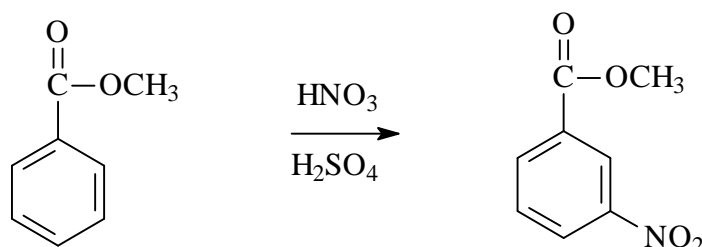


## Organic Chemistry Laboratory

### #11 Nitration of Methyl Benzoate

#### Pre lab reading:

You should review recrystallization and filtering techniques discussed in Chapter 13 of the Zubrick text.



#### Procedure:

**CAUTION:** Concentrated sulfuric acid and nitric acid can cause burns on contact with skin. Take particular care in measuring these reagents. If a spill occurs, clean it up immediately and consult your instructor. If any comes in contact with your skin immediately wash the skin thoroughly with cold water then soap and water. Aromatic nitro compounds are generally toxic, and some may cause contact dermatitis; avoid contact with any of these compounds.

Place 14.5 mL of concentrated sulfuric acid in a 125 mL Erlenmeyer flask, cool it to 0 °C in an ice bath, and then add 6.3 mL of methyl benzoate with swirling. Prepare a mixture of 5 mL of concentrated sulfuric acid and 5 mL of concentrated nitric acid, and cool it in an ice bath. Add the cold acid mixture drop by drop over a period of 5 minutes to the methyl benzoate solution, which is constantly swirled in the ice bath. Allow the resulting mixture to stand at room temperature for an additional 10 minutes with occasional swirling and then pour it with stirring over about 50 grams of crushed ice. Collect the resulting solid by suction filtration and wash it thoroughly with water to remove the acids. Finally, wash the product with two 5 mL portions of ice-cold methanol. The product can be recrystallized from a small amount of methanol.

#### Prelab questions:

Calculate the theoretical yield of methyl nitrobenzoate for the above procedure.

#### Report:

Fill in a data report sheet on the last page of this handout.

### Questions:

1. Which compound in each of the following pairs is more reactive towards aromatic nitration? Explain your answers.
  - a. phenol or nitrobenzene
  - b. methyl benzoate or phenol
2. If all the methyl benzoate is not nitrated in this procedure, where is the unreacted starting material removed in the purification process?
3. There are three isomers of methyl nitrobenzoate; *ortho*, *meta*, and *para*. How can you be certain that you made the *meta* isomer?

## Preparation of Methyl Nitrobenzoate

Name: \_\_\_\_\_

Lab section: \_\_\_\_\_

Date: \_\_\_\_\_

### DATA

Weight of methyl benzoate	_____ g	_____ mol
Theoretical yield of methyl <i>meta</i> -nitrobenzoate	_____ g	_____ mol
Weight of methyl <i>meta</i> -nitrobenzoate	_____ g	_____ mol
Percent yield of methyl <i>meta</i> -nitrobenzoate	_____ %	
Observed melting range	_____ °C	