Lab 5: Reactions of Organic Compounds and Qualitative Analysis

Objectives:
- To better understand several chemical reactions.
- To identify an unknown chemical by testing its chemical and physical properties.

Introduction:
In this experiment, you will investigate solubility behavior and some typical reactions of carboxylic acids, amines, aromatic compounds, alkenes, alcohols, aldehydes, and ketones. You will be testing the reaction and solubility of an unknown compound and comparing it to known compounds in order to determine its identity. Your unknowns will be one of the compounds found on page 5.

The tests that you will investigate include: solubility, ignition, bromine, chromic acid oxidation, acetyl chloride, and Benedicts. You should first perform each of these tests with the test compounds that are suggested for each test and then perform the same tests with your unknown.

Remember to record results and observations for all test compounds as well as your unknown. Record both the visual result and your conclusion. Do not just say positive or negative result – you should describe what you observed. When you have performed all of the tests, identity the unknowns from the list on page 5. Assuming that your test results are correct, you should be able to narrow down your choice to one unknown in most cases. However, in a few cases there may be two compounds that match your test results. Sometimes a test result may not work as expected, either because of student error or because the chemistry just doesn’t work as expected for a particular compound. If you have results that don’t seem to fit the rest of your results, you may want to repeat a test.

A. Solubility
Using the following procedure, determine the solubility of the unknown or test compound in each of the following solvents: water, 5% HCl, and 5% NaOH. Each test should be done using a fresh sample of the compound.

Place about 2 mL of the solvent in a small test tube. Add 1 drop of an unknown liquid from a pipet or a small amount (about one grain of rice in size) of an unknown solid from the end of a spatula directly into the solvent. Watch carefully as the sample enters the liquid. Gently tap the test tube with your finger to ensure mixing and then observe whether any mixing lines appear in the solution. The disappearance of the liquid or solid or the appearance of the mixing lines indicates that solution is taking place. Add several more drops of the liquid or a little more solid to determine the extent of the compound’s solubility. A common mistake in determining the solubility of a compound is testing with a quantity of the unknown too large to dissolve in the chosen solvent. Use small amounts. It may take a couple minutes to dissolve solids. If a compound is found to dissolve in water, the pH of the aqueous solution should be estimated with pH paper. Dip a glass stirring rod into the solution and touch the pH paper with the glass rod – do not dip the pH paper into the solution. Note that distilled water typically has a pH of 5-6.
due to dissolved carbon dioxide. A pH in this range indicates a neutral unknown. If a compound is soluble in water, then it will also be soluble in HCl and NaOH and you do not need to test it in these solvents.

**Test Compounds:** methyl alcohol, cyclohexanone, benzoic acid, ethyl p-aminobenzoate and your unknown

Use the following chart to interpret your solubility results.

B. Ignition

Working in a hood, adjust a Bunsen burner so that the flame is blue. Place a small amount of the compound on a spatula and place it in the flame of the burner. Observe whether a sooty yellow flame results. Compounds giving the sooty yellow flame have a high degree of unsaturation and are likely aromatic.

**Test Compounds:** ethyl benzoate, cyclohexanol and your unknown
C. Bromine in Methylene Chloride

Dissolve 50 mg of a solid unknown or 4 drops of a liquid unknown in 1 mL of methylene chloride. Add a 2% solution of bromine in methylene chloride, dropwise, with shaking. If you find that the red color remains after adding one or two drops of the bromine solution, the test is negative (no carbon, carbon double bond present). If the red color disappears, continue adding the bromine in methylene chloride, but do not add more than 10 drops. The test is positive if more than 5 drops of the bromine solution were added, with discharge of the red color of bromine.

**Test Compounds:** cyclohexene, cyclohexanol, toluene and your unknown

\[
\text{Red} \quad \text{Orange} \quad \text{Colorless}
\]

**Discussion:** A successful test depends on the addition of bromine, a red liquid, to a double bond to give a colorless dibromide.

D. Chromic Acid Oxidation

Dissolve 1 drop of a liquid or 10 mg (approximate) of a solid unknown in 1 mL of reagent-grade acetone. Add several drops of the chromic acid reagent, a drop at a time while shaking the mixture. A positive test is indicated by a green precipitate and a loss of the orange color in the reagent. It may take a minute or two for the precipitate to form. In some cases, you may find that some of the original orange color remains together with a green (or sometimes brown) precipitate. This should be interpreted as a positive test.

**Test Compounds:** 1-butanol, 2-butanol, tert-butyl alcohol (a tertiary alcohol) and your unknown

**Discussion:** This reagent will give a positive test with aldehydes and with primary or secondary alcohols. This is the reaction with an aldehyde. Benedict’s Test will help you distinguish between aldehydes and alcohols.

Edited by Nick Buker 02/10/10
E. Benedict's Test

Add 1 mL of Benedict's solution to a test tube. Also add about 10 drops of a liquid unknown or about 0.2 g of a solid unknown. Note: Solids must be at least partially soluble in water to give a good test. Prepare a water bath at 70 to 90° C. Place the test tube in the hot water bath for about 5 minutes. Formation of a red-orange precipitate indicates a positive test.

Test compounds: cinnamaldehyde, 2-butanone, 1-butanol, 50% glucose and your unknown

\[
\text{RCHO} + \text{Cu}^{2+} \rightarrow \text{RCOOH} + \text{Cu}_2\text{O}
\]

Discussion: Aldehydes will react with Benedict's reagent, but ketones and alcohols do not react. This test is based on the following reaction:
List of Possible Unknowns

- Acetone
- Acetophenone
- Benzyl Alcohol
- Propene
- Butanal
- Butyl amine
- Chloroacetic Acid
- Cyclohexene
- Isopropyl Alcohol
- Heptanal
- 2-Heptanone
- Hexanoic Acid
- 1-Octanol
- p-Toluic acid
- p-Toluidine
CHEM 131 LAB 5 REPORT:                        Names_________________________

Turn in reports as a group                Due Tuesday, Nov 23

Results (10 pts)

Give the name and structure of your unknown.

Make a table describing the observations and summarizes the results of the tests run on your unknown (attach table to the report).

Discuss your results and comment on how each test allowed you to narrow the list of possibilities. Explain your logic for eliminating various choices by performing each test.
Name __________________________

Qualitative analysis lab

Pre Lab Questions- 10 pts: (Answers submitted individually at the beginning of lab)

1. Predict which unknowns will be water soluble.

2. Which unknowns will react in the Chromic acid oxidation test (give a positive test)?

3. Which unknowns will react in the Benidicts test (give a positive test)?

4. Which unknowns will react in the Bromine test (give a positive test)?