

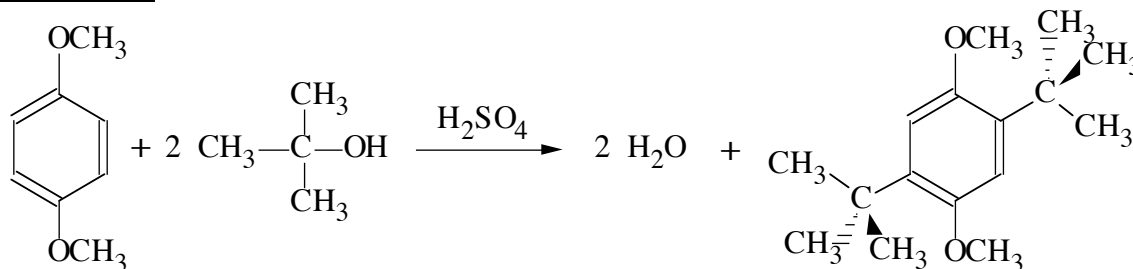
Friedel-Crafts Alkylation of 1,4-Dimethoxybenzene

Source: AEM Handout adapted from K. L. Williamson *Macroscale and Microscale Organic Experiments*. 2nd ed. Lexington, MA: D. C. Heath, 1994. p 440-441.

In any synthetic procedure, a chemist must use a series of techniques that can be classified as: (1) carrying out the reaction, (2) isolating the product (perhaps using extraction, crystallization, distillation, etc.), (3) purifying the product (again, perhaps using recrystallization, distillation, etc.), and (4) characterizing the product and estimating its purity (perhaps using melting point, spectroscopy, various types of chromatography, etc.). Steps (2) and (3), the isolation and purification of the reaction product, are often called the "work-up" of the reaction mixture. The types of techniques that are used for each of these steps depend on the energy requirements of the reaction and on the physical and chemical properties of both the starting materials and the products.

In this Friedel Crafts Alkylation reaction, the reaction is carried out in acetic acid solvent, and while the starting materials are quite soluble in this acetic acid, the product is not. As the product is formed from the starting materials, it crystallizes from the reaction mixture. After these crystals are isolated from the reaction mixture, they can be purified by recrystallization, and characterized by mp. Your instructor might also ask you to take NMR and/or IR spectra.

overall reaction:



1,4-dimethoxybenzene
MW: 138.17 g/mol
mp: 56-58°C

t-butyl alcohol
MW: 74.12 g/mol
mp: 25.5°C
d: 0.79 g/mL

1,4-di-*t*-butyl-2,5-
dimethoxybenzene
MW: 250.37 g/mol
mp: 104-105°C
(No other information required
about this substance)

Experimental:

Reaction Procedure. In a 25 mL Erlenmeyer flask, dissolve approximately 0.480 g (know the exact mass that you use; weigh by difference) of 1,4-dimethoxybenzene in 1.8 mL of acetic acid (solvent) with gentle warming. Add to it 0.9 mL *t*-butyl alcohol. (Know the exact mass that you use; weigh by difference using a syringe.) Cool the mixture in an ice bath, and then add to it dropwise from a Pasteur pipette 1.8 mL concentrated H₂SO₄ (catalyst). Continue stirring throughout this addition process, and after each drop, stir the reaction mixture thoroughly with your glass stirring rod. At the end of this addition, considerable solid reaction product should have separated. Stir the mixture thoroughly before removing it from the ice bath, and allowing it to warm up to room temperature (RT). Once warm, it should remain at RT for at least 20 minutes to complete the reaction.

Reaction Workup. Cool the mixture again in the ice bath to induce complete crystallization of the product. Very carefully add a drop of water and stir with a glass rod, and continue to add the water dropwise (with the solution still in the ice bath) until 11.3 mL have been added. Filter the crystals from the reaction mixture using vacuum filtration and "wash" the crystals thoroughly with ice cold water.

Product Purification and Characterization. Recrystallize the product from methanol. (Review the steps for recrystallization from the previous experiments.) Determine the purity of your product by mp; calculate the theoretical & percent yields. Obtain and fully analyze NMR and/or IR spectra of your product, as directed by your instructor; spectra of the starting materials will be provided. The product will be turned in to be graded.

Clean-Up: check each filtered solutions' pH, neutralize to pH 6-8 (as needed), and flush down the drain with lots of water.