Hydrolysis of Acetonitrile

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 acetonitrile to acetic 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.

Procedure

In a 50 mL round-bottom flask add 10 mmol of acetonitrile (____ g; ____ mL), 13 mL of a 10% solution of NaOH (d = 1.11 g/mL) 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. (Acetonitrile: 41.05 g/mol; d=0.982 g/mL)

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?). 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 till the next lab period. Once dried record the weight and melting point of the acetic acid recovered.
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Name: ____________________________

Reaction Mechanism:

Show complete calculations for the following where necessary.

Volume of benzonitrile: ________________  Theor. mmol benzoic acid: ________________

mmol of benzonitrile: ________________  Theor. mass benzoic acid: ________________

Volume of NaOH: ________________  Mass recovered benzoic acid: ________________

mmol of NaOH: ________________  mmol recovered benzoic acid: ________________

% yield of benzoic acid: ________________

Observed melting point of recovered acetic acid: ________________

Literature melting point: ________________
**Post Lab Questions:**

1. Write out a complete mechanism for the base-promoted hydrolysis of benzonitrile. Be sure to indicate the formation of the intermediate amide by circling it. Name the final product.
2. Complete the following hydrolysis reactions.

\[
\text{OH} \quad \text{CN} \quad 1. \text{NaOH, heat} \quad 2. \text{H}_3\text{O}^+
\]

\[
\text{H}_3\text{O}^+
\]

\[
\text{Ibuprofen}
\]

\[
\text{O}_2\text{N} \quad \text{H} \quad \text{H} \quad \text{OCH}_3 \quad 1. \text{NaOH, heat} \quad 2. \text{H}_3\text{O}^+
\]

\[
\text{precursor to Fenclorac}
\]

3. Attach a completely analyzed IR – all bonds should be labeled with wavenumbers on the IR with the structure of the product as well.