

CHEM 330
Organic Chemistry Laboratory
Dr. Ramberg

Friedel-Crafts Reaction

New Techniques: Doing a simple chemical reaction, NMR spectroscopy

Reading (in Pavia): chapter 26, esp. pp. 411-414 on sample preparation.

Prelab: Determine the melting point for this product, and calculate the theoretical yield for this reaction. Predict the approximate chemical shifts you would expect in the NMR spectrum of your product. Would there be any splitting?

For the lab report: Report your percent yield and your observed melting point. Assign the relevant peaks to the NMR spectrum of your product. Can you identify any impurities from your NMR spectrum? How could you improve your yield?

Procedure: With gently warming, dissolve approximately 320 mg (record exact mass) of 1,4-dimethoxybenzene in 1.2 mL of acetic acid. Add 1.1 equivalents of t-butyl alcohol to this solution. Cool in an ice bath, and add 1.2 mL concentrated H_2SO_4 , continuing to stir while adding. After adding each drop, stir thoroughly with a glass stirring rod. You should have a precipitate at this point. Stir the solution thoroughly after completing the addition of acid, and allow it to warm to room temperature. Allow it to sit for at least 20 minutes to complete the reaction. Cool the mixture once more in an ice bath, and add 7.5 mL water dropwise, maintaining a 0° temperature. Vacuum filter the precipitate and wash the crystals with ice-cold water. Recrystallize your product from methanol and obtain a melting point. Add a small amount (a small heap on the end of your spatula) to an NMR tube and dissolve it in CDCl_3 . Label the tube, and turn it in with the remainder of your product in a clean, labelled vial for grading.