Electronic Supplementary Material (ESI) for RSC Advances.


# Co(II), a catalyst for selective conversion of phenyl rings to carboxylic acid groups.

Shashi Bhushan Sinha,[a] Jesús Campos,[a] Gary W. Brudvig,[*a] Robert H. Crabtree[*a]

[a]Department of Chemistry, Yale University, 225 Prospect Street, New Haven, Connecticut 06520, United States.

* E-mail: gary.brudvig@yale.edu , robert.crabtree@yale.edu

## - Supporting Information -

CONTENTS

## Experimental

All commercially available chemicals and reagents were used without any further purification unless otherwise indicated. All reusable glassware and stir bars were extensively soaked in concentrated base bath to ensure a minimal risk of reaction contamination by reactive transition metals. NMR spectra were recorded on Agilent DD2-400, -500, -600 or Bruker AMX-500 spectrometers at ambient probe temperatures. All yields were determined by $^{1}H$ NMR spectroscopy using 3-(Trimethylsilyl) propionic-2,2,3,3-$d_4$ acid sodium salt as an internal standard held in small sealed capillaries to avoid oxidation of the standard. These capillaries were placed inside the NMR tubes throughout the whole reaction, and NMR spectra were collected before and after the reaction for each entry. Mass balance was calculated as the sum of the amounts of products (excluding small species derived from the oxidation of the phenyl rings, such as formate or acetate) and recovered starting material divided by the initial amount of substrate subjected to oxidation.

As a representative substrate, 2-picolinic acid was isolated using a modification of a previously reported procedure (ref 15a,b) and carrying out the reaction at higher scale (0.42 mmol of 2-phenylpyridine). After completion of the reaction, the pH of the solution was adjusted to 3-4 by using KOH. The water was removed under reduced pressure. The crude was dissolved in hot 2-butanol and sonicated for 15 minutes. The precipitate was filtered off and the procedure was repeated thrice to remove the excess salts. 2-butanol was removed under reduced pressure to afford the 2-picolinic acid in ~20% yield (10.5 mg, 0.08 mmol).

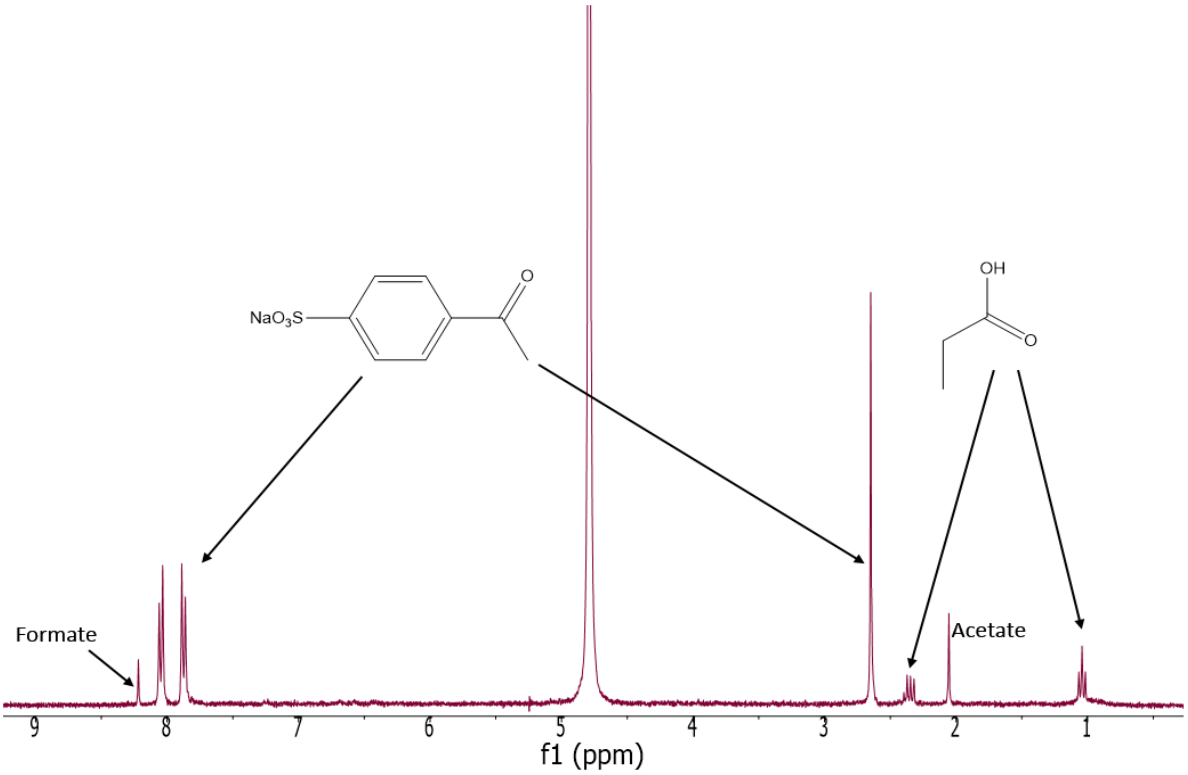


**Figure S1**. $^{1}$H NMR spectra after completion of the reaction for the oxidation of **1**, using the Co(II)-Oxone system. Reaction Conditions: 4 equiv. Oxone, 5 mol% catalyst, room temperature.

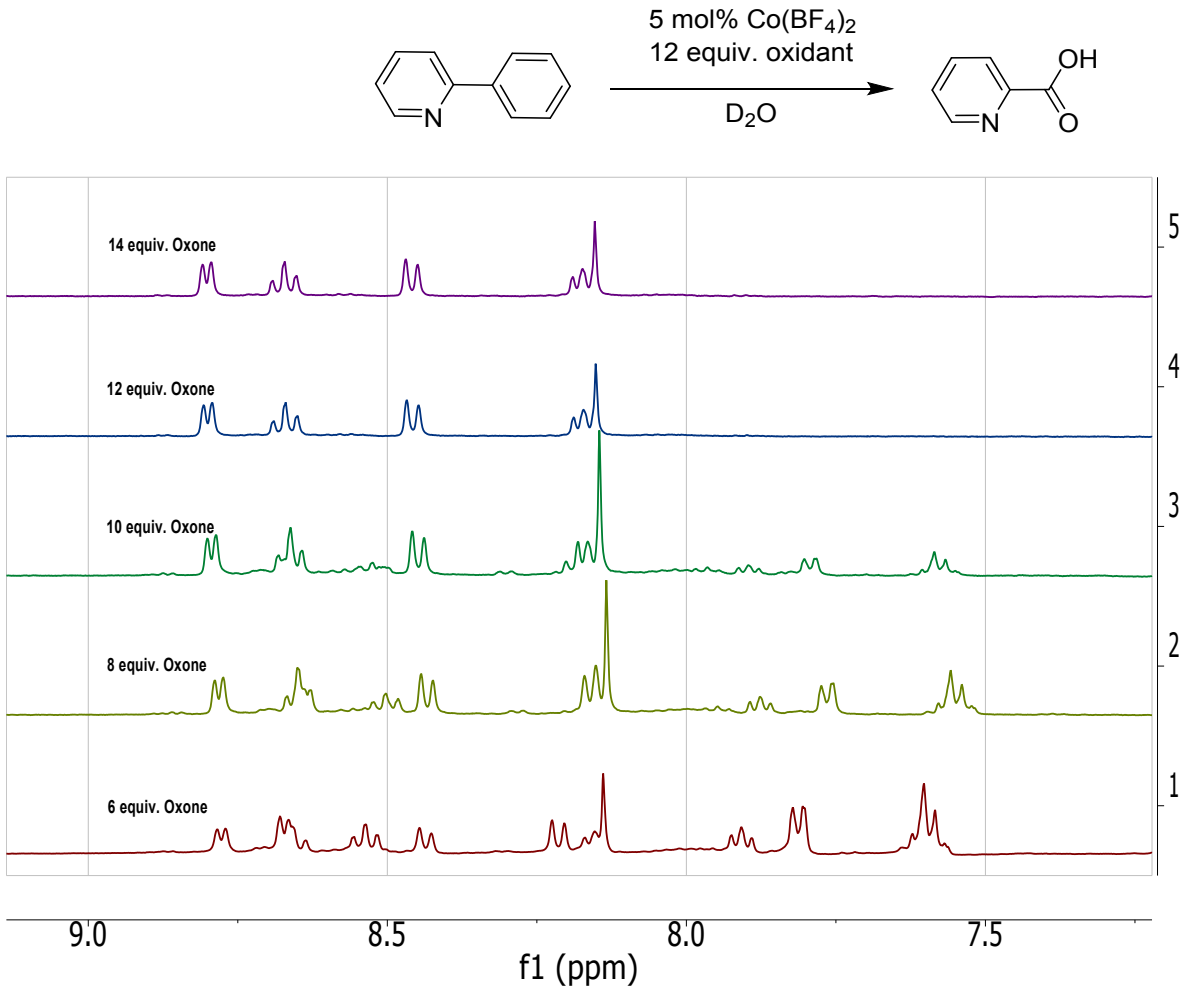


**Figure S2**. $^{1}$H NMR spectra (after 18 h heating) demonstrating that 12 equiv. of Oxone is required for complete oxidation of 2-phenyl pyridine to 2-picolinic acid. Reaction conditions: 2-phenylpyridine heated with 5 mol% catalyst and different equivalents of Oxone.

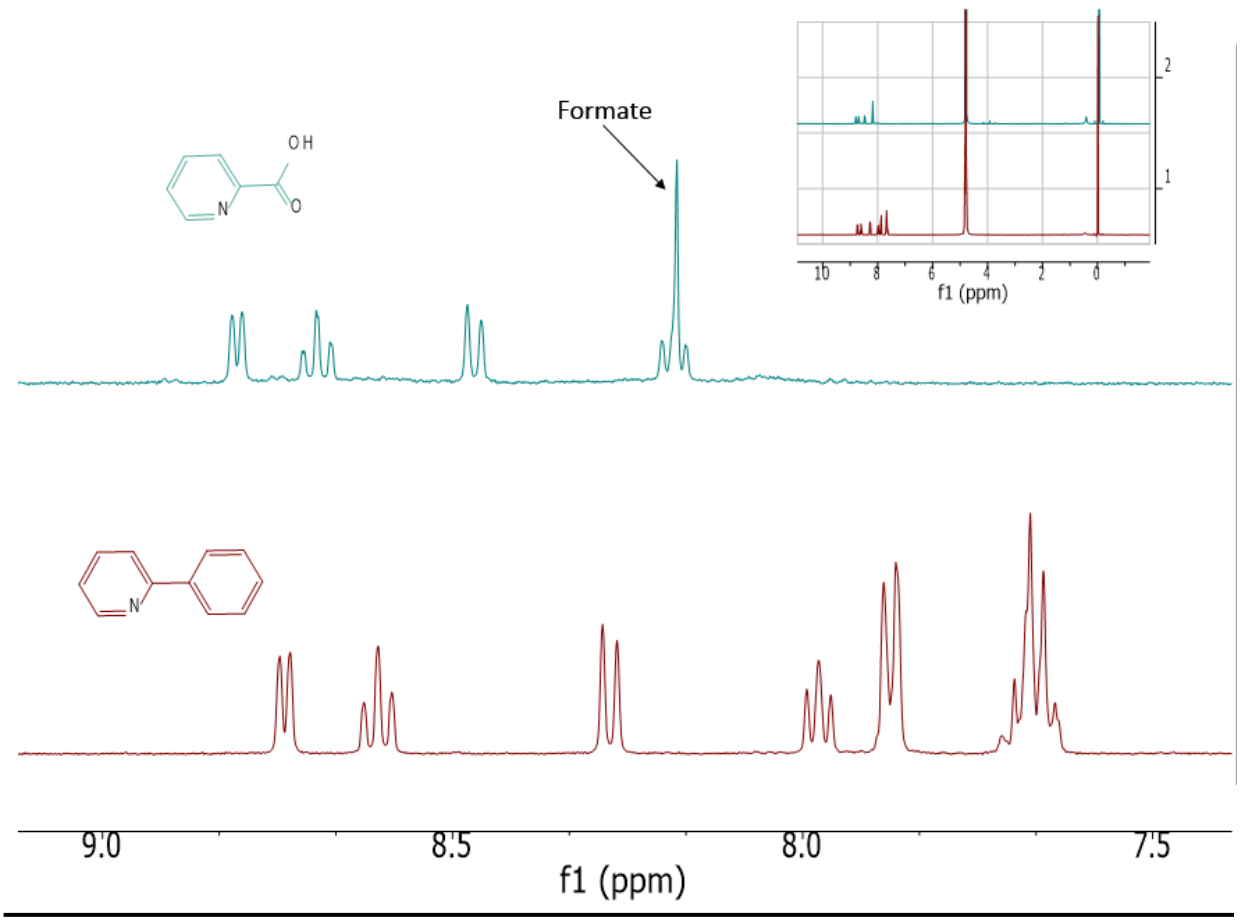


**Figure S3**. $^{1}$H NMR spectra for complete conversion of 2-phenylpyridine into picolinic acid using 5 mol% catalyst and 12 equiv. Oxone.

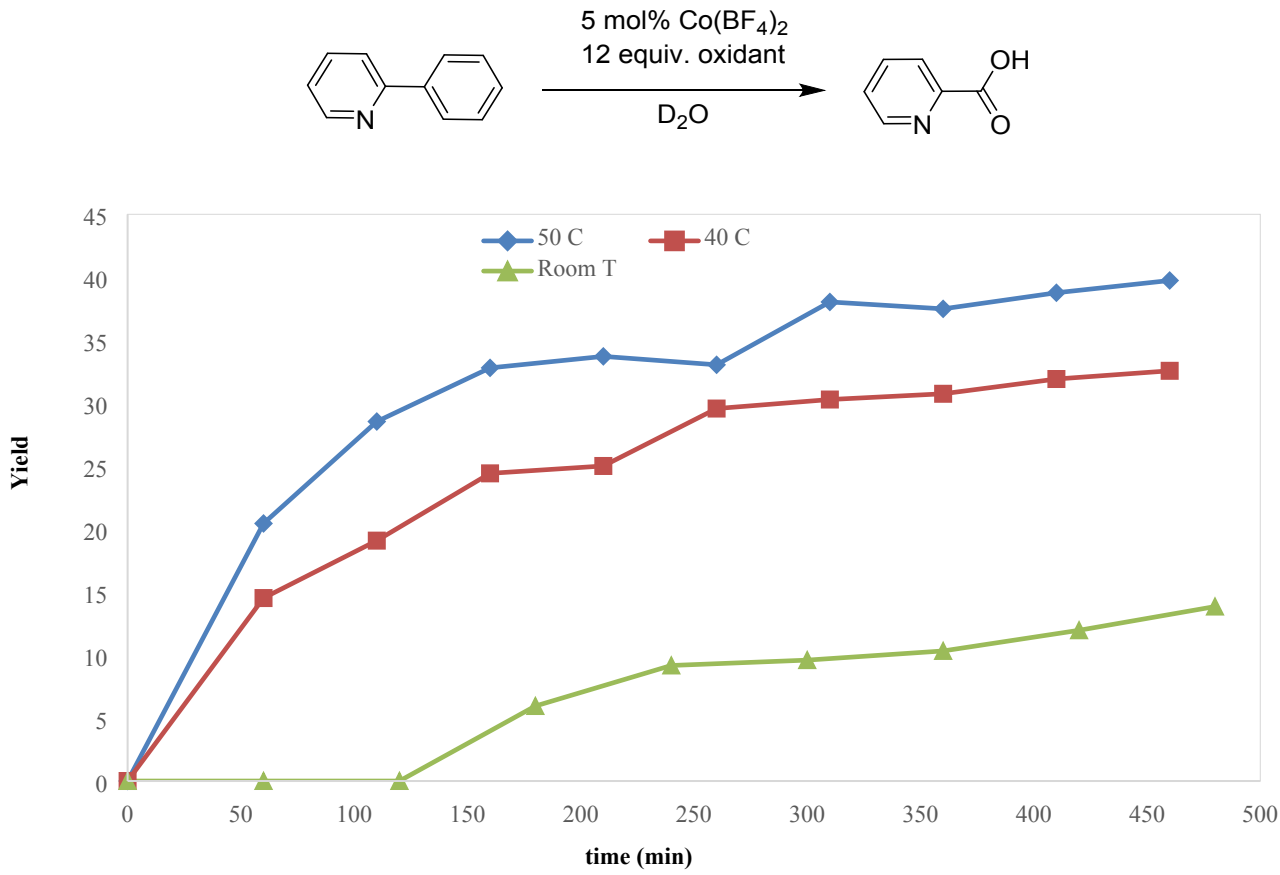


**Figure S4.** Rate of the reaction for degradation of –Ph ring increases considerably on going from 25°C to 50°C.