Laboratory 22: Properties of Alcohols, Aldehydes and Ketones

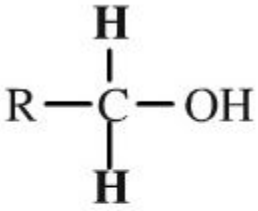
Introduction

Alcohols represent an important class of organic molecules. In this experiment you will study the physical and chemical properties of alcohols. Solubility in water, and organic solvents, combustibility, and reactivity with various chemical reagents will be examined.

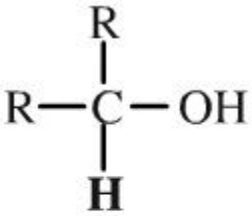
Discussion

Structure of Alcohols

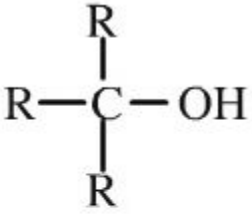
Alcohols are organic compounds containing a hydroxy (hydroxyl) (R−OH) functional group bonded to a carbon atom that is not bonded to a carbonyl carbon (C=O). Those molecules will be explored in a future experiment. If the hydroxy group is bonded to an aromatic ring (benzene ring), a class of compounds called phenols are form, which have properties different than regular alcohols, and will not be used in this experiment.
Alcohols can be separated into three subclasses, primary (1°), secondary (2°) and tertiary (3°) based on the number of alkyl (R-) groups attached to the carbon atom with the hydroxy (−OH) group attached.



Primary Alcohol



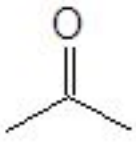
Secondary Alcohol



Tertiary Alcohol

Physical Properties of Alcohols

The most common alcohols are colorless liquids at room temperature. Their melting and boiling points are considerably higher than those of alkanes, alkenes, and alkynes of similar size due to the ability to hydrogen bond (See Hein 13.8). The ability to form hydrogen bonds also makes low molar mass alcohols soluble in water, however, as the alkane portion of the molecule increases in size the solubility decreases, because the hydrogen bonds formed by the alcohol group can not counteract the non-polar alkane part. Solubility in acetone will also be tested, as it is a widely used organic solvent because of its ability to dissolve a wide variety of compounds.



Acetone

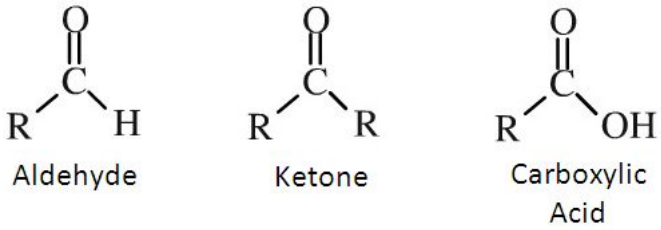
Chemical Properties

Chemically alcohols undergo two main categories of reactions: oxidation and dehydration. There are several common oxidizing agents (compounds that will oxidize organic molecules). These include:

1. Potassium Permanganate: $\xrightarrow[\Delta]{KMnO_4/H_2O}$
2. Chromic Acid: $\xrightarrow[\Delta]{K_2Cr_2O_7/H_2SO_4}$
3. Generic: $\xrightarrow{[O]}$

Generally we will be unconcerned with the identity of the oxidizing agent, and will use the generic representation. Due to the complex reaction mechanisms and balancing required, we will focus on the organic compounds formed in the reactions only. Depending on the classification of alcohol (primary, secondary, tertiary) they will be oxidized to different classes of molecules. The reaction results in the replacement of the $-OH$ group by either an Aldehyde, Carboxylic Acid or Ketone group.

- Primary Alcohol $\xrightarrow{[O]}$ Aldehyde + H_2O $\xrightarrow{[O]}$ Carboxylic Acid
- Secondary Alcohol $\xrightarrow{[O]}$ Ketone + H_2O $\xrightarrow{[O]}$ NR
- Tertiary Alcohol $\xrightarrow{[O]}$ NR



Chromic Acid/Jones Reagent

A specific test for oxidizing alcohols is the Chromic Acid test. A color change from orange to green or to blue-green within 5 seconds is a positive test, indicating the alcohol has been oxidized.

$$\text{Primary/SecondaryAlcohol} \xrightarrow[\Delta]{K_2Cr_2O_7/H_2SO_4} \text{Aldehyde/Ketone (Color Change : Orange/Red to Blue/Green)} \quad (1)$$

$$\text{TertiaryAlcohol} \xrightarrow[\Delta]{K_2Cr_2O_7/H_2SO_4} \text{NR} \quad (2)$$

Lucas Test

Another useful chemical property of alcohols is their reaction with a solution of hydrochloric acid in the presence of zinc chloride to form alkyl chlorides. This reaction is commonly referred to as the Lucas Test. This test is important because of the different rates of reactions between primary, secondary and tertiary alcohols. Generally Tertiary alcohols react immediately, secondary alcohols require 5-15 minutes to react, and primary alcohols will show little or no reactivity after 30 minutes. The evidence of a positive reaction is the conversion of a soluble alcohol to an insoluble alkyl chloride which forms a milky suspension in the solution.

$$R{-}OH + HCl(aq) \xrightarrow{[ZnCl_2]} R{-}Cl(s) + H_2O(\text{Rate : (Fast or Slow or Very Slow)} \quad (3)$$

Procedure

A. Solubility Tests - Water

1. Test the solubility of each of the listed substances with water by adding 1 mL (20 drops) of the substance to be tested to 5 mL of water in a test tube.
2. Mix each of the test tubes vigorously for 15 seconds. Wait 30 seconds. Sketch a picture of your results. In the sketch, label each liquid in the test tube. Note which pairs are miscible and which are not.
3. Wait an additional 5 minutes and record your results.
4. For any solutions that are insoluble note the relative density of the hydrocarbons with respect to water.
5. Dispose of the solutions in the waste bottle labeled "E22 Waste".

B. Solubility Tests - Acetone

1. Test the solubility of each of the listed substance with acetone by adding 1 mL (20 drops) of the substance to be tested to 5 mL of acetone in a clean dry test tube.
2. Mix each of the test tubes vigorously for 15 seconds. Wait 30 seconds. Sketch a picture of your results. In the sketch, label each liquid in the test tube. Note which pairs are miscible and which are not.
3. Wait an additional 5 minutes and record your results.
4. For any solutions that are insoluble note the relative density of the hydrocarbons with respect to acetone.
5. Dispose of the solutions in the waste bottle labeled "E22 Waste".

C. Oxidation - Chromic Acid

1. Be especially careful with the chromic acid solution as it is very corrosive. If spilled on the skin rinse immediately with cold water and inform your instructor.
2. In a clean dry test tube place 1 mL of acetone.
3. Add 5 drops of the alcohol to be tested.
4. Swirl the tubes gently to mix them.
5. Add 2 drops of the chromic acid reagent to the test tube.
6. Note any results after 5 seconds. Record any color changes.
7. Dispose of the contents of the test tubes in the waste bottle labeled "E22 Waste".
8. Write the complete chemical reaction.
9. Based on your observations is your alcohol a primary, secondary or tertiary alcohol.

D. Oxidation - Lucas Test

1. Be especially careful with the Lucas reagent as it is very corrosive. If spilled on the skin rinse immediately with cold water and inform your instructor.
2. In a clean test tube place 1 mL of the Lucas Reagent.
3. To the solution add 4 drops of the alcohol to be tested.
4. Swirl the tubes gently to mix them.
5. Note any results after 1 minute, 5 minutes, 15 minutes, and 30 minutes. Record any color changes or the formation of a cloudy mixture.
6. Dispose of the contents of the test tubes in the waste bottle labeled "E22 Waste".
7. Write the complete chemical reaction.
8. Based on your observations is your alcohol a primary, secondary or tertiary alcohol.

E. Identity of an Unknown Alcohol

1. You must complete the tests all of the previous tests before doing testing your unknown.
2. Determine which class of alcohol (primary, secondary, or tertiary) that your unknown belongs to by reacting it with chromic acid and the Lucas reagent as directed in parts E and F.
3. Be sure to record the identity of your unknown solution.
4. Record any required observations about your unknown.

Name: _____ Class: _____ Date: _____

Results

A. Solubility Tests - Water

Mixture	**Observation/Sketch**	**Miscible/Immiscible**	
		30 seconds	**5 minutes**
ethanol			
1-butanol			
1-hexanol			
1-decanol			

Table 1: Results - Solubility in Water

B. Solubility Tests - Acetone

Mixture	**Observation/Sketch**	**Miscible/Immiscible**	
		30 seconds	**5 minutes**
ethanol			
1-butanol			
1-hexanol			
1-decanol			

Table 2: Results - Solubility in Acetone

C. Chromic Acid Test - Oxidation of Alcohols

Compound	**Observations (+/-) Color Changes**	**Write the Complete Reaction**	**Conclusion** 1°, 2°, 3°
Control (H_2O)			
ethanol			
1-butanol			
2-butanol			
1-hexanol			
1-decanol			
2-methyl-2-propanol			
cyclohexanol			

Table 3: Results - Chromic Acid Test

D. Lucas Reagent - Conversion of Alcohols to Alkyl Chlorides

Compound	**Observations: A milky ppt**				1°, 2°, 3°	**Complete Chemical Reaction**
	1 min	**5 min**	**15 min**	**30 min**		
Control (H_2O)						
ethanol						
1-butanol						
2-butanol						
1-hexanol						
1-decanol						
2-methyl-2-propanol						
cyclohexanol						

Table 4: Results - Lucas Reagent

E. Analysis of an Unknown Alcohol

Unknown ID: _____

Observations	**Conclusions** (1°, 2°, 3°) + **Explanation**
Chromic Acid Test:	
Lucas Reagent:	

Table 5: Results - Unknown Alcohol

Questions

A. Solubility - Water

1. Which compounds were completely soluble in water:

2. Which compounds were insoluble in water:

3. Comment on any trends noticed in your observations of solubility and insolubility in water.

B. Solubility - Acetone

1. Which compounds were completely soluble in acetone:

2. Which compounds were insoluble in acetone:

3. Comment on any trends noticed in your observations of solubility and insolubility in acetone.

4. Comment on the differences in the solubility results between water and acetone. What properties of acetone make it a much better solvent for alcohols than water. (Hint: Draw/consider the structure of acetone compared to water.)

C. Chromic Acid Test

1. What was the purpose of the control?

2. What is the evidence of a positive test?

3. Did any of the compounds tested behave unexpectedly. Explain. Why might this have occurred?

D. Lucas Reagent

1. What was the purpose of the control?

2. What is the evidence of a positive test?

3. Did any of the compounds tested behave unexpectedly. Explain. Why might this have occurred?

4. What type of reaction occurred? Write a complete chemical reaction for ethanol.

E. Unknown

1. Is it necessary to perform both the Chromic Acid test and the Lucas test to classify your unknown?

2. What advantages does the Chromic Acid test have over the Lucas test?

3. What advantages does the Lucas test have over the Chromic Acid test?

4. Why might we perform both tests to help identify an unknown?

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Name: _____ Class: _____ Date: _____

Prelab Questions

1. Draw the Lewis structure of the following molecules used in this laboratory:
   (a) Methanol

   (b) Ethanol

   (c) 1-butanol

   (d) 2-butanol

   (e) 1-hexanol

   (a) 1-octanol

   (b) 1-decanol

   (c) cyclohexanol

2. How can you use the Lucas test to tell if an alcohol is a primary, secondary or tertiary alcohol?

3. The Chromic Acid test can be used to differentiate between what two types of alcohols. Which ones give a positive test and which give a negative test?

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