Reactions of Alcohols, Phenols and Aldehydes

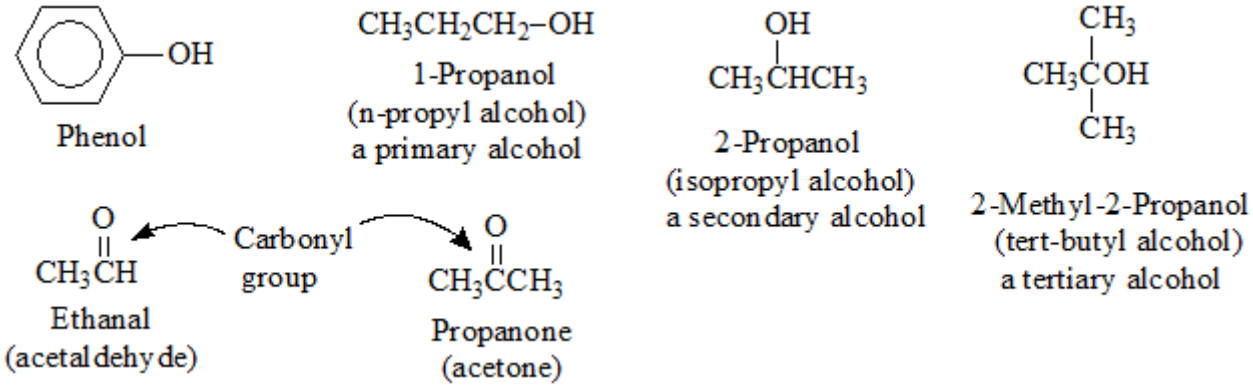
Experiment #10

Objectives: To observe reactions of alcohols, phenols and aldehydes 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. 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. Alcohols are not oxidized by Cu^{2+} ion under these conditions. A stronger oxidant such as chromic acid in Bordwell-Wellman reagent oxidizes primary and secondary alcohols and also oxidizes phenols and 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; they are not oxidized with these reagents.

This experiment will demonstrate some reactions of alcohols, phenols and aldehydes. 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.



Materials: Acetaldehye; 1-propanol; 2-propanol (isopropyl alcohol); 2-methyl-2-propanol (tert-butyl alcohol); 3% phenol; Lucas reagent; Bordwell-Wellman reagent; Fehling's reagent A and B; 0.3 M ferric chloride solution; unknown alcohols, aldehydes and phenol.

Notice: Each student must have her/his 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 C.

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 alcohols. The general equation for the reaction is:

$$ROH + HCl \xrightarrow{ZnCl_2} RCl + H_2O$$

- 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 within a few minutes, place the test tube in a warm water bath for a few minutes and observe.

- Primary alcohols give no reaction in a reasonable length of time, even when placed in a warm water bath.

Aldehydes, ketones and phenol should give no reaction with Lucas reagent, even after warming.

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

Label 6 (or 7 if working in pairs) small test tubes with numbers 1 thru 6 (or 7). Record your observations for each tube on the report sheet as you complete that step.

1. Place 1 ml of Lucas reagent in each of the labeled clean test tubes.

2. Add 6 drops of 1-propanol to test tube #1. Shake the test tube to mix the reagents and notice whether the mixture gets cloudy immediately. If it does not get cloudy immediately, place it in the warm water bath for a few minutes.

3. Add 6 drops of 2-propanol (isopropyl alcohol) to test tube #2, shake and note how long it takes the tube to get cloudy. If it does not get cloudy immediately, place the tube in a warm water bath for a few minutes and take note of any cloudiness.

4. In test tube #3 place 6 drops of 2-methyl-2-propanol (t-butyl alcohol), shake and note

how long it takes the tube to get cloudy.

5. In test tube #4 place 6 drops of acetaldehyde and shake. If it does not get cloudy immediately, place the tube in a warm water bath for a few minutes and take note of any cloudiness.

6. In test tube #5 place 6 drops of 3% phenol solution and shake. If it does not get cloudy immediately, place the tube in a warm water bath for a few minutes and take note of any cloudiness.

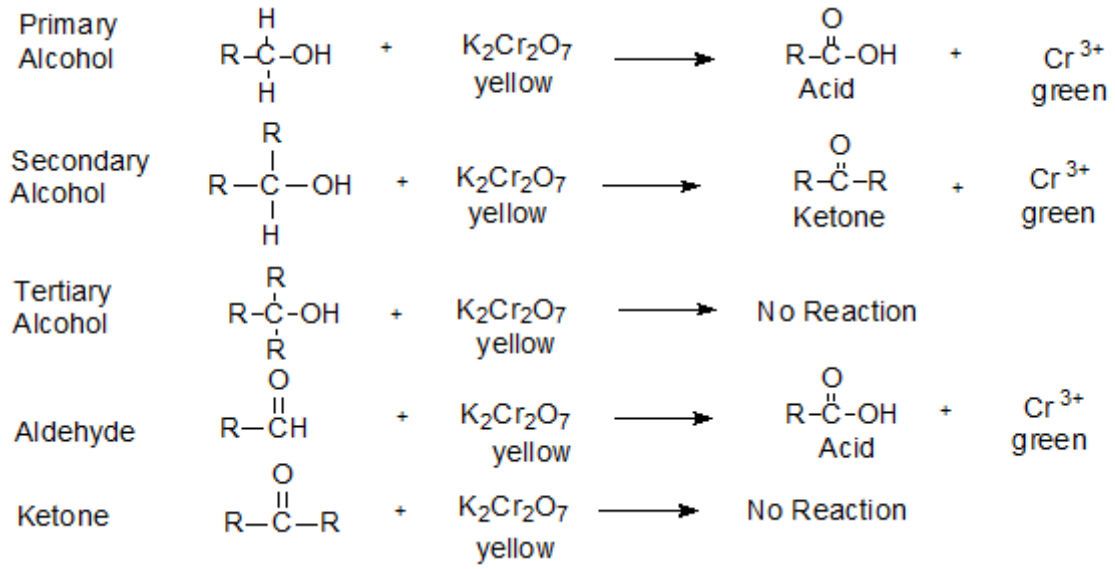
7. In test tube #6 add 6 drops of your unknown (and to tube #7 add 6 drops of your partner's unknown if you work in pairs). Shake the tubes and note whether they get cloudy immediately. If not, place them in the warm water bath and notice whether they get cloudy within a few minutes.

Be sure to record all of your data on the report sheet for this part.

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

B. Bordwell-Wellman Test for Primary and Secondary Alcohols and Aldehydes.

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 shades of green serves as an indicator for the presence of a primary alcohol, secondary alcohol, or aldehyde. 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!!!

After rinsing the test tubes used in part A, you can reuse them for this part.

1. Place 0.5 mL of 1-propanol in test tube #1; 0.5 mL of 2-propanol in test tube #2; 0.5 mL of 2-methyl-2-propanol (tert-butyl alcohol) in test tube #3; 0.5 mL of acetaldehyde in test tube #4; 0.5 mL of 3% phenol solution in test tube #5; and 0.5 mL of your unknown in test tube #6 (and 0.5 mL of your partner's unknown in tube #7 if you work in pairs).

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. 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. You can rinse the test tubes from part B and reuse them for this part.

1. Using your 10 mL graduated cylinder, add 8 mL of Fehling's Solution A to a small beaker and mix 8 mL of Fehling's Solution B with it. Notice the change in color of the Cu^{2+} ion when these solutions are mixed.

2. Use a plastic pipet to add about 1.5 mL of the mixture prepared in step 1 to each of the clean small test tubes.

3. Add 5 drops of 1-propanol to test tube #1; 5 drops of 2-propanol to test tube #2; 5 drops of 2-methyl-2-propanol (tert-butyl alcohol) to test tube #3; 5 drops of acetaldehyde to test tube #4; 5 drops of 3% phenol solution to test tube #5; and 5 drops of your unknown(s) to the remaining test tubes (#6 and #7 if you work in pairs).

4. Mix all the tubes well and 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.

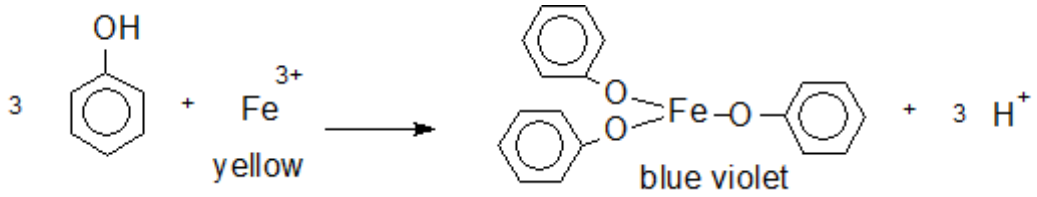
6. Record your observations on the Report Sheet for this section.

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.

D. 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 in another small test tube. Do the same for your partner's unknown if you work in pairs.

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

4. Answer question 5 about this reaction on the Report Sheet.

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

Name _____ Section _____

Reactions of Alcohols, Phenols and Aldehydes
Experiment #10 **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. Briefly 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 alcohol and one example of oxidation of an aldehyde. Be sure to indicate what oxidizing agent is used.

Name _____ Section _____

Reactions of Alcohols, Phenols, Aldehydes and Ketones
Experiment #10 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, B and D. Chemical Tests of Alcohols and Aldehydes
Record your observations, noting any precipitate, cloudiness or color change.

#		Lucas Reagent	Bordwell-Wellman	Fehling's Reagent
1	1-propanol			
2	2-propanol			
3	2-methyl-2-propanol (tert-butyl alcohol)			
4	acetaldehyde			
5	3% phenol solution			
6	unknown #_____			

1. If your unknown reacted with Lucas reagent, can you say whether it is a primary, secondary or tertiary alcohol? If it did not react with Lucas reagent, just write that it did not react, but could possibly be a primary alcohol.

2. If your unknown reacted with Bordwell-Wellman reagent, can you say whether it is a primary or secondary alcohol, considering whether it reacted with Lucas reagent as well?

Name _____ Section _____

3. If you got no reaction with Lucas reagent, but your unknown reacted with Bordwell-Wellman reagent, is it possible to say from these two tests whether your unknown is an alcohol or an aldehyde? Explain in the context of these two tests.

4. From the results of the Fehling's test, would you say that your unknown is an aldehyde? Explain.

5. From your results in part D with addition of ferric chloride solution, would you say that your unknown is a phenol? Did your unknown change color when you added ferric chloride solution?

Considering the results of all of these tests, what conclusion can you make about your unknown? Is it a primary alcohol, a secondary alcohol, a tertiary alcohol, an aldehyde, a phenol, or none of these.