Inorganic Chemistry
including bioinorganic chemistry

Inorg. Chem., 1997, 36(20), 4294-4295, DOI:10.1021/ic970516s

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.[1] 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.[2]

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 $CHCl_3$ (~ 0.5 mM) for a few seconds followed by immersion in pure $CHCl_3$ to remove all but the irreversibly adsorbed porphyrin. The $CHCl_3$ (EM Science, Omnisolv) was pre-treated by passage through a 2 x 25 cm chromatographic column loaded with activated basic alumina (Aldrich).

References

1. Neya, S.; Yodo, H.; Funasaki, N. *J. Heterocycl. Chem.*, **1993**, *30*, 549.
2. Adler, A. D.; Longo, F. R.; Kampas., F.; Kim, J. *J. Inorg. Nucl. Chem.*, **1970**, *32*, 2443.