Laboratory Evaluation for
Microwave Synthesis of Tetraphenylporphyrin


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Submitted by Pete Hanson at Wittenberg University based on evaluations completed by participants at the Green Chemistry in Education Workshop in Eugene, Oregon July 15-20, 2006.

Evaluation

**Microwave Synthesis of Tetraphenylporphyrin**

This experiment is easily performed within 3 hours, and provides students with the opportunity to use microwave energy to conduct an organic reaction. It serves as a green alternative to the traditional porphyrin synthesis, which requires reflux of the starting materials in propionic acid. Because the product mixture contains distinctively colored compounds (green, red, purple, yellow and brown), the experiment can also be used to provide a sample that will visually demonstrate separation by column chromatography.

The educators who performed this reaction at the GCEW in 2006 commented that a tarry residue was produced as a byproduct of the reaction. The result was that attempted separation of the entire product mixture by column chromatography was difficult. Desorption of the product from the solid support with the specified amount of dichloromethane, followed by application of 20% of the obtained solution to the column, gave excellent chromatographic separation as well as material that can be used in the porphyrin metallation experiment appearing in Doxsee and Hutchison (Green Organic Chemistry - Strategies, Tools, and Laboratory Experiments 2004).

There are opportunities for improvement of this experiment. The dichloromethane used to desorb the product mixture from the silica support might be replaced with a greener solvent (ethyl acetate, acetic acid or even ethanol?), or perhaps only a portion of the initial crude product, still on silica, could be directly applied to the chromatography column. It might also be possible to "scale down" the experiment, since the amount of material produced using the specified amounts can produce more material than can be effectively separated on the column used (1 cm diameter). Synthetic efficiency might be improved by investigating the effect of microwave power (we used a 600 W oven) and duration of the irradiation on the outcome of the reaction.