

# Inorganic Chemistry

including bioinorganic chemistry

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Synthesis of Co(II) porphine. The porphine ligand was synthesized from pyrrole (Aldrich, freshly distilled) and formaldehyde (Fisher, 37 wt.% solution in water) in propionic acid (Aldrich, 99%)/pyridine (EM Science, Omnisolv) 100/1 at 90 °C in an open beaker.<sup>1</sup> The combined crude material from three 2 L batches was chromatographed twice on silica gel, reacted with 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (Aldrich, 98%) to oxidize any chlorin, chromatographed again and finally recrystallized to give 230 mg (0.26%) of pure porphine. Cobalt(II) was inserted into porphine by reacting a 20% molar excess of cobalt acetate tetrahydrate (EM Science, GR) with 20 mg of porphine in 5 ml of refluxing *N, N'*-dimethylformamide (EM Science, GR) under argon for 30 min.<sup>2</sup>

Adsorption of Co(II) porphine on graphite electrodes. The cobalt porphine was adsorbed on edge plane pyrolytic graphite electrodes that had been polished with wet 600 grit SiC paper to obtain a roughened surface. The freshly polished electrodes were dipped in a saturated solution of cobalt porphine in CHCl<sub>3</sub> (~ 0.5 mM) for a few seconds followed by immersion in pure CHCl<sub>3</sub> to remove all but the irreversibly adsorbed porphyrin. The CHCl<sub>3</sub> (EM Science, Omnisolv) was pre-treated by passage through a 2 x 25 cm chromatographic column loaded with activated basic alumina (Aldrich).

#### References

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