Experiment 15

Organic Chemistry

Pre-lab Assignment

Before coming to lab:

- Read the lab thoroughly.
- Answer the pre-lab questions that appear at the end of this lab exercise. The questions should be answered on a separate (new) page of your lab notebook. Be sure to show all work, round answers, and include units on all answers. Background information can be found in Chapter 24, especially sections 24.1- 24.4 in your textbook (Brown and LeMay).
- Follow the guidelines in the "Lab Notebook Policy and Format for Lab Reports" section of the lab manual to complete in your lab notebook the following sections of the report for this lab exercise: Title, Lab Purpose, Procedure and Data Tables.

Purpose

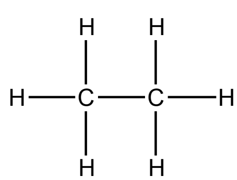
The goal of this lab is to determine become familiar with the the various types of organic compounds and to perform a series of tests to identify two unknown organic compounds.

Background

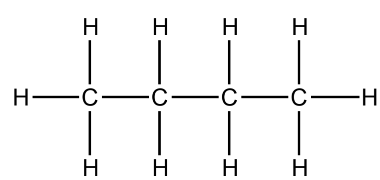
Hydrocarbons

Organic chemistry is the study of carbon compounds. Because this branch of chemistry covers such a large number of compounds, organic compounds are it is broken down into many subdivisions or families. One of these groups of compounds is the hydrocarbons. Hydrocarbons are organic compounds made up of only hydrogen and carbon. In this experiment we will investigate the properties of four families of hydrocarbons.

The first family of hydrocarbons we will study is the alkanes. These are compounds made up of carbons using sp^3 hybridized orbitals only and containing only single bonds. The compounds are saturated with hydrogens and show very few chemical reactions. Examples of alkanes are:

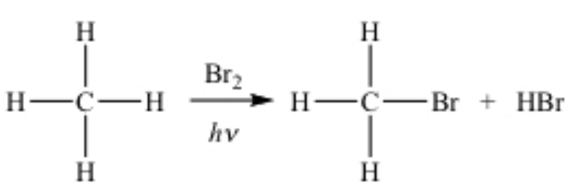


ethane



butane

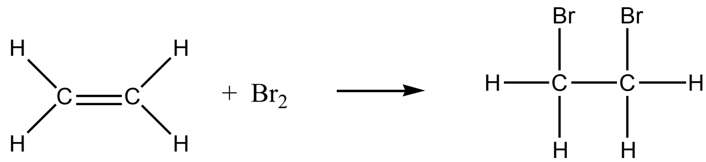
Alkanes burn to form CO_2 and H_2O, as do almost all organic compounds, and undergo free radical **substitution reactions** with Br_2 when exposed to ultraviolet or sunlight. In a substitution reaction, one atom replaces another.



methane

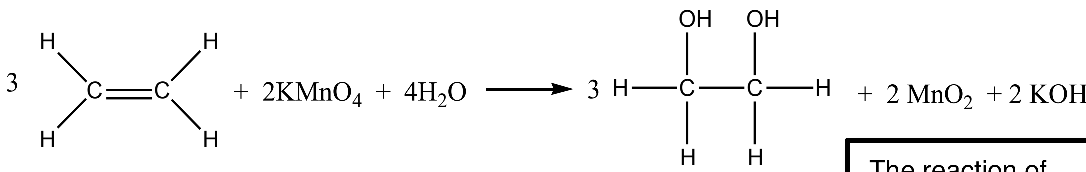
Methane undergoing a substitution reaction with bromine. One of methane's hydrogens is replaced with a bromine.

The second family of hydrocarbons we will study are the alkenes which contain a carbon-carbon double bond. The carbons use sp^2 hybridization on the double bonded carbons. The double bond is very reactive and undergoes rapid reactions called **addition reactions** without the need of sunlight. The reactions cause loss of the double bond and addition of two atoms to these carbons. Two reagents, which react with the double bond, are bromine and potassium permanganate (Baeyer Test).



ethene

The addition of Br_2 to ethene.

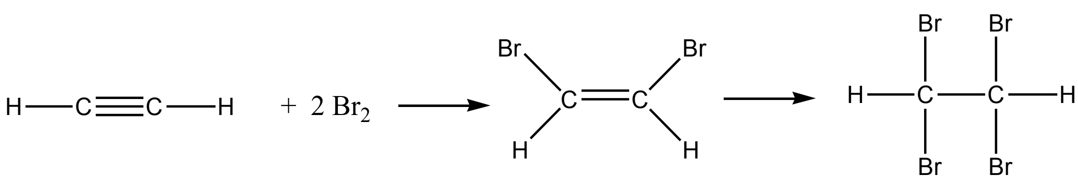


ethene

potassium permanganate

The reaction of $KMnO_4$ with ethene (Baeyer Test).

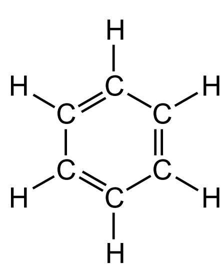
The third family of hydrocarbons we will study are the alkynes which contain a carbon-carbon triple bond. The carbons use sp hybridized orbitals and like the alkenes, undergo rapid bromine addition reactions. The reactions go through two steps:



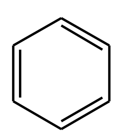
ethyne (acetylene)

The final family of hydrocarbons we will study are the aromatic family. These compounds contain the highly stable benzene ring as a part of their structure.

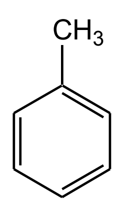
Examples of aromatic compounds are shown below.



benzene



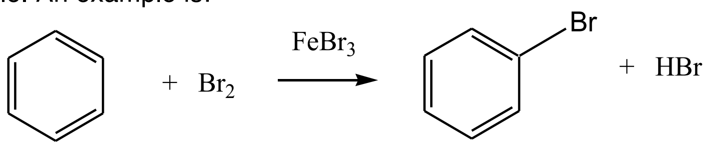
benzene (line drawing)



toluene

All of the carbons in the benzene ring use sp^2 hybridized orbitals.

Aromatic compounds undergo substitution reactions requiring a catalyst and prolonged heating since they are stable. An example is:



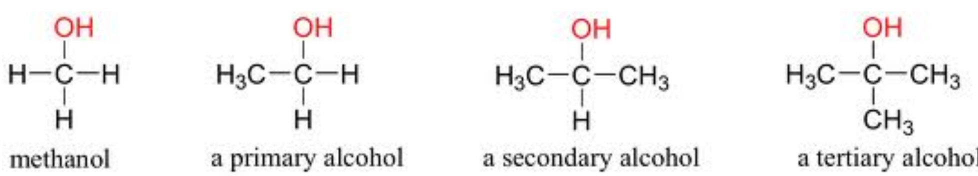
The catalyst $FeBr_3$ is shown above the arrow. Note the second product is HBr gas is formed during substitution unlike an addition reaction where only one product is formed. Most aromatics burn poorly due to the stable benzene ring and give an unusual sooty flame. This is an easy way to test for the aromatic ring.

Functional Groups

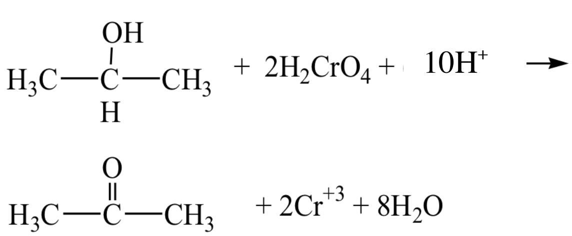
Many organic compounds contant elements besides carbon and hydrogen. We classify these organic compounds into families based on the unique set of atoms that they contain. These unique sets of atoms are called functional groups and they impart special chemical properties to the parent compound. In this experiment, we will study four common functional groups all of which contain not only carbon and hydrogen but also oxygen. They are the alcohol, aldehyde, ketone and carboxylic acid families.

Alcohols

Alcohols contain the functional group "-OH" group attached to a carbon with no other oxygens present. This group is called the hydroxyl group. Alcohols are divided into subgroups based on how many other carbons the carbon directly attached to the -OH is bonded to. Alcohols will be either primary alcohols (if the C with the -OH is bonded to one other carbon), secondary alcohols (if the C with the -OH is bonded to two other carbons, and tertiary alcohols (if the C with the -OH is bonded to three other carbons). Examples are provided below.



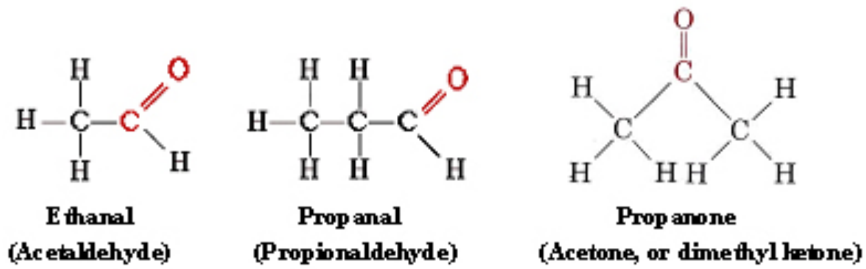
We will test for the presence of an alcohol group by oxidizing it with chromic acid; however, only primary and secondary alcohols can be oxidized in this way.



Aldehydes and Ketones

Both aldehydes and ketones have a carbonyl, C=O group (carbon double bonded to an oxygen). Aldehydes have the carbonyl at the end of a chain while ketones have a carbonyl between carbon atoms in the middle of a chain.

Some examples are provided below.



aldehyde | aldehyde | ketone

We will first test for the presence of a carbonyl group by forming a precipitate with 2,4-D (2,4-dinitrophenylhydrazine). We will then distinguish aldehydes from ketones by using the Tollen's test. The Tollen's test takes advantage of the fact that aldehydes can be oxidized under mild conditions but ketones cannot.

$$H_3C\text{—}\overset{\overset{\displaystyle O}{\|}}{C}\text{—}H \;+\; 2Ag(NH_3)_2OH \longrightarrow$$

$$H_3C\text{—}\overset{\overset{\displaystyle O}{\|}}{C}\text{—}O^-NH_4^+ \quad 2Ag \;+\; NH_3 \;+\; H_2O$$

Oxidation of an aldehyde using the Tollen's test - $Ag(NH_3)_2OH$

A positive Tollen's test will result in silver forming in the solution. This is sometimes called the silver mirror test since the silver will reflect light.

Carboxylic Acids

The last functional group we will investigate is the carboxylic acid, $\overset{\overset{\displaystyle O}{\|}}{C}\text{—}OH$ group. We will react the acid with sodium bicarbonate solution as our test.

$$H_3C\text{—}\overset{\overset{\displaystyle O}{\|}}{C}\text{—}OH \;+\; NaHCO_3 \longrightarrow$$

$$H_3C\text{—}\overset{\overset{\displaystyle O}{\|}}{C}\text{—}O^-Na^+ \;+\; H_2O \;+ CO_{2(g)}$$

The reaction of the carboxylic acid ethanoic acid with sodium bicarbonate. Note that CO_2 gas is produced.

Procedure

Safety: Chromic Acid and Bromine (Br_2) are highly toxic. Be sure to wear goggles, gloves, and a lab apron when using them.
Potassium permanganate stains clothing. Be sure to wear goggles, gloves, and a lab apron when using it.

Waste: Dispose of all waste in the proper containers. There is more than one waste container for this experiment so if you are not sure which one to use, asks your lab instructor.

Part A Identification of a hydrocarbon

1. Reaction with Bromine
a. Dissolve 5 drops of the compound to be tested in 1 ml of dichloromethane.
b. Add to this solution, dropwise, a 2% Br_2/CH_2Cl_2 solution. Mix and observe after each drop, adding a total of 3 drops. A positive result for this test will be a loss of the orange color of the original Br_2 solution.
c. Expose those that do not change color to sunlight for a few minutes.
d. Record your observations for this test in your notebook. Also, **for those known compounds that reacted,** write the balanced chemical equation in your notebook using structural formulas for organic compounds.

Compounds to be tested with Bromine: Hexane, 1-hexene, toluene, unknown

2. Reaction with potassium permanganate (Baeyer Test)
a. To 1 ml of 0.5% potassium permanganate solution, add 5 drops of compound to be tested compound.
b. Shake the test tube well for 1-2 minutes. A positive test is loss of the purple color and formation of a brown precipitate.
c. Record your observations for this test in your notebook. Also, for those known compounds that reacted, write the balanced chemical equation in your notebook using structural formulas for organic compounds.

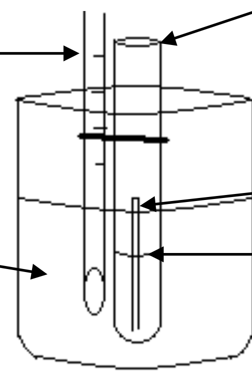
Compounds to be tested with $KMnO_4$**:** Hexane, 1-hexene, toluene, unknown

3. Combustion Test
a. Place 5-10 drops of the reagent in an evaporating dish and ignite with a match. Do not drop the match in the evaporating dish as the burning wood will skew your results.
b. Record your observations for this test in your notebook. Also write the balanced chemical equations in your notebook for the known compounds. You may use the molecular formula for the organic compounds to make the balancing easier. Remember that the product of the combustion of a hydrocarbon is carbon dioxide and water.

Compounds to be combusted: Hexane, 1-hexene, toluene, unknown

4. Boiling Point Determination

a. Fill a 150 ml beaker half full of mineral oil. (Be sure the beaker is **absolutely dry** before using or the oil will splatter).
b. **Securely** suspend a thermometer with a small test tube attached using a rubber band as shown in the drawing.
c. Place 1/2 ml (10 drops) of unknown in the test tube and inset a capillary tube, open end down, into the test tube.
d. Heat the oil slowly while stirring. (The oil should not boil or splatter).
e. When a rapid and continuous stream of bubbles escapes the capillary tube, discontinue heating but continue to stir the liquid. When bubbles cease to escape and just begin to enter the capillary tube, record the temperature, the boiling point of the liquid.
f. Record the boiling point of your unknown in your notebook.



5. Identify your unknown from the following list of hydrocarbons.

Write the name and draw the structure of your unknown in your notebook. Be sure to include your unknown number.

Alkane Boiling Points | **Alkene Boiling Points**

n-pentane	36°	1-pentene	30°
n-hexane	69°	1-hexene	63.5°
cyclohexane	81°	cyclohexene	83°
n-heptane	98°	1-heptene	93°
n-octane	126°	1-octene	122.5°
n-nonane	151°	1-nonene	146°

Aromatic Boiling Points | **Alkynes Boiling Points**

benzene	80°	1-pentyne	40°
toluene	111°	1-hexyne	72°
ethyl benzene	136°	1-heptyne	100°
o-xylene	144°	Mesitylene (1,3,5-trimethylbenzene)	165°

Part B Identification of an alcohol, aldehyde, ketone, or carboxylic acid

1. Chromic Acid Test

a. Dissolve 8 drops of the compound to be tested in 1-mL of acetone.
b. Add 2 drops of chromic acid test reagent and mix gently. A positive test is the appearance of a blue green color within a few seconds. Use extreme caution with the chromic acid.
c. Record your observations for this test in your notebook. Also, for those known compounds that reacted**,** write the balanced chemical equation in your notebook using structural formulas for organic compounds.

Compounds to be tested: acetic acid, ethyl alcohol, 2-butanone and your unknown

2. 2, 4-D test

a. Place 1 drop of the test compound in a small test tube.
b. Add 1 ml of 2, 4-D reagent. Shake the mixture vigorously. A positive result is the formation of a yellow to red precipitate. Most aldehydes and ketones will react immediately. However, some may require up to 15 minutes.
c. Record your observations for this test in your notebook. It is not necessary to write equations for this reaction.

Compounds to be tested: acetone, benzaldehyde, ethyl alcohol, and your unknown.

3. Tollen's Test

a. Tollen's reagent must be prepared immediately before use in a thoroughly cleaned test tube. To do this, mix 1 ml of Tollen's solution A with 1 mL of Tollen's solution B in a large test tube. A precipitate of silver oxide will form. While stirring the mixture, add enough 10 % ammonium hydroxide to the mixture to just dissolve the silver oxide.
b. Dissolve 5 drops of the compound to be tested in a minimum amount of bis(2-ethoxyethyl)ether. Add this solution, a little at a time, to the Tollen's reagent. Shake the solution well. A silver mirror on the test tube or a dark precipitate is a positive test for an aldehyde. (Caution: Some alcohols may also give a positive test.
c. Record your observations for this test in your notebook. Also, for those known compounds that reacted**,** write the balanced chemical equation in your notebook using structural formulas for organic compounds.

Compounds to be tested: acetone, benzaldehyde, ethyl alcohol, and your unknown

4. Sodium Bicarbonate Test

a. Dissolve 3 drops of the compound to be tested in 1 ml of 5% aqueous sodium bicarbonate solution. **Do not shake**. A positive result is the formation of a gas. You will need to look closely at your samples. Add more of the compound you are testing if you are not sure.
b. Record your observations for this test in your notebook. Also, write the balanced chemical equations in your notebook for the compounds that reacted. You may use the molecular formula for the organic compounds to make the balancing easier.

Compounds to be tested: acetic acid, ethyl alcohol and unknown.

5. **Boiling Point Determination**
Determine the boiling point of your unknown as described in part A Step 4.

6. Identify your unknown from the following list.
Write the name and draw the structure of your unknown in your notebook. Be sure to include your unknown number.

Aldehyde Boiling Points

Propanal	48°
2-methylpropanal	64°
butanal	75°
3-methylbutanal	92°
2-butenal	104°
2-ethylbutanal	117°
hexanal	130°
heptanal	153°
benzaldehyde	179°

Ketone Boiling Points

Acetone	56°
2-butanone	80°
2-pentanone	101°
4-methyl-2-pentanone	117°
Cyclopentanone	131°
2-heptanone	151°
2-octanone	173°

Alcohol Boiling Points

methanol	65°
2-propanol	82°
2-butanol	99°
1-butanol	118°
3-methyl -1-butanol	130°
1-hexanol	157°
1-heptanol	176°

Carboxylic Acid Boiling Points

acetic acid	118°
propanoic acid	141°
2-methylpropanoic acid	154°
2-methylpropenoic acid	163°
3-methylbutanoic acid	176°

Pre-Lab Questions

1. How does an alkane differ from an alkene structurally?

2. What two tests are can be used to test for the presence of a double or triple bond (i.e. will result in a positive test while an alkane will have a negative result)?

3. Of the tests in this experiment, what test is the easiest to use to determine if a compound contains an aromatic ring?

4. A compound on burning gives a sooty flame. The boiling point test gives a boiling point of 110±2°. What is the identity of the compound?

5. How does an aldehyde differ from a ketone structurally?

6. An unknown is added to aqueous $NaHCO_3$ solution. No gas is formed. Of the types of compounds discussed in Part B of the lab, which category of compounds can you eliminate as a possibility regarding this unknown?

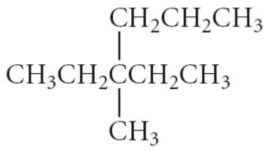
7. How does the alcohol functional group differ from the carboxylic acid functional group structurally?

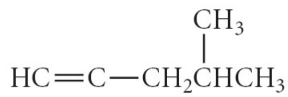
8. An unknown gives a precipitate when 2, 4-D is added but does not react with Tollen's reagent. The boiling point is 116±2°. The unknown is:

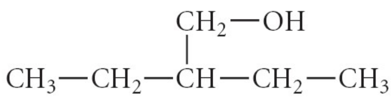
9. Write the balance chemical reaction between propanoic acid and sodum bicarbonate.

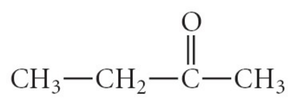
Post-Lab Questions

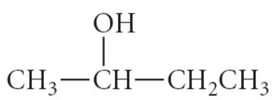
1. Name the following compounds

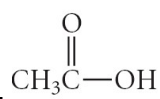
a. 

b. 

c. 

d. 

e. 

f. 

2. Draw the structure of the following compound and classify each as an alkane, alkene, alkyne or aromatic compound:

a. cyclopentane
b. n-hexane
c. 2-heptene
d. 1-pentyne
e. toluene
f. 1,3-diethylbenzene

3. Draw the structure of the following compound and classify each as an alcohol, aldehyde, ketone, or carboxylic acid.

a. 2 –butanol
b. 3-pentanone
c. butanal
d. propanoic acid

4. Now that you have identified your unknowns for Part B and Part C, write the complete balanced equations for any reactions you observed the unknowns undergo in this experiment.

5. Write the balanced chemical equation that represents the addition of Cl_2 to $CH_3CH{=}CH_2$