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#### Research paper

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### **ACCEPTED MANUSCRIPT**

## Modifying the steric and electronic character within

Re(I)-phenanthroline complexes for electrocatalytic CO<sub>2</sub>

reduction

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**Abstract**: We have synthesized and characterized a series of fac-[Re(R<sub>2</sub>phen)(CO)<sub>3</sub>Cl] complexes (R<sub>2</sub>phen = 2,9-disubstituted-1,10-phenanthroline) that function as electrocatalysts for the reduction of CO<sub>2</sub> to CO. The 2,9-disubstituted phenanthroline ligands contain proton (**phen**), methyl (**2,9-Me<sub>2</sub>phen**), trimethylphenyl (**Mes<sub>2</sub>phen**), or trimethoxyphenyl ((**2,4,6-tmp**)<sub>2</sub>**phen** and (**3,4,5-tmp**)<sub>2</sub>**phen**) groups as steric and electronic modifiers to provide insight into factors impacting catalytic activity. Cyclic voltammograms (CVs) recorded in CO<sub>2</sub>-saturated CH<sub>3</sub>CN or DMF solutions reveal that following two-electron reduction and chloride dissociation to form the active [Re(R<sub>2</sub>phen)(CO)<sub>3</sub>]<sup>-</sup> intermediate, current enhancement indicative of CO<sub>2</sub> reduction to CO was observed and confirmed by controlled potential electrolysis (CPE). Using current enhancement values ( $i_{cat}/i_p$ , where  $i_{cat}$  and  $i_p$  are the current response under CO<sub>2</sub> and N<sub>2</sub>, respectively) to estimate catalytic activity, it was observed that catalysts with more cathodic Re<sup>10</sup> potentials displayed greater activity, in accord with an electronic effect driving catalysis.

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