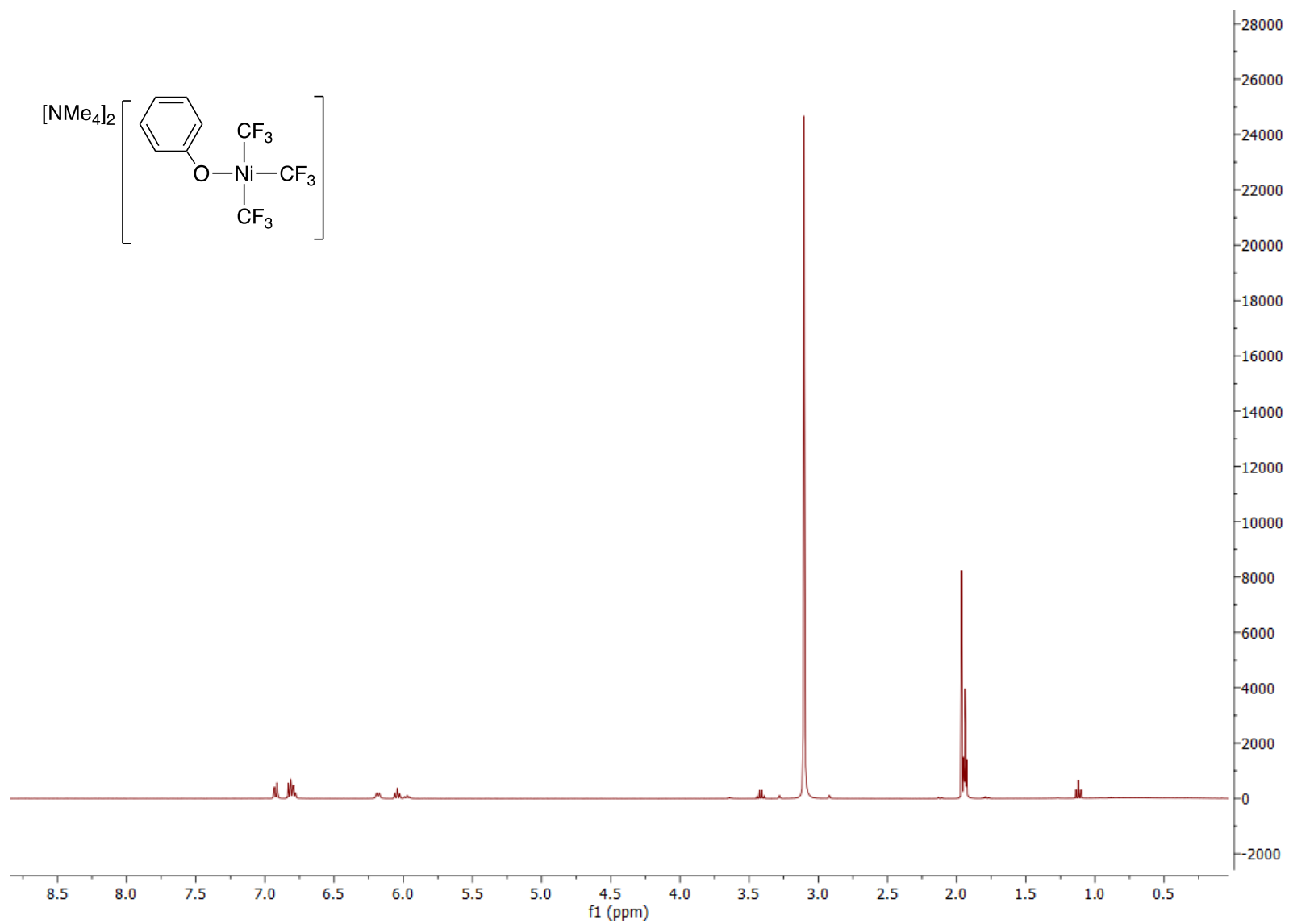
Figure S2. ^1H NMR of $[\text{NMe}_4]_2[(\text{PhO})\text{Ni}(\text{CF}_3)_3]$ (**2**) at room temperature in CD_3CN 

Figure S3. ^1H NMR of $[\text{NMe}_4]_2[(\text{PhO})\text{Ni}(\text{CF}_3)_3]$ (**X**) in CD_3CN at room temperature (top), **2** in CD_3CN at -30°C (middle) and NMe_4OPh in CD_3CN at room temperature

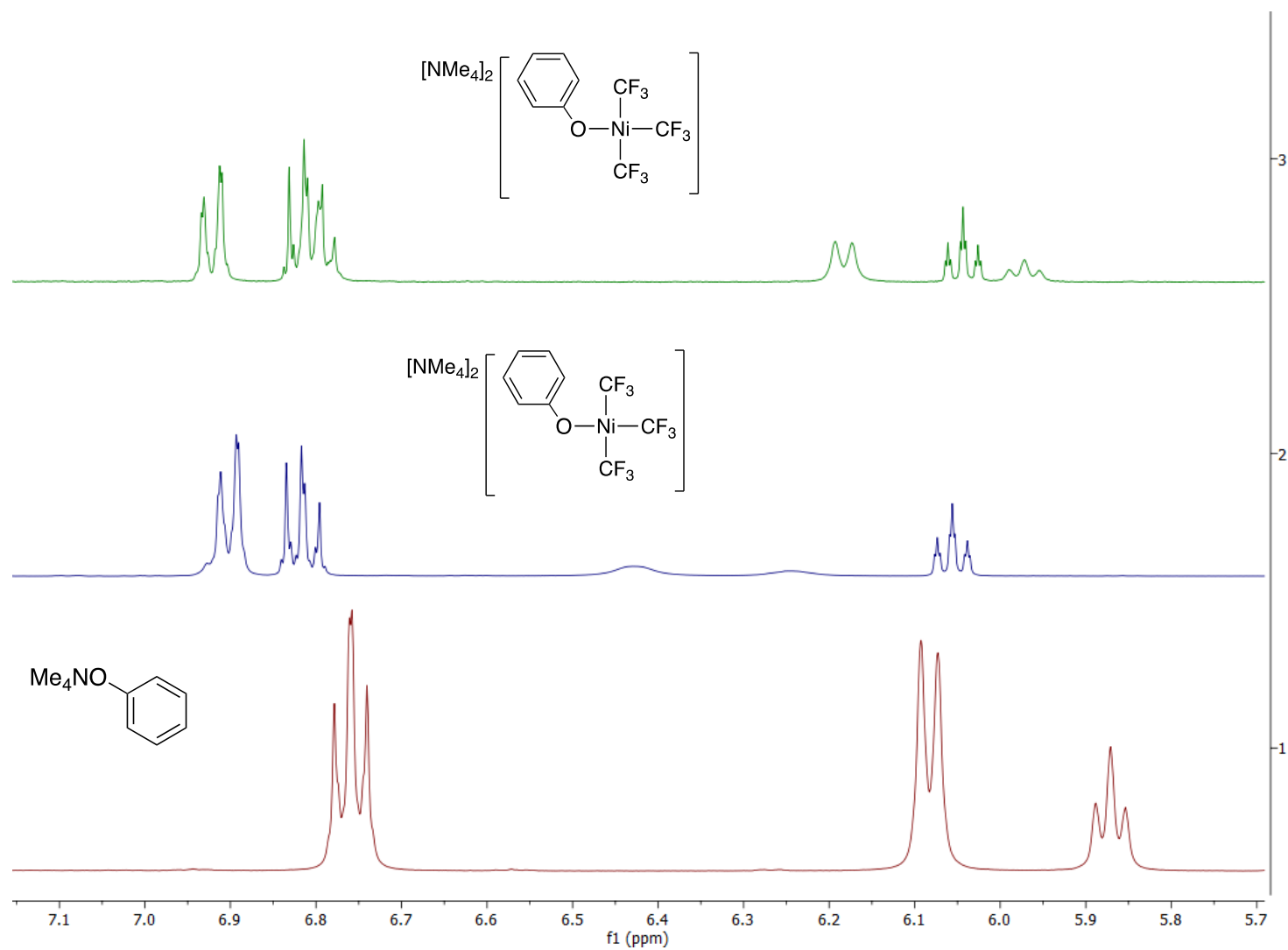


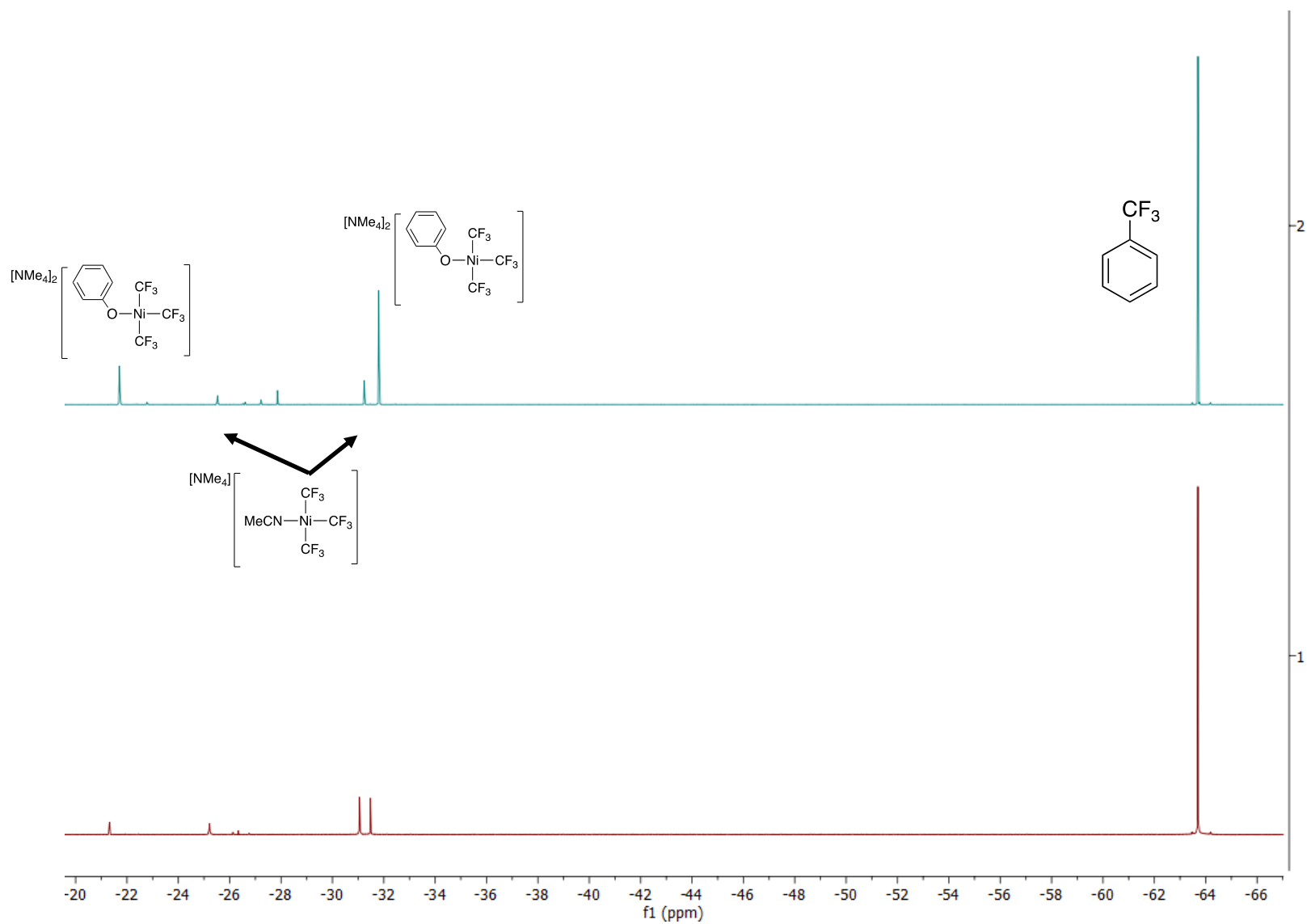
Figure S4. ^{19}F NMR of $[\text{NMe}_4]_2[(\text{PhO})\text{Ni}(\text{CF}_3)_3]$ (**2**) at -30°C (top) and room temperature (bottom) in CD_3CN 

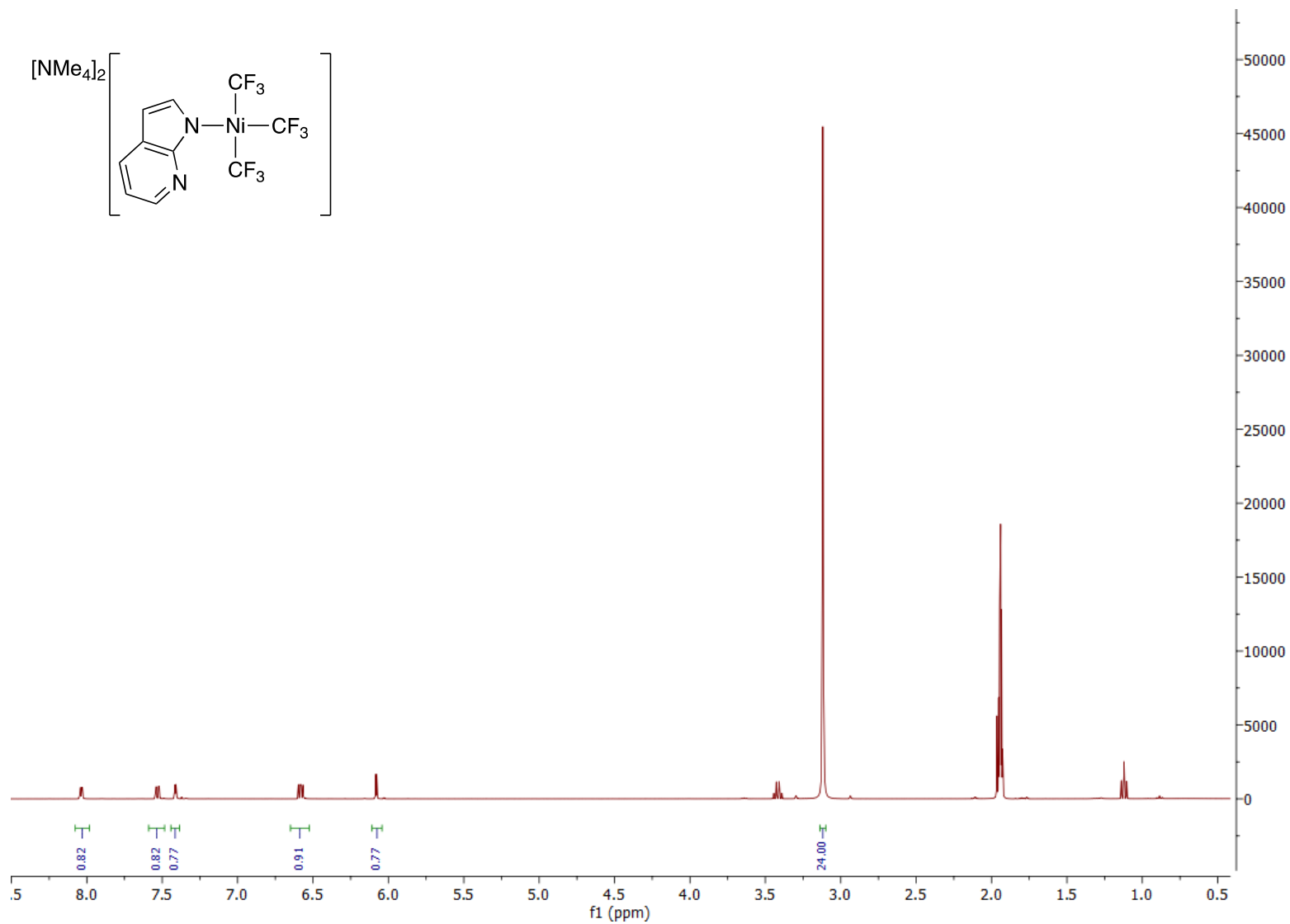
Figure S5. ^1H NMR of $[\text{NMe}_4]_2[(7\text{-azaindole})\text{Ni}(\text{CF}_3)_3]$ (**3**) in CD_3CN at room temperature

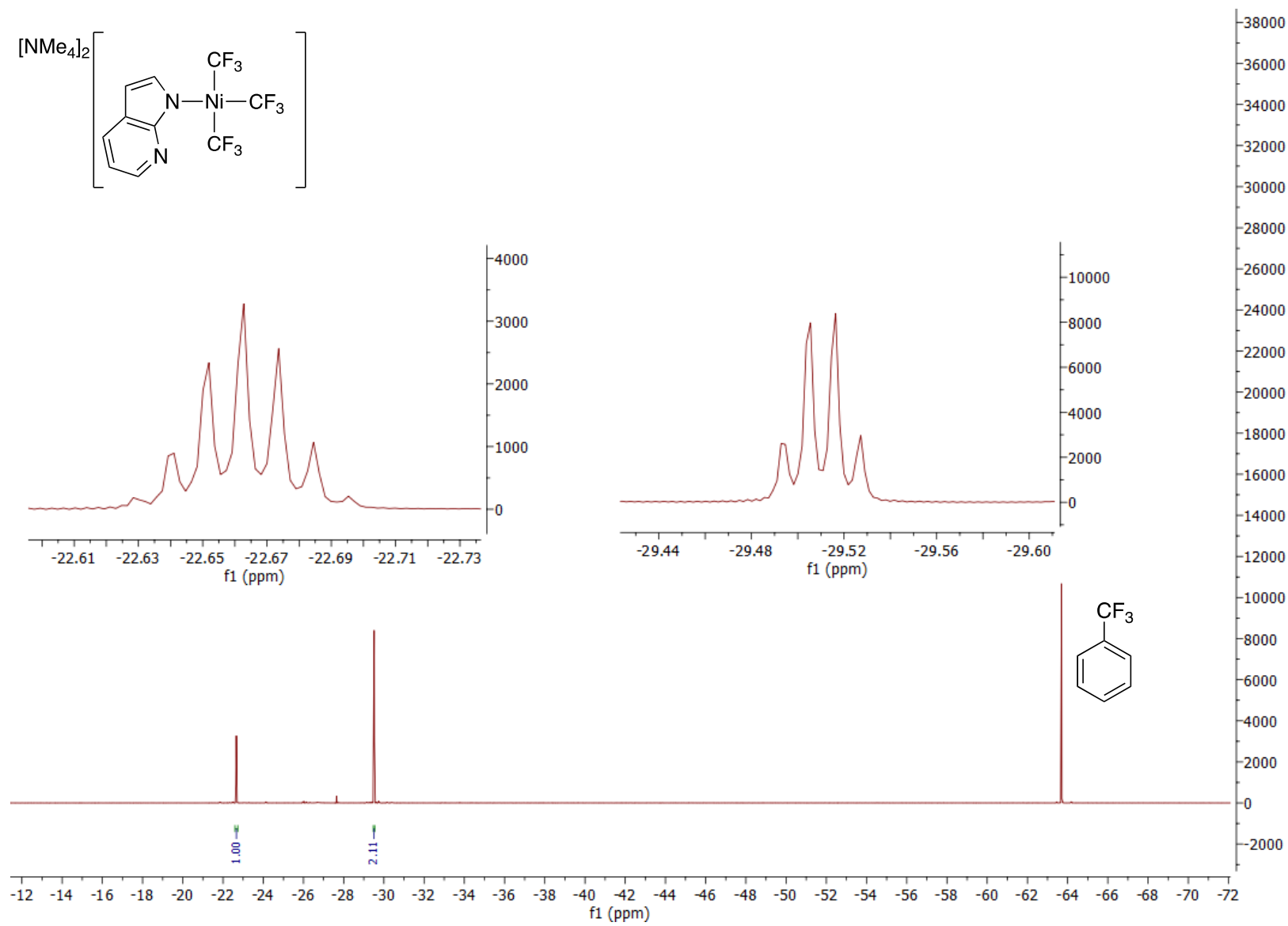
Figure S6. ^{19}F NMR of $[\text{NMe}_4]_2[(7\text{-azaindole})\text{Ni}(\text{CF}_3)_3]$ (**3**) in CD_3CN at room temperature

Figure S7. Cyclic voltammogram of $[\text{NMe}_4]_2[(\text{PhO})\text{Ni}(\text{CF}_3)_3]$ (**2**, blue), $[\text{NMe}_4][\text{PhO}]$ (red), and $[\text{NMe}_4][(\text{MeCN})\text{Ni}(\text{CF}_3)_3]$ (**1**, black) in MeCN. Complex, 10 mM; electrolyte, 100 mM $[\text{NBu}_4][\text{PF}_6]$; working and counter electrode, platinum; silver pseudoreference; scan rate 100 mVs^{-1} .

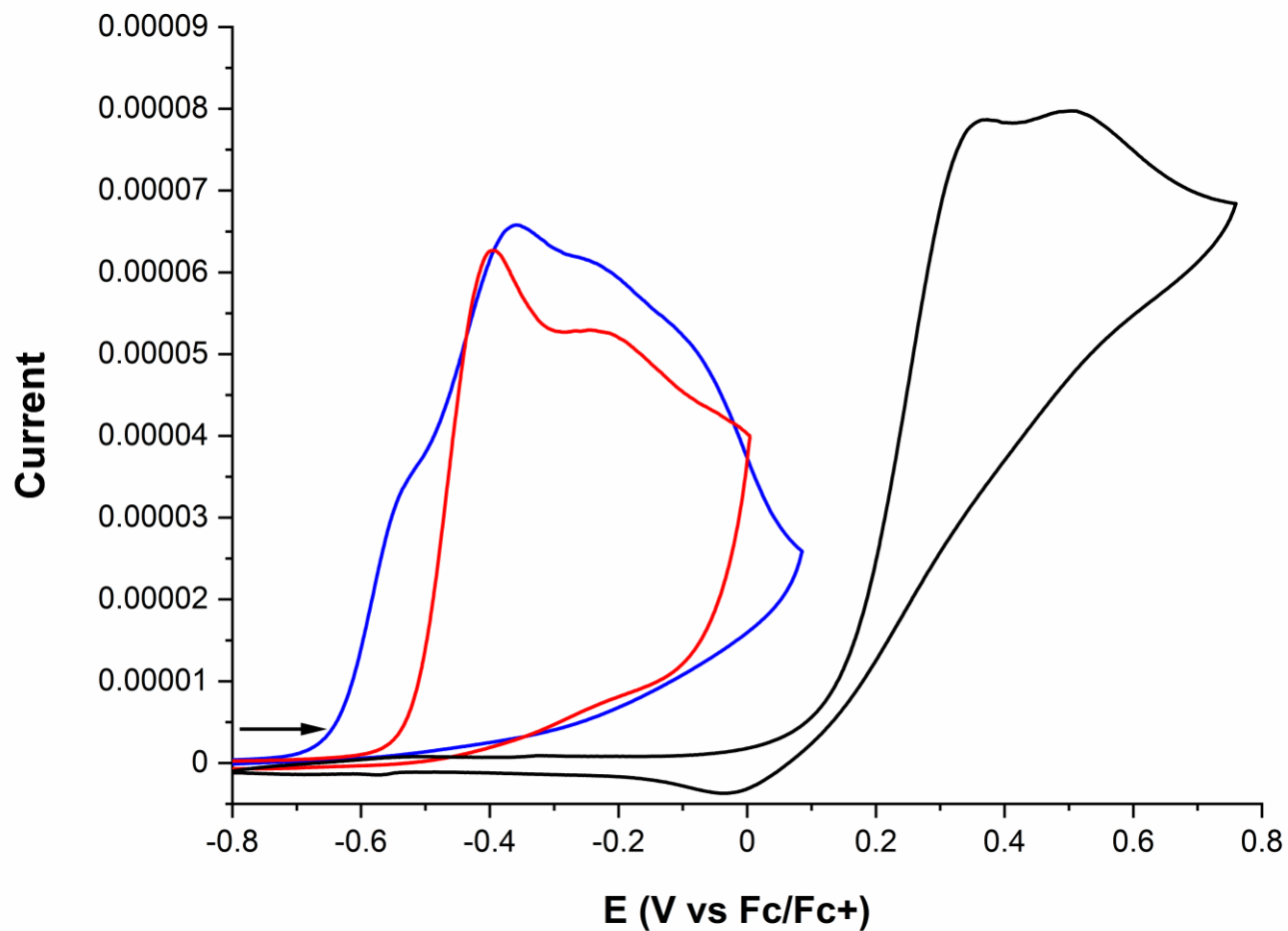


Figure S8. Cyclic voltammogram of $[\text{NMe}_4]_2[(7\text{-azaindole})\text{Ni}(\text{CF}_3)_3]$ (**3**, blue), $[\text{K}][7\text{-azaindole}]$ (red), and $[\text{NMe}_4][(\text{MeCN})\text{Ni}(\text{CF}_3)_3]$ (**1**, black) in MeCN. Complex, 10 mM; electrolyte, 100 mM $[\text{NBu}_4][\text{PF}_6]$; working and counter electrode, platinum; silver pseudoreference; scan rate 100 mVs^{-1} .

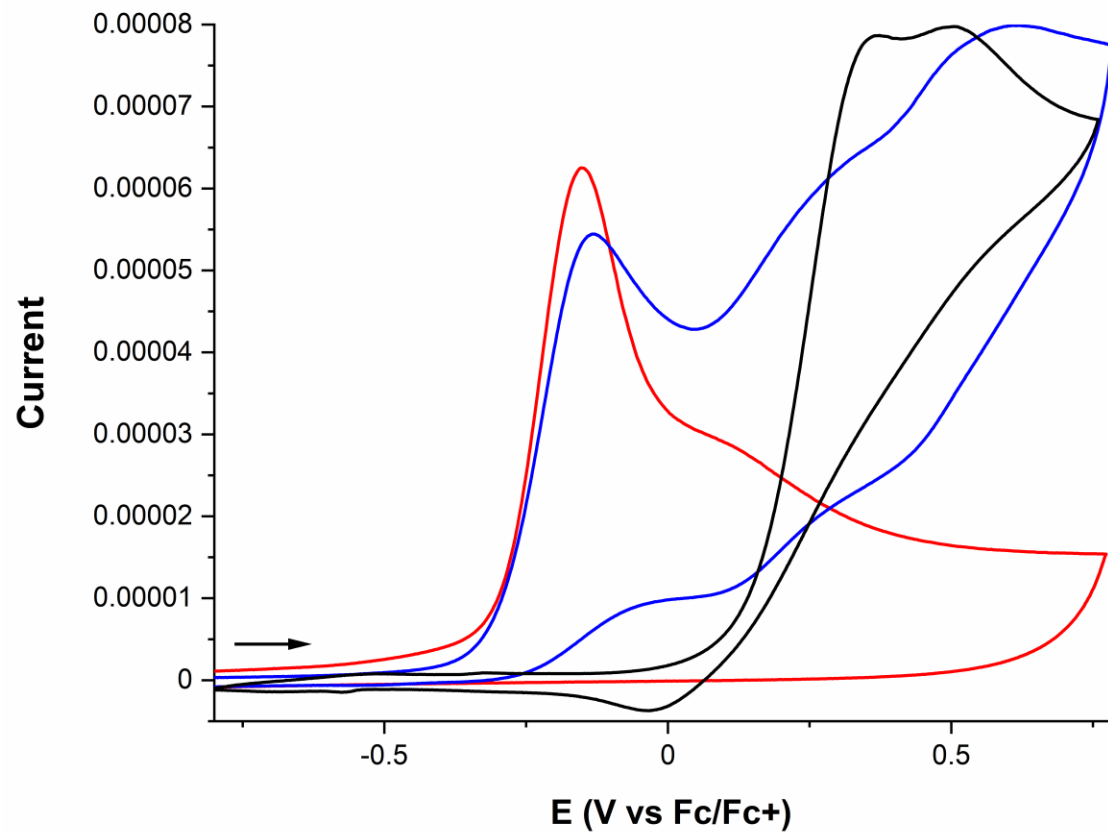


Figure S9. Cyclic voltammogram of first oxidation waves of $[\text{NMe}_4]_2[(\text{PhO})\text{Ni}(\text{CF}_3)_3]$ (**2**, black) and $[\text{NMe}_4]_2[(7\text{-azaindole})\text{Ni}(\text{CF}_3)_3]$ (**3**, blue) in MeCN. Complex, 10 mM; electrolyte, 100 mM $[\text{NBu}_4][\text{PF}_6]$; working and counter electrode, platinum; silver pseudoreference; scan rate 100 mVs^{-1} .

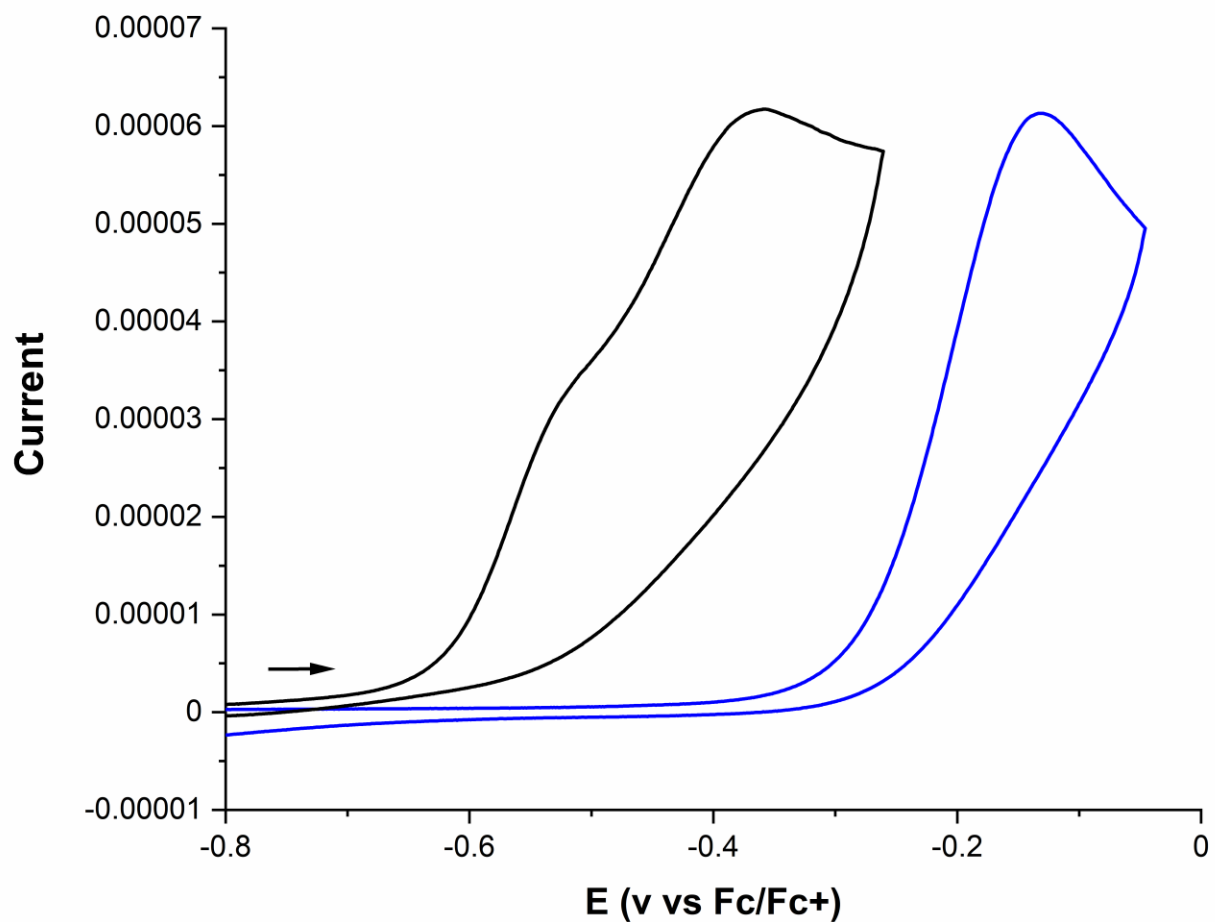


Figure S10. Cyclic voltammogram (full spectrum) of $[\text{NMe}_4]_2[(\text{PhO})\text{Ni}(\text{CF}_3)_3]$ (**2**, black) and $[\text{NMe}_4]_2[(7\text{-azaindole})\text{Ni}(\text{CF}_3)_3]$ (**3**, blue) in MeCN. Complex, 10 mM; electrolyte, 100 mM $[\text{NBu}_4][\text{PF}_6]$; working and counter electrode, platinum; silver pseudoreference; scan rate 100 mVs^{-1} .

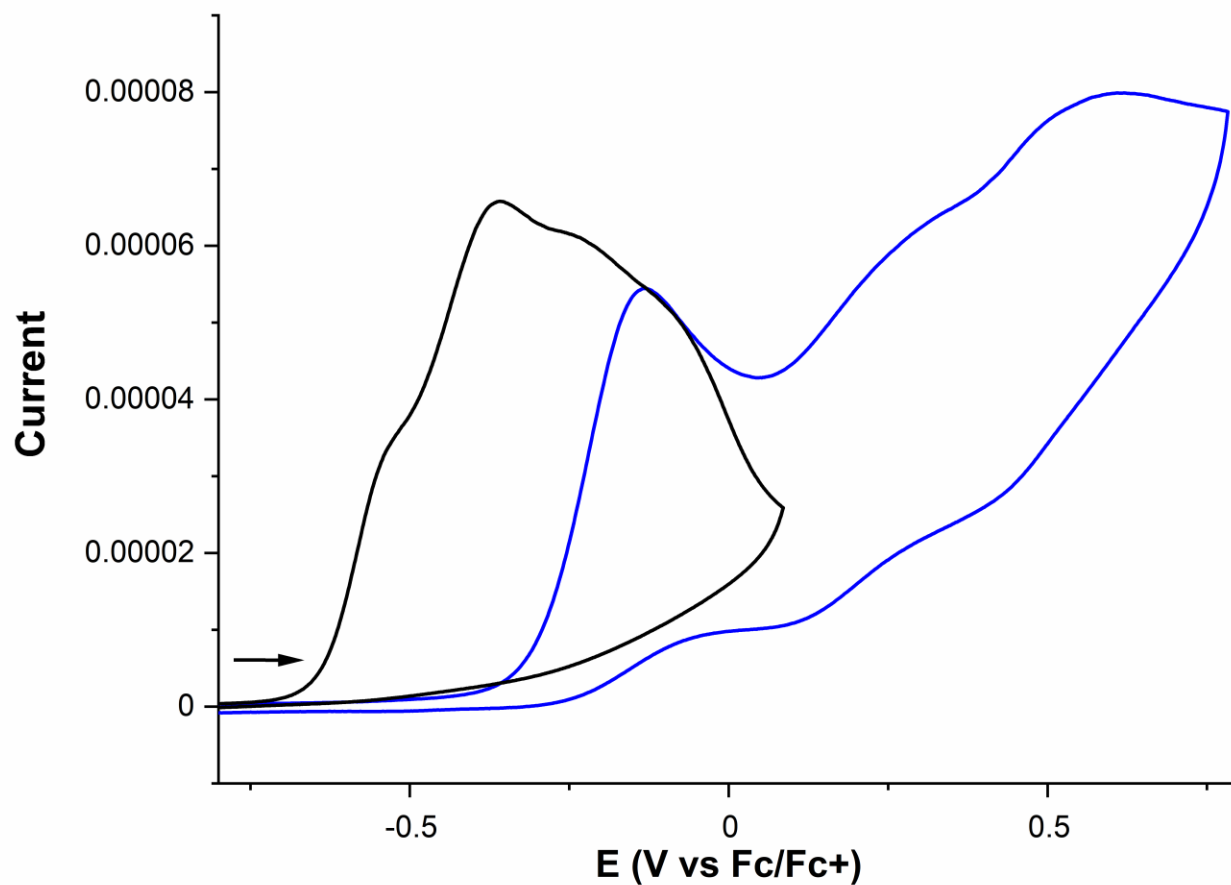


Figure S11. Preliminary X-ray Structure of Compound **3**. Structure has four independent molecules of **3**, two of which have unique disorders. A molecule of [NMe₄]Cl is also co-crystallized, with the chlorine disordered over two sites. Data has been deposited as CCDC 2203186.

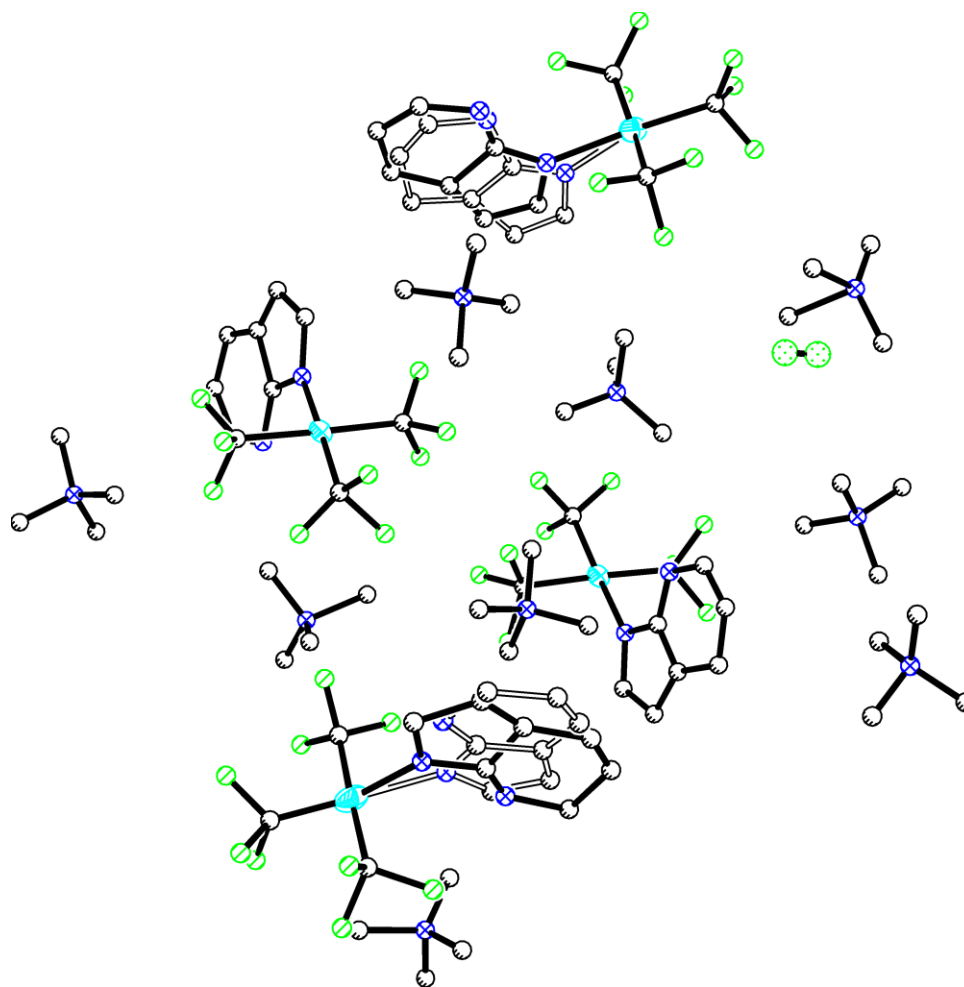


Figure S12. Preliminary X-ray Structure of Compound **3**, showing the two non-disordered cations in the unit cell.

