

Organic Chemistry Laboratory

#2 - Isolation of Acetylsalicylic Acid from Aspirin Tablets

Aspirin, like most tablets, contains the active ingredient (acetylsalicylic acid) plus a binder to give the tablet its shape. The active ingredient in aspirin will be recovered by recrystallization from ethanol. The aspirin tablets you will be using contain 325 mg of acetylsalicylic acid per tablet. (However, check the bottle to be certain, as occasionally substitutions have to be made.)

Pre-lab Reading:

Read Chapters 11 & 13 in the Zubrick manual.

Pre-lab assignment:

Calculate the maximum amount (in grams) of acetylsalicylic acid that could be obtained from 10 aspirin tablets. If 2.4 grams were actually obtained, what is the percent recovery? For the purpose of this exercise you may assume that each tablet contains 325 mg of acetylsalicylic acid. You should record this information in your notebook.

Procedure:

Obtain 10 aspirin tablets and place in a 125 mL Erlenmeyer flask. Add to the flask 10 mL of 95% ethanol and a boiling stone then heat the mixture to boiling on a hot plate. Swirl the hot mixture until the tablets disintegrate. Only the acetylsalicylic acid will be soluble in the hot ethanol so the binder will remain visible as a white powder in the solution. Gravity filter the hot solution through fluted filter paper into a clean Erlenmeyer flask. This should remove the insoluble material. *Be careful at this stage since you can overflow the filter paper allowing impurities into the filtrate!* After the gravity filtration is complete add 50 mL of cold water to the filtrate and set aside for at least 10 minutes. Place the flask in an ice-water bath for an additional 5 minutes. Collect the acetylsalicylic acid by vacuum filtration using a Buchner funnel and clean filter paper. Wash the crystals with some cold water and air-dry the solid by continuing the vacuum and drawing air through the crystals. Transfer your crystals to a pre-weighed container and determine the mass of your product. You must have at least 1.0 g of solid at this point! Consult your instructor if you do not have enough product. Set your compound aside in the weighing boat until next lab period. This will allow your compound to completely dry. During the next period you will obtain a final weight and melting point for your compound. Finally transfer your compound to a labeled vial and submit to your instructor. Determine the percent yield of compound and compare your melting range with the literature value for acetylsalicylic acid.

Data and Observations:

Record the physical constants for acetylsalicylic acid in your notebook. You may use the CRC Handbook, Aldrich Catalog or any other reference book available (see Chapter 3 to review these handbooks.). You should view MSDS for acetylsalicylic acid and be somewhat familiar with the compound. The MSDS is available on the web site of several manufacturers and obtainable through the course web site under *Chemistry Links*.

You should include a table of data that resembles the one used on the report sheet.

Report:

Fill in the data on the report sheet contained in this handout.

Questions:

1. Do you believe your recrystallized acetylsalicylic acid to be very pure? Explain.
2. The solubility of benzoic acid in water is 68 g per 1 L at 95 °C and 1.7 g per 1 L at 0 °C. What is the minimum amount of water needed to recrystallize a 9.68 g sample of benzoic acid? How many grams of pure benzoic acid will be collected after crystallization is complete? How many grams of benzoic acid will remain in solution after the crystals are collected? (Show your work!)
3. Why was 50 mL of cold water added to the ethanol solution during the recrystallization procedure?

Report: Recrystallization

Name: _____

Lab section: _____

Date: _____

Number of tablets actually used _____

Total mass of acetylsalicylic acid (ASA) _____ g

Mass of recrystallized ASA (wet) _____ g

Final mass of ASA (dry) _____ g

Melting range of recrystallized ASA _____ °C

Melting range of recrystallized ASA (corrected) _____ °C

Literature melting range of ASA _____ °C

Literature source _____

Percent recovery of ASA _____ %

Prelab: Recrystallization

Name: _____

Lab section: _____

Date: _____

Calculate the maximum amount (in grams) of acetylsalicylic acid that could be obtained from 10 aspirin tablets. If 2.4 grams were actually obtained, what is the percent recovery? For the purpose of this exercise you may assume that each tablet contains 325 mg of acetylsalicylic acid. You must show your calculations.