Chem 306 Name_____

Partner_____

Section (Circle) M Tu W Th Date_____

ALCOHOLS AND PHENOLS

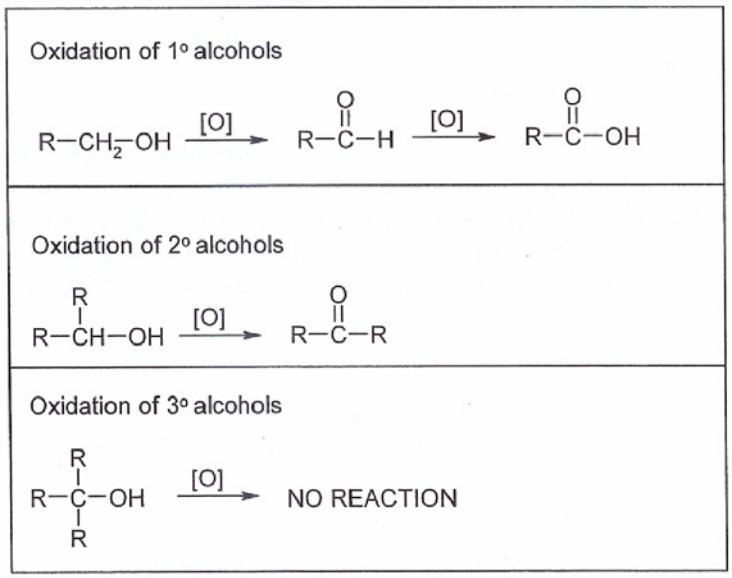
Purpose: In this laboratory activity you will investigate some chemical and physical properties of alcohols and phenols

Introduction:

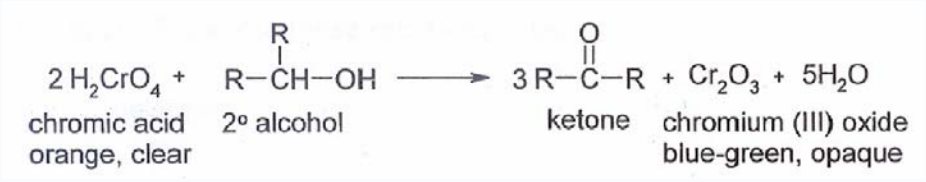
Organic molecules containing the alcohol functional group and/or a phenol structural feature are very common in nature and household products.

Ethanol, CH_3CH_2OH, is the "alcohol" in alcoholic beverages and is also used as a solvent for perfumes, mouthwash, food flavorings, and pharmaceuticals. Methanol, CH_3OH, the simplest alcohol, is used as a solvent, fuel, and as a denaturant for ethanol. Some alcohols, called diols and glycols, contain more than one hydroxyl group. Ethylene glycol, antifreeze, and glycerin (also known as glycerol) are common examples. As you will discover in this lab activity, there are many other alcohols found in foods and consumer products.

One of the most important reactions of alcohols in nature is oxidation. Oxidation reactions are very important in biochemistry. Many intermediates in carbohydrate and lipid metabolism are alcohols that undergo oxidations in our cells. The oxidation of alcohols in the body is facilitated by enzymes. For example, dehydrogenase enzymes in the liver and cells of the body work to oxidize alcohols. Both primary and secondary alcohols are easily oxidized to form carbonyl compounds. Tertiary alcohols are resistant to oxidation.

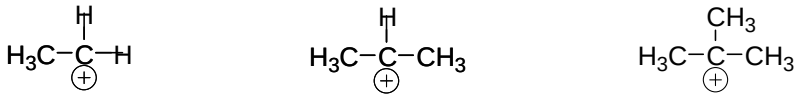


In this lab the chromium (VI) ion, Cr 6+, will be the oxidizing agent for the alcohol oxidation reactions. (Chromium (VI) oxide, CrO_3, is combined with sulfuric acid, H_2SO_4, to make a chromic acid reagent, H_2CrO_4.) In the laboratory, a change in the physical appearance of an oxidizing agent can indicate the presence of an oxidizable functional group such as alcohols. The chromic acid reagent, an orange color, will form a blue-green color when it oxidizes and alcohol. (What gets reduced here?) We will use this reaction as a chemical test for primary and secondary alcohols.



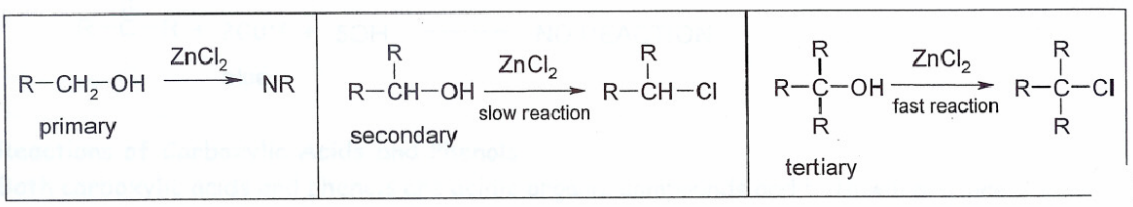
Chromium (VI) is toxic and should be treated with care. Chromium (III) is an essential mineral in your diet. You may notice chromium chloride (or other chromic salt) listed as an ingredient in your vitamins. If a chemist wrote the vitamin label it would say chromium (III) chloride, because then it is clear which form of this metal is present.

Another chemical test that can be used to classify alcohols is the Lucas Test. This test is used to distinguish between water-soluble primary, secondary, and tertiary alcohols. In secondary and tertiary alcohols, the hydroxyl group (-OH) is substituted with a chloro group (- Cl). The mechanism of this reaction includes a carbocation intermediate. Reactions will occur faster if they proceed via a stable carbocation intermediate. In general, tertiary carbocations are more stable than secondary carbocations and secondary carbocations are more stable than primary carbocations. In fact, primary carbocations rarely form at all.

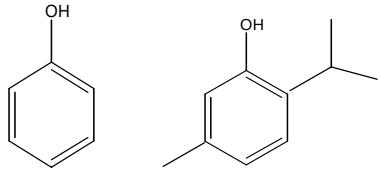


Primary carbocation (least stable) | *Secondary carbocation* | *Tertiary carbocation (most stable)*

Lucas Reagent is a mixture of zinc chloride, $ZnCl_2$, and concentrated hydrochloric acid, HCl. Upon addition of this reagent, a tertiary alcohol reacts rapidly and immediately gives an insoluble white layer. A tertiary alcohol reacts rapidly because it readily forms a stable tertiary carbocation. A secondary alcohol reacts slowly and, after heating slightly, gives the white layer within 10 minutes. A primary alcohol does not react. Any formation of an emulsion or layered phase is a positive test.

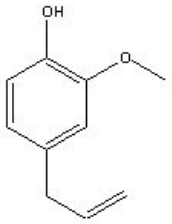


From the standpoint of structure, alcohols and phenols look much alike: both contain the hydroxyl functional group. However, their chemical properties are quite different. Phenols are slightly acidic and resist oxidation. (Can you explain why?)

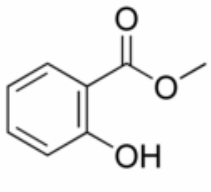


Phenol

Thymol



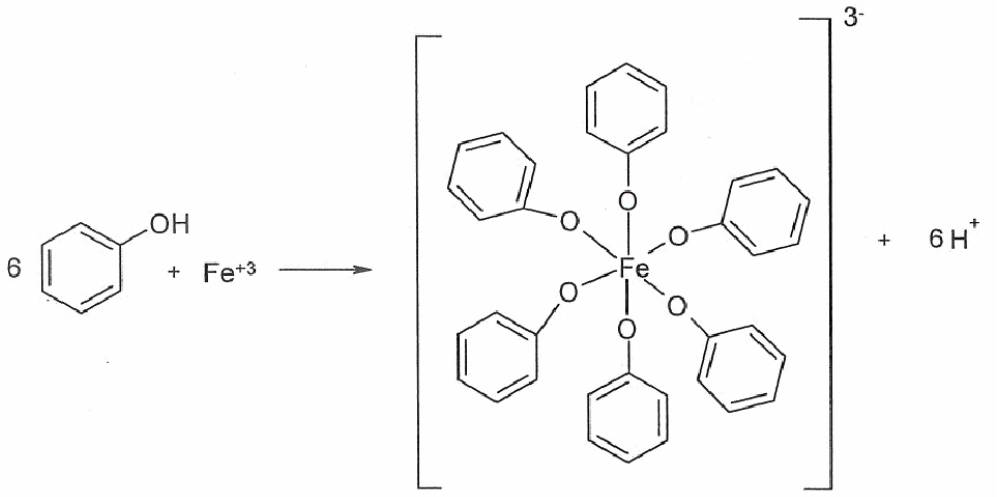
Eugenol



Methyl Salicylate

Many phenols have a strong odor which you may associate with disinfectants. Phenol itself is a strong disinfectant and is present in low concentrations in mouthwashes and sore-throat remedies. How do phenols kill bacteria? Phenols interfere with the structure of proteins in bacteria thereby destroying the function of crucial proteins needed for the survival of microbes. Thymol, a terpene and a phenol, is found in thyme essential oil and has strong antiseptic properties. It is also used as a preservative in halothane, an anesthetic. Some phenolic compounds such as methyl salicylate and eugenol (found in clove oil) can act as topical anesthetics via a topical cooling sensation that partially blocks nerve transmission. Phenols, specifically polyphenols, can also serve as antioxidants that trap free radicals and prevent the process of auto-oxidation, the oxidation of an organic compound by O_2.

Most phenols react with ferric ion, Fe 3+(aq), to form a colored complex (often purple but can also form red, blue, or green complexes). Therefore, Fe 3+(aq) can be used as a chemical test to detect a phenol structure.



purple complex

Experiment:

Please keep all test-tubes containing alcohols/phenols in the hood. You may bring your test tubes to your bench if they are stoppered. When disposing of chemicals in the waste bottle, rinse your test tubes with a small volume of deionized water (in the squirt bottles).

I. PHYSICAL PROPERTIES OF ALCOHOLS AND PHENOLS

A. Viscosity of Alcohols and Diols

Viscosity is the resistance of a substance to flow. For example, water has less resistance to flow than honey. Therefore, honey is more viscous than water.

Compare the viscosity of glycerin, ethylene glycol, and ethanol by letting each drop slowly from a medicine dropper. Which is the most viscous? Record your results.

B. Solubility

Into separate test tubes place 10 drops of each of the following alcohols:
1 – butanol,2 – butanol, t – butyl alcohol. Dilute the alcohols with water by adding 1 mL of distilled water to each test tube. Stopper your tests tubes and mix gently. Are all of the solutions homogeneous? Record your results

C. pH

Test the pH of each of the aqueous solutions in part B above and the aqueous phenol solution provided. Do the test by first dipping a clean glass rod into the solutions and then transferring a drop of liquid to a small piece of broad pH indicator paper (pH range 1 – 12) and read the value of the pH by comparing the color to the chart on the dispenser. Be sure to clean the stirring rod between samples!! Record your results.

II. CHEMICAL PROPERTIES OF ALCOHOLS AND PHENOLS

A. Oxidation Test for Alcohols

Caution! Chromic acid ($H_2SO_4/CrO_3 = H_2CrO_4$) will cause severe burns! In case of skin contact, wash with plenty of water.

To each of three test tubes add 1 mL of acetone (as a solvent). To one test tube add three drops of 1 – butanol, to another add three drops of 2 – butanol,, and to the third test tube add three drops of 2 – methyl – 2 – propanol. Swirl each tube to mix, then add three drops of chromic acid to each, swirl and observe. Development of a blue-green color indicates an oxidation. Record your observations.

B. Substitution (Lucas) Test for Alcohols

Caution! The Lucas Reagent will cause severe burns! In case of skin contact, wash with plenty of water.

To each of three test tubes add 1 mL of Lucas Reagent (conc. $HCl/ZnCl_2$). Then add about 30 drops of 1 – butanol to one, 30 drops of 2 – butanol to another, and 30 drops of 2 – methyl – 2 – propanol to the third. Swirl and note the length of time it takes each to become cloudy or to form an oily layer. You will need to monitor this reaction carefully as the oily layer can take some time to form. Record your observations.

C. Fe^{+3} Test for Phenols

Obtain five test tubes. Place 0.5 mL of each of the following in a test tube: ethyl alcohol, phenol solution, Bengay, clove oil, and Listerine. Add 1 mL of ethyl alcohol to the clove oil to help dissolve any phenols in the iron chloride solution. Add 3 drops of a 1.0% (w/v) $FeCl_3$ (aq) solution to each test tube including the pure alcohol sample. Why are we testing a pure sample of the alcohol? Mix and look for the purple color (or other possible colors described in the introduction☺) which indicates the presence of a phenol.

III. UNKNOWN DETERMINATION

Obtain an unknown from your instructor. Each pair of students will analyze two unknowns. Your unknown may be a primary, secondary, or tertiary alcohol, or a phenol.

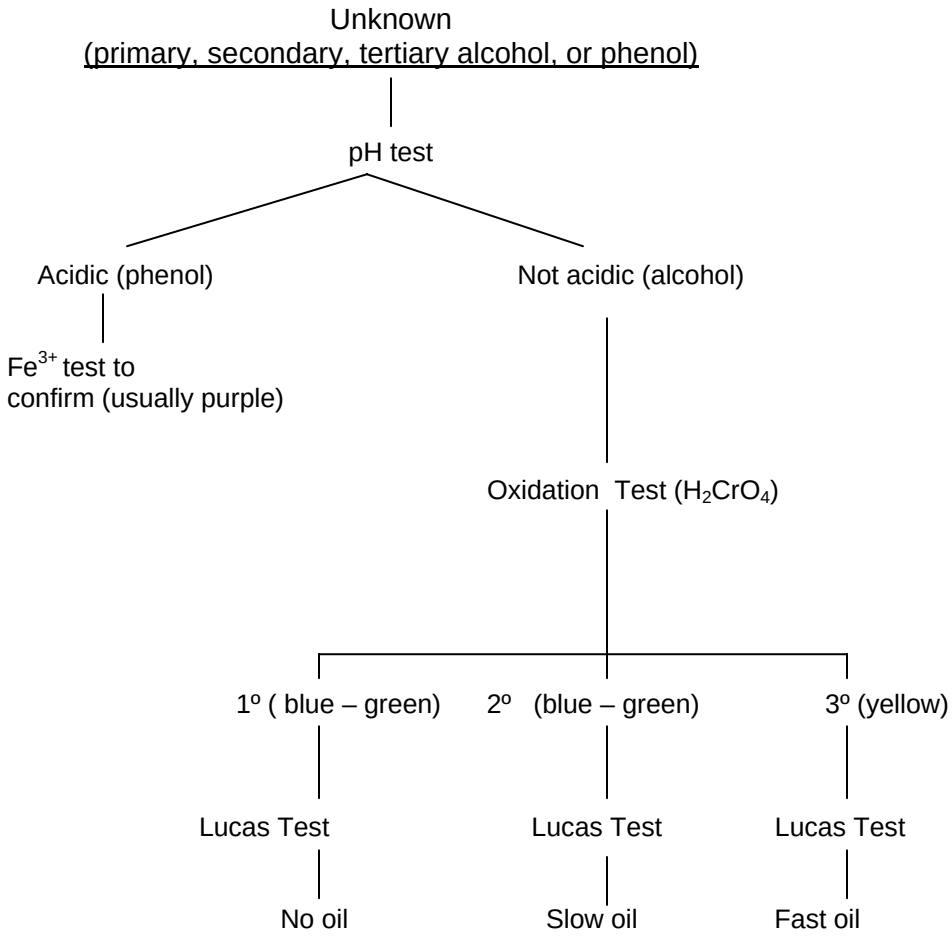
Carry out the tests in the sequence shown in the flow chart (pg 6) by following the branches showing your results. Use the general instructions for the test that you used for the known compounds, substituting your unknown for the known compounds. If you and your partner determine that both unknowns are phenols, please choose another unknown.

If your unknown begins to solidify, gently warm it in a hot water bath. Record your results.

IV. ALCOHOLS AND PHENOLS IN HOUSEHOLD PRODUCTS

Look over the ingredient labels on the household products in lab (bubble gum, hand lotion, mouthwash, analgesic lotion, toothpaste, hair conditioner, lip balm, etc.) Find products with one of the following type of organic compounds: a phenol, triol, any alcohol, and a diol or glycol.. Use your text, lecture notes, and/or the resources in lab to research the function of each of these ingredients in the product. Record your findings on the data table You should have an example of one of each of these compounds (any alcohol, a diol/glycol, triol, and a phenol) in your data table

Flowchart for Unknown Determination


Unknown
(primary, secondary, tertiary alcohol, or phenol)
pH test
Acidic (phenol)
Not acidic (alcohol)
Fe^{3+} test to
confirm (usually purple)
Oxidation Test (H_2CrO_4)
1º (blue – green)
2º (blue – green)
3º (yellow)
Lucas Test
Lucas Test
Lucas Test
No oil
Slow oil
Fast oil

Partner_____

Section (Circle) M Tu W Th Date_____

ALCOHOLS AND PHENOLS – Report Sheet

Please turn in this portion of your lab before your leave today. Please use complete sentences where appropriate.

I. PHYSICAL PROPERTIES OF ALCOHOLS AND PHENOLS

A. Viscosity of Alcohols and Diols

1. Draw the structure of the following.

Glycerin	Ethanol	Ethylene glycol

2. List the above liquids in order of increasing viscosity.

3. Explain the differences in viscosity as a function of hydrogen bonding. Include a diagram of two hydrogen bonded ethanol molecules in your answer. (Show all lone pairs and partial charges on participating atoms. Clearly label the hydrogen bond.)

4. Propylene glycol and butylene glycol are common additives in household products. They often function as moisturizers and humectants. Use what you know about the structure of these compounds to explain how they function to retain moisture. Include the structure of these diols in your answer.

B. Solubility

1. Record your observations here! Include a sketch.

2. The structure of each of the alcohols tested includes a non-polar hydrophobic region and a polar hydrophilic region, a hydroxyl group. Use what you know about molecular structure, polarity, and intermolecular forces to explain your observations above.

3. Cetyl alcohol (1 – hexadecanol or palmityl alcohol), $CH_3(CH_2)_{15}OH$, was first obtained from whale oil. Now it is readily manufactured from plant oils such as palm and coconut oils. Draw the skeletal structure of cetyl alcohol. Circle the hydrophobic portion of the molecule. Explain why this long-chain alcohol is used in hair conditioners/detanglers.

C. pH

Record your data in the table below.

	1 - butanol	2 - butanol	t – butyl alcohol	phenol
pH				

II. CHEMICAL PROPERTIES OF ALCOHOLS AND PHENOLS

A/B. Oxidation and Substitution Test for Alcohols

	Structure	Is this a 1°, 2° or 3° ROH?	Oxidation Test (observations)	Lucas Test (observations)
1 – butanol				
2 – butanol				
t – butyl alcohol				

Write chemical equations representing the oxidation reactions performed in this section. Include the structure of the alcohol and its oxidation product.

Which of the following alcohols with produce a ketone upon oxidation: cyclohexanol, 3 – methyl – 3 – hexanol, methanol? _____

Write the equation(s) for the oxidation reaction(s) of the alcohol(s) identified above. Include the structure of the alcohol and its ketone oxidation product.

Which of the following alcohols with produce an aldehyde upon oxidation: ethanol, cyclopentanol, sec-butyl alcohol? _____

Write the equation(s) for the oxidation reaction(s) of the alcohol(s) identified above. Include structure of the alcohol and its aldehyde oxidation product.

The Lucas Test reaction proceeds via a carbocation intermediate. Can you explain why the reaction with t-butyl alcohol occurs faster than the reaction with 2 – butanol?

C. Fe^{+3} Test for Phenols

Record your results below.

	Ethyl alcohol (pure sample)	Phenol (aq)	Bengay	Clove Oil	Listerine
Observations					

What is the phenol compound found in clove oil? Draw its structure.

Find two phenol compounds found in Listerine. Write their names and draw their structures below.

Write the name and draw the structure of the phenol compound found in Bengay.

Work Check

III. IDENTIFICATION OF AN UNKNOWN

1. Unknown Number _____

Describe the results of your pH test.

List the tests you ran and describe each result.

What functional group does your unknown have? If the unknown is an alcohol, specify what type of alcohol (1°, 2° or 3°)?

2. Unknown Number _____

Describe the results of your pH test.

List the tests you ran and describe each result.

What functional group does your unknown have? If the unknown is an alcohol, specify what type of alcohol (1°, 2° or 3°)?

IV. Alcohols and Phenols in Household Products

Product	Compound	Structure	Function of Compound

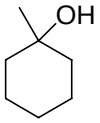
Work Check

Alcohols and Phenols **Name** _____
Pre-lab
Chem 306

1. The following questions are about the chromic acid reagent:
    a. What is its chemical make-up and what is its function in this lab?

    b. Describe the color change involved with the use of this reagent and the chemical basis for the color change.

    c. Which of the following alcohols will react with the chromic acid reagent? _____



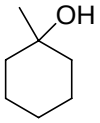
A.

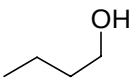
B.

    d. Write a balanced equation for the reaction of the alcohol above that reacts with chromic acid. You can use the reaction in your lab as a guide.

2. The following questions are about the Lucas reagent:
    a. What is its chemical make-up?

    b. Which of the following alcohols will **not** react with the Lucas reagent?_____





3. What reagent is used to test for a phenol? _____

4. A compound is not acidic and forms a blue-green color upon treatment with chromic acid. Does this indicate a primary, secondary, or tertiary alcohol? Describe the test and outcome that would help narrow your conclusion.