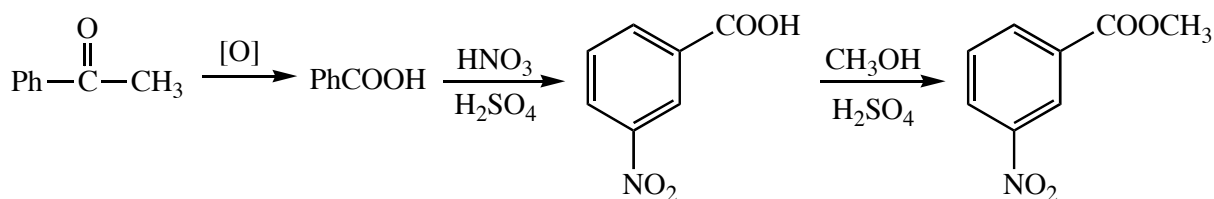# Multi-Step Synthesis of Methyl *m*-Nitrobenzoate

AEM – last update July 2013

This **Fisher Esterification** is the final step of the multi-step synthesis of methyl *m*-nitrobenzoate. Abbreviated versions of the three reaction steps are given here, with more complete versions of the overall reactions given with each lab prompt separately.
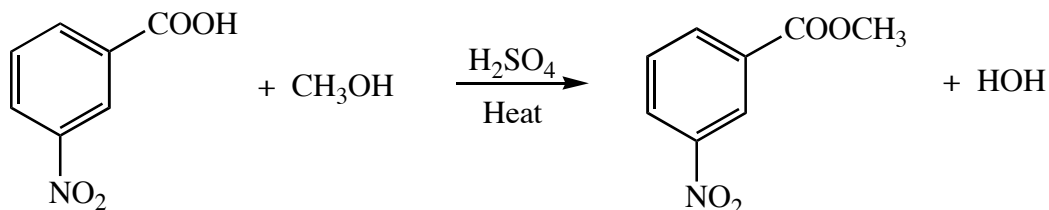
All Three Steps of the Multi-Step Reaction Sequence:

Your m-nitrobenzoic acid should be completely dry by now. In addition, you should have measured its mass and characterized it by mp, IR, and/or NMR before you go on to the esterification experiment.

Step 3: Fischer Esterification of m-Nitrobenzoic Acid to Produce Methyl m-Nitrobenzoate

Overall Reaction:

As in the previous step, the amounts of reagents used for this procedure will depend on the mass of *m*-nitrobenzoic acid that you use in the reaction. Use between 1 and 2 g of it! It is also critical that your *m*-nitrobenzoic acid is completely dry, since this reaction is an equilibrium process and water in a wet sample will drive the reaction in the reverse direction, reducing your yield of product.

For each g of *m*-nitrobenzoic acid you will need 8 mL of *anhydrous* methanol; for each 20 mL of methanol, you will need 1 mL of concentrated H_2SO_4. Note that this concentrated acid

should be measured using your graduated cylinder for safe handling, even if that requires some estimation! Consider the total volume of this mixture, and choose a roundbottom flask that holds about twice that volume; in other words, choose a flask so that it is about half full. Put the three materials in the proportions described above into the roundbottom flask with a couple of boiling chips, and attach a reflux condenser to form a reflux apparatus. Heat to reflux for 1 hour.

When the roundbottom flask has cooled enough to hold, pour the reaction mixture into an ice/water slurry (use a total volume of slurry of about 5 times the volume of methanol used) and stir. Use suction filtration to isolate the product and wash with water; the ice should melt during this process, but you can rinse it off and remove it if needed. The crude product should be recrystallized from methanol. Once it is completely dry, determine its mass, and calculate its theoretical and percent yield. Also, determine its purity by mp, IR, and/or NMR as preferred by your professor.

Clean up. Check the pH of all the solutions, neutralize as needed, and flush down the drain with lots of water.

Multi-Step Synthesis Yield Calculation.

When you carry out a series of reaction steps, you usually want to know the efficiency of the whole process. To do so, you can use the percent yields for each step to compute the overall percent yield. This is easiest to explain with an example. Suppose you carried out four reactions in sequence with the percent yields given below.

Step 1: 87.5%;   Step 2: 91.2%;   Step 3: 79.3%;   and Step 4: 81.9%

The overall percent yield is computed as shown, here.

Overall Percent Yield: 0.875 x 0.912 x 0.793 x 0.819 x 100 = 51.8% overall.

Be sure to compute the overall percent yield for the three steps of you synthesis of methyl *m*-nitrobenzoate.