

then allowed to slowly cool to room temperature. Upon cooling, the flask was placed in an ice bath to facilitate recrystallization of the tetraphenylporphyrin product. The recrystallized product was isolated through vacuum filtration with a Buchner funnel. The product was thoroughly washed first with methanol and then with hot deionized water. After washing, the product was placed in a desiccator for one week to facilitate further drying prior to massing the product.

### Synthesis of nickel-tetraphenylporphyrin

A 100mL round-bottomed flask containing 50mL of DMF, .500g (.813mmol) of the TPP synthesized the previous week (obtained from the desiccator), and a stir bar were placed on a heater/stir plate combined setup without heat. To this setup was added .263g(105.69mmol) of Ni(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)<sub>2</sub> and the solution was heated to a gentle reflux for 30 minutes. After 30 minutes, 50mL of room temperature deionized water was added to the round-bottomed flask, which was immediately placed in an ice-bath to chill for 15 minutes. The chilled product was vacuum-filtered through a Buchner filter and washed several times with deionized water. The product was dried under vacuum pull for 10 minutes and placed in a desiccator for 1 week prior to massing and characterizing the product.

### Summary of Physical Properties and Spectral Data:

Tetraphenylporphyrin (Week 1 product)

percent yield: 15.2%

IR (HATR)  $\nu$  in cm<sup>-1</sup>: 3309.4 (s, N-H str); 3056.1 (s); 2357.3 (m); 1442.0 (m); 968.0 (s); 718.7 (s)

Magnetic Susceptibility:  $\chi_g = -5.823 \times 10^{-8}$

Vis (H<sub>2</sub>O):  $\lambda_{max} = 646$  nm;  $\epsilon = 3149.8$  M<sup>-1</sup>cm<sup>-1</sup>;  $\lambda_{max} = 590$ ;  $\epsilon = 4102.9$  M<sup>-1</sup>cm<sup>-1</sup>  $\lambda_{max} = 549$ ;

$\epsilon = 5855.1$  M<sup>-1</sup>cm<sup>-1</sup>;  $\lambda_{max} = 514$ ;  $\epsilon = 14186.2$  M<sup>-1</sup>cm<sup>-1</sup>

<sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>):  $\delta$  in ppm, -2.695 (2H, pyrrole N-H); 7.28 (CDCl<sub>2</sub>H); 7.806 (12 H, H-phenyl, J=20.4 Hz); 8.270 (8 H, H-phenyl); 8.899 (8 H, pyrrole-H)

Nickel-Tetraphenyl porphyrin (Week 2 product)

percent yield: 88.13%