Experiment 5

Carboxylic Acids and Saponification of Methyl Benzoate

In the first part of this experiment you are going to evaluate the solubility of salicylic acid in water, hot water and in the presence of aqueous NaOH. As you would expect carboxylic acids should react with water to form a water soluble carboxylate anion but solubility is also dependent of the size of the alkyl or aryl group attached to the carboxylic acid functional group. In general the following acid-base reaction occurs:

\[
R-\text{COOH} + \text{H}_2\text{O} \leftrightarrow \text{RCOO}^-\text{(aq)} + \text{H}_3\text{O}^+\text{(aq)}
\]

The structure for salicylic acid is given below:

Salicylic acid

Additionally, carboxylic acids react with bases to form water soluble salts as shown below:

\[
\text{R-COOH} + \text{NaOH (aq)} \rightarrow \text{RCOO}^-\text{Na}^+\text{(aq)} + \text{H}_2\text{O}
\]

Note that salicylic acid contains, in addition to the carboxylic acid functional group, a phenol group hydrogen that can also be titrated by base.

The resultant sodium carboxylate/phenolate salt can further react with acids to reform the free acid (COOH) and phenol (Ph-OH) as shown below:

\[
\text{R-COO}^-\text{Na}^+\text{(aq)} + \text{HCl (aq)} \rightarrow \text{RCOOH} + \text{NaCl (aq)}
\]

\[
\text{Ph-O}^-\text{Na}^+\text{(aq)} + \text{HCl (aq)} \rightarrow \text{Ph-OH} + \text{NaCl (aq)}
\]
The use of bases and acids serves as solubility switches to convert an insoluble form of a compound to a soluble form and vice versa. Observing the solubility or insolubility of reactants and/or products serves as a means to monitor acid base reactions.

The concept of a solubility switch will also serve as the basis for following the base (NaOH) catalyzed hydrolysis (saponification) reaction of an ester (methyl benzoate) to the subsequent water soluble carboxylate salt (sodium benzoate) and alcohol (methanol). The structure for methyl benzoate is shown below:

\[ \text{methyl benzoate} \]

\[ \text{(insoluble)} \]

The resultant mixture containing the sodium benzoate is then reacted with acid to form benzoic acid.

Procedure:

**Part 1:** Set up a boiling water bath with a 250 mL beaker on a hot plate and an ice water bath in a beaker. Place a small amount of salicylic acid (~pea sized amount) into a clean small test tube and add 5 mL of distilled water. Stir well and record your observations in your notebook. Carefully place the test tube into the boiling water bath. Record your observations. Remove the test tube and cool the solution in the ice water bath. Note: you may have to scratch the inside surface of the test tube with a glass rod; your instructor will demonstrate this technique. Record your observations. Add 3 M NaOH to the mixture drop by drop and agitate by finger flicking the test tube after each drop. Be sure to keep track of the number of drops that you add. Record your observations and write the overall equation for this reaction. Finally, add to the solution as many
drops of 3M HCl as you used for the 3 M NaOH addition; then add several more drops of the 3M HCl. Record your observations.

**Part 2:** Place 3 drops of methyl benzoate into a clean test tube and add 2 mL of distilled water. Record your observations in your notebook. Add 12 drops of 10% NaOH and mix the contents of the test tube. Place the test tube into the boiling water bath for 30 minutes (or longer) and every 5 minutes stir the contents vigorously with a clean glass rod, replacing the test tube back into the boiling water bath. Stop the reaction when you judge the solution to be homogeneous and record your observations. Cool the mixture to room temperature by running cold tap water along the outside of the test tube. Add 15 drops of 3 M HCl, mixing by finger flicking the test tube after each addition. Record your observations.
Experiment 5 Report

Name ________________________________

Part 1:

1. a. Does salicylic acid dissolve in cold water?

b. Does salicylic acid dissolve in hot water?

c. Does salicylic acid dissolve in aqueous sodium hydroxide?

d. Write an overall equation for the reaction of salicylic acid with aqueous sodium hydroxide?

e. Write a net ionic equation for the reaction in part (d).

f. What did you observe when hydrochloric acid was added to the test tube containing the product formed in part (e)?

g. Write an overall equation for the chemical reaction that occurs in part (f).

h. Write a net ionic equation for the chemical reaction that occurs in part (f).

2. Which is more soluble in water, salicylic acid or sodium salicylate?
Part 2:

1. Write an overall chemical equation for the saponification of methyl benzoate.

2. Are the initial products of the saponification of methyl benzoate soluble or insoluble in water?

3. What did you observe when hydrochloric acid was added to the reaction mixture following saponification?

4. Write an overall chemical equation for the reaction described in question 3 above.

5. Write a net ionic equation for the reaction in question 4 above.