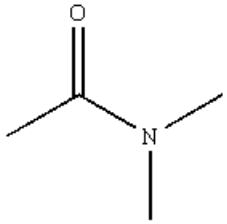
Experiment 8 - Amide Preparation

OBJECTIVE

In this experiment, the amide, 2-(N-acetylamino)benzoic acid (N-Acetylanthranilic acid) will be prepared by the reaction of 2-aminobenzoic acid (anthranilic acid) with acetic anhydride.

INTRODUCTION

Amides are another one of the many functional groups encountered in the study of Organic Chemistry. The amide is identified by having a carbonyl unit (C=O) bound to a Nitrogen unit (NR_2, R can be H).



AMIDE GROUP

Figure 1

An amide can be produced in a variety of ways: 1) From the reaction of an acyl halide with a 1° or 2° amine, 2) the reaction of a carboxylic acid with an amine (usually requires heating), and 3) the reaction of an acid anhydride (commonly acetic anhydride) with an amine.

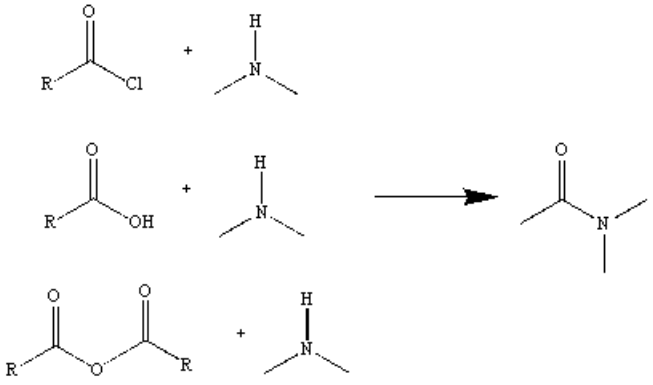


Figure 2

The amide linkage is seen in Biochemical examples where amino acids condense together to form peptides and proteins. The peptide bond is an example of an amide linkage.

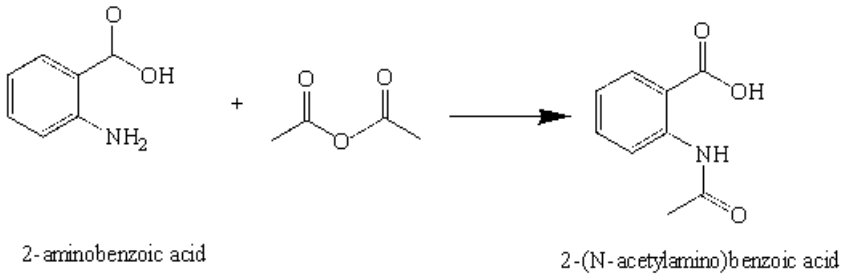


Figure 3

PRE-LAB

Complete the pre-lab assignment in WebAssign.

PROCEDURE

Make sure that the reaction is performed under the sink hoods (i.e., not out in the middle of the bench top). Acetic anhydride is a lachrymator (what does that mean?). In a 25 mL Erlenmeyer flask, place 1 g of anthranilic acid and 3-4 mL of acetic anhydride. Warm the mixture to boiling (gently) on the hot plate. Any solid should dissolve. Heat for a period of 15 minutes. Allow to cool, add 2 mL of water, and heat again to boiling. Slowly let the reaction mixture cool to room temperature (i.e., no ice bath, or cold tap water). The crystalline product should form as the mixture cools. Isolate the product by vacuum filtration and wash with a small amount of cold water. Determine the yield, percentage yield, and melting point of the product.

The filtrate may be neutralized with aqueous $NaHCO_3$, diluted with water, and flushed down the drain.

IN-LAB QUESTIONS

Download and print the following worksheet. You will use this worksheet to record your answers to the In-Lab questions.

Questions

Record the following data.

Question 1: Amount of anthranilic acid used _____ g, _____ mol

Question 2: Theoretical Yield of product _____

Question 3: Actual Yield of product _____

Question 4: Percentage Yield _____

Question 5: Melting Point _____ (observed), _____ (actual)

Question 6: Record your calculations.

