

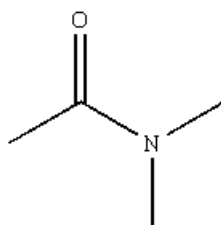
Experiment 8 - Amide Preparation

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 (NR₂, 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.

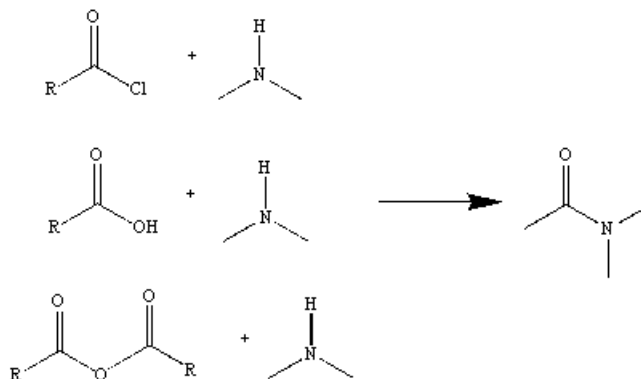


Figure 2

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.

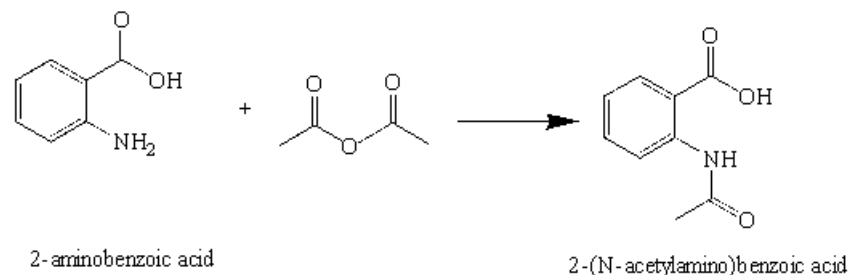


Figure 3

PRE-LAB

Complete the pre-lab assignment in WebAssign.

PROCEDURE

Note: Make sure that the reaction is performed under the bench hoods (i.e., not out in the middle of the bench top). Acetic anhydride is a lachrymator (look up what this means).

- 1 Place 1 g of anthranilic acid and 3-4 mL of acetic anhydride in a 25 mL Erlenmeyer flask.
- 2 Warm the mixture to boiling (gently) on a hot plate set to 260°C. All solid should dissolve.
- 3 Heat for a period of 15 minutes.
- 4 Allow to cool to room temperature.
- 5 Add 2 mL of water to the mixture.
- 6 Heat the mixture to boiling again.
- 7 Slowly let the reaction mixture cool to room temperature (i.e., **no** ice bath or cold tap water) so that the solid formed will have larger crystals.
- 8 Isolate the product by vacuum filtration (here is a video that shows you how to do a vacuum filtration¹).
- 9 Wash with a small amount of cold water while still on the Buchner funnel.
- 10 Determine the yield, percentage yield, and melting point of the product. Here is a video that shows you how to take a melting point².

¹<https://www.youtube.com/watch?v=zHfnwOHCDWM>

²<https://www.youtube.com/watch?v=oKPqXAT0bG8>

Waste Disposal

- 1 The filtrate should be placed in a beaker designated by your TA to be neutralized.
- 2 Any solid product should be added to the "SOLID WASTE" container.
- 3 Melting point capillaries and test tubes should be thrown in broken glass.

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.