**MICROSCALE ACETYLATION OF BENZOIN WITH ACETIC ANHYDRIDE**

**Required Pre-lab Readings**: McMurry, sect. 16.3 pp 654-655 & sect 16.5, pp 806-807

Padias, pp 121-125

**Techniques you must be prepared to use**: microscale recrystallization

Esters are most often associated with their characteristic smells. A number of common esters and their smells are shown below. Esters also have other important uses ranging from cell membranes to plastics.



Esters can be synthesized by the acid catalyzed reaction of an alcohol with either a carboxylic acid, an acid chloride, or an anhydride. In this experiment, you will perform the acetylation of benzoin, an alcohol, to benzoin acetate using acetic anhydride as shown below:



As you read in the pre-lab assignment, a strong acid is used to protonate the anhydride to begin the reaction. The alcohol then acts as the nucleophile and attacks the electrophilic carbonyl carbon of the anhydride. These types of reactions are reversible. How do you prevent the reverse reaction from occurring in the lab?

This experiment is another example of a microscale reaction Remember, microscale experiments have advantages and disadvantages. Advantages include being less expensive, less polluting, and using less equipment than larger scale reactions. They are also generally faster, so if anything goes wrong, one has time to repeat the experiment. Some of the disadvantages are that reagents and products must be measured more carefully and one must work more carefully to avoid losses in transferring materials.

**Synthesis**

**Caution:** Take care in handling the chemicals in this experiment.

Prepare a boiling water bath in a 400 mL beaker on a hot plate. Meanwhile, weigh out 0.50 mmol of benzoin (\_\_\_\_ mg) on an analytical balance and place it in a 6 in. test tube. To the same test tube, add, in order, 6 drops of glacial acetic acid, about 1 mL of acetic anhydride, and 1 drop of concentrated sulfuric acid, with stirring.

Heat the mixture in the boiling water bath for 15 minutes. Remove the test tube from the bath and add 10 mL of cold water. Chill in an ice bath, with occasional stirring, until precipitation of the product is complete. If an oil forms, scratch the inside of the test tube with a glass stir rod to induce precipitation. Remove the water layer with a pipet.

To recrystallize the residue, add 2-4 mL of methanol and warm to dissolve the product. Cool slightly, then chill in an ice bath. Again, if an oil results, diligent scratching will produce a solid. Isolate the final product using a microscale Hirsch funnel and wash the product with a small amount of ice-cold methanol. Record the mass and the melting point of the solid.

 Please turn in your product along with your lab report. The vial must have a label on it with the following information: 1) name, 2) lab day, 3) name or structure of compound, 4) weight.

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**DATA SHEET**

|  |  |  |  |
| --- | --- | --- | --- |
| Name: |  | Section: |  |

Overall Reaction: (chemical drawing software)

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Mass of benzoin: |  |  | Theo mmol benzoin acetate: | |  | |
|  |  |  |  | |  | |
| mmol of benzoin: |  |  | Theo mass benzoin acetate: | |  | |
|  |  |  |  | | |  |
| Volume acetic anhydride |  |  | Mass recovered benzoin acetate: | | |  |
|  |  |  |  | | |  |
| mmol acetic anhydride |  |  | mmol recovered benzoin acetate: | | |  |
|  |  |  |  | |  | |
|  |  |  | % yield benzoin acetate: |  | | |

Observed melting point of recovered benzoin acetate:

Literature melting point:

Literature source:

Show complete calculations: (notebook)

**Post Lab Questions:**

1. Why is a strong acid, such as sulfuric acid, used in a Fischer esterification reaction when there is already a carboxylic acid present?

2. Write out the complete mechanism for this reaction: (Hint: remember that under strongly acidic conditions, you should not generate even moderately weak bases in your mechanism.)

3. Use the a,b,c… system to label the nonequivalent protons in the structure below. Then complete the chart with the information you would expect from the 1H NMR spectrum of the compound shown.



|  |  |  |  |  |
| --- | --- | --- | --- | --- |
|  (ppm) | Multiplicity | J, Hz | Number of H’s | Identification |
|  |  |  |  |  |
|  |  |  |  |  |
|  |  |  |  |  |
|  |  |  |  |  |
|  |  |  |  |  |
|  |  |  |  |  |
|  |  |  |  |  |

4. Complete the following esterification reactions:

|  |  |  |  |
| --- | --- | --- | --- |
| a. |  |  |  |
|  |  |  |  |
| b. |  |  |  |
|  |  |  |  |
| c. |  |  |  |
|  |  |  |  |
| d. |  |  |  |