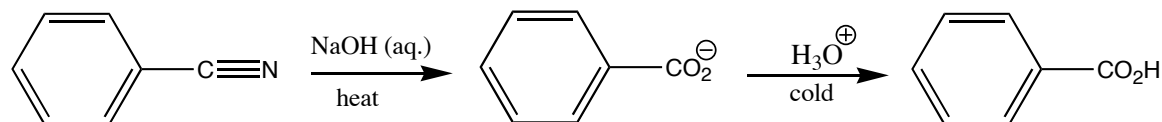


Hydrolysis of Benzonitrile

Required Pre-Lab Readings: Ege Section 15.5 and Chapter 15 Introduction

Techniques you must be prepared to perform: Reflux (pp 56-58) and Recrystallization (pp 100-105).



Introduction

Nitriles are derivatives of carboxylic acids and, as is the case of all derivatives of carboxylic acids, they can be hydrolyzed (literally, "to break with water") to the carboxylic acid. In the case of nitriles either added acid or base and elevated temperatures are required. The hydrolysis of acid derivatives is of particular importance in biology. The hydrolysis of fats (esters) and proteins (amides) are just two of many examples of hydrolysis reactions that occur in animals.

In this lab you will complete a base promoted conversion of benzonitrile to benzoic acid. The mechanism involves initial attack of the nucleophilic hydroxide on the electrophilic carbon. Proton transfer from the solvent (water) results in an intermediate that tautomerizes to the corresponding amide. The amide proceeds on to the carboxylate by a mechanism analogous to the base hydrolysis of esters (see Ege, p. 618).

Procedure

In a 50 mL round-bottom flask add 10 mmol of benzonitrile (____ g; ____ mL), 13 mL of a 10% solution of NaOH and a stir bar. Add a water-cooled reflux condenser and reflux gently for 90 minutes. During reflux you may notice an odor at the top of the condenser (What causes this odor?). After refluxing the oily drops of the starting material should have mostly disappeared.

Remove the mixture from heat, chill the solution in an ice/water bath for several minutes and add cold 6 M HCl (with stirring as always) until the solution is acidic and precipitation of the product is complete (Approximately how much HCl solution will you need?). Do not add the acid one or two drops at a time (think about what you should see). Collect the product by vacuum filtration and wash with cold water. Once the product is isolated, recrystallize the material from water and allow to air dry for a week in the lab drawer. Once dried record the weight and melting point of the benzoic acid recovered.

Hydrolysis of Benzonitrile

Name: _____

Section Number: _____

Overall Reaction:

Volume of benzonitrile: _____

Theo moles benzoic acid: _____

Moles of benzonitrile: _____

Theo mass benzoic acid: _____

Volume of NaOH: _____

Mass recovered benzoic acid: _____

Moles of NaOH: _____

Moles recovered benzoic acid: _____

% yield of benzoic acid: _____

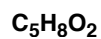
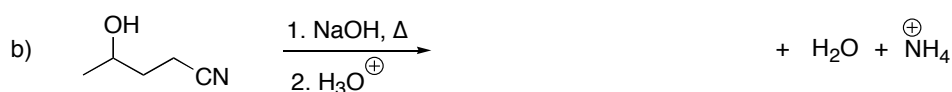
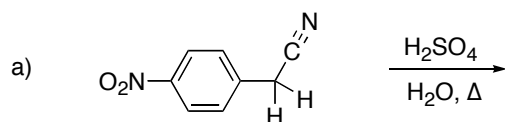
Observed melting point of recovered benzoic acid: _____

Literature melting point: _____

Literature source:

Show complete calculations for yields: (use a separate sheet if necessary)

Show the products of the following reactions:



Write out a complete mechanism for the base-catalyzed hydrolysis of benzonitrile. Be sure to indicate the formation of the intermediate amide by circling it. (Hint: This mechanism contains a tautomerization and runs in a similar fashion to the hydrolysis of an ester)