Reactions of Alcohols, Phenols, Aldehydes and Ketones

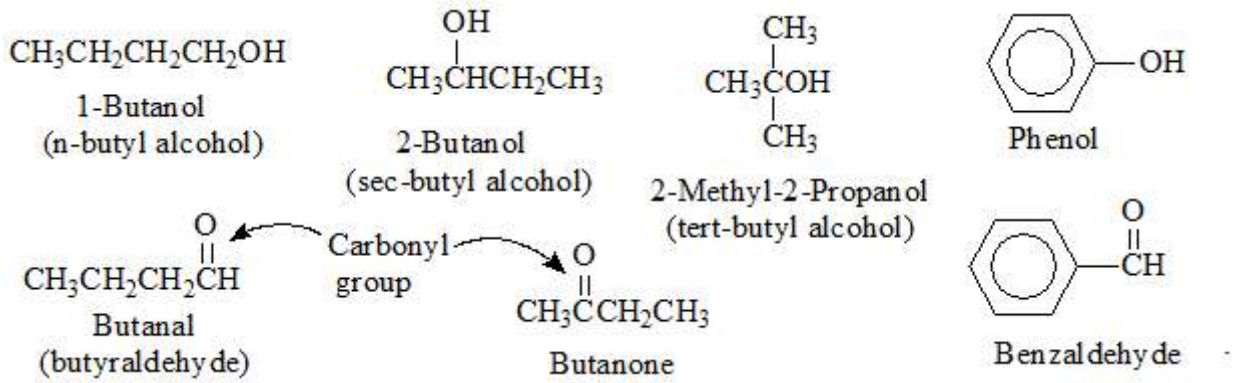
Experiment #9

Objectives: To observe reactions of alcohols, phenols, aldehydes and ketones and attempt to determine to which of these functional groups an unknown substance belongs.

Introduction

The general formula of an alcohol is ROH in which the R is an aliphatic hydrocarbon group. Phenols are aromatic alcohols, in which R is an aromatic ring. This experiment will demonstrate some reactions of alcohols and phenols. Although alkyl alcohols have an -OH group, they do not ionize in water, whereas phenols ionize like acids (donating a proton to water). The -OH group of alkyl alcohols can be positioned on different carbon atoms of the carbon chain and are classified as primary (1°), secondary (2°), or tertiary (3°) alcohols depending on whether the -OH group is attached to a carbon with 1, 2 or 3 other carbon atoms attached to it. These different alcohols react differently with Lucas reagent and with Bordwell-Wellman reagent; hence these reagents can be used for identifying the class of alcohol. The relative acidity of phenols can be used to characterize this class of alcohols relative to the alkyl alcohols.

Aldehydes and ketones both contain the carbonyl functional group. Aldehydes are much more susceptible to oxidation because a hydrogen atom is attached to the carbonyl, which is the basis for some of the chemical reactions that distinguish between these two classes of compounds. The oxidation of aldehydes can be performed with a mild oxidizing agent, such as Cu^{2+} ion in Fehling's reagent. Fehling's reagent is a deep blue Cu^{2+} solution that forms a brick-red precipitate of Cu_2O in the presence of aldehydes. A stronger oxidant such as chromic acid in Bordwell-Wellman reagent also oxidizes aldehydes, but does not oxidize ketones. The chromic acid in Bordwell-Wellman reagent is an orange-yellow solution that forms the green Cr^{3+} ion when it is reduced by alcohols or aldehydes. Ketones give no reaction.



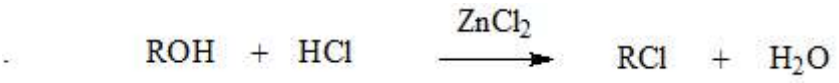
Notice: Each student must have his/her own unknown and be sure to record the number of your unknown on the report sheet. If you work in pairs, each pair must have 2 unknowns. Keep the tubes containing your unknown at your bench until you finish all parts of the lab.

Procedure

Add 100 mL of tap water to a 250 mL beaker and heat it on a hot plate for Parts A and D.

A. Lucas Test for Primary, Secondary and Tertiary Alcohols.

The Lucas reagent is a solution of zinc chloride in concentrated hydrochloric acid. This solution must be made freshly to get proper results. The test depends on a difference in the rate of reaction of these alcohols. The general equation for the reaction is:



- Tertiary alcohols react IMMEDIATELY. The test tube will get hot, and because the alkyl chloride is insoluble in water two layers may be apparent, or a cloudy dispersion forms.
- Secondary alcohols will become cloudy in 5 to 10 minutes. If cloudiness does not appear place test tube in a warm water bath and observe.
- Primary alcohols give no reaction in a reasonable length of time.

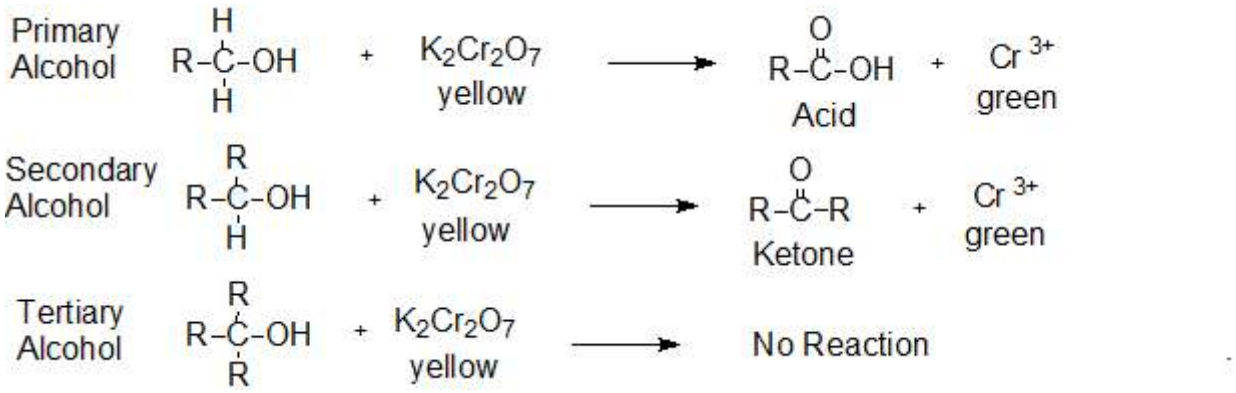
CAUTION! Lucas reagent contains concentrated hydrochloric acid - Handle It With Care

1. Place 1 ml of Lucas reagent in each of four (5 if you work in pairs) clean test tubes.
2. Add 6 drops of n-butyl alcohol (1-butanol) to one test tube. Shake the test tube to mix the reagents and notice whether the mixture gets cloudy and how long it takes.
3. In a second test tube place 6 drops of sec-butyl alcohol (2-butanol), shake and note how long it takes the tube to get cloudy.
4. In a third test tube place 6 drops of t-butyl alcohol (2-methyl-2-propanol), shake and note how long it takes the tube to get cloudy.
5. In the fourth (and fifth for pairs) test tube place 6 drops of your unknown and shake.
6. Record observations on the Report Sheet for this section.

Dispose of these reagents in the "Liquid Waste" container in the hood.

B. Bordwell-Wellman Test for Primary, Secondary and Tertiary Alcohols

The Bordwell-Wellman reagent contains potassium dichromate dissolved in sulfuric acid. The orange-yellow color is due to the $Cr_2O_7^{2-}$ ion. The oxidation number of chromium is +6. This reagent will oxidize primary and secondary alcohols and chromium is reduced to the greenish colored chromium(III) ion, Cr^{3+}. This color change from orange-yellow to green serves as an indicator for the presence of a primary or secondary alcohol. A primary alcohol is oxidized first to an aldehyde, which will be further oxidized to an acid.



USE EXTREME CARE WITH THIS REAGENT, IT IS VERY CORROSIVE!
WASH IMMEDIATELY IF YOU GET ANY ON YOUR SKIN OR CLOTHING!!!

1. Place 0.5 ml of acetone in each of four (or 5 if you work in pairs) small test tubes.

2. Add 0.5 mL of n-butyl alcohol to one test tube; 0.5 mL of sec-butyl alcohol to a second test tube; 0.5 mL of tert-butyl alcohol to a third test tube; and 0.5 mL of your unknown to the remaining test tube(s).

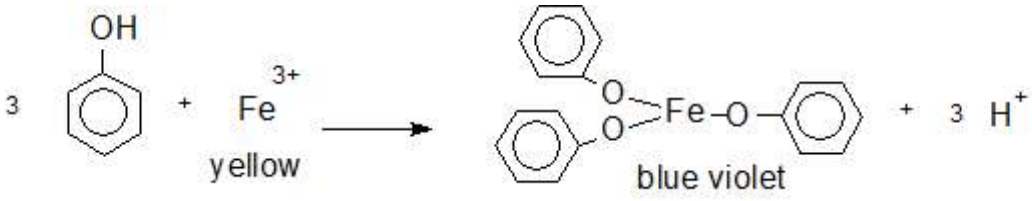
3. To each test tube add 1 drop of Bordwell-Wellman reagent and shake.

4. Record your observations on the Report Sheet for this section.

Dispose of these reagents in the Organic Liquid Waste container in the hood.

C. Phenols

Phenols form highly colored coordination complexes with ferric ion. A blue-violet colored solution results.



1. Place 0.5 mL of 3% phenol solution in a small test tube.

2. Place 0.5 mL of your unknown and 0.5 mL of water in another small test tube.

3. Add 1 drop of ferric chloride solution to each and shake.

4. Answer questions about this reaction on the Report Sheet.

Dispose of these reagents in the Organic Liquid Waste container in the hood.

D. Fehling's Test for Aldehydes

The water bath you set up earlier should be boiling. Set the hot plate temperature control to a medium setting. Use 5 clean small test tubes if working alone, or 6 test tubes if working in pairs.

1. Add 5 mL of Fehling's Solution A to a small beaker and mix 5 mL of Fehling's Solution B with it. Notice the change in color of the Cu^{2+} ion when these solutions are mixed.

2. Add 1.5 mL of the mixture prepared in step 1 to each of 5 (or 6) clean small test tubes.

3. Add 5 drops of the following test compounds (aldehydes/ketones) to the Fehling's reagent in each test tube. Acetone in tube 1; benzaldehyde in tube 2; butanal (butyraldehyde) in tube 3; butanone in tube 4; and unknown in tube 5 (and 6).

4. Make sure the tubes are properly labeled before placing them in the boiling water bath.

5. After heating the mixtures in boiling water for 5 minutes, take note of any changes in color or formation of a red precipitate at the bottom of the tube.

6. Record your observations on the Report Sheet for this section.

7. Turn off the hot plate, the water bath is no longer needed.

Dispose of these reagents in the "Liquid Waste" container in the hood.

E. Oxidation with Bordwell-Wellman reagent

CAUTION!! Concentrated sulfuric acid in the Bordwell-Wellman reagent is very corrosive. Handle it with care.

1. Add 0.5 mL of each of the following test compounds (aldehydes/ketones) to 5 (or 6 if working in pairs) clean test tubes: acetone in tube 1; benzaldehyde in tube 2; butanal (butyraldehyde) in tube 3; butanone in tube 4; and unknown(s) in tube(s) 5 (and 6).

2. Add 2 drops of Bordwell-Wellman reagent solution to each of the test tubes containing the test aldehydes, alcohol or ketone from step 1.

3. Mix well and note any color changes after a minute or two. If there is a reaction, the color should change from yellow/orange to green or brown.

4. Record your observations on the Report Sheet for this section.

Dispose of these reagents in the "Liquid Waste" container in the hood.

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Name _____ Section _____

Reactions of Alcohols, Phenols, Aldehydes and Ketones
Experiment #9 Prelab Exercise

1. Draw structures of one primary alcohol, one secondary alcohol and one tertiary alcohol and give the correct name for each structure you have drawn.

2. Describe the difference between alcohols and phenols.

3. Describe the difference between an aldehyde and a ketone, and indicate how each differs from an alcohol.

Name _____ Section _____

4. Describe what is meant by oxidation and reduction in relation to organic compounds, giving one example of oxidation of an organic compound and one example of reduction of an organic compound. The compound you use for the example may be the same or different for the oxidation and the reduction reactions. Be sure to indicate what oxidizing agent is used and what reducing agent is used for each example.

Name _____ Section _____

Reactions of Alcohols, Phenols, Aldehydes and Ketones
Experiment #9 Data & Report Sheet

Unknown Number _____

Important: Be sure to enter your unknown number. You will not receive credit for lab if you have the same unknown number as another person in this lab section.

Parts A and B. Chemical Tests of Alcohols

Record your observations, noting any precipitate, cloudiness or color change.

		Lucas Reagent	Bordwell-Wellman Reagent
#1	n-butyl alcohol		
#2	sec-butyl alcohol		
#3	tert-butyl alcohol		
#4	Unknown # _____		

A-1. After looking at the results in the above table, would you conclude your unknown is a primary, secondary or tertiary alcohol?

A-2. Show the chemical reactions for the Lucas test with each of the butyl alcohols (n-, sec- and tert-butyl alcohol or 1-butanol, 2-butanol and 2-methyl-2-propanol) that react with this reagent. You do not need to show cases where there is no reaction. Indicate whether the reaction is fast or slow.

Name _____ Section _____

A-3. Show the chemical reactions for the Bordwell-Wellman test with each of the butyl alcohols (1-butanol, 2-butanol and 2-methyl-2-propanol) that react with this reagent. You do not need to show cases where there is no reaction.

C. Phenols

C-1. What is the color of the ferric chloride solution before adding it to phenol?

C-2. What is the color of the solution after the reaction between phenol and ferric chloride takes place?

C-3. From the results with ferric chloride, would you classify your unknown as a phenol?

Name _____ Section _____

Parts D and E. Tests for aldehydes with oxidizing agents.

Fill in your observations for the Fehling's test and Bordwell-Wellman test in the table below. [Indicate whether you observed a positive reaction (+) or no reaction (-) in each test **and** note any color changes or other changes such as cloudiness].

	Fehling's Test	Bordwell-Wellman Test
Acetone		
Benzaldehyde		
Butyraldehyde		
Butanone		
Unknown # _____		

D-1. After looking at the results in the above table, would you conclude your unknown is an aldehyde, a ketone or an aromatic aldehyde (like benzaldehyde)?

D-2. Did Fehling's reagent and Bordwell-Wellman reagent react differently with benzaldehyde compared to butyraldehyde?

D-3. Why would benzaldehyde react differently than other aldehydes such as butyraldehyde?

Name _____ Section _____

D-4. You used Bordwell-Wellman reagent to test for alcohols and for aldehydes and ketones. Which of the alcohols reacted with this reagent and what compounds of the aldehydes and ketones reacted with this reagent?

D-5. Are you able to determine whether your unknown is an alcohol, aldehyde, ketone or phenol from the observations you made in each of the tests? If so, which category of these functional groups is your unknown? (See comment below)

Comment regarding identifying your unknown:You should be able to distinguish between different alcohols and aldehydes and ketones by comparing the different tests in each group. For example, primary and secondary alcohols as well as aldehydes react with Bordwell-Wellman reagent, but the alcohols do not react with Fehling's reagent. Similarly, a tertiary alcohol and ketones do not react with Bordwell-Wellman reagent, but a tertiary alcohol will react readily with Lucas reagent and a ketone does not.