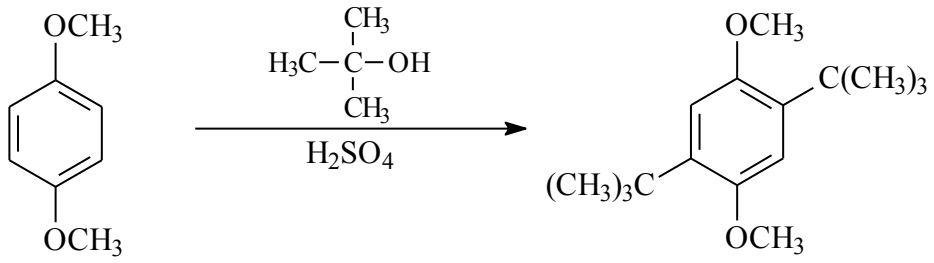
Chemistry 30121 Laboratory

Preparation of 1,4-Di-*t*-butyl-2,5-dimethoxybenzene



Reaction: Obtain a 125-mL Erlenmeyer flask containing 1.0 g of 1,4-dimethoxybenzene from your teaching assistant. Add 2 mL of *t*-butyl alcohol and 5 mL of acetic acid to the flask and swirl the mixture until all of the solid dissolves. Place the flask in an ice/water bath contained in a 400-mL beaker. Swirl the solution periodically during five minutes to facilitate the cooling process. Measure approximately 5 mL of conc. sulfuric acid into a 50-mL beaker by using the graduations on the side of the beaker; *i.e.,* the level of the acid should be half way between the bottom of the beaker and the 10-mL mark. Add the acid to the Erlenmeyer flask **1 mL at a time using an eye dropper** in two-minute intervals (total addition time = 8 minutes). Each addition should be all at once, not dropwise. Swirl the flask rapidly with periodic cooling in the ice/water bath to maintain a reaction temperature below room temperature. After the first addition, you should notice a pale pink color in the reaction mixture that will darken slightly to a purplish tan during the course of the reaction. The reaction mixture will also thicken as the product begins to precipitate from solution. <u>The pink color will not develop if the reaction mixture is too cold.</u> After all of the sulfuric acid is added, swirl the flask at room temperature for about 5 minutes. Then return the flask to the ice/water bath; add 50 mL of ice cold water, and swirl the flask to dilute the sulfuric acid. After about 5 minutes, add another 50 mL of ice cold water and remove the flask from the ice/water bath. At this stage, there should be a substantial amount of white solid suspended in the aqueous solution.

The crude product is isolated by vacuum filtration using a Buchner funnel. Apply only a very gentle vacuum at first to avoid breaking the filter paper which might be weakened by the strongly acidic solution. Use several portions of distilled water to aid in transferring the product from the Erlenmeyer flask into the Buchner funnel. This will also serve to wash the crystals. Finally, apply a full vacuum and press the solid with a spatula to remove as much water as possible.

Recrystallization: Transfer the crude product into a 50-mL Erlenmeyer flask and add 8-10 mL of methanol. For most people, this will be an insufficient amount of solvent to dissolve the solid at the boiling point of methanol. If so, add extra methanol 1 mL at a time until all of the solid dissolves.* Move the flask to the metal rim of the sand bath and allow it to cool very slowly to room temperature. The purified product is then collected by vacuum filtration. Allow air to pull through the crystalline mass for five minutes to dry the product. Transfer the material to a watch glass and spread the solid to facilitate additional drying. Weigh the purified product, obtain a melting point, and turn in the material on a piece of waxed weighing paper. Do not forget to write your name and the weight of the product on the paper.

*The sample may dissolve very slowly if the crude product contains large chunks of material. If so, use a stirring rod to break up the chunks; and let the solution boil gently for at least 5 minutes before adding additional methanol in order to avoid excess solvent.