

Properties of Alcohols and Phenols

Experiment #3

Objectives: (A) To observe the solubility of alcohols relative to their chemical structure and (B) chemical tests will be performed to distinguish primary, secondary and tertiary alcohols and a color test will be performed for phenol.

Introduction

The general formula of an alcohol is ROH in which the R is an aliphatic hydrocarbon group. Alcohols may be looked upon as derivatives of water, HOH. One hydrogen of water is substituted by an alkyl group, R. Like water, alcohols show hydrogen bonding. As the chain of the R group increases the hydrocarbon character of the compound overshadows the polar character of the OH group. Consequently, the solubility and boiling point of an alcohol are affected by the length of the carbon chain and the shape of the molecule. The short chain alcohols are soluble in water, whereas the longer chain alcohols are insoluble in water. In general a molecule which is more compact (i.e., more branched) will be more soluble in water and will have a lower boiling point than the straight chain isomer. Phenols are aromatic alcohols, in which R is an aromatic ring. This experiment will demonstrate some of the properties of alcohols and phenols.

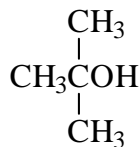
Although alkyl alcohols have an -OH group, they do not ionize in water, whereas phenols ionize like acids (donating a proton to water). The -OH group of alkyl alcohols can be positioned on different carbon atoms of the carbon chain and are classified as primary (1°), secondary (2°), or tertiary (3°) 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. This property will be exploited in the identification of phenol.



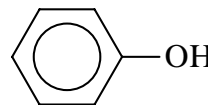
1-Butanol



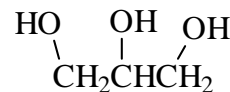
2-Butanol
(sec-butyl alcohol)



1,1-Dimethyl ethanol
(tert-butyl alcohol)



Phenol



Glycerol
(glycerin)

Apparatus and Reagents

Test tubes, Bordwell-Wellman reagent, n-butyl alcohol (1-butanol), secondary-butyl alcohol (2-butanol), tertiary-butyl alcohol (1,1-dimethylethanol), glycerol, ferric chloride solution, Lucas reagent, phenol (3% solution), 10% sodium hydroxide solution, acetone, unknown.

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 unknown tubes at your bench until you finish all parts of the lab.

Part A. Solubility

To test the solubility in water of alcohols, add several (6 to 8) drops of alcohol to about 2 mL water, shake the test tube vigorously [**Use a cork to stopper the test tube; do not use your thumb**] and notice if two layers form. If two layers form, the liquids are immiscible, hence, insoluble or of only limited solubility. You should make sure you mix viscous alcohols (such as glycerol) well because they may not dissolve immediately.

Procedure

1. Place 2 ml of water into each of 5 small test tubes.
2. To one test tube add 5 drops of n-butyl alcohol (1-butanol). To a second test tube add 5 drops of sec-butyl alcohol (2-butanol). To a third test tube add 5 drops t-butyl alcohol (1,1-dimethylethanol), and to a fourth test tube add 5 drops glycerol, to a fifth test tube add 5 drops of your unknown. If you are working in pairs, you should have a sixth test tube with the second unknown.
3. Stir each mixture by holding the top of the tube and tapping the bottom with your finger. Note whether these substances are soluble in water. You may have to stir the viscous glycerol sample vigorously. Record your observation on the Report Sheet, Table 1.

You may dispose of these solutions in the sink and flush with water.

Part B. Lucas Test for Primary, Secondary and Tertiary Alcohols.

The Lucas reagent is a solution of zinc chloride in concentrated HCl. 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 chloride is insoluble 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 hot 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

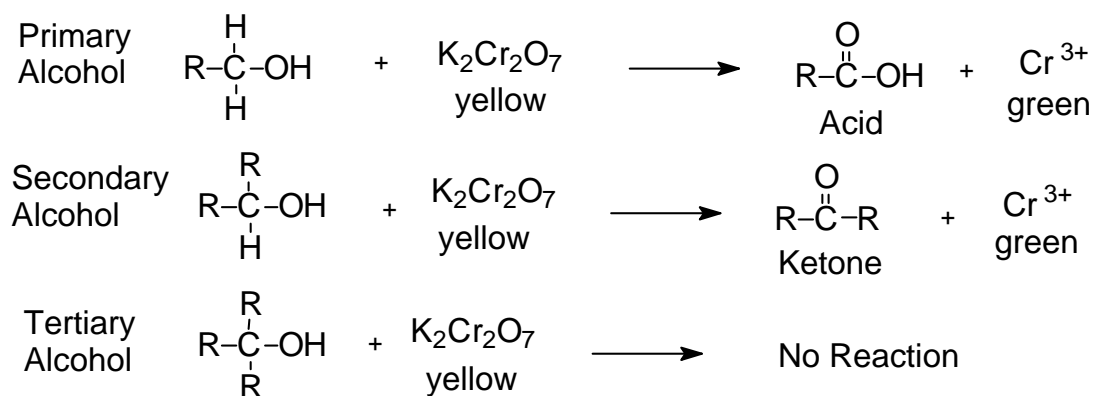
Procedure

1. Place 1 ml of Lucas reagent in each of four (5 if you work in pairs) clean test tubes.
2. Add 4 drops of 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 4 drops of 2-butanol, shake and note how long it takes the tube to get cloudy.
4. In a third test tube place 4 drops of t-butyl alcohol, shake and note how long it takes the tube to get cloudy.
5. In the fourth (and fifth for pairs) test tube place 4 drops of your unknown and shake.
6. Record observations on the Report Sheet, Table 2.

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

Part C. Bordwell-Wellman Test

The Bordwell-Wellman test solution contains potassium dichromate dissolved in sulfuric acid. It is an orange-yellow solution. The orange-yellow color is due to the $\text{Cr}_2\text{O}_7^{2-}$ ion. The oxidation number of chromium is +6. This reagent will oxidize primary and secondary alcohols and, in turn, becomes reduced. The chromium in dichromate is reduced to chromic ion, Cr^{3+} . A greenish colored solution results. 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!!!**

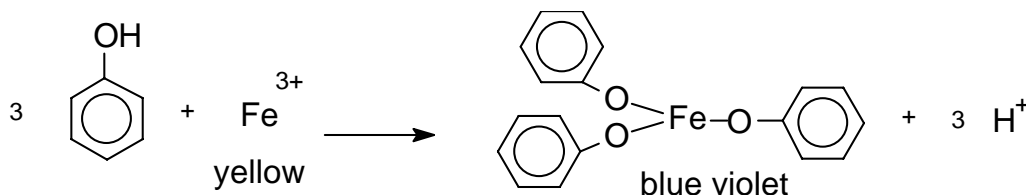
Procedure

1. Place 1 ml of acetone in each of four (or 5 if you work in pairs) small test tubes.
2. Add 2 or 3 drops of n-butyl alcohol to one test tube; 2 or 3 drops of 2-butanol to a second test tube; and 2 or 3 drops of t-butyl alcohol to a third test tube; and 2 or 3 drop 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, Table 2.

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

Part D. Phenols

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



Procedure

1. Place 10 drops of 3% phenol solution in a test tube.
2. Place 10 drops of your unknown in 10 drops of water, if it was soluble in part A, in another test tube. If your unknown was not soluble in water, would you expect it to be phenol?
3. Add 1 drop of ferric chloride solution to each and shake.
4. Record your observations on the Report Sheet.

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

Name _____

Section _____

Alcohols and Phenols

Experiment #3

Pre-Lab 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. Explain why many alcohols are soluble in water. What part of an alcohol will limit its solubility in water? What part of an alcohol may make it soluble in water.
3. Describe the difference between alcohols and phenols.

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Alcohols and Phenols

Experiment #3

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.

Table 1. Solubility Test

Solubility in water

| | | |
|----|--|--|
| #1 | 1-butanol | |
| #2 | 2-butanol | |
| #3 | t-butyl alcohol (1,1-dimethylethanol) | |
| #4 | glycerol | |
| #5 | unknown # _____ | |

QUESTIONS

1. Would you predict that methanol, ethanol and propanol are soluble in water? Explain your answer.

Table 2. Chemical Tests of Alcohols

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

Lucas Reagent

Bordwell-Wellman Reagent

| | | | |
|----|-----------------|--|--|
| #1 | n-butyl alcohol | | |
| #2 | 2-butanol | | |
| #3 | t-butyl alcohol | | |
| #4 | unknown | | |

Name _____ Section _____

2. Write the structural formulas for 1-butanol, 2-butanol and t-butyl alcohol.
3. Show the chemical reactions for the Lucas test and the Bordwell-Wellman test with each of the three butyl alcohols.
4. What is the color of the ferric chloride solution before adding it to phenol?
5. What is the color of the solution after the reaction between phenol and ferric chloride takes place?
6. From the results with ferric chloride, would you classify your unknown as a phenol?
7. What is your unknown number and what class of alcohol is your unknown (primary, secondary, tertiary, or phenol):