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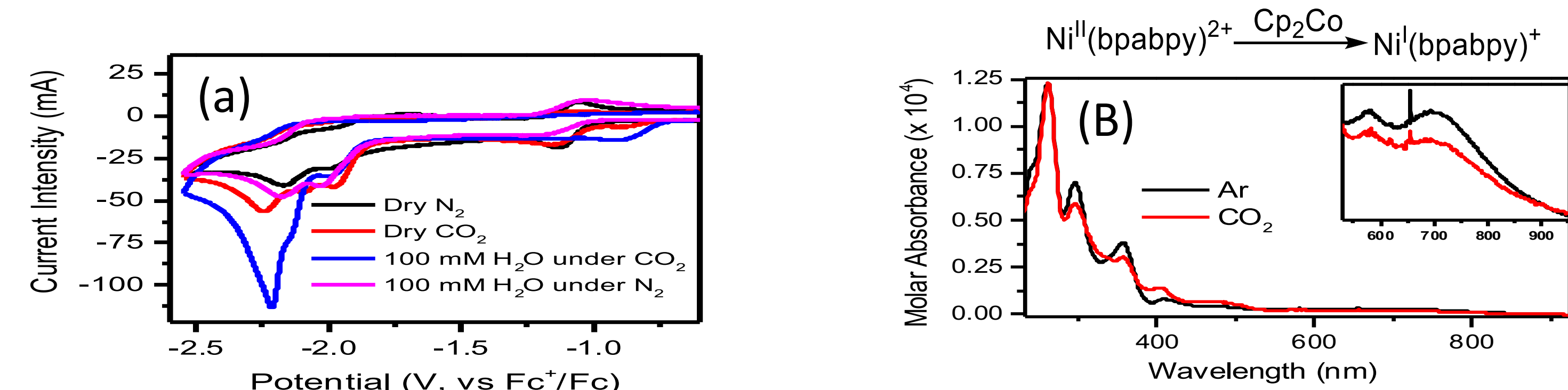
# A Ni<sup>II</sup> Complex Electrocatalyst for CO<sub>2</sub> Reduction and H<sub>2</sub> Evolution

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**Figure 1 – CV Curves, UV-vis Absorption**



(A) CV curves of Ni<sup>II</sup> complex with/without CO<sub>2</sub> and H<sub>2</sub>O. (B) UV-vis absorption of Ni<sup>II</sup> complex in the absence (black)/presence (red) of CO<sub>2</sub>.

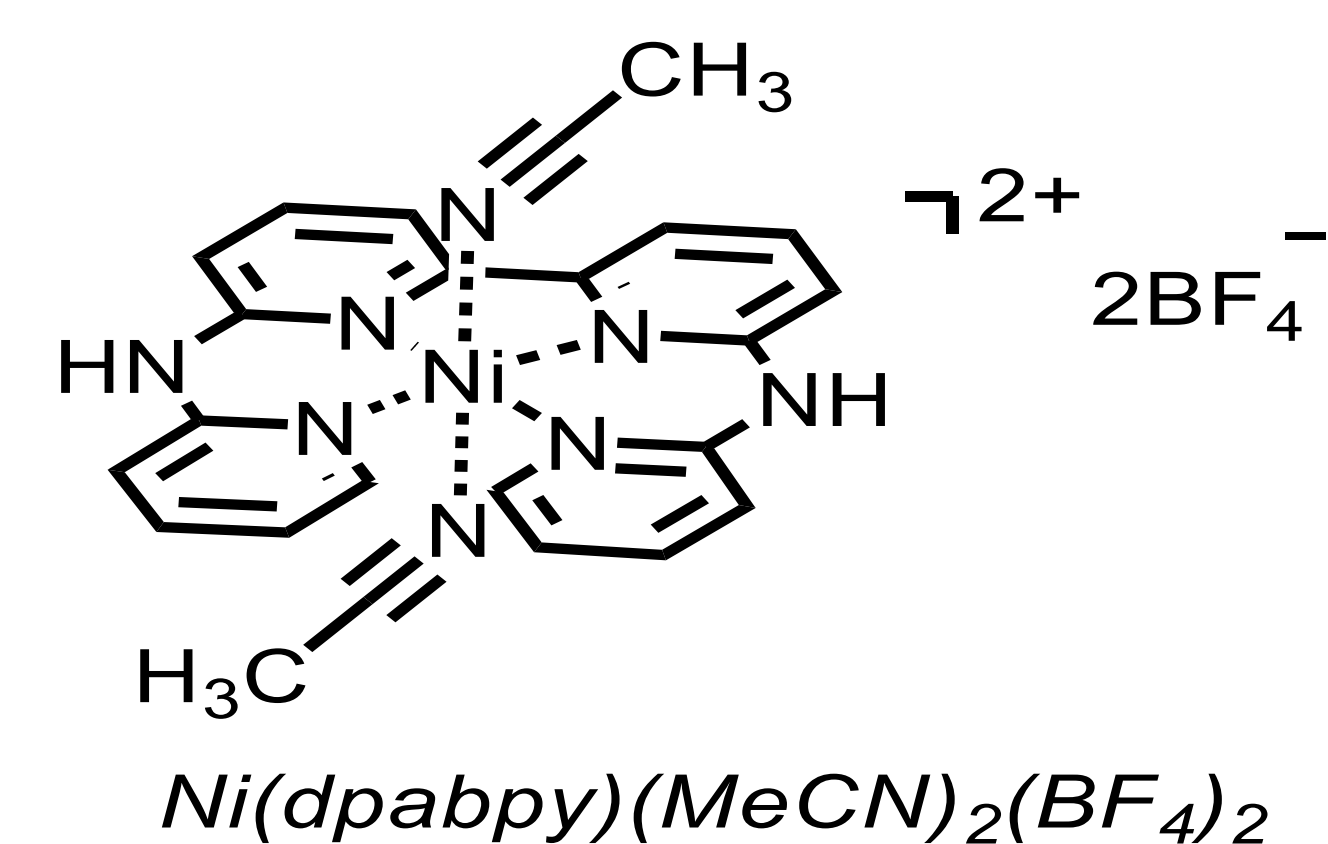
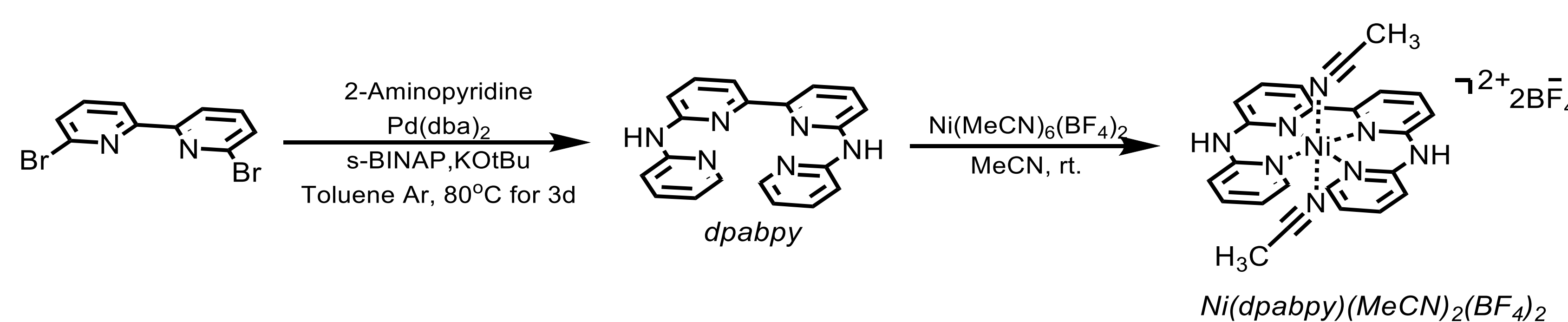
## Results and Discussion

The Ni<sup>II</sup>(bpbppy)<sub>2</sub>(BF<sub>4</sub>)<sub>2</sub> complex was synthesized as shown in **Scheme 1**. The structures of the products were confirmed by NMR and single crystal X-ray diffraction. The electrochemical study of the Ni<sup>II</sup> complex showed a reversible Ni<sup>II</sup>/Ni<sup>I</sup> redox peak at -1.09 V (vs. Fc<sup>+/0</sup>) under N<sub>2</sub>.

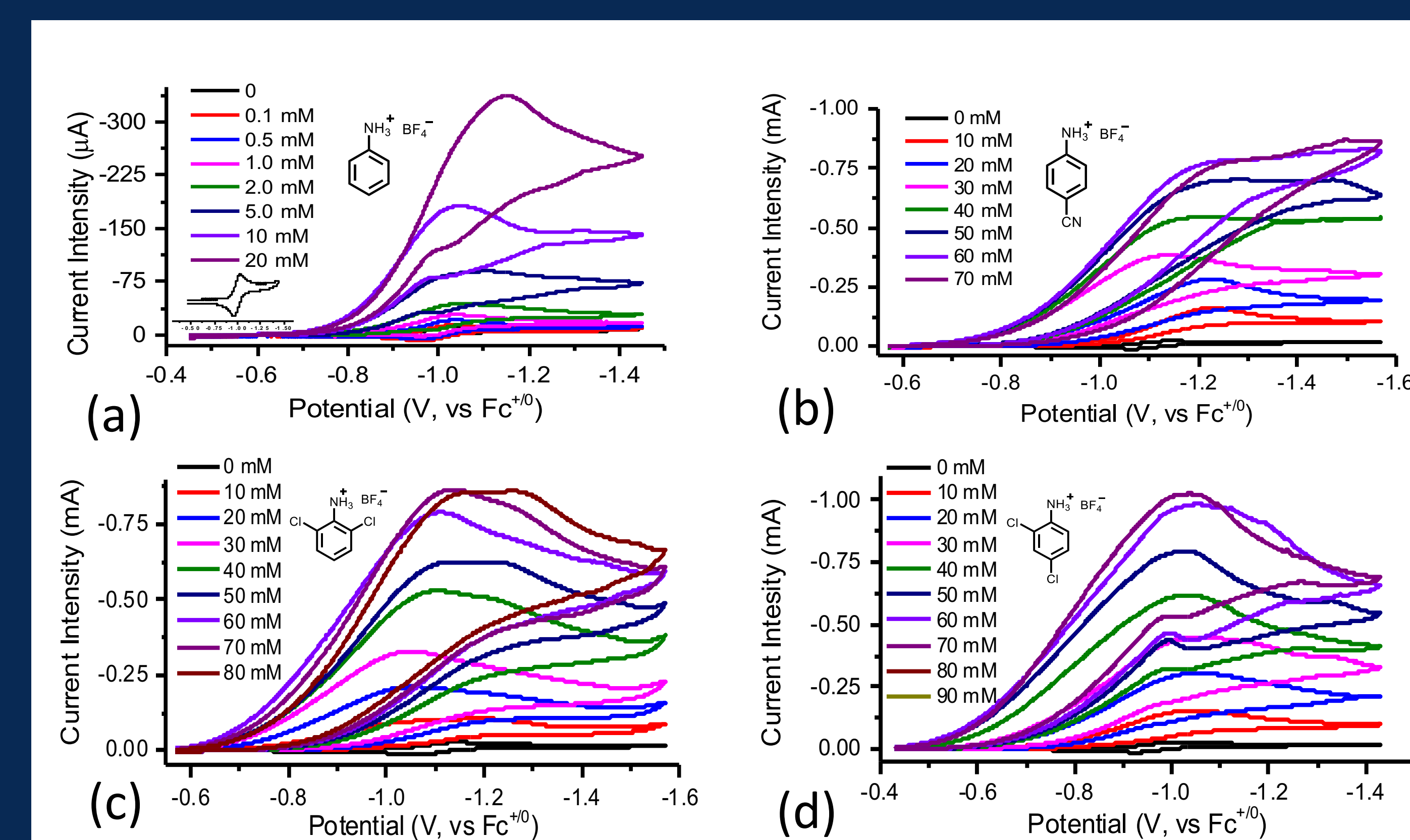
In the presence of CO<sub>2</sub>, the Ni<sup>II</sup>/Ni<sup>I</sup> peak turned to irreversible. In the presence of H<sub>2</sub>O, the typical CO<sub>2</sub> reduction peak, at -2.24 V (vs. Fc<sup>+/0</sup>), signal was remarkably enhanced (**Figure 1a**). The UV-vis absorption shows the coordination of CO<sub>2</sub> to Ni<sup>I</sup> complex.

GC and NMR were used to confirm that CO is the only product for the CO<sub>2</sub> reduction. In the presence of 5 mM of H<sub>2</sub>O, CO<sub>2</sub> was selectively reduced to CO with 57% Faradaic efficiency, further increase of the concentration of H<sub>2</sub>O showed a remarkable H<sub>2</sub> evolution reaction and the selectivity of CO formation was decreased. As shown in **Figure 2**, in the presence of acid, the proton reduction peaks were increased for more than 20 times. To our surprise, in the case of 2,4-Cl<sub>2</sub>PhNH<sub>3</sub><sup>+</sup>, there is a super-low overpotential for the H<sub>2</sub> evolution (only 0.04 V) with 239 s<sup>-1</sup> TOF.

**Scheme 1- Synthesis of Ni<sup>II</sup>(bpbppy)(BF<sub>4</sub>)<sub>2</sub>**



**Figure 2 – H<sub>2</sub> Evolution with Acids**



CV (a) PhNH<sub>3</sub>BF<sub>4</sub>, (b) CNPhNH<sub>3</sub>BF<sub>4</sub>, (c) 2,6-Cl<sub>2</sub>PhNH<sub>3</sub>BF<sub>4</sub>, (d) 2,4-Cl<sub>2</sub>PhNH<sub>3</sub>BF<sub>4</sub>.

## Conclusion

In conclusion, a Ni<sup>II</sup> complex with a N-tetradentate ligand bpbppy was prepared as a catalyst for CO<sub>2</sub> reduction and H<sub>2</sub> evolution. In the presence of 5 mM H<sub>2</sub>O, the Ni<sup>II</sup> complex showed good selectivity and efficiency for CO formation. The Ni<sup>II</sup> complex also showed a very low overpotential and high efficiency for the H<sub>2</sub> evolution.

**Table 1- H<sub>2</sub> Evolution Overpotential (OP) and Turnover Frequency (TOF) with Acid**

Acid	OP (V)	TOF (s <sup>-1</sup> )
PhNH <sub>3</sub> BF <sub>4</sub>	0.17	140
CNPhNH <sub>3</sub> BF <sub>4</sub>	0.39	195
2,6-Cl <sub>2</sub> PhNH <sub>3</sub> BF <sub>4</sub>	0.36	179
2,4-Cl <sub>2</sub> PhNH <sub>3</sub> BF <sub>4</sub>	0.04	239

