#### **Experiment 4**

# **Properties of Alcohols, Aldehydes and Ketones**

#### **Objectives**

In this experiment, you will do a series of chemical reactions designed to characterize alcohols, aldehydes and ketones. You will be able to determine if a reaction has occurred by several means, including color changes and chemical odors. Many of these compounds have distinctive odors, and the new chemicals you make will have different odors, some of which will be familar to you.

#### A. Combustion

As observed in Experiment 2, one of the main uses of organic chemicals, including alcohols, is combustion. In fact, ethanol and methanol are used as the fuel in some race cars, and is often added to gasoline. What are the combustion products of alcohols, besides heat?

#### Combustion of Methanol: $CH_3OH + O_2 \rightarrow$ (what are the products?)

Obtain about 1 mL (no more) of methyl alcohol in an evaporating dish and ignite the alcohol with a match or burning splint (decide if you should do the experiment in the hood or out in the lab). Repeat this experiment with small volumes of ethanol and isopropyl alcohol as well.

Record your observations and any observable difference between the three alcohols. Compare to the combustion of the hydrocarbons used in Experiment 2.

### **B. Oxidation of Alcohols**

Many alcohols react with oxidizing agents to produce new chemical compounds. Primary alcohols produce aldehydes and secondary alcohols produce ketones. Tertiary alcohols, however, are not easily oxidized. In addition, many alcohols, aldehydes and ketones have distinctive odors. We will use these odors to determine if a reaction has occurred.

CAUTION: If you are instructed to smell chemicals, be careful. Waft the vapors to the nose with your hand, as demonstrated by your instructor. Dispense and use chromic acid solution in hood.

You will use 2-mL portions of ethyl alcohol, isopropyl alcohol, and t-butyl alcohol in separate

test tubes. After you dispense the different alcohols into the tubes, and immediately before you add the acid, smell the odor of the alcohol in the tube by wafting the vapors to your nose (your instructor will show how to do this). Then, to the first test tube, add dropwise about 1 mL of your chromic acid solution (*see instructions below on how to make this solution, if not prepared for the entire lab*). Observe any changes in color, whether heat is generated, if gases are produced, etc. Smell the vapors of each tube after you add the acid, and observe if the odor has changed. Write down all observations for each alcohol.

Write equations for the probable product that would be produced, if the alcohol was oxidized to produce an aldehyde, ketone or acid. How does the odor from your reaction tubes compare to the real aldehyde, ketone or acid? Since aldehydes are easily oxidized, you will get a mixture of oxidation products (and odors) from the oxidation of ethyl alcohol.

## C. Formation of Esters

As discussed in class, the chemical combination of an alcohol and acid produces an ester (with the removal of a water molecule during the combination). As indicated above, alcohols, aldehydes, ketones and acids often have distinctive odors. Esters have unique odors as well. You will do reactions and determine if an ester has been produced.

Take three clean test tubes and mix the following reagents in them.

- **Tube 1:** 3 mL of ethyl alcohol, 0.5 mL glacial acetic acid using a pipet or dropper, and 10 drops of concentrated sulfuric acid.
- **Tube 2:** 3 mL of isoamyl alcohol (isopentyl alcohol), 0.5 mL glacial acetic acid using a pipet or dropper, and 10 drops of concentrated sulfuric acid.
- **Tube 3:** Salicylic acid crystals (about 1 cm deep in the tube), 2 mL of methyl alcohol, and 10 drops of concentrated sulfuric acid.

Heat the tubes by placing them in a boiling water bath for about 3 minutes. (Make your water bath by putting about 3-4 cm of water in a 250-mL beaker with a few boiling stones in the bottom.) When the water is boiling, add the tubes to the beaker and continue to heat for 3 minutes. Remove the hot tubes and place into your test tube rack. The anticipated products should be:

- Tube 1: Ethyl acetate (what does it smell like?)
- Tube 2: Isoamyl (isopentyl) acetate (what does it smell like?)
- Tube 3: Methyl salicylate (what does it smell like?)

After heating, pour a small amount of each product onto a piece of filter paper and carefully smell it (like you would smell a cologne at Nordstroms) and describe the odor. Dispose of the filter paper in the solid waste container.

## D. Tollens Test (Silver mirror test) for Aldehydes

This test is based on the ability of an aldehyde (which is easily oxidized) to reduce silver ions in solution, forming either a black deposit of free silver or a silver mirror within the test tube. The aldehyde group is oxidized to an acid during this reaction. Tollens reagent is made by reacting silver nitrate solution with dilute ammonium hydroxide. Rinse all glass equipment with distilled water before use.

*The NH*<sub>4</sub>*OH used in this experiment must be prepared within one hour of use, it cannot be made before the day of the experiment*.

Using the freshly prepared Tollens Reagent, add about 5 mL to three very clean test tubes. To each of these tubes, containing the Tollens Reagent, add one of the following:

- **Tube 1:** 2 drops of 10% formaldehyde
- Tube 2: 2 drops of acetone
- Tube 3: 5 drops of 10% glucose

Allow the tubes to stand undisturbed and note the results. The solution containing the glucose may take 10-15 minutes to react.

## E. Properties of Aldehydes and Ketones

### **Odors of Aldehydes and Ketones:**

Carefully detect the odor of samples of the following chemicals, if available:

- acetone
- benzaldehyde
- camphor
- vanillin
- cinnamaldehyde
- 2,4-pentanedione

# Solubility of Aldehydes and Ketones:

Place 2 mL of water in each of 4 separate test tubes. Add 5 drops of propanal (propionaldehyde), benzaldehyde, acetone, and cyclohexanone. Record your observations.

## **Questions and Problems**

- 1. Write the complete balanced reaction for combustion of the following alcohols:
  - ∘ Methanol: CH<sub>3</sub>OH + O<sub>2</sub> $\rightarrow$
  - ∘ Butanol: C<sub>4</sub>H<sub>9</sub>OH + O<sub>2</sub>  $\rightarrow$
- 2. Write equations, including structures, for the reactant alcohol and any product you would produce when each of these alcohols is treated with the oxidizing agent, chromic acid:
  - Oxidation of ethyl alcohol:
  - Oxidation of isopropyl alcohol
  - Oxidation of *t*-butyl alcohol
- 3. When alcohols react with acids, esters are produced. Many esters have pleasant odors. Show the reaction, and structure of the product when each of the following alcohols react :
  - Ethyl alcohol with glacial acetic acid to produce ethyl acetate (what does it smell like?)
  - Isoamyl alcohol (isopentyl) with glacial acetic acid to produce isoamyl (isopentyl) acetate (what does it smell like?)
  - Salicylic acid with methanol acid to produce methylsalicylic acid (what does it smell like?)
- 4. Carbonyl-containing compounds, especially aldehydes, are easily oxidized. Show the organic compound that would be produced, in an oxidation reaction when each of the following is reacted with the Tollens reagent:
  - Formaldehyde
  - Acetone
  - $\circ$  Glucose
- 5. Show the step-by-step reaction products, and names, when ethanol is oxidized. You will produce an aldehyde, which also ends up reacting.

#### Chemicals needed for each student (or group of students):

- Chromic acid solution (*prepared by stockroom for entire lab*): For each group, combine 3 mL of 5% sodium (or potassium) dichromate (Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> or K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>) in a beaker (or large test tube).
  - *Students*: Prepare your solution by adding about 1 mL of concentrated sulfuric acid to the 2 mL of dichromate solution in a beaker or test tube. (If a precipitate appears, swirl the mixture until it dissolves. Cool the solution to room temperature, since hot chromic acid is dangerous and converts alcohols into alkenes.)
  - Stockroom: For ten groups, combine 30 mL of the dichromate solution with 10 mL of concentrated sulfuric acid. (If a precipitate appears, swirl the mixture until it dissolves. Cool the solution to room temperature, since hot chromic acid is dangerous and converts alcohols into alkenes.)
  - Dispense and use in the hood.
- Tollens Reagent (prepared by each student group, with Instructor adding the

*ammonium hydroxide*): For each group, add 8 mL of 0.1 M silver nitrate to a beaker or flask (measure in a 10-mL graduated cylinder). Add dilute ammonium hydroxide (*the NH4OH must be prepared within one hour of being used, it cannot be prepared on a previous day*) 1 drop at a time until the brown precipitate of silver oxide that is formed just dissolves (mix by swirling after each drop is added). (*The liquid may remain slightly brownish in color, but it cannot still have a precipitate*.) Now, add 7 mL of distilled water and mix. Use about 5 mL of Tollens reagent for each of the three tubes used in the experiment.

- propanal (propionaldehyde)
- benzaldehyde
- 10% formaldehyde
- 10% glucose (dextrose)
- concentrated sulfuric acid
- 0.1 M silver nitrate
- 5% sodium or potassium dichromate (Na<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> or K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>)
- glacial acetic acid (concentrated acetic acid)
- salicylic acid
- ethyl alcohol
- isopropyl alcohol (2-propanol)
- *t*-butyl alcohol

The following chemicals are not used for reactions. They should be in small capped vials for odor detection:

- Acetone
- Benzaldehyde
- Camphor
- Vanillin
- Cinnamaldehyde
- 2,4-Pentanedione

#### **Equipment needed:**

• No specialized equipment is needed.

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